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LEAKAGE TESTING HANDBOOK

by J. William Marr

Prepared by
GENERAL ELECTRIC
Schenectady, N. Y.
for Jet Propulsion Laboratory

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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NOTICE

This Leakage Testing Handbook S-67-1014 prepared by Dr. J. W. Marr for NASA, is currently issued so that it can be utilized at the earliest possible time by the many agencies and industries concerned with leakage testing and specifications. A separate report covering the characteristics of many commercially available leakage detectors is also available as S-67-1013.

In this edition, errors possibly exist in the text. However, it is hoped that these errors will not detract from the general usefulness of the book.

Corrections and extensions including the more recent advances in the technology of leakage testing will be included in a revised edition of the Handbook, to be published in 1968, under a continuation of Contract NAS 7-396.

Your comments and criticisms of the book, its general format and the handling of the subject matter, are solicited. Also, it is hoped that you will suggest additional topics pertinent to leakage testing which you feel should be included in the revised edition.

The form below is provided for your convenience.

- -

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FOREWORD

This Leakage Testing Handbook was sponsored by the National Aeronautics and Space Administration's Jet Propulsion Laboratory, Pasadena, California under Contract No. NAS 7-396 with the General Electric Company's Missile and Space Division in Philadelphia, Pennsylvania. This work was conducted at the Company's Research and Development Center in Schenectady, New York.

The NASA Project Manager is Mr. F. E. Compitello, Code RPL, Liquid Propulsion Technology, Office of Advanced Research and Technology. The NASA Technical Manager is Mr. R. S. Weiner, Liquid Propulsion Section, Jet Propulsion Laboratory. The author acknowledges their support and cooperation.

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Section 1.0

INTRODUCTION

There is no standard method of measuring leakage. Moreover, there is little agreement among manufacturers of closed systems on specifications. Each manufacturer must develop his own standards and techniques for leakage measurement. Frequently these arbitarily derived specifications are included on equipment, but only a very few military manuals describe specific methods whereby necessary tests can be performed (Ref. 1, 2, 3, 4, and 5).* In a study of available literature on leakage testing it was found that, although there are numerous articles in the literature describing individual tests and pieces of equipment, only one booklet (Ref. 6) exists on the subject.

Currently, specifications for leakage testing are often so ambiquous that the manufacturer himself is not sure of the method or the procedure to follow. Usually the procedures are not well defined and the manufacturer is forced to make assumptions based on meager knowledge in order to obtain the results required in the specifications. Furthermore, the specification writer is not generally familiar with the methods that are available and what sensitivities may reasonably be expected. He is therefore forced to make estimates concerning methods and then attempt to define proofs that must be met by the manufacturer to show that the required sensitivities are in fact obtained.

This situation exists even though there is extensive uncollected information in the literature on leakage phenomena and leak detection methods. This book has been written to unify and consolidate available information on leak testing. It is the intent of the author that this book serve as a ready reference for the engineers who will design the equipment, write the specifications, and perform the leakage tests. The book covers fundamental concepts of leakage testing, leakage phenomena often encountered during testing, and the testing methods which are available.

It is not the purpose of this book to instruct the operator as to the exact steps to be taken during leakage testing. These steps will have to be defined by the engineer in each individual case. Hopefully, with the help of this book, standard specifications and instructions will be written by appropriate committees.

The book is divided into two parts:

- The first part describes the theory of leakage testing.
- The second part describes the individual methods of leakage testing, their limitations, sensitivities, and methods of use.

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* The list of references, cited in the text is located at the end of this book. General reading references are listed at the end of each section.

In addition, the book contains a Bibliography and three Appendices. The Bibliography includes a list of references specifically cited in the text and a list of publications concerning leak testing. Appendix A gives the physical properties of tracer gases and safety precautions necessary in their use. Appendix B is a glossary of terms used in leak testing. Appendix C consists of certain relevant leakage specifications.

The engineer, desiring to be familiar with leak detection, can study the first part of the book for a proper understanding of leakage testing theory; he can select individual chapters of the second part for the various methods which are appropriate for his applications.

1.0.1. SUMMARY

1.0.1.1. PART I

The first section of the book discusses the distinction between leakage measurement and leak location. This is the very important first distinction which must be clearly apprehended before leak testing is initiated.

The second section of the book discusses the various reasons for leak testing. These reasons should be used for setting necessary leakage specifications.

The third section contains a description of the various techniques of using leak testing equipment. The advantages and disadvantages of these techniques are discussed individually, as well as the effect of location of pumps. The units that are used to describe leaks and leakage are also discussed.

Section 4 is a description of the flow characteristics encountered in leaks. This section explains how leakage may be predicted from the values under other conditions.

Section 5 is a description of the various pieces of accessory equipment, manifolding, and pumping which are generally used for leak testing. A description is also given of other testing considerations such as safety, accessibility of the test equipment, and proper testing procedures.

The sixth section describes the standards used in leak testing, their availability, their calibration, and their manufacturers.

The seventh section describes the procedure for writing a leakage specification.

The eighth section of the first part of the book describes a technique for choosing between different procedures to use in leakage testing. This section is the first one which discusses in detail the individual testing methods. It is intended to serve as a guide in choosing the appropriate testing technique.

1.0.1.2. PART II

The second part of the book is a detailed description of the individual testing methods. Each section discusses one method, together with all its modifications. The individual sections are divided into as many as six subsections.

The first subsection discusses principles of the procedure. They describe general characteristics of the testing procedure, its applicability, and its sensitivity. The second subsection describes equipment available for testing, cost, and a list of manufacturers. The third subsection describes the advantages and limitations of the procedures. The principles of operation are described in the fourth subsection. The fifth subsection discusses instrumentation. The sixth subsection describes the testing procedure, the specific equipment involved in testing, and the maintenance necessary to keep it in operation. Finally, a subsection of general references is included. The list includes only those general references which offer good general information on the subject. References cited specifically in the text appear at the back of the book.

The testing procedures described in the second part of the book are only those in common use for leakage testing. New methods, and methods not generally used, are not listed because further development is necessary before they can be considered universally adaptable.

A separate volume entitled "Characteristics and Sources of Commercially Available Leak Detectors," (GE Report No. S-67-1013) has been prepared under the same contract (NAS 7-396). This volume describes in detail equipment currently available for leak detection. The Tables of Characteristics of the commercial leak detectors have been abstracted from this report.

Section 1.1

LEAKAGE CATEGORIES

Leakage testing can be divided into three categories: Leakage measurement, leak location and leakage monitoring. Leakage measurement is the measurement of the total leakage of a system or subsystem. Leakage measurement is the only method which can reliably determine if the leak exists.

Leak location is the procedure of pinpointing the precise location of individual leaks. Theoretically, by pinpointing and measuring the individual leaks, it is possible to obtain the total leakage of the system. However, this method of measuring total leakage is extremely unreliable, since until located, no individual leak is known to exist.

Leakage monitoring is the continuous measurement for the presence of contaminants coming into or out of the system. The major distinction between leakage monitoring and the other two leak testing techniques is that monitoring is usually performed over extremely long periods of time during system storage or operation. Leakage monitoring equipment differs from the equipment used for leak detection in that it is smaller, consumes less power, might have a remote readout and more inherent stability. Leakage monitoring is not described in this book.

It is extremely important that leakage measurement techniques and leak location techniques are not interchanged indiscriminately. In testing a system, the most reliable technique is to measure total leakage and then, if necessary, locate the individual leaks by a leakage location scheme. An attempt to use leakage location for measuring the total system leakage produces results which are unreliable!

Section 1.2.

REASONS FOR LEAKAGE TESTING

Rapid nondestructive methods for detection of leaks in sealed containers are of great industrial and military importance, The probable operational reliability of many devices is greatly reduced if sufficiently large leaks exist.

Leakage testing is performed for three basic reasons:

1. To prevent material loss.

2. To prevent contamination.

3. To detect unreliable components.

Leakage testing is expensive, but adds no tangible value to the product. Therefore, the above three reasons are the only ones for which leakage testing is done. They have to be considered individually so that a rational decision can be made on the sensitivity to be prescribed for any particular testing.

1.2.1. MATERIAL LOSS

The first consideration in specifying the leak tightness of the system is that the system does not leak sufficient material to cause system failure during the operational life of the system. The allowable leakage rate then becomes the allowable total leakage divided by the operational life of the system. Of course conversion might have to be made between the tracer gas leakage during testing and the material leakage during system operation. These conversions are discussed in Section 1.4.

1.2.2. CONTAMINATION

Contamination failure of the system might cause environmental damage, personnel hazard, or degraded appearance.

1.2.2.1. ENVIRONMENTAL DAMAGE

The environmental damage to a system may be caused by material leaking either in or out of the system. For example, system damage may be caused to a liquid rocket motor when the oxidizer leaks out of the storage tank and reacts with parts of the motor. On the other hand, electronic components can fail when air or water vapor enters a sealed protective container. Calculations of the very small amount of material necessary for contamination failure to occur are difficult to make. However, in most cases, such calculations are not impossible if the failure can be defined. For example, if some decision can be made as to the amount of reaction which is allowable between the oxidizer and the rocket engine parts, the amount of total leakage from it and the leakage per unit time can be defined. Similarly, in an electronic component, if failure results from absorption of a monolayer of leaking molecules on the surface, then knowing that 10^{15} molecules form one monolayer on a square centimeter of surface (Ref. 7) makes it possible to calculate the allowable leakage rate for this particular component. If failure results from a pressure rise, then the maximum allowable pressure, the planned system operation time, and the system volume are all that are necessary for calcualation of the allowable leakage rate. Neilson (Ref. 8) has demonstrated a method of calculating the allowable leakage into sealed small electronic components.

1.2.2.2. PERSONNEL HAZARD

Material leakage can cause personnel hazard during system operation. If the tolerable concentrations are known, and these are often reported in literature (Ref. 9), it is again quite easy to calculate the maximum tolerable equipment leakage.

1.2.2.3. APPEARANCE

An appearance specification is a specification for maximum leakage that is made because leakage of a higher value will spoil the appearance of the system. It is often made when no more stringent specification is necessary. A leakage specification of oil out of an oil pan of a new car is a good example. This leakage specification is not caused by concern that too much oil will be lost or that damage to the car motor will occur; rather it is because the prospective buyer would not be inclined to buy a car which would immediately dirty his driveway and which, at the time of the sale, was dropping oil on the showroom floor.

When appearance sets the leakage specification of the system, the leakage is often only a nuisance. However, even leaks which are largely a nuisance may alter the effectiveness of the total system. For example, during the East Coast power blackout on November 9, 1965, a large steam generator failed during shutdown because the auxiliary steam supply used for lubrication was not available. This steam supply had been shutoff earlier by some workers who were bothered by excessive leakage of steam through some valve packing. This steam leakage was not critical, but was enough of a nuisance that the system was shut down for repair. The repair did not take place in time and the bearings of the generator burned during emergency shutdown of the system.

1.2.3. RELIABILITY

The third basic reason for leak testing is to measure the reliability of the tested system. This is not a direct measure of reliability, but might show a fundamental fault of the system by a higher than expected leakage measurement.

A high leakage value might indicate that a gasket is improperly aligned or missing. In the same manner, a high leakage value might show the presence of a misaligned or misthreaded flange. It is therefore possible to detect installation errors by high leakage values. The leakage measurement to detect these errors need not be extremely sensitive, since the leakage expected from serious error will be relatively high (10^{-2} to 10^{-5} atm cc/sec). However, the absence of high leakage does not necessarily indicate the absence of improperly installed components.

Many leaks are caused by material flaws such as cracks and fissures. Some of these flaws can be detected by leakage measurement. Even more can be detected by x-rays. But neither technique will detect all flaws. Leak detection is therefore complementary to other detection techniques in finding basic material flaws.

Since reliability is not a direct function of the leakage in a system, it is difficult to establish a leakage acceptance level. Part of this decision may be influenced by the fact that the added cost of increased sensitivity will detect only a small number of additional leaks. This is because most leaks in welds, brazes, and other joints tend to be relatively large. The only case where small (less than 10^{-7} atm cc/sec) leaks are encountered is in parts that receive special clean room treatment during manufacture. This is partly due to the clogging of leaks by water vapor and liquids which may be present. See Section 1.4.

Occasionally it is desirable to locate every existing leak irrespective of size. The reasons for this are: (1) stress leaks have a habit of growing, i.e., very small leaks today may be troublesome later, and (2) high temperature leaks may be very small at test temperatures.

The cost of the procedure compared to the number of leaks found should be the criterion whereby a decision on whether or not to seek greater reliability is made. For example, a reliability of 10^{-6} atm cc/sec over a reliability of 10^{-5} atm cc/sec may not be justified. The cost of obtaining the slight increase is prohibitive in relation to the value of increase to detection reliability.

The expected leak tightness of the sealing operations which will be used must also be considered. The specification will then have to be written with advice from an experienced engineer who is to make a judgment on the reasonable value of allowable leakage.

1.2.4. SENSITIVITY

In specifying the sensitivity of the leakage procedure, an optimum sensitivity value should be sought. Large deviations from this optimum value increase the cost and the difficulty of performing the measurement. Secondly, any increase in the sensitivity specified for a particular test automatically increases the cost of testing. Therefore, a compromise has to be reached between testing cost and leakage tolerance. Finally, the specification has to be written in a language which is easy to implement in testing.

1.2.4.1. OPTIMUM VALUE AND DIFFICULTY

Each modification of a test procedure has an optimum value at which it is most readily used. Deviation from this optimum value of sensitivity makes it more difficult to perform the measurement and decreases the confidence in the results. Figure 1 is a diagram showing the influence of increasing sensitivity on the ease of operation of the equipment. In most cases, after reaching a plateau, further increase of sensitivity rapidly decreases the ease of operation. Bubble testing by immersion in water is an example of how the optimum value affects the ease of performing the test. This procedure has a sensitivity range between 10^{-1} to 10^{-4} atm cc/sec. In measuring for 10^{-1} atm cc/sec leaks, a component may be placed in water and quickly removed. When immersed, bubbles will evolve from the pressurized component at such a rapid rate that there is no question of the existence of a leak. In checking for leaks in the 10^{-2} to 10^{-3} atm cc/sec range, care must be taken that the immersed component is submerged long enough for any bubbles coming from around crevices to have a chance to collect and rise. In measuring leaks in the 10^{-4} atm cc/sec range, the component, after being immersed, has to be completely stripped of attached air bubbles so that the forming bubbles of leaking gas may be detected. The 10^{-4} atm cc/sec range is near the limit of detectability of this technique, although longer waiting periods theoretically could obtain higher sensitivity. Longer waiting periods are impractical because the rate of bubble evolution approaches the rate at which the gas is dissolving in the testing fluid.

Therefore, requiring a sensitivity greater than 10^{-4} atm cc/sec makes the procedure exceedingly difficult. For instance, this procedure could be used at higher sensitivity by saturating the liquid with the gas. It would then be prudent to change completely the operating procedure to one which is most effective at that higher sensitivity. In the same manner, trying to check leaks greater than 10^{-1} atm cc/sec becomes difficult because of rapid gas evolution and rapid decay of pressure in the system. However, the difficulties on the lower range of sensitivity are usually not so great as in the more stringent sensitivity range.

1.2.4.2. COST CONSIDERATIONS

The cost of leakage testing increases as the required sensitivity increases. Figure 2 illustrates this increase in cost. The investment for a leakage determination of 10^{-3} atm cc/sec is negligible - approximately $5.00 - while the investment for 10^{-12} atm cc/sec is four decades higher. Even after the technique is established, an increase in sensitivity within this technique will result in an increase in measurement cost. This increase is usually caused by the greater complexity with increasing sensitivity. This becomes particularly drastic when the required sensitivity is higher than the sensitivity plateau described in Section 1.2.4.1.

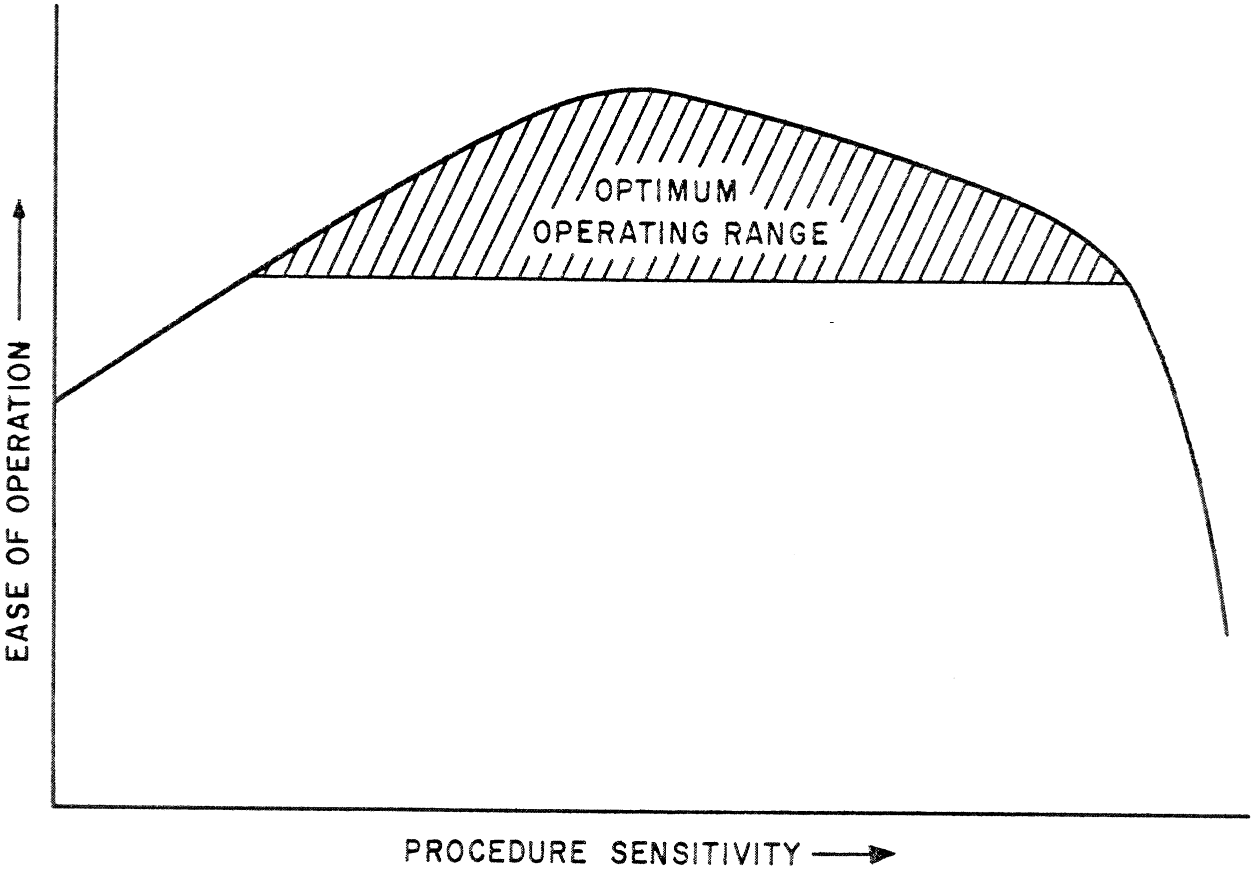

Figure 1. Ease of Operation Versus Procedure Sensitivity.

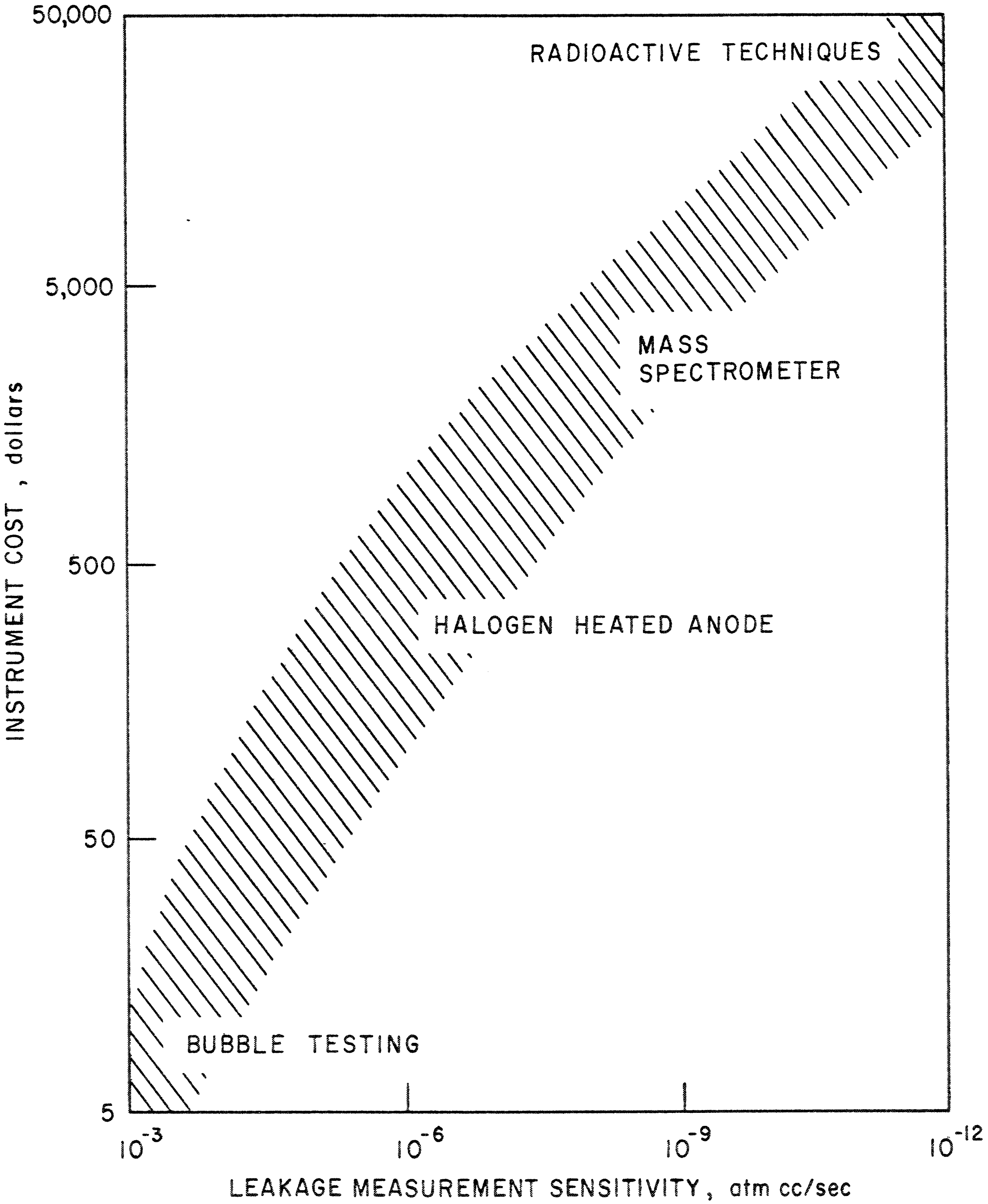

Figure 2. Effect of Required Sensitivity on Leak Detection Equipment Cost. (Reprinted from J.W. Marr, "Leakage Phenomenon", Valve Technology Seminar, Midwest Research Institute, Kansas City Mo., October 1965.

1.2.4.3. ZERO LEAKAGE

Nothing made by man can be considered to be absolutely leak-tight. Even in the absence of minute porosity, the permeation of certain gases through crystals, polymers, and glasses still occurs. Leak tightness is, therefore, a relative item. It is necessary to establish a practical leakage level for any given component under test. Because leak tightness is a relative term and has no absolute meaning, the sensitivity of the equipment involved is a practical guide. A preliminary decision has to be made concerning the definition of leak tightness for the particular situation. Any increase in sensitivity of a technique brings with it an increase in the expenditure of time and, therefore, an increase in cost. This increase in cost reaches a maximum when the specification reads in one of the following ways:

- No detectable leakage
- No measurable leakage
- No leakage
- Zero leakage

Such a specification is not only expensive, but also very confusing unless the method is precisely described. With specifications such as above, the operator is always working against background instrument noise. He is continually forced to make a decision as to whether the reading he is obtaining is caused by the random fluctuations of his instruments or by the actual detection of specific leakage. It is much easier to discriminate whether a measured leakage is above or below a given standard than to discriminate leakage from random instrument noise. It is therefore suggested that zero leakage be defined as a measurable quantitative value that is insignificant in the operation of the system. Such a procedure carries with it the added advantage that the system or the measurement sensitivity can be compared with a standard, and in that way a qualification of the system can be made during the test operation.

The Jet Propulsion Laboratory, for example, requires that certain components have zero liquid leakage. However, they define this term (Ref. 10) by the operating pressure of the system and the maximum measured gas leakage.

Section 1.3

FUNDAMENTALS OF TESTING

Design of any specific leakage test involves various methods of arranging the leakage testing equipment in relation to the system being tested. Other fundamentals that must be considered include the effect of the testing equipment size on the detector response, the units used for leakage measurement, the dimensions of these units, and the conversion of one type of unit to another. Finally, the concept of leak size must be compared with leak testing sensitivity. It is the purpose of this section to discuss these fundamentals of testing without consideration of any specific piece of equipment, since the concepts described are applicable for all equipment.

1.3.1. LEAKAGE UNITS

1.3.1.1. DIMENSIONS OF LEAKAGE UNITS

The units of gas leak rate are

$$\frac{\text{Pressure x Volume}}{\text{Time}}$$

The leak rate is occasionally referred to as mass flow, since at constant temperature these units are proportional to the mass flow rate. At constant temperature, these units describe the number of molecules leaking, regardless of the nature of the gas involved. Since the nature of the leaking gas and the temperature are usually known, it is possible to use the ideal gas formula to determine the actual mass of the leakage.

A wide variety of units for leakage are used. The justification for this diversity of units is the relative ease with which this variety of units can be adapted for the individual engineering problems. In most cases, however, the dimensions of these units remain: pressure multiplied by volume per unit time.

For example, an operator has a gas cylinder with pressure gage calibrated in pounds per square inch. If the gage is read daily, it is convenient for him to express the leakage as a pressure difference multiplied by cylinder volume per time increment. These results come out as psi cubic feet per day. Expressing the leakage as the volume of gas is insufficient, since the volume of gas leaving the cylinder at high pressure is considerably different from the volume of gas entering the atmosphere at lower pressure.

As another example, a leak into a vacuum system has an effect on the system pressure. The amplitude of the effect does not depend on the volume of the gas going in, but on the number of molecules entering the system. The number of molecules depends, however, on the volume of the gas at a particular pressure. A change of pressure directly changes the amount of gas going into the system.

Incorrect dimensions lead to considerable confusion in performing calculations. All equations used in this book involve gas leakage rates in volume multiplied by pressure per unit time; this fact must be kept in mind when verifying the dimensional validity of these equations.

1.3.1.2. CONVENTIONAL LEAKAGE UNITS

In scientific work on leakage, mass flow is usually expressed in torr* liters per second (torr l/sec) or atmosphere cc's per second (atm cc/sec) at 25 degrees C. Atmosphere cc's per second seems to be the more common of the two units in engineering work. However, in scientific work, torr liters per second are preferable for several reasons. First, torr liters per second involves units more closely akin to the metric system which is favored in scientific work. Secondly, pressure is usually expressed in torr rather than atmospheres. Moreover, since many of the leaks produce an effect on pressure in vacuum systems, the calculations may readily be made without use of a conversion factor. A list of units and conversion factors is given in Table 1.

Other conventional usages are as follows:

1. Military standards are written in atmosphere cc's per second.

2. The Atomic Energy Commission writes its standards in standard cc's per second, defining standard cc as the volume of a gas at zero degrees centigrade and one atmosphere pressure.

3. The George C. Marshall Space Flight Center of the National Aeronautics and Space Administration defines leakage in SCIMs. SCIM is standard cubic inch per minute at one atmosphere pressure.

4. The English used lusec as a unit for leakage rate. A lusec is a micron liter per second, a micron being defined as 1/1000 torr. With the emphasis on the use of a torr as a unit of pressure, this unit of leakage rate is slowly being phased out.

5. The American usage of micron liters per second as a unit of leakage is also being phased out with the emphasis on torr as a unit of pressure.

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*A torr is the suggested international standard to replace the English term, millimeters of mercury. A torr is defined as 1/760 of a standard atmosphere and differs by only one part in seven million from the International Standard millimeter of mercury.

Table 1A

LEAKAGE CONVERSION FACTORS (VOLUME)

To convert to Torr Liters/sec	From	Multiply by	Divide by		
To convert from Torr Liters/sec	To	Divide by	Multiply by		
To convert to Atm cc/sec	From			Multiply by	Divide by
To convert from Atm cc/sec	To			Divide by	Multiply by
	Torr Liters/Sec	1.00	1.00	1.32	0.76
	Torr Liters/Min	1.67 x 10^{-2}	60.	2.19 x 10^{-2}	45.6
	Torr Liters/Hour	2.78 x 10^{-4}	3.60 x 10^{3}	3.65 x 10^{-4}	2.74 x 10^{3}
	Torr Liters/Day	1.16 x 10^{-5}	8.64 x 10^{4}	1.52 x 10^{-5}	6.57 x 10^{4}
	Torr Liters/Week	1.65 x 10^{-6}	6.05 x 10^{5}	2.18 x 10^{-6}	4.60 x 10^{5}
	Torr Liters/Year	3.18 x 10^{-8}	3.14 x 10^{7}	4.18 x 10^{-8}	2.39 x 10^{7}
	Atm cc/Sec	0.76	1.32	1.00	1.00
	Atm cc/Min	1.27 x 10^{-2}	78.9	1.67 x 10^{-2}	60.0
	Atm cc/Hour	2.11 x 10^{-4}	4.74 x 10^{3}	2.78 x 10^{-4}	3.60 x 10^{3}
	Atm cc/Day	8.80 x 10^{-6}	1.14 x 10^{5}	1.16 x 10^{-5}	8.64 x 10^{4}
	Atm cc/Week	1.26 x 10^{-6}	7.96 x 10^{5}	1.65 x 10^{-6}	6.05 x 10^{5}
	Atm cc/Year	2.42 x 10^{-8}	4.14 x 10^{7}	3.18 x 10^{-8}	3.14 x 10^{7}
	Micron cc/Sec	1.00 x 10^{-6}	1.00 x 10^{6}	1.32 x 10^{-6}	7.60 x 10^{5}
	Micron cc/Min	1.67 x 10^{-8}	6.00 x 10^{7}	2.19 x 10^{-8}	4.56 x 10^{7}
	Micron cc/Hour	2.78 x 10^{-10}	3.60 x 10^{9}	3.65 x 10^{-10}	2.74 x 10^{9}
	Micron cc/Day	1.16 x 10^{-11}	8.64 x 10^{10}	1.52 x 10^{-11}	6.57 x 10^{10}
	Micron cc/Week	1.65 x 10^{-12}	6.05 x 10^{11}	2.18 x 10^{-12}	4.60 x 10^{11}
	Micron cc/Year	3.18 x 10^{-14}	3.14 x 10^{13}	4.18 x 10^{-14}	2.39 x 10^{13}
	Torr cc/Sec	1.00 x 10^{-3}	1.00 x 10^{3}	1.32 x 10^{-3}	7.60 x 10^{2}
	Torr cc/Min	1.67 x 10^{-5}	6.00 x 10^{4}	2.19 x 10^{-5}	4.56 x 10^{4}
	Torr cc/Hour	2.78 x 10^{-7}	3.60 x 10^{6}	3.65 x 10^{-7}	2.74 x 10^{6}
	Torr cc/Day	1.16 x 10^{-8}	8.64 x 10^{7}	1.52 x 10^{-8}	6.57 x 10^{7}
	Torr cc/Week	1.65 x 10^{-9}	6.05 x 10^{8}	2.18 x 10^{-9}	4.60 x 10^{8}
	Torr cc/Year	3.18 x 10^{-11}	3.14 x 10^{10}	4.18 x 10^{-11}	2.39 x 10^{10}
	Psi cc/Sec	5.17 x 10^{-2}	19.3	6.80 x 10^{-2}	14.7
	Psi cc/Min	8.62 x 10^{-4}	1.16 x 10^{3}	1.13 x 10^{-3}	8.82 x 10^{2}
	Psi cc/Hour	1.44 x 10^{-5}	6.96 x 10^{4}	1.89 x 10^{-5}	5.29 x 10^{4}
	Psi cc/Day	5.99 x 10^{-7}	1.67 x 10^{6}	7.88 x 10^{-7}	1.27 x 10^{6}
	Psi cc/Week	8.55 x 10^{-8}	1.17 x 10^{7}	1.13 x 10^{-7}	8.89 x 10^{6}
	Psi cc/Year	1.64 x 10^{-9}	6.08 x 10^{8}	2.16 x 10^{-9}	4.62 x 10^{8}
	Micron In^3/Sec	1.64 x 10^{-5}	6.10 x 10^{4}	2.16 x 10^{-5}	4.64 x 10^{4}
	Micron In^3/Min	2.73 x 10^{-7}	3.66 x 10^{6}	3.59 x 10^{-7}	2.78 x 10^{6}
	Micron In^3/Hour	4.55 x 10^{-9}	2.20 x 10^{8}	5.99 x 10^{-9}	1.67 x 10^{8}
	Micron In^3/Day	1.90 x 10^{-10}	5.27 x 10^{9}	2.50 x 10^{-10}	4.01 x 10^{9}
	Micron In^3/Week	2.71 x 10^{-11}	3.69 x 10^{10}	3.57 x 10^{-11}	2.80 x 10^{10}
	Micron In^3/Year	5.21 x 10^{-13}	1.92 x 10^{12}	6.86 x 10^{-13}	1.46 x 10^{12}
	Torr In^3/Sec	1.64 x 10^{-2}	61.0	2.16 x 10^{-2}	46.4
	Torr In^3/Min	2.73 x 10^{-4}	3.66 x 10^{3}	3.59 x 10^{-4}	2.78 x 10^{3}
	Torr In^3/Hour	4.55 x 10^{-6}	2.20 x 10^{5}	5.99 x 10^{-6}	1.67 x 10^{5}
	Torr In^3/Day	1.90 x 10^{-7}	5.27 x 10^{6}	2.50 x 10^{-7}	4.01 x 10^{6}
	Torr In^3/Week	2.71 x 10^{-8}	3.69 x 10^{7}	3.57 x 10^{-8}	2.80 x 10^{7}
	Torr In^3/Year	5.21 x 10^{-10}	1.92 x 10^{9}	6.86 x 10^{-10}	1.46 x 10^{9}
	Psi In^3/Sec	0.85	1.18	1.12	0.90
	Psi In^3/Min	1.41 x 10^{-2}	70.8	1.86 x 10^{-2}	53.8
	Psi In^3/Hour	2.35 x 10^{-4}	4.25 x 10^{3}	3.10 x 10^{-4}	3.23 x 10^{3}
	Psi In^3/Day	9.81 x 10^{-6}	1.02 x 10^{5}	1.29 x 10^{-5}	7.75 x 10^{4}
	Psi In^3/Week	1.40 x 10^{-6}	7.14 x 10^{5}	1.84 x 10^{-6}	5.42 x 10^{5}
	Psi In^3/Year	2.69 x 10^{-8}	3.71 x 10^{7}	3.55 x 10^{-8}	2.82 x 10^{7}
	Atm In^3/Sec	12.5	8.03 x 10^{-2}	16.4	6.10 x 10^{-2}
	Atm In^3/Min	2.08	4.82	0.27	3.66
	Atm In^3/Hour	3.46 x 10^{-3}	2.89 x 10^{2}	4.55 x 10^{-3}	2.20 x 10^{2}
	Atm In^3/Day	1.44 x 10^{-4}	6.94 x 10^{3}	1.90 x 10^{-4}	5.27 x 10^{3}
	Atm In^3/Week	2.06 x 10^{-5}	4.86 x 10^{4}	2.71 x 10^{-5}	3.69 x 10^{4}
	Atm In^3/Year	3.96 x 10^{-7}	2.53 x 10^{6}	5.21 x 10^{-7}	1.92 x 10^{6}
	Micron L/Sec	1.00 x 10^{-3}	1.00 x 10^{3}	1.32 x 10^{-3}	7.60 x 10^{2}
	Micron L/Min	1.67 x 10^{-5}	6.00 x 10^{4}	2.19 x 10^{-5}	4.56 x 10^{4}
	Micron L/Hour	2.78 x 10^{-7}	3.60 x 10^{6}	3.65 x 10^{-7}	2.74 x 10^{6}
	Micron L/Day	1.16 x 10^{-8}	8.64 x 10^{7}	1.52 x 10^{-8}	6.57 x 10^{7}
	Micron L/Week	1.65 x 10^{-9}	6.05 x 10^{8}	2.18 x 10^{-9}	4.60 x 10^{8}
	Micron L/Year	3.18 x 10^{-11}	3.14 x 10^{10}	4.18 x 10^{-11}	2.39 x 10^{10}
	Psi L/Sec	51.7	1.93 x 10^{-2}	68.0	1.47 x 10^{-2}
	Psi L/Min	0.86	1.16	1.13	0.88
	Psi L/Hour	1.44 x 10^{-2}	69.6	1.89 x 10^{-2}	52.9
	Psi L/Day	5.99 x 10^{-4}	1.67 x 10^{3}	7.88 x 10^{-4}	1.27 x 10^{3}
	Psi L/Week	8.55 x 10^{-5}	1.17 x 10^{4}	1.13 x 10^{-4}	8.89 x 10^{3}
	Psi L/Year	1.64 x 10^{-6}	6.08 x 10^{5}	2.16 x 10^{-6}	4.62 x 10^{5}
	Atm L/Sec	7.60 x 10^{2}	1.32 x 10^{-3}	1.00 x 10^{3}	1.00 x 10^{-3}
	Atm L/Min	12.7	7.89 x 10^{-2}	16.7	6.00 x 10^{-2}
	Atm L/Hour	0.21	4.74	0.28	3.60
	Atm L/Day	8.80 x 10^{-3}	1.14 x 10^{2}	1.16 x 10^{-2}	86.4
	Atm L/Week	1.26 x 10^{-3}	7.96 x 10^{2}	1.65 x 10^{-3}	6.05 x 10^{2}
	Atm L/Year	2.42 x 10^{-5}	4.14 x 10^{4}	3.18 x 10^{-5}	3.15 x 10^{4}
	Micron Ft^3/Sec	2.83 x 10^{-2}	35.3	3.73 x 10^{-2}	26.8
	Micron Ft^3/Min	4.72 x 10^{-4}	2.12 x 10^{3}	6.21 x 10^{-4}	1.61 x 10^{3}
	Micron Ft^3/Hour	7.87 x 10^{-6}	1.27 x 10^{5}	1.03 x 10^{-5}	9.66 x 10^{4}
	Micron Ft^3/Day	3.28 x 10^{-7}	3.05 x 10^{6}	4.31 x 10^{-7}	2.32 x 10^{6}
	Micron Ft^3/Week	4.68 x 10^{-8}	2.14 x 10^{7}	6.16 x 10^{-8}	1.62 x 10^{7}
	Micron Ft^3/Year	9.00 x 10^{-10}	1.11 x 10^{9}	1.18 x 10^{-9}	8.44 x 10^{8}
	Torr Ft^3/Sec	28.3	3.53 x 10^{-2}	37.3	2.68 x 10^{-2}
	Torr Ft^3/Min	0.47	2.13	0.62	1.61
	Torr Ft^3/Hour	7.87 x 10^{-3}	1.27 x 10^{2}	1.03 x 10^{-2}	96.6
	Torr Ft^3/Day	3.28 x 10^{-4}	3.05 x 10^{3}	4.31 x 10^{-4}	2.32 x 10^{3}
	Torr Ft^3/Week	4.68 x 10^{-5}	2.14 x 10^{4}	6.16 x 10^{-5}	1.62 x 10^{4}
	Torr Ft^3/Year	9.00 x 10^{-7}	1.11 x 10^{6}	1.18 x 10^{-6}	8.44 x 10^{5}
	Psi Ft^3/Sec	1.46 x 10^{3}	6.83 x 10^{-4}	1.93 x 10^{3}	5.19 x 10^{-4}
	Psi Ft^3/Min	24.4	4.10 x 10^{-2}	32.1	3.11 x 10^{-2}
	Psi Ft^3/Hour	0.41	2.46	0.54	1.87
	Psi Ft^3/Day	1.69 x 10^{-2}	59.0	2.23 x 10^{-2}	44.8
	Psi Ft^3/Week	2.42 x 10^{-3}	4.13 x 10^{2}	3.19 x 10^{-3}	3.14 x 10^{2}
	Psi Ft^3/Year	4.66 x 10^{-5}	2.15 x 10^{4}	6.13 x 10^{-5}	1.63 x 10^{4}
	Atm Ft^3/Sec	2.15 x 10^{4}	4.65 x 10^{-5}	2.83 x 10^{4}	3.53 x 10^{-5}
	Atm Ft^3/Min	3.59 x 10^{2}	2.79 x 10^{-3}	4.72 x 10^{2}	2.12 x 10^{-3}
	Atm Ft^3/Hour	5.98	0.17	7.87	0.13
	Atm Ft^3/Day	0.25	4.01	0.33	3.05
	Atm Ft^3/Week	3.56 x 10^{-2}	28.1	4.68 x 10^{-2}	21.4
	Atm Ft^3/Year	6.84 x 10^{-4}	1.46 x 10^{3}	9.00 x 10^{-4}	1.11 x 10^{3}

Examples:

1 Atm cc/Sec = $\frac{\text{Torr Liters/Year}}{4.18 \text{ x } 10\text{-}8}$

1 Torr Liter / Week = Atm cc/Sec x 2.18 x 10^{-6}

Table 1B

LEAKAGE CONVERSION FACTORS (WEIGHT)

Atm cc	1	1.3	3.7×10^{-20}	2.2×10^{4}	2.2×10^{7}	6.4×10^{5}	1.0×10^{7}
Torr Liters	0.76	1	2.8×10^{-20}	1.7×10^{4}	1.7×10^{7}	4.8×10^{5}	7.7×10^{6}
Molecules	2.7×10^{19}	3.5×10^{19}	1	6.0×10^{23}	6.0×10^{26}	1.7×10^{25}	2.7×10^{26}
Gram-moles	4.5×10^{-5}	5.9×10^{-5}	1.7×10^{-24}	1	10^{3}	28	4.5×10^{2}
Kilogram-moles	4.5×10^{-8}	5.9×10^{-8}	1.7×10^{-27}	10^{-3}	1	2.8×10^{-2}	0.45
Oz-moles	1.6×10^{-6}	2.1×10^{-6}	5.9×10^{-26}	3.5×10^{-2}	35	1	16
Lb-moles	1.0×10^{-7}	1.3×10^{-7}	3.7×10^{-27}	2.2×10^{-3}	2.2	6.3×10^{-2}	1

The weight leakages are stated in weight-mole units, i.e. the number of moles of the material expressed in the stated weight units. To obtain the weight of material, multiply the weight-mole by the molecular weight of the material.

The conversions between volume and weight or molecular numbers are made for a gas temperature of 0°C. To convert volume to a weight at any other temperature, multiply result of calculation at 0°C by $\frac{273}{273 + T}$. Where T is the temperature in degrees centigrade.

Example:
Convert 5×10^{-4} atm cc of air at 100°C to grams:

$(5 \times 10^{-4}) \times (4.5 \times 10^{-5}) \times 29. \qquad \times (273/373) = 4.78 \times 10^{-7}$ GRAMS

$(\text{ATM CC}) \times \frac{(\text{GRAMS-MOLES})}{(\text{ATM CC})} \times (\text{MOLECULAR WT.}) \times (\text{TEMPERATURE CORRECTION}) = (\text{GRAMS})$

6. Refrigeration servicemen define their leakage specifications in ounces per year of refrigerant. This standard is rational, since refrigeration systems are charged with several ounces of refrigerant and the systems must operate for years. A leakage of an ounce per year of refrigerant is equivalent to a helium leakage of 1.8×10^{-4} atm cc/sec.

Attempts have been made to initiate a clear, concise and exact form of describing leaks for use in specifications and other documents (Ref. 11). Roberts (Ref. 12) suggested a nomenclature which eliminates the exponent and units. For example:

2-8G 50-15/2

Here the various parts of the specification are covered as follows:

2-8	Maximum leak size permitted is 2×10^{-8} atm cc/sec
G	Gas leakage (for liquid leakage: L followed by viscosity in centistokes. Example: "L (80)," 80 being viscosity of light oil at 20°C)
50-15	High-side pressure, psia, and low-side pressure, psia
/2	Maximum accuracy factor of detection method. Here it is required that the demonstrable accuracy of the leak detector -- leak tracer -- leak standard system fall within the range of 1×10^{-8} to 4×10^{-8} atm cc/sec.

1.3.2. LEAK CONDUCTANCE

In vacuum technology, a leak is a hole or porosity in the wall of an enclosure capable of passing gas from one side of the wall to the other under pressure or concentration differential existing across the wall (Ref. 13).

A leak is not usually indicated by specific physical dimensions. Rather, it represents a physical hole of a certain length and width. However, since a leak is not intentionally manufactured into an item, its dimensions are generally unknown. In leak detection the dimension used to describe the leak is the leak rate.

Although the flow characteristics of a leak may be expressed in many ways, the most common is in terms of conductance of the leak. Conductance is defined by:

$$C = \frac{Q}{P_1 - P_2} \qquad \text{Equation 1.3-1}$$

where C = conductance
Q = leakage
$P_1 - P_2$ = pressure drop across the leak

Conductance has the dimensions of volume/unit time and may be thought of as the volume of gas that enters the leak/second. Conductance is the flow rate-pressure x volume/unit time divided by pressure drop. Unfortunately, the conductance of a leak in laminar flow (Section 1.4) is pressure dependent, so the above equation is not wholly accurate.

The leak rate (Q) in Equation 1.3-1 depends on the pressure difference. The higher the pressure difference, the greater the leak rate through a given leak. Therefore, leakage measurements of the same leak under differing pressure conditions give varying values of flow. Since conductance (C) is defined by both leak rate (Q) and the pressure differential (Equation 1.3-1), conductance or leak rate at a given pressure should be the unit specified in describing a leak size.

1.3.3 DETECTOR SENSITIVITY

A detector's sensitivity is a measure of the concentration of flow rate of tracer gas which gives a minimum measurable signal. This depends on a minimum number of molecules entering the detector. Sensitivity of a detector is not dependent on the pressure in the system being tested. Increase in system pressure increases the flow of tracer gas through a particular sized leak; this, in turn, increases the flow rate into the detector. This increases the response of the detector for that particular sized leak. Thus, an increase in system pressure enables location of holes of smaller dimensions without changing the fundamental instrument sensitivity.

1.3.4. LEAKAGE TESTING TECHNIQUES

Leakage measurement gives the value of total leakage from the whole system being measured. It is the only technique which provides a reliable indication of whether a leak exists or not. Leakage measurement techniques do not provide information concerning the location of the leak, but simply of its existence. Leak location techniques are described in Section 1.3.4.2. However, leak location techniques do not give reliable information concerning the existence of a leak. Without assurance that the leaks exist, leak location techniques become arbitrary, since the operator will have a tendency to overlook unexpected or small leaks. The presence of leakage by a measurement technique should always be ascertained prior to use of leak location techniques.

1.3.4.1. LEAKAGE MEASUREMENT

The requirements for a leakage measurement technique are that gas enclose the whole suspected leak area and the detector measure the tracer gas concentration on the opposite side.

Leakage measurement techniques fall into two major classes: static and dynamic testing. The difference is that the total amount of gas coming from the system passes through the detector in the dynamic method and only a small fraction of the gas around the system is measured in the static method. With either one of these techniques a modification entitled back-pressurizing may be used. Back-pressurizing is a method of testing the leakage from completely sealed systems.

Leakage measurement consists of placing tracer gas in or around the whole system area being tested. A pressure differential is established across the system boundary by either pressurizing the tracer gas or evacuating the other side. The tracer gas concentration on the lower pressure side of the system is measured.

It should not make any difference whether the leakage measurement technique uses a tracer gas envelope, where the system is evacuated and tracer gas placed around it, or pressurized system testing, where the system is pressurized and placed in an evacuated vessel. Both methods are illustrated in Figure 3.

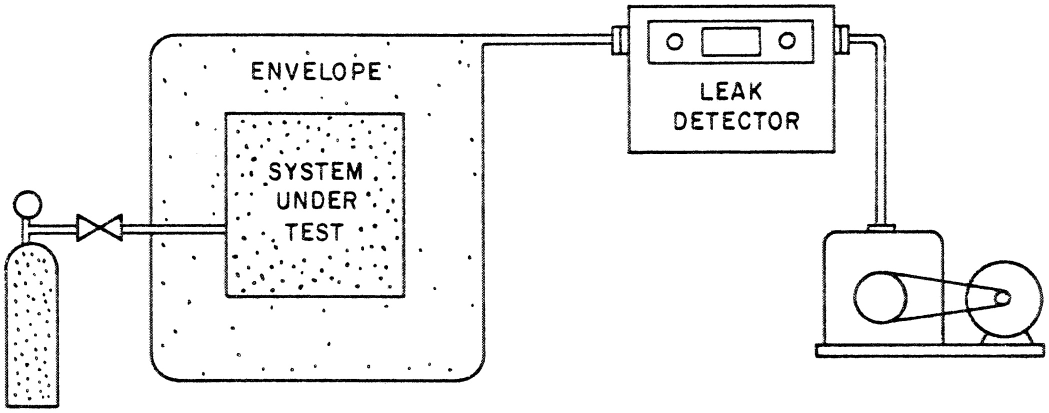
The test sensitivity should be the same, providing that the pressure differential in either case remains the same, and the concentration of the tracer gas has not been changed. The need to choose between one of the above two methods might be based on the characteristics of the leaks in one direction as opposed to another. These leak characteristics are described in Section 1.4.4. On the other hand, the choice might be due to the ability of the system to withstand pressure in only one direction, or on the ease with which tracer gas may be applied to one side of the system.

The factors which affect the sensitivity and response of a testing technique are:

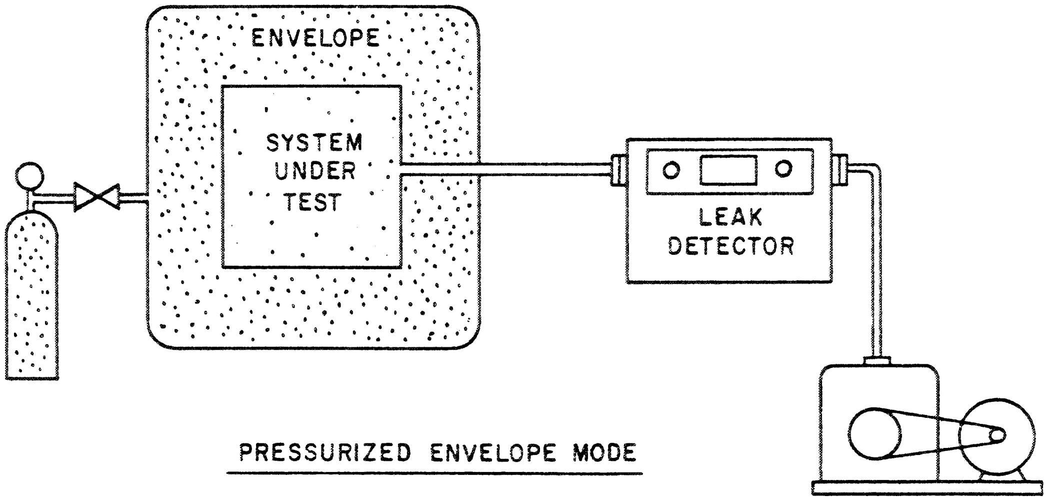
1. The physical properties of the search gas which govern the rate at which the gas enters the leak. This is discussed in Section 1.4.
2. The speed at which the gas is removed by the pump or the combination of pumps.
3. The sensitivity of the detector for the particular gas used.
4. The volume under test.
5. The position of the sensing element of the detector in the pumping system.

1.3.4.1.1. Static Testing

In static testing, the device to be tested is pressurized with the tracer gas and placed in the leak-tight chamber. The concentration of tracer gas in this chamber is then monitored as a function of time. Depending on the instrument being used for monitoring, this leak-tight chamber can either be evacuated or remain at atmospheric pressure. The important characteristic which defines static testing is that the concentration of the



PRESSURIZED SYSTEM MODE



PRESSURIZED ENVELOPE MODE

Figure 3. Leakage Measurement Modes using the Dynamic Testing Technique.

tracer gas increases in the test chamber during the testing period. In general, static testing is more sensitive than dynamic testing since the sensitivity of a static test may be increased by increasing the time of testing or accumulation time. Static testing for this reason is occasionally called "accumulation testing."

The basic equation for leakage measured by static testing is:

$$Q = V \frac{dP}{dt} \qquad \text{Equation 1.3-2}$$

where Q = leakage
V = volume of system collecting the leaking gas
P = tracer gas pressure
t = accumulation time (time of test duration)

Occasionally, the static technique is employed using an instrument which is calibrated in leakage rate, but which employs a probe for use in the dynamic technique. In such a case the device to be tested is placed in a leak-tight chamber and the tracer gas allowed to accumulate for a period of time. After accumulation, the dynamic probe is placed in the test chamber and the leakage rate noted. The following proportionality results:

$$Q \propto \frac{V}{t} Q_i \qquad \text{Equation 1.3-3}$$

where Q_i is the indicated leakage rate; and it is clear that for a given indicated leakage, the actual leakage is inversely proportional to accumulation time.

The static method of leakage measurement is useful under the following conditions:

1. Where a reading of total leakage is wanted.
2. Where the leak to be measured is smaller than the maximum sensitivity of the leak detector in the dynamic mode.
3. Where the background contamination is severe, making it difficult to stabilize the leak detector for ordinary dynamic testing.
4. Where there is a leak area or a number of possible leak points that are not feasible or economical to check individually.

In the use of this method of leakage measurement, the accumulation chamber should be reasonably tight to prevent any losses, including losses from permeation during the accumulation time. For this reason, if a high concentration of tracer gas is expected, the accumulation chamber should be made of nonpermeable material. In all cases, it is advisable to use a standard to compensate for the permeation losses. If the accumulation chamber is an odd shape, some means should be provided to circulate the air within the chamber. This will tend to prevent uneven concentration. Circulation is also necessary when the chamber is of a large volume or when high-density gas that tends to stratify is being used.

1.3.4.1.2. Dynamic Testing

In dynamic testing, the system being tested is continually being pumped. The tracer gas flowing into the pump passes through a detector section where its concentration is measured. When equilibrium is established, the equation for the response of the detector under such condition is:

$$Q = PS \qquad \text{Equation 1.3-4}$$

where Q = leakage of system
P = pressure of the tracer gas
S = pumping speed of system

The dynamic technique of leakage measurement is much more rapid than the static technique. The response time of the system is dependent on the speed at which the tracer gas reaches the detector and the response time of the detector. It is the ideal method of measuring leakage as a function of time, or as a function of other parameters where leakage changes occur during tests. Unfortunately, the sensitivity of the dynamic method is usually less than that of the static one.

1.3.4.1.2.1. Magnitude of Response

The magnitude of the response in the dynamic method is dependent on the sensitivity of the detector to the tracer gas used, and the speed at which the gas is removed by the pump. In most cases, where the tracer gas partial pressure is converted by the detector to an electric current, Equation 1.3-4 becomes:

$$Q = S_d K a \qquad \text{Equation 1.3-5}$$

where Q = leakage rate
S_d = pumping speed of the detector section of the instrument
K = amplification factor, which, when multiplied by detector current, gives tracer gas partial pressure
a = detector current

In order to increase the pressure reading Ka (and thus the instrument sensitivity), it is possible to alter one of two parameters. First, K may be increased up to a limit generally imposed by electronic instrument noise. Alternatively, it is possible to increase the tracer partial pressure within the system by decreasing the pumping speed. Thus, the sensitivity of the technique increase directly with the decrease of pumping speed. Unfortunately, this increase in sensitivity brings with it a decrease in the speec of response.

There are times, however, when it becomes necessary to add additional pumps to the evacuation system being used for testing. This is particularly the case when the detector is pressure-sensitive. The additional pumps are therefore necessary to remove gases resulting from outgassing during the leakage measurement procedure. These additional pumps (Figure 4) increase the total pumping speed of the system and in that way reduce the partial pressure of the tracer gas. These additional pumps, although necessary, reduce the sensitivity of the dynamic leakage measurement technique. The amount of reduction is directly proportional to the increase in pumping speed of the system. It should be pointed out that this increase in pumping speed is not the total pumping speed, but the apparent speed due to the conductance losses, as it appears in the detector section of the system.

1.3.4.1.2.2. System Response Time

Although the pumping speed of a dynamic leak detector determines the magnitude of the response at equilibrium (Equation 1.3-4), several other factors influence the system response time. Volume is the most important of these factors. The complete subject is discussed in detail in several papers (Ref. 14, 15, 16, and 17). The discussion here shows the fundamental points involved.

When tracer gas is introduced into a system through a leak at rate Q, the rate of tracer pressure buildup in the system is:

$$\frac{dP}{dt} = \frac{Q}{V} \qquad \text{Equation 1.3-2}$$

where $\frac{dP}{dt}$ = change in tracer gas partial pressure with time
Q = leakage rate
V = system volume

However, if the system is kept evacuated during tracer gas introduction, i.e., tracer gas is continually pumped out, a mass balance gives:

$$V\frac{dP}{dt} = Q - PS \qquad \text{Equation 1.3-6}$$

where P = tracer gas pressure
S = pumping speed at system outlet

When integrated, the solution to this equation is(for the case of tracer gas pressure equal to zero initially):

$$-\frac{V}{S}\ln\left(1 - \frac{PS}{Q}\right) = t \qquad \text{Equation 1.3-7}$$

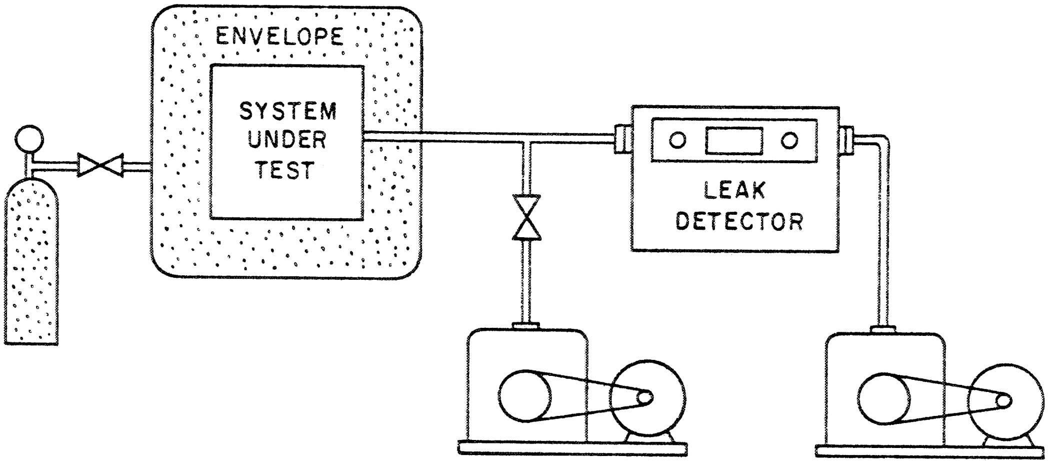


Figure 4. Leakage Measurement Involving the Use of an Auxiliary Pump.

or

$$P = \frac{Q}{S} \left(1 - \exp \frac{-St}{V}\right) \qquad \text{Equation 1.3-8}$$

At long exposure time, the exponential term approaches zero and the equation reduces to:

$$P = \frac{Q}{S} \qquad \text{Equation 1.3-4}$$

That means, at equilibrium, the tracer gas pressure is dependent only on leak size and system pumping speed.

Difficulty arises when the time required for reaching equilibrium is considered. For example, a leak detector may be pumping on a 10-liter system with a one-liter-per-second pump. The response of such a system is plotted as Curve 1 on Figure 5. In ten seconds, the detector responds with 63 percent of the ultimate signal. The equilibrium signal is attained in approximately one minute.

If the system to be pumped is 100 liters, the response time is considerably slower. As seen on Curve 2 of Figure 5, in 10 seconds only about 10 percent of the total response is observed. The equilibrium value is not reached for 10 minutes.

Such a response time might not be acceptable for leakage measurement, and is of dubious value for leak location. It is possible to decrease the response time by increasing the pumping speed of the system. In Curve 3 of Figure 5, the pumping speed for the 100-liter volume was increased from 1 liter/second to 5 liters/second. This, however, decreases the magnitude of the signal. It may be observed that in this particular case the response rate for Curves 2 and 3 are similar at the beginning of the test.

The necessity for the extra pumping speed is also seen during evacuation. When the tracer gas is removed from the leak, the decay of tracer gas in the system is proportional to the system pumping speed and tracer gas pressure:

$$\frac{dP}{dt} = \frac{PS}{V} \qquad \text{Equation 1.3-9}$$

Integrating the above equation, the tracer gas partial pressure decay with time is:

$$t = \frac{V}{S} \ln \frac{P_t}{P_o} \qquad \text{Equation 1.3-10}$$

where t = evacuation time
P_o = original tracer gas partial pressure
P_t = tracer gas partial pressure at time t

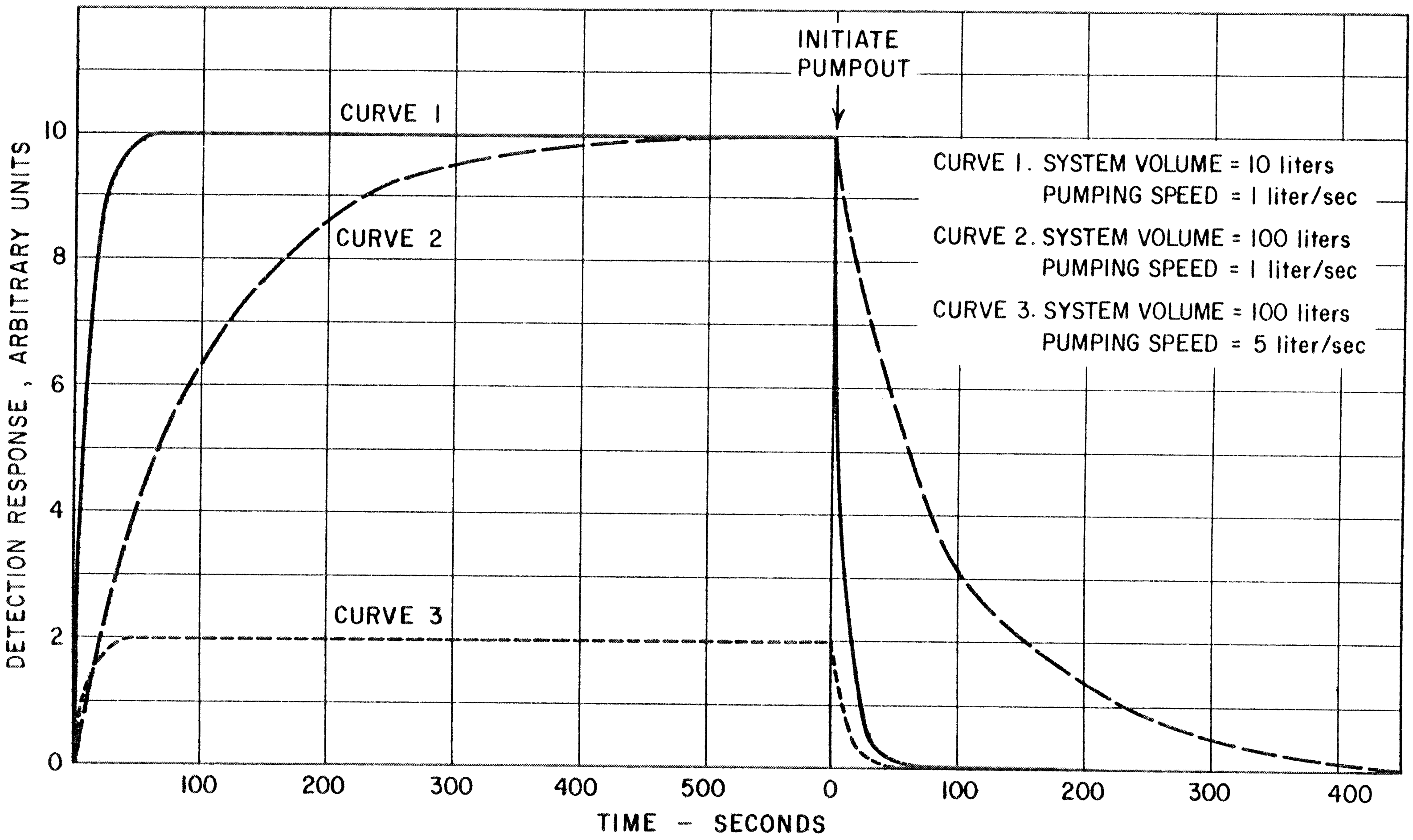


Figure 5. System Response Time.

This may be expressed in the form

$$\frac{P_t}{P_o} = \exp \frac{-St}{V} \qquad \text{Equation 1.3-11}$$

Another way of expressing system performance is by means of system response time and clean-up time. Response time is the time required for the system to yield an output equal to $1-\frac{1}{e}$ of the maximum or equilibrium signal. This is the value attained when in Equation 1.3-8.

$$\frac{V}{S} = t \qquad \text{Equation 1.3-12}$$

The numerical value of $1-\frac{1}{e}$ is 0.63.

Clean-up time is the time required, when no tracer gas is present, for the leak detector output to be reduced to 1/e (or 37 percent) of its initial value, or when the value of the exponent in Equation 1.3-11 reaches unity.

It may be seen on Curves 1 and 3 of Figure 5 that the tracer gas is pumped rapidly from the system once it is removed from the vicinity of the leak. However, in Curve 2, the gas remains for a considerable period.

If the requirements of short response and clean-up times are not met, the leak-testing process is delayed to a large and sometimes intolerable extent. As an example, consider the case of a section of weld being probed at a constant rate. If the response is poor, the leakage indication will appear some time after the probe has moved well beyond the leak, and the tester will then have to backtrack the probe slowly until a second signal is obtained. However, this second signal cannot be observed distinctly until the first signal has been removed, or "cleaned up." Therefore, the clean-up time is equally as important as the response time.

Although the leak size varies in practice, its value does not affect the shape of the pressure versus time curve. The important factor in determining the shape of these curves is the ratio S/V. The reciprocal quantity V/S, having the dimensions of time, is generally called the time constant of the system.

Figure 5 shows that greater time constants result in greater indicated pressure changes during the probing time. On the other hand, a smaller time constant gives a more rapid removal of search gas from the system when probing has ceased.

Once the time constant $\frac{V}{S}$ (volume/pumping speed) of the system has been calculated, and the equilibrium response is known, it is possible to construct the response curves similar to those shown in Figure 5. Table 2 lists values which can be used to construct response curves for any system.

Table 2

DETECTOR RESPONSE TIMES

	Detector Response	Detector Cleaning
Fraction of Time Constant	Fraction of Ultimate Signal	Fraction of Starting Signal
0.001	0.0009	0.999
0.002	0.0019	0.998
0.003	0.0029	0.997
0.004	0.0039	0.996
0.005	0.0049	0.995
0.006	0.0059	0.994
0.007	0.0069	0.993
0.008	0.0079	0.992
0.01	0.0099	0.990
0.02	0.0198	0.980
0.03	0.0296	0.970
0.04	0.0392	0.961
0.05	0.0488	0.951
0.06	0.0582	0.942
0.07	0.0676	0.932
0.08	0.0769	0.923
0.09	0.0861	0.914
0.1	0.095	0.905
0.2	0.181	0.819
0.3	0.259	0.741
0.4	0.329	0.670
0.5	0.393	0.607
0.6	0.451	0.549
0.7	0.503	0.496
0.8	0.550	0.449
0.9	0.593	0.407
1	0.6321	0.3679
2	0.8647	0.1353
3	0.9502	0.0498
4	0.9816	0.0183
5	0.9932	0.0067
6	0.9975	0.0025
7	0.9991	0.0009
8	0.9997	0.0003
9	0.9999	0.0001
10	1.0000	4×10^{-5}

1.3.4.1.2.3. Detector Location

In recent years, many papers dealing with dynamic leakage measurement have been published. To increase the sensitivity of this method, it is often suggested that the detector be connected between the diffusion pump and the fore pump (see Figure 6.) While some authors conclude that such an arrangement does not show any improvement, (Ref. 14 and 15), or that an improvement may be obtained under certain conditions (Ref. 16), others assert that, by this procedure, the change in probe pressure in the fore-pressure line can be considerably greater than in the test chamber (Ref. 17, 18 and 19). These differences in interpretation form the basis for extensive discussions in current technical periodicals. (Ref. 15, 20, 21, 22, 23 and 24).

D. J. Santeler (Ref. 25) summarizes these discussions by stating that whether or not an advantage is gained depends on what factor is limiting the ultimate sensitivity of the test. In a clean system, the ultimate sensitivity may be limited by the partial pressure sensitivity of the detector. In this event, the pressure amplification obtained on the forepressure side will result in a sensitivity gain. In a contaminated system, or when searching for extremely small leaks in the presence of large leaks, the sensitivity is frequently limited by the resolution of the detector in distinguishing tracer gas partial pressure from the background. In this event, forepressure leak detection results in amplification of both tracer signal and background, and unless selective pumping means are employed, no gain in the concentration ratio is realized.

If the leak detector is connected on the forepump side of the diffusion pump, the forepump removes gas from the system by a batch process. The pressure of its inlet side tends to fluctuate. These fluctuations would contribute to the noise level of a sensitive detecting element which is directly connected with the mechanical pump. By placing the detecting element between two diffusion pumps, this effect is greatly reduced. An alternative approach is to place a ballast tank or a throttling valve between the pumps and the detector.(See Figure 6)

1.3.4.2. LEAK LOCATION TECHNIQUES

There are two distinct techniques of locating leaks with instruments: the use of a tracer probe and the use of a detector probe (Figure 7). In the tracer probe technique, gas from one side of the system is pumped past the detector. A stream of tracer gas is being played on the suspected leak area.

In the detector probe method, tracer gas blankets one side of the system being tested. The detector probe is then passed over the suspected area to determine if tracer is coming through. In both detector and tracer probe techniques, the response equations are those of dynamic testing. The choice between the two techniques depends on several factors.

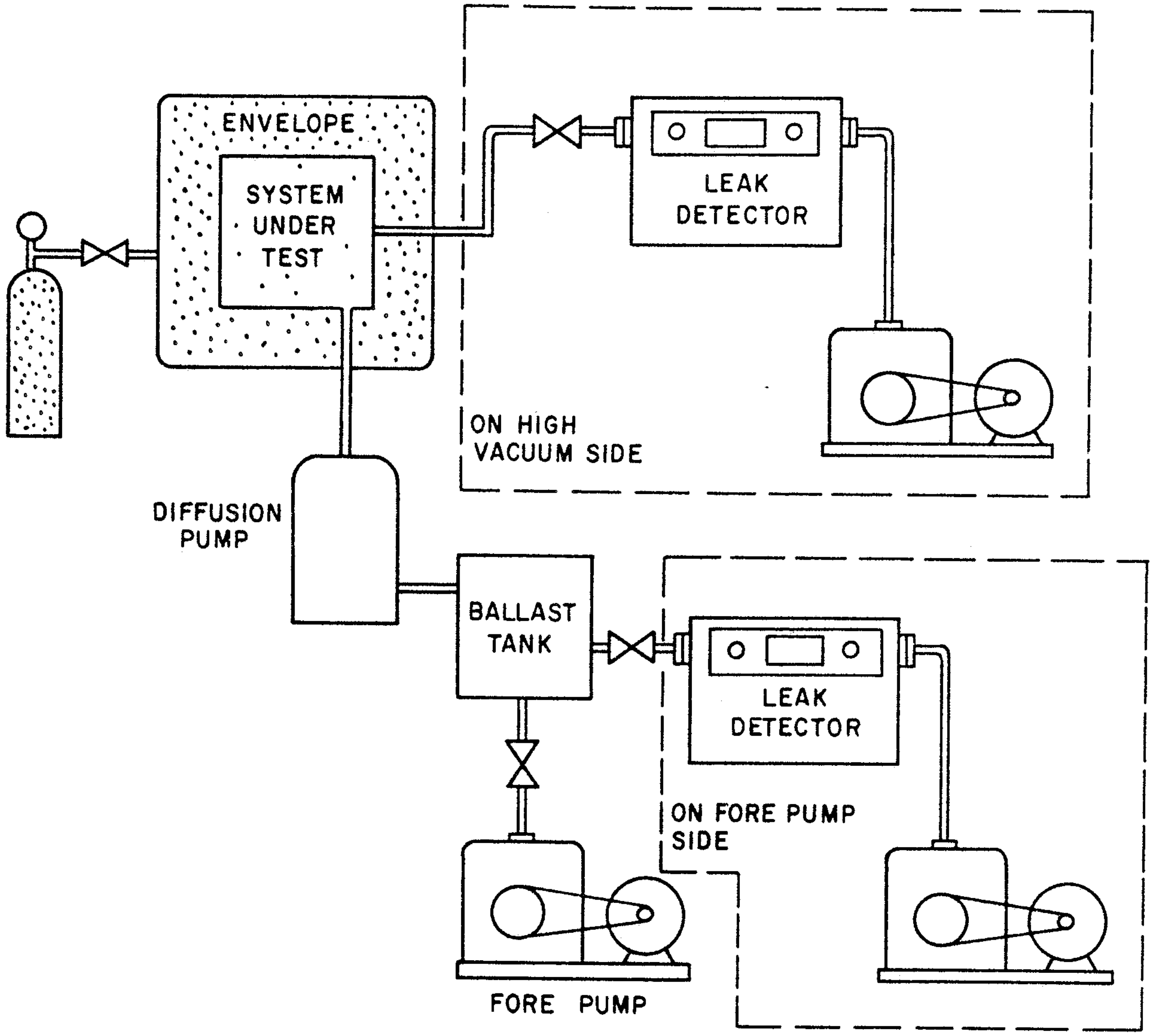
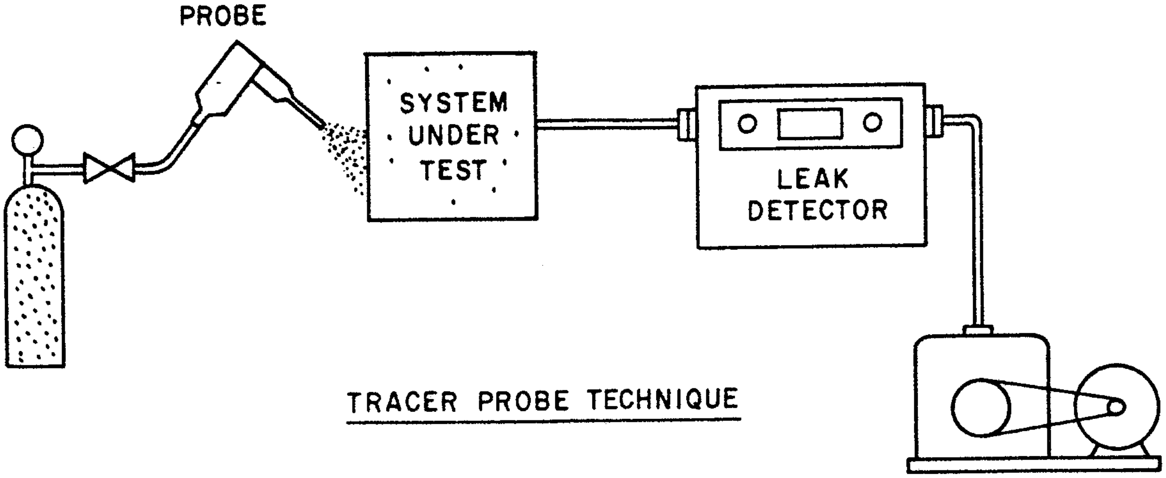
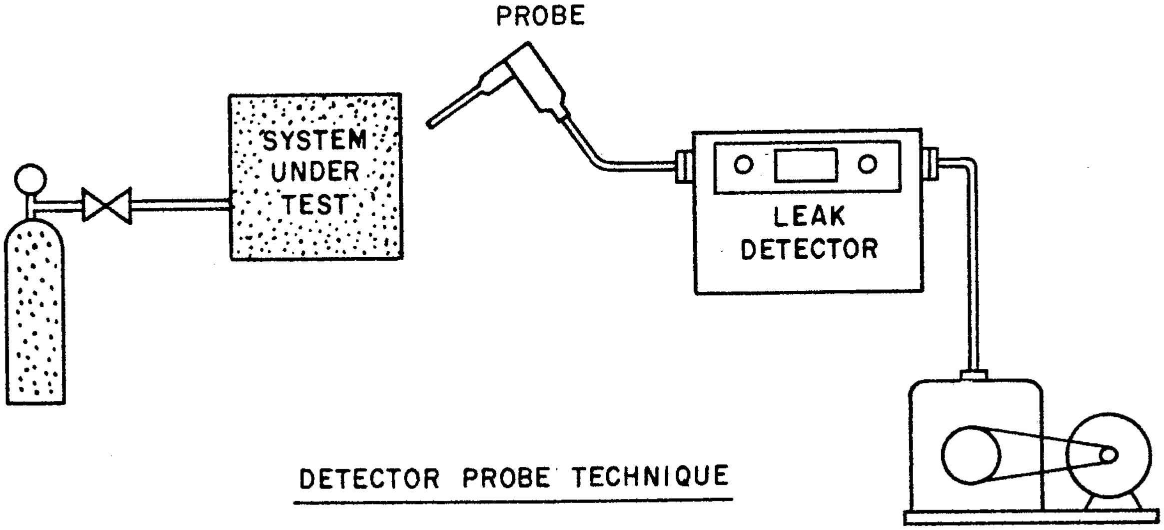


Figure 6. Alternate Sites for Leak Detector on Vacuum System.



TRACER PROBE TECHNIQUE



DETECTOR PROBE TECHNIQUE

Figure 7. Leak Location Techniques.

The detector probe techniques should be used if:

1. The system has to be tested in a pressurized condition.
2. The tracer gas is one which may readily be absorbed on the leak surfaces. If a tracer probe is used the gas will be absorbed in the leak and small leaks will go unnoticed because an equilibrium is not established between the leak surfaces and the tracer gas. See Section 1.4.4.4. for details.
3. The detector has a sensing element that may be operated under atmospheric pressure.

The tracer probe technique should be used if:

1. The detector sensing head has to be evacuated for use. When an evacuated detector probe is used, not much gas can be brought into the sensing element.
2. Leak location is performed after dynamic leakage measurement. It is possible to remove the gas enclosure and, after the tracer gas has been pumped out of the system, use a tracer probe to locate the individual leaks.

Other factors may dictate choice of one technique over the other:

1. It may be desirable to check in one specific direction because of the effect of check valve leaks. See Section 1.4.4.1.
2. The system might have long, narrow passages. If so, it is preferable to pressurize rather than evacuate them, since the rate of travel of molecules in evacuated passages is slow.

1.3.4.3. SEALED UNIT TESTING

Two methods of testing sealed units are:

1. Fill with tracer gas under pressure during manufacture. Test for gas leaks.
2. Seal unit, subject to high-pressure tracer gas, and remove tracer gas from outside envelope. Test for gas leaking from sealed unit.

1.3.4.3.1. Unit Sealed With Tracer Gas

For the system where the unit is filled with tracer gas during manufacture, the effect of leak size on detector signal may be represented in the generalized graph of Figure 8. Here the leak rate, derived from the signal of the detector, is plotted against the true leak size. The curve represents the situation

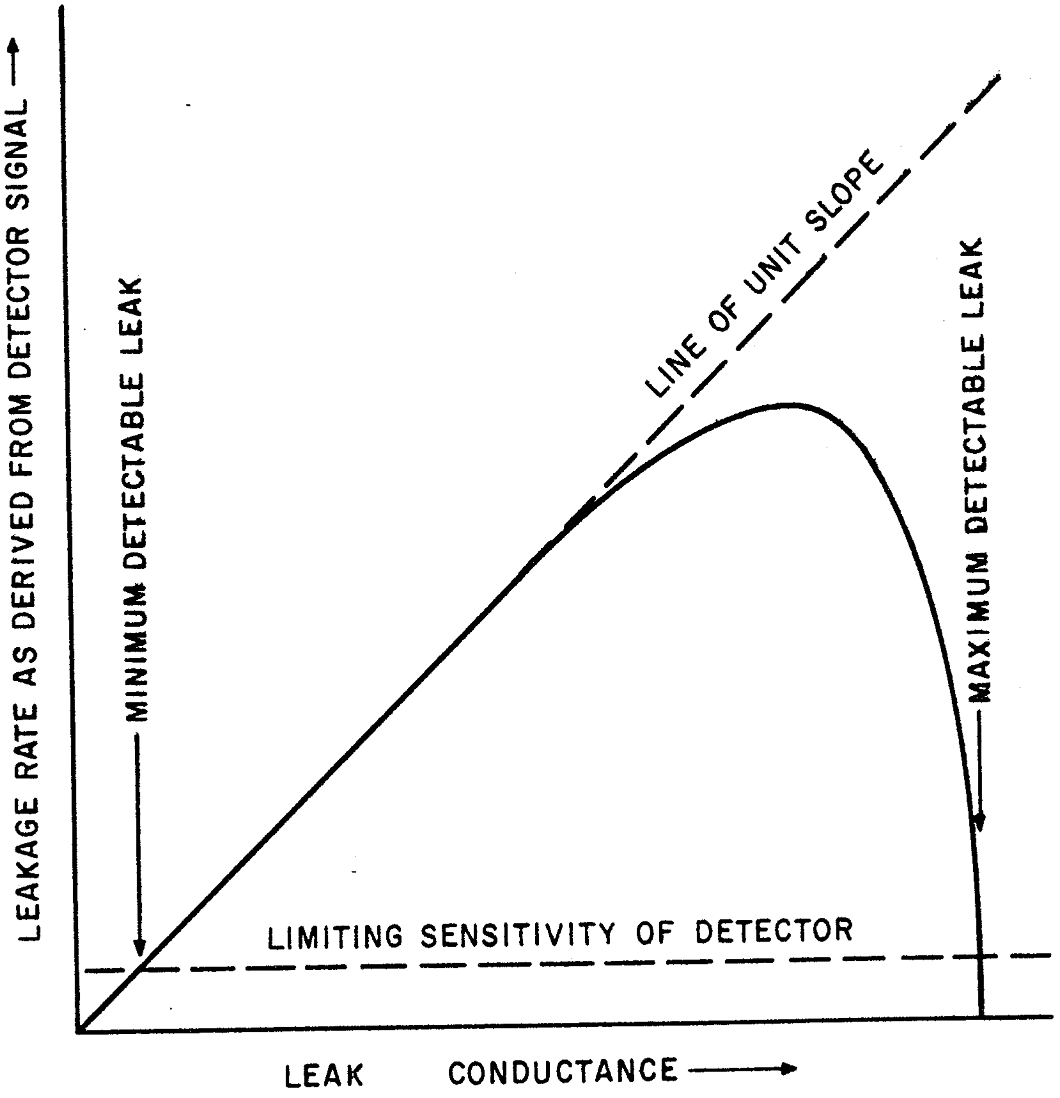


Figure 8. Detector Response for a Unit Sealed with Tracer Gas (Reprinted with permission from D.A. Howl and C.A. Mann, "The Back-Pressurizing Technique of Leak-Testing", Vacuum 15, No. 7, 347-52 (1965), Pergamon Press Copyright 1965).

where the pressure of the tracer gas is initially atmospheric and where a fixed time elapses between manufacture and testing. Because of the time lapse, the curve departs from the line of unit slope, reaches a maximum, and falls for larger leak conductances. This is caused by the diffusive loss of gas from a leaking specimen. The minimum detectable leak is thus fixed by the detector, and the maximum detectable leak by the time lapse before testing (among other factors such as the internal geometry of the unit and the rate of release of absorbed tracer gas from the internal surfaces).

It follows from this description that extremely large leaks cannot be detected by this procedure. Some independent method of testing, such as immersion bubble testing, must be used to detect extremely large leaks. It also follows that this method will not be quantitative on large leaks. The leak size on which this method stops being quantitative may be calculated from the magnitude of tracer gas loss prior to leakage measurement.

1.3.4.3.2. Back-pressurizing Technique

Back-pressurizing is a term coined by Howl and Mann (Ref. 26). Their article is the most comprehensive treatment of this technique.

In this technique the test unit is held for a period in a pressure vessel containing tracer gas at a high pressure. The gas enters the unit through any leak and is detected subsequently when the unit is taken out of the pressure vessel and the gas issues again from the leak. The rate of the flow depends not only on the conductance of the leak, but on how much gas has entered during the pressurizing period.

The leak conductance signal response curve for the back-pressurizing process is shown in Figure 9. For very small leaks, where the final signal is a function of the leak conductance and the partial pressure of the tracer after pressurizing, itself a function of leak conductance, it is obvious that the curve is concave and tangential to the x-axis. The curve crosses the line of unit slope where the leak conductance and pressurizing conditions are such as to raise the pressure of the tracer to one atmosphere. The curve remains above this line for a range determined by these two factors and, in addition, by the time lapse before testing. The physical significance of this curve being above the unit slope is that the pressure of the tracer in the specimen is above atmospheric at the time of test. The curve subsequently falls as in Figure 8.

Although the minimum and maximum detectable leaks are shown as greater in Figure 9 than in Figure 8, this is not of general significance. Back-pressurizing may be more or less sensitive than a test with previous filling with tracer, depending on the pressurizing and test conditions. The analysis which follows indicates how the sensitivity may be determined, or the conditions specified for a given sensitivity. The analysis includes the effect of absorbed tracer gas on the outside of the test specimen. This can be one of the main practical disadvantages of the method.

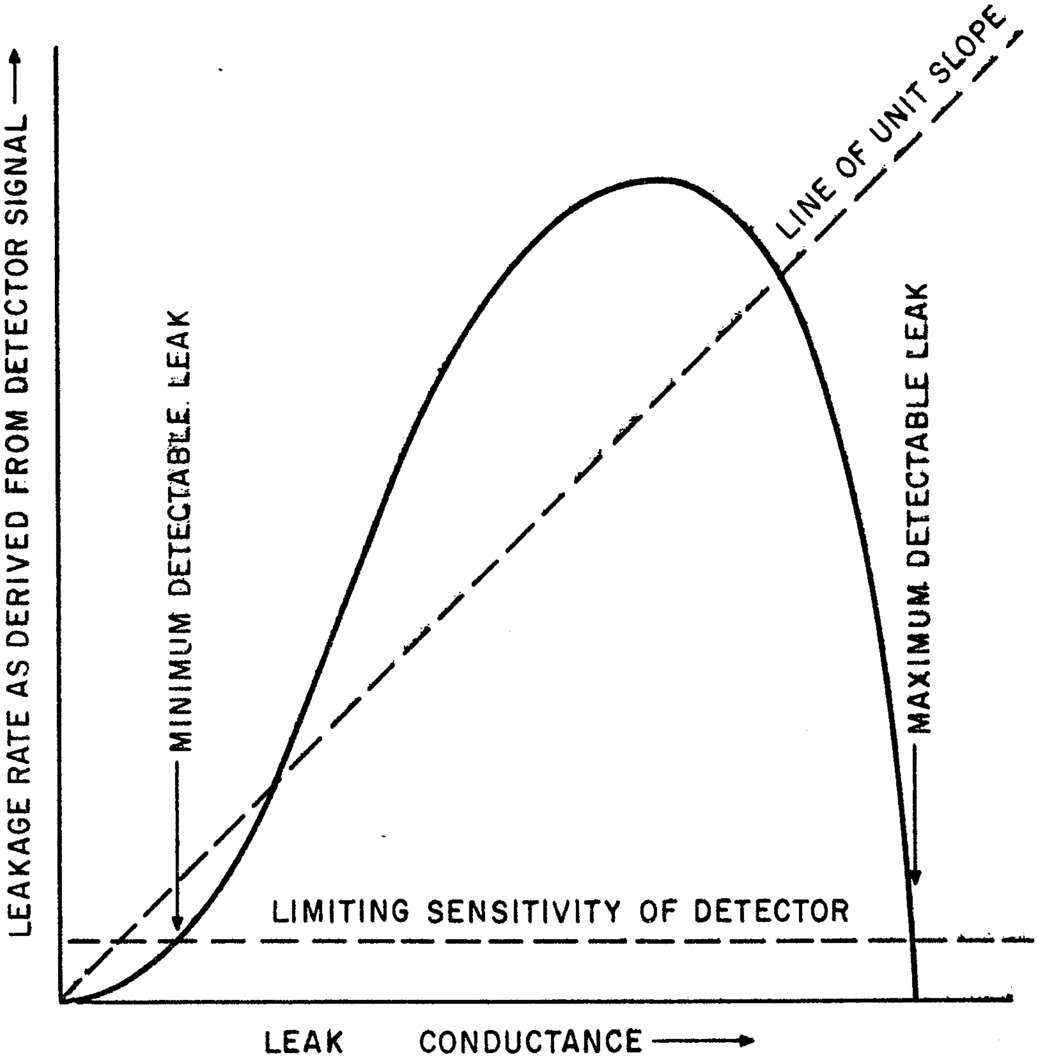


Figure 9. Relation of Leak-rate Signal to Leak Conductance for Back-pressurizing Technique (Reprinted with permission from D.A. Howl and C.A. Mann, "The Back-Pressurizing of Technique of Leak-Testing", Vacuum 15, No. 7, 347-52 (1965), Pergamon Press Copyright 1965).

The back-pressurizing technique of leak testing consists of three distinct stages:

1. The application to the external surface of the test specimen of tracer gas at a high pressure and the inflow of the tracer gas through any leak.

2. The period between the release of the external tracer gas pressure and the leak test, during which some tracer gas is lost from the test specimen through the leak.

3. The leak test itself.

Pure molecular flow and pure laminar flow through the leak are considered separately. (See Section 1.4). In leak-testing practice the leak with molecular flow is the more important, since it is necessary to detect leaks down to 10^{-7} torr liter per second or less, while leaks of 10^{-3} torr liter per second may still show molecular flow characteristics. For capillary leaks it can be shown that the leak rate size must be less than 10^{-6} torr liter per second for pure molecular flow and greater than 10^{-4} torr liter per second for pure laminar flow. However, leaks in practice may depart very greatly from circular cross-sections, so that these limits do not necessarily apply.

1.3.4.3.2.1. Molecular Flow Leaks

Assuming that the flow in and out of the system is molecular, the equation derived (Ref. 26) for the measured flow after performance of the three stages described above is:

$$Q = C_A P_E \left[1 - \exp\left(- \frac{C_A t_E}{V}\right)\right] \exp\left(- \frac{C_A t_R}{V}\right) \qquad \text{Equation 1.3-13}$$

where Q = measured gas leakage

C_A = leak conductance of the tracer gas

P_E = tracer gas pressurizing pressure

t_E = pressurizing time

V = internal free volume of the system

t_R = residence time at one atmosphere after pressurizing

Figure 10 shows a plot of the measured leakage Q against leak conductance C_A for typical values of P_E, V, t_E, and t_R. Most leak detector systems using tracer gases are designed to detect leaks very much smaller than 10^{-5} torr liter per second. An assessment of the smallest detectable leak must therefore be based upon the molecular flow equation. Furthermore, for the very small leaks (less than 10^{-6} torr liter per second), it is possible to reduce this equation to a very simple form

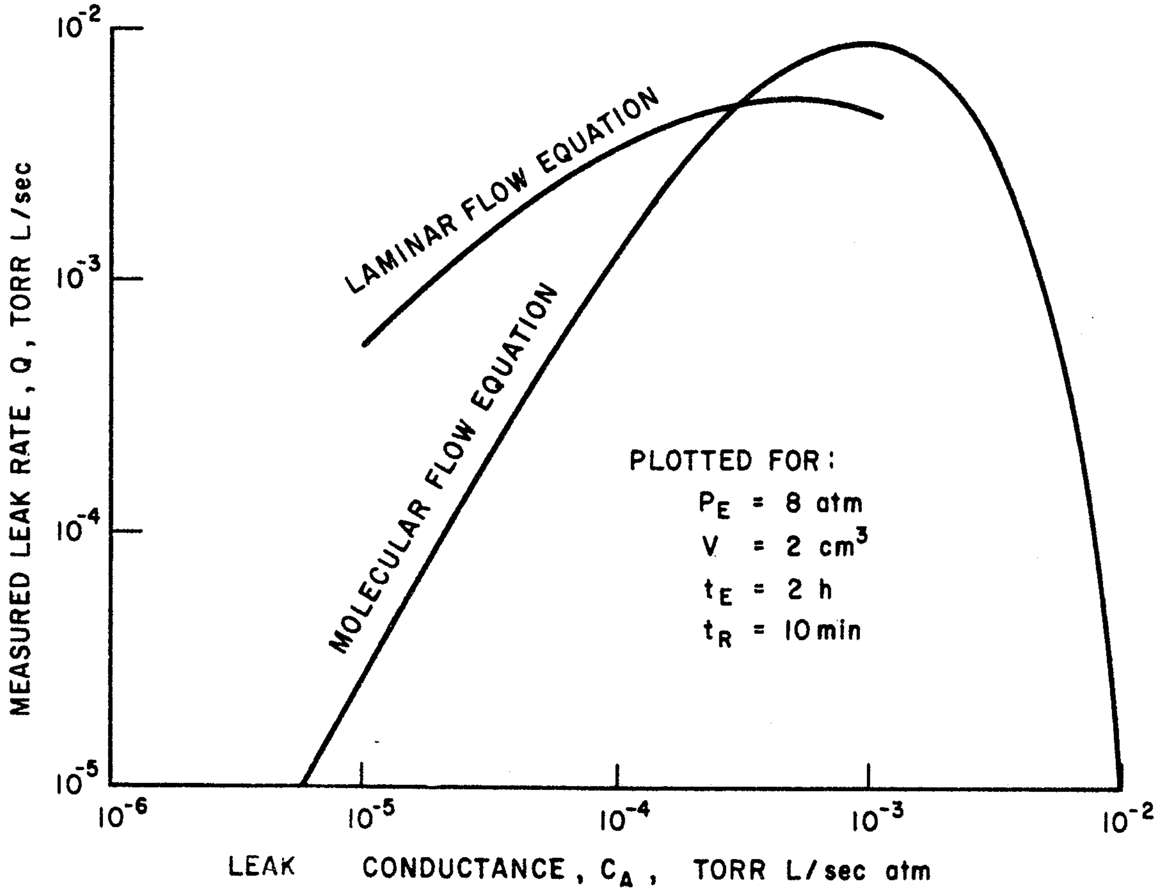


Figure 10. Computed Relation of Leak-rate Signal to Leak Conductance for a Particular Set of Conditions. (Reprinted with permission from D. A. Howl and C. A. Mann, "The Back-Pressurizing Technique of Leak-Testing", Vacuum, Vol. 15, No. 7, 1965, pp. 347-352; Copyright 1965 Pergamon Press.)

$$Q = \frac{P_E t_E}{V} \; C_A{}^2 \qquad \text{Equation 1.3-14}$$

This may be rewritten

$$P_E t_E = \frac{QV}{C_A{}^2} \qquad \text{Equation 1.3-15}$$

The smallest detectable leak C_A depends upon the minimum leak rate Q, which gives a signal on the detector appreciably above the background. The above equation defines the value of the product $P_E t_E$ necessary to detect leaks as small as C_A in a system with a background signal appreciably smaller than Q.

Figure 11 shows the product of $P_E t_E$ plotted against C_A for various values of system volume, the minimum detectable signal being taken as 2.5×10^{-8} torr liter per second.

It is common for the noise level or background signal of the detector to result mainly from the presence of tracer gas which has been absorbed on the surface of the system during pressurizing. The amount of this adsorption, and of the subsequent desorption during the leak test, is dependent on the gas used and on the material of the outer surface of the specimen, and on its finish. Generally, bright metal surfaces absorb least, oxidized metal surfaces absorb significantly, and such porous materials as graphite absorb excessively. As a first step in any back-pressurizing test, the signal from desorbed tracer gas should, if possible, be measured experimentally, using a specimen of the material which will be used.

The effect of significant absorption can be reduced by heating the specimen. This can be done either after pressurizing or, to minimize delay before testing, by heating while pressurizing. While hot, the tracer gas desorbs rapidly. In tests on nuclear fuel elements using helium (Ref. 26), temperatures in the range 200 C - 400 C have been found satisfactory in reducing background signals to negligible levels, i.e. to levels comparable to the noise level of the detector, below which no further gain in sensitivity would be achieved.

1.3.4.3.2.2. Laminar Flow Leaks

If the flow in and out of the system is laminar, the equation for measured flow after performance of three stages of back-pressurizing described in Section 1.3.4.3.2. is:

$$Q = \left(\frac{P_E - P_o}{P_E}\right) C_A P_o \left[\frac{1 + \dfrac{P_E - P_o}{P_E + P_o} \exp\dfrac{-2C_A t_R}{V}}{1 - \dfrac{P_E - P_o}{P_E + P_o} \exp\dfrac{-2C_A t_R}{V}}\right]^2 \qquad \text{Equation 1.3-16}$$

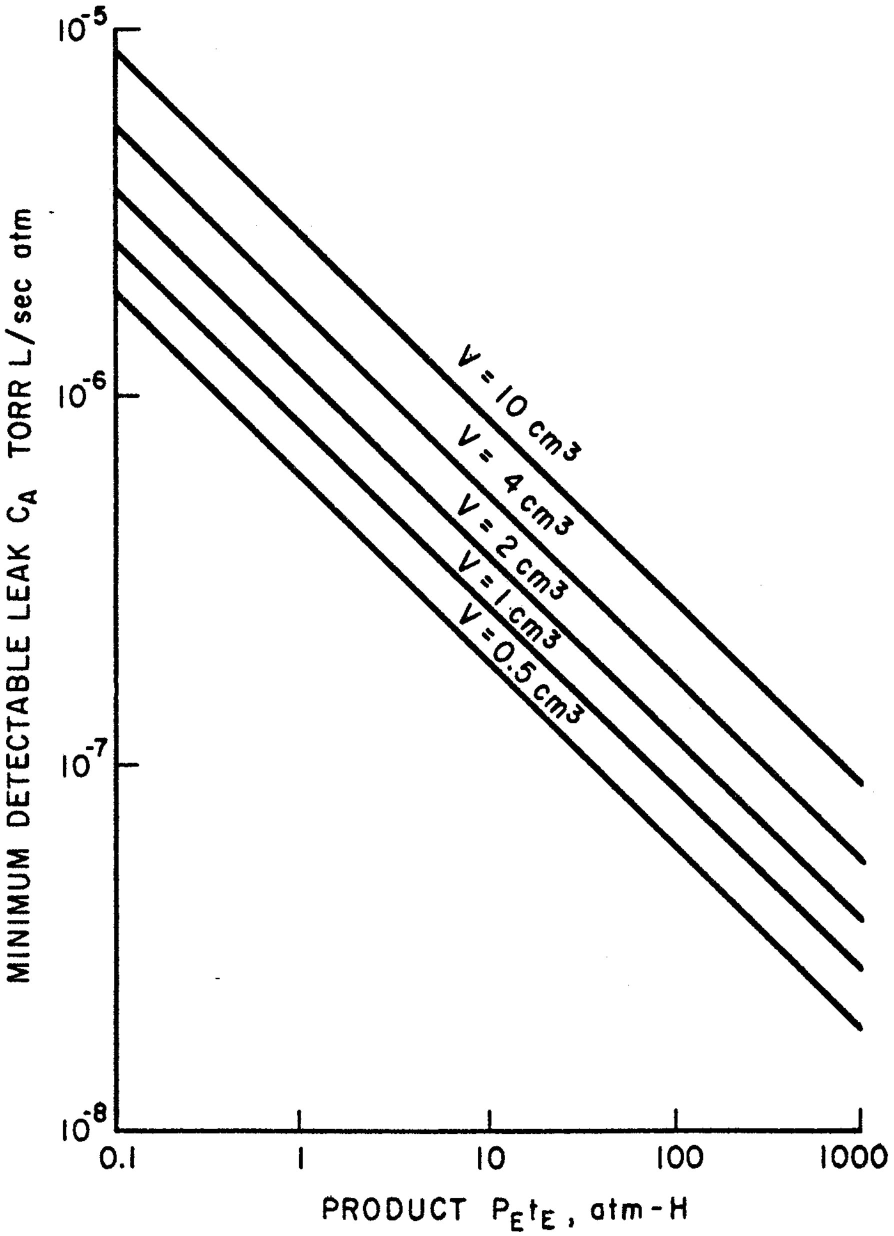


Figure 11. Required Values of $P_E t_E$ Calculated for Q = 2.5×10^{-8} Torr Liter Per Second. (Reprinted, with permission, from D.A. Howl and C.A. Mann, "The Back-pressurizing Technique of Leak-testing," Vacuum, Vol. 15, No. 7, 1965, pp. 347-352; Pergamon Press Copyright 1965.)

where Q = measured gas leakage
C_A = leak conductance of the tracer gas in units of pressure times volume per unit time at one atmosphere pressure differential
P_o = atmospheric pressure
P_E = tracer gas pressurizing pressure
t_R = residence time at one atmosphere after pressurizing
V = internal free volume of the system

The above is a simplified equation which assumes that:

$$2 \frac{C_A}{P_o} \quad t_E \; P_E > 3 \qquad \text{Equation 1.3-17}$$

This implies that Equation 1.3-17 is valid for leaks greater than the 10^{-5} torr liter per second atm range if the volume of the system is in the cubic centimeter range.

The above equation for laminar flow leaks breaks down when, in the second stage, with the test specimen in the atmosphere, the diffusion of air into the test specimen against the outflow of gas becomes appreciable. This is arbitrarily assumed to occur when the pressure in the system is less than twice atmospheric pressure.

Calculations using Equation 1.3-16 for laminar flow are compared with the results using Equation 1.3-13 for molecular flow on Figure 10. Equation 1.3-16 is plotted only between 4×10^{-5} and 8×10^{-4} torr liter per second atm, the range in which it is applicable.

In general, the size of the leaks which may be detected depend on loss of gas during the second stage. The range of detectable leaks can only be extended appreciably by reducing t_R the time between back-pressurizing and leak testing.

1.3.5. GENERAL REFERENCES

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Section 1.4

FLOW CHARACTERISTICS

1.4.1. GAS FLOW

Mass transfer attributed to leakage can occur in two modes: pneumatic flow and permeation. Pneumatic flow occurs when leakage is by passage of fluid through finite holes. Permeation is passage of a fluid into, through, and out of a solid barrier having no holes large enough to permit more than a small fraction of the molecules to pass through any one hole.

1.4.1.1. PNEUMATIC FLOWS

Pneumatic gas flow which occurs in a leak may be placed in four categories: laminar, molecular, turbulent and choked. Table 3 shows the approximate types of flow modes: 10^{-1} to 10^{-6} atm cc/sec for laminar, above 10^{-2} atm cc/sec for turbulent and below 10^{-5} atm cc/sec for molecular type of flow. As may be seen from the table, laminar flow is predominant in the area of leakage interest.

1.4.1.1.1. Laminar Flow

The laminar flow of a fluid in a pipe is defined as a condition where the velocity distribution of the fluid in the cross-section of the pipe is parabolic. Laminar flow is one of the two classes of viscous flow, the other class being turbulent flow. Because turbulent flow is rarely encountered in leaks, the term viscous flow is sometimes incorrectly used to describe laminar flow in leak detection work.

The most familiar laminar flow equation is one developed by Poiseuille (Ref. 27). For the flow through a straight tube of circular cross-section, this equation is:

$$Q = \frac{\pi}{8} \left(\frac{d}{2}\right)^4 \frac{P}{\eta \ell} \left(P_1 - P_2\right) \qquad \text{Equation 1.4-1}$$

where Q = leakage rate
d = diameter of the leak
ℓ = length of the leak
P_1 = upstream pressure, P_2 = downstream pressure
η = viscosity of the gas
P_a = arithmetic mean between P_2 and P_1 $= \frac{P_1 + P_2}{2}$

In the range where this equation is applicable, it has been substantially verified experimentally. The above equation is applicable where the length and diameter of the flow passage are known. This is not the case for most leaks, but the equation may be rewritten in this form:

Table 3

PROPERTIES OF THE VARIOUS FLOWS

	Turbulent*	Choked*	Laminar*	Molecular	Permeation
Leakage region	$>10^{-2}$ atm cc/sec		10^{-1} to 10^{-6} atm cc/sec	$<10^{-5}$ atm cc/sec	Dependent on thickness
Flow-pressure relationship	$Q \propto (P_1{}^2 - P_2{}^2)^{\frac{1}{2}}$	$Q \propto P_1$	$Q \propto (P_1^2 - P_2^2)$	$Q \propto (P_1 - P_2)$	$Q \propto (P_1 - P_2)$
Flow-property relationship	$Q \propto \sqrt{\frac{1}{M}}$	$Q \propto \sqrt{\frac{1}{M}\left(\frac{1}{\gamma + 1}\right)^{\left(\frac{\gamma + 1}{\gamma - 1}\right)}}$	$Q \propto \frac{1}{\eta}$	$Q \propto \sqrt{\frac{1}{M}}$	Varies with gas and material

P_1 = Upstream pressure

P_2 = Downstream pressure

---

* Ideal gas, compressible flow. For turbulent flow, constant friction factor is assumed.

$$Q = K \frac{P_a \left(P_1 - P_2\right)}{\eta} \qquad \text{Equation 1.4-2}$$

where K represents the constants in the above equations and the two geometry factors.

$$K = \frac{\pi}{8} \left(\frac{d}{2}\right)^4 \frac{1}{\ell} \qquad \text{Equation 1.4-3}$$

Laminar flow takes place when the Reynolds' number of flow is lower than a defined critical value. The Reynolds' number is a unitless quantity which defines the flow conditions and is given by:

$$N_{Re} = \frac{d\rho F}{\eta} \qquad \text{Equation 1.4-4}$$

where N_{Re} = Reynolds' number
ρ = fluid density
η = absolute viscosity
F = average flow velocity across a plane in the tube

By substituting the ideal gas equation in the above formula, the expression for the Reynolds' number for an ideal gas becomes:

$$N_{Re} = \frac{Q}{d} \left(\frac{4M}{\pi \eta RT}\right) \qquad \text{Equation 1.4-5}$$

where M = molecular weight
R = gas constant
T = absolute temperature

The critical value of Reynolds' number has been shown to be dependent upon the entrance conditions, roughness of the walls of a tube, and shape of the flow path. In general, for smooth tubes with well-rounded entrances, the critical value is about 1,200. For flow corrections necessitated by turns, constrictions, and surface roughness refer to Ref. 28 and 29.

The kinetic theory states that the viscosity of a gas is given by the relationship (Ref. 30):

$$N = \frac{m\bar{F}}{3\sqrt{2}\pi\sigma^2} \qquad \text{Equation 1.4-6}$$

where $\bar{F}$ = average velocity of the individual molecules
m = molecular mass
σ = molecular diameter

The average velocity of a molecule is:

$$\bar{F} = \left(\frac{8RT}{\pi M}\right)^{1/2} \qquad \text{Equation 1.4-7}$$

the mass of the individual molecules is:

$$m = \frac{M}{A^{O}} \qquad \text{Equation 1.4-8}$$

where A^{O} = Avogadro's number; i.e. number of molecules/mole
Substituting Equation 1.4-7 and 1.4-8 in Equation 1.4-6:

$$= \frac{2(MRT)^{1/2}}{3\pi^{3/2} A^{0}_{\sigma}{}^{2}} \qquad \text{Equation 1.4-9}$$

This equation shows that the viscosity of a gas is independent of pressure and is proportional to the square root of temperature.

The two most important characteristics of laminar leaks are that the flow is proportional to the difference between the squares of the pressures upstream and downstream of the leak and that the leakage is inversely proportional to the leaking gas viscosity. Table 4 shows the viscosity of most gases is similar. Therefore, a change of gas will not markedly increase the sensitivity of the leak detection method unless this change of gas implies a change of instrument sensitivity. However, as shown on Figure 12, increasing the pressure difference across the leak by a factor of a little over three will increase the flow rate through this leak by a factor of ten. Obviously then, when the leaks to be measured are in the laminar flow range, the simplest method of increasing detection sensitivity is by an increase of pressure across the leak.

1.4.1.1.2. Molecular Flow

Molecular flow is flow through a duct under conditions where the mean free path is greater than the largest dimension of a transverse section of the duct. (Ref. 13) In such a flow, each atom moves independently by random movement. Net flow is from volume of high concentration to one of low concentration.

The mean free path is the average distance that a molecule travels between successive collisions with the other molecules of an ensemble. The mean free path λ is:

$$\lambda = \frac{1}{2\pi n\sigma^{2}} \qquad \text{Equation 1.4-10}$$

where n = number of molecules in a unit volume
σ = molecular diameter

If $n = gA^{O}/MV$ Equation 1.4-11

where g = mass of the gas
A^{O} = Avogadro's number; i.e. number of molecules/mole
M = molecular weight of the gas
V = volume

Table 4

VISCOSITY OF GASES AT O C
(Ref. 29)

Gas	Viscosity, Centipoises
Acetylene	0.0092
Air	0.0169
Ammonia	0.0094
Argon	0.0208
Benzene	0.0069
Carbon dioxide	0.0135
Carbon disulfide	0.0089
Carbon monoxide	0.0171
Ethane	0.0085
Ethyl alcohol	0.0082
Ethylene	0.0093
Halogenated hydrocarbon F-11	0.0103
Halogenated hydrocarbon F-12	0.0118
Halogenated hydrocarbon F-21	0.0108
Halogenated hydrocarbon F-22	0.0120
Halogenated hydrocarbon F-113	0.0098
Helium	0.0178
Hydrogen	0.0083
Hydrogen sulfide	0.0118
Methane	0.0100
Nitric oxide	0.0178
Nitrogen	0.0168
Nitrous oxide	0.0133
Oxygen	0.0191
Propane	0.0077
Sulfur dioxide	0.0116
Water	0.0088
Xenon	0.0210

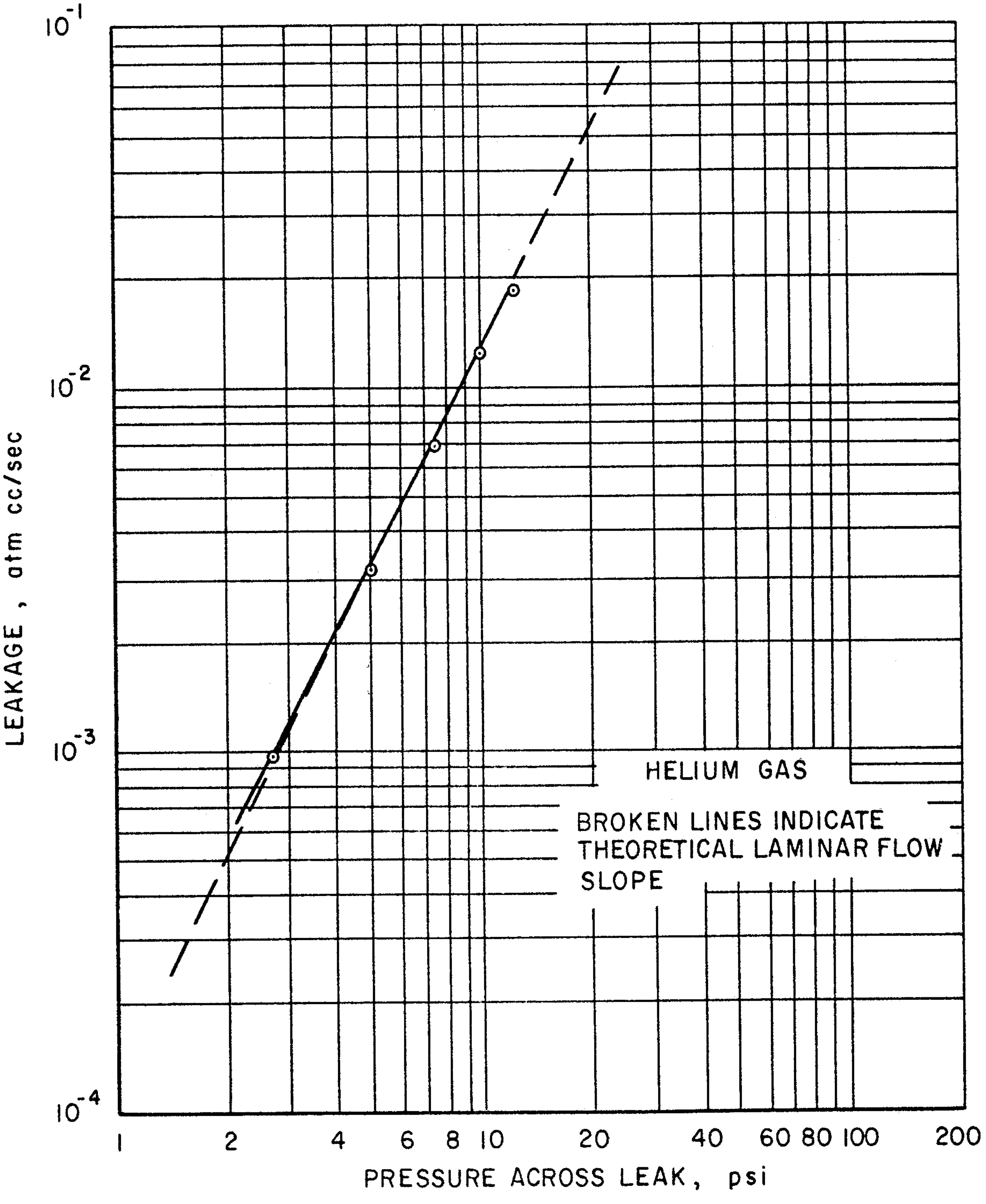

Figure 12. Laminar Flow in a Typical Hardware Leak. (Reprinted from J.W. Marr, Study of Dynamic and Static Seals for Liquid Rocket Engines, Final Report Phase II, Contract NAS 7-102. (Ref. 45))

and replacing V from the ideal gas law with the value in Equation 1.4-11:

$$\lambda = \frac{RT}{\sqrt{2}\pi P A^{o} \sigma^{2}} \qquad \text{Equation 1.4-12}$$

Equation 1.4-12 shows that at constant pressure the mean free path is proportional to temperature. However, if the amount of gas in a volume is kept constant, the mean free path is independent of temperature (Equation 1.4-10).

The magnitude of molecular diameters and mean free paths is shown in Table 5. As a convenient calculation guide, the mean free path of air at room temperature is:

$$\lambda_{\text{air}} = \frac{5 \times 10^{-3}}{P} \text{ centimeters} \qquad \text{Equation 1.4-13}$$

when P is expressed in torr.

The original mathematical derivations of molecular flow are attributed to Knudsen (Ref. 32). The rate of flow in a long pipe is:

$$Q = \frac{\sqrt{2\pi}}{6} \sqrt{\frac{RT}{M}} \frac{d^{3}}{\ell} (P_1 - P_2) \qquad \text{Equation 1.4-14}$$

where d = diameter of the pipe
ℓ = length of the pipe
P_2 and P_1 = pressures at the two ends

For this formula to apply, the pipe must be of a circular cross-section. For pipes and ducts of a non-circular cross-section the conductance is less than for pipes of circular cross-section and equal area. Equation 1.4-14 applies only if the pipe is much longer than its diameter. Any difficulty experienced by a molecule in entering the pipe must be negligibly small compared to the difficulty in traversing its length. If difficulty is experienced in entering the pipe, the kinetic theory shows that the rate of free molecular escape of gas from the container into a small aperture of area A is:

$$Q = \frac{1}{\sqrt{2\pi}} \sqrt{\frac{RT}{M}} A (P_2 - P_1) \qquad \text{Equation 1.4-15}$$

In the case of an aperture, the opening does not have to be circular to use Equation 1.4-15.

In molecular flow, the conductance of lines and apertures is independent of pressure. Calculations may be made of the effect of turns, apertures, and change in pipe diameter to calculate the overall flow in a leak. Formulas are available in the literature (Ref. 30 and 33) on the effect of structure on the flow rate.

Table 5

MEAN FREE PATHS AND MOLECULAR DIAMETERS FOR VARIOUS MOLECULES (Ref. 30)

Molecule	Mean Free Path cm x 10^{-3} at 1 Torr and 25 C	Molecular Diameter cm x 10^{-8}
H_2	9.31	2.75
He	14.72	2.18
Ne	10.45	2.60
A	5.31	3.67
O_2	5.40	3.64
CO_2	3.34	4.65
H_2O	3.37	4.68
benzene	1.34	7.65
methane	4.15	4.19
ethane	2.53	5.37
propane	1.82	6.32
n-butane	1.46	7.06
n-pentane	1.19	7.82
n-hexane	1.03	8.42

These equations demonstrate the general form. They are not applicable in most leakage situations, because the leak length and diameter are not known. The molecular flow of a gas mixture is inversely proportional to the square root of their individual masses. Therefore a certain amount of separation takes place through a leak.

Since, in molecular flow the molecules travel independently of each other, it is possible for random molecules to travel from a part of a system at low pressure to another part of the system at a higher pressure. When an ultra-high-vacuum system is being tested by a mass spectrometer leak detector, the mass spectrometer leak detector operates at a pressure of 10^{-7} torr where the ultra-high vacuum system might be operating at a pressure of 10^{-9} torr. When tracer gas enters the ultra-high-vacuum system through a leak, it will eventually arrive from the 10^{-9} torr system to the mass spectrometer operating at 10^{-7} torr by the process of molecular flow. This does not infer that the total flow is from a system at low pressure to one at high pressure, because the mass spectrometer operating at 10^{-7} torr is sending gas molecules into the system at the lower pressure. In the summation of the flows, the total flow is from the high-pressure system to the low-pressure one. The high-pressure system is contributing gas molecules to the ultra-high-vacuum system. Tracer gas flow in the direction opposing the major flow of molecules is possible because of the random mode of molecular flow. The gas molecules, when traveling from one system to the other, do not come in contact with molecules traveling in the other direction.

1.4.1.1.3. Transition Flow

The transition from laminar flow to molecular flow is gradual. The mathematical treatment of this region is extremely difficult. However, a treatment of this region is necessary because a leak from a volume to a vacuum necessarily involves a transition from laminar to molecular flow. Equation 1.4-14 shows that the conductivity of a passage in molecular flow is proportional to the cube of the passage diameter and independent of pressure. Conversely, Equation 1.4-1 shows that the conductivity of the same passage in laminar flow is proportional to the fourth power of the diameter of passage, and proportional to the pressure.

Knudsen (Ref. 32) derived a semiempirical formula for the flow of gases through long tubes in the transition region. This formula is:

$$C = \frac{\pi}{8}\left(\frac{d}{2}\right)^4 \frac{P_a}{\eta \ell} + \frac{1}{6} \quad \frac{2\pi RT}{M} \frac{d^3}{\ell} \quad \frac{1 + \quad \frac{M}{RT} \frac{dP_a}{\eta}}{1+1.24 \quad \frac{M}{RT} \frac{dP_a}{\eta}} \qquad \text{Equation 1.4-16}$$

The formula is valid providing that:

1. The flow is not turbulent in any part of the pipe.
2. The pressure difference between the ends is not so great that the mechanism of the flow (i.e. laminar or molecular) changes along the pipe.

Although the first of these conditions is usually satisfied in the leak, the second generally is not; that is, the transition from laminar to molecular flow does take place. The above equation at low pressures becomes an equation of molecular flow; whereas, at high pressure this equation reduces to one of strictly laminar flow.

Knudsen used the above equation to represent his experimental data. This equation has the effect of molecular flow added to the effect of laminar flow; consequently, it is not an actual representation of the flow mechanism taking place in the leak. The phenomenon is better visualized by realizing that both are occurring at the same time.

Burrows (Ref. 34) combined the equation for laminar flow (Equation 1.4-1) with the one for molecular flow (Equation 1.4-14) to obtain a general equation:

$$Q = \frac{\pi}{8} \left(\frac{d}{2}\right)^4 \frac{P_a}{\eta \ell} (P_1 - P_2) + \frac{\sqrt{2\pi}}{6} \sqrt{\frac{RT}{M}} \frac{d^3}{\ell} (P_1 - P_2) \quad \text{Equation 1.4-17}$$

In a way this equation is an accurate representation of the events occurring in the leak. Both laminar and molecular flow always occur in a leak. However, laminar flow is insignificant at low pressures and the molecular flow mode contributes little to total flow at high pressures.

The above equation is not completely accurate because of a "slipping" of molecules (Ref. 35) in transition flow. In laminar flow, the velocity of the molecular layers is proportional to their distance from the wall, the first layer being stationary. In the transition region slipping of the gas over the walls of the tube occurs; that is, the flow velocity at the walls is not zero. At pressures below the viscous limit, the slip correction becomes an appreciable contribution to the total conductance.

With further reduction in pressure, the dependence of the conductance on pressure becomes more complex. The flow characteristics begin a progressive change from those of viscous slip flow to those of molecular flow, where the conductance becomes independent of the pressure. The complete transition from viscous to molecular flow takes place over roughly two orders of magnitude change in pressure. This effect of slip can change the predicted flow rate by at least 20 percent. Because of this effect, Equation 1.4-16 better represents flow in the transition region. However, it is not capable of handling the total transition region (Ref. 30).

Other authors have (Ref. 34) attempted to derive equations to represent this phenomenon of transition from one type of flow to another. One simple method (Ref. 37) is to calculate laminar flow through one section of the tube, molecular flow through another, and approximate the in-between region.

1.4.1.1.4. Turbulent Flow

In viscous flow above a critical value of the Reynolds' number (about 2100 in the case of circular pipe flow), flow becomes unstable, resulting in innumerable eddies or vortexes in the flow. Any particle in turbulent flow follows a very erratic path, whereas in laminar flow the particle follows a smooth line.

The laws for turbulent flow are quite different from the laws for laminar flow. The equation relating mass flow rate (Q) in units of pressure x volume/time may be written:

$$Q = \pi d^{5/2} \left[\frac{RT(P_1^{\,2} - P_2^{\,2})}{16FM\ell} \right]^{1/2} \qquad \text{Equation 1.4-18}$$

where f, the friction factor, is defined on page 125 of Ref. 28. The friction factor depends on roughness of the channel walls, and can be considered a constant in fully developed turbulent flow.

Turbulent flow, because it requires relatively high velocity, occurs only in rather large leaks.

1.4.1.1.5. Choked Flow

Choked flow, or sonic flow, as it is sometimes called, occurs under certain conditions of geometry and pressure. Assume a passage in the form of an orifice or a venturi, and assume that the pressure upstream is kept constant. If the pressure downstream is gradually lowered, the velocity through the throat or orifice will increase until it reaches the speed of sound through the fluid. The downstream pressure at the time the orifice velocity reaches the speed of sound is called the critical pressure. If the downstream pressure is lowered below this critical pressure, no further increase in orifice velocity can occur, with the consequence that the mass flow rate has reached its maximum. This condition is known as "choked" or "sonic" flow.

Choked flow can occur under the following two conditions. (1) the flow passage must be in the form of an orifice or venturi in which only negligible frictional losses occur upstream of the orifice or throat of the venturi; (2) the ratio of downstream to upstream pressure must be below a certain "critical" value, which is

$$r_c = \left(\frac{2}{\gamma + 1}\right)^{\frac{\gamma}{\gamma - 1}} \qquad \text{Equation 1.4-19}$$

The velocity of sound through a gas can be written:

$$F_c = \frac{2\gamma}{\gamma + 1}\,\frac{RT_1}{M} \qquad \text{Equation 1.4-20}$$

where F_c = velocity sound

T_1 = absolute temperature upstream of the orifice where the velocity is low

$\gamma = C_p/C_V$ Equation 1.4-21

where C_p = heat capacity at constant pressure

C_v = heat capacity at constant volume

The mass flow rate under a choked flow condition is:

$$Q = \frac{\pi d^2 P_1}{4M} C_0 \sqrt{RT_1 \gamma \left(\frac{2}{\gamma + 1}\right)^{\frac{\gamma + 1}{\gamma - 1}}} \qquad \text{Equation 1.4-22}$$

where d = orifice diameter

P_1 = upstream pressure

C_0 = orifice discharge coefficient

For an ideal monatomic gas, the value of γ is 1.67. For polyatomic molecules, the heat energy supplies is used for increasing not only the kinetic energy of translation, but also the kinetic energy of rotation and vibration. Since the same amount of extra energy is required at both constant pressure and constant volume, γ decreases with molecular complexity. Characteristic values of γ are shown in Table 6. Additional values may be calculated (Ref. 38) or obtained from other references (Ref. 39).

Because of the stringent requirements, choked flow is rarely encountered as the predominant flow mode, except in very large leaks.

Table 6

MOLAR HEAT CAPACITY OF GASES

(in calories per mole at 25 C and 1 atm)

Gas	C_p	C_v	$C_p/C_v = \gamma$
A	4.97	2.98	1.67
He	4.97	2.98	1.67
H_2	6.90	4.91	1.41
O_2	7.05	5.05	1.40
N_2	6.94	4.95	1.40
CO_2	8.96	6.92	1.29
NH_3	8.63	6.57	1.31
ethane	12.71	10.65	1.19
propane	17.60	15.60	1.13

1.4.1.1.6. Distinction Between Modes of Flow

Equations have been presented for the various possible modes of flow that can be encountered in a leak. The following rules may be used to predict the mode most likely to occur. In distinguishing between laminar and molecular flow, the size of the passage and the mean free path are the two important parameters. The distinction may be specified by a dimensionless parameter called the Knudsen number. The Knudsen number is defined as the ratio of the mean free path of the molecule to a characteristic dimension of the channel through which the gas is flowing.

$$N_K = \frac{\lambda}{d} \qquad \text{Equation 1.4-23}$$

where N_K = Knudsen number
λ = mean free path
d = channel diameter

The type of flow encountered in the various Knudsen number ranges is shown in Equation 1.4-24.

$$\frac{\lambda}{d} < 0.01 \text{ laminar flow}$$

$$\frac{\lambda}{d} > 1.00 \text{ molecular flow} \qquad \text{Equation 1.4-24}$$

$$0.01 > \frac{\lambda}{d} > 1.00 \text{ transition flow}$$

Flow in the viscous region is determined by the Reynolds' number which was described in Equation 1.4-5.

$$N_{Re} = \frac{d\rho F}{\eta} = \frac{Q}{d} \; \frac{4M}{\pi\eta RT} \qquad \text{Equation 1.4-5}$$

The distinction between laminar and turbulent flow is shown in Equation 1.4-25

$$N_{Re} > 2100 \qquad \text{Turbulent flow}$$

$$N_{Re} < 1200 \qquad \text{Viscous flow} \qquad \text{Equation 1.4-25}$$

$$1200 < N_{Re} < 2100 \qquad \text{Either turbulent or viscous depending on duct conditions}$$

Choked flow takes place when the pressure ratio between outlet and inlet reaches a certain minimum value. This, of course, is dependent on other characteristics such as aperture dimension, etc. The formula for the critical pressure ratio is defined by Equation 1.4-26.

$$r = \frac{P_1}{P_2} \qquad \text{Equation 1.4-26}$$

The critical ratio below which choked flow takes place is:

$$r_c = \frac{2}{\gamma + 1}^{\gamma/(\gamma-1)} \qquad \text{Equation 1.4-19}$$

Choked flow cannot take place when P_1 is so low that molecular flow exists.

1.4.1.2. PERMEATION

Permeation is passage of a fluid into, through, and out of a solid barrier having no holes large enough to permit more than a small fraction of the molecules to pass through any one hole. The process always involves diffusion through a solid and may involve other phenomena such as adsorption, dissociation, migration and desorption.

The general formula for permeation is:

$$q = K_p A \frac{\Delta P}{\ell} \qquad \text{Equation 1.4-27}$$

where q = rate of mass flow
K_p = permeation rate constant
A = area normal to flow
ΔP = pressure drop along the flow path
ℓ = length of flow path

The ΔP in this case does not represent absolute pressures, but the difference in partial pressure of the leaking fluid between the two sides of the barrier. The permeation rates for some characteristic materials are shown in Tables 7-10 (Ref. 40). Additional data may be obtained in other tabulations (Ref. 41) and the original references mentioned within.

In these tables, where sufficient data is available to allow calculation, the permeability is given in parametric form:

$$\log_{10} K_p = A - B/T \qquad \text{Equation 1.4-28}$$

where the units of K_p are in cc(STP) - mm/cm^2-sec-atm in the case of polymers and glasses and cc(STP) - mm/cm^2-sec(atm)$^{1/2}$ in the case of metals; T is in K. This assumes the Arrhenius type exponential law is followed in termperature and that the permeation rate is proportional to the pressure difference across the membrane in the case of polymers and glasses, and to the difference in the square root of the pressure in the case of metals. Although these assumptions are often safe, there are many exceptions. For convenience in quick comparisons the permeability at 25 C (77 F), P_{25}, is also given in these tables. Note that values for "A" in the tables are given as the sum of two numbers, and that "P" in the tables corresponds to K_p.

Table 7

PERMEABILITY OF POLYMERIC MATERIALS TO H_2, He, N_2, O_2 AND CO_2

P in cc(STP)-mm/cm^2-sec-atm, B in ^oK

MATERIAL	TRADE NAME OR CODE	H_2			He			N_2			O_2			CO_2		
		A	B	$P_{25} \times 10^7$	A	B	$P_{25} \times 10^7$	A	B	$P_{25} \times 10^7$	A	B	$P_{25} \times 10^7$	A	B	$P_{25} \times 10^7$
Natural Rubber		0.663-1	1508	39	0.146-1	1420	23	0.672-0	2032	6.6	0.903-1	1442	18	0.556-1	1355	102
Rubber Hydrochloride	Pliofilm 140-N_2	0.206-3	1228	1.21										0.134-0	2227	0.49
	Pliofilm FM-1							0.386-1	2185	0.11	0.767-2	1836	0.40	0.160-1	1879	0.71
Poly(butadiene)		0.342-1	1442	32				0.724-1	1792	4.9	0.362-1	1551	14.5	0.839-2	1136	105
Poly(butadiene) hydrogenated	Hydropol				0.241-0	1836	12.0	0.695-1	2447	3.0	0.214-0	2141	8.6	0.946-0	1901	36.8
Poly(dimethyl butadiene)	Methyl rubber	0.000-0	1748	13	0.892-2	1442	11	0.342+2	2906	0.36	0.518+1	2469	1.6	0.892-2	1442	11
Poly(isobutylene	Oppanol B-200 (butyl rubber)	0.672-0	2076	4.9	0.544-1	1726	5.6	0.740+1	2979	0.22	0.820-0	2338	0.90	0.991-0	2207	3.8
Poly(butadiene-acrylonitrile)	Perbunan (German)	0.996-1	1770	11.5	0.079-1	1530	8.7	0.041+1	2404	0.89	0.398-0	2054	3.2	0.462-0	1814	23
	Perbunan 18	0.830-2	1355	19.2	0.098-1	1486	12.9	0.542-0	2163	1.92	0.100-0	1879	6.2	0.818-1	1530	48
	Hycar OR 15	0.187-0	1923	5.4	0.364-1	1683	5.2	0.375+2	3016	0.18	0.662+1	2622	0.73	0.453+1	2294	5.7
	Hycar OR 25	0.967-1	1792	9.0	0.302-1	1617	7.5	0.684+1	2688	0.46	0.949-0	2294	1.78	0.750-0	1967	14.1
Poly(butadiene-styrene)	Buna S, GR-S	0.491-1	1486	30.5	0.114-0	1442	17.5	0.079-0	1901	4.8	0.477-1	1595	13	0.146-0	1246	94
Poly(chloroprene)	Neoprene G	0.954-1	1770	10.3			3.4	0.748-0	2316	0.89	0.732-0	2163	3.0	0.518-0	1857	19.5
Poly(vinyl chloride-methylacrylate)	Mipolam MP	0.176-0	1945	4.4						0.2	0.505-0	2568	0.70	0.832-0	2141	4.0
Poly(alkylpolysulfide)	Thiokol B	0.869-0	2316	1.2							0.255+2	2950	0.22	0.839+1	2513	2.4
Poly(esteramide-diisocyanate)	Valcaprene A	0.280-0	1967	4.8				0.519+1	2666	0.37	0.055+1	2382	1.15	0.042+1	2054	14.1
Polyethylene (density 0.914)	DE2400 Alathon 14	0.220-0	1921	5.96	0.662-1	1814	3.75	0.524+1	2579	0.74	0.822-0	2229	2.20	0.801-0	2032	9.6
(density 0.964)	Grex	--	--	--	0.144-2	1551	0.87	0.004-1	2076	0.11	0.651-2	1836	0.31	0.718-3	1573	0.28
Poly(ethylene glycol terephthalate)	Mylar A	0.678-4	1201	0.445	0.890-4	1198	0.74	0.995-5	1639	0.0031	0.960-5	1398	0.019	0.509-4	1356	0.90
Polystyrene	Dow 0641	0.077-5	72	68.5				0.713-7	-18	5.92	0.235-6	-10	18.5	0.178-5	219	277
Polyvinyl chloride	Geon 101	0.818-6	418	2.6										0.611-7	217	0.762
+ 20% DOP		0.549-5	216	66.7										0.896-6	-332	1025
Polyvinyl (chloride 95%, Acetate 5%)	Bakelite VB1930	0.133-5	478	3.4										0.472-5	799	0.49
+ 25% DOP		0.074-1	1621	4.3	0.942-2	1570	4.8	0.023+2	2820	0.37	0.933-0	2320	1.4	0.150-0	1931	4.1
Polyvinylidene chloride	Saran 517			0.050			0.011	0.960+1	3671	0.00044	0.922+1	3474	0.0018	0.399-0	2688	0.024
Poly(caprolactam)	Nylon 6							0.386-0	2447	0.0064	0.995-2	2273	0.023	0.085-1	2120	0.093
Poly(tetrafluoroethylene)	Teflon			18			530			2.4			7.6			
Poly(chlorotrifluoro-ethylene)	Trithene, Kel-F	0.008-2	1535	0.74				0.608-0	3060	0.0025	0.890-1	2514	0.028	0.940-4	1513	0.11
plasticized		0.160-0	2018	2.4	0.015-1	1485	10.7	0.971+1	3021	0.054	0.390-0	2353	0.31	0.017+1	2346	1.4
Ethyl cellulose (plasticized)	Ethocel 610	0.877-4	751	22.7				0.787-4	905	5.6	0.022-3	824	18.1	0.524-5	305	32
Cellulose acetate (plasticized)	Celanese P-912	0.220-0	1149	6.7				0.995-4	1420	0.17	0.580-4	1093	0.82	0.075-1	1555	7.0
Cellulose acetate-butyrate	Kodapak II	0.231-3	893	16	0.533-3	1038	10.9	0.765-2	1710	1.1	0.041-2	1370	2.8	0.484-3	921	24
Polypropylene (d = 0.907)								0.111+2	2906	0.23	0.449+1	2491	1.2	0.386-0	1988	5.2
Cellulose nitrate							5.2			0.88			1.5			1.6

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Table 8

PERMEABILITY OF POLYMERIC MATERIALS TO PROPANE AND TO WATER

P in cc (STP)-mm/cm^2-sec-atm, B in $^{\circ}$K

MATERIAL	TRADE NAME OR CODE	H_2O			PROPANE		
		A	B	$P_{25} \times 10^7$	A	B	$P_{25} \times 10^7$
Natural Rubber				2600	0.141-1	1202	128
Rubber Hydrochloride	Pliofilm NO	0.0707-0	1726	19			
Poly(butadiene), hydrogenated	Hydropol				0.241+2	2273	41
Poly(isobutylene)	Butyl rubber				0.951-0	2338	1.3
Poly(chloroprene)	Neoprene GN						5.5
Poly(alkylpolysulfide)	Thiokol 3000 ST						1.1
Polyethylene(density 0.922)		0.869-0	1748	68.4			
(density 0.914)	Alathon 14				0.662-1	2447	7.2
(density 0.960)				9.1			
(density 0.964)	Grex				0.459-0	2338	0.41
Polypropylene (d = 0.907)		0.960+1	2207	35.7			
Poly(ethylene glycol terephthalate)	Mylar A	0.508-5	153	98.8			
Polystyrene		0.80-5	0	630			
Poly(vinyl chloride)	Geon 101	0.78-4	514	110			
Poly(vinyl chloride-acetate)	Vinylite VYNWO	0.37-2	874	275			
Poly(vinylidene chloride)	Saran 517	0.941-7	2404	7.5			0.00027
Polyamide	Nylon 6-6			53-516**			
Poly(chlorotrifluoroethylene)	Kel F 300	0.222-1	2050	0.22			
Ethyl cellulose (plasticized)	Ethocell 610	0.995-4	0	9880			
Cellulose acetate		0.621-4	0	4180			
(plus 15% DBP)		0.750-4	0	5620			2.8
Cellulose nitrate				4800			0.0058
Poly(methylmethacrylate)	Lucite			2200			

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**Humidity dependent.

Table 9

PERMEABILITY OF GLASSES

P in cc (STP)-mm/cm^2-sec-atm, B in^{o}K

SYSTEM	A	B	P_{25}
H_2-Fused silica	0.398-5	1873	1.12×10^{-12}
-Vycor (Corning 7900)	0.520-5	1826	3.9×10^{-12}
He-Fused silica (GE)	0.401-4	1049	0.76×10^{-7}
-Vycor (Corning 7900)	0.579-4	1049	1.14×10^{-7}
-Pyrex (Corning 7740)	0.654-4	1398	0.91×10^{-8}
-Soda lime (Corning 0080)	0.822-4	2403	5.7×10^{-12}
-X-ray shield (Pittsburgh Plate Glass)	0.662-5	2731	3.1×10^{-14}
N_20Fused silica	0.967-4	4820	6.2×10^{-20}

Table 10

PERMEABILITY OF METALS

P in cc (STP)-mm/cm^2-sec (atm)$^{1/2}$, B in ^{o}K

SYSTEM	A	B	P_{25}
H_2-Aluminum	0.589-1	3017	7.5×10^{-11}
Copper	0.621-1	3933	2.6×10^{-14}
Hastalloy B	0.701-1	3657	2.7×10^{-13}
Inconel	0.847-1	3649	4.0×10^{-13}
Iron	0.554-2	1836	2.6×10^{-8}
Kovar	0.352-1	3497	4.1×10^{-13}
Monel	0.161-1	2797	5.9×10^{-11}
Nickel	0.520-1	2885	6.9×10^{-11}
Niobium	0.749-1	1137	8.7×10^{-5}
Palladium	0.929-1	2296	1.7×10^{-8}
Platinum	0.512-1	3936	2.1×10^{-14}
Steel			
Cold drawn	0.554-2	1879	1.8×10^{-8}
Low carbon	0.312-2	2288	4.2×10^{-10}
Stainless 303	0.435-1	3540	4.6×10^{-13}
304	0.586-1	3715	1.3×10^{-13}
316, 321	0.273-1	3252	2.3×10^{-12}
347	0.634-2	3180	9.2×10^{-13}
410	0.188-1	3260	5.7×10^{-12}
N_2-Iron	0.093-1	5204	4.3×10^{-19}
Molybdenum	0.359-0	9837	4.4×10^{-33}
CO-Iron	0.554-2	4066	8.1×10^{-16}
O_2-Silver	0.754-1	4941	1.5×10^{-17}

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If any system is to be relatively leaktight, the materials of construction have to exclude leakage by permeability. As an example, the permeation rate at room temperature of a natural rubber gasket 0.1 inch thick, 0.1 inch wide, and 5 inches in diameter with a one-atmosphere hydrogen pressure differential is 1.6×10^{-5} atm cc/sec. In some uses, this permeation might represent an unacceptable leakage rate.

Permeation presents a problem in leak checking equipment where the construction materials have a high permeability to the tracer gas. For example, if a component containing a rubber diaphragm one millimeter thick and one square inch in surface area is leak checked using helium gas, a leakage of approximately 1×10^{-5} atm cc/sec will be measured across the diaphragm. This leakage is due to permeation of helium through the diaphragm and not any actual holes. It represents the maximum sensitivity of helium leak testing that can be performed on this component. However, if the component is to be used with another fluid to which the membrane is impermeable, the apparent leakage due to permeation measured during leak testing has little meaning under operating conditions.

Another example of this type of false reading is a rubber O-ring. Depending on material, a rubber O-ring usually represents a permeability of approximately 10^{-7} atm cc/sec atm per linear inch of exposed surface. Figure 13 is an example of the permeation rates of O-rings of various materials. This permeability does not have to be taken into consideration during routine leak checking if leakage measurement occurs in a time too short to permit the saturation and mass transfer of helium through the O-ring.

To eliminate permeability as a factor in leakage measurement, three procedures may be used.

1. The leakage measurement may be taken rapidly, not allowing the material to be saturated with gas. This is only possible if the material is relatively thick. For example, a rubber diaphragm will rapidly saturate and almost immediately show leakage. On the other hand, O-rings are relatively thick and will not saturate rapidly enough to give a reading within a reasonable period of time (five minutes). If the diffusivity and solubility of the fluid in the material is known, it is possible to calculate the rate of increase of leakage. This calculation is described by Baer (Ref. 41). However, in many cases (where the leakage path is long) this calculation is not necessary. Rather than calculations, experimental results can determine very quickly if leakage through a thick gasket is inconsequential for short-time periods.

2. The maximum permeability of all components and the resulting mass transfer produced during leak testing may be calculated. Equation 1.4-27 is used for these calculations. In this way, the permeability value will be

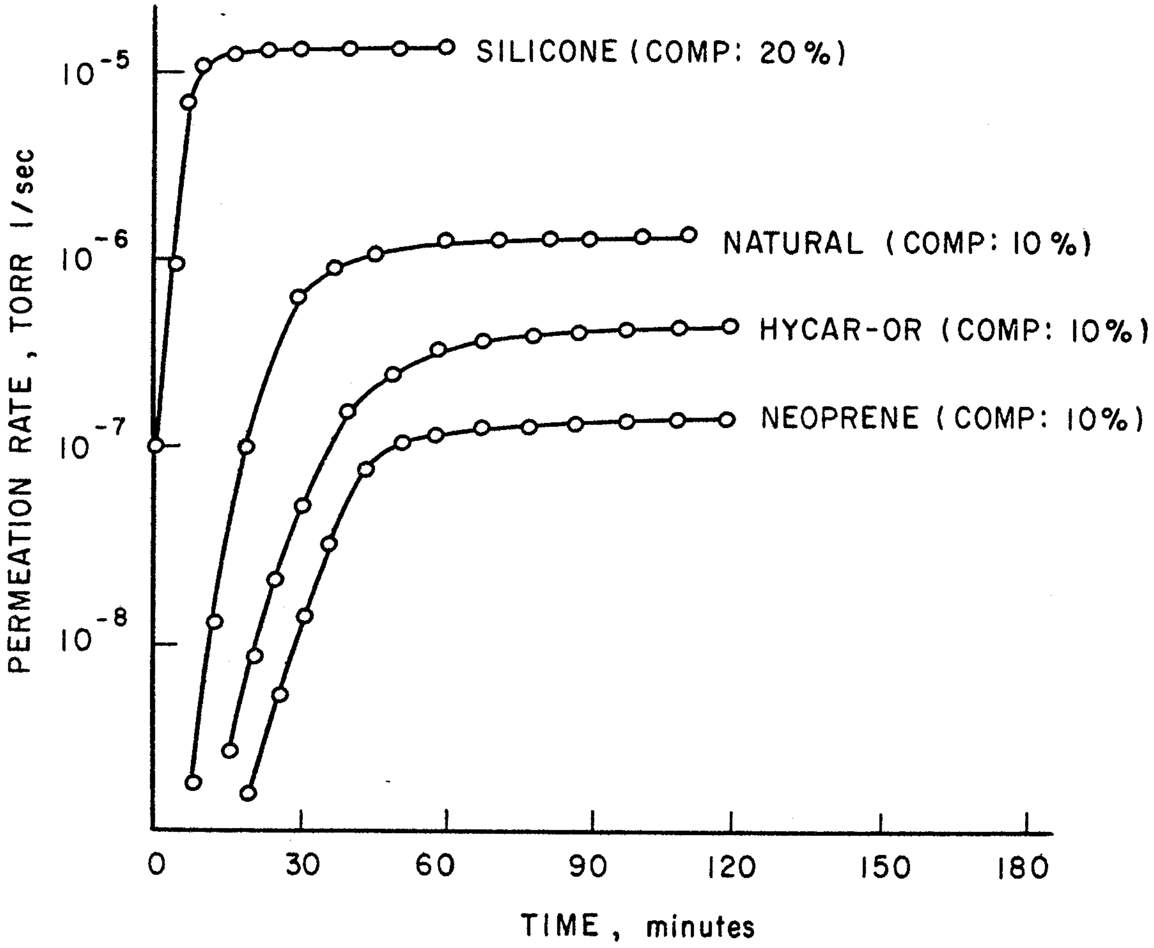

Figure 13. Permeation Rate Versus Time of Rubber Gasket for a 4x4mm Cross Section. (Reprinted with permission from I. Kobayeshi and H. Yada, "Leak Test of Rubber Gaskets," Fundamental Problems in Vacuum Techniques, Vol. I, Pergamon Press, London, 1960, p. 248.)

known and only leakage above this value will be considered as leakage flow.

3. The last method and the most difficult one is to quantitatively measure the leakage at various pressure differentials. Providing the leak being measured is pneumatic (i.e. it is through a hole in the component) and laminar, the flow is proportional to the square of the pressure differential across the leak. However, if the flow is strictly due to permeation, then the flow through the leak will be directly proportional to the difference in concentration across the leak. In this way, the presence of holes in the component can be differentiated from permeation.

1.4.2. LIQUID FLOW

The flow of a liquid through a leak may be one of two modes: laminar or turbulent. The flow characteristics and the requirements for attaining the flow modes are similar for gases and liquids. The criterion is a certain Reynolds' number. The viscous flow equations for gas are different from those for liquids because of the modifications necessary due to gas compressibility.

1.4.2.1. LAMINAR FLOW

Poiseuille's equation for laminar flow leakage is:

$$Q_v = \frac{\pi}{8}\left(\frac{d}{2}\right)^4 \frac{P_1 - P_2}{\eta \ell} \qquad \text{Equation 1.4-29}$$

where Q_v = volume flow in units of volume per unit time.

1.4.2.2. TURBULENT FLOW

Turbulent flow equation for a liquid can be expressed:

$$Q_v = \frac{\pi}{4}\, d^{5/2} \sqrt{\frac{P_1 - P_2}{2 \rho f \ell}} \qquad \text{Equation 1.4-30}$$

Turbulent flow occurs only in very large liquid leaks.

1.4.3. CORRELATION BETWEEN FLOW TYPES

1.4.3.1. CONVERSION OF FLOW RATES FOR VARYING GASES

The equations discussed thus far show the flow relationship in ideal cases. Leaks, however, are not intentional and, therefore, far from ideal. They are not the long straight tubes for which equations are easy to obtain. The general approach to leakage correlation is to predict which flow mode occurs and to predict changes which will occur upon change in conditions within the flow mode.

Table 3 shows the properties of the various flow leaks and how fluid parameters influence the flow rate. This table is derived from the various flow equations presented in the text.

As may be seen from Table 3 the influence of varying the gas is not so great as that of varying the flow mode. Once the flow mode is determined, the conversion to another gas should be relatively easy to make, providing the relationships in the above table are in fact correct. The major difficulty is identifying the predominant flow mode.

The data necessary for the conversion is relatively easy to obtain. For example, the viscosity of many gases is published. Even if the viscosity is not known, approximation should not produce a large error. As shown in Table 4, the viscosity of gases at constant temperature varies by less than half an order of magnitude between the most viscous and the least viscous.

For molecular flow, the molecular weight of the gases is easily available and should cause no problem in the conversion. If choked flow does occur, gamma, necessary for conversion of choked flow leakage, is 1.67 for monatomic gases and rapidly approaches one as the complexity of the gas molecule increases.

Working with a variety of leaks of different sizes and under different conditions, some of the flow modes may readily be eliminated. For example, if the leak rate is small, it is relatively easy to assume that no turbulent flow will take place. If the leak is one going from high pressure to a slightly lower pressure, but not a vacuum, it is likely that molecular flow is not the flow mechanism. In the above case, the flow may be of a laminar nature and therefore conversion to a second flow pressure is relatively easy. Choked flow is rarely encountered in small leaks.

Another example - convert one leakage rate flowing into a vacuum to an anticipated rate for a different pressure into the same vacuum. Calculations described in Ref. 34, show that if the leak is of relatively small size (10^{-5} atm cc/sec or less) molecular flow will play a major role in such a leak. However, should the leak be relatively large (10^{-3} atm cc/sec or greater), the leakage will be predominately of laminar nature. If one can accurately predict the type of flow which will predominate in a leak, one could therefore make accurate conversions to a different set of conditions. Unfortunately, the state-of-the-art is such that these predictions are usually not possible.

Many authors (Ref. 30, 34,37, and 43) have predicted the following predominant flow modes in leaks:

turbulent	$>10^{-2}$ atm cc/sec
laminar	10^{-1} - 10^{-6} atm cc/sec
transition	10^{-4} - 10^{-6} atm cc/sec
molecular	$<10^{-6}$ atm cc/sec

Assuming the flow mode has been identified, the following are sample calculations for correlation of flow rates with the use of different gases and pressure.

This sample calculation is for laminar flow. The general equation for laminar flow is:

$$Q = \frac{\pi}{8}\,\frac{d}{2}^{4}\,\frac{P_1 + P_2}{\eta\,2\,\ell}\,(P_2 - P_1) \qquad \text{Equation 1.4-1}$$

where Q = leakage (mass flow in units of pressure x (volume/time)
d = average diameter of leak
P_2 = pressure on the entrance side of the leak
P_1 = pressure on the exit side of the leak
η = viscosity and the leaking fluid or fluid mixtures
ℓ = leak length

The dimensions of d and ℓ are usually not known. An apparent conductance K may be calculated by the formula, where this apparent conductance is the product of $\frac{\pi}{8}\,\frac{d}{2}^{4}\,\frac{1}{\ell}$ and any unit conversion factors. From this calculation, an apparent geometry factor can be calculated.

$$K = \frac{\pi}{128}\,\frac{d^4}{\ell} \qquad \text{Equation 1.4-3}$$

If K is calculated only for conversion from one flow to another, the constant does not have to be in compatible units, providing that the same units are used both in solving for K and using the K in correlation equations.

Using the apparent conductance K calculated above, the flow of any gas at operating pressure may be predicted using Equation 1.4-2.

$$Q = \frac{K}{2\eta}\,(P_1^{2} - P_2^{2}) \qquad \text{Equation 1.4-2}$$

A similar apparent conductance may be calculated for other flow modes using the equations given earlier in this section. Such calculations are correct only if the flow mode has been correctly chosen.

When there is doubt about the correctness of flow identification, the following procedure is recommended:

1. If pressure is increased, correlate as laminar.
2. If pressure is decreased, correlate as molecular.
3. If gas is changed, correlate as molecular.

Correlation should be performed so that, if an error is made, actual leakage will be no greater than that predicted in the correlation. Correlation of leaks resulting from increased pressure across a leak is not recommended. Reasons for this statement are given in Section 1.4.4.

1.4.3.2. CONVERSION BETWEEN GASES AND LIQUIDS

It is possible to predict liquid leakage from leakage measured under other known conditions. This method is generally applicable on a leak or series of leaks, most of which have a conductance between 1 and 10^{-5} atm cc/sec atm. The procedure consists of calculating an apparent conductance of the leak and, by assuming laminar flow conditions, calculating the expected leakage. The derivation of the equation is as follows.

The equations for flow of gases:

$$Q = \frac{\pi}{8} \left(\frac{d}{2}\right)^4 \frac{P_a}{\eta \ell} (P_1 - P_2) \qquad \text{Equation 1.4-1}$$

and for liquids:

$$Q_v = \frac{\pi}{8} \left(\frac{d}{2}\right)^4 \frac{P_1 - P_2}{\eta \ell} \qquad \text{Equation 1.4-29}$$

both have the similar geometry factor K:

$$K = \frac{\pi}{8} \left(\frac{d}{2}\right)^4 \frac{1}{\ell} \qquad \text{Equation 1.4-3}$$

In substituting the geometry factor, the equations reduce to:

$$Q = \frac{K}{\eta} P_a (P_1 - P_2) \qquad \text{Equation 1.4-2}$$

for gases and:

$$Q_v = \frac{K}{\eta} (P_1 - P_2) \qquad \text{Equation 1.4-31}$$

for liquids. In use, the system leakage is measured and Equation 1.4-2 is solved for K. This calculated K is then used in Equation 1.4-31 so that the liquid leakage may be predicted. Such a procedure will be accurate only in laminar flow leaks. Should the measured leakage be molecular rather than laminar, the error introduced in the calculation will predict a greater liquid leakage than will actually be found. The equation may therefore be used with confidence, since any error will add a margin of safety into the results.

The following restrictions apply to the use of the above method:

1. The leakage is the result of a finite hole or holes and not the result of permeation.

2. The gas flow is laminar, i.e. the flow through the leak is in the range of 1 to 10^{-6} atm cc/sec or is made up of a number of leaks in that flow range.

3. The calculations should at best be considered accurate to only a factor of two. Error in both the measurements and the deviations from the flow equations preclude more accurate solution.

If Equations 1.4-2 and 1.4-31 are combined, the ratio of gas to liquid leakage through the same leak at the same pressure is:

$$\frac{Q}{Q_v} = \frac{\eta_{liq.} \, P_a}{\eta_{gas}} \qquad \text{Equation 1.4-32}$$

This is a convenient method of determining what liquid leakage will exist if the gas leakage is measured at the same pressure.

Experiments were recently performed (Ref. 44) to check the validity of the above correlation. Liquid leakage was measured for leaks with previously measured gas leak rates. It was found that leaks having a gas conductance in the 10^{-3} atm cc/sec range had a liquid leakage approximately one-half that predicted by Equations 1.4-31 or 1.4-32. Leaks in the 10^{-4} atm cc/sec range leaked liquid at a rate approximately one-tenth the rate predicted by theory. Liquid leakage in leaks in the 10^{-5} atm cc/sec atm range were approximately one-twentieth of that predicted by the above equations.

Based on the above, it would appear that the equations for correlation will produce conservative answers, i.e. the actual liquid leakage will always be smaller than that predicted by theory.

It is believed that the liquid flow is lower than calculated for two reasons:

1. No correction was made for any molecular flow component of the measured gas leakage.

2. Physical adsorption completely immobilized a layer of liquid adjacent to the leak wall and therefore reduced the apparent leak diameter.

1.4.3.3. GRAPHICAL CORRELATION

Santeler and Moller (Ref. 43) used the above equations to construct a graph for solution of these correlations. Such a graph is shown on Figure 14. The application of this graphical method is discussed in the following paragraphs.

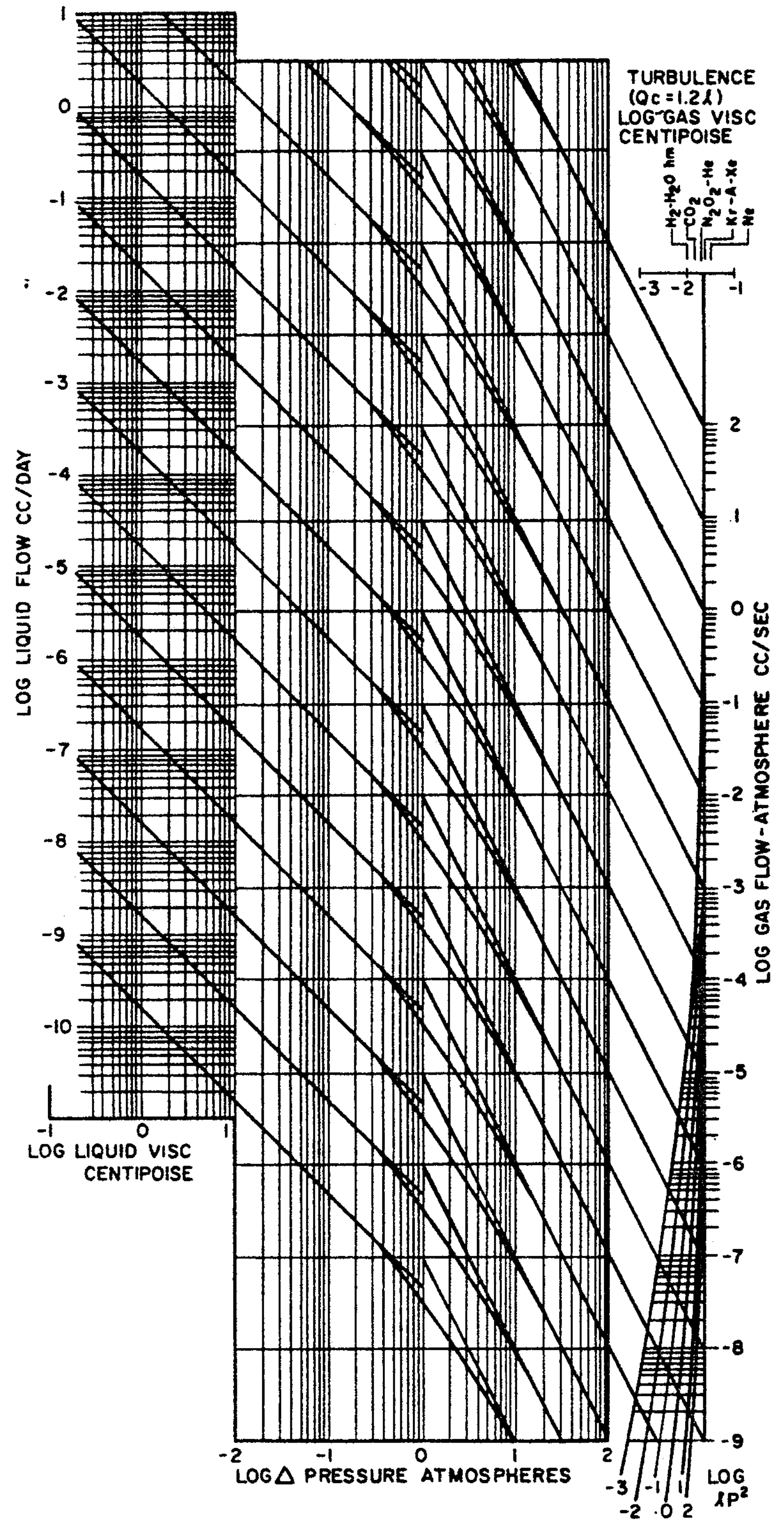

Figure 14. Fluid Flow Conversion Graph

1.4.3.3.1. General Description

Using the equations previously discussed the nomograph (Figure 14) was constructed. In the nomograph, ΔP is plotted on the lower axis. The respective gas and liquid flows are on the right and left margins. The horizontal guide lines are characteristic of a given geometry independent of pressure or type of gas. That is, they represent the K factor in the equations. On the left the diagonal lines are a 45 degree slope which fits the relation $Q = \frac{K'}{\eta} \Delta P$ for liquids. On the right side the slope becomes 2:1 which satisfies the relation $Q = \frac{K}{\eta} \Delta P^2$. In order to handle different gases and liquids, two viscosity scales were added to the graph. The scale on the right is for gases while the left scale is for liquids. This liquid viscosity scale has been selected and oriented on the graph so that its decade extensions coincide with the pressure decades. This permits easy extrapolations to higher values of liquid viscosity.

For gaseous flow the units are atm cc/sec while for liquid flow the units are cc/day. Pressure is expressed in atmospheres and length in centimeters. The units used for the viscosity of both liquids and gases are centiposies.

In any use of the graph to make a conversion from one set of pressure and flow conditions for a given gas or liquid to another set of pressure and flow conditions for a new gas or liquid, enter the graph at the right side at the log of the given gas flow or at the left side at the log of the given liquid flow. Move horizontally to the viscosity of the given liquid or to the log ℓP^2 of the given gas. Then move parallel to the diagonal slope lines to the given differential pressure (log ΔP).

In all cases involving liquids, the diagonal movement will be along a straight line, as evidenced by the lines leading from the left side of the graph. Also, diagonal movement is along a straight line for gases if the gas flow is to a vacuum. The curved portions of the diagonal slope lines are used only in cases involving gas differential pressures with no flow to a vacuum.

After the given differential pressure is located by keeping these slope restrictions in mind, move horizontally to the new differential pressure (log ΔP).

Then move parallel to the diagonal slope lines to the log of the new liquid viscosity (i.e., up and to the left along a straight line), or, in the case of a new gas, to the log of ℓP^2 (i.e., down and to the right). Stop at the horizontal line which intersects the new viscosity or the new ℓP^2. The horizontal line is the desired log of the new gas or the new liquid flow rate as determined by the graph.

1.4.3.3.2. Corrections For Molecular and Transitional Flow

Only laminar flow has been considered thus far. Leaks in size of 10^{-4} atm cc/sec and smaller can be in the transition range between laminar and molecular flow. Very small leaks around 10^{-9} and 10^{-10}, are predominately molecular. The use of a laminar conversion for leaks of either of these types could result in considerable error, since molecular flow is a linear function of pressure rather than the squared function for which the graph is constructed. To make this graph applicable to the conversion of leaks which fall in these two ranges, a correction factor must be applied. It would be desirable if this factor could be applied directly on the basis of the leak size, but unfortunately, this is not the nature of gas flow. It is perfectly feasible for one leak to be in laminar flow while a second leak which passes an identical gas flow under the same pressure conditions could be in molecular flow. With a change in pressure conditions, the flow of gas through the two leaks will become widely different. To apply a correction for molecular flow, either the radius or length of the leak must be estimated. In this handbook, the correction will be based on an assumed length of the leak. The reasons for this selection are two-fold: (1) the length has a linear effect on the equations for both laminar and molecular flow and (2) it is much easier by visual inspection to predict a probable length of leak. Certainly, considerable error might exist in this estimate of length, since the actual flow path may vary from essentially an orifice to a long winding restriction.

A correction on the basis of an assumed length alone is not sufficient. The assumption of a value for the length permits an estimate of the extent to which transition or molecular flow exists, but a pressure correction must then be applied to account for the changed flow condition.

As a parameter for gas flow, the expression ℓP^2 where ℓ is the length in centimeters, and P is the inlet pressure in atmospheres has been chosen. The family of lines on the right side of Figure 14 has been drawn for dry air. The viscosity correction has reduced to a scale on the upper right margin.

1.4.3.3.3. Sample Solutions Incorporating the ℓP^2 Correction

Next, consider how these extra factors are used. Assume that a gas leakage of 10^{-8} has been measured with the conditions of air at one atmosphere to vacuum. It is desired to know what the air leakage would be for pressure conditions of 132 psi gage, or 10 atmospheres absolute, to vacuum. The solution is shown in Figure 14. A value must first be assumed for ℓ, for instance, $\ell = 0.1$ cm. The inlet pressure for the test conditions was one atmosphere. Therefore $\ell P^2 = 0.1$. Starting with the flow of 10^{-8} atm cc/sec, move to the line $\log \ell P^2 = -1$, then up to 2:1 sloped line to the pressure of one atmosphere. Here obtain the horizontal characteristic line and move to the new pressure differential condition of 10 atmospheres. Since the low side pressure is still a vacuum, follow down the 2:1 slope. Since the pressure conditions have changed, ℓP^2 has changed to 10 rather than 0.1. By going to the $\log \ell P^2 = 1$ line, the corrected flow of 4×10^{-7} atm cc/sec is obtained. Had the existence of laminar flow been assumed, uncorrected solution would have been obtained. That is, the expected flow would have been 1×10^{-6} atm cc/sec.

1.4.3.3.4. Turbulent Flow Limitations

The graph is developed on the basis of laminar flow and corrected for conditions of transition and molecular flow. There remains only to explore the opposite end of the scale, at high flow rates, to determine under what conditions turbulence can occur.

The point or region of transition from viscous laminar flow to turbulent flow has never been accurately established. In past practice, transition has been considered to begin at a Reynolds' number - defined as $\left(\frac{\rho F d}{\mu}\right)$ - of approximately 2100. Theoretical considerations of the nature of the flow at this point of transition indicate turbulence should occur when the flow in atm cc/sec exceeds 1.2 times the length of the leak in centimeters. This criterion for incipient turbulence has been included on the conversion graph by the Equation $Q = 1.2\ell$.

1.4.4. ANOMALOUS LEAKS

Four types of leaks were encountered which did not fit in the categories already discussed. These were check valve leaks, surface flow leaks, self-cleaning leaks, and geometry change leaks. The errors in leak measurement due to the presence of these types of leaks could be greater than any errors due to the miscalculation discussed in the flow sections.

1.4.4.1. CHECK VALVE LEAKS

During a study (Ref. 45) of leakage phenomena, examples of check valve and geometry change leaks were found.

Figure 15 is a plot of the leakage-pressure differential obtained on a damaged needle valve. It was observed that although the typical laminar flow curve was obtained at a high pressure differential, below this pressure, the leakage abruptly stopped. Upon increasing the pressure, the leak reappeared. This phenomenon was repeatable.

This type of leak would be particularly hard to detect since the leak cannot be seen below a critical pressure.

1.4.4.2. GEOMETRY CHANGE IN LEAKS

The shape of a leak may change with changes in system pressure. As pressure increases, the expansion of system parts resulting from stresses induced by the increased pressure can cause leakage rates of known leaks to increase beyond the predictions of laminar flow theory. An example of this increase of leak rate with geometry change is shown in Figure 16.

1.4.4.3. SELF-CLEANING LEAKS

In performing work on project NAS 8-4012 (Ref. 46), gaskets under compression are subjected to a high helium pressure and the

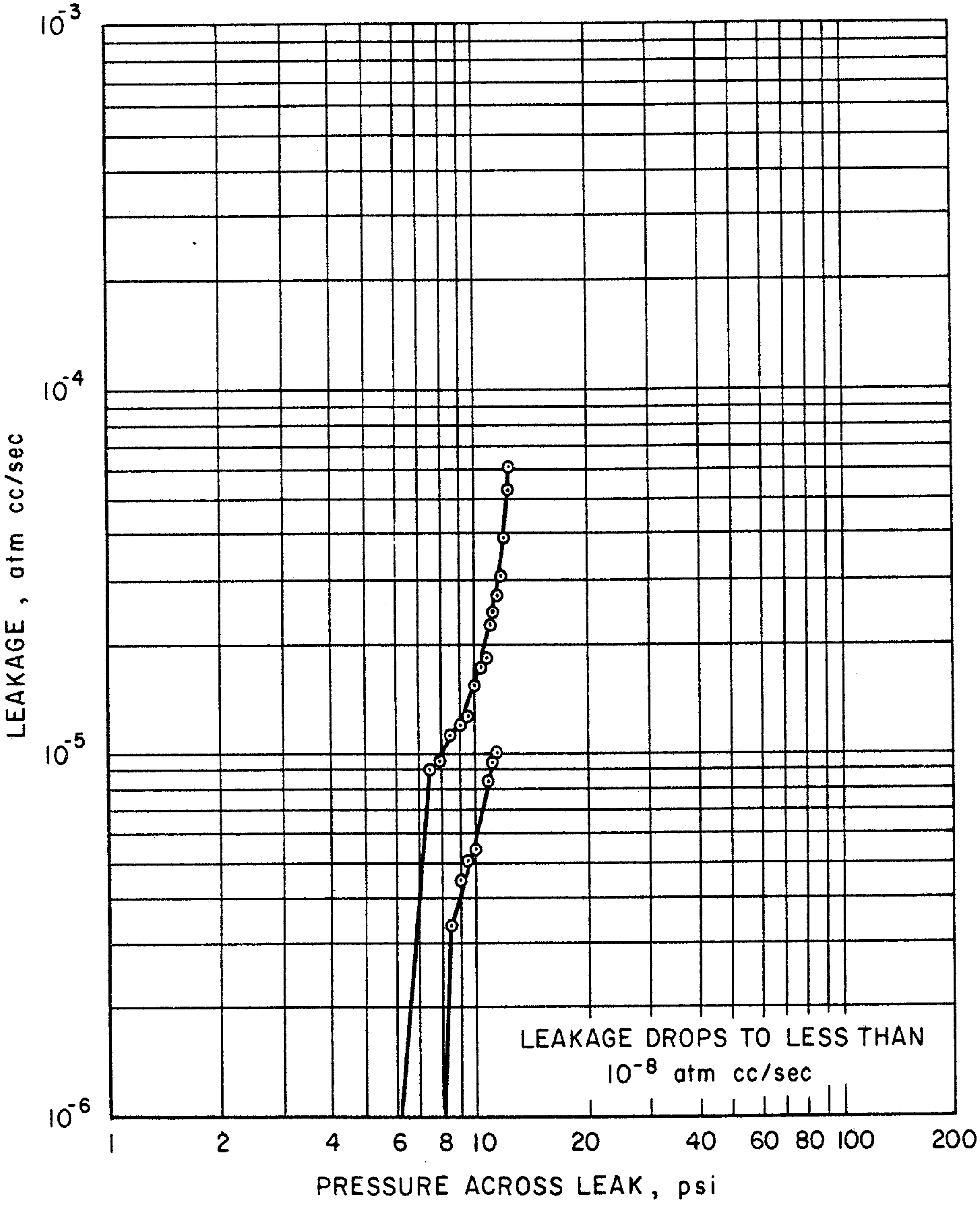

Figure 15. Check-valve Effect in a Hardware Leak. (Reprinted from J.W. Marr, Study of Static and Dynamic Seals in Liquid Rocket Engines, Final Report Phase II, Contract NAS 7-102. (Ref. 45))

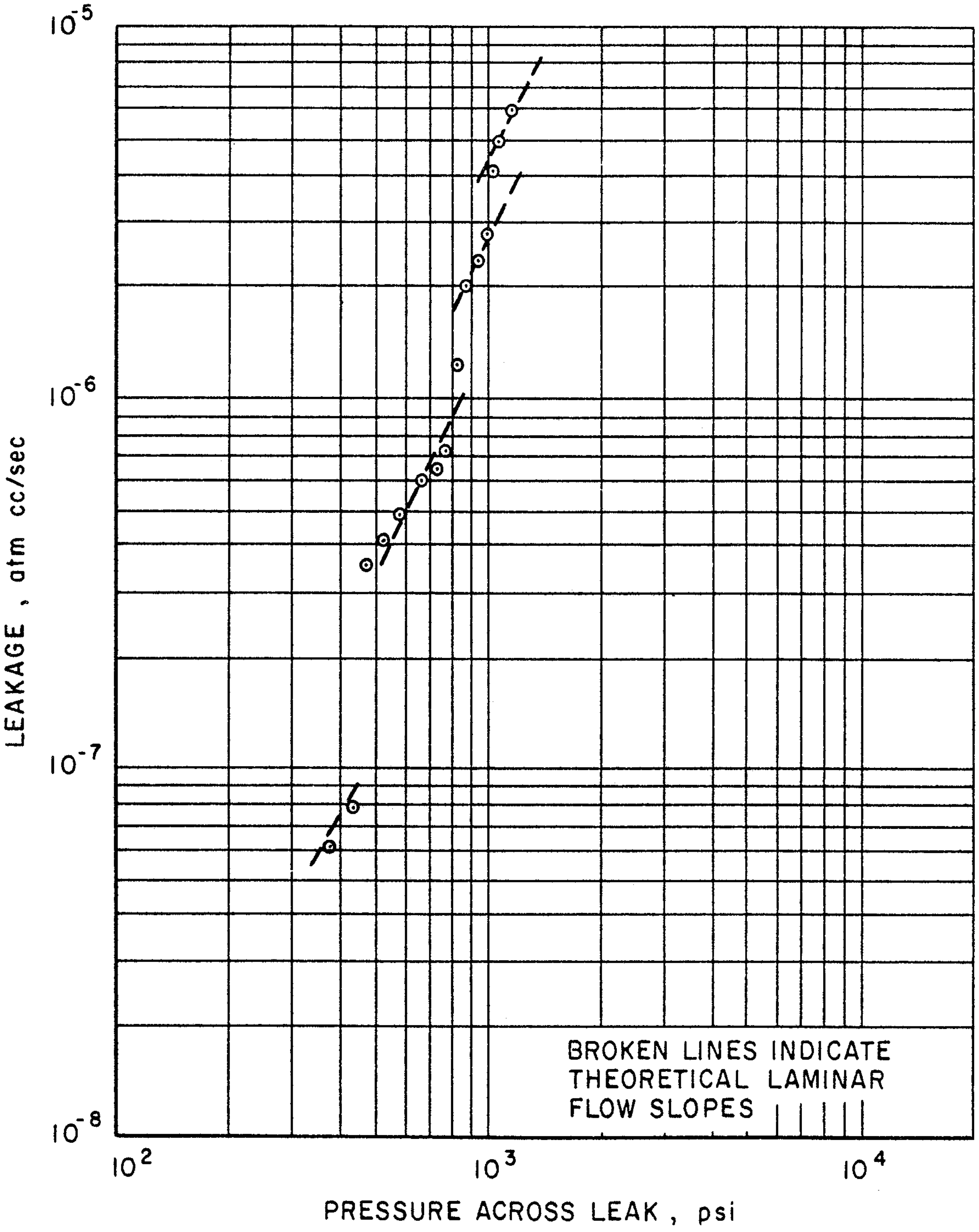

Figure 16. Geometry Change in a Gasket Leak (Reprinted from F. O. Rathbun, Jr., Design Criteria for Zero-leakage Connectors for Launch Vehicles. Report on National Aeronautics and Space Administration Contract NAS 8-4012, for George C. Marshall Space Flight Center, December 1963. (Ref. 46))

leakage quantitatively determined. It was difficult to explain why slope of the pressure-leakage line should be greater than two, since no flow regime would produce such a slope. However, these curves consist of a series of lines with a slope corresponding to that for laminar flow.

Since the increase could result from a permanent deformation of the gasket, an experiment was run using an aluminum gasket too sturdy to be deformed. Figure 17 shows the data obtained during this experiment. During the original increase in pressure, the leakage increased at a rate greater than the square of the pressure increase. However, upon releasing the pressure, the leakage decrease was proportional to the square of the pressure decrease. A second increase in pressure produced an increase which retraced the leakage encountered during the pressure decrease.

It is believed that the original pressure increased cleaned the leakage passages. Further pressure cycling does not effect the maximum leakage. This suggests that whenever possible, leak testing should be done at the proposed operating pressure, in order that potential leaks may be formed and observed.

1.4.4.4. ABSORBED OR SURFACE FLOW LEAKS

The flow of gases and non-condensing vapors through fine capillaries and micropores cannot be dealt with by means of simple methods analogous to those applicable to molecular and laminar flow (Ref. 34). The narrow passages and large surface areas involved cause surface adsorption and surface flow to become important factors. The adsorption may be physical, where only relatively weak van der Waals attractions are involved, or the adsorption may be regarded as chemical, where the surface of the solid provides binding sites for the atoms, and the electronic structure of the solid permits the formation of a chemisorption bond. The nature of the binding sites, the bonds between the gas atoms, and the surface influences the degree of surface migration of the atoms.

The flow along a fine capillary or micropore is assumed to consist of two mechanisms working simultaneously:

1. Molecular flow along the bore of the capillary, whereby molecules are supposed to collide with the wall, re-evaporate, and collide with the wall again without intermolecular collisions, and
2. Surface flow along the wall of the capillary, whereby molecules are adsorbed and diffuse along the surface of the wall.

Both these mechanisms promote gas flow from regions of higher gas concentrations to regions of lower gas concentrations.

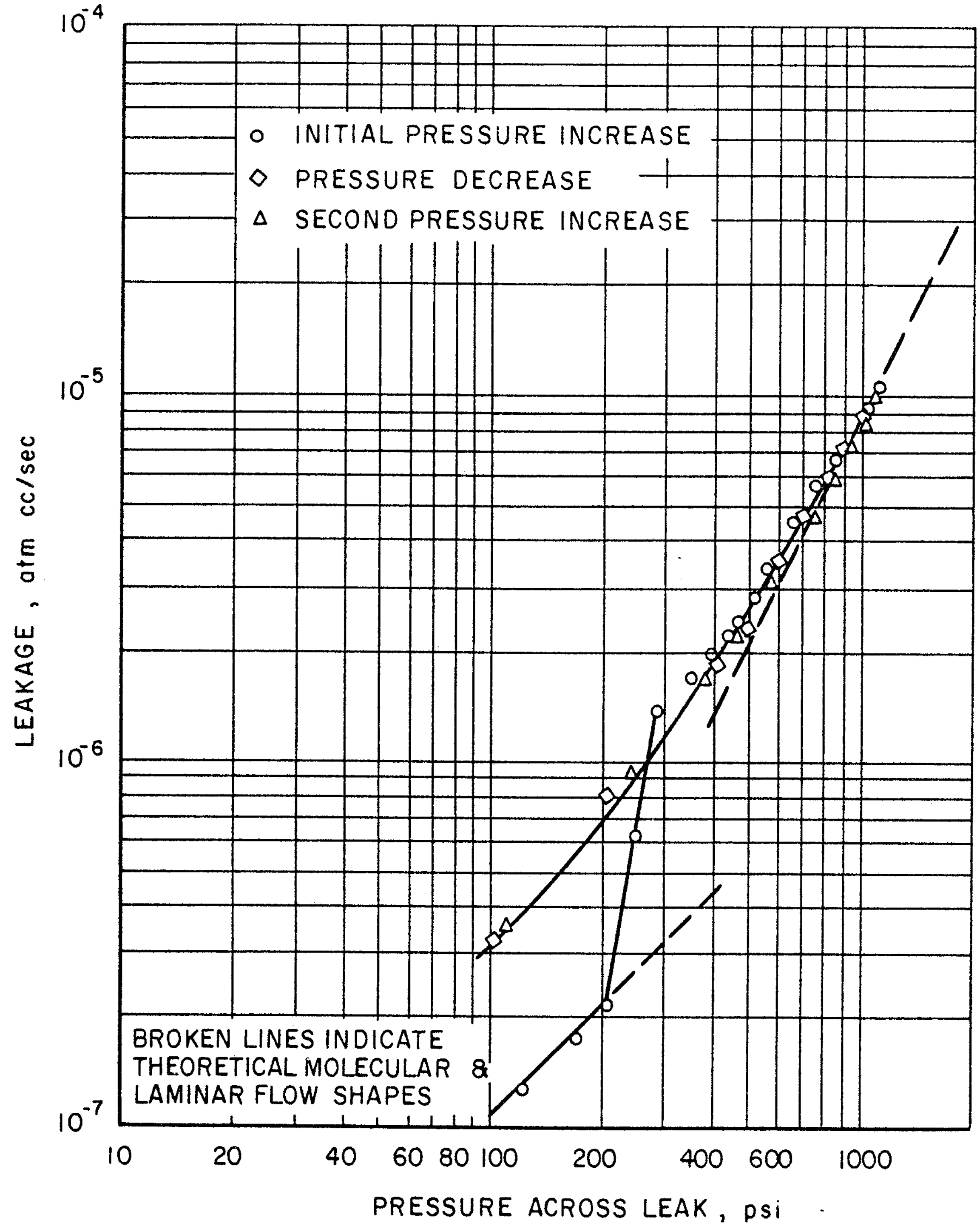

Figure 17. Self-cleaning Effect in Leaks. (Reprinted from F.O. Rathbun, Jr., Design Criteria for Zero-leakage Connectors for Launch Vehicles. Report on National Aeronautics and Space Administration Contract NAS 8-4012, for George C. Marshall Space Flight Center, December 1963. (Ref. 46))

For a given set of conditions, the proportion of molecules that follow the above mechanisms depends on a variety of factors. These factors include:

- The sticking probability (the probability that a molecule striking the surface will become adsorbed)
- The length of time the molecule remains adsorbed (the mean surface lifetime of the molecules
- The coefficient of surface diffusion of the molecules

These features are, in turn, influenced by other characteristics, such as the number of sites occupied by the adsorbed molecules or whether a complete monolayer is involved.

The nearer the properties of a gas approach those of a condensable vapor, the greater the proportion of surface flow. Therefore, a reduction of temperature or an increase of pressure may sometimes promote a total flow in excess of that predicted by the laminar-molecule theory.

Although the final leak rate achieved with a condensable gas will be higher than predicted from flow theory, Hayashi (Ref. 47) predicted that there will be an initial delay of flow due to condensation of the tracer gas on the leak surfaces. This delay is important if a tracer probe technique is used for testing. For example, if butane, a readily condensable gas, is used in a tracer probe, some small leaks will be missed because of the delay caused by the adsorption. Two remedies can be suggested to counter this problem.

1. The use of a non-condensable gas.
2. The use of a detector probe with condensable gases. With use of a detector probe, the gas is continually in contact with the leak and equilibrium is established.

1.4.5. LEAK CLOGGING

1.4.5.1. SURFACE TENSION AND WETTING

Very small holes that may give rise to appreciable leakage rates of gas under high vacuum conditions are sometimes partially or wholly blocked by solid particles having sizes in the micron range, or by liquids as a result of surface tension effects. The blockage may be of a temporary nature and so give rise to misleading test results.

Preferably the system should be kept out of contact with sources that may cause blockage until after proving and rectification have been completed. These ideal conditions cannot always be met in practice. A phenomenon frequently observed is, that if a container to be tested for leaks has at any time after manufacture been in contact with a liquid, no leaks small in size (i.e., 10^{-7} atm cc/sec or smaller) will be found. The small leaks are clogged with liquid because of surface tension. If water has entered a capillary tube, the pressure necessary to force it out of the tube is given by the following equation (Ref. 48):

$$P = \frac{4\theta}{d} \qquad \text{Equation 1.4-33}$$

where θ = surface tension of the liquid usually expressed in dynes per centimeter
d = tube diameter in centimeters
P = pressure expressed in dynes/cm^2 *

A leak with a diameter of 1×10^{-4} centimeter will have a leakage rate for helium approximately $1 \times 10^{-6} \ell$ atm/sec. If water, with a surface tension of 72 dynes/cm, enters such a leak, the pressure required to force it out is approximately 2.8 atmospheres. Since the pressure differential during leak testing is only 1 atmosphere, this leak is essentially clogged by the use of water. In actual conditions, clogging is more likely to take place than in the above example. Most leaks are neither circular nor regular in diameter. They usually contain constrictions and occasionally consist of slits. The rate of flow in a leak is approximately proportional to the leak cross-sections.

If clogging were to take place, the pressure required to remove a liquid is inversely proportional to the smallest dimension in the cross-section. Therefore, clogging of an orifice or of a slit is more likely to take place than may be assumed from the above example. If the leak in the above example were in fact a slit with the ratio of width to height of 10 to 1, the pressure necessary to remove water from such a slit would be approximately 28 atmospheres. Leakage would be of the same order of magnitude as mentioned in the above example.

Clogging by surface tension can occur in several different ways. As a first breakdown, the clogging can be divided into two forms - with liquid in the system and with gas in the system after it has been in contact with liquid.

With liquid in the system, clogging can occur if the surface of the leak is not wet by the liquid. In that case, no liquid will flow even though a hole exists. On the other hand, if the liquid wets the leak, the equilibrium flow rate through the leak will be that predicted by flow theory. This type of clogging can not be predicted or depended on as a permanent phenomenon, since the wetting properties of the leak might be different from the bulk surface and might change with oxidation, weathering and other treatment.

After the liquid has been removed from the system, clogging can occur from liquid remaining in the leak. It is immaterial

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*One atmosphere is equal to 1.063759×10^{-6} dynes/cm^2

whether the liquid wets the surface. Clogging as a result of the liquid surface tension occurs in either case.

Unfortunately, clogging is not of a permanent nature. If the temperature of the container is raised, the surface tension of the liquid decreases and quite possibly the liquid inside the leak evaporates.

Leakage much smaller than 10^{-8} atm cc/sec is rarely seen. Such leakages are in the range of detectability, but are a rare phenomenon. They are found on new systems which have never been in contact with liquid. These leaks have to be carefully treated or they will clog during normal handling. It may be calculated that a leak 10^{-8} atm cc/sec has a diameter in the order of 10^{-5} centimeter. To place this number in dimensions of atomic size, its diameter is about 1,000 Angstrom units.* Molecules of contaminants (for example, either hand or machine oil), are in the order of 100 Angstroms in length. Should these molecules be adsorbed in the leak, they would reduce the size of the hole and therefore reduce the leak rate to a point of apparent clogging. According to the equation for molecular flow (Equation 1.4-14), the flow which should predominate in this range is proportional to the cube of the leak diameter. Therefore, a small decrease in leak diameter will produce a drastic decrease in leakage.

1.4.5.2. PRESSURE REMOVAL AND EVAPORATION

A pressurizing test may suffer some restriction in its application to certain types of vacuum equipment, because the design may permit use of only small over-pressures. In these applications the presence of water may easily block a leakage path owing to its high surface tension. For example, when a particular capillary contains water that wets the capillary wall, this capillary will be completely blocked against a gas pressure difference of 1 1/2 atm. A thin mineral oil would be more easily moved in such a capillary, requiring only about 1/2 atm. Even though the applied pressure difference may be sufficient to move the oil, the viscosities of oils are relatively high so that, in practice, an oil may usually be regarded as permanently blocking a capillary of any appreciable length. This is due to the extremely slow rate of flow of oil. Consequently, an oil should never be used for pressure testing before leak testing.

On the other hand, water will evaporate much more readily into a vacuum than would a mineral oil because of the higher vapor pressure of water. The rate of evaporation is accelerated if the temperature is raised. For leak testing evacuated systems, a number of points should be considered.

- A preliminary water pressure test is permissible, but not recommended.

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*One centimeter is equal to 10^{8} Angstrom units

- Oil should never be used for over-pressure test.

- A compressed air test should never follow a water pressure test before vacuum testing for leaks. This is because the residual water that is held in small capillaries may be forced back by the compressed air into larger voids. Under vacuum conditions, the liquid would evaporate slowly through the capillary restriction.

- Even a preliminary water pressure test is inadvisable for ultra-high vacuum systems. In such a system detection of very small leaks through fine capillaries becomes essential and the time required to ensure clearance of such capillaries (by evaporation of the residual water) may become unduly prolonged.

To summarize, when a liquid is in a capillary, the liquid that has the lower surface tension is more readily moved by a gas-pressure difference, while a liquid that has a higher vapor pressure is more readily evaporated out. As a consequence, capillaries that may be blocked by water under the conditions of an over-pressure test, may often be readily cleared by evaporation of the water into a vacuum. Moreover, apparatus which must undergo a rigorous check for leaks must be dried in a vacuum because small leaks are readily clogged by the water adsorbed on the walls.

Following the general rule, it is preferable that the vessel or component to be leak-tested should be kept out of contact with sources that may cause blockage, until after leak-testing and leak-rectification have been completed.

Solid particles in the millimeter size-range are not, in general, a source of trouble. But solid particles in the micron range may enter and block a capillary temporarily. They may subsequently be displaced, giving rise to an intermittent leak that is difficult to establish and locate. Blockage by solid particles may occur, for example, if the fluid used in connection with magnetic crack-detection work is applied before testing for leaks. Magnetic fluid contains iron particles, of which the greater proportion are usually in the 2 1/2- to-10-micron size-range, and are, therefore, comparable with the cross-sectional dimensions of the capillaries considered here.

One of the disadvantages of porosity will be understood by considering the effect of a hypothetical condition of a thousand capillaries 1/4 inch long situated close together in parallel, each having a radius of 10^{-5} centimeter. The total leak rate through this assembly would be approximately 1.3×10^{-7} torr liter/sec-- that is, about the same order of leakage as a single capillary of 10^{-4} centimeter radius. However, if water has access to the porous assembly, the pressure difference required to remove it from the capillaries would be (from Equation 1.4-33)

$$\frac{2 \times 72}{1 \times 10^{-5}} = 14 \text{ atm or } 210 \text{ lb/in.}^2, \qquad \text{Equation 1.4-34}$$

that is, ten times the pressure difference needed to remove such blockage from a capillary of 10^{-4} centimeter radius.

Small holes in mild steel vessels or components may become permanently blocked by "rusting-up" caused by moisture. In practice it is usually impossible to prevent atmospheric moisture from entering capillaries. Most metals are attacked, more or less, by water in the presence of air. This action gives rise to an oxide film that has a greater specific volume than the metal. This effect applies particularly to mild steel, where the oxide film has a specific volume about seven times greater than that of the parent metal. Consequently, the smaller capillaries, which have the greater tendency to retain water, readily "rust-up." Stainless steel is less likely to be affected in this way.

When a liquid blocks a capillary its removal from the capillary may be influenced by any one, or a combination, of the following factors:

- Wetting combined with surface tension, which resists motion.
- Viscosity, which controls the tendency to flow.
- Vapor pressure, which controls the tendency to evaporate.

Thus, a thin mineral oil in a capillary can be moved with a smaller pressure difference than when water is present, owing to the lower surface tension of the oil. Under similar conditions, a very much longer time would be taken by the oil to pass out of the capillary, because of its greater viscosity compared with water. Burrows (Ref. 34) states the equation needed to calculate the evaporation rate and liquid flow in a capillary.

1.4.5.3. SAFETY

Although the prevention of clogged leaks dictates leak testing be done prior to contact of the system with liquid, the need for safety might overrule this procedure. Pressurizing a system with a liquid does not create the explosion hazard involved with gases under high pressure. Therefore, safety requirements may dictate pressure-testing of a system with a liquid before gases are introduced for leak-testing.

The amount of energy stored in a pressurized tank is a function of the quantity of gas contained in the vessel. Because of this, a high-volume, low-pressure vessel can contain the same stored energy as a low-volume, high-pressure vessel and, therefore, present a hazard of similar magnitude. Because of the similarity in explosive effect, it was decided to equate the energy available in the tank to that in a quantity of TNT (Ref. 49).

The hazard potential or TNT equivalent of a pressurized vessel is determined by the calculation of the amount of energy available from the explosive expansion of a volume of compressed gas (Ref. 50).

$$E = 454 \left[\frac{V \left(\frac{P_1}{P_2}\right)\left(\frac{T_1}{T_2}\right)}{359} \right] RT \ln \left(\frac{P_1}{P_2}\right) \qquad \text{Equation 1.4-35}$$

where E = energy (calories),
P_1 = original (absolute) pressure of the compressed gas,
P_2 = final or atmospheric pressure,
R = molar gas constant (1.987 cal/gm-mole $^{\circ}$K for an ideal gas),
V = pressure vessel volume (ft^3),
T_1 = ambient air temperature ($^{\circ}$K),
T_2 = temperature of the compressed gas ($^{\circ}$K).

The standard for energy release is set at 1,000,000,000 cal/ton of TNT or 1,140 cal/gm.

Once the energy equivalent has been established, minimum safe distance for non-operating personnel can be computed, using over-pressure data from reference TNT explosions (Figure 18).

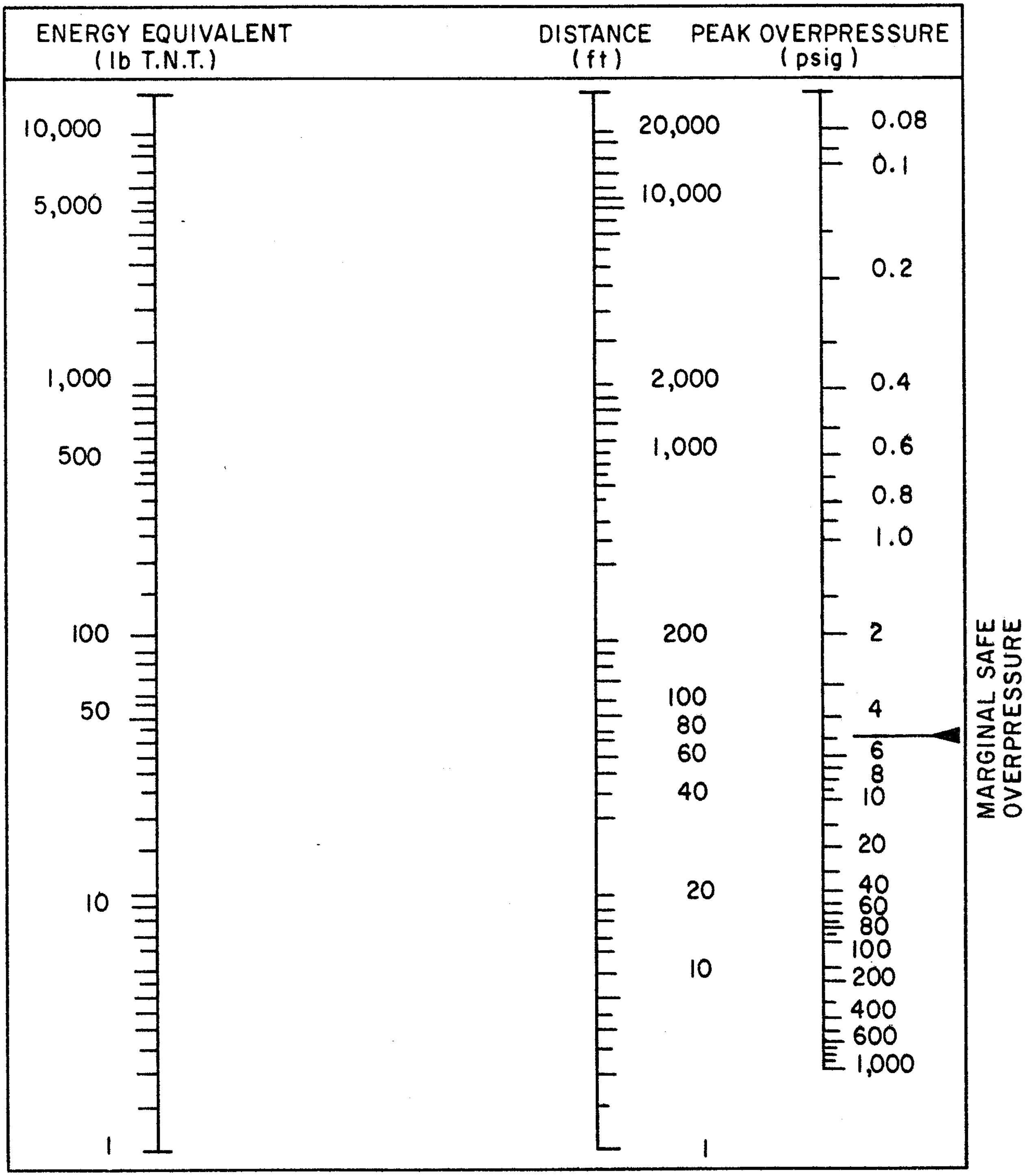

Figure 18. Nomograph for Calculating Magnitude of Blast Hazard from Equivalent Weights of TNT. (Reprinted with permission from R.E. Olson, "Safety Hazards of High Pressure Leak Checks", paper Presented at 56th. Air Force Industry Conference on Missile Leaks and Spills, Los Angeles, Calif.)

1.4.6. GENERAL REFERENCES

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Section 1.5.

METHODOLOGY OF TESTING

1.5.1. PLAN FOR TESTING

1.5.1.1. ACCESSIBILITY

If a system is to need leak testing, such a requirement should be given weight during the design period. Too often a system is completely constructed before any thought is given to necessary leak testing. At the end of construction, it may be impossible either to pressurize the system with tracer gas or to make connections to the system for evacuation. Although leak testing may be accomplished without either pressurizing or evacuation, other procedures are difficult and expensive.

Therefore, if leak testing is to be required, a system should be accessible for either:

1. Pressurizing with tracer gas, or

2. Evacuation

A suitable connecting point in any system may be the connection for the vacuum gage.

Well-designed test connections should be used. The connections can be a major source of difficulty on account of leakage, excessive tracer gas contamination, and dirt. Minimum amounts of elastomers and plastic should be used. Rubber tubing is particularly bad because it absorbs tracer gas, which is difficult to remove by pumping and also eventually becomes contaminated with other materials. Therefore, only short lengths of rubber tubing should be used. A good, low-vapor-pressure grease should be applied in a light film to gaskets. Any excess should be removed, since large quantities of grease act as a source of tracer gas contamination and also as a dirt catcher. The system may become so dirty because of excess grease that a good vacuum cannot be attained.

1.5.1.2. LEAKS IN SERIES

It has long been recognized (Ref. 51) that when designing a device which will require leak testing, no design which may develop voids in series with possible leak paths is acceptable. For example, if two heavy plates are to be welded together, it is best to make either a full penetration weld or a partial penetration weld from only one side. It is undesirable to allow partial penetration welds from opposite sides since a void can be left between the welds. It would then be difficult to check and assure that there is not a leak through both welds since the speed of response of the leaking system may be very slow.

The leak path (Figure 19) may be considered one or more chambers connected by very fine capillaries. The pressure in this chamber (the intermediate volume) is atmospheric immediately after pressurization of the system. The driving force, (or pressure difference) between the chamber and the outside atmosphere is zero at this time. As the inner capillary, (between the system and the chamber) conducts tracer gas to build up the pressure in the chamber, the pressure difference between the chamber and the outside atmosphere increases. With this increased pressure difference, there is a slow increase in the flow rate. Since the leak detector can only measure a specific minimum flow rate, no leak will be detected until this minimum is reached. This minimum flow rate will correspond to a particular pressure difference, depending on the size of the capillaries through which the tracer gas passes.

Consideration of the time delay in leak detection is important when an intermediate volume is present in the total leak path. A time lapse effect may prevent a leak from being detected. A review of leak test procedures to establish the time lapse to be expected between component pressurization and measurable leakage is necessary. This can be accomplished by solving the derived equations (Ref. 51) for typical values of parameters associated with a particular configuration.

In any system construction, sealed voids should not be allowed to form. If a volume is to be enclosed, wholly or partially, sufficient openings should remain to allow rapid evacuation or gas diffusion from the enclosed volume. Where such gas transfer can be accomplished only through very limited openings, much time may be wasted in leak testing adjacent volumes. Instructions for welding without enclosing sealed voids may be found in current publications (Ref. 52 and 53)

1.5.1.6. COMPONENT TESTING

When a vacuum system is to be constructed, it is advisable to test each individual item for leakage prior to assembly, and then to start construction of the system at the pumps. If possible, the vacuum-tightness of the system should be checked after each new item has been added. In other words, each newly-made vacuum joint should be tested as construction proceeds.

In constructing any system, it is easier to leak test each component before assembly rather than to test after assembly. If this procedure is followed, any test of the complete system is a test of the joints made in assembly. Any leaks indicated by the system test will be located only in the joints.

It is always good practice in repairing leaks found during leak hunting to make permanent repairs whenever possible. Temporary repairs should only be made if the system must be operated for a limited length of time before permanent repairs can be made. A permanent repair means reworking the part involved or replacing the part. Some common sources of leaks and

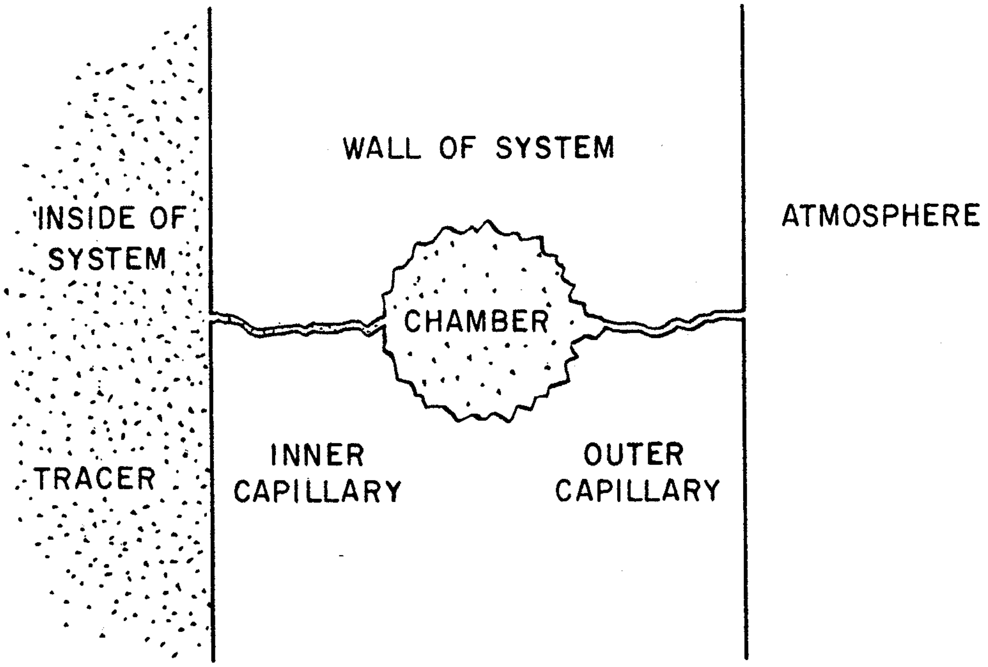

Figure 19. Sketch of Typical Leak Paths in Series.

possible methods of repair are indicated below (Reprinted with permission from Vacuum Technology, by A. Guthrie; John Wiley and Sons, Inc., New York, 1963, pp. 500-501):

1. Static gasket seals.

   Tighten seal but not too much.

   If tightening does not work, shut down the system and examine the gasket and gasket surfaces.

   Replace any damaged gasket and smooth rough surfaces (fine emery cloth).

   When the gasket is not damaged, clean (acetone, etc.), coat with light film of good quality vacuum grease (when permitted), and reassemble seal.

   Do not use sealing materials such as Glyptal to stop the leak. This procedure is temporary and the gasket can't be used again.

2. Movable, gasketed seals (Wilson, chevron, etc.).

   Add a small quantity of good quality vacuum grease to the moving member and operate this member a few times through the seal.

   If vacuum grease doesn't work, try tightening the retaining rings on the seal.

   If tightening doesn't work, dismantle the seal and examine the component parts. Replace all damaged parts.

3. Flare fittings (and similar metal-to-metal seals).

   Tighten the compression nut moderately. Too much tightening is likely to twist the tubing passing through the fitting.

   If tightening doesn't work, take the joint apart.

   Try annealing the copper flare.

   If annealing doesn't work, use a thin coating of a suitable sealant (Glyptal, etc.) on the surfaces that make contact.

4. Soldered, brazed, and welded joints.

   For temporary repair, use a cement. This method works for small leaks where the diffusion pumps are in their operating range. A "thin" cement (clear glyptal, Eastman Kodak Resin 910, etc.) can be used for the smallest leak. For somewhat larger leaks use a "thicker" cement such as red Glyptal.

- For permanent repair, rework the joint or replace the part.

5. Leaks through metal parts.

   - For temporary repair, use a cement as in the case of soldered, brazed, and welded joints.
   - For permanent repair, rework the part involved (as appropriate) or replace the part.
   - Peening the leak is not reliable.

6. Glass-to-metal seals.

   - A temporary seal can be made with a cement or wax (heat part appropriately).
   - Rework wax seals. Such seals are not recommended.
   - For permanent repair, replace the seal.

7. Glass-to-glass joints, cracks, and pinholes in a glass system.

   - For small leaks, use a cement (at room temperature) or heat the glass and apply a suitable wax (picein, sealing wax, etc.).
   - For a large leak, rework the glass or replace the part of the system where the leak is located.

1.5.1.4. SAFETY CONSIDERATIONS

Four hazards to be taken into account when designing the leakage test are: the flammability of the tracer gas, the possibility of asphyxiation or poisoning and the danger of explosion or implosion of a pressurized system. As long as the designer is aware of these considerations from the beginning, it is possible to test with little inconvenience or danger. Included in Appendix A, along with physical properties of tracer gases, is a list of the safety precautions to be observed with the following gases: ammonia, argon, carbon dioxide, dichlorodifluoromethane, helium, hydrogen chloride, hydrogen, krypton, methane, neon, nitrogen, nitrous oxide, oxygen and sulfur dioxide.

1.5.1.4.1. Flammability

Hydrogen, methane, butane, and many of the liquids used in leakage testing are flammable. When these tracer fluids are used in leakage testing, they are usually mixed to some extent with air. These explosive mixtures can become dangerous and procedures should be taken to provide adequate ventilation and to prevent accidental ignition.

The following precautions will apply in all cases of flammable gases. Use of hydrogen enhances the risk, and the precautions given below are essential. (Ref. 6)

1. Before admitting hydrogen the system under test should be evacuated or purged with nitrogen. This will avoid an air-hydrogen mixture anywhere in the explosive range (4 per cent to 74 per cent of hydrogen in air, by volume). The same should be done with a rigid hood. With flexible hoods the volume between hood and vessel under test can usually be made very small - much less than the 10 cubic feet referred to in 2 below. If this is done, evacuation or purging is not essential.

   A hydrogen/nitrogen mixture is safer than pure hydrogen, if it can be used.

2. Testing should be done in a well-ventilated place, isolated as far as practicable from other processes. A room with a high roof, adequately vented at its apex, and with enough low-level air inlets, is desirable. A small room, with low roof, and a minimum of openings for ventilation, should not be used for testing with hydrogen.

   Special precautions will be necessary when the volume between the hood and system under test approaches 10 cubic feet, as there is then risk of a major explosion. Safety can then only be insured by testing the system in a blast enclosure, or cell. This should have strong walls and a light roof; persons should not be allowed to enter during a test. For safety, and to avoid having to provide a test cell unnecessarily, the volume between a hood and the enclosed system should be as small as possible.

3. Test areas, particularly test cells, should be free from obvious sources of ignition. Smoking must be prohibited, particularly when hydrogen is used. Electrical equipment may be a difficulty. If there is a possibility that, in the event of a leak, such equipment will be in an explosive atmosphere, then either the equipment must be repositioned outside the danger area, or special equipment used. Such special equipment is either pressurized with inert gas or is intrinsically safe.

   A discharge of static electricity is a possible cause of ignition. Therefore all metal parts liable to become charged should be grounded, and any flexible non-metallic hood should be made of 'anti-static' material. This would mean that the resistance of the leakage path from any point on the hood would not exceed 10^7 ohms. It is advisable for personnel when testing with hydrogen to avoid wearing clothing liable to produce static charges, and to wear shoes with conducting soles.

Another precaution is the use of reduced sparking tools.

4. If possible, hydrogen cylinders should be in a storage area separate from any occupied building. The storage area should be well ventilated. Also permanent piping should be provided to convey hydrogen to the test area.

   The above precautions are also relevant where other flammable tracer gases are used; although hydrogen easily presents the greatest risk. Where a large system is being tested, or large volumes of hydrogen used, it may be advisable to provide monitoring equipment giving a continuous indication of the hydrogen in air content in the test area. Intrinsically safe detectors are available.

1.5.1.4.2. Asphyxiation

Most tracer gases are not toxic. However, if a question exists about the toxicity of any particular gas, a competent authority should be consulted (Ref. 8). Toxicity of some tracer gases is included in Appendix A.

None of the tracer gases will support human life, i.e., if they replace oxygen in a volume, this volume cannot be entered without proper respiratory equipment. In this case, proper equipment consists of a gas mask which contains its own oxygen supply.

The oxygen required for breathing might be removed from an area accidentally. For example, if one of the halogenated hydrocarbons is used as a tracer gas, it will stagnate and settle to the lowest area in the system. If an operator is attempting to use a detector probe in this low area, the tracer gas which settles may eventually displace enough of the air to produce asphyxiation. In this case, adequate ventilation is necessary. This ventilation, however, must be performed carefully. If the tracer gas is dispersed too rapidly from the place where it is escaping from the system, leak location will be difficult.

1.5.1.4.3. Explosion

If the system is pressurized with tracer gas, rupture of its walls can produce considerable damage. This has been discussed in Section 1.4.5.3. That section describes in detail the damage which may occur as the result of rupturing systems. It would seem that if the system being pressurized is small, little precaution is necessary in pressurizing. However, the damage from rupture of a gas-filled volume results from the total amount of gas. Therefore, both a small system at high pressure and a large system at low pressure may be equally dangerous.

Large systems need proof-testing prior to leakage measurement. The proof-test may be performed by pressurizing with gas to a high pressure while the test area is evacuated, or by a hydrostatic test using water (Ref. 55). The disadvantage of the gas proof-test is that if the system bursts during testing, considerable damage can result. On the other hand, should hydrostatic testing be performed, any small leaks in the system will be clogged with water. Therefore, if at all possible, hydrostatic testing should not be performed on systems where allowable leakage is less than 10^{-6} atm cc/sec. The reasons for this are discussed in Section 1.4.5. It should be pointed out that the Boilermaker's Code specifies that all vessels should be proof-tested to 1.5 times the operating pressure (Ref. 55). Such a requirement is a good safety rule in proof-testing systems prior to leakage measurement.

In pressurizing a system, a pressure regulator fitted with a safety over-pressure device should be included, so that a pressure in excess of the design pressure can never be applied to the system.

1.5.1.4.4. Implosion

Although they are not pressurized, glass bell jars which are evacuated can be a dangerous source of flying glass from implosions. Should a crack occur in the bell jar, unequal force will shatter it. The pieces of flying glass, propelled by a pressure difference of approximately 15 pounds per square inch will travel great distances unless they should happen to meet a safety shield or glass coming from the other direction. This is particularly serious when the capacity of the vessel exceeds about one cubic foot. For this reason all evacuated bell jars should be enclosed in some safety shield.

Safety shields should be used on bell jars under all conditions. One mistake often made with bell jar safety shields is not using them unless the system is to be evacuated to a high vacuum. The increase of the pressure on the bell jar going from one torr to 10^{-3} torr is negligible. The major part of atmospheric pressure is exerted on the jar when rough evacuation takes place. The increase in pressure difference by evacuating the bell jar further is very small.

1.5.1.5. EFFECTS OF GAS PROPERTIES

Various properties of tracer gases affect the results of leak testing. Gas density has an effect on the tendency of the tracer gas to stratify or form layers in the test volume, giving uneven readings for various leak locations. The diffusion rate of a tracer gas has an important effect on test results, particularly if there are restrictions or long, narrow, blind passages or crevices in the test volume. Variations in tracer gas concentration can give varying leak indications.

1.5.1.5.1. Effect of Tracer Gas Concentration

In many leakage measurements, it is desirable or necessary to dilute the tracer gas used. Diluted tracer gas might be dictated by:

1. Expense involved in use of pure gas.
2. More linear or more stable response obtained at lower gas concentration.
3. Pure gas producing a sensitivity much greater than needed.
4. Danger of fire or explosion with a flammable tracer gas (a dilute gas mixture reduces danger).
5. Inability to completely evacuate the system prior to filling with tracer gas, so that a dilute tracer is obtained during the pressurizing process.

Concentration of tracer gas in the system depends on its partial pressure. This may be expressed by:

$$P = P_A + P_B + P_C + \dots \qquad \text{Equation 1.5-1}$$

where P = system pressure
$P_A\ P_B\ P_C$ = partial pressure of the individual gas species.

Using the above equation, the fractional concentration of gas A (N_A) in the system is:

$$N_A = \frac{P_A}{P} = \frac{P_A}{P_A + P_B + P_C + \dots} \qquad \text{Equation 1.5-2}$$

It is possible to synthesize a gas mixture by pressurizing a constant volume to pressures of the individual gases. Alternatively, a synthetic mixture may be made by combining volumes of the gases at the same pressure.

Most leak detectors are to some extent specific to a particular tracer gas. As a general and approximate rule, the response of a leak detector to a measured dilute tracer gas concentration is proportional to the concentration. For example, if a one-percent tracer gas leak is measured and the leakage is determined as 1×10^{-6} atm cc/sec, the total leakage at that time is approximately 1×10^{-4} atm cc/sec. The magnitude and direction of any deviation from the above rule depends on the predominant flow mode in the leak. The expected predominant flow modes are discussed in Section 1.4.3.1.

If the leakage is laminar, the flow will be inversely proportional to the viscosity of the gas mixture. The mixture will have a viscosity proportional to the viscosity of the individual species in a relation:

$$\eta_{mixture} = N_A \eta_A + N_B \eta_B + N_C \eta_C + \cdot \quad . . . \text{Equation 1.5-3}$$

where η = viscosity
N = fractional concentration of the gas

The leakage of pure tracer gas may be calculated from the relationship:

$$Q_{pure} = \frac{Q_{tracer}}{N_{tracer}} \frac{\eta_{mixture}}{\eta_{pure}} \qquad \text{Equation 1.5-4}$$

where Q is the leakage as measured. In most cases, the calculations are simplified by the fact that the viscosities of most gases are similar (Table 4, Section 1.4). Therefore, the measured leakage for laminar flow is approximately related to the total leakage by the equation:

$$Q_{pure} = \frac{Q_{tracer}}{N_{tracer}} \qquad \text{Equation 1.5-5}$$

If the flow in the leak is molecular, leakage of the tracer gas will be inversely proportional to the square root of the molecular weight of the gas. The two gases will flow through the leak at rates independent of each other. Therefore, a change of concentration can be obtained by leakage. Since flow is proportional to concentration, the flow of a pure tracer gas can be calculated from the measurement of leakage of the mixture:

$$Q_{tracer} = \frac{Q_{mixture}}{N_C} \qquad \text{Equation 1.5-6}$$

Using the molecular weight relationships, the flow of any other gas may be calculated

$$Q_A = Q_B \sqrt{\frac{M_B}{M_A}} \qquad \text{Equation 1.5-7}$$

where M = molecular weight

1.5.1.5.2. Stratification

If a tracer gas is added to air already in the system, a uniform mixture is not readily achieved. The tracer gas will settle towards top or bottom of the system depending on its density. This stratification is more pronounced with high-molecular-weight gases and with gases of a low diffusion coefficient.

Precautions should be taken to mix the tracer diluent gas during pressurization of the system. This may be done by providing some means of circulating the air within the system.

Alternatively, both the tracer and diluent gases may be added simultaneously through a rake or screened aperture. There should be no pooling problem inside the system, provided precautions are taken to mix the tracer thoroughly with diluent gas in pressurization of the system. The equilibrium distribution law gives the ratio of concentration at the top of a tank relative to that at the bottom as:

$$C_h/C_o = \exp(-Mhg/RT) \qquad \text{Equation 1.5-8}$$

where M = molecular weight
h = height of tank
R = universal gas constant
T = absolute temperature
g = local gravitational acceleration

1.5.1.5.3. Diffusion

A tracer gas of low diffusivity gives the advantage of easy leak location since the concentration of tracer gas builds up at the leak exit. This allows detection by a probe and location of the leak. With low diffusivity, gas does not leave the leak location at a rapid rate.

A tracer gas of high diffusivity is necessary to fill cul-de-sacs or blind passages within a reasonable time. A low diffusion rate would not allow tracer gas to traverse a tortuous leak passage, making leak detection unreliable.

The magnitude of the blind-duct problem is demonstrated by Equation 1.5-9.

$$t \approx \frac{L^2}{\pi^2 K} \ln \frac{4}{\pi} \frac{1}{1-N_L/N_o} \qquad \text{Equation 1.5-9}$$

where K = diffusion coeff, feet^2/hour
L = passage length, feet
N_o = tracer concentration in system
N_L = tracer concentration at end of duct
t = time, hours

Time calculated by the above equation is proportional to system pressure. Therefore, if the system pressure is doubled, the time required to reach a desired concentration is also doubled. Table 11 gives the diffusivity of certain gases used in leak detection. Figure 20 is a plot of diffusion time in a blind duct for a number of tracer gases.

Two procedures may be used to fill a duct with tracer gas. If the duct can be opened (perhaps by a valve) at the end remote from the gas source, filling the duct with tracer gas will be expedited. The other procedure, which is much more difficult, is to evacuate the system prior to admission of tracer gas.

Table 11

DIFFUSIVITY OF TRACER GASES IN AIR
(1 ATM, 0 C)

Gas	Chemical Formula	Molecular Weight	Diffusion Coefficient* Sq.Ft.per Hr.
Acetylene	C_2H_2	26.0	0.55
Ammonia	NH_3	17.0	0.66
Argon	A	39.9	0.61
Benzene	C_6H_6	78.1	0.30
Butane	C_4H_{10}	58.1	0.33
Carbon dioxide	CO_2	44.0	0.52
Carbon disulfide	CS_2	76.1	0.36
Carbon monoxide	CO	28.0	0.67
Carbon tetrachloride	CCl_4	154.	0.28
Ethane	C_2H_6	30.1	0.49
Ethyl alcohol	C_2H_5OH	46.1	0.38
Ethylene	C_2H_4	28.0	0.52
**Halogenated hydrocarbon F-11	CCl_3F	137.	0.30
Halogenated hydrocarbon F-12	CCl_2F_2	121.	0.32
Halogenated hydrocarbon F-21	$CHCl_2F$	103.	0.33
Halogenated hydrocarbon F-22	$CHClF_2$	86.5	0.37
Halogenated hydrocarbon F-112	CCl_2F-CCl_2F	204.	0.25
Halogenated hydrocarbon F-114	$CClF_2-CClF_2$	171.	0.28
Helium	He	4.00	2.7
Hydrogen	H_2	2.02	2.6
Hydrogen sulfide	H_2S	34.1	0.53
Krypton	Kr	83.8	0.51
Methane	CH_4	16.0	0.72
Neon	Ne	20.2	1.1
Nitric oxide	NO	30.0	0.70
Nitrogen	N_2	28.0	0.68
Nitrous oxide	N_2O	44.0	0.52
Oxygen	O_2	32.0	0.68
Propane	C_3H_8	44.1	0.39
Sulfur dioxide	SO_2	64.1	0.42
Water	H_2O	18.0	0.85
Xenon	Xe	131.	0.42

*Values calculated from empirical equation of J.C. Slattery and R.B. Bird (AIChE Journal, Vol. 4, No. 2, 1958, pp. 137-42.

**ASHRAE Standard Designation

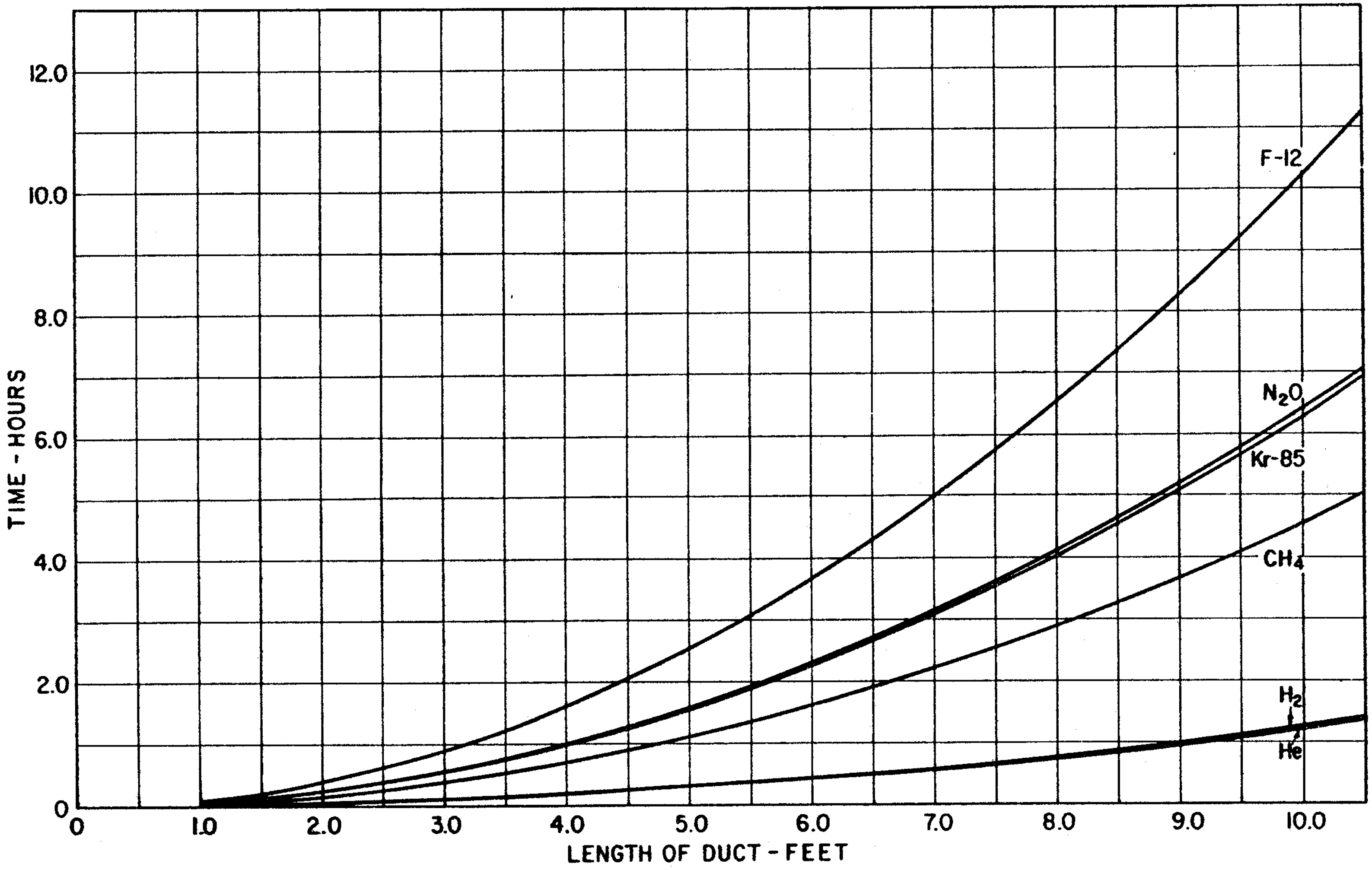

Figure 20. Diffusion of Tracer Gases in Blind Ducts. Time required to reach one-tenth the original tracer gas concentration at end of duct.

1.5.2. PROCEDURAL TECHNIQUES

The two types of procedures are discussed in this section: leakage measurement and leak location (by probe technique). Although there are many similarities in principle, each procedure is best followed with allowance for individual equipment and operation perculiarities. Many of these are demonstrated on the following pages.

1.5.2.1. LEAKAGE MEASUREMENT

On large systems where rapid testing of a large number of objects is required, it has been found that testing time can be reduced by the use of a testing hood to surround the entire test object with an atmosphere of tracer gas. This eliminates probing time and gives a response proportional to the total leakage if several leaks exist. The possibility of missing leaks by careless probing is also removed. After locating leaky test objects the hood may be removed and the leaks located accurately by probing with a small jet of tracer gas. A valuable adjunct to the testing hood is a thermal conductivity gage, calibrated to read directly the concentration of tracer gas in the hood.

1.5.2.1.1. Hood Testing

In hood testing, the form of hood depends on the task in hand. In a semi-permanent assembly designed for routine over-all testing of a large numbers of components, the hood may be a metal chamber or bell-jar. When testing one object of a type, almost any item can be accommodated in a polyethylene bag. It can be dangerous, however, to use flammable or explosive tracer gases in conjunction with plastic hoods which can acquire an electrostatic charge.

A typical hood setup is shown in Figure 21. For small objects several manifolds are attached to one pump-leak detector combination; this allows the objects to be setup or taken down on one manifold while the units on the other are under test. For larger equipment, individual pump-leak detector combinations are required.

The test is made in the following manner:

With the valve leading to the calibrated leak in an open position, tracer gas is forced into the hood until its concentration there is sufficient to give a predetermined reading on the leak detector. This reading (A) is proportional to the total leakage, which consists of unknown vessel leakage and known leakage from the calibrated leak.

The valve leading to the calibrated leak is then closed, and a second reading (B) taken. This reading is, of course, proportional to the unknown leakage alone. From these two readings the following equation may be derived:

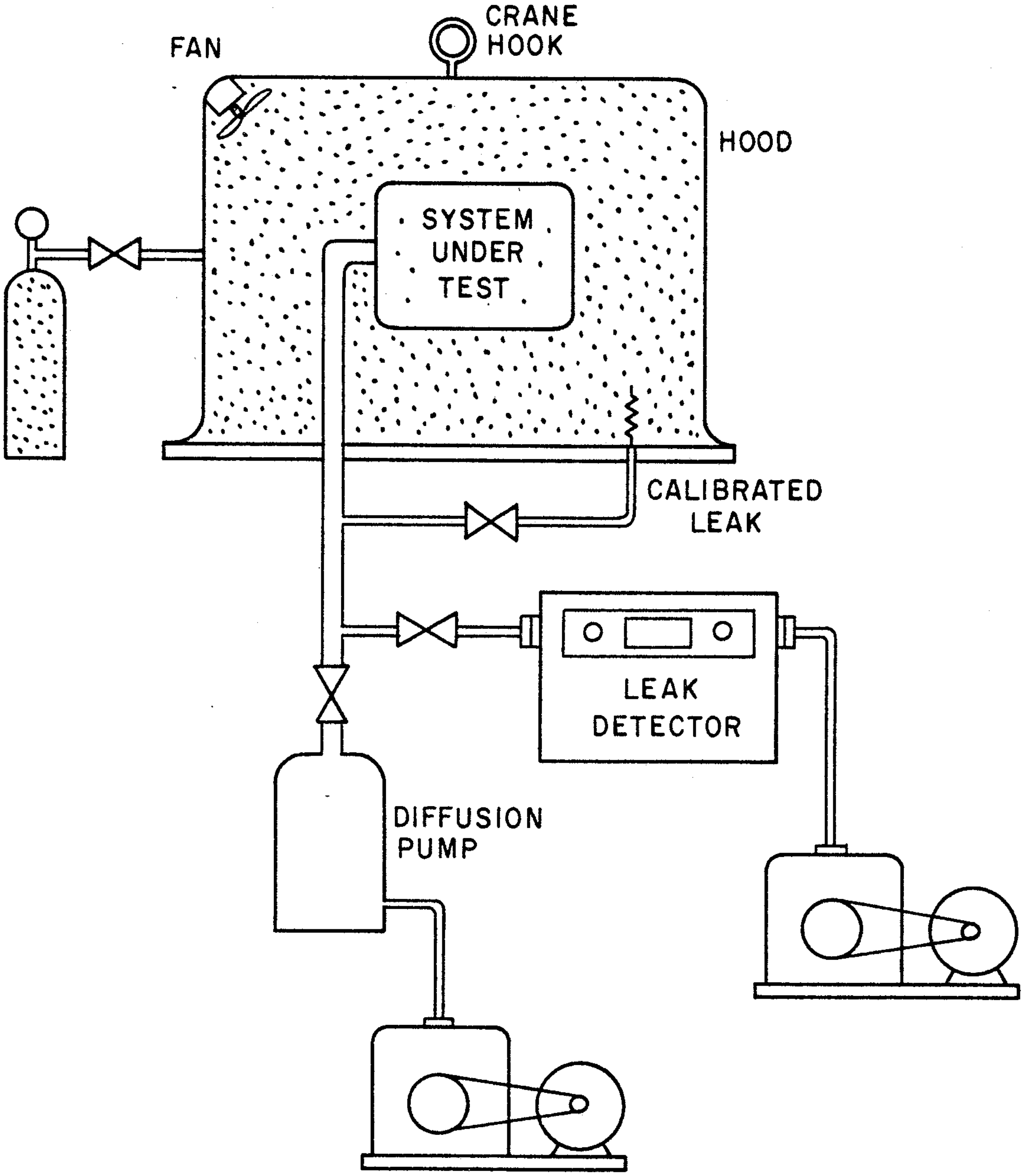

Figure 21. Typical Setup for Hood Testing.

$$\text{Unknown leakage} = \frac{\text{reading (B)}}{\text{reading (A)} - \text{reading (B)}} \times \text{calibrated leak size}$$

This is the calibration procedure described in Section 1.6.

This equation gives the unknown leakage in terms of the two readings and the value of the calibrated leak. Knowledge of tracer gas concentration in the hood in not necessary. The only requirement is that this concentration remain substantially constant while the readings are taken. Normally, this requires from 5 to 10 minutes. Well-constructed hoods show practically no decrease in tracer gas concentration for considerably longer periods.

Figure 22 illustrates a type of manifold which allows continous testing of a number of small vessels. The three testing lines make possible three simultaneous operations: loading and unloading, rough evacuation, and leakage measurement. Measurement of leakage can be performed individually on each vessel, or several vessels may be grouped in a single test. These measurements are made by the hood method. If rejected vessels are set aside for later attention, testing may be as rapid as the loading and unloading can be performed. The calibrated leaks shown serve both to test the effectiveness of the testing manifold and as a reference standard.

1.5.2.1.2. Small Component Testing

For rapid testing, a component can be leak tested on a rubber pad on a base plate, by sealing the component to the pad, (usually with the help of a vacuum grease) and spraying some tracer gas around the component. This is an unsatisfactory method of testing. If leakage is indicated, the experimenter does not know whether the component or the sealing surfaces (between pad and component or pad and fixture) are leaking. Furthermore, vacuum grease absorbs and retains the tracer gas. This produces what is known as background: the presence of tracer gas in the setup, even before testing begins. Background must be compensated for in analyzing the test results. In order to obtain accurate and reliable results, fixtures must therefore be well engineered.

A putty, which can be used for sealing in rapid testing, is Duxseal or Apiezon Q. The component is sealed to a fixture with Duxseal and pumped down. The system is then allowed to stabilize at its maximum sensitivity. When performance is optimum, tracer gas is passed over the entire surface of the part, and then lightly over the Duxseal joint. If a leak is observed in the Duxseal area, the setup is broken and must be remade, using new Duxseal. Duxseal is used only once and then discarded. Duxseal both absorbs tracer gas and allows permeation by tracer gas. The time constant for Duxseal with helium has been determined as 20 minutes; hence, this is the maximum duration of the detection cycle.

A good discussion of fixtures for leak testing is given by Smith (Ref. 56). Every fixture has two sealing surfaces; where

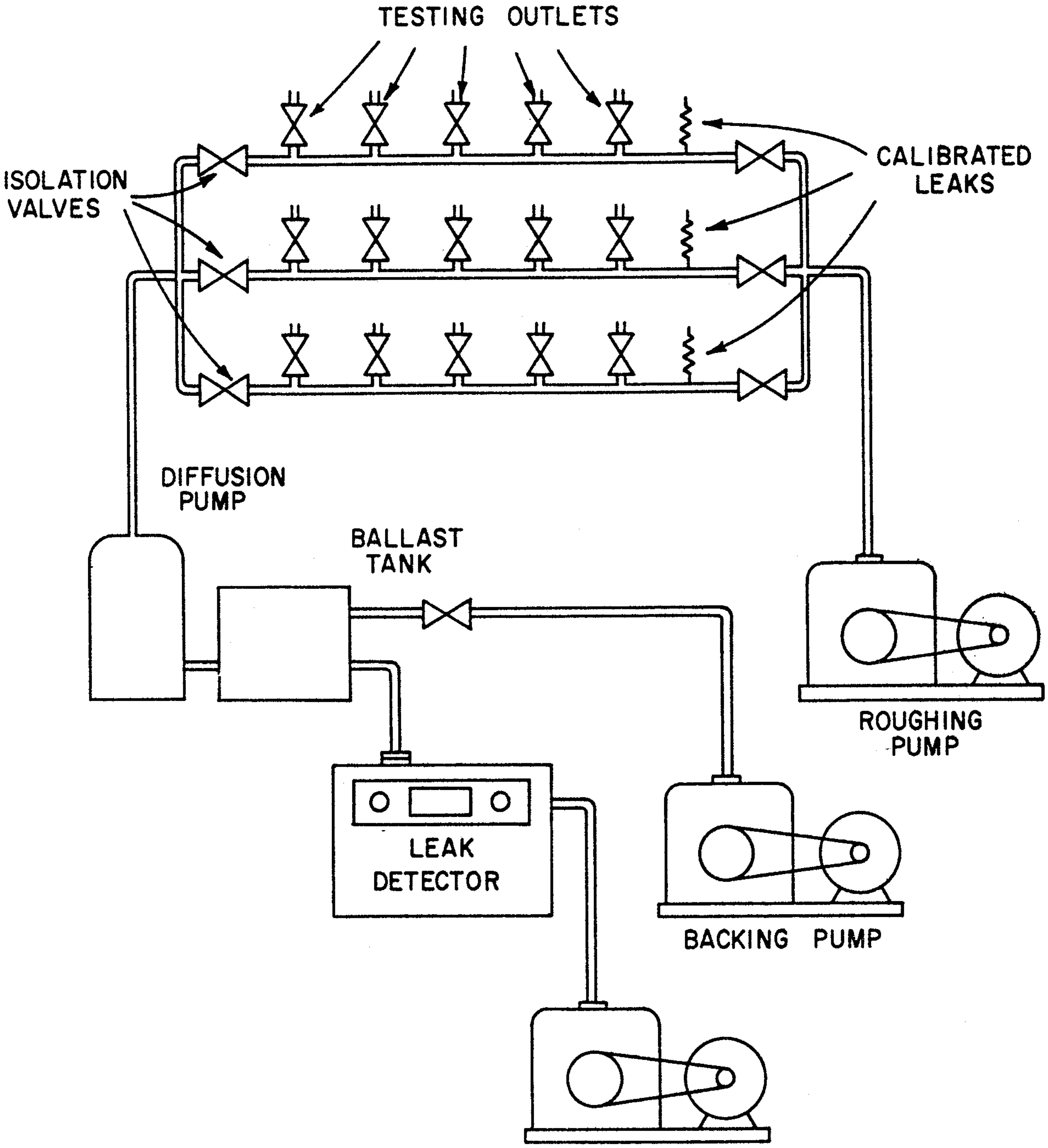

Figure 22. Typical Manifold for Rapid Testing of Small Parts.

it is sealed to the detector and where the component to be tested is sealed to the fixture. Both of these seals must be virtually leak proof for most tests. Figure 23 is an example of such a test fixture. Note that the two sealing surfaces cannot leak. The seal between the envelope and the base plate does not have to be leak tight.

Gaskets made of elastomers and some other materials are limited in their use because they become permeated with helium. During the first few minutes that helium is entering one side of such a gasket, no helium leaves the other side. However, leakage starts after a few minutes and quickly builds up to a steady value. Figure 13 shows the leak rate of some elastomer O-ring plotted versus time. Because of this permeability, elastomers and similar materials are limited to sealing test setups for the measurement of leaks larger than 5 x 10^{-7} atm cc/sec unless special fixturing and techniques are used. If rapid testing is the design purpose, or if no long-term measurements are desired, elastomers can be used when detecting leaks as small as 1 x 10^{-9} atm cc/sec.

The size and type of leaks which must be detected are major considerations in designing fixtures. In the realm of 10^{-4} atm cc/sec to 5 x 10^{-7} atm cc/sec elastomers and flat gaskets are the easiest and most practical to use. The elastomers used in O-ring configurations are made of viton or butyl rubber; flat gaskets are made of neoprene.

O-rings are mainly used for sealing between the base plate and the fixture, or around a circular section. When sealing around a circular part, the fixture must incorporate a means of forcing the O-ring in against the side of the part. One way to accomplish this is to use a retainer ring in the fixture to hold the O-ring in place. Without the retainer, the O-ring tends to spread or roll away.

Sealing surfaces should be of good quality and approximately 64 rms minimum or better. Shaft surfaces should not have longitudinal marks or scratches. Tooling marks which are oriented such that they bridge the sealing interface are harder to seal than marks which are perpendicular to the shortest-leak paths. To reduce the problem of sealing it is better to provide a good surface rather than try to compensate for a mediocre surface by vacuum grease.

Metal gaskets are often used for sealing when testing for leaks smaller than 5 x 10^{-7}. These gaskets are usually made of copper or aluminum and heat treated to a dead soft condition. Tests, lasting 24 hours, performed on these gaskets showed their permeability rate for helium was far below detectable leak rates.

The big disadvantage in using metal gaskets is in the large forces required to assure sealing. Fixtures can be designed to withstand these forces, but many components do not have the physical strength to withstand them. Therefore the dimensions of a component, and the mechanical properties of material used in it, must be known when designing fixtures.

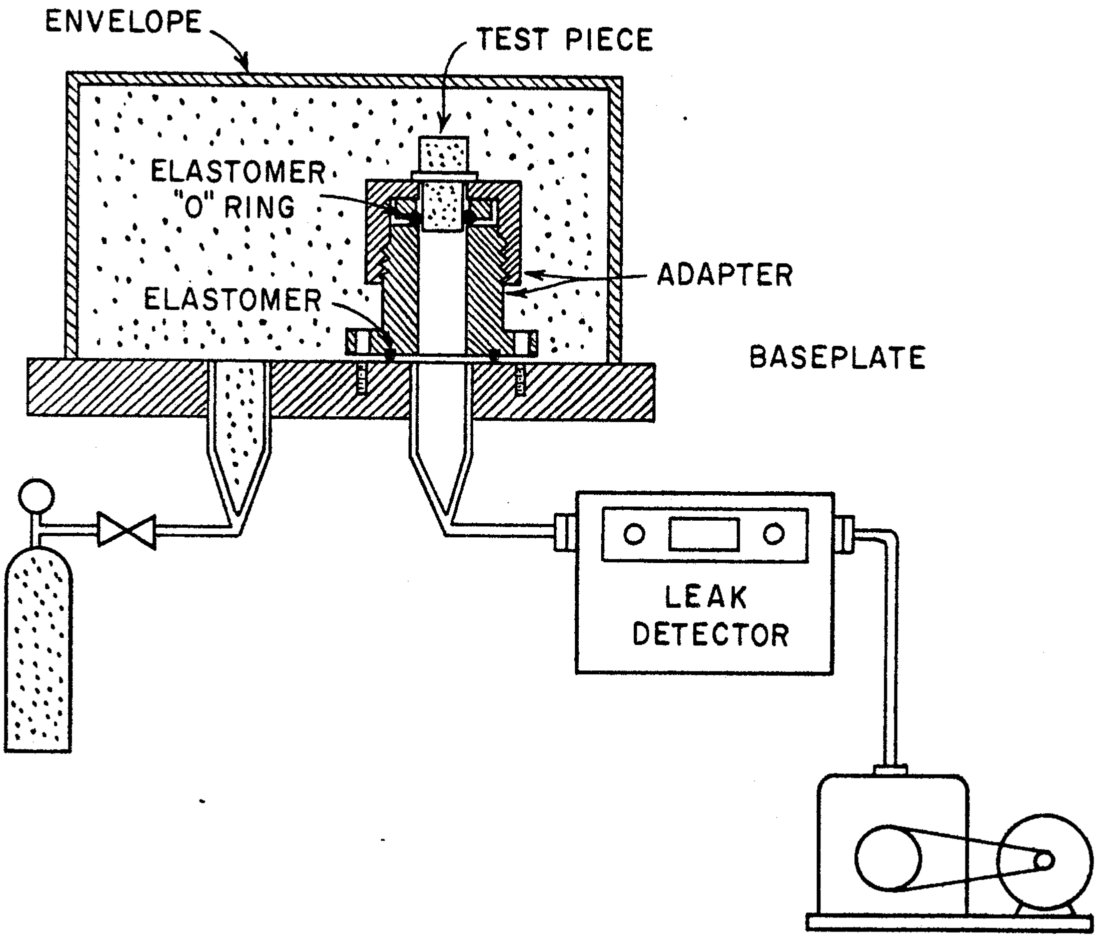

Figure 23. Reliable Test Fixture for Leakage Measurement. (Reprinted with permission, from G.C. Smith, ISA Journal, Vol. 10, 1963, p. 56; Copyright Instrument Society of America, Pittsburgh, Pa., 1963.)

When using metal gaskets the surfaces which they seal against must be of much better quality than those used with elastomers. Surface finishes of 16 rms or better are required for repeatable sealing.

The split-type fixture makes it possible to use elastomers and neoprene gaskets for leak testing in the realm of 5×10^{-9} atm cc/sec. Design of the fixture is such that any leakage of tracer gas through seals will be to the atmosphere and will not be sensed by the detector. Detector seals (Figure 24) between the test piece and the detector base plate do not have to seal against the tracer gas since they are not in contact with it. Therefore any tracer gas entering the detector must come through leaks in the test piece. This method is generally more practical than metal to metal seals, and it does not impose severe mechanical loads on the test piece in order to secure good sealing. However, it cannot always be used due to the shape of some test pieces.

Considerable test time can be saved by keeping the internal volume of the fixture as small as possible, and avoiding materials of construction which have high vapor pressures or high outgassing rates. This reduces the time required to evacuate the fixture to the operating pressure of the detector. Additional time can be saved by using a pump of ample capacity for evacuating the fixture and component.

The time required to perform the actual leak test is largely determined by the test specifications. Response time of a detector to tracer gas leakage in small parts, such as are considered here, is usually instantaneous. The fixture designer should see to it that nothing impedes the flow of tracer gas to the detector tube, and thereby add to test time.

Fixtures should be small. This allows the use of a small envelope for containing the tracer gas and a saving in cost of fixture, envelope, and tracer gas results. In addition to requiring less storage space, small fixtures save time during evacuating and backfilling.

Before a finished fixture is released for use, it should be thoroughly inspected visually, and leak tested. Leak testing is done by substituting a solid metal replica for the component, which will be used with the fixture. Any leaks found will be in the fixture, provided the sealing surfaces are of good quality. If the fixture leaks it can be improved until its leaks are much smaller than the maximum allowable leakage for the component.

1.5.2.1.3. Weld Testing

Garrod and Nankivell (Ref. 57) discuss testing of the individual welds in large systems. The principle of test is illustrated schematically in Figure 25. A vacuum box which covered temporarily the section of weld under test, was evacuated by a pump and also connected to the mass spectrometer leak detector. An atmosphere of helium, at roughly atmospheric pressure, was

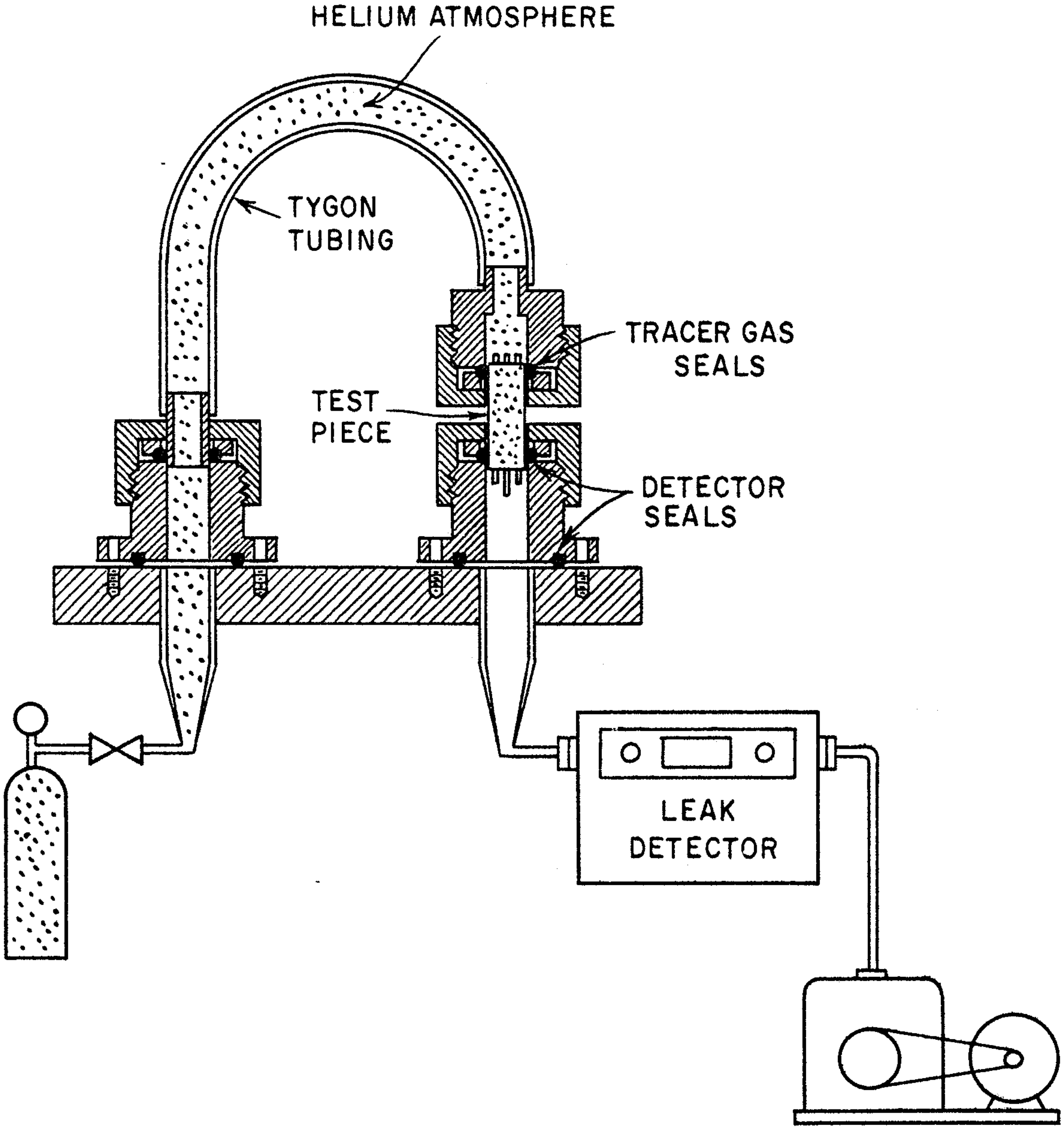

Figure 24. Split Fixture for Parts Testing. (Reprinted with permission from G.C. Smith, ISA Journal, Vol. 10, 1963, p. 56, Instrument Society of America, Pittsburgh, Pa., Copyright 1963)

maintained on the opposite side of the weld under a cover of sheet polyethylene, fastened at its edges to the steel plate by suitable adhesive tape. If a leak were detected, it could be located by reducing progressively the length of the helium envelope, and finally isolated by a fine helium probe.

The major design requirements for the vacuum boxes may be summarized as follows:

1. Working pressure in a box should not exceed 10^{-1} torr.

2. Attachment of a box to any welded section should be an easy, reliable, and relatively rapid process.

3. Design should aim for a minimum number of different types and sizes of box to cover all types of weld.

The principal difficulty in meeting these requirements is in making a satisfactory seal between the box and the section of the system under test. The surface of the steel plate is usually pitted and generally has a poor finish compared with normal vacuum engineering standards. Furthermore, the welds often impose a relatively abrupt change in contour on the seal where the two ends of the box cross the weld.

The method adopted, which has proven satisfactory in operation, is illustrated schematically in Figure 26. The vacuum box consisted of a flexible metal plate, to which were cemented two rectangular rubber gaskets. These gaskets were made from non-interconnecting cellular rubber. This material provided sufficient sponginess to effect a satisfactory seal with the steel plate and across the weld, but at the same time was non-porous. The section of the box inside the inner gasket was connected via flexible neoprene hose to the leak detector, which also contained the roughing pump for evacuating this volume. The annular space between the two gaskets was connected to a second rotary pump. By this means, leakage across the inner gasket was reduced sufficiently so that the inner volume of the box could be maintained at a pressure in most cases, of about 10^{-2} torr. For releasing the box from the welded plate after a leak test, air was admitted to each section of the box through driers and inlet valves. For transportability and convenience in use, the rotary pump and the air inlet system were mounted on a trolley.

1.5.2.1.4. Pipe Testing

The vacuum box can take any convenient form that makes a reasonably tight seal to the pipework. The box or shroud case consist of two brass half-shells with silicone rubber seals molded to them. The offset clamp can have an over-center lock with adjustable tension similar to a pair of "vice-grip" pliers (Ref. 58). Different sizes and configurations would be needed for various fittings. Such a fitting may be used to hold the tracer gas or as the detector connector.

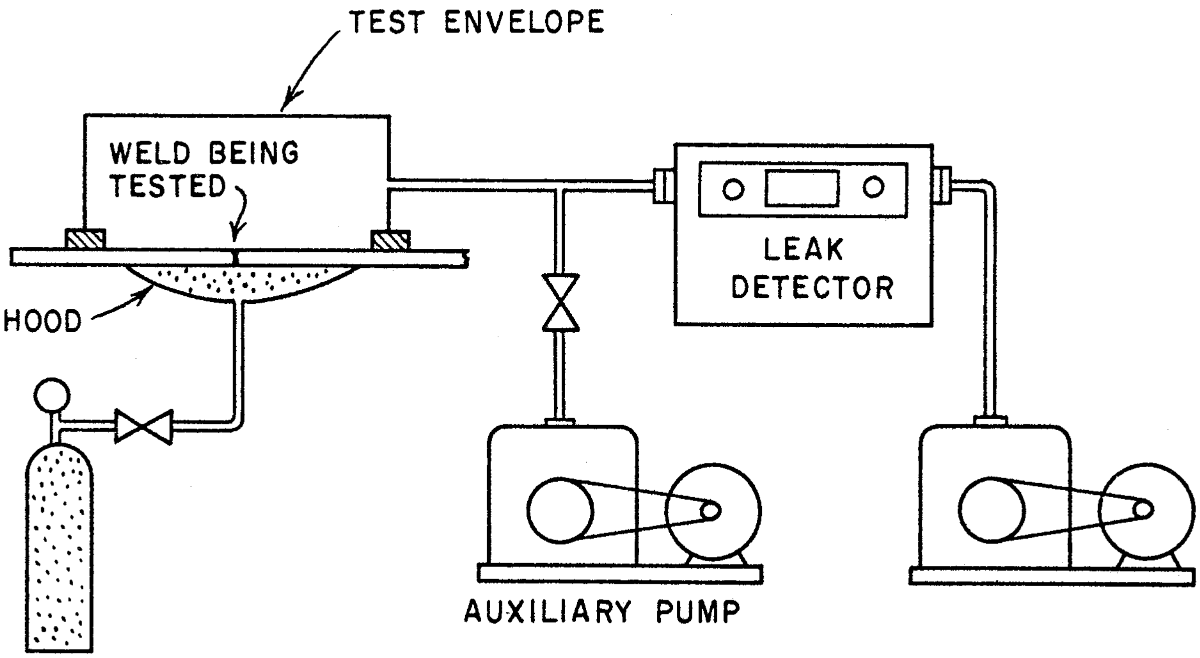

Figure 25. Vacuum Box for Weld Testing (Reprinted, with permission, from R.I. Garrod and J.F. Nankiwell, Vacuum, Vol. 11, No. 3, 1961, pp. 139-145, Copyright 1961, Pergamon Press, Inc.)

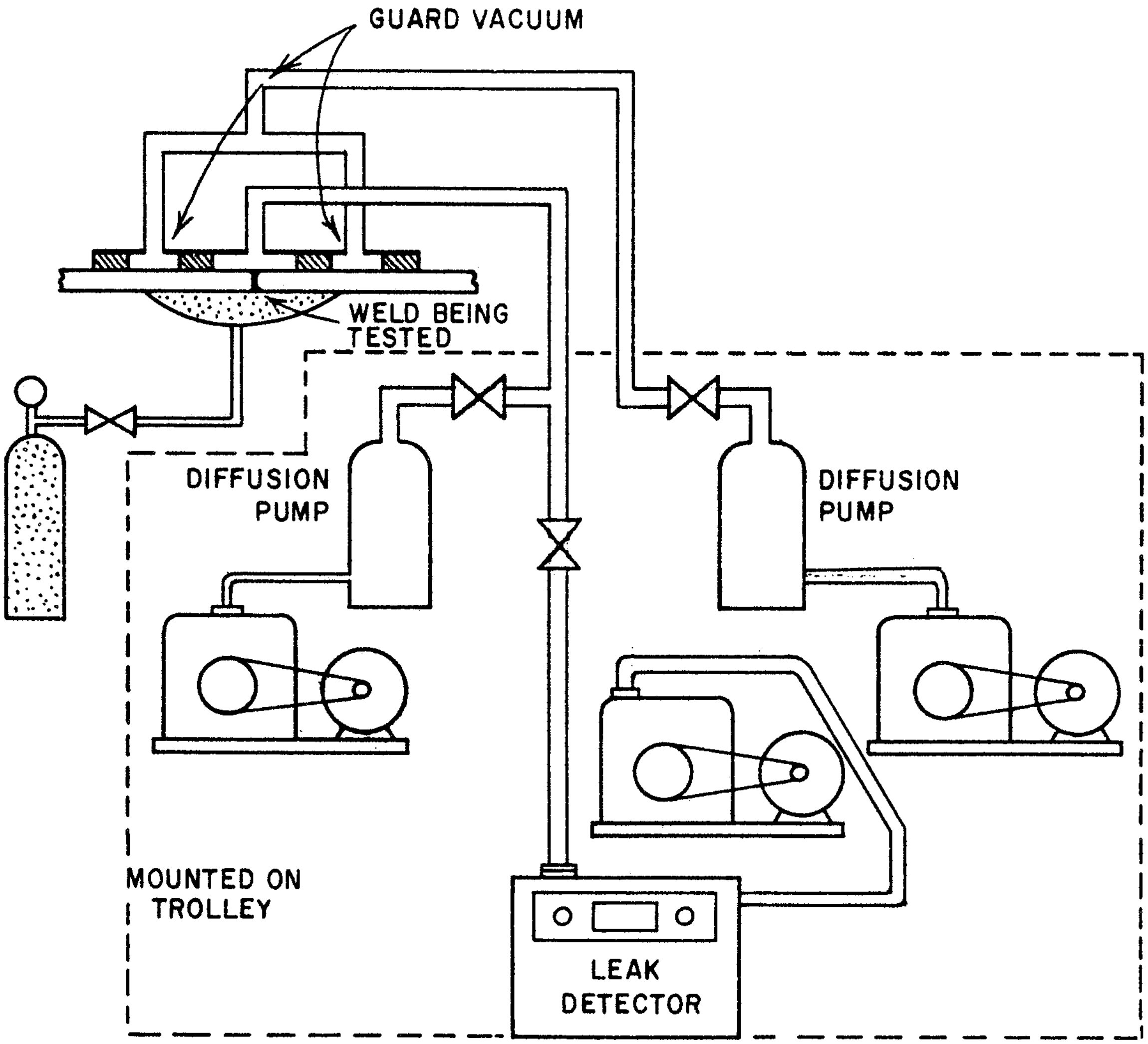

Figure 26. Use of Vacuum Box for Weld Testing (Reprinted with permission from R. I. Garrod and J. F. Nankiwell, Vacuum, Vol. 11, No. 3, 1961, pp. 139-145, Copyright 1961, Pergamon Press, Inc.)

1.5.2.2. LEAK LOCATION

1.5.2.2.1. General Procedure

There are two techniques of leak location - detector probe and tracer probe. Certain points should be observed in carrying out the probing:

1. If the tracer gas is lighter than air, the system should be probed from the upper parts of the test object to the lower. In this way the tracer gas, which rises, will flow back only over areas already tested. Of course, the reverse is true if the tracer gas is heavier than air.

2. For tracer probe testing of individual joints, time may be saved by using a generous flow of tracer gas from a flexible rubber tubing (1/4 in. ID). When a leak is indicated, its exact location can be determined by means of a finer probe. On the other hand, with large leaks in the system, enough tracer gas may enter the system to saturate it and the leak detector for a while. With many small leaks, diffusion of tracer gas to these small leaks may make leak detection difficult. The use of a fine probe will narrowly limit the area covered by tracer gas.

3. A very large leak will register on the detector, even when the probe is some distance from it. The leak should be located and repaired (permanently or temporarily). Vacuum putty may be used for temporary repairs.

4. When a point appears to leak, but does not give a consistant response, a large leak in some other location is possible.

5. To distinguish between two possible points of leakage, close to one another, one of them can be covered (with tape, for example). A fine probe and a minimum flow of tracer gas will help.

6. If a tracer liquid is to be used, there is less likelihood of blocking a leak if a pad of cotton is moistened (but not saturated) with the liquid and used, rather than a liquid spray.

7. The probe should be moved over the test area at about one foot per minute if the time constant of the system is near the optimum value, and more slowly if the time constant is greater than this.

   Response and cleanup time constants, when using a detector probe, depend on the length of tubing used in connecting the probe to the detector. As the length of tubing is increased, both response and cleanup time also increase.

8. In detector probe testing, if large leaks are known to be present, it is wasteful of search gas to use a high pressure. Furthermore, the space outside the test vessel in the neighborhood of a large leak will become flooded with search gas which will give confusing results when adjacent areas are probed. It is advisable, therefore, to pressurize the test vessel cautiously, watching the pressure gage for an indication of the presence of large leaks.

1.5.2.2.2. Design of Probes

The "sniffer" used in detector probe testing can be quite a simple device when it is to be connected to a leak detector with a pumping system, since the pumps provide the suction necessary for drawing a gas sample through the sniffer into the leak detecting element. Figure 27 shows the Atlas-MAT "Schnuffelsonde" (Ref. 59) with a screw adjustment for controlling the gas throughput.

The important points in this design are that a sleeve on the end of the probe collects the gas which will travel to the detector, and that the flow is controlled at the entrance to the probe i.e., there are no blind ducts at the entrance.

When tracer probe techniques are employed to locate the exact position of small leaks in equipment having re-entrant cavities, difficulties arise owing to the escape of search gas from the area under test. Under extreme conditions, the whole region may become charged with search gas, which by its entry through neighboring leaks makes localization almost impossible. Extrator fans which remove excess search gas from the region where the test is conducted effect only a partial improvement, and their installation is costly.

Pacey (Ref. 60) described a probe design which overcomes this difficulty. It consists of two coaxial stainless-steel tubes, search gas being supplied through the inner, the excess being removed through the outer by means of a small vacuum pump, which is vented outside the test region. The flow of search gas is adjusted to provide coverage of leaks, without exceeding the capacity of the extraction pump.

Standard hypodermic needles provide a ready source of suitable tubes. Size 0 is used for the outer, and size G for the inner, both having Luer type fittings. The arrangement adopted is shown in Figure 28. The outer needle, after the removal of most of its fitting to provide an attachment for the flexible tube leading to the vacuum pump at D, is softened by heating in a small gas-air flame, and when cool is bent through an angle of about 30°. A hole is made at the bend to allow insertion of the inner tube. Concentricity at the probe end is brought about by winding a 5-turn helix, shown at B, of 0.25 mm diameter wire around the inner tube.

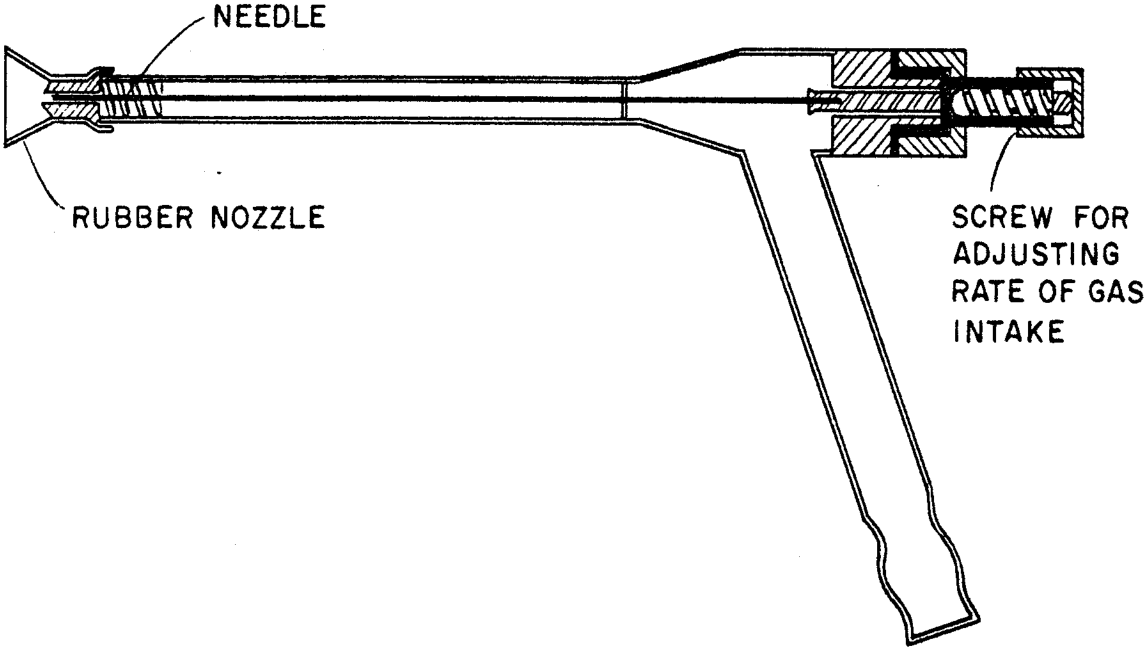

Figure 27. Detector Probe for Leak Location. (Reprinted with permission, from H.W. Drawin and K. Kronenberger, Vakuum-Technik, Vol. 8, 1959, p. 128, Rudolf A. Long-Verlag, Wiesbaden, Germany.)

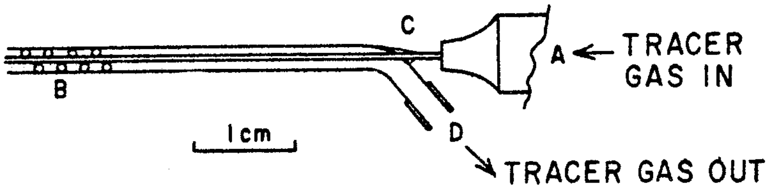

Figure 28. Self-extracting Tracer Gas Probe. (Reprinted, with permission, from D.J. Pacey. Journal of Scientific Instruments. Vol. 41, 1964, p. 398, The Institute of Physics and The Physical Society, London.)

After springing the helix into place in the outer tube, the inner is inserted through the hole already made and soft-soldered in position, using a zinc chloride flux, allowing a fillet of solder to form at C to provide adequate mechanical strength. The probe is attached to a suitable tubular holder by a taper engaging with the fitting of the smaller needle through which search gas is supplied.

If a leak closes at atmospheric pressure and thus does not exist under ordinary testing conditions, it is very difficult to locate. A device (Ref. 61) has been developed to locate such leaks by applying gas pressure to selected, small portions of the surface. Application of this device to successive small portions of the system allows one to localize the leak.

1.5.3. SYSTEM CLEANLINESS

The need for cleanliness of the system as a whole, including the test object, cannot be overemphasized. The larger the system, the more important is cleanliness.

Tracer gas accumulation in surface dust and oil may cause false but large leak signals. Excessive amounts of rubber, for example, gaskets and rubber tubing, should not be used since they absorb tracer gas. When a large leak is encountered, appreciable amounts of tracer gas are absorbed by the rubber and give false leak indications on succeeding tests.

Lubricants and vacuum greases should be minimized, since they too have an affinity for tracer gas. Good vacuum seals are obtainable without the use of greases. Furthermore, greases are dirt catchers and as such will contaminate the vacuum system.

Outgassing properties of leak detection systems are described in numerous books on vacuum technology. A pressure rise in a sealed vacuum indicates a leak, but so does outgassing. Pressure rise caused by a leak will be a straight line when plotted against time; outgassing will graph differently because it gradually decreases. This is discussed in more detail in Section 2.3.4.

It is possible for a system to exhibit the symptoms of a leak when it is in fact vacuum-tight. Virtual leakage, as it is called, is due to the presence of a condensable vapor in the system. For example, if water vapor is present in a system equipped with "dry ice" traps, the system pressure will approach a limit of 10^{-3} torr, which is the vapor pressure of water at the temperature of solid carbon dioxide. This is the simplest form of a virtual leak. In a liquid-nitrogen cooled trap, the vapor pressure of water is about 10^{-15} torr, which is negligible by present-day standards. If, however, some of the water vapor has been condensed as ice just below the refrigerant level, then, as the liquid nitrogen evaporates and its level falls, the temperature of this ice will rise until it begins to sublime, producing a virtual leak. This effect will not occur, of course, in a trap in which the refrigerant level is automatically maintained.

The characteristic of virtual leaks is that the system pressure is raised to some particular value which tends to be constant. When the presence of a virtual leak is suspected, volatile material may be present in the vacuum system.

The use of a dry gas bleed in vacuum systems and leak detectors is a highly recommended procedure. (Ref. 62) The purpose of the gas bleed is to create a laminar flow of gas through the entire system during those periods when the equipment is not in active use. This aids the outgassing of condensable materials, such as water vapor, by preventing readsorbation. It also minimizes the back migration of both hydrocarbons from the mechanical pump and condensables evaporating from the trap. In the case of mass spectrometer leak detectors, the background signal will be lowered and the maintenance reduced.

When installing a dry gas bleed system, the point of connection is chosen so that the gas flows from the cleanest parts of the system, past any traps or baffles, and then through the vacuum pumps. In the case of the leak detector, locating the bleed between the spectrometer tube and the inlet cold trap, and leaving the inlet valve open to the manifold and its pump during the bleed operation, permits gas flow toward both pumps. In this way, the inlet trap can be cleared of condensables without passing them through the spectrometer tube.

In operation, gas flow is adjusted so that the vacuum system pressure is between 0.2 and 0.5 torr. This can best be accomplished by initially collapsing a piece of 1/4-inch copper tube until the vacuum pressure is in the proper range. A good vacuum valve between the vacuum system and the restriction can be used to turn the bleed off and on.

When the gas bleed procedure is employed on a vacuum system, the cold traps, if any, should not be maintained cold, but allowed to warm to room temperature. As the required system pressure for effective bleeding is above their normal operating pressure, diffusion pumps should be shut down before applying the bleed gas, leaving only the mechanical pump(s) operating.

1.5.4. GENERAL REFERENCES

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Section 1.6.

TESTING STANDARDS

1.6.1. CALIBRATION VERSUS QUALIFICATION

Use of the term "calibration" implies the existence of a universally-accepted standard such as those at the National Bureau of Standards. No such primary standards for calibration of leaks exist. Individual manufacturers are forced to establish their own standards based on the first principles of physics.

Military specifications (Ref. 63) require that all "calibrations" be directly traceable to standards maintained by the National Bureau of Standards. In respect to leakage specifications, this puts equipment users in an impossible situation. Since no primary standards exist, equipment cannot be calibrated in accord with the government specifications.

A better approach to leak specification would be to avoid use of the word "calibration." Rather, a qualification should be written which define desired performance of specific "qualification" rather than a "calibration," desired performance will be obtained without requiring unobtainable measurements or comparison with non-existent standards.

In many cases, accuracy is not of prime importance in leakage measurement. Rather, most practical situations require that some particular leakage value not be exceeded. It need only be established that no leakage in the tested system is greater than this allowable maximum. This practical approach to leakage specification requires some arbitrary standard. However, if any doubt exists, one need only reduce the leakage of this arbitrary standard by a sufficient safety factor to ensure that test qualification will meet the practical leakage requirement.

1.6.2. COMMON TYPES OF CALIBRATED LEAKS

Calibrated leaks, leaks which deliver gas at a known rate, are sold by a number of vendors. The most common use of such leaks is in the measurement of sensitivity of leak detectors. However, calibrated leaks are also used to measure the speed of vacuum pumps and to calibrate pressure gages. A leak standard makes feasible the establishment of leak-rate specifications and provides a uniform standard for calibrating leak detectors at each location of product inspection. This assures agreement of all tests.

Calibrated leaks may be divided into two distinct categories - the reservoir leaks, those which contain their own gas supply, and the non-reservoir leaks to which tracer gas is added during test. A possible cataloging of leak types is shown in Figure 29. The calibrated leaks with their own gas supplies are the only ones which are of absolute accuracy. These leaks are said to

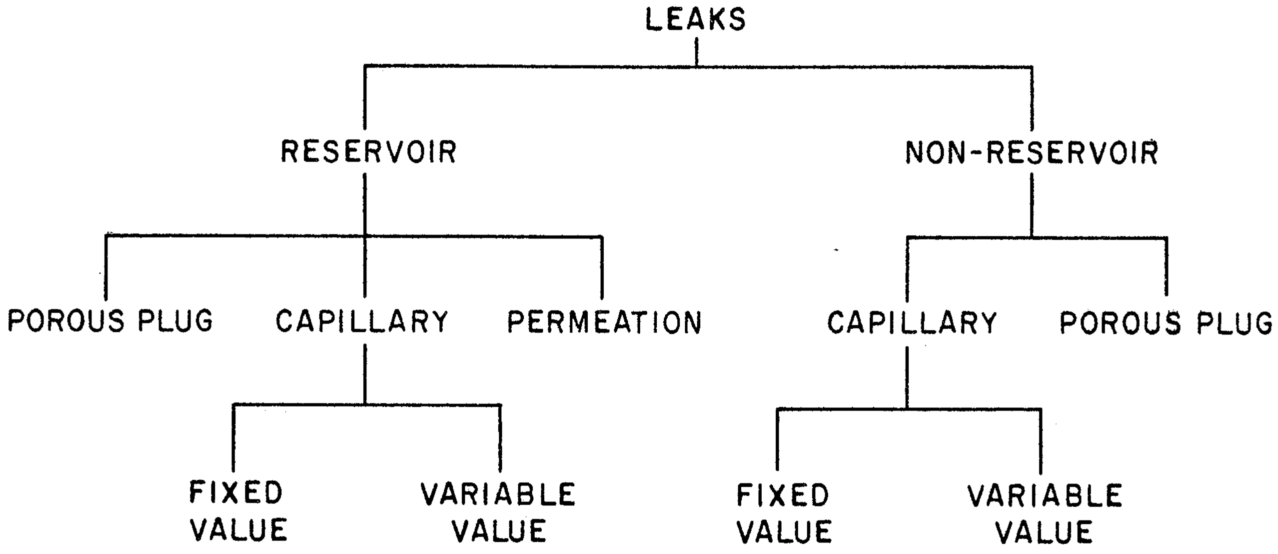


Figure 29. Categories of Artificial Leaks.

deliver gas into the leak detector at an absolute and known rate. They are, therefore, very good for determining the absolute sensitivity of the detector. However, such a reservoir calibrated leak does not necessarily give the total picture during operation of the leak detector.

The other type of leak is simply a hole or a series of holes and passages which permit gas to pass through at a known rate. The users of this type of calibrated leak must provide gas at a known concentration and pressure. However, any error made in supplying gas of a certain purity or pressure is probably the same one that will be made during operation. For this reason, the sensitivity of the leak detector during calibration should be approximately the same as the sensitivity during leakage testing.

In proper leak-detection practice, the sensitivity of leak detectors is checked frequently by calibrated leaks. For sensitivity checks, a calibrated leak without reservoir is preferable because it closely imitates the behavior of an actual leak. The calibrated leak without a reservoir is open to local atmospheric pressure; therefore it requires no sensitivity correction for this and other environmental factors. Tracer gas is sprayed on the calibrated leak under the same conditions which exist when the leak detector is used to measure a leak in any system or enclosure under test.

In the case of a reservoir-containing leak, the measured sensitivity of the leak detector is independent of the pressure and gas purity in the leak-detection area. If the calibrated leak is to be used for the measurement of an absolute value, as in the case of the calibration of a pressure gage or measurement of the speed of a pump, a leak carrying its own gas supply is desirable.

1.6.2.1. PERMEATION LEAKS

Permeation leaks employ the principle gas diffusion through a thin wall (Ref. 64). Tracer gas permeated from the area of high concentration to air or vacuum on the other side at a rate governed by the permeability of the thin membrane. The major advantage of permeation leaks is that they deliver extremely small quantities of gas, the commercially available helium range being 10^{-6} atm cc/sec to 10^{-10} atm cc/sec. Because a long period of time is necessary to achieve permeation equilibrium, these leaks usually come with a self-contained gas supply. However, at small leak rates, the leakage remain constant over a long period of time. The two disadvantages of these calibrated leaks are that they can only be made for the few gases that permeate through membranes, and that their leakage has a temperature coefficient of approximately three percent per degree Centigrade. The most common of these is the helium permeation leak, an example of which is shown in Figure 30. The helium permeation leak consists of a small helium-filled metal or glass cylinder with a built-in glass membrane at one end. Helium

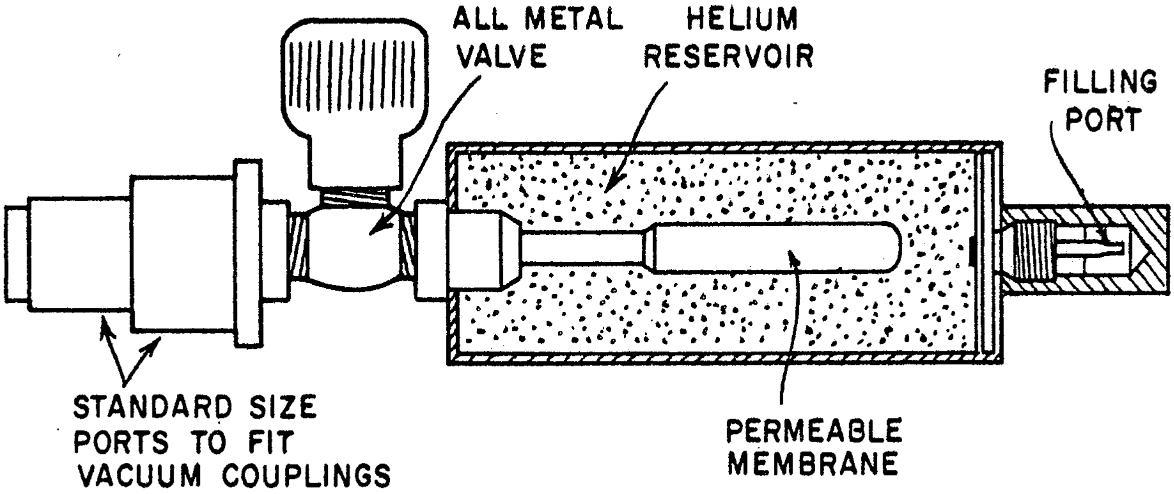


Figure 30. Helium Permeation Leak. (Reprinted with permission from Veeco Instruments, Inc., Plainview, N.Y.)

diffuses through this glass at a measurable rate, a rate so small that the helium reservoir will not diminish more than 10 percent in approximately 10 years. Each leak is individually calibrated and labeled. The leak illustrated in Figure 30 also contains a valve to enable it to remain permanently on the system, the valve being only open during the calibration procedure. The end of the leak is made standard size to fit one of the several types of quick-coupling vacuum connectors.

1.6.2.2. POROUS PLUG LEAKS

The porous-plug calibrated leaks are not commercially available but have frequently been cited in literature (Ref. 65, 66, and 67). They consist of a metal, ceramic, or glass plug containing extremely fine pores. The major advantage of this type of calibrated leak is that flow through the plug is molecular. Therefore, the change of flow resulting from a change of gas may be calculated from the kinetic theory of gas flow. These leaks can be made as a reservoir or non-reservoir type, with the implications of the choice as discussed above.

1.6.2.3. CAPILLARY LEAKS

Another type of commercial calibrated leak is a single orifice in Pyrex glass or metal, encased in a stainless-steel fixture. Tracer gas leaks through the orifice at the rated leakage, when placed at a specific pressure differential.

1.6.2.3.1. Fixed Value Leaks

Capillary type calibrated leaks are made from constricted glass tubing (Ref. 68), or collapsed thin metal tubing. These leaks can be produced from large sizes down to approximately 10^{-7} atm cc/sec. Although smaller leaks of this nature can be made, they become extremely difficult to handle because of clogging. These leaks can be calibrated to deliver one or a variety of gases. They are made to be used with an independent gas supply, i.e., simply a capillary which is attached to the system and the gas sprayed over the capillary. Alternatively, a leak can be made with a self-contained gas supply which can be permanently attached to the leak.

In another variation, the same orifice leak may be obtained with its own tracer gas reservoir leak factor gage (Figure 31). The gage reads in multiplying factors from 1 through 5, the pressure being changed in order to change the leak rate.

1.6.2.3.2. Variable Value Leaks

The General Electric Type LS-20 shown in Figure 32 is an example of a variable value leak. A schematic of the system is shown in Figure 32. This leak contains a reservoir of halogenated hydrocarbon, which is valved into a ballast tank in gaseous form. Also connected to the ballast tank is a glass capillary tube and pressure gage. The amount of leakage through the calibrated leak

Figure 31.

Reservoir Leak with Leak Factor Gage. (Reprinted with permission of the Instrument Department General Electric Company West Lynn, Massachusetts.)

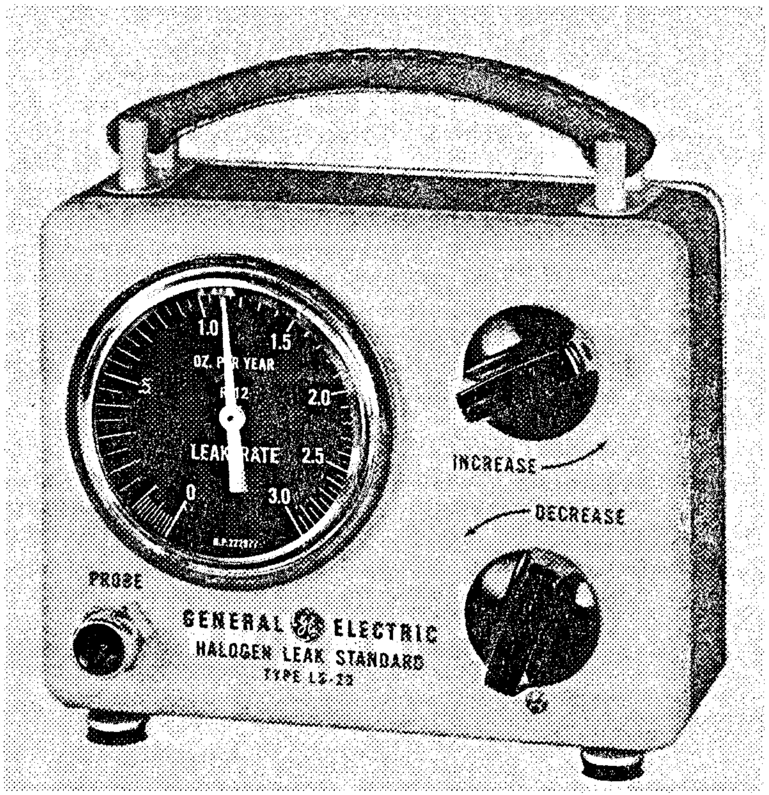


Figure 32.

Variable Valve Leak for Heated Anode Leak Detector. (Reprinted with permission of the Instrument Department. General Electric Company. West Lynn, Massachusetts.)

is dependent on the amount of pressure in the ballast tank. Since the flow through the leak is laminar, the gage is marked in leakage units, where leakage is proportional to the square of the pressure drop. The halogen leak standard is commonly used in heated-anode halogen leak detectors. It is an excellent standard to use with probe-type instruments, since the probe may be placed directly in the leak exit and the calibration approximates operating conditions.

Although not commercially available, a similar instrument could be used in conjunction with the mass spectrometer leak detector employing vacuum probe. This type of instrument could readily be made using the schematic shown in Figure 33. The major difference between this type of instrument and the halogen leak (General Electric Type LS20)is that the LS20 is portable, since halogenated hydrocarbon gas may be liquefied in the reservoir. Helium cannot be liquefied at room temperature, so the reservoir section would have to be made of a sturdy material, increasing the bulk of the equipment. Another difficulty with this type of system is that no commercially available leaks are calibrated with helium to an exit pressure of one atmosphere. These leaks, however, could be made by several manufacturers on demand. The equipment would find great use in calibrating vacuum-probe helium mass spectrometer tests.

Other variable value leaks are sold by leak detector manufactures as "variable leaks." These are either elegant needle valves or crushed tubing whose conductance is changed by flexing. Although the conductance of these leaks can be made quite repeatable, they should not be considered calibrated leaks because of a complete lack of standardization in these devices.

1.6.3. INACCURACY OF LEAKAGE MEASUREMENT TECHNIQUES

The inaccuracy of leak detectors may be caused by:

1. Inaccuracy in calibrating the leak.
2. Nonlinearity of the leak-detection instrument.
3. Variation in pressure differential applied across the leak.
4. Impurity of gas applied to the leak.
5. Variation in the amount of gas reaching the detector.

The accuracy of the calibrated leaks available from vendors has been measured (Ref. 69 and 70). There are no primary standards against which calibrated leaks may be compared. It is, for example, impossible to obtain calibrated leaks with a calibration which can be traced back to the National Bureau of Standards. Individual manufacturers, therefore, are forced to establish their own standards based on first principles of physics.

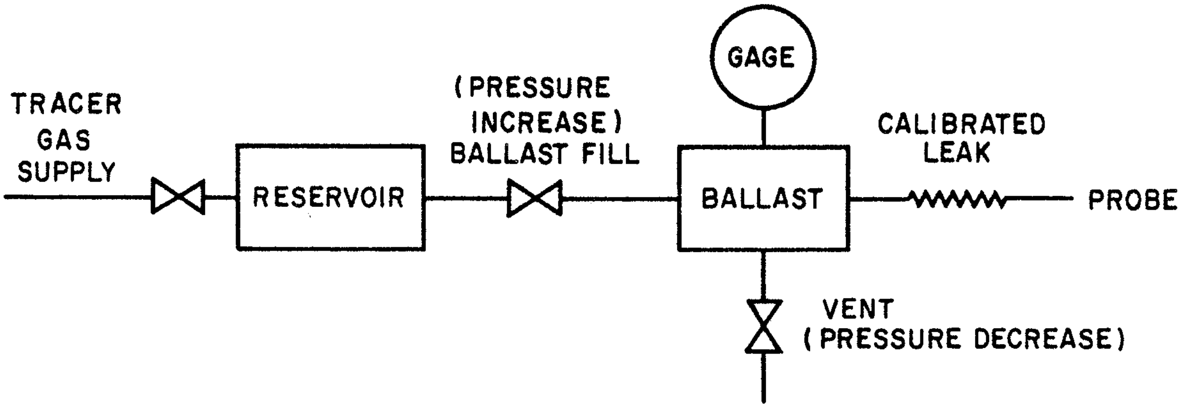


Figure 33. System for LS 20 Variable Valve Leak

Comparisons between leaks of various manufacturers have shown that their accuracies do not agree to more than plus or minus 50 percent of a mean value. This may be seen by the experimental plot of Figure 34 which shows the calibrated leakage readings as plotted to the response of a very linear mass spectrometer.

The straight line drawn in the graph is the least mean square value of leakage versus spectrometer response. This line does not imply the correct value, but the general pattern around which the values of the leaks congregate.

Even the leaks of any single manufacturer vary by approximately 10 percent. This is usually the guarantee which is presented upon purchase of the permeation type calibrated leak. Leaks of a variable type, for example, the General Electric LS20 are claimed to be accurate only to plus or minus 20 percent, since considerable error is encountered, such as variations in meter readings.

Most commercial leak detectors display the response to a detected leak as a current reading on a sensitive microammeter. It is usually assumed by the operator that current reading twice the magnitude of a previously observable one represents a leak of twice the size. This linearity is not necessarily correct because of the structure of the pumping system, the background usually associated with the leak-detection practice, and the electronic circuitry associated with the detection system. Such deviation from linearity in mass spectrometer leak detectors is currently being studied by the American Society for Testing and Materials project. (Ref. 70).

A typical response error curve is shown in Figure 35. The instrument response is not linear with leakage. This error is added to the error which occurs because of the difference in leak calibration. Because of this lack of linearity, the further apart the two leaks are in nominal value, the greater the error in the calibration.

Because such deviations exist, it is recommended that when the leakage measurement is done to a specified high tolerance, a calibrated leak of the exact specified value be used as a standard.

Leakage is dependent on the pressure differential across the leak. When leak detection is done by a tracer probe, the pressure differential is usually one atmosphere, the gas being sprayed over the suspected area without aid of additional pressure. Should leak detection be performed at high altitudes, the atmospheric pressure is less than one standard atmosphere (the magnitude of this reduction is as much as 20 percent in places such as Boulder, Colorado). If the leaks which are being located are of a laminar nature, the flow through the leak is proportional to the square of the pressure differential. The values obtained for these readings are 40 percent less than those obtained at one-atmosphere pressure differential. Therefore, a leak measured

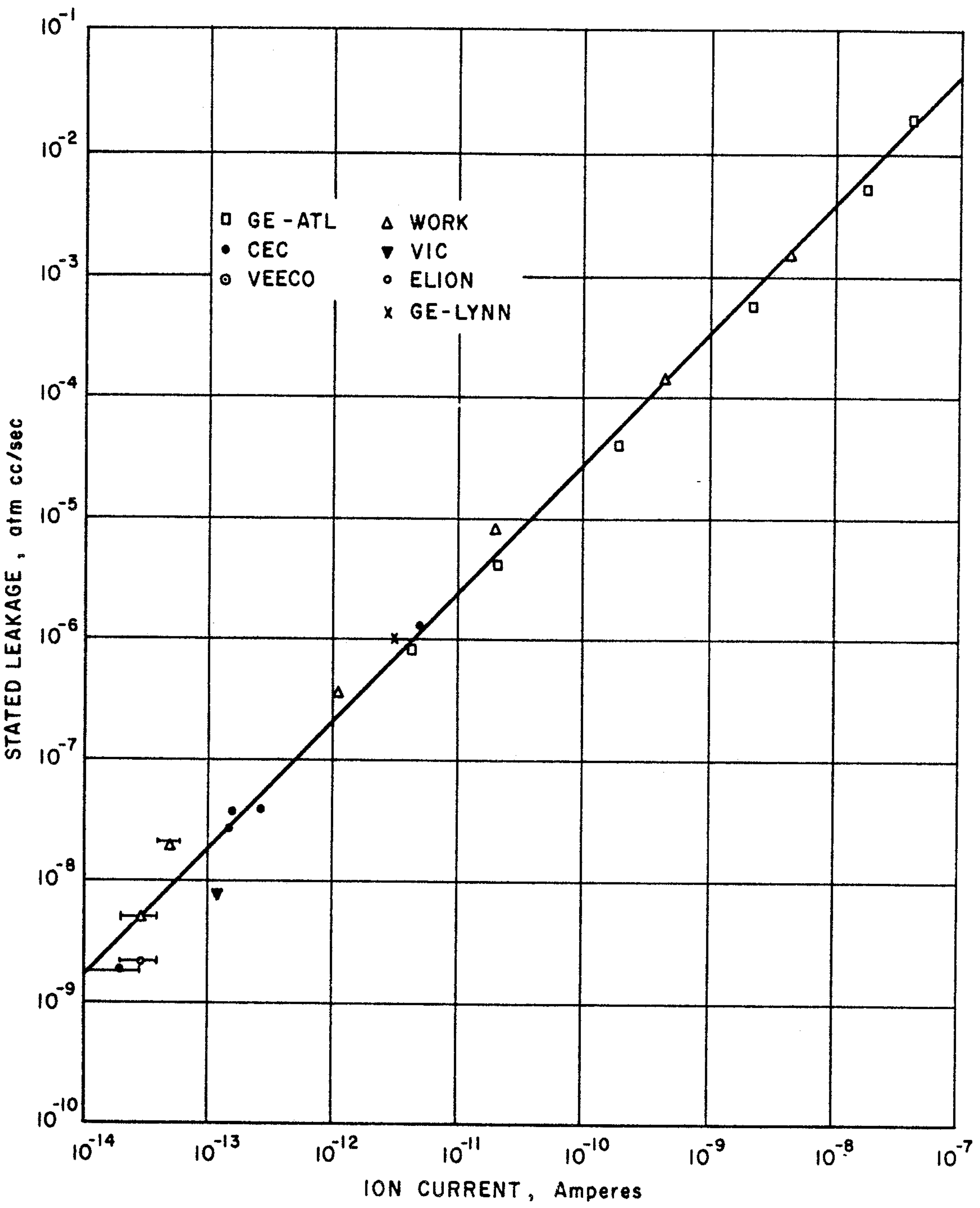


Figure 34. Comparison of Leakage Values for Leaks Supplied by Various Vendors. (Ref. 45)

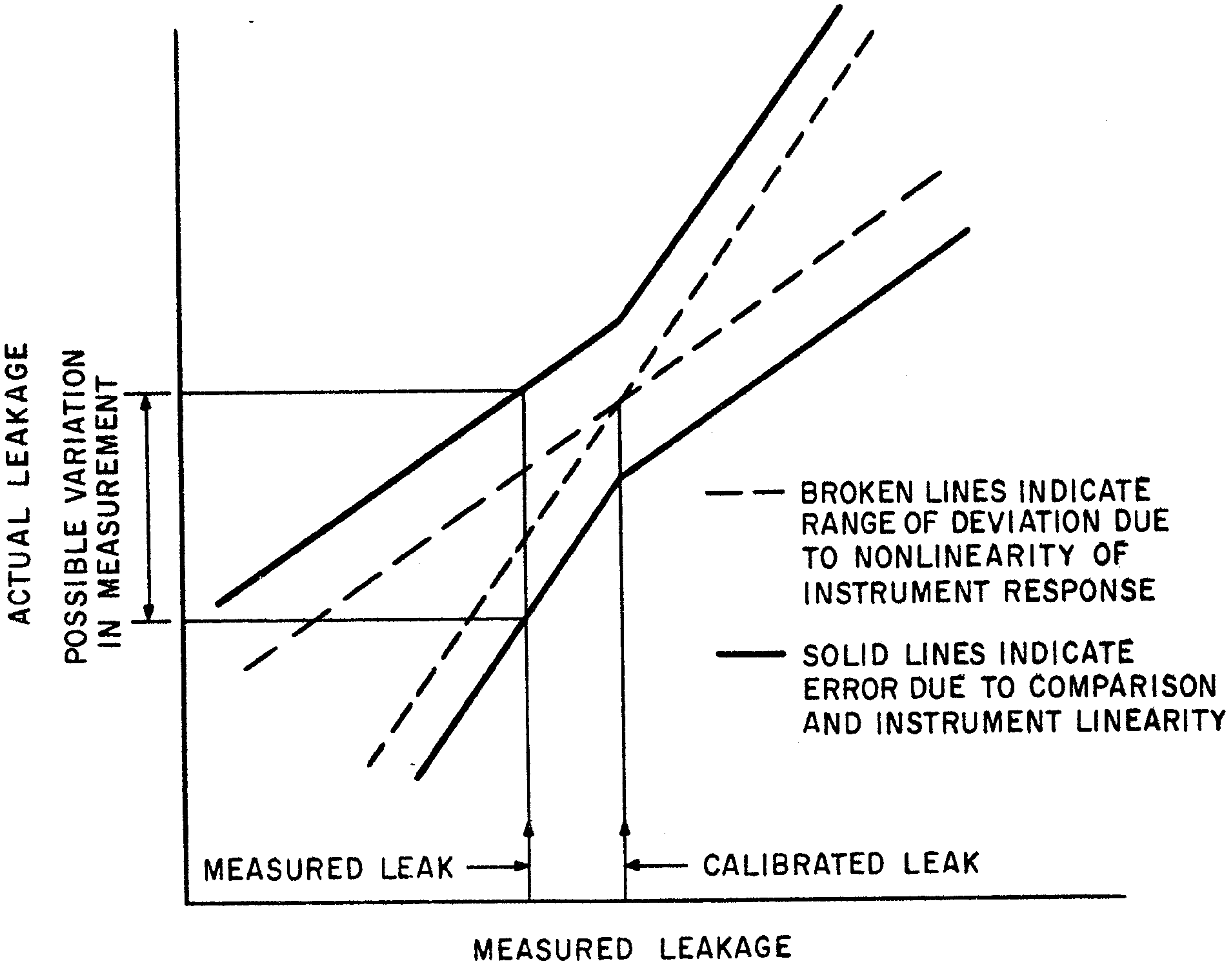


Figure 35. Typical Response Error Curve in Leakage Measurement. (Reprinted from J.W. Marr, "Leakage Phenomenon," Presented at Valve Seminar, Midwest Research Institute, Kansas City Missouri, October 1965.)

to atmosphere in Boulder, Colorado will be only 60 percent as large as one in, say, Cape Kennedy, Florida.

Certain calibrated leaks contain their own gas supply, whereas others have the tracer gas sprayed on the leak at one-atmosphere pressure differential during use. Calibrated leaks with a self-contained gas supply always deliver to the detector a fixed amount of gas which is interpreted as the sensitivity of the leak detector. On the other hand, leaks where gas is added during use produce the calibrated amount of leakage only when a one-atmosphere pressure differential is supplied. The latter leaks therefore deliver less than the calibrated amount of leakage when used at high altitudes where the atmospheric pressure is lower. However, at these altitudes, the pressure of the tracer gas across the leak is lower. In such cases a leak <u>without</u> its own gas supply describes more accurately the sensitivity of the leakage test.

Another source of inaccuracy is the impurity of the tracer gas used for leakage measurement. If a tracer probe is used for leak detection, the gas is sprayed over the suspected area. In such a case it is quite possible that the tracer gas is diluted with air as it approaches the leak. Therefore, the response of the leak detector will be reduced by the amount of air impurity entering the detector with the tracer gas. In this case, the use of a calibrated leak with a self-contained gas supply is undesirable, since it would not reproduce the leakage measurement method. In other words, the gas should be sprayed on the calibrated leak in the same manner as on the tested leak. The gas in a self-contained calibrated leak would be purer than the gas encountered by simple spraying from a tracer probe.

Tracer gas traveling to the detector may be absorbed on system surfaces. This would decrease the response of the detector. Therefore, calibrated leaks should be positioned on the system as near as possible to suspected leak sites to improve accuracy. Alternatively, they may be positioned as far away from the detector as possible to show minimum sensitivity.

Because of the variations discussed in this section, the accuracy of any leakage measurement is probably no greater than a factor of twice around stated value. This implies that if a leak is measured as 1×10^{-5} atm cc/sec, the actual value of this leak is between 2×10^{-5} and 0.5×10^{-5} atm cc/sec. Therefore, if the maximum allowable leakage of a particular system is 2×10^{-5}, the specification may be written with a leakage tolerance of 1×10^{-5}, knowing that the accuracy of this measurement is a factor of two. There is reasonable assurance that if the measured leakage is no higher than the one stated on the specification (1×10^{-5}), the actual system leakage will be no greater than the allowable one (2×10^{-5}). This method of specifying leakage is much more sensible than specifying a slightly higher leakage value (2×10^{-5}) and thereby requiring an unreasonably high accuracy (± 10%).

1.6.4. AVAILABILITY

The calibrated leaks commercially available are shown in Table 12. Only a limited number of calibrated leaks are available. Gases which may be used with the calibrated leaks are limited. Only one type of leak is designed to deliver gas at atmospheric pressure, that is, the General Electric LS20 leak for the leak detector. Some manufacturers will produce calibrated leaks on request. One of these is Hastings-Raydist, Inc., formerly the RH Work Company.

1.6.5. LEAK CALIBRATION TECHNIQUES

Calibrated leaks are available with eight decades of leakage values. Because of this large range of leakage, calibration is a difficult task. This task is made more difficult by the fact that there are no established standards in this field, and therefore each vendor is compelled to develop his own primary standards. The difficulty is compounded because no standards for the low-pressure measurements exist.

The five methods of measuring leakage rates are:

- Isobaric volume change.
- Pressure rise method.
- Pressure drop across a known conductance.
- Pressure measurement at constant pumping speed.
- Measurement of alpha radiation.

1.6.5.1. ISOBARIC VOLUME CHANGE

A schematic diagram of the equipment used in the isobaric volume change method is shown in Figure 36. One side of the leak is attached to a vacuum system while the other side is attached to a gas reservoir at atmospheric pressure. To this reservoir is attached a capillary of known cross-section. A slug of indicating fluid is placed in this capillary. As gas leaks from the volume into the vacuum, the slug of fluid travels down this capillary in order to keep the pressure in the reservoir constant. The leakage rate is determined by measurement of the volume displaced by the travel of the slug down the capillary, as stated in Equation 1.6-1.

$$Q = \frac{P(V_2-V_1)}{t} \qquad \text{Equation 1.6-1}$$

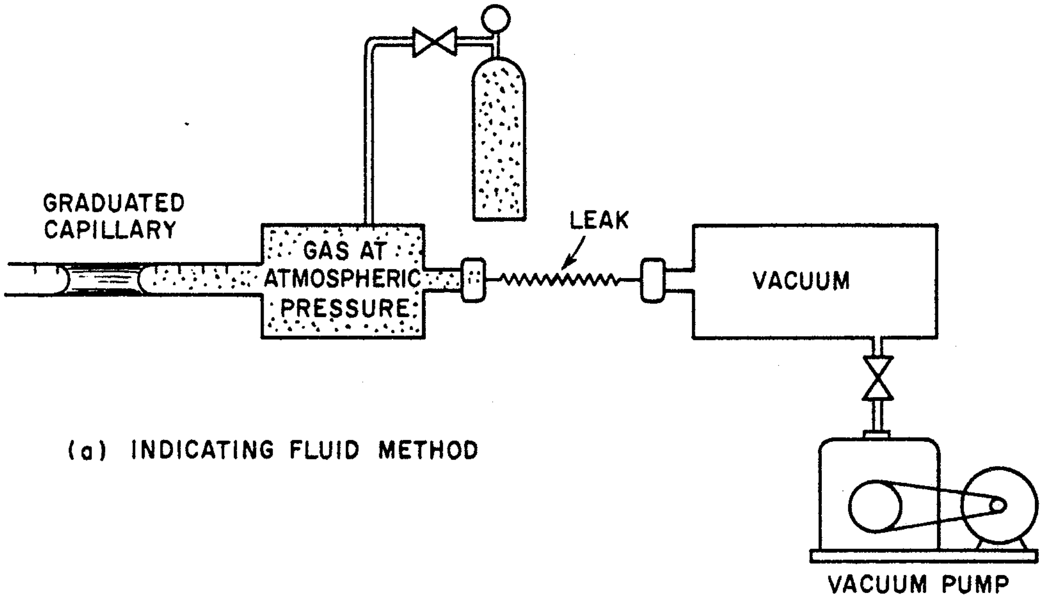
where Q = leakage
P = pressure in the gas volume
V_2-V_1 = volume displaced during travel of the indicating fluid
t = time

Table 12

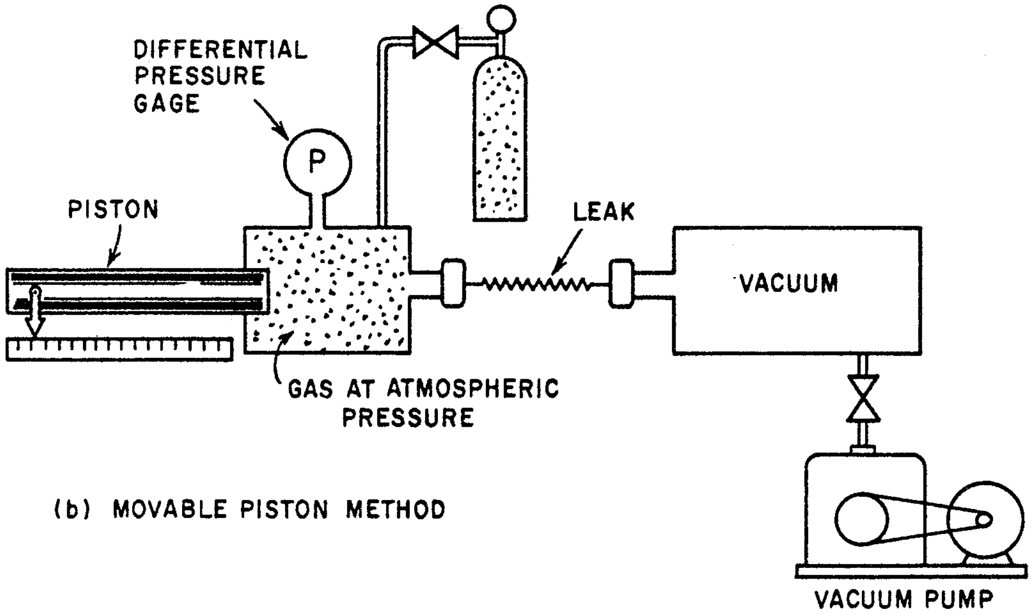
CALIBRATED LEAKS COMMERCIALLY AVAILABLE

Name	Type	Gas Supply	Gas	Leakage Range (atm cc/sec)
Consolidated Electrodynamics Corporation Pasadena, California	Permeation	s.c.	Helium	10^{-6} to 10^{-10}
	Capillary-glass	s.c.	Argon	10^{-6} to 10^{-7}
	Capillary-glass	s.c.	Neon	10^{-6} to 10^{-7}
Edwards High Vacuum Incorporated Grand Island, New York	Capillary-glass	s.c.	Hydrogen	10^{-5} to 10^{-6}
The General Electric Company - Vacuum Products Business Section Schenectady, New York	Capillary-metal	ind.	Air	10^{-6} to 10^{-7}
The General Electric Company - Instrument Department West Lynn, Massachusetts	Capillary-glass	s.c. or ind.	Helium, halogenated hydrocarbons	10^{-2} to 10^{-7}
Heraeus-Engelhard Vacuum, Incorporated Monroeville, Pennsylvania	Capillary	s.c.	Argon-Helium	10^{-6} to 10^{-8}
	Permeation	s.c.	Helium	10^{-8}
Veeco Instruments, Incorporated Plainview, Long Island, New York	Permeation	s.c.	Helium	10^{-9}
	Capillary-metal	ind.	Air	10^{-3} to 10^{-5}
Vacuum Instruments Corporation Huntington Station, New York	Permeation	s.c.	Helium	10^{-9}
Hastings-Raydist, Incorporated Hampton, Virginia	Permeation	s.c.	Helium	10^{-6} to 10^{-10}
	Capillary-glass	s.c.	Specified by Purchaser	10^{-2} to 10^{-7}

s.c.	self-contained tracer gas supply
ind.	independent tracer gas supply



(a) INDICATING FLUID METHOD



(b) MOVABLE PISTON METHOD

Figure 36. Leak Calibration By Isobaric Volume Change. (Reprinted from J.W. Marr, Study of Static and Dynamic Seals in Liquid Rocket Engines, Final Report Phase II, Contract NAS 7-102. (Ref. 45))

The primary limitation of this method is the size of the capillary involved. It is difficult to obtain a liquid which may be placed in a small capillary tube and which subsequently can be forced out the other end. For this reason, the practical limitation of such a method is in the range of 10^{-5} atm cc/sec. It would theoretically be possible to use a slightly larger capillary, and to take longer periods of time between readings. However, in this case the errors might arise from permeation either through the liquid slug or through the walls. An error might also be introduced by a change in barometric pressure or a change of ambient temperature. This becomes particularly critical in the calibration of small leaks, since a slight temperature change might produce a volume change greater than that due to the efflux of the gas.

It is desirable that the indicating fluid is not permeable to the gas being calibrated. For this reason, mercury is the preferred indicating fluid. Because of its high surface tension, mercury cannot be placed in a small capillary; this drastically limits the size of the leak which may be calibrated with mercury. For these measurements it is desirable to use a liquid with a low vapor pressure so that the leak is not contaminated by the calibration fluid. Unfortunately, most liquids of low vapor pressure are also of high viscosity and make it difficult to obtain an accurate measurement of the flow of liquid displaced in the capillary. These fluids also tend to form bubbles at the end of the capillary. The added pressure necessary to remove the bubble of liquid from the end of the capillary prevents the experiment from being isobaric.

Haywood (Ref. 71) overcame the need for an indicating fluid by the use of a piston to replace the effluxing gas. In this method, differential pressure gage is used to measure the pressure in the gas volume, and the piston is pushed into the volume at a rate to keep the pressure constant. The pressure gage need not be calibrated. This method can readily measure leakage as low as 10^{-6} atm cc/sec.

1.6.5.2. PRESSURE RISE METHOD

The second method of calibrating leaks is by means of the pressure rise technique (Ref. 72). A leak and its gas supply are attached to an evacuated chamber of known volume (Figure 37). The leaking gas is allowed to accumulate in this volume, and the pressure rise is measured at various intervals. The leakage may then be computed by the equation:

$$Q = V\frac{dP}{dt} \qquad \text{Equation 1.6-2}$$

where Q = leakage
V = volume of evacuated chamber
P = pressure in chamber
t = time

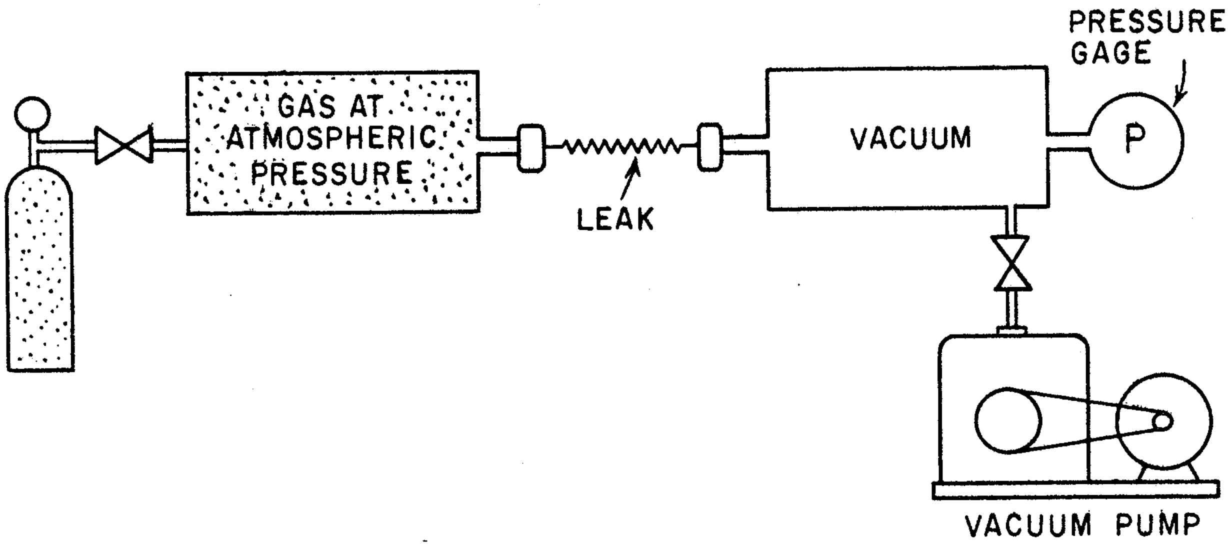


Figure 37. Leak Calibration By Pressure Rise Technique. (Reprinted from J.W. Marr, <u>Study of Static and Dynamic Seals in Liquid Rocket Engines, Final Report Phase II, Contract NAS 7-102.</u> (Ref. 45))

The major difficulties with this type of calibration technique are in measurement of pressure. The pressure in an evacuated system usually does not stay constant, but gradually increases due to outgassing of the walls of the chamber. The pressure rise due to this desorption has to be taken into account in the calculations.

A second difficulty in pressure measurement is the lack of a suitable gage. A McLeod gage may be used for the pressure measurement, but it is suitable only for the higher pressure ranges. If a long period is used to obtain a readable pressure change, outgassing becomes a particularly difficult problem. Ionization gages are difficult to use for this work because of lack of adequate calibration, and because of outgassing at the low pressures at which they work. Another alternative is to use a mass spectrometer as the pressure gage (Ref. 73). In this case the effect of outgassing may be overlooked since the desorbing gas is usually not the one being measured.

1.6.5.3. PRESSURE DROP ACROSS A KNOWN CONDUCTANCE

A third method of measuring leakage rates is by measuring the pressure drop across a known conductance. This technique is illustrated in Figure 38. The pressure drop across a known conductance is proportional to the flow. As previously stated,

$$Q = C\,(P_1 - P_2) \qquad \text{Equation 1.6-3}$$

In molecular flow the conductance C may be designed from theoretical grounds and such a conductance may be accurately constructed.

Again, the major difficulty with this technique is in obtaining accurate pressure measurements. Ionization gages have been used for the pressure measurement, but their readings are often questionable. Because their sensitivities are more often in doubt, this type of measurement has also been used to calibrate ionization gages (Ref. 74).

1.6.5.4. PRESSURE MEASUREMENT AT CONSTANT PUMP SPEED

A fourth method of calibrating the flow of a leak is by measuring the pressure it produces in a vacuum system which is pumped at a known speed (Figure 39). This is the limiting case of Equation 1.6-3, when P_2 is zero. The equation then being used takes the form

$$Q = SP \qquad \text{Equation 1.6-4}$$

where S = the pumping speed of the system, usually governed by an orifice
P = the ultimate pressure

The system is usually constructed so that the pumping speed is controlled by molecular kinetics considerations and may be

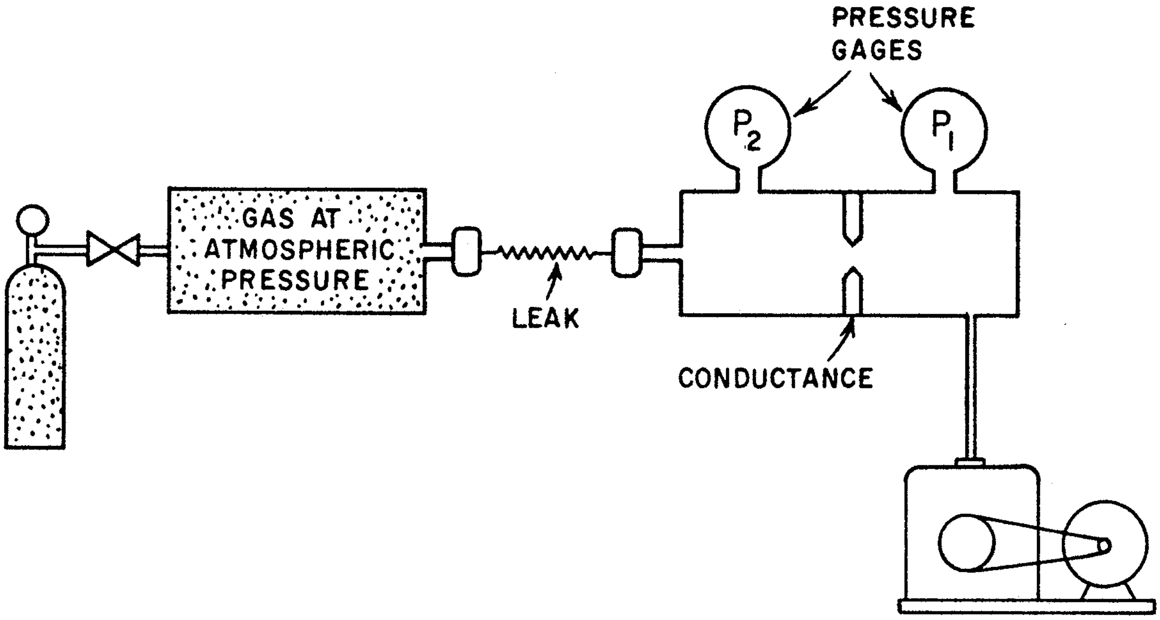


Figure 38. Leak Calibration By Pressure Drop Across a Known Conductance. (Reprinted from J.W. Marr, Study of Static and Dynamic Seals in Liquid Rocket Engines, Final Report Phase II, Contract NAS 7-102. (Ref. 45))

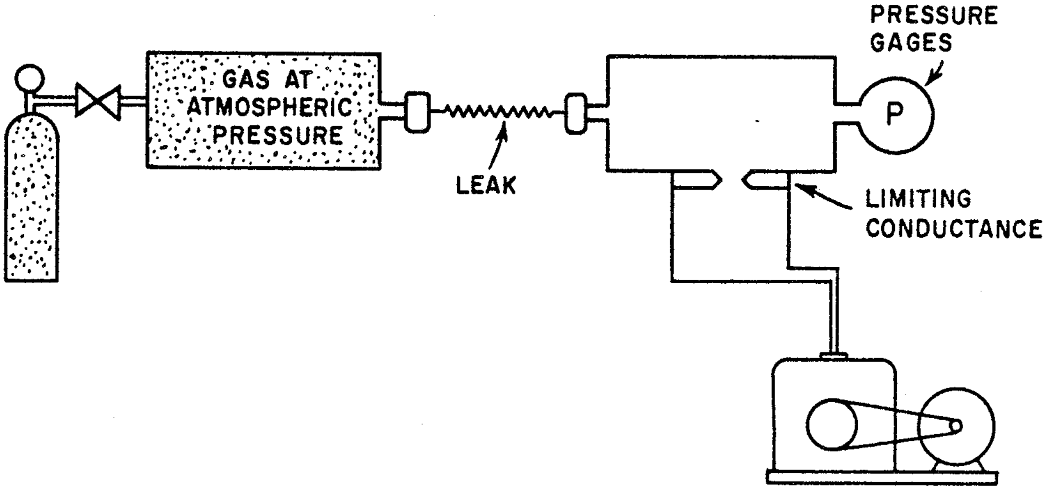


Figure 39. Leak Calibration By Pressure Measurement at Constant Pumping Speed. (Reprinted from J.W. Marr, Study of Static and Dynamic Seals in Liquid Rocket Engines, Final Report Phase II, Contract NAS 7-102. (Ref. 45))

rigorously calculated from theoretical grounds. The disadvantage of such a method is, again, that the pressure of the system must be accurately measured. If the leakage Q and the pumping speed S are known, P can be derived using the above equation. This type of system has also been used to calibrate pressure gages. (Ref. 75).

If a mass spectrometer is used as the pressure gage, some accuracy is gained since the error due to outgassing is eliminated. The system described above has essentially the same flow pattern as the mass spectrometer leak detector. In a mass spectrometer leak detector, Equation 1.6-4 takes the form:

$$Q = SKa \qquad \text{Equation 1.6-5}$$

where Q = leakage
S = pumping speed, which is constant
K = conversion faction for pressure from collector current reading
a = collector current

In most cases, a and K are not known, but a proportionality constant is used which consists of the product of these two numbers. Providing that the response of the leak detector is linear, it can be used to calibrate leaks by comparison to standards calibrated by other methods.

1.6.5.5. MEASUREMENT OF ALPHA RADIATION

A fifth type of primary standard for leak calibration has recently been described by Professor B.R. Sundheim (Ref. 76) of New York University. Radioactive polonium is an alpha emitter. Alpha particles are helium atoms stripped of their electrons. When alpha particles come in contact with other atoms, they acquire the necessary electrons to become helium molecules. Since the radioactivity of the polonium sample can be measured accurately the number of alpha particles evolved is known and therefore, the amount of helium gas being formed can be predicted. In this way pressure-measuring instruments may be calibrated accurately. The major advantage of this technique is that it provides a check on the calibration by measurements which are made through independent techniques. The only source of error is possible absorption or entrapment of the helium within the leak, and this may be obviated by good design and/or the establishment of steady-state conditions.

The above technique is limited to helium gas calibration. It can be used effectively only in the low-leakage ranges ($\ll 10^{-8}$ atm cc/sec), because of the shielding that would be necessary in the use of this technique at higher concentrations of radioactive materials. However, the technique is an excellent supplement for the other methods described, for it is most effective in the range which is difficult to measure by other techniques.

1.6.6. METHODS OF USE

Since there are many variables which can affect the sensitivity of testing, it is necessary that calibration or qualification be performed. This must serve to determine the users ability to detect leakage in the item under consideration, and in some cases, to provide means for quantitative measure of the leak rate. It is, therefore, imperative that the test be calibrated, not merely the instrument.

Figure 40 shows two alternative positions for a reference leak during leakage measurement. If the reference leak is connected as in Method 1, tracer gas is applied to the hood with the valve open, and reading A is taken, representing leakage through the system and reference leak. Tracer gas is then applied to the hood with the valve closed and reading B is taken, representing leakage through the system only. Reading A is repeated to check the calibration. If the response is linear, the leakage through the system is

$$\frac{B}{A - B} \times \text{reference leak} = \text{system leakage} \qquad \text{Equation 1.6-6}$$

Method 1 is used if the leak does not contain its own gas supply. For leaks carrying their own gas supply Method 2 is used.

If the reference leak is connected as in Method 2, search gas is first applied to it and reading C is taken. Search gas is then applied to the hood and reading D is taken. Reading C is repeated to check the calibration. If the response is linear, the leakage through the system is

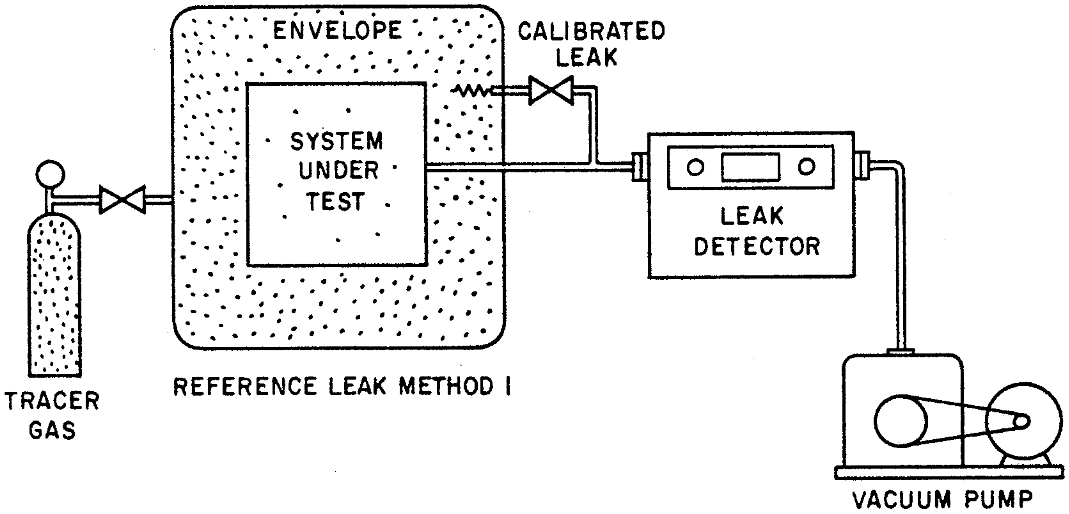
$$D/C \times \text{reference leak} = \text{system leakage} \qquad \text{Equation 1.6-7}$$

Method 1 gives better assurance of the same concentration of tracer gas in all phases of the test (which is essential for valid results), but it is only as accurate as the linearity of the detector response, since the readings are additive. Either method will show whether the system leakage rate is greater or less than the reference leak.

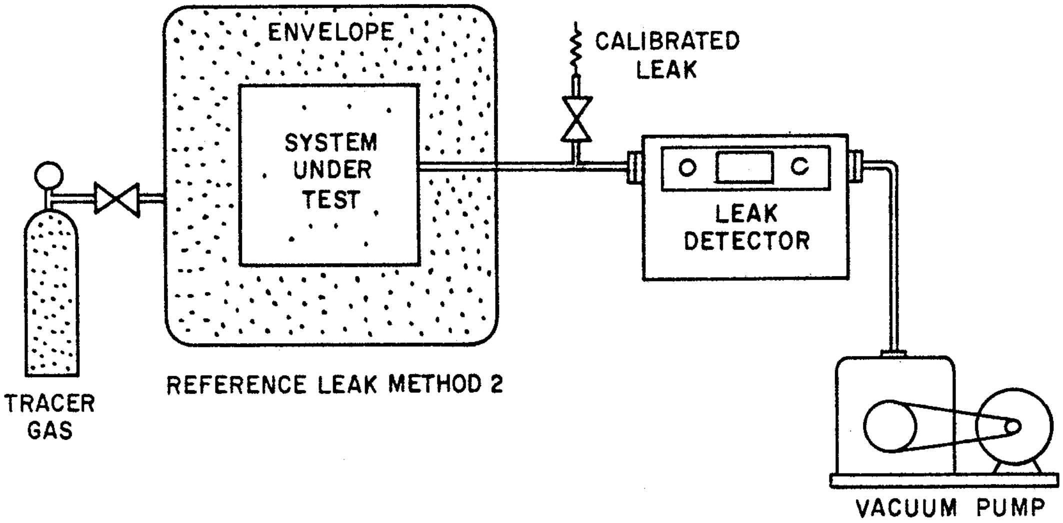
In practice, Method 1 is easy to incorporate in a rigid hood in a permanent test position. It is not always easily arranged with a flexible hood.

In all cases it is necessary to continue the application of tracer gas until the reading settles down to a steady value. For example, with the reference leak connected as in Method 1, tracer gas is passing through the reference leak into the space between it and the valve while reading B is being taken. When reading A is repeated there will be a momentary surge until this little concentration of tracer gas has been cleared.

Obviously, for a true calibration, the leak should be located at the most remote point of the test. It is extremely important when establishing sensitivity that the tracer gas be applied to the leak for the same period of time as it is applied to each suspected area. The reasons for this are tracer gas



REFERENCE LEAK METHOD I



REFERENCE LEAK METHOD 2

Figure 40. Alternative Positions for a Calibrated Leak on a System.

absorption on the walls and the effect of time constant due to large volumes.

For accurate results the unknown leak should be placed in the same port as the standard leak and under identical conditions.

If the object to be tested is located immediately adjacent to the inlet throttle valve, calibrate by placing the known leak in that position. If the object to be tested is a vacuum chamber, or an object to be located in a vacuum chamber, and the leak detector will be connected to the fore-line, calibrate by locating the known leak in a representative position in the vacuum chamber. In this case output versus time must be known.

The above procedures are used in leakage measurement. For leak location with a detector probe (Section 1.3.1.2.2), the testing procedure should be calibrated by a method that obtains test sensitivity and not merely instrument sensitivity. For example, if a helium mass spectrometer leak detector is used in the detector probe (reverse-probe) mode, a calibrated leak placed in the manifold of the instrument will obtain the instrument sensitivity. This would be higher than test sensitivity because it would not take into account the helium loss by diffusion between the leak and the detector probe, the loss of sensitivity due to absorption of helium on the walls of the probe, or the loss of sensitivity due to the higher pressure in the mass spectrometer.

A better way to qualify the instrument is to detect leakage from a synthetic leak (for example those described in Section 1.6.2.3.) or to measure the sensitivity of the detector to helium concentration in the atmosphere. The latest data (Ref. 77) indicate 5.24 parts per million of helium in air.

When using glass calibrated leaks placed inside a vacuum chamber, the helium permeation through the glass envelope of the calibrated leak as well as that through the quartz membrane must be considered. The rate given by the manufacturer is for helium permeation through quartz membrane only.

When a system is calibrated by using a pure helium leak with a one-atmosphere pressure differential across it, the rate obtained during an actual test must be that for pure helium across a one-atmosphere pressure differential. If, in the actual test, the helium is reduced in concentration, either by being mixed with another gas, or increased in pressure, the calibration must be adjusted accordingly.

The reference leak should be of the approximate value of the permissible leakage rate, and must be so if the response of the detector to leakage is not linear. The smaller the reference leak, the greater the difficulties associated with it. If the reference leak is substantially different from the permissible leakage (a contingency which may result from the difficulty of making small reference leaks), the response of the detector to different leakage rates becomes important. This is discussed in Section 1.6.3.

Appendix C includes a tentative specification for calibration of mass spectrometer leak detectors. This American Vacuum Society technique (Ref. 78) makes it possible to compare the sensitivity of various instruments.

1.6.7. GENERAL REFERENCES

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Section 1.7.

GUIDELINES FOR WRITING SPECFICATIONS

This chapter discusses the determination of correct values for specifying leakage and suggests rules to be applied in writing leakage specifications. It is not the object of this chapter to write a universal leakage specification procedure. Rather, the aim is to provide guidelines and necessary steps for deriving the specifications. The guidelines describe the necessary criteria for use in leakage measurement.

1.7.1. MAGNITUDE OF TOLERABLE LEAKAGE

A suggested approach is:

1. Determine the leakage rate which causes system failure during the operational life of the system. If more than one type of system failure is possible, determine leakage rates for the larger ones only.
2. Determine the leakage which produces hazard to either equipment or personnel.
3. Determine the system leakage rate which produces an unacceptable appearance of the system.
4. Determine the system leakage rate which represents good construction on the part of the manufacturer. This is reliability determination by means of leakage. This leakage value, to an extent, is obtained by an arbitrary method. Experience may indicate which is an acceptable leak rate, or analysis of the various joints may determine a reasonable leakage value for specification of these joints.

The smallest of the numbers derived represents the allowable maximum leakage on which specifications should be based. These numbers should not be added. Thus far the leakage is specified in terms of the operating fluid. It is necessary to convert this leakage to leakage of tracer gas. Conversion should be done by the method which produces the most stringent leakage specification. The safety factor thereby attained allows for incorrect estimations. Because the accuracy of any leakage standard is low, an additional safety factor of two or more is added to the leakage value. It is much better to add the safety factor to the maximum allowable leakage than to require that the leakage measurement be performed to a high accuracy.

Setting the maximum tolerable leakage should include these considerations:

1. Is the leakage measurement reasonable in cost? If the

requirements are set so high that unusual techniques or equipment are necessary to perform the measurement, it might be prudent to re-evaluate the specified maximum allowable leakage and lower the requirements.

2. Is the maximum tolerable leakage less than the leakage inherent in normal operation of the system? If this is the case, the leakage standard may be revised upward. For example, if the allowable leakage specified is less than the leakage expected from permeation there is not much point in more stringent leakage specifications. Requirements may be relaxed, and the speed of leakage measurements can be increased.

1.7.2. SPECIFICATION CONTENT

1.7.2.1. MEASUREMENT VALUES AND CONDITIONS

Allowable leakage should be specified with the gas used for system operation. The leakage value should be given in units of pressure times volume per unit time with a specified pressure differential across the leak. (Sections 1.3.2 and 1.3.3) Although the leakage value is specified with one gas, leakage measurement can be performed with other gases. (Section 1.4.3) This may be done by setting a formula for correlation between the gases. A correlation which produces a conservative values should be used.

The temperature for the leakage test should be specified as the operating temperature. Although leakage is not drastically affected by temperature changes, the hole size may be greatly affected because of thermal expansion of the materials.

Perhaps the most important condition to specify is the pressure difference across the leak. This should be the highest operating pressure of the system. Whenever possible, leakage measurements should not be performed at low pressures only. A test must be made at the maximum operating pressure to reveal any self-cleaning leaks, check valve leaks, and leak expansion. (Section 1.4.5) The testing should be done both at maximum operating pressure and at one or several pressures below this maximum to completely eliminate the possibility of check valve leaks. However, leakage at maximum operating pressure must be measured first.

1.7.2.2. USE OF STANDARDS (Section 1.6)

A tolerance factor should be included when writing the specification since it is reasonable to expect that the actual leakage will range considerably above and below the measured value (Section 1.6.3). The leakage measurement should be specified very loosely.

Whenever possible, the determination of instrument sensitivity should be called a qualification rather than a calibration to eliminate the necessity of referring to non-existent Bureau of Standards values.To qualify the instrument, the standards and their method of use should be carefully detailed. The standards may come from leak detector manufacturers or from sample leaks produced in the specifier's laboratory. If the leakage measurement is to be done on several pieces of equipment in various locations, the standards used for the various equipments should be similar and procedures should be standardized. The leakage standard should be of a value similar to the specified leakage.

The qualifying test shows that, under the conditions of leakage measurement, it is possible to determine the specified leakage. It is important in detailing the procedure that the test is being qualified and not the instrument itself. The ideal types of leaks to use in this qualification are those which imitate the leakage procedure, i.e., orifices which let the gas into the leakage instrument. Ideally, standard leaks use the same gas which is used for leakage measurement. The gas would be introduced at the same pressure and in the same concentration into the qualifying leak as into the system being tested.

1.7.3. SYSTEM PREPARATION

1.7.3.1. CLEANING

It is desirable to obtain the system in a clean condition. However, if leaks smaller than 10^{-6} atm cc/sec are to be measured, it should be specified that the system should not be cleaned prior to leakage testing. This prevents cleaning fluids from clogging the smaller leaks in the system. (Section 1.4.5)

1.7.3.2. TESTING TECHNIQUE

Providing the allowable leakage is specified and an acceptable correlation is given for the various test gases, it should not be necessary to specify the exact equipment to be used in the test. However, it is necessary to state in some manner whether leakage measurement or leak location is required. (Section 1.1) Leakage measurement is the only reliable method of testing to establish maximum allowable leakage. It might be necessary to specify the enclosure to insure that all gas leaking from the system is collected by the leakage measurement equipment. These specifications should be rigid enough so that there is no question that the gas which leaks out of the system is being measured.

Only in rare instances, where leakage measurement cannot be performed, may leakage location techniques be used for leakage measurement. It should be stressed that this will drastically affect the reliability of the test. When a leakage location technique is used for leakage measurement, the sampling speed should be specified and extremely rigorous methods of qualifying the technique (not the equipment) should be used.

1.7.3.3. SAFETY STATEMENT

Some statement must be made as to the safety precautions necessary while tests are being performed. (Section 1.5.1) If large pressurized vessels are being used, warning concerning proof-testing must be given. Decision must be made by the individual specifying the test whether it is permissible to test at high pressure prior to proof-testing. Proof-testing using a hydrostatic pressure will clog most small leaks. It is, therefore, necessary to decide whether to take the chance of accidental explosion or whether to perform the leakage measurement after testing by pressurizing with gas to a higher pressure.

1.7.3.4. DESIGN FOR TESTING

Testing requirements should be considered early in design of the system. In addition, the system should be designed only after determination of the leakage specification. (Section 1.5.1) The system design should take into consideration such items as location of ports or connections for leakage measurements. The system must be capable of being tested rapidly. It should not contain sealed voids which could drastically increase the length of the testing procedure.

Section 1.8

CHOICE OF PROCEDURE FOR LEAK TESTING

1.8.1. DISCUSSION

One of the most difficult and important decisions in leak testing is which method should be used. A correct choice will optimize sensitivity, cost, and reliability of the procedure; an incorrect choice will be less sensitive and reliable, and much more difficult.

The simplified approach to this problem advocated by numerous authors (Ref. 6, 54 and 79), is to rank the various leak testing methods by means of sensitivity. The engineer then has only to decide what degree of sensitivity is required and choose the method from those offering adequate sensitivity.

There are two major difficulties with such a procedure. First, whenever such a list is produced, doubt arises that the sensitivity range for any particular test is correct. This occurs because of the variety of techniques which may be used to implement any one of the testing methods. Each technique may have different sensitivity. For example, a mass spectrometer leak detector at sensitivity of 10^{-11} atm cc/sec may be used during routine testing with dynamic leakage measurement techniques. This sensitivity may be increased to 10^{-14} atm cc/sec by the static leakage measurement technique. However, the static leakage measurement technique is not the standard method of using this instrument. Therefore, the sensitivity that is stated is subject to question.

The second difficulty in using this list is that individual testing methods are usually optimized for one particular type of testing. Therefore, it is a mistake to compare various methods as to their sensitivitdes under seemingly similar conditions. For example, if the helium mass spectrometer leak detector is compared with the halogen heated anode leak detector for leak location, it is found that in the tracer probe technique on an evacuated vessel the mass spectrometer is a more sensitive instrument by a factor of 10,000. On the other hand, if these two instruments are to be used for leak location on a pressurized system, sensitivity of the heated anode halogen detector is superior by approximately the same amount. The reason for this becomes obvious upon examination. The mass spectrometer leak detector is constructed for operation under vacuum conditions, whereas the halogen heated anode leak detector is primarily an instrument for operation at 1 atmosphere. Therefore, when sensitivity of any two instruments is compared it must be done with one particular test in mind.

In view of these facts, it is recommended that choice of leak testing method be a step by step process. The user should be given a number of simple choices as to the testing procedure.

When these choices are made, the final selection of testing method will be made from only three or four different methods. In this way the choices will be greatly simplified. Figure 41 illustrates the alternative procedures available; each is coordinated with the text by subsection number. This suggested procedure is only a basic guide. Further consideration will produce modification in the original procedure, as the problems involved are clarified.

1.8.2. TYPE OF LEAK TESTING

The first question to be asked is, "should this test reveal the presence of a suspected leak (leakage measurement), or show the location of a known leak (leak location)?" This distinction is discussed in Section 1.1.

1.8.2.1. LEAKAGE MEASUREMENT

Leakage measurement can be divided into two different types: open units which are accessible on both sides and units which are sealed. The second category usually consists of mass-produced items which are to be tested in large quantities. These include transistors, relays, ordinance units, and sealed instruments. Sealed units in multiple quantities should be tested under the guidelines presented in Section 1.8.2.1.2. Single units are discussed in Section 1.8.2.1.1.

1.8.2.1.1. Units Accessible On Both Sides -- Single Sealed Units

With units which are accessible on both sides, the second decision that must be made is whether the unit should be evacuated during the testing or whether the testing can be performed at atmospheric pressure. If one side is evacuated, more rapid results can be obtained because the gases can be expected to reach the detector quickly, with little possibility of stratification. However, evacuation does not always produce the most sensitive and reliable results. If the system is extremely large, high pumping speed is necessary. Such auxiliary pumping will reduce the amount of tracer gas reaching the detector. Other restraints may prevent evacuation to low enough pressure. The structure of the equipment may prevent operation of a detector in a vacuum.

1.8.2.1.1.1. Units Which May Be Evacuated

The first approach to testing units which may be evacuated is to determine whether there is an inherent tracer in the system. Perhaps during normal operation the system contains one of the tracer gases (e.g., helium or halogenated hydrocarbon). If so, the use of a testing method specific for that gas might be preferred, since considerable savings can be realized if the system need not be filled with a tracer gas.

If no inherent tracer is available, the next approach should consider if there is a gage which might be used for leakage measurement already present in the system. This gage might be simply a pressure or ionization gage or, in some fortunate

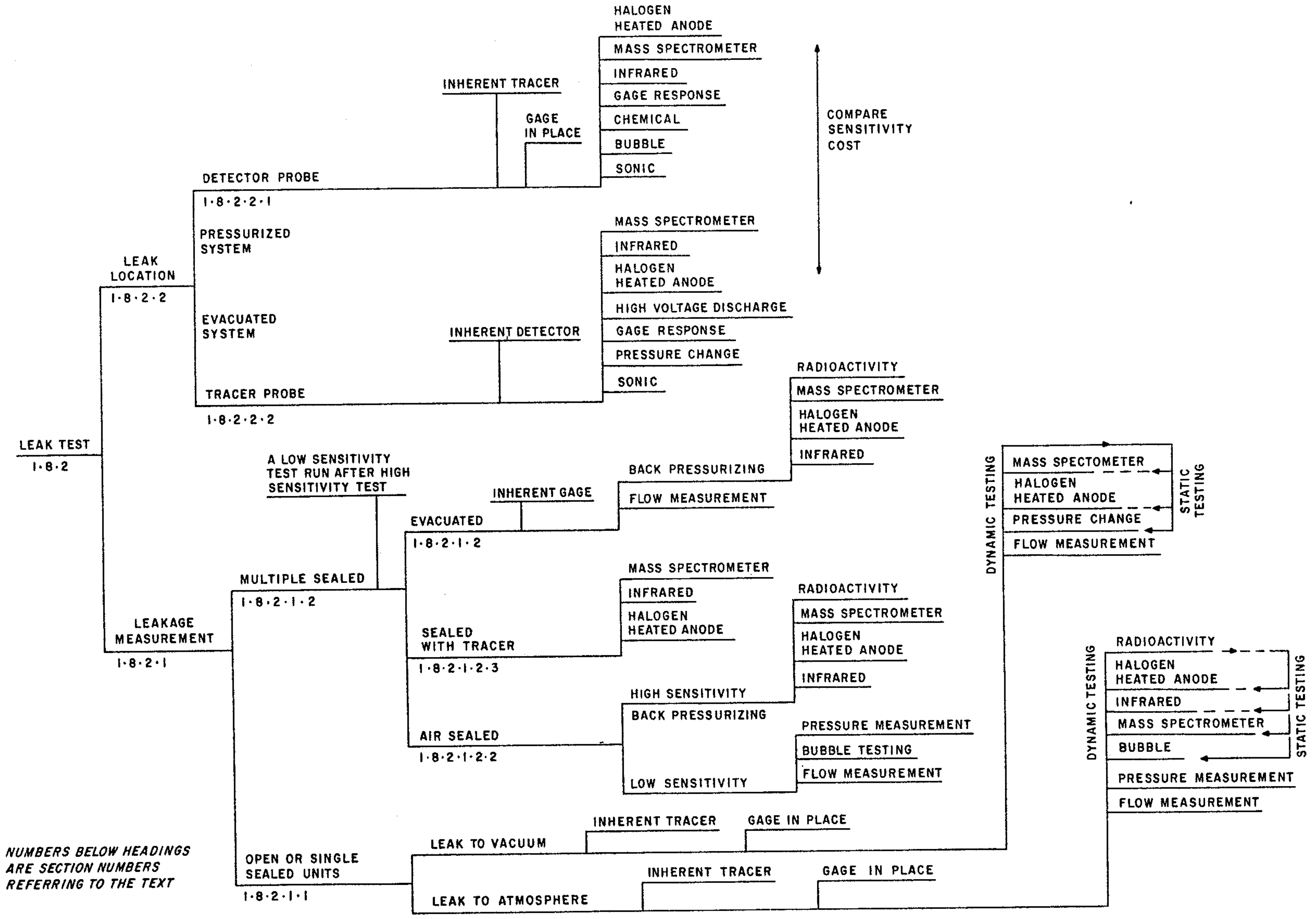

Figure 41. Typical Step-by-step Procedure for Choice of Leakage Test.

circumstances, a mass spectrometer which is in the system as part of the analytical setup. Not only gages which are normally used for leak detection should be considered. Any gas concentration equipment may be used for leakage measurement if it happens to be available. Even equipment not originally intended for pressure measurement may be used if it happens to be available. For example, it is possible to measure the pressure rise in a leaking vacuum tube by means of the current increase in a triode. This is not the original function of the triode, but since the circuit is schematically the same as that of an ionization gage, it may be used for these purposes during testing.

If there is no inherent tracer or gage within the system, standard testing methods must then be considered. In order of increasing sensitivity for testing an evacuated system, these include:

- Flow measurement
- Pressure measurement
- Halogen heated anode detector
- Helium mass spectrometer leak detector

These should be considered individually and accepted or rejected because of their limitations or advantages.

In most cases, all the possible methods should be considered. A more sensitive method might represent higher initial investment, but on the other hand might produce a great saving during the testing programs. Alternatively, it might provide greater reliability in the results.

Once the methods have been chosen, a second consideration must be made. It is usually preferable to perform the tests using a dynamic testing technique. However, a static technique of leakage measurement should be considered. Although this will usually be slower, higher sensitivities can be achieved; these may be worth the extra effort.

1.8.2.1.1.2. Systems Leaking At Atmospheric Pressure

The choice of testing method for systems leaking at atmospheric pressure should be made in the same pattern as suggested for evacuated systems. The first point to be considered is the possibility of an inherent tracer in the system. The second thing to consider is whether a gage exists in the system which may be used to measure pressure or tracer gas concentration. Again, this might not necessarily be the original purpose of the particular gage, but it may be adapted to this use for the leakage measurement procedure.

If an inherent tracer or a gage does not exist, then one of the standard methods of leakage measurements must be considered.

These are again listed in order of increasing sensitivity:

- Flow measurement
- Pressure measurement
- Bubble testing (immersion)
- Helium mass spectrometer
- Infrared testing
- Heated anode halogen detector
- Radioactivity

Whenever applicable, a dynamic testing method should be used. After the various methods have been considered and those whose limitations are unacceptable are rejected, a static-testing technique should be considered. A static technique will increase testing time but will also increase sensitivity.

1.8.2.1.2. Multiple Sealed Units

Sealed units which are made in large quantities present several specific problems in testing. It is imperative that a large number of units be tested rapidly. It is also imperative that no defective units be allowed to pass.

Most of the testing procedures for sealed units involve some evacuation as part of the testing. If the leak in the unit is exceptionally large, the tracer gas will escape rapidly during this partial evacuation. Consequently, high sensitivity tests will be ineffective since they cannot be usefully employed if the tracer gas is already escaped from the system. It is therefore recommended that all parts be tested for large leaks after the high sensitivity tests that are discussed below have been conducted.

The standard way of performing large-leak testing is by the bubble-testing procedure. Bubble testing will reveal large leaks in the system; however, it should only be performed after the high sensitivity testing, since the liquids involved could clog smaller leaks and prevent their detection.

The routine method for high sensitivity testing of sealed units involves the use of a back pressurizing technique. This has been described in Section 1.3. Multiple unit testing may be divided into three sub-categories:

- Testing of evacuated units (Section 1.8.2.1.2.1.)
- Testing of air sealed units (Section 1.8.2.1.2.2.)
- Testing units sealed with tracer gas (Section) 1.8.2.1.2.3.)

In testing sealed units, testing procedures sensitivity varies with each procedure. In order of increasing sensitivity, the procedures are: bubble testing, flow measurements, pressure measurements, infrared methods, heated anode halogen methods, helium mass spectrometer and radioactive testing. The last four methods are applicable for a back pressurizing technique.

1.8.2.1.2.1. Evacuated Unit Testing

With evacuated units the choice of testing technique is relatively simple. If the system contains a gage which may be used to show the presence of gas contamination, it is the first testing method to be tried. If such a gage does not exist, a flow measurement technique may be considered. Finally, if this is not sensitive enough, a back pressurizing technique must be used. The sensitivity of the back pressurizing technique has been given in the previous section. The only other consideration in the choice of a technique is that, after testing, the units must be passed through a bubble testing procedure to locate the exceptionally large leaks.

1.8.2.1.2.2. Units Sealed With Ambient Gas

Testing procedures for these units may be divided into two categories: Low sensitivity testing by either bubble testing technique, flow measurement, or pressure measurement and high sensitivity testing using the back pressurizing technique. Again, the testing procedure must include a final check for large leaks.

1.8.2.1.2.3. Units Sealed With Tracer Gas

Units sealed with tracer gas may be tested by dynamic or static test for the concentration of that gas on the inside of the unit. Generally, this will involve a test which is more sensitive than an equivalent test that involves a back pressurizing technique would be. Such testing is usually cheaper since it involves fewer steps. As in the case with the other methods a final test by means of bubble testing must be performed to locate exceptionally large leaks.

1.8.2.2. LEAK LOCATION

Leak location can be subdivided into a tracer probe technique and a detector probe technique.

The tracer probe technique (Section 1.8.2.2.2.) is usually used when the system is evacuated and the tracer gas comes from the outside. The detector probe technique (Section 1.8.2.2.1.) is used when the system is pressurized and testing is done at ambient atmosphere. Usually the tracer probe technique is more rapid because the gas traveling in the vacuum reaches the detector more rapidly. On the other hand, a higher pressure differential may be used with the detector probe, and therefore leaks of a smaller conductance may be found.

In using either technique it is important that leak location be attempted only after the presence of a leak has been ascertained. In choosing between the atmospheric and vacuum techniques both alternatives must be examined. When satisfactory choice of one technique has been made, it is a good idea to compare it with a satisfactory choice in the other technique to see if reduced cost or easier test method might be possible.

1.8.2.2.1. Testing at Atmospheric Pressure (Detector Probe Technique)

In testing a system which is leaking into atmosphere the first consideration is whether the leaking fluid may be used as a tracer. This will always be the procedure when using either the sonic method or the bubble-testing method. However, the tracer might be of a composition which will also prove satisfactory for use with the other testing methods. In order of sensitivity these methods of leak location are: chemical testing, gage response, infrared, mass spectrometer, and the halogen heated anode detector.

1.8.2.2.2. Testing of Evacuated Systems (Tracer Probe Technique)

In testing of evacuated systems the first consideration is whether there is an inherent detector within the system. This may be a pressure gage of some type or, more desirably, a gage which is specific for a tracer gas which may be used. If such a gage does not exist, the methods in order of increasing sensitivity which may be used for leak location are: sonic, pressure change gage response, high voltage discharge, halogen heated anode detector, infrared detector, and mass spectrometer.

These methods must be individually examined to see if their limitations and advantages are suitable to the particular system being tested.

The methods discussed above for leak location are those of primary use. Other methods, for example, radioactivity, are not generally employed because of problems associated with their use. However, if one of the above methods is not satisfactory, there are more complicated methods that may be considered.

Section 2.1

MASS SPECTROMETER

2.1.1. PRINCIPLES OF PROCEDURE

The mass spectrometer ionizes molecules and separates them in terms of their mass in a magnetic and electrostatic field. In leak testing, , the mass spectrometer is used as a detector for tracer gas. The mass spectrometer and its associated pumping system are optimized to detect the gas being used as the tracer. Detection and measurement usually consist of observation of the intensity of the ion current in the spectrometer tube.

2.1.1.1. CHARACTERISTICS

Several manufacturers produce mass spectrometer leak detectors. These devices have in common sensitivities of 1×10^{-10} to 5×10^{-11} atm cc/sec of helium. They use hot filaments as electron sources. Ions are produced in a source chamber by electron bombardment and accelerated electrostatically through an analyzer magnet field. Tracer gas ions are deflected either 60, 90 or 180 degrees in the various machines and collected on a target plate connected to the grid of an electrometer tube. Output of the electrometer tube is amplified and presented on a multirange output meter. Liquid nitrogen traps, oil diffusion pumps, and inlet throttle valves are used to allow attachment to vacuum systems operating at high pressures.

2.1.1.2. APPLICABILITY

The mass spectrometer leak detector is a commercially available instrument. It is probably the most commonly used leakage testing procedure. Often, no other technique is considered if a mass spectrometer can be used.

The mass spectrometer leak detector may be used in four leak testing modes. Since techniques in the dynamic leakage measurement and the tracer probe leak location modes are similar, these will be treated together. The other two methods are the static leakage measurement technique and the detector probe technique.

Because a vacuum is necessary for operation of the mass spectrometer, leak detection using this technqiue involves the use of a high-vacuum system. However, the components being tested do not necessarily have to be within the vacuum.

2.1.1.3. SENSITIVITY

The fundamental sensitivity of the helium mass spectrometer leak detector is approximately 1 part helium in 10 million parts of gas. When this instrument is used in the dynamic mode, this fundamental sensitivity implies a sensitivity of approximately 5×10^{-11}atm cc/sec. This sensitivity, of course, is reduced if additional pumps are necessary, or if the mass spectrometer is used in the detector probe mode.

2.1.2. PRINCIPLES OF OPERATION

In the sector field mass spectrometer used for isotopic and quantitative analysis of gases, gas molecules entering the evacuated body of the instrument are ionized by a bombarding beam of electrons. The resultant positive ions are accelerated by the influence of a high voltage into a magnetic field whose direction is perpendicular to the plane of the mean path of the ions. Under the influence of this magnetic field, ions of different masses travel on arcs of different radii. Ions of a particular mass pass through a slit and fall on a collecting electrode; from it they leak to ground through a high-value resistor. The ion current through the resistor is amplified and read on a meter. By varying the magnetic field or the accelerating electric field, ions of any chosen mass can be collected.

In analytical mass spectrometers, it is not customary to vary the electric field, since this tends to produce so-called mass discrimination effects; rather, the mass spectrum is scanned by varying the magnetic field with an electromagnet. A mass spectrometer designed expressly for leak detection, however, does not have to be capable of scanning a mass spectrum. The instrument can be tuned for use with a particular search gas by adjusting the electric field. A permanent magnet can be used to provide the deflecting field. Furthermore, since resolving power is of little consequence in a leak-detecting mass spectrometer, the radius of curvature of the ion path can be quite small. The operation of a spectrometer tube is shown in Figure 42.

When a beam of positive ions, accelerated by a potential V, is passed through a magnetic field of strength H, the ions are sorted out, according to their values of e/m, where m is the mass of the ion, and e the charge. The radius of curvature R of the path of any given type of singly charged ion is determined by the relation

$$R = 143.9 \frac{(MV)^{1/2}}{H} \qquad \text{Equation 2.1-1}$$

where R is expressed in centimeters, V in volts, H in oersteds, and M is the molar mass of the ion in grams.

For example, for R = 4.0 cm and H = 1500 oersteds, the values of V for helium (M = 4) and nitrogen (M = 14, since N^+ is formed), are found to be 434 volts and 124 volts, respectively. Thus, for a given value of the radius of curvature, the ions reaching a collector may be differentiated by varying the value of V. The magnitude of the ion current at given voltage setting will depend on the concentration of the ions in the beam, that

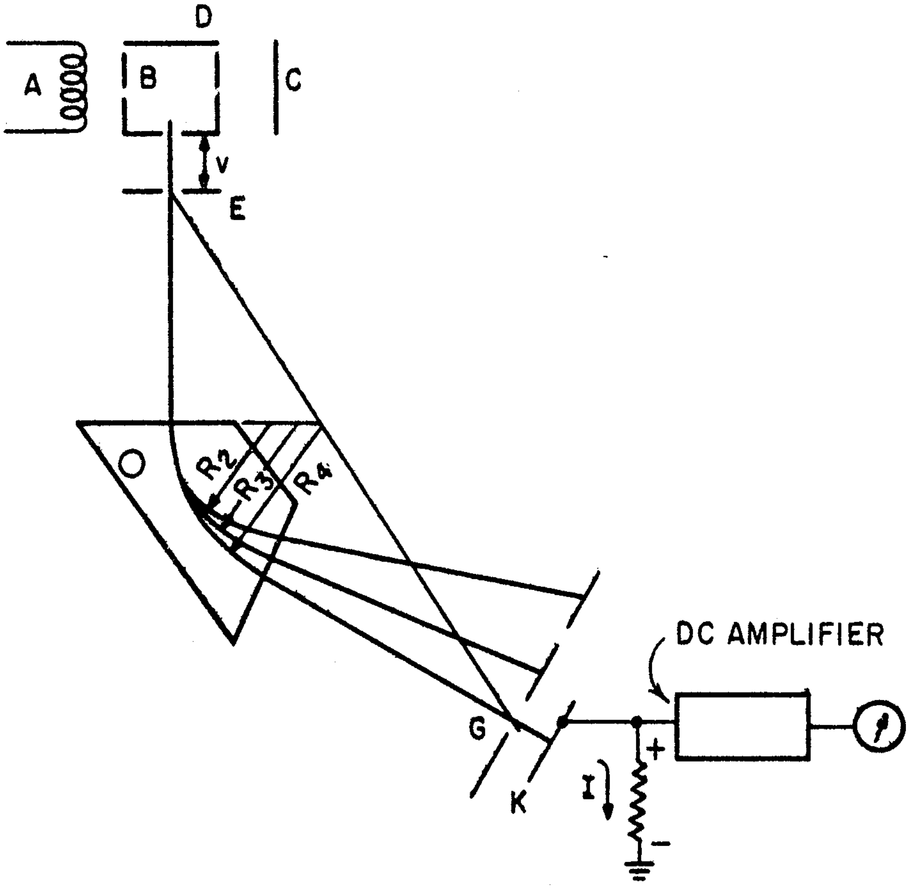

Figure 42. Schematic Diagram of Spectrometer Tube Operation.

is, on the rate at which the corresponding molecules leak into the spectrometer.

The ions are repelled through the slit B by a small positive voltage, and accelerated between B and E by the potential V. Tracer gas ions sorted according to the value of R, the radius of curvature, enter the slit G and are discharged on the collector plate, causing a positive current, I, to flow through the high resistance to ground.

The mass spectrometer operation is detailed in the ensuing paragraphs.

A beam of electrons of stabilized intensity from a heated filament A is attracted by a potential difference of a few volts towards an ionization chamber B, passes through this chamber and is collected on an anode C. The mass spectrometer unit is contained within the same vacuum system as the component being tested. Therefore, the electrons, on their way through the ionization chamber, produce positive ions from the gas molecules within the system.

A repeller plate D which covers one end of the ionization chamber carries a positive voltage and repels the positive ions towards the grounded plate E. The ions are further accelerated by a voltage difference between B and E, and emerge through the slit in the form of a narrow beam. Under the influence of the field H of a permanent magnet that is mounted on the mass spectrometer chamber, the positive ions are made to follow circular orbits and eventually encounter a plate carrying a slit G.

The center of this slit and of slit E lie at opposite ends of a 60-degree sector of radius 4.0 cm. The magnetic field is approximately 1500 oersteds, so that singly-charged helium ions (mass 4) pass through the slit G when the accelerating voltage (i.e. the voltage difference between the B and E) is about 434 volts. Behind the collector lies a suppressor plate J carrying a slit, and then a collector plate K. Ions which reach K leak to ground via a 10^{11}-ohm resistor, develop a voltage across it which is applied to the grid of an electrometer tube. (Both this tube and the high-value resistor are mounted in the vacuum system of the collector assembly.) After further amplification, this voltage produces a deflection on the leak rate indicating meter which is proportional to the partial pressure of helium in the system, assuming that only helium ions reach the collector.

A suppressor plate is placed just ahead of the collector plate. This plate is operated at a potential near that of the ionizing region so that those ions which have lost energy by collision cannot pass through the slit in the suppressor plate and do not reach the collector plate. Without the suppressor voltage, the instrument operating with a small concentration of helium in air at the maximum allowable tube pressure gives a spectrum in the region of helium as shown by the upper curve of Figure 43. The sloping background curve is due to ions which have been

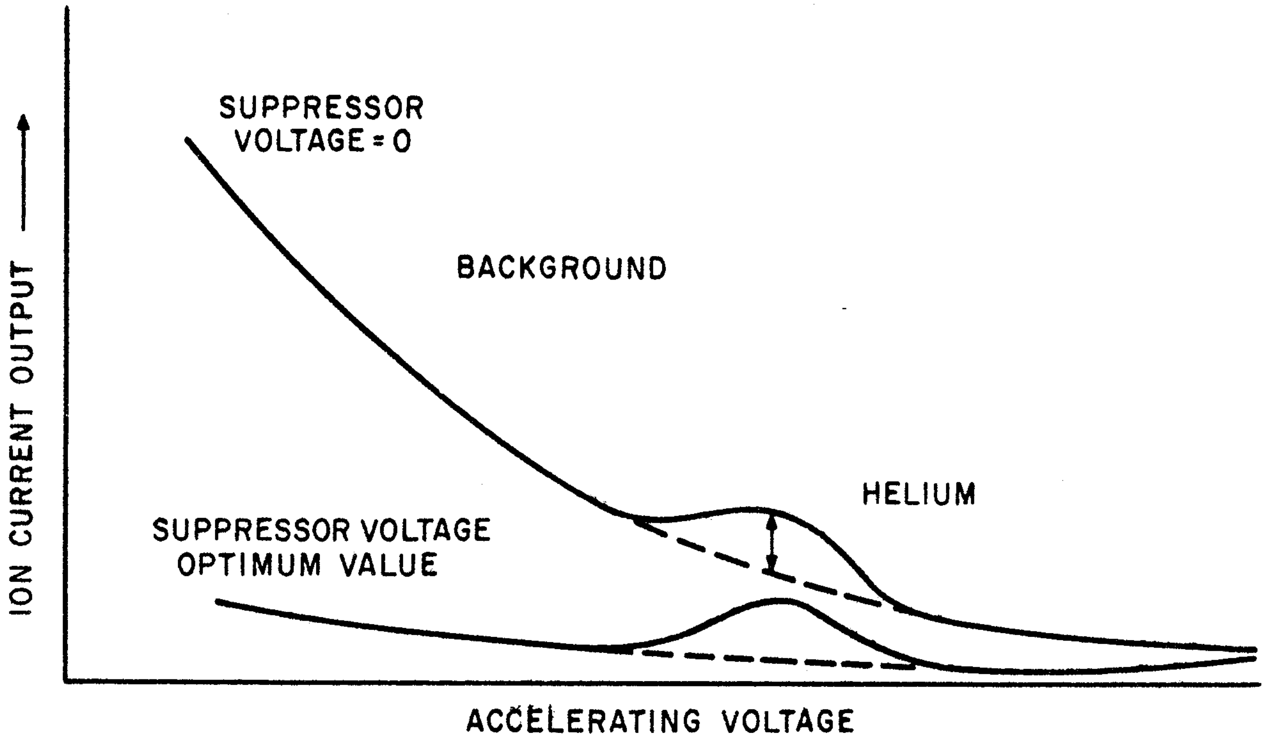

Figure 43. Effect of Suppressor Voltage on Ion Current Output. (Reprinted, with permission, from A.I.E.E. Technical Paper 46-142 (May 1946), W.G. Worcester and E.G. Doughty, The Institute of Electrical and Electronics Engineers, Inc., New York)

slowed down or deflected by collisions during the transit from the ion source, and which therefore happen to pass through the collector slit. All such ions have less than normal energy, since each collision results in loss of energy. A potential barrier through which these scattered ions cannot pass can be erected by applying a positive potential, essentially equal to the accelerating voltage, on the suppressor plate. A spectrum results, similar to the lower curve of Figure 43, in which the presence of helium is much more easily discerned than in the upper curve.

The first stage of the amplifier is mounted inside the mass spectrometer tube. This keeps the high resistance and the grid connections dry to minimize leakage currents. It also makes the leads as short as possible from collector plate to the grid of the first tube, provides electrostatic shielding for the input stage. The remainder of the amplifier is mounted in the leak detector control panel.

The ideal amplifier emits no signal when the target collects no current. Actually, a small signal is emitted which can be split into a fluctuation and a drift.

The fluctuation is an uncertain signal, the average value of which is zero by definition. It is this fluctuation which limits the sensitivity of the detector, because a signal can only be detected if its amplitude is at least equal to the fluctuation.

The drift consists of an output voltage which varies monotonically over a long period of time. It does not limit the sensitivity of the detector, because the zero reading can always be reset.

Other gases may be used, but helium is most often chosen as the tracer gas for the following reasons:

1. Helium's molecular weight yields a rate of diffusion through a leak greater than any other gas except hydrogen.

2. Helium occurs in the atmosphere to an extent of only one part in two hundred thousand parts of air (Ref. 77).

3. There is little possibility that an ion from another gas will give an indication that can be mistaken for helium. One exception is pure deuterium gas.

4. Helium's low molecular weight makes possible a simple construction for the mass spectrometer.

When helium is inapplicable or unavailable, several alternative gases are possible: argon, hydrogen, and neon. In such cases, a modified mass spectrometer has to be used. In principle, it should be possible to convert a commercial helium leak detector over to the detection of some other gas. However, in practice this can be expensive and unsatisfactory. Consequently, considerable attention has been given to other designs of mass spectrometer. The problems encountered stem from the higher concentrations of other gases in the atmosphere and from the difficulty in separating the desired ions from unwanted ions.

Hydrogen and argon have been used fairly extensively with mass spectrometers. However, the normal concentration of helium in air is 1 part in 200,000; the normal concentrations of hydrogen and argon are about 1 part in 10,000 and 1 part in 100, respectively. Consequently, the background due to air seeping into the system is considerably higher with these gases. Suitable means must be used to compensate for this background.

Hydrogen is more widely available and costs less than helium. It is more readily ionized than helium, yielding a higher sensitivity for leakage measurements. One disadvantage is that hydrogen is present as background in a vacuum system. Moreover, it is formed in the spectrometer ion source as a breakdown product of hydrocarbons and water vapor, especially when these come into contact with the spectrometer filament.

Special precautions must therefore be taken in the construction of the ion source. Vapor contamination from the equipment under test is kept out of the spectrometer by a liquid nitrogen trap in the inlet line to the ion source. The ion-source construction is "clean," in that organic materials are avoided and the demountable seals use metal foil gaskets. Finally, the mass-spectrometer pumping system is comprised of a mercury diffusion pump with its own liquid nitrogen trap. This results in a significantly lower hydrogen background.

Cossuta and Steckelmacher (Ref. 80) have attained a sensitivity of 4 x 10^{-10}atm cc/sec on a lens mass spectrometer using hydrogen as the tracer gas. With helium, the sensitivity was about half that for hydrogen. The background stability was greater in the mass-4 than in the mass-2 region. Therefore, the limiting sensitivity was about the same for both helium and hydrogen.

Argon could be used as a substitute, but at a reduced sensitivity. It retains many advantages such as high ionization efficiency, chemical inertness, low cost, and reasonably low background in air. The principal disadvantage is that argon at mass 40 is poorly resolved from the background peaks. Both the nitrogen and oxygen peaks of air and hydrocarbons normally present in the vacuum system increase the general background in the mass 40 region. Thus argon is difficult to detect and measure. Some of these background difficulties may be overcome by use of the doubly ionized argon peak at mass 20. Although this produces a lower sensitivity than measurements in the mass 40 region, the mass 20 peak appears in a relatively clean region of the spectrum. Thus background ceases to be a problem. Slow diffusion of argon limits its use for leak location.

2.1.3. EQUIPMENT DESCRIPTION

2.1.3.1. TYPICAL MASS SPECTROMETER LEAK DETECTOR

The typical mass spectrometer leak detector contains:

1. A vacuum pumping system for pumping down the analyzer and associated lines.
2. A cold trap for pumping condensable vapors.
3. An appropriate vacuum coupling for connecting a standard leak (or the object to be tested).
4. Flanges for connecting objects or systems to be tested.
5. Valves for controlling the pumping down of the test object, throttle valve, and for special methods of testing ("accumulator" valve).
6. A leak indicator, which is usually a meter with an audio signal added.
7. Vacuum gages.
8. Analyzer and appropriate electronic circuitry.
9. The leak detector pumping system to evacuate the test object.

A schematic diagram of a typical vacuum system is shown in Figure 44.

2.1.3.1.1. Vacuum Pumping System

The mass spectrometer tube operates in a high vacuum. Therefore, a complete, self-contained vacuum system must be provided as part of the leak-detector system.

A motor-driven, oil-filled rotary pump is used to maintain low pressures into which the diffusion pump in the mass spectrometer can operate. This fore pump is capable of reducing pressure to about 2 x 10^{-2} torr.

Since the vacuum produced by the fore pump is not sufficient for proper operation of the mass spectrometer, an oil diffusion pump is used in series with the fore pump. In an oil diffusion pump, high-speed jets of oil vapor impart a momentum on gas molecules toward the output port. The vapor jets are produced by heating a pool of oil in the base of the pump. The vapors rise in a column and are deflected down and out toward the walls where they are condensed and returned to the pump. The diffusion pump is usually air-cooled by an electric blower. An oil diffusion pump cannot exhaust against atmospheric pressure, thus the fore pump connected to the exhaust port provides pressures lower than 10^{-1} torr into which it can exhaust.

The exposure of hot organic pump fluids to atmospheric

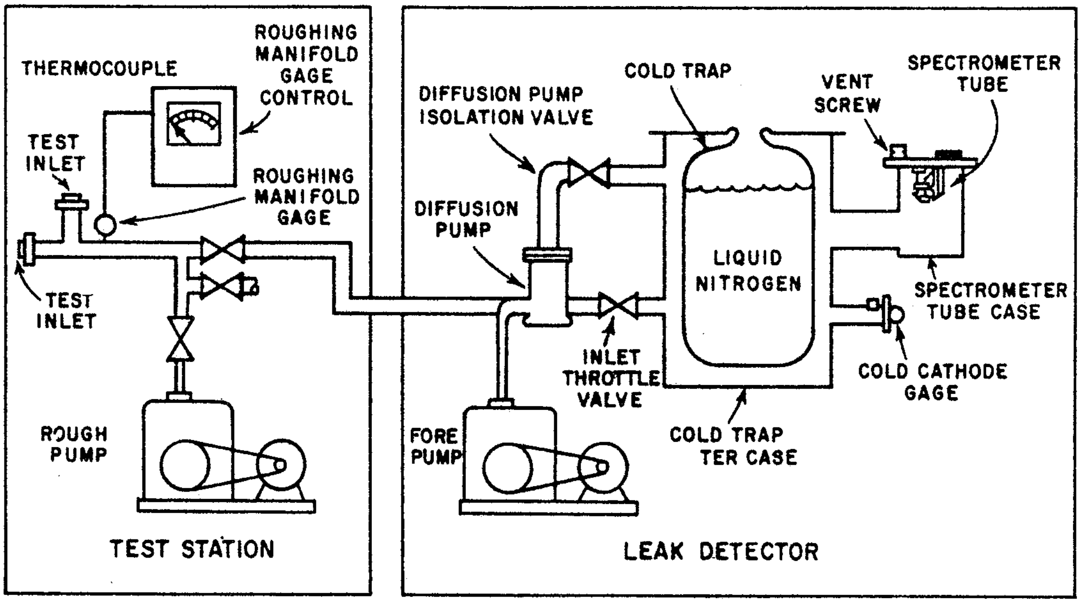

Figure 44. Mass Spectrometer Leak Detector Pumping System. (Reprinted with permission from Vacuum Products Business Section, General Electric Company, Schenectady, New York.)

pressure would result in decomposition of the fluid. Therefore, the system must first be exhausted to a pressure less than 10^{-1} torr before the diffusion pump is turned on. Similarly, it is important to shut off the diffusion pump first, and then wait to allow the oil to cool before turning off the mechanical fore pump or venting the diffusion pump to atmosphere.

The fore pump, diffusion pump, and blower should be interlocked for maximum protection of the diffusion-pump oil. A thermal, self-restoring circuit breaker should protect the fore pump while providing intermittent operation to maintain vacuum in certain instances. The blower should be fused in conjunction with the diffusion pump so that a failure of the blower causing the fuse to blow will disrupt heating power to the diffusion pump. The diffusion pump then cannot be turned on unless the fore pump is started.

One company, (Vacuum Instruments Corporation) manufactures a detector with a novel pumping system. Instead of a diffusion pump, the pumping is by means of chemical gettering. It is claimed that by the preferential chemical reaction of the residual gases, the concentration of helium in the analyzer tube is increased.This is supposed to increase the inherent sensitivity of this type of leak detector.

2.1.3.1.2. Cold Trap

The purpose of a cold trap in a vacuum system is to condense vapors such as water and oil, and entrap them onto a cold surface. This reduces the pressure to a negligible value. For example, the vapor pressure of water at 20 C is 17.5 torr, but its vapor pressure at liquid nitrogen temperature (-196 C) is approximately 10^{-15} torr.

The lower the temperature of the trap, the more effective it is in reducing pumping time and assuring a clean vacuum system. The recommended refrigerant is liquid nitrogen. Liquid air, though equally effective, may be an explosion hazard because of its liquid oxygen content. The volume of the liquid nitrogen cold trap should be large enough to permit 24-hour effective operation with one filling. Good vacuum practice prescribes that the cold trap be maintained at a reasonable level. If the cold trap is neglected and allowed to go dry, there should be no serious concern. The most notable effect will be the inability of the system to maintain low enough pressures during operation. This should not, however, be made a routine practice as the operating efficiency of the system is considerably reduced, and the system will exhibit a high background and short filament life.

If liquid nitrogen is not available, a slurry of dry ice and acetone may be used as a refrigerant. This is not so efficient and should only be used when other alternatives are not available. This mixture also can be a fire hazard!

Welch Scientific Company manufactures a refrigeration unit specifically for leak detector cold traps. This unit uses the adiabatic expansion of compressed air as the refrigeration principle. It may be operated on compressed gas cylinders or on a filtered shop air supply.

2.1.3.1.3. Test Inlet

The vacuum manifold connecting the test piece to the system usually consists of several vacuum compression-seal couplings. It is good practice to leave in place a standard leak in one of these inlets so a rapid qualification (or calibration) may be performed after each leak detection procedure.

2.1.3.1.4. Flanges for Connection to System

Ideally, all systems to be tested will be of a size which fit the test inlet of the detector. This is usually not the case, so a series of fittings are available to connect to the test inlet. Manifolds described in Section 1.5 are often used with the mass spectrometer leak detector where routine testing of similar parts is required.

2.1.3.1.5. Control Valves

Two high-vacuum, bellows-sealed control valves are incorporated in the vacuum system: an inlet throttle valve and a diffusion-pump isolation valve.

The inlet throttle valve is especially designed with a throttling plate that effectively varies the size of the valve opening as it is withdrawn from a fully closed to a fully open position. Its purpose is to restrict the inlet, so that a continuous gas sample can be drawn from a large vessel or from a test piece with large leaks, without letting the leak-detector vacuum system exceed operating pressures.

The diffusion-pump isolation valve serves a dual purpose. First, it provides a means of isolating the diffusion pump from the vacuum chamber, cold trap, and mass spectrometer tube. It is extremely convenient in servicing and maintenance. For example, by closing the valve and venting the vacuum system, the cold trap and mass spectrometer can be removed for cleaning, the filament can be changed, and other maintenance functions can be performed without shutdown of the diffusion pump and fore pump. Thus, the leak detector can be restored to operational status within minutes after reassembly.

On the General Electric LC-20 Leak Detector, the construction of this valve, shown in Figure 45, permits two discrete sensitivities as well as allowing accumulation techniques of leak detection. The valve has a sensitivity choke which provides a constant and fixed sensitivity increase of approximately three times by changing the pumping speed at the spectrometer tube. Opening the valve two to three turns from its closed position

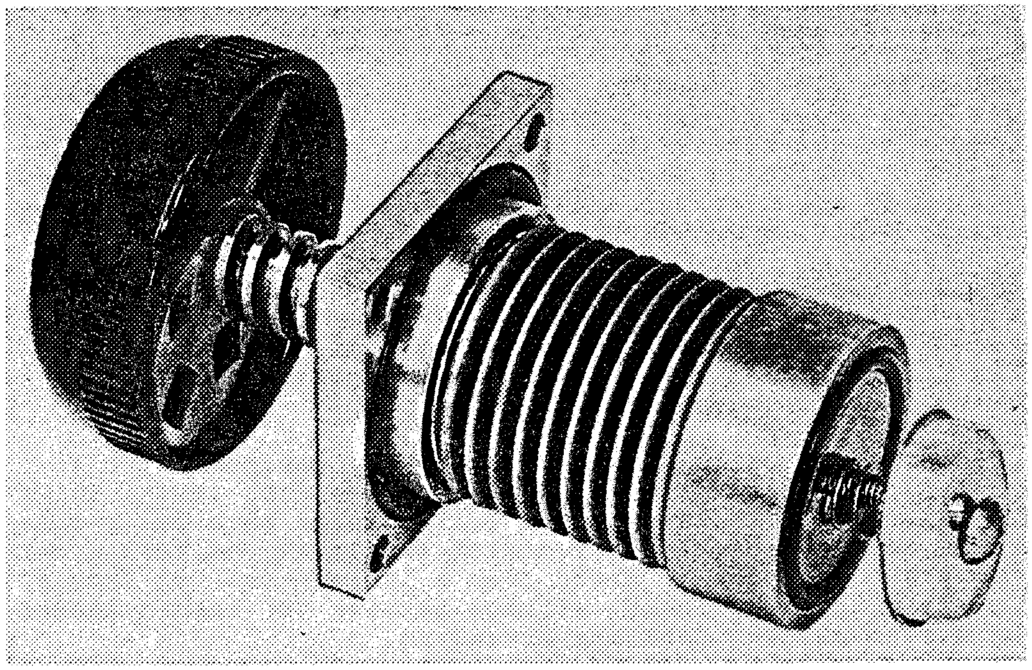

Figure 45. Exposed View of a Diffusion Pump Isolation Valve. (Reprinted with permission of the Vacuum Products Business Section, General Electric Company Schenectady, New York.)

produces a fixed low pumping speed which results in the higher sensitivity. For faster pumping, this valve may be fully opened and the choke lifted off its seat. The sensitivity and pumping speed can thus be changed without disassembly and with reproducible results. It will not be necessary to recalibrate each time the valve position is changed. However, it is important that both calibration and operation be performed at the same valve setting.

As an alternative to the two-position valve, it is possible to use a permanent orifice to reduce pumping speed. Although such an orifice would increase the system sensitivity, it would also increase the system response time (Section 1.3).

2.1.3.1.6. Leak Indicator

The leak indicator is usually a milliammeter operated by the amplified ion current. The detector usually has a multiposition stepping switch so that current of four decades may be read on the meter.

For the convenience of the operator who may be unable to see the output meter while he is probing the test system, a loudspeaker, audio oscillator, and amplifier can be installed on the equipment. An audible note is emitted whose pitch is related to the meter reading and hence to the magnitude of the ion current, i.e., the pitch of the note rises when a leak is probed.

2.1.3.1.7. Vacuum Gages

Mass spectrometer leak detectors usually employ two types of gages: Pirani or thermocouple gages for low-vacuum (atm to 10^{-3} torr) measurement and ionization gages for high-vacuum (less than 10^{-4} torr) measurement. The Pirani or thermocouple gage indicates pressure in the fore pump line and shows when the diffusion pump line may be opened.

The ionization gage is usually of the cold-cathode or Phillips discharge type. This type of gage is more rugged and does not contain a hot filament which could burn out if accidentally exposed to atmosphere.

In the cold-cathode gage, discharge current is a result of a high voltage between anode and cathode and is a function of pressure. The external permanent magnet facilitates ionization by forcing the electrons into a spiral path between the two electrodes. The discharge current is displayed on a meter on the control panel and usually is also monitored by a filament protective circuit. The protective circuit senses when the pressure exceeds safe operating levels and instantaneously removes spectrometer filament power. In some automatically operated leak detectors, the protective circuit will shut the throttle valve if the pressure rises above a safe operating level.

2.1.3.1.8. Analyzer and Appropriate Electronic Circuit

In the twenty-five years that mass spectrometer leak detectors have been in operation, many changes in analyzer and circuit design have taken place. Perhaps the most obvious of these have been the gradual replacement of vacuum tubes by transistorized electronic circuits.

2.1.3.1.8.1. Pressurized Ion Source

Early analyzer tubes were designed with the ionization chamber at a higher pressure than the rest of the spectrometer tube (Ref. 81). This "pressurized source" increases helium gas concentration in the ionization chamber, which increases the concentration of ionized helium atoms formed. Unfortunately, increased pressure also greatly increases the width of the peaks at the base, especially on the high-voltage side of the peak. This produces an increase in background which more than offsets the advantage gained because of the increased height of the peaks. The background is increased because at the higher pressure more nitrogen and oxygen ions lose energy by collision in passing through the region between the source and the magnet, and are collected simultaneously with ions of lower mass.

The insertion of a baffle (Figure 46) between the ionization chamber and the rest of the tube insures a high pressure in the ion source without a correspondingly high pressure in the rest of the tube. Thus, this baffle materially increases the effective sensitivity of the tube.

2.1.3.1.8.2. Modern Spectrometer Tube

The modern mass spectrometer tube source is not pressurized,and the whole tube operates in a volume connected to the cold trap. Usually the entire mass spectrometer tube and detector assembly are completely self-contained on a single, easily demountable flange, Figure 47. All elements of the tube including magnets and filament are self-aligning, eliminating critical alignment problems. The entire assembly can be lifted out for cleaning and parts replacement.

Design of the tube is shown in Figure 48. A beam of electrons from a heated filament is attracted by a small potential difference toward an ionization chamber. The electrons pass through a hole into the chamber. Electrons hitting any molecules in the chamber ionize them. The source magnet makes the path of the electrons parallel to the chamber wall, increasing the probability of collision. This in turn increases the system sensitivity. An ion repeller pushes the positive ions out of the slit in the ionization chamber. An ion lens, consisting of two focusing half-plates which can be separately adjusted to the proper potential, concentrate the ion beam at the center of the slit. Use of such an ion lens increases the intensity of the ion beam and therefore increases the sensitivity of the leak detector.

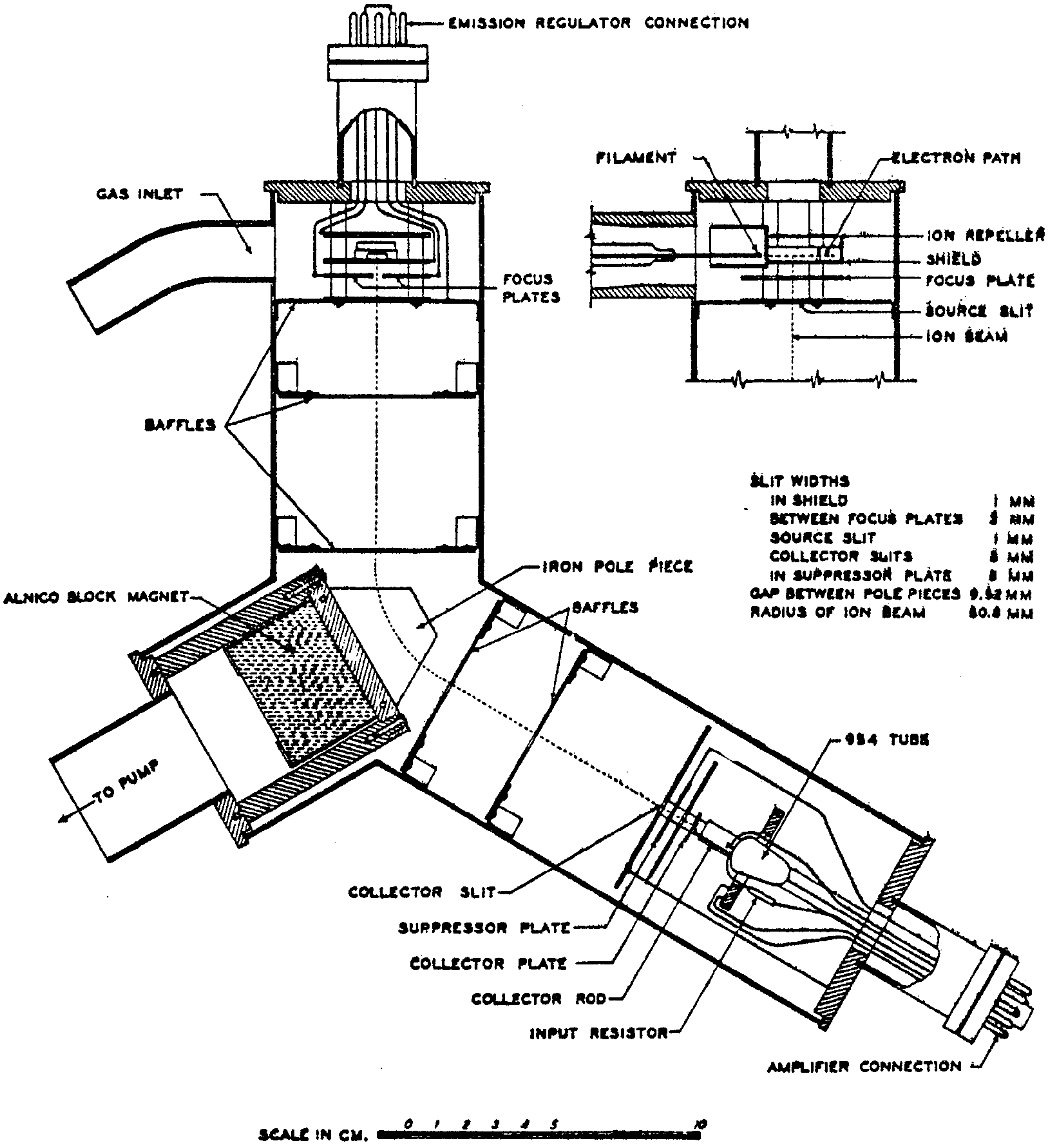

Figure 46. Pressurized Source Mass Spectrometer Tube. (Reprinted with permission, from A. O. Nier and others, Journal of Applied Physics, Vol. 18, January 1947, pp. 30-33; copyright 1947, American Institute of Physics. (Ref. 81)).

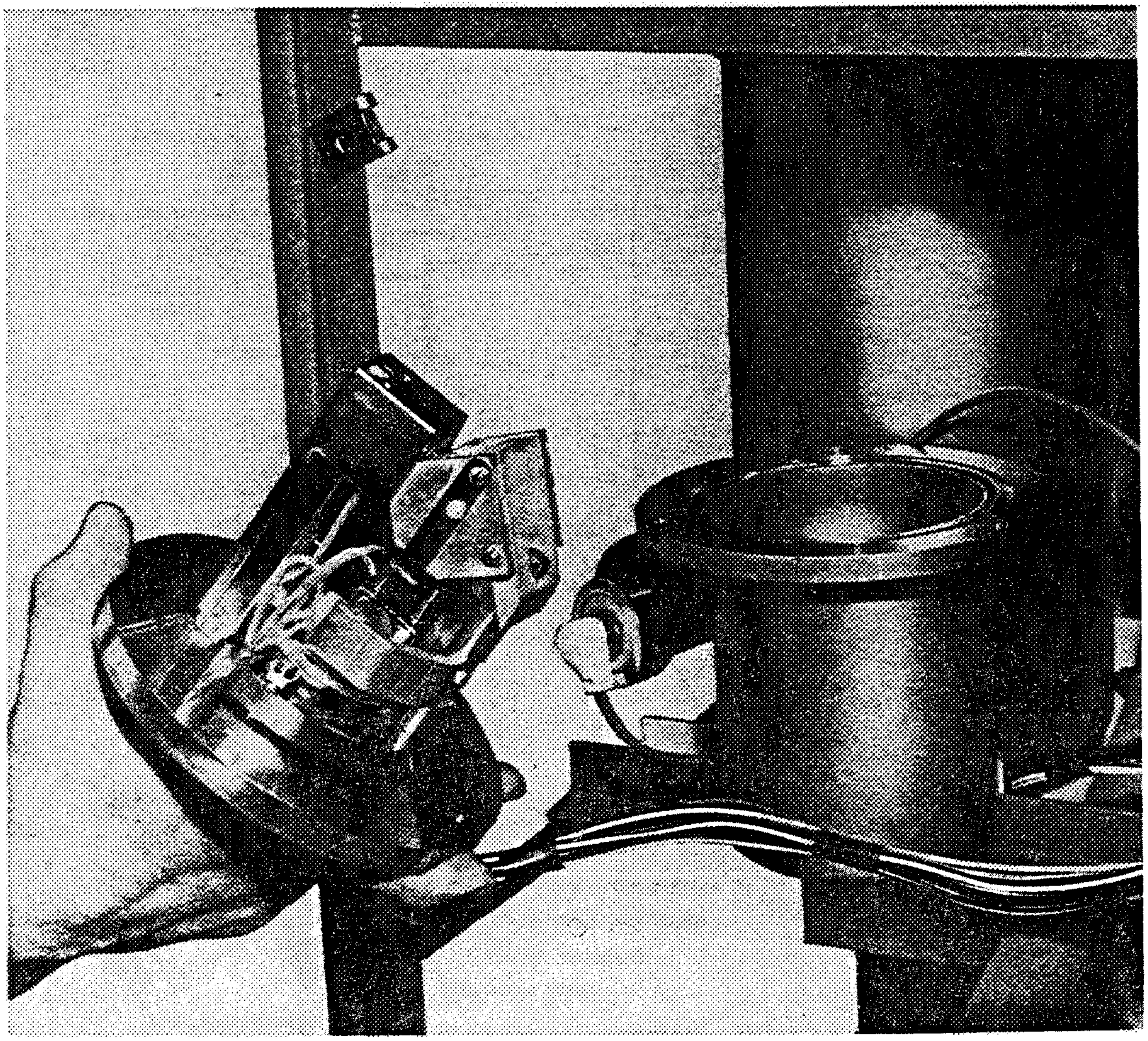

Figure 47. Analyzer Tube of a Modern Mass Spectrometer Leak Detector. (Reprinted with permission of the Vacuum Products Business Section, General Electric Company, Schenectady, New York.)

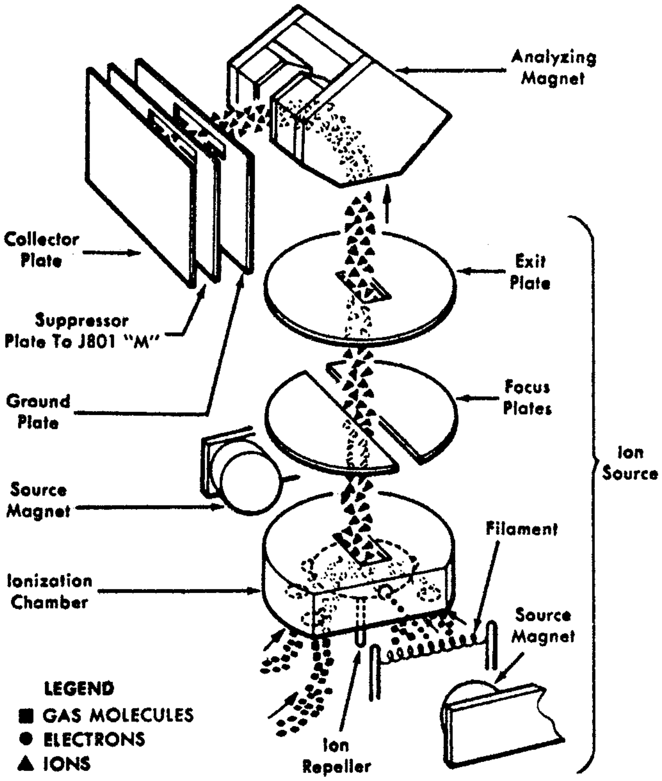

Figure 48. Operation of Modern Mass Spectrometer Analyzer Tube. (Reprinted with permission from the Vacuum Products Business Section, General Electric Company, Schenectady, New York)

The ions are accelerated by the exit plate and deflected by the analyzing magnet. The helium ion beam passes through slits in the ground and suppressor plates to the collector plate.

2.1.3.1.8.3. Ionization Sources

One of the problems encountered in the mass spectrometer leak detector is that of obtaining a reasonable life from the filament. A pure tungsten filament can be used. Although the required emission current is usually only a fraction of a milliampere per square centimeter, the temperature necessary to obtain this current from bare tungsten is at least 2000 K. In the mass spectrometer leak detector the operating pressures are between 10^{-3} and 10^{-4} torr, with occasional bursts of gas bringing the system up to much higher pressures. Even with protective circuits to take care of these bursts, tungsten filaments have a very short life. It is possible to increase the life of the filaments (Ref. 82) by coating them with thoria. These filaments emit electrons at a lower temperature (Ref. 83), making it possible to operate the filaments cooler with a resultant longer life. Some commercial leak detectors are now sold containing thoria-coated tungsten or iridium filaments. Even these filaments sometimes burn out during operation. Consequently, several leak detectors are built with dual filaments. If one filament burns out, the second may be put into operation by electrical switching on the control board.

Ionization of the tracer gas varies with emission current. The emission current varies rapidly with changes in voltage, changes in heat conductivity of the surrounding gas, changes in the surface conditions of the filament due to the presence of different gases, and other similar causes. The emission is therefore kept constant with an automatic regulator.

The ion sources of the analyzer tubes become dirty due to decomposition products of organic molecules which migrate into the ionization chambers. The primary need for the cold trap is to prevent this migration. One leak detector (Veeco Vacuum Corporation) contains a self-cleaning ion source in the mass spectrometer head. In that instrument, the ion repeller is a grid which runs red-hot to burn off contamination deposits which try to form on the electrodes (Ref. 84). On other instruments, the ion repeller is platinum-clad and may be cleaned after system shutdown by immersion in a soft flame.

The need for maximum over-all sensitivity implies the use of the most sensitive available method of ion current detection; but the detector must also meet the important requirements of ruggedness, simplicity of operation, and portability. A d-c amplifier with 100 percent feedback meets these requirements satisfactorily, although it is somewhat less sensitive than certain other methods, such as an electrometer tube and sensitive galvanometer. The amplifier, when used with an input impedance of 10^{11} ohms and an output instrument rated 30 millivolts full scale, is capable of measuring ion currents of the order of 10^{-14} ampere.

The amplifier usually consists of an electrometer tube connected as a space-charge tetrode, followed by two stages of voltage gain and a cathode follower output tube. The output appears on an ordinary panel-type milliammeter rated one milliampere full scale.

2.1.3.1.9. Leak Detector Pumping System

The leak detector pumping system could be used to evacuate the test object. However, even with adequate valving, this method is not satisfactory. Without the addition of more valves and lines, the pumping has to be done slowly through the throttle valve to avoid damage to parts such as gages and pumps. The usual procedure is to use an auxiliary mechanical pump. A capacity of five cubic feet per minute is adequate for most purposes.

Some mass spectrometer leak detectors incorporate an automatic test station in the detector cabinet. A mechanical pump or some other facility is provided for roughing the test piece to low enough pressures to transfer it to the leak detector system.

In the automatic mode, the operator need only connect to the test piece and push the test switch. The test piece is then evacuated to a pre-set vacuum level. When this level is reached, the test valve will open automatically and the roughing valve will close. The test piece is then ready to be leak tested. Upon completion of the leak test procedure, the test piece is automatically vented to atmosphere. The piece can then be extracted and a new piece inserted.

2.1.3.2. ALTERNATIVE TYPES OF ANALYZERS

A leak detector having a higher sensitivity is always of interest. This is mainly a matter of reducing the background fluctuation, since it is easy to use greater amplification for the output signal. Limitation of sensitivity in mass spectrometer leak detectors is due to background caused by gas scattering of the ion beam, nonhomogeneity of the beam, and space charge defocussing. Greater amplification can be utilized in the same proportion as background is reduced, and still maintain the same signal-to-noise ratio in the output.

Peters (Ref. 85) designed a mass spectrometer using two magnetic analyzers in series. The ions are accelerated from the first analyzer into the second through an interstage slit. Those ions which are gas-scattered in the first analyzer, and which would have produced a broadened peak in a single-stage instrument, are resolved into separate peaks by the second analyzer because interstage acceleration adds different amounts of momentum to the different masses. With operating pressure in the spectrometer tube in the 10^{-5} to 10^{-6} torr region, the background was three orders of magnitude less than in a commercial single-stage mass spectrometer. It was therefore possible to use an electron multiplier as an ion detector. A leak detector employing this principle has recently been manufactured by Veeco Instruments Inc. The smallest detectable leak in this detector is in the range of 5 x 10^{-14} atm cc/sec.

With higher mass spectrometer sensitivity, however, unusual problems are encountered. Residual background from atmospheric helium, diffusion of helium through glass, helium holdup from previous testing, and helium accumulation in the associated vacuum measuring gages can present serious limitations. One of the most significant problems is handling the item to be tested. Leaks which the new machine would be capable of detecting can be easily - though temporarily - sealed by the grease and moisture of a fingerprint (Section 1.4.5).

One approach to the problem of reduction of background noise is embodied in an ion detector described by Daly (Ref. 86). It can distinguish between helium ions and unwanted "air" ions. This detector is used with a conventional single-stage mass spectrometer. Its operation depends on the fact that the penetration of low energy particles in aluminum is proportional to the energy and inversely proportional to the square root of the atomic number of the ion. The foil thickness can be chosen so that the helium ion just penetrates to the back and releases there a secondary electron, which may be used to record the arrival of the helium ion at the foil. No signal will be obtained from the N^+ and N_2^+ ions because of their failure to penetrate a foil of this thickness. A great reduction of background can be achieved by this technique. This detector is not commercially available at present.

A number of mass-spectrometer types other than magnetic sector may be used for leak detection. For example, Varadi (Ref. 87) describes a radio-frequency mass spectrometer leak detector. In this type of analyzer tube ions are accelerated through a voltage, and then sent through a series of grids which have a radio-frequency (RF) voltage applied to them. If the transit time of the ions in going through the grid structure is in proper phase with the frequency of the applied RF voltage, they will gain maximum energy. A repelling potential placed on a grid between the RF structure and the collector prevent all but those ions which have gained maximum energy from getting through to the collector.

This instrument does not require a magnetic field and is fairly simple in construction. One disadvantage of this type of instrument is that it requires a large amount of electronic circuitry.

Barrington (Ref. 88) developed an inverted magnetron leak detector. In this analyzer tube, the anode is mounted centrally inside a grounded cylinder which acts as the cathode. A magnetic field of about 1000 gauss is applied axially. A discharge is initiated on application of a potential difference of about 1500 volts between the electrodes. The electrons are trapped by the magnetic field and circulate around the anode; positive ions, however, are accelerated radially towards the cathode and, because of their much larger mass-to-charge ratio, are deflected only slightly by the magnetic field. A small exit hole is provided in the cathode; some ions emerge and are deflected further by the fringing magnetic field. This comparatively crude arrangement functions as a mass spectrometer because the energy spread of the emerging ions is small. The Geophysical Company of America (GCA) is manufacturing a leak detector employing this principle.

An omegatron leak detector is described by Nicolliam (Ref. 89). The omegatron is basically a small compact cyclotron. The electron beam produces ions of the residual gases in the volume, and the magnetic field causes these ions to move in circular paths at their cyclotron frequency. When the applied radiofrequency voltage is in resonance with the cyclotron frequency of a particular ion, the ion will gain energy and move out in a spiral path, eventually striking the collector.

Theoretically, any mass spectrometer may be used as a leak detector. Mass spectrometers designed for leak detection are portable, rugged, and have the ability to remain tuned to the tracer gas signal. There are times, however, when a mass spectrometer not of the leak detection design is used for leak detection (Ref. 90).

2.1.3.3. EQUIPMENT AVAILABLE

Table 13 shows the manufacturers of mass spectrometers commerically available specifically for leak detection. Also shown on Table 13 are the general characteristics of the mass spectrometer leak detector. These general characteristics are typical and are not intended to represent any one company's products.

2.1.4. READING INTERPRETATION

Leak detector sensitivity is specified in two ways:

- By the smallest detectable tracer gas concentration in air at a specified source pressure (this is usually expressed in parts per million). For the helium mass spectrometer leak detector, this value is approximately 0.1 part per million.

- By the minimum partial pressure of air which, if changed to the same pressure as the search gas, would produce the minimum detectable indication, e.g., in indication of three times random noise.

The second definition is commonly applied to helium leak detectors. Manufacturers often use another form of this definition: the smallest pure helium leak which can be detected at a specified source pressure (usually atmospheric), and under specified test conditions. This is often called the smallest leak detectable and is given in units of leakage, such as std cc/sec, atm cc/sec or torr liters/sec. For the helium mass sprectrometer

Table 13

COMMERCIAL MASS SPECTROMETER LEAK DETECTORS*

General Characteristics	Average Values
Sensitivity	1 x 10^{-11} atm cc/sec: 0.1 parts per million helium in air
Tracer Gas	Primarily helium; also, hydrogen, argon, neon, butane
Output Signal	Millivolt meter or micro-amp meter
Power Requirement	1000 watts, 105/125 60 cycle AC; 220/250 60 cycle AC
Size	Width= 25 inches, Height= 40 inches, Depth= 20 inches
Weight	500 pounds
Price	$5000
Necessary Accessory	Liquid Nitrogen

Manufacturers

Atlas Mess and Analysen Technik GMBH (Haraeus-Engelhard Vacuum, Incorporated, distributor)
Consolidated Electrodynamics Corporation (subsidiary of Bell and Howell Company)
Denton Vacuum, Incorporated
General Electric Company (Vacuum Products Operation)
National Research Corporation (Equipment Division) subsidiary of Norton Company
Vacuum Industries, Incorporated (subsidiary of GCA Corporation)
Vacuum Instrument Corporation
Varian Associates (Mikros Division)
Vecco Instruments, Incorporated (formerly Vacuum Electronics Corporation)

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* More specific information of this equipment may be obtained from General Electric Company Report S-67-1013 (Ref.[186])

leak detector, this value is approximately 5 x 10^{-11} atm cc/sec.

Another term that is often used is minimum detectable leak. This is the smallest leak that can be clearly detected by a given leak detector in the presence of noise or background. Alternatively, it is the product of the minimum detectable pressure change and the pumping speed at the detector. These definitions are used interchangeably here, with the same types of units.

It must be kept in mind that helium flows more readily through a leak than air. To convert the value of helium leakage to the corresponding value for air, simply divide by 2.7. The sensitivities of most commercial helium leak detectors are expressed in terms of helium. Some advertising brochures state the minimum detectable leakage as the air leakage, even though the measurements were performed with helium gas. The advantage of stating leakage in terms of air units is that the value of the minimum detectable leak will appear as a number smaller by a factor of 2.7 than the equivalent helium leak. This difference in specifications should be carefully noted when the advertised sensitivity of various detectors is being compared.

The maximum sensitivity is limited by the characteristics of the electronic circuitry (usually the amplifiers) used to measure the helium ion current. This limitation is caused by noise and drift. Noise shows up as a random fluctuation in the electronic output, and will be indicated by an erratic motion of the needle of the detector or output meter. Drift shows up as a gradual wandering of the output meter needle. For the purpose of discounting any insignificant fluctuations in the reading, the measurement is usually made over a period of an hour or so under steady conditions, an electrical recorder often being employed. The drift for most commercial leak detectors is usually less than 1/2 of 1 percent per minute of full scale on the output meter. This type of drift ordinarily introduces no particular problem, although it may be necessary occasionally to "zero" the output meter while leak locating. The combination of noise and drift gives a smallest readable deflection (in terms of scale divisions), which is also called the minimum detectable signal. The smallest readable deflection is specified for the most sensitive setting of the leak detector output meter. The minimum detectable signal (or smallest detectable signal) is often taken as three times the mean peak-to-peak fluctuations of the output meter needle, averaged over ten fluctuations. The signal-to-noise ratio is then 3. Appendix III lists two tentative specifications for measuring the above terms on a mass spectrometer leak detector.

The term background is also used in connection with leak detectors. This refers to the spurious output from the response of the detector to other gases than the probe gas. The background may be inherent in the detector or accidental. It must be subtracted from the reading indicated on the output meter in order to get the true reading of the leak. The following is a useful rule of thumb: as the magnitude of the leak being sought becomes smaller and smaller, the tracer gas background level becomes more and more important. Hence, background levels should always be checked before and after a test, and calculations adjusted accordingly.

The sensitivity of any particular mass spectrometer leak detector is a function of both total pressure and helium concentration in the detector. The most thorough study of these two variables was made by Choumoff and Laplume (Ref. 91). The sensitivity of the mass spectrometer leak detector decreases with an increase of system pressure. An example of this variation of sensitivity with pressure is shown in Figure 49. Depending on the individual instrument, sensitivity may be regarded as essentially constant below a particular pressure which is in the region of 10^{-2} torr.

Pressure affects sensitivity in two important cases. One, if excessive outgasing or leaks in the system under test raise the system pressure above the critical value in the region of 10^{-2} torr, sensitivity decreases rapidly. In this case, auxiliary pumping may be used to reduce system pressure. However, such pumping will also reduce sensitivity. Two, if too large a detector probe is used, sensitivity decreases rapidly because pressure within the mass spectrometer detector will rise to the 10^{-2} torr region. A compromise must be sought between too large a detector opening, which reduces sensitivity, and too small an opening which does not allow entry of enough helium for detection.

Figure 50 shows a typical mass spectrum. If quantitative leakage measurement is required, the helium peak must be measured accurately and the background (dotted line) subtracted. The background is a tailing from other gases present in the system, and is particularly large at high pressures. Background is reduced somewhat by the suppressor plate, but can rarely be eliminated entirely. For accurate peak height measurement, the amplifier current should be measured at the maximum peak height. The accelerating voltage is then varied from the maximum peak value by an equal amount in both directions. The average of these two readings may be used as a representative background to be subtracted from the maximum peak value.

On Figure 50, for example, if a peak is found at 190 volts, the average of readings at 180 and 200 volts will give a background value.

Three types of false signals may be encountered in testing: helium coming from inert gas welds, permeation without actual leakage, and signals from deuterium gas. The inert gas welding signal will be observed prior to tracer gas being put into the system. Unless the system contains many porous parts, it will usually be a rapidly decaying signal. Permeation in quantities large enough to be detected will only appear in the presence of large plastic or rubber gasket fittings and diaphragms. This

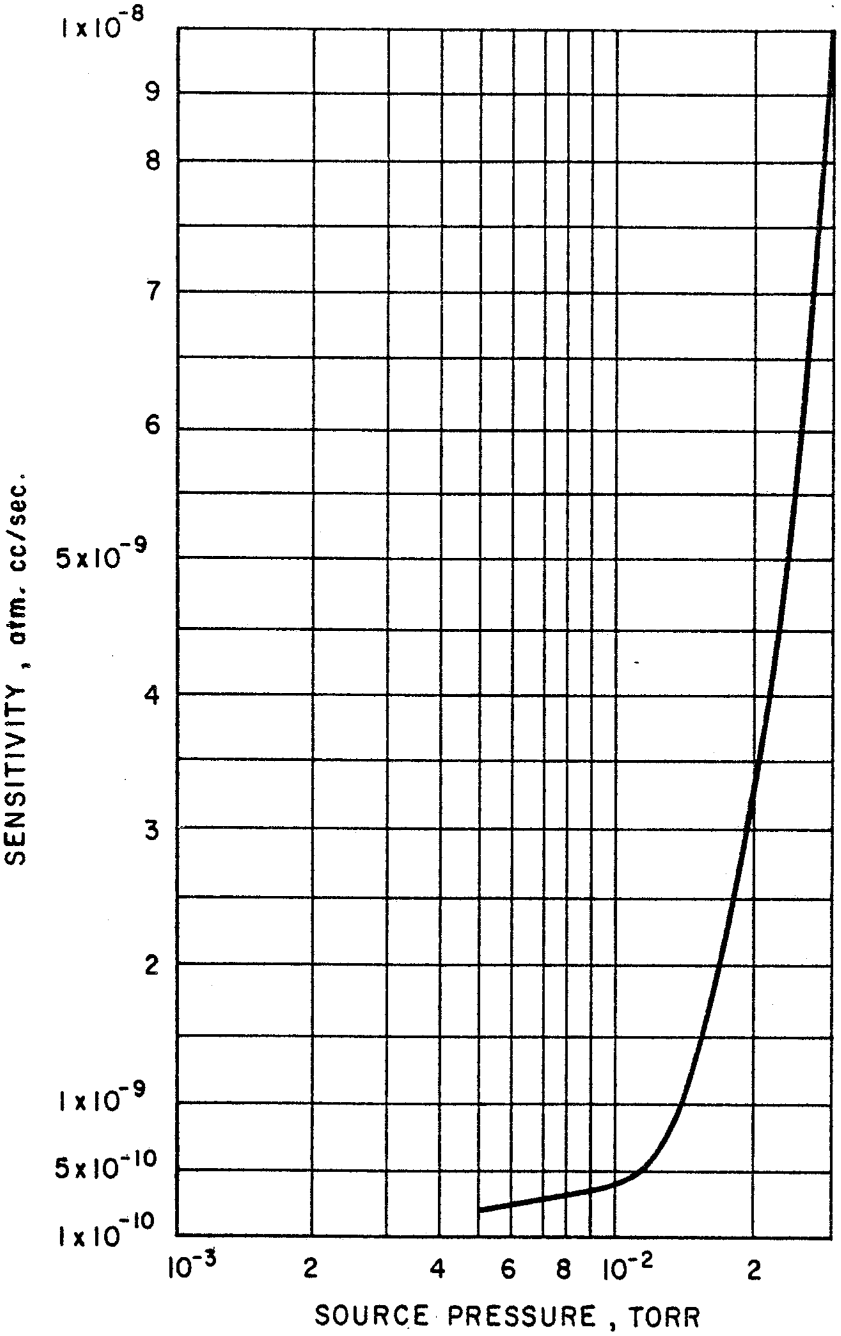

Figure 49. Sensitivity Versus Pressure for a Mass Spectrometer Leak Detector. (Reprinted, with permission, from Advances in Vacuum Science and Technology, Proceedings of the First International Congress on Vacuum Techniques (10-13 June, 1958, Nemur, Belgium), Vol I, "Fundamental Problems in Vacuum Techniques Ultra-high Vacuum", by S. Schoumoff and J. Laplume; Copyright 1960, Pergamon Press, New York)

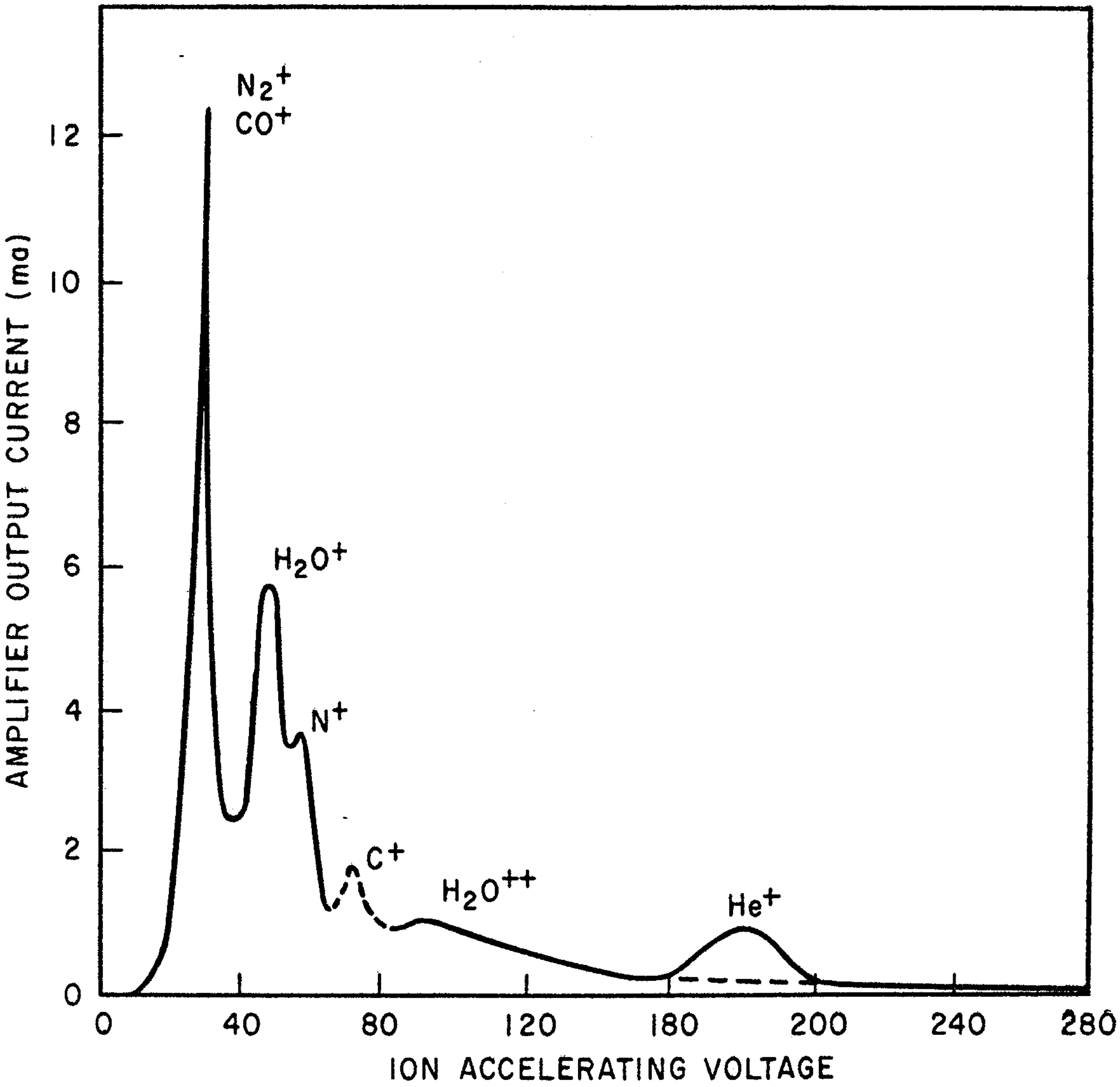

Figure 50. Typical Mass Spectrum Taken with a Mass Spectrometer Leak Detector. (Reprinted, with permission, from A.O. Nier and others, "Mass Spectrometer for Leak Detection", Journal of Applied Physics, Vol. 18, 1947, pp. 30-33.)

signal will usually take some considerable time to build up. See Section 1.4.1.2. for permeation references and calculations. Deuterium gas will be encountered in installations using it or heavy water. This is a rare occurrence.

2.1.5. METHODS OF TESTING

The mass spectrometer leak detector can be used in the following techniques:

1. Dynamic leakage measurement

2. Leak location
   Tracer probe Technique
   Detector probe Technique

3. Accumulation leakage measurement (static testing)

   Detector probe (system at atmospheric pressure)
   Evacuated analyzer (system evacuated)

If it is desired to utilize the high sensitivity of the equipment, care must be taken that the small leaks are not clogged prior to detection. This clogging is discussed in Section 1.4.5.

2.1.5.1. DYNAMIC LEAKAGE MEASUREMENT TECHNIQUE

This is the typical technique for which the mass spectrometer leak detector is designed. Under ideal conditions, the full sensitivity (approximately 5 x 10^{-11} atm cc/sec) of the leak detector is utilized. In this technique the test object is connected through appropriate valving to the leak detector. The magnitude of the response and the time of response follow the equations given in Section 1.3.4.1.2. This is particularly true, since these equations were first developed for use with the mass spectrometer. The Procedural Techniques described in Section 1.5.2. also apply.

The evacuated side cannot contain volatile materials or materials sensitive to vacuum because it has to be at a relatively low pressure (10^{-2} torr). The evacuated side has to be constructed in such a way as to be capable of being maintained at low pressure.

The procedure consists of evacuating one side of the tested component to a pressure of at least 10^{-2} torr, attaching the mass spectrometer to the pumping system, and enveloping the other side of the component with helium gas. The gas may be at any predetermined pressure; although an increase in pressure differential will increase the leakage. It is however, important that during every analysis, the pressure differential be kept at the same level. See Section 1.4.3. on predicting leakage change with change in pressure differential.

The major portion of time required for detection using this procedure is the pumpdown time to evacuate the component. The equipment usually contains a mechanical pump with approximately 20 liters/min pumping speed. A pressure of 10^{-2} torr is needed for testing. Equations given in References 53 and 54 may be used to calculate the pumpdown time. Additional pumps can provide more rapid pumpdown. This procedure can be highly automated. Therefore, an operator can be trained to use the instrument in less than one hour.

2.1.5.2. LEAK LOCATION TECHNIQUES

2.1.5.2.1. Tracer Probe Technique

Leaks may be located using the tracer probe technique by evacuating the system and probing with tracer gas. (Section 1.3.4. and Section 1.5.2.).

Often the tracer probe technique is used after dynamic leakage measurement. The tracer gas envelope is removed, tracer gas is pumped out of the system, and searching with the probe is begun. One disadvantage is that the leakage can only be determined at one atmosphere pressure. Increased sensitivity cannot be gained by use of a pressure differential greater than one atmosphere.

Detection time with this technique is the pumpdown time to evacuate the component and to spray all suspected leak areas. The equipment usually contains a mechanical pump with approximately 20-liter/min pumping speed. A pressure of 10^{-2} torr is needed for testing. References 53 and 54 may be used to calculate the pumpdown time. Spraying can be done with a large tracer gas jet at a rate of several yards per minute. Location of individual leaks using a fine tracer gas stream can be an extensive effort.

Testing of a large object may be initiated by loosely fitting a film of vinyl plastic around it to retain the helium. In this way it is possible to determine if any leaks exist. Helium gas is lighter than air. Consequently, in systematic leak checking, gas is sprayed on the top of the component first and then gradually down all other suspected areas. This is done so that helium gas will not drift up to an undetected leak and cause an erroneous signal. The detection procedure should be carried out in an area free from drafts and breezes.

2.1.5.2.2. Detector Probe Technique

The detector probe technique, (also called "sniffer" probe, pressure probe, sampling probe, and reverse probe) consists of pressurizing the test system, then locating leaks by sucking tracer gas through the probe into the detector. This technique should be used only for leak location, and not for leakage measurement.

The detector probe is a probe tube at the end of several feet of flexible tubing with an orifice of approximately 10^{-4} atm cc/sec at the tip. The method is useful for systems that will not stand internal vacuum. For maximum sensitivity, the orifice should be large enough to admit a large sample of gas from the vicinity of the probe. On the other hand, the orifice conductance must be rather low to maintain a low enough pressure in the detector. Small samples of the atmosphere at the probe tip are admitted to the detector. This results in a sensitivity of around a thousand times less than in tracer probe testing. With faulty techniques, additional sensitivity loss of 10:1 or even 100:1 will be realized.

In the vicinity of a small leak, helium coming from the leak commences to build up a high helium concentration in the air. However, since helium diffuses rapidly in air, a steep helium concentration gradient is established in the vicinity of the leak. When the probe enters the leak area, it samples the mixture which exists at its orifice. Therefore, it is absolutely necessary that this orifice be at the extreme end of the probe. If the flow rate through the probe were high, it would sweep in the helium. However, it is only 10^{-4} atm cc/sec and hence the flow has no effect on distrubing the gradient. If the probe misses the leak by as small a distance as 1/4 inch, the sensitivity is reduced by approximately 10 to 1 because of the steep concentration gradient.

To overcome the effect of the gradient, a small rubber suction cup can be placed over the end of the probe. While this reduces the problem of close approach, it creates one of time constant. As the defining volume covers the leak during probing, the helium concentration begins to increase in the volume. The sampling probe is constantly monitoring this concentration. After a while, the helium concentration will build up to an equilibrium and full mass spectrometer sensitivity will be attained. However, the time constant is in the order of half an hour, but the probe is over the leak for only a few seconds. Hence this ratio represents a lost sensitivity factor. In addition, helium diffuses from the small volume to the atmosphere. This causes additional sensitivity loss.

Both the probing speed and the distance between the system and the detector probe are critical. Figure 51 (Ref. 92) is an example of the effect of probing speed and distance on the sensitivity of the technique. This data represents one manufacturer's results with the probe obtained under ideal laboratory conditions. Such high sensitivity cannot be obtained under field conditions. At a probing speed of three feet per minute, even under ideal laboratory conditions, the probe must be within 1/4 inch of the leak to be able to detect and locate the leak. If a weld which is wider than 1/4 inch is being checked for leaks, at least two parallel passes must be made to check the weld completely.

Mass spectrometers will respond to the five parts per million of helium already in the atmosphere when operated at the

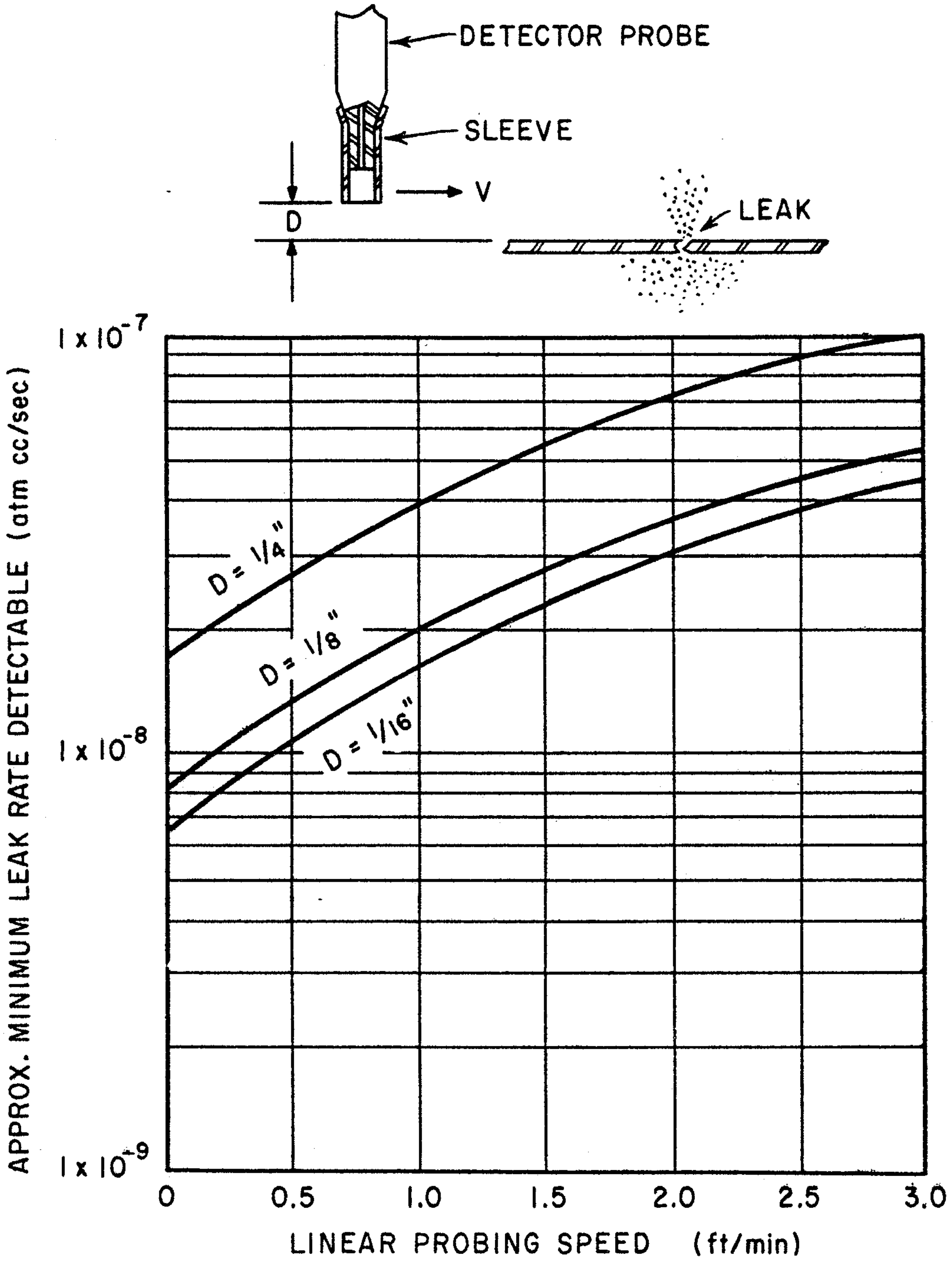

Figure 51. Effect of Probing Speed and Probe Distance on Sensitivity. (Reprinted with permission from Consolidated Electrodynamics Corporation, Pasadena, Calif.)

high pressures required for reverse probe testing. The signal observed is a function of the flow rate through the probe. Hence, the problem of working over a background helium signal exists.

On the other hand, this air signal may be used to tune and calibrate the leak detector and to optimize the probe orifice size. For best sensitivities the leak detector should be operated at about 1 x 10^{-4} torr detector pressure. Lower pressures result in low speed and longer time constants, while at higher pressures the inherent sensitivity of the leak detector begins to decrease. The use of long vacuum leads between the machine and probe should be avoided if possible. The additional volume created by these leads adds to the time constant and hence reduces the apparent sensitivity. The true sensitivity is not reduced, but slow response interferes with prompt detection of leaks as the probe is moved over the suspected area.

When pressure testing large objects, it is preferable to use a short hose and to move the leak detector rather than to use a long hose and a stationary leak detector. Response is approximately linear with probe length in the absence of adsorption and outgassing. That is, doubling the length should double the response time. Actually, doubling the length will probably double the outgassing rate, which further acts to reduce the apparent sensitivity.

Figure 52 (Ref. 92) is an example of the effect of probe length on the cleanup and response time of the leak.

The flexible hose to the probe preferably should be of the metal bellows type. However, rubber tubing (preferably neoprene) is often used. The problem of sorption of helium can be ignored provided metal lines are used. Rubber vacuum hose acts as a good sponge for helium, removing it from the stream and reducing the sensitivity. This helium is later given off and adds to the helium background. The complete removal of helium from a vacuum hose is a tedious process and frequently requires several hours. Cleanup rate can be improved by passing a dry gas through the line at a fraction of a torr of pressure.

Certain points regarding detector probe testing can be summarized as follows:

1. Sometimes a specially shaped sniffer probe is required. This can be made by flattening the end of a piece of copper tubing. Needle valves are also used to vary the suction through the probe. A long needle with slight taper is desirable. The valve must be at the end of the orifice, so there is no volume between the valve and the point where the sample enters the probe.

2. Sometimes the flow through the probe is too large to be handled by the pumping system of the leak detector. An auxiliary pump then is used. This will reduce the

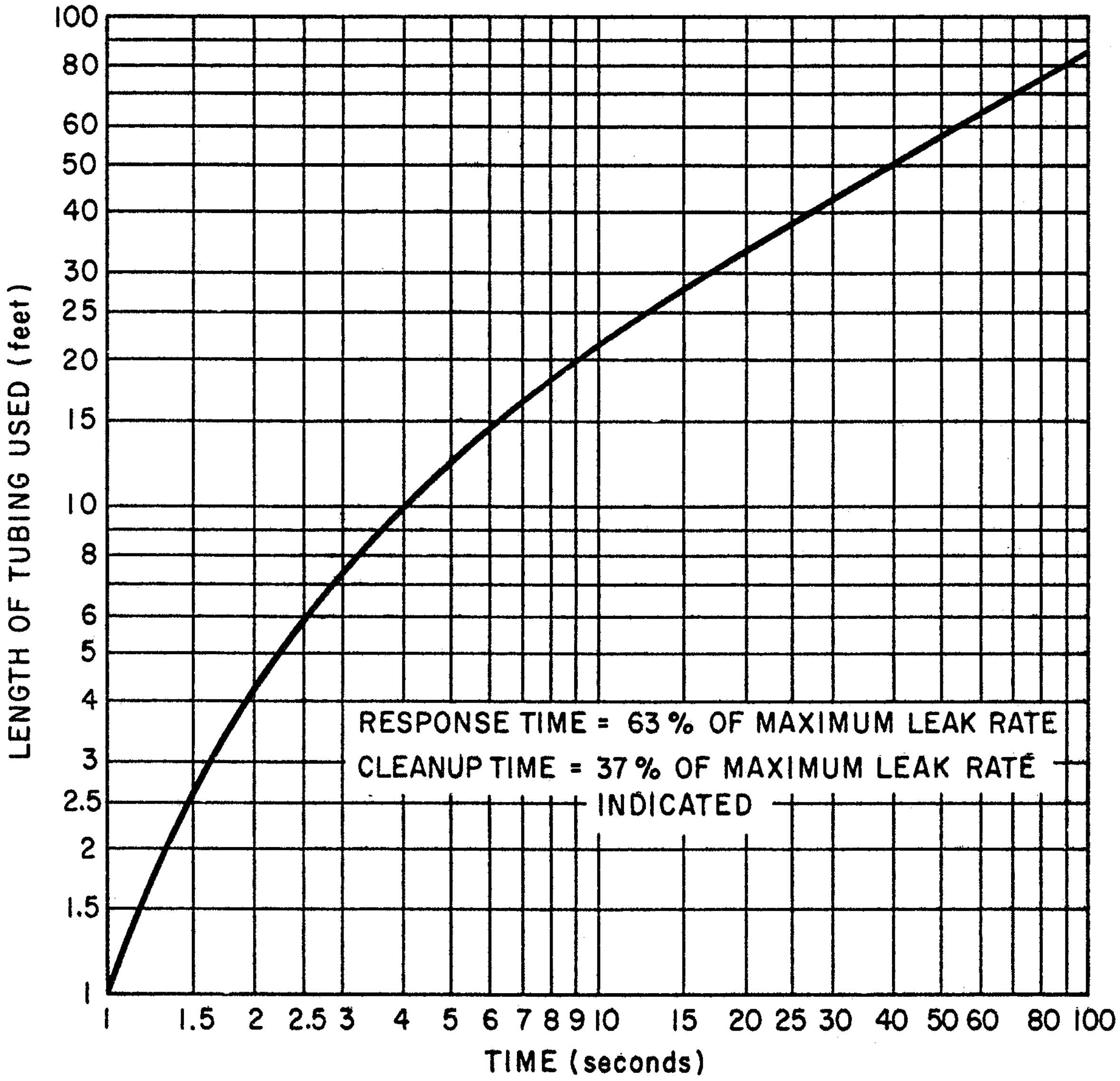

Figure 52. Response and Cleanup Time Versus Probe Length for a Mass Spectrometer Leak Detector. (Reprinted with permission from Consolidated Electrodynamics Corporation, Pasadena, Calif.)

detector sensitivity.

3. The sensitivity can be increased by restricting the slight suction of the probe to the neighborhood of the area under test. A rubber fitting (even a small piece of rubber tubing) over the end of the probe will act as a suction cup.

4. After probing in one location, the probe may use up all the helium issuing from a leak there. The probe should be removed until a detectable concentration of helium has built up again around the leak.

5. The test object should be kept out of drafts so helium can build up in a concentration around a leak.

6. The minimum detectable conductance of the leak can be changed by pressurizing the vessel to be tested. If such an increase in pressure implies too great an expense in tracer gas, the gas may be diluted with some loss of sensitivity. For example, if a system pressure is increased by a factor of 10, providing the flow through the leak is laminar, the flow through the leak will increase by a factor of 100 (Section 1.4.1.1.1.). Decreasing the tracer gas by a factor of 10 will decrease the sensitivity by 10. (Section 1.5.1.5.1.).

The best method of establishing the over-all sensitivity is to probe past a known calibrated leak which has been pressurized with helium. The reading can be extrapolated down to the minimum detectable leak by the ratio of the signal observed to the minimum signal required for detection under given operating conditions. Unfortunately, this type of leak is not commercially available. The design of a variable leak for qualification of this technique is discussed in Section 1.6.2.3.2.

Two types of sensitivity can be measured when using the probe technique. The fundamental sensitivity of the detector may be measured by placing a calibrated leak in the manifold of the detector and measuring the sensitivity. This is done to tune the analyzer tube. The over-all sensitivity of the technique is determined by placing the probe near a calibrated leak. This is the only reliable method of qualifying the detector probe technique.

2.1.5.3. ACCUMULATION TESTING (STATIC LEAKAGE MEASUREMENT)

Accumulation testing for leakage measurement is done with a detector probe, or by a vacuum accumulation technique. In the vacuum accumulation technique, the system is evacuated. In the detector probe technique, the system is pressurized with helium gas.

2.1.5.3.1. Accumulation Technique (Detector Probe)

Leakage may be measured on large or complex systems by pressurizing the system and placing it in a sealed volume or room. A schematic of this technique is shown on Figure 53. The detector probe is placed in the room and the tracer gas concentration monitored. This technique has been described by Summers and Koch (Ref. 93).

Any leakage of helium from the pressurized system will be picked up by the leak detector. Since the concentration of helium in the room will be increasing with time due to the leakage of helium from the system, the output reading will also increase with time. This can be converted to a leak rate in atmosphere-ppm/unit-time using the calculated instrument sensitivity, or it can be converted to leak rate units, i.e., atm cc/sec. To make this conversion, a knowledge of the room or chamber volume, less system volume, is necessary. The leak rate of the system under test can be found by multiplying the helium percent increase per test time by the free volume of the room.

The percentage of helium in air is 0.000524 percent or one part helium in 200,000 parts air (Ref. 77). The leak detector will indicate a constant, readable output reflecting this helium concentration, and the sensitivity of the instrument can be determined in ppm/div. To avoid confusion it may be desirable to "zero" this reading out of the system, after calibration, and before leak testing the system. However, such a calibration will ignore leakage out of the room. For accurate measurement, a calibration should be performed using a calibrated leak discharging into the room.

2.1.5.3.2. Accumulation Technique (Vacuum)

This may be considered a static leakage measurement technique. The test object is evacuated, isolated by a valve from the auxiliary pump, and covered with helium in the hood. The higher the helium concentration and the longer the test object remains in the hood, the more helium will pass through a given leak and accumulate in the test system. Suppose a vacuum gage is included in the system between the leak detector and the test system. If there is a considerable rise in pressure during the accumulation period, a gross leak exists. If this is not the case, the leak detector valve can be opened rapidly and at a uniform rate. A detectable leak will show up as a rise in the output signal to a peak, followed by a decline as the accumulated helium is pumped out by the leak detector.

The difficulty with this procedure is that it is not quantitative. The accumulation-time is limited by the outgassing of the test object. If the testing is conducted for a long period of time, the pressure rise due to outgassing will be so high that the spectrometer will give false readings. Lineweaver (Ref. 94) describes this technique in testing envelopes of TV

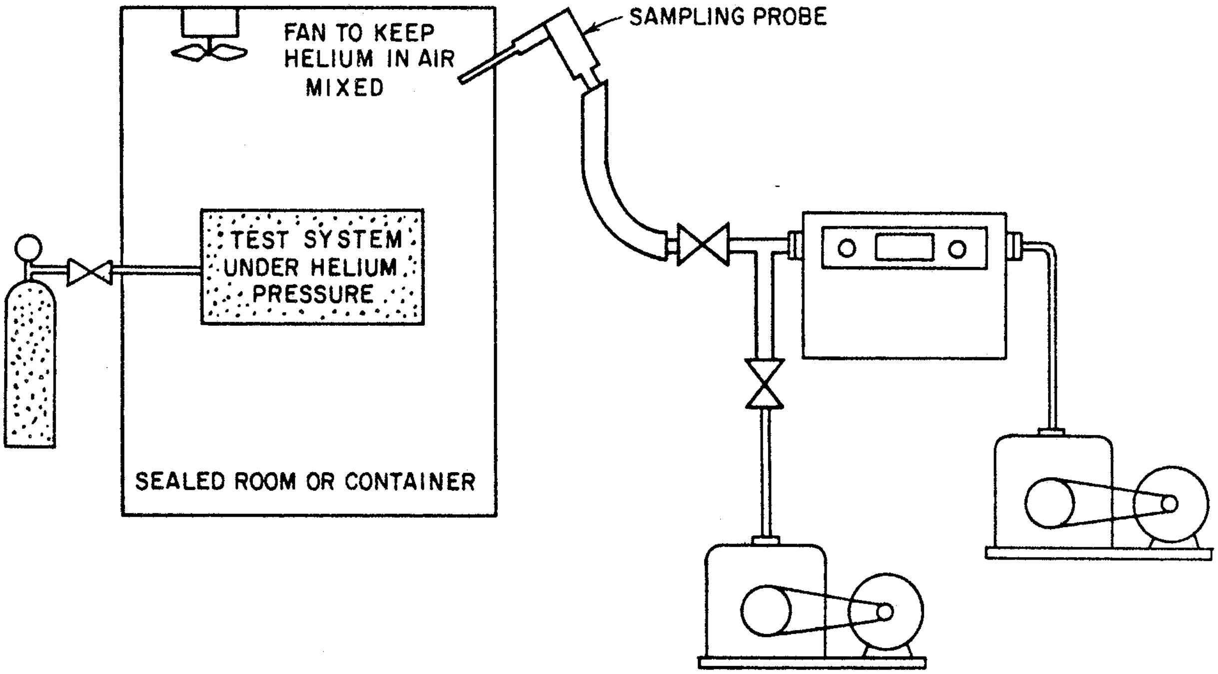

Figure 53. Detector Probe Accumulation Testing.

picture tubes. Accumulation times can be raised from minutes to days by previously outgassing the system.

A more quantitative measurement may be made by constructing the vacuum system and the analyzer tube using ultrahigh-vacuum techniques (Ref. 90). The accumulation may then take days, with the analyzer tube and test system isolated from the pumping system.

2.1.6. OPERATION AND MAINTENANCE

The specific steps involved in using a commercial leak detector will differ from model to model and from manufacturer to manufacturer. However, the details of operation for each model are usually spelled out carefully by the manufacturer. Maintenance problems are generally minimal. The openings for air circulation through the leak detector should not be blocked and vibrations should be avoided. A maintenance schedule should be set up for changing mechanical and diffusion pump oils. Gasketed joints that are disassembled periodically, such as the flanges for connecting test objects, the liquid nitrogen trap, and the standard leak, should be inspected on disassembly. Spare parts, such as filaments, gaskets, and certain electronic parts, should be kept on hand. The manufacturer can provide a list of parts that are most likely to be needed.

A great number of machine adjustments may be used to vary the sensitivity of the leak detector. Zero adjust will change the sensitivity to some extent. Internal pressure variation will also change sensitivity. Perhaps the greatest effect on sensitivity is the cleanliness level of the detector and the system being tested.

A philosophy needs to be developed for both personnel and equipment. Leak detection is no better than the operator. The following illustrate some of the established operator rules that have been known to minimize maintenance and increase efficiency. An operator should:

1. Refuse to test systems or parts which appear unclean or which visually appear to have contaminants on their surfaces, i.e., oil, chips, lint, threads, water stains, etc.

2. Use only jigs and fixtures developed for the specific parts and standard setups.

3. Acquaint the supervisor or associate process control engineer with any parts which have been damaged or appear abnormal in any way prior to its test.

4. Determine that the apparatus has been checked out by the maintenance man before operating the detector at the start of each shift. The maintenance check is recorded in a log book attached to each station.

5. Report all variations in machine performance to a maintenance man.

6. Maintain all cold traps diligently.

The cleanliness of the detector can be greatly enhanced by use of the nitrogen bleed technique described in Section 1.5.3. This technique should be used whenever the system is left overnight and use is anticipated the next day. Under no circumstances should the pumps of the leak detector be used for drying wet systems or evacuating dirty systems. Overnight, use of the nitrogen bleed technique keeps the leak detector pumping system unavailable to night-shift personnel who might not be knowledgeable in its proper use.

No matter what the circumstances of vacuum testing, a reliable, informed operator is needed. Although most commercial leak detectors are now almost completely automatic, some individual, responsible for the equipment operation and use, should be familiar with the principles of vacuum technology. Information that is helpful will be found in References 53 and 54. It is desirable to check sensitivity at the start and at the end of a work shift. It is also advisable to rapidly check if the detector is operational after each testing task by admitting tracer gas into the detector through a calibrated leak.

The leak detector should be leak-tight when being used. It is possible to locate leaks in the detector system by a tracer probe technique, using the mass spectrometer itself to locate its own leaks. When checking the mass spectrometer leak detector, it might be found that a leak is indicated when helium is brought close to the exhaust of the mechanical pump. This is because the diffusion rate of helium is high enough so the gas will flow against the general gas flow. This counter diffusion is a normal occurrence. The operator of the equipment should be aware of it. False signals due to this counter diffusion may be avoided by preventing large amounts of helium gas from haphazardly reaching the area near the mechanical pump.

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Section 2.2.

HEATED ANODE HALOGEN DETECTOR

2.2.1. PRINCIPLES OF PROCEDURE

This detector makes use of a red-hot platinum or ceramic filament which emits positive ions. The presence of small traces of halogen vapors increases the emission of positive ions markedly. It is this increase in emission that is measured to indicate the presence of a leak.

The halogens are chlorine, iodine, bromine, and fluorine. Materials containing these elements are usually called halides. The most common halide materials used in leak detection are those containing chlorine.

2.2.1.1. CHARACTERISTICS

In general, emission of ions means loss of material from the cathode emitting them. One unique feature of ion emission, however, is that it can be made to occur readily in the air.

Owing to the fact that platinum and some ceramic materials can be operated at a red heat with little oxidation and loss from evaporation, such material is very useful as an ion-emitting source. The volume of ion emission from such materials varies greatly, depending upon temperature, area and nature of the surface, and purity. In emission, current drops slowly with time, eventually reaching a small but finite equilibrium value.

It has been found by C. W. Rice (Ref. 95) that the steady emission of ions in air is increased to a marked extent when certain vapors strike the electrode surfaces. In the presence of even a small amount of a halogen compound vapor there is a marked increase in ion current.

The essential elements of a heated anode halogen leak detector are shown in Figure 54.

1. A two-element sensing structure is shown in Figure 55 (Ref. 96). One suitable arrangement is in the form of concentric cylinders. The vapor to be detected is passed between these two closely spaced cylinders. The inner cylinder or cathode is kept red hot by an internal wire heater and the outer cylinder or anode is operated at a negative potential.

2. The detector includes means of forcing air containing the tracer gas between the cylinders at a constant low velocity.

3. The circuit (Figure 56) contains low-voltage for the heater and another supply delivering a few hundred

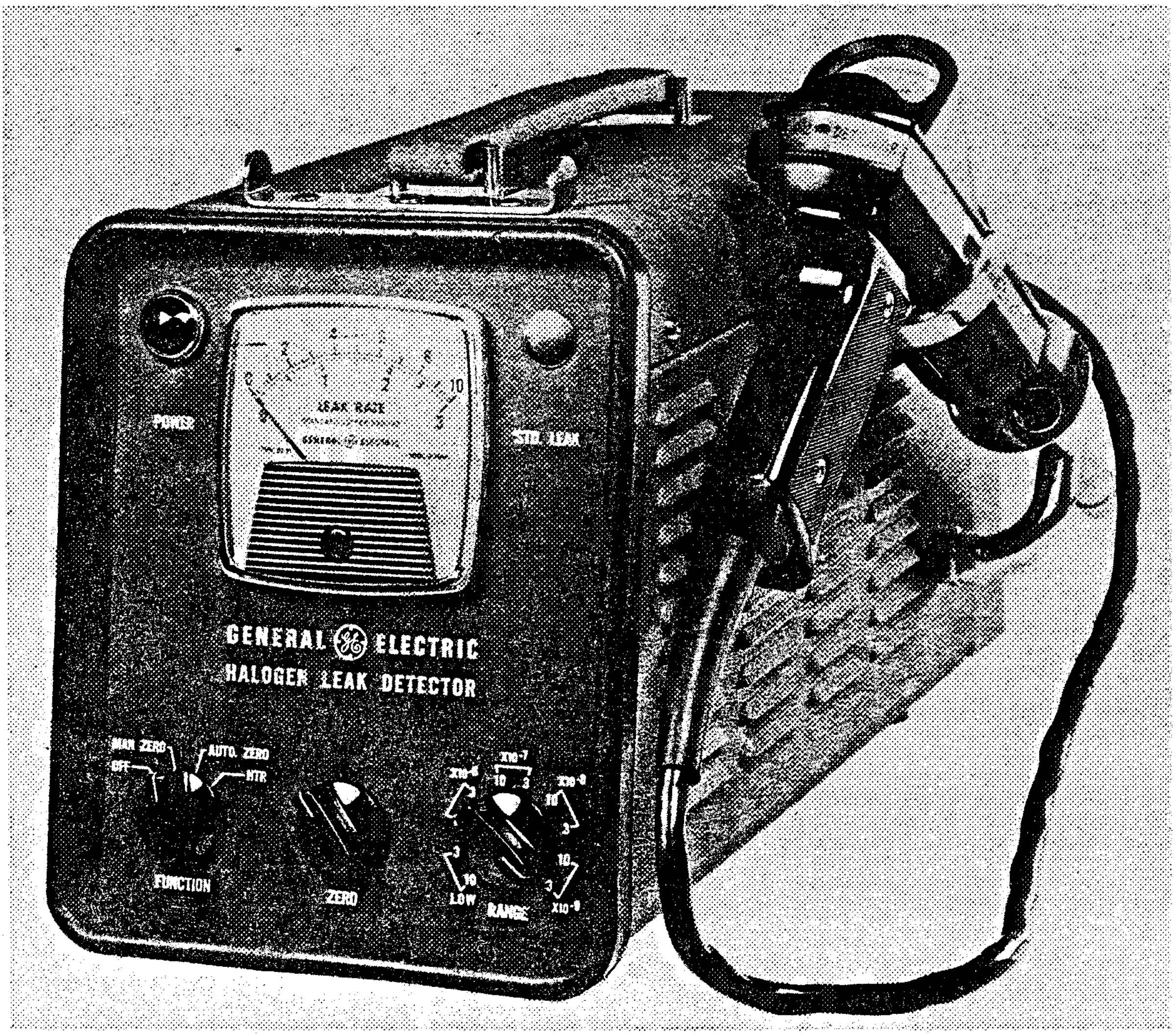

Figure 54. Heated Anode Halogen Leak Detector. (Reprinted with permission of the Instrument Department General Electric Company, West Lynn, Massachusetts.)

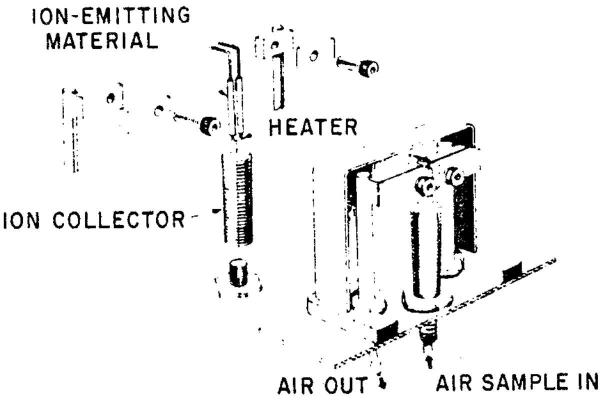

Figure 55. Detector Element of Heated Anode Halogen Leak Detector. (Reprinted, with permission, from W.E. Briggs, A.C. Jones, and J.A. Roberts, "Leak Detection Techniques", 1958 Fifth National Symposium on Vacuum Technology Transactions, Symposium Publications Division, Pergamon Press, New York)

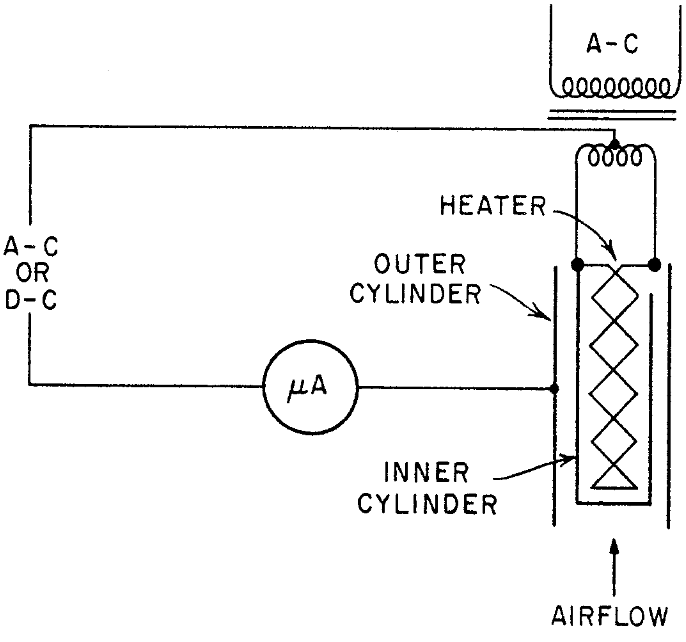

Figure 56. Basic Circuit of Heated Anode Halogen Leak Detector. (Reprinted, with permission, from W.C. White and J.S. Hickey, Electronics, Vol. 21, March 1948, McGraw-Hill Publications, New York)

microamperes at somewhere between 50 and 500 volts a-c or d-c for use as the interelectrode potential.

4. Sufficient current amplification is contained in the detector circuit to make a small increase in d-c current due to ion emission variation readily detectable.

There are several ways by which increase of current due to exposure to a tracer gas may be indicated. The simplest is by means of a microammeter or a galvanometer. Another method is to utilize the change in voltage across a high resistance to operate an amplifier, which in turn operates a relay.

A third method is to add a relaxation type circuit incorporating a capacitor and glow-discharge tube with a loudspeaker as an indicating element. The current through the sensing element builds up a charge in the capacitor. When the voltage is sufficiently high, the glow-discharge tube operates and the pulse of current resulting from the discharge of the capacitor produces a click in the loudspeaker. The repetition rate of the clicks is an indication of the amount of current.

With any circuit used, it is desirable to include a protective resistor to prevent injury to the sensing element and indicating device resulting from an overdose of tracer gas or a short circuit between electrodes. This resistor may be of the order of 100,000 ohms.

2.2.1.2. APPLICABILITY

Equipment for the heated anode halogen detector procedure is primarily built for detector probe leak location. However, this equipment may be used without modification in static leakage measurement. The major advantage of halogen equipment is that it is designed to operate in air at ambient pressure.

A modification of the equipment allows operation in vacuum. It may be used for leak location as a tracer probe. The equipment as sold may also be used for dynamic leakage measurement. For this application the detector section must be installed in the system being tested.

2.2.1.3. SENSITIVITY

Sensitivity of detector probe instruments is in the order of one part per billion halogen in air. This corresponds to a leakage rate of 1×10^{-9} atm cc/sec using the standard pumps (approximately one cc/sec) on the detector. The vacuum-operated detector works at a pressure of 10^{-1} to 10^{-3} torr. It is capable of detecting halogen gas in concentrations as low as 0.2 part per million.

Sensitivity of the detector varies with different halogen compounds. Table 14 (Ref. 97) gives this sensitivity variation.

Table 14

SENSITIVITY OF HEATED ANODE HALOGEN LEAK DETECTORS TO VARIOUS HALOGEN COMPOUND GASES (Ref. 189)

Limited experimental data indicates that to give the same leak signal as R-12, the leak rate of another gas will be the R-12 leak rate multiplied by a factor as shown below.

Generic Name	Trade Designation	Chemical Formula	Approximate Multiplying Factor By Volume	Approximate Multiplying Factor By Weight
Dichlorodifluoromethane	R-12, F-12	CCl_2F_2	1	1
Trichlorofluoromethane	R-11, F-11	CCl_3F	1 1/4	3/4
Chlorotrifluoromethane	R-13, F-13	$CClF_3$	35	40
Chlorodifluoromethane	R-22, F-22	$CHClF_2$	1	3/4
Trichlorotrifluoroethane	R-113, F-113	CCl_3F_3	1/6	---
Dichlorotetrafluoroethane	R-114, F-114	$C_2Cl_2F_4$	1	1 1/4
	FC-43		131	---
	FC-75		30	100
Sulfurhexafluoride		SF_6	200	240
Methyl chloride		CH_3Cl	1 1/4	3
Perfluorocyclobutane	Freon C-318	C_4F_8	+	---
Perchloroethylene		C_2Cl_4	*	---
Trichloroethylene		C_2HCl_3	*	---
Carbon tetrachloride		CCl_4	*	---

\+ It is believed sensitivity to this gas is 1% of R-12 (multiplier 100) compared with R-12, but supporting experimental data is not available.

* It is believed sensitivity to this gas is almost equal to sensitivity to R-12, but experimental supporting data is not available.

2.2.2. EQUIPMENT AVAILABLE

As far as it can be ascertained, General Electric is the major manufacturer of halogen leak detectors in the United States. Devco Engineering, Inc. is the only other manufacturer.

A brief description of the features of the General Electric models is presented below. The characteristics of the Devco models are similar. The various models of halogen leak detectors have certain features which make them adaptable to specific applications and at the same time increase their versatility and flexibility. The price of these units varies from \$100 to \$1000.

1. Model H. This is the original detector manufactured by General Electric and is no longer sold as a complete unit. It has been superseded by the H-2 model described below.

2. Model H-2. The same control unit is used in models H-2 through H-5. This control unit houses the necessary electronic circuitry, switches, and power supply. Attached to this unit are the various types of detector probes used with the different models. The H-2 uses a "gun" detector that is held in the hand to probe areas where leakage is suspected in pressurized systems. The leak signal can be seen both on a meter in the hand-held gun and on a meter in the control unit. An adjustable audible alarm is also provided.

   The control features include the following:

   a. A speaker circuit which permits the operator to adjust the loudspeaker to give an audible signal at any predetermined leak rate within the leak detector's range.

   b. A sensitivity selector which permits the operator to change from a full-scale sensitivity of 3×10^{-9} atm cc/sec to a full-scale sensitivity of 3×10^{-5} atm cc/sec in ten equal steps.

   c. An automatic-manual balance selector which allows the operator to read total concentration of halogen in the area when the selector switch is in the manual position. In the automatic balance position the leak detector responds only to sudden changes in halogen concentration.

   The automatic balance feature is particularly useful in regions where a high halogen concentration is present in the air. A detected leak is signaled by a sudden pointer deflection on the leak indicating instrument. If the detector probe is held "on" the leak, the pointer will maintain the leak rate level until the leak detector rebalances to the increased concentration of halogen surrounding the leak. The pointer will then return to zero and automatically maintain this position despite varying background concentrations of halogen.

In the manual balance position the leak detector responds to sudden changes in halogen concentration and also responds to any evidence of halogen in the surrounding atmosphere. For example, if the detector probe is held "on" the leak, the pointer will maintain the leak rate reading; it will not return to zero setting. To compensate for constant-level halogen concentrations, the balance control is simply reset to zero.

3. Model H-3. The only difference between this model and the H-2 is that the detector gun is designed to be mounted permanently in a fixed location. This model is only used in assembly line testing.

4. Model H-4. This model has a detector probe designed to be connected to a vacuum system in which a leak is to be detected. The optimum operating range of this detector is between 5 and 20 x 10^{-3} torr, but it may be used satisfactorily at absolute pressures between 1 and 500 x 10^{-3} torr.

5. Model H-5. This model utilizes a pencil-shaped probe which can be inserted into otherwise inaccessible regions. The sensitive element is located in the control unit, rather than in the detector probe itself.

   A feature of the H-5 and H-6 not found in other models is the air-proportioning nozzle in the detector probe. The purpose of such a nozzle is to provide halogen-free air to the sensitive element. This air is provided via a charcoal filter in the control unit that removes all traces of halogen; the air is then pumped through the probe cord to the proportioning nozzle. At the nozzle this clean air can be mixed in any proportion with the contaminated air that is being sucked from the environment. This mixture is then drawn past the sensitive element located in the control unit. In this manner it is possible to get high sensitivity and stability in a contaminated atmosphere. Although some sensitivity is lost because leaking gas is replaced by air, it is more than compensated for by the increased stability and sensitivity.

6. Model H-6. This model uses a control unit different from that of the preceding models. It is smaller and simpler than the others and contains no adjustment switches other than a continuously variable sensitivity adjustment switch. The automatic balance feature is an integral part of the detector and remains in the circuit at all times.

The pencil-type probe contains a neon lamp that flashes in the presence of halogen gases. Because of the automatic balance, the lamp flashes only once when brought near a leak, except when the halogen concentration is so high that the automatic balance oscillates and flashes a few times before coming to rest. After the initial flash or series of flashes, the detector is no longer responsive to the leak. The sensitivity may be re-established by allowing the detector to intake a lower concentration of halogen gas. It will then respond again when placed near the leak. This detector obviously gives only a qualitative response.

Both the H-6 and H-7 have limited duty cycles. The detector cannot be continuously operated for more than one hour at a time and ON time must equal OFF time.

7. Model H-7. The H-7 combines a control unit and a standard leak of 0.5 oz/yr into one compact unit only slightly larger than the H-6. The detector signal is an auditory one rather than a flashing light. The H-7 does not have the automatic balance of the H-6. This detector also gives only a qualitative response, although with the use of a calibrated leak, it can be used for pass-fail testing.

2.2.3. PROCEDURE ADVANTAGES AND LIMITATIONS

The greatest advantage of the heated anode halogen detector is that the detector operates in air. It can therefore be operated efficiently as a detector probe and may be used without complex pumping equipment. The detector is relatively cheap and portable. It may be used by inexperienced personnel without extensive instruction.

Another major advantage is that the detectors are specific for halogen compounds. Although halogen contamination in the atmosphere will sometimes present a problem, the specificity leaves no doubt when the tracer gas is being measured.

A third advantage of the halogen leak detector stems from the fact that halogen-containing gases are soluble in oil. Oil effectively plugs small leaks against internal pressure, since even with high pressure differences there is no clearing force because of the small area of the hole. Oil however, has a high solubility for halogen gases. Consequently, the halogen gases diffuse through the oil clogging the leak and can be picked up by the sensitive halogen detector.

The detector can give a quantitative measurement of leakage both for a total system or for an individual leak.

The major disadvantage of this detector is that it responds to any gases which contain halogen compounds. For example, the detector will respond to solder fluxes, cleaning compounds, and aeresol container propellants. Care must therefore be taken that these compounds are not present on the test area.

The sensing element is hot and therefore cannot be used in an explosive atmosphere.

The halogenated hydrocarbons used in testing are of a high molecular weight and therefore stagnate. Moreover, these compounds do not readily diffuse into all crevices in the system because of their molecular weight.

The sensor element deteriorates with time or upon contact with too high a halogen gas concentration. The detector sensitivity must therefore be checked periodically.

Rubber tubing, and tubing made from plastics must be avoided in any test system, since halogen gas is easily absorbed by these materials and may be released later to show false concentration readings.

2.2.4. PRINCIPLES OF OPERATION

2.2.4.1. LINEARITY AND SPEED OF RESPONSE

The linearity of the response is dependent on halogen concentration. A positive ion current flows at all times from the emitter to the collector of the diode. When a trace of halogen gas is present, the ion current increase is linear to the concentration of halogen from 0 to 1 ppm. For concentrations from 1 ppm to about 1000 ppm, the increase is exponentially proportional. Above 1000 ppm, no further increase of ion current occurs, and the element desensitizes rapidly.

One characteristic of the procedure is the necessity of transferring the gas sample from the probe to the heated anode, which may be some distance away. A time lag therefore ensues between the positioning of the probe and the response of the instrument.

2.2.4.2. TRACER GAS HANDLING

The most popular tracer gases for leak detection are the Freon* series F-12 (dichlorodifluoromethane - CCl_2F_2) and F-22 (monochlorodifluoromethane - $CHClF_2$). These gases are stored as liquids under pressure at room temperature and exert vapor pressures of 70 psig and 122 psig at 70°F above the liquid. If the pressure in the cylinder is reduced through a valve and introduced into a chamber, some or all of the liquid will vaporize to fill the chamber. Liquid will continue to vaporize until the pressure in the gas chamber is equal to the vapor pressure cited above or until no more liquid is left.

During the vaporization process, these refrigerant gases cool considerably. If these gases are being vented into a

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* Trademark, E. I. DuPont De Nemours and Company

rather large chamber, the refrigerant cylinder may cool to the point where vaporization is extremely slow. When this occurs, it might be advisable to hasten vaporization by placing the cylinder in a tank of warm water.

It is sometimes desirable to dilute the gases for the following reasons:

1. Since F-12 and F-22 liquefy at 70 psig and 122 psig respectively at room temperature, a pressurized system being tested cannot have a refrigerant gas pressure greater than these pressures. If leak testing is to be done at higher pressures, additional pressurized air must be added on top of the refrigerant to obtain the desired test pressure in the system. This dilution with air reduces the sensitivity, but the higher pressure increases the leak rate in more than a compensatory fashion. The decrease in sensitivity is proportional to the decrease in concentration; but a more than compensating increase in sensitivity is realized because it is proportional to the square of the pressure rise.

   If Freon (F-22) gas is used, a slight penalty in sensitivity is incurred. This could be offset by the difference in vapor pressures between F-12 and F-22. Pure F-12 cannot be introduced into a system above its vapor pressure of 70 psig, whereas F-22 can be added up to 122 psig. If the bulk of testing is to be done below 70 psig, F-12 is recommended; above 70 psig, F-22 would function better.

2. It may be desired to quantitatively measure the leakage. In that case, the halogen concentration reaching the detector must be relatively low, less than one part per million.

3. In testing large systems, the cost of the tracer gas may be considerable. If only large leaks are of interest, the dilution of the tracer gas will reduce the over-all cost and, as an added feature, decrease the amount of background contamination by the leaks.

The low diffusion coefficient of the heavier tracer gases presents another problem to the efficient use of the halogen detector. In order to produce a dependable signal at a leak, the tracer gas mixture with which the system is charged must have uniform composition. Any blind passages in the system must be flushed with well-mixed tracer gas; otherwise leaks in these cul-de-sacs will simply leak air and escape detection. Those not flushed will remain at low halogen concentration for long periods, because of the low diffusion rate of tracer gas in air (about 0.3 moles/ft^2 hr per unit molar concentration gradient). The time required for a blind duct one yard long to reach 50 percent of full halogen concentration (Ref. 97) is on the order of three hours if diffusion alone is acting. Figure 57 shows

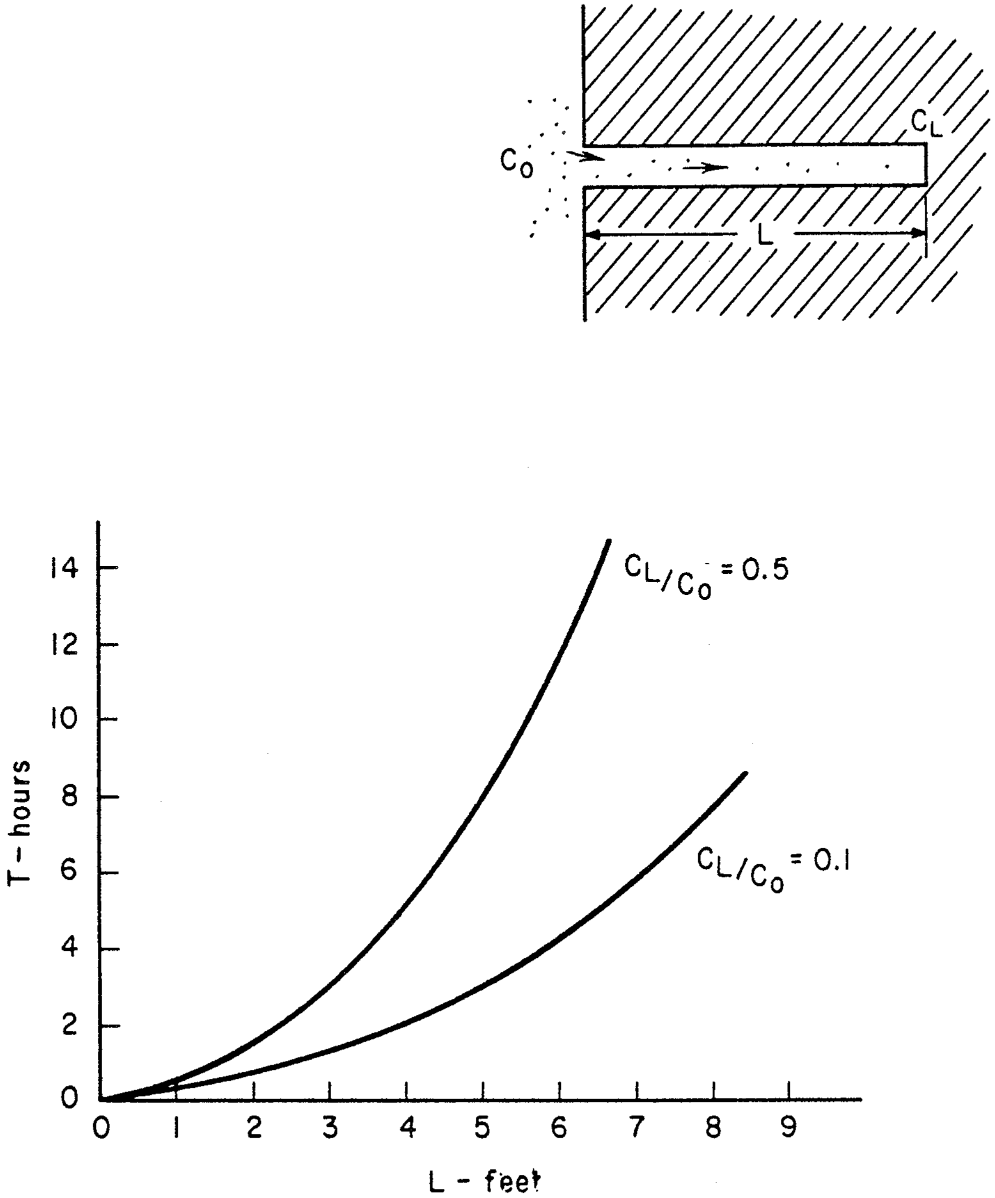

Figure 57. Diffusion of Halogenated Hydrocarbon F-12 in a Blind Duct. (Reprinted, with permission, from R.C. Quisenberry, Leak Detection Techniques Improvement Study for Space Vehicles, Second Formal Report, Ohio University, NASA Contract NAS8-2563, Athens, Ohio, June 1963.)

the time required for various lengths of blind duct to reach 10 percent and 50 percent of open-end halogen concentration. On the other hand, settling out of heavier halogen from an initially well-mixed tracer gas is not significant.

Experience has shown that many of the halogen compound vapors cling for several minutes or longer to surfaces and, therefore, may cause a sluggish recovery response in the detector. This must be kept in mind in connection with the construction of the detector probe and materials of the system being tested.

2.2.4.3 CONTROL OF ATMOSPHERE

Halogenated gases are about three times as dense as air. If tracer gas emerges from a relatively large leak, it will flow into all nearby nooks and crannies and remain there for long periods of time. Its presence in confined spaces may give "ghost" readings up to 24 hours after the original leak has been repaired. The nature and persistence of these "ghost" signals are highly dependent upon the geometry of the stagnant pocket and the ventilation around this space. Pure halogenated gas in an open beaker will be undetectable about 15 minutes after it is filled. An open-mouth Erlenmeyer flask, on the other hand, will contain detectable amounts after sitting on a table top for 24 hours or more. If this same flask were placed in a light breeze near an open window the halogenated gas would vanish in a few minutes.(187)

The detector is suitable for exposure to vapors, provided this exposure is limited to a relatively short time. If the sensing element is exposed to a halogen vapor for too long a time, or to a too highly concentrated vapor, it may lose its sensitivity. Prolonged operation at full temperature in a current of pure air and with voltage between electrodes will restore its sensitivity if the contamination has not been too great. If the contamination has been too great it may be necessary to clean or replace the electrodes. The ease of contamination varies greatly from one compound to another. For instance, carbon tetrachloride contaminates the electrodes more easily than other halogenated hydrocarbons.

The elements used in industrial leak detectors may be expected to last 500 to 1,000 hours with proper maintenance and operation. Those elements used in the servicemen's leak detectors are much smaller and will not last as long. In general, little can be done to extend the life of these units. The elements used in servicemen's leak detectors, however, are much lower in cost.

The halogen detector responds to solid particles of the iodides, chlorides, bromides and fluorides. Therefore, it detects smoke from burning materials containing such compounds. The tobacco used in cigarettes must contain such a compound, since the detector responds readily to cigarette (and to some extent cigar and pipe) smoke. There is a belief that the response of the detector is to the alkali ash in the smoke. The platinum diode is not effected by cigarette smoke. When looking for small leaks, a NO SMOKING rule in the vicinity should be enforced.

The inherent sensitivity of the halogen detector cannot be reached in practice if there is background contamination. A large leak near a small one may completely obscure the signal from the small one. In many factory test areas, a background level of halogen gas will build up, due to leaks in the units being tested, leaks in the refrigerant supply tank, dumping of gas, and other sources that may allow halogens to enter the area.

In a testing set-up, the chief precaution is that of making certain that the testing is carried on in an ambient that is sufficiently free of halogen vapors. In many instances, an exploration of the available floor space will indicate a location that is free of these vapors. If such a location is not available, it may be necessary to partition off a testing area and provide proper ventilation to bring in outside air that is halogen free. When speaking of halogen-free air, it should be understood that the presence of halogen vapors in proportions of ten parts per million may be sufficient to contaminate the air enough to cause a loss of sensitivity in the testing equipment.

The automatic balance feature of the control unit will balance out a certain amount of halogen background, provided the concentration is constant or changing slowly. However, if this background level builds up to a point where normal air currents present in the room cause sudden changes in background concentration, sudden leak signals may result even when the detector has not encountered a leak.

There are three methods of combating this condition:

- First, eliminate sources of background.
- Second, provide a controlled enviornment of fresh air in the testing area.
- Third, use a proportioning probe detector.

2.2.4.3.1. Halogen Background Elimination

In some instances, elimination of the halogen background may be an inexpensive and simple step to control testing atmosphere. For example, it should be possible to control indiscriminate dumping of refrigerant charges, leaky lines, degreasers using halogen solvents, paint fumes, etc.

2.2.4.3.2. Controlled Fresh Air Environment

This second approach can be a very elaborate one or a very simple and inexpensive one, depending upon how bad the background problem is. Manufacturers of refrigerators and air conditioners have found it desirable to construct a small room or booth for testing, since the background level of tracer gas is usually very high in their testing areas due to the many sources of halogen gas. This room or booth is fed fresh air from the outdoors at a very low velocity to prevent excessive drafts and eddy currents of air within the room. The booth, in effect, isolates the leak testing area and helps to keep background contamination from interfering with the leak testing.

Basically, the booth is a four-sided structure with a roof. Through the roof, fresh air is introduced so as to provide an air change once or twice a minute. A test booth is illustrated in Figure 58.

Some important things to keep in mind in constructing the booth are:

1. Fresh air should be supplied to the booth from the outside. In some cases, this air should go through an activated-charcoal filter bed. This will remove any halogen gas present in the air.

2. The fresh air from the blower should be diffused before entering the booth to prevent drafts or eddy currents of air within the booth. A false ceiling made of peg board is ideal to provide an even flow of air in the booth. When the booth is kept under this even positive air pressure, contaminated air will not come in from the shop or factory.

3. If a conveyor goes through the booth, canvas or rubber curtains help to maintain the integrity of the booth.

4. An air conditioner in the booth will be helpful in providing comfort for the booth personnel and to help clear the air. It will, of course, also help remove humidity. Care should be taken to baffle the air conditioner so that the cool air is distributed as evenly as possible in the booth.

5. Construction techniques will vary from factory to factory. Some fabricate the booth out of wooden studs. Sheathing can be a type of hardboard or sheet rock. Others use the metal, movable partitions commonly found in offices.

The roof of the booth quite often is fabricated of metal. The pipe is standard air conditioning air handling pipe (four or more inches in diameter) right up and through the roof or through the side of the building.

The filter chamber should be large enough to allow a liberal amount of activated charcoal to be used. The larger this chamber, the less often the charcoal will have to be changed. Thought

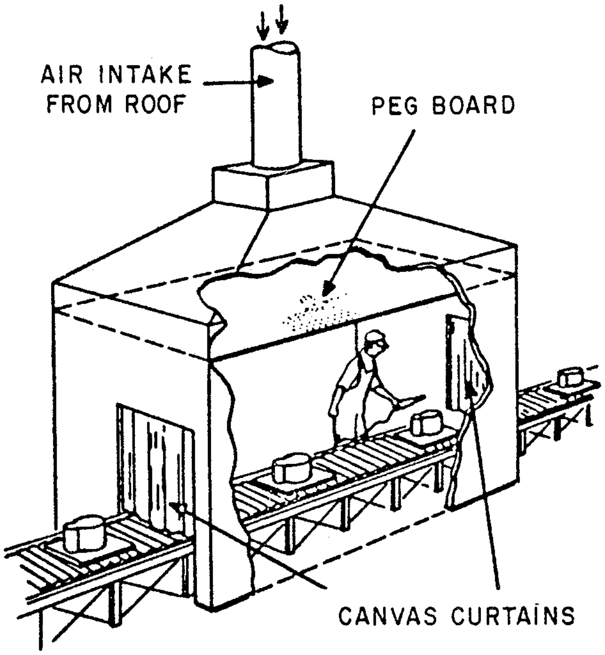

Figure 58. Controlled Atmosphere Test Booth.

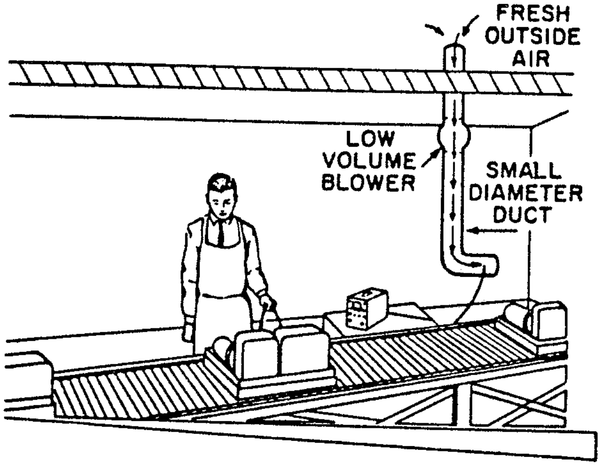

Figure 59. Fresh-Air Ducting for a Proportional Probe.

should be given to ease of change. One way to contain the charcoal is to use a drawer. The bottom of the drawer can then be a fine-mesh screen to allow air-flow through the charcoal.

In other areas where the background level is much lower, it may not be necessary to use any special ventilating techniques, other than the normal ventilation required for good health. A simple ventilating device that can be used, if it is impractical to build a special room or booth, is a portable 16-inch or 20-inch fan placed in a window or doorway. Arranged in this manner, the fan will usually clear out the halogen background to the point where leak testing can be accomplished satisfactorily.

2.2.4.3.3. Proportioning Probe

The third method to overcome leak test problems commonly associated with area contamination is the proportioning probe. During test, air is drawn from the atmosphere into the probe. If halogen content is excessive, incoming air is mixed with pure air to effectively reduce contamination. The probe's adjustable valve controls this important proportioning process.

In a "clean" test area, no pure air blending is needed. The probe's proportioning valve can be opened wide. All air then enters the probe tip from the atmosphere and is passed directly into the control unit and over the sensitive element.

In a heavily contaminated area, the proportioning valve is closed part way to restrict the sample intake. At the same time, pure air from the probe's fresh air filter is pumped to the probe and is mixed with the incoming sample. The mixture travels into the control unit and passes over the sensitive element. If the probe has passed near a leak, a signal proportional to the size of the leak is given.

With its built-in pure air filter, the probe requires no costly construction of specially ventilated test facilities to combat area contamination. However, to avoid halogen-saturation and subsequent replacement of the probe's expendable air filter, a supply of fresh air can be ducted directly to the filter. Figure 59 illustrates a simple, inexpensive method. A small pipe can be led from the test area to the outdoors or any other area that is relatively free of halogen contaminants. A small, low-volume centrifugal blower is then installed to force a supply of fresh air to the test end of the pipe. One end of an extension tube is connected to the filter intake and the other end is allowed to rest in the pipe exhaust.

Pipe size, blower capacity, size and number of exhaust holes can vary greatly. The only criterion is that a positive exhaust pressure be maintained in the pipe to prevent halogen contamination from entering the extension tube.

If no satisfactory supply of uncontaminated air is available from the outdoors or the plant area, one of the following alternatives should be used:

Shop air - which can be metered to bleed off into a small container at a rate that will maintain an uncontaminated atmosphere around the filter tube extension resting in the container. This method is useful only when the intake to the shop compressor is in an area which is free of halogen contamination.

High-pressure tank of air or nitrogen - which can be metered to bleed off into a small container at a rate that will maintain an uncontaminated atmosphere around the filter tube extension resting in the container. This method has the advantage of portability when the probe has to be used for leak testing in more than one area.

Larger filter - simply replace the small air filter supplied with a larger, separately mounted unit. This could consist of a container, air-tight except for intake and exhaust, filled with commercially available activated charcoal.

2.2.4.3.4. Measuring Contamination

It is possible to measure the extent of atmospheric contamination if a leak standard, a halogen detector, and a pure air supply are available.

In a contaminated area, with the leak detector operating at a known air flow, the leak detector should be allowed to intake pure air for about one minute. Then the detector should sample the contaminated area. If the leak detector gives a signal, the area is contaminated. The magnitude of the leak detector setting is noted but the sensitivity of the leak detector should not be adjusted.

The leak detector and leak standard should be moved to an area where there is no atmospheric contamination. The leak standard must be adjusted so that when the leak detector "sniffs" the leak standard port, the leak signal indication is the same as when the leak detector "sniffed" air in the contaminated area.

The indicated leak rate in atm cc/sec (on the leak standard) multiplied by 10^6 and divided by the air flow of the leak detector in cc/sec equals the contamination in parts per million.

2.2.4.4. SAFETY CONSIDERATIONS

In these instruments the leak detection element operates at about 1600 F and voltages of 300 volts are present in the amplifier circuit. The following safety precautions must be observed.

1. Never enter an area where there is an explosive vapor. If there is any question, first test the area with an explosion meter.

2. Never test in enclosed spaces, such as bearing housings, oil tanks, or piping without first testing with an explosion meter.

3. Since voltages as high as 300 volts are present, the case should be kept at ground potential by using a three prong grounded receptacle.

2.2.5. METHODS OF TESTING

This procedure is applicable for two types of operation: testing in atmosphere and testing in vacuum. In atmosphere, the equipment is used for static leakage measurement and for detector probe leak location. In vacuum the equipment is used for tracer probe leak location and for dynamic leakage measurement.

The use of the leak detector is not limited to apparatus into which a detector gas can be injected. In a liquid-filled sealed unit the same leak-testing techniques can be employed, provided one of the halogen compounds can be added to the liquid at the time of filling without detriment to the liquid. The compound used must be one that will quickly vaporize. In some types of electrical and thermal apparatus, such as liquid-filled indicating thermometers and liquid-cooled equipment, the liquid used may itself contain a halogen compound and therefore will not require any additives.

The one requirement is that the halogen compounds used have an appreciable vapor pressure. For example, Pyranol cannot be detected on a halogen leak detector at room temperature. However, if the compound is heated, the vapor pressure is increased to a point where detection is possible.

2.2.5.1. TESTING IN ATMOSPHERE

In order to make optimum use of the capabilities of the halogen leak detector, the following procedure is advised.

1. Check and repair larger leaks first. This should be done before the system is fully pressurized. This prevents excessive contamination of the ambient and will be a time-saver.

2. Charge the system with tracer gas after larger leaks have been corrected.

3. Use pre-mixed tracer gas. Its concentration should be checked by appropriate metering to avoid over-or under-rich mixtures. During charging with tracer, dead-end ducts should either be opened to allow trapped air to escape or time should be allowed for diffusion to take place. Alternatively, the system should be evacuated before charging with tracer gas.

2.2.5.1.1. Leak Location

For leak location using a detector probe, the system is pressurized with tracer gas and probed with the detector. Certain precautions are necessary in this probe exploration. Too rapid a search may miss the very small leak. If this risk is to be avoided, the speed at which the probe is moved must be in proportion to the minimum leak tolerance. In testing equipment for a leak specification of the order of 10^{-5} atm cc/sec, the travel can be one to two inches per second; but probe speed should be reduced to 1/2 inch per second for smaller leaks.

If the tracer gas escapes into the room after the test has been completed and the test piece opened, sufficient time must be allowed to permit the ambient to again become free of halogen vapors. If the waiting period cannot be tolerated, there are two alternatives that may be employed. The test piece can be removed to an outside area before permitting the gas to escape, or a vacuum system can be used to remove the gas before opening the test piece. The use of the vacuum system has been found to be the more satisfactory. Although some gas usually remains in the test piece after evacuation, and therefore tends to contaminate the air, the time lost in regaining clear air is relatively small.

If large volumes are to be tested, it is possible to recompress the tracer gas for reuse.

The degree of mechanization employed in a testing setup is dependent upon the production rate, the uniformity of test pieces, and the total time available for hand work by the operator.

It is not possible to depend on natural dissipation of escaping gas in a high-speed production test; it therefore becomes necessary to remove gas in the shortest possible time. The design must include adequate vacuum lines to remove the gas from the test piece after testing, and proper forced ventilation must be employed to clear the surrounding air. A hood has been found to be most suitable for this purpose.

When sufficient incoming air flow is used to clear away leaking gas, steps must be taken to properly direct the air so that it does not remove the gas at the point of the leak, before the detector has had time to pick it up and indicate the leak.

2.2.5.1.2. Leakage Measurement

Leakage measurement using the detector operated in air is a static accumulation procedure. Because the system will be leaking to atmosphere, the collecting container does not have to be completely tight. For example, some manufacturers package their refrigerators and air conditioners in cartons, and make a final leak check on their product in the warehouse before shipment (Ref. 98). This is accomplished by making a hole near the bottom of the carton with a punch the size of a pencil, and then inserting the probe of the detector. The leak detector will sample air from the carton. A leak signal will occur if the unit in the carton is leaking.

Even though the over-all leakage rate of the unit in the carton may be very low, a sustantial leak signal will be obtained when performing this test if the unit has been in the carton any length of time.

The approximate total leak rate can be determined as follows:

1. Note the signal from the leak detector when sniffing the carton.

2. Adjust the leak standard so that when sniffing the standard the signal is the same as for the carton.

3. Compute:

$$\text{Total leak rate} = \frac{VR}{T}$$

where V = volume of the carton in cubic feet, minus volume of the unit in the carton in cubic feet.

R = indicated leak rate in oz/year of leak standard with the air pump set for one cubic foot per hour.

T = time in hours the unit has been sealed in the carton.

In some circumstances a tray under the test piece may collect the leaking gas, thus making it necessary only to test the air in the tray. In areas where drafts may blow the gas away, it may be necessary to completely enclose the test piece. In either case, sufficient time must be allowed for the gas from the smallest leak to collect in sufficient quantities to be detected.

2.2.5.1.3. Reading Interpretation

It is desirable that the surfaces to which the nozzle is applied be free from loose lint, dust, or light grit. These might be sucked into the sensitive element forming a partial short circuit and thus giving a false indication of a leak. This problem is usually prevented by including some form of air filter in the suction tube.

The device is quite sensitive to variations in air flow. If the air flow is too rapid, the vapor apparently has only limited opportunity to strike the hot cathode and dissociate. The sensitivity of the device therefore decreases as the air flow is increased. Part of the decreased sensitivity is also due to the additional cooling of the cathode with increased air flow. On the other hand, if the air flow is at too low a rate, the device will be extremely responsive to vapors. However, considerable time will elapse before the current returns to its normal no-vapor condition, even after the inlet is again given a supply of pure air.

The response of the sensing element increases markedly with temperature over a narrow range. Below approximately 850 C, the emission current is too small to be easily utilized; over approximately 950 C it becomes unstable and random fluctuations will hide any signal.

2.2.5.2. TESTING IN VACUUM

The halogen leak detector may be used for leakage measurement or leak detection in vacuum by inserting the detector head directly into equipment having integral pumping systems. When inserted in the high-vacuum chamber, best sensitivity will be obtained if the valve to the diffusion pump is throttled. If the leak is not large enough to keep the optimum operating range, a small air bleed can be inserted to do so. For checking leakage in the fore pump portion of the system, as well as in the high-vacuum chamber, the detector may be located just ahead of the forepump.

The optimum pressure for vacuum operation is between 10^{-3} and 10^{-1} torr (Ref. 99). In vacuum operation, the detector is somewhat sensitive to pressure variation.

When vacuum leak testing small items on a short time cycle basis, the detector should be maintained in a "ready" condition in a small vacuum system of its own. This is desirable because of the large thermal inertia of the detecting diode. The system should preferably maintain the element at about 5×10^{-3} torr pressure, and provide a small, steady flow of air through it to maintain sensitivity. The units to be tested should be evacuated to a pressure somewhat above that in the detector system, and a sampling valve opened between the two before starting the leak testing procedure.

Great care should be taken to make all vacuum piping short and large in diameter, as the tracer gas moves primarily by diffusion, there being little or no pressure differential to cause flow.

Sensitivity multiplication can also be obtained in vacuum leak testing by using either pressurized, external tracer gas, or allowing accumulation in the system, or both.

The sensitivity of the detector unit will decrease as a result of prolonged exposure to pressures less than 10^{-3} torr. Exposing the element to air for a while, or bleeding a small amount of air into the system should restore sensitivity.

If, after the leak detector has been used for some time, there is a lack of sensitivity, or there is erratic or off-scale indication, the sensitive element must be removed and cleaned or replaced.

In using the tracer probe for leak location, probing should be initiated at the bottom of the system. Movement should continue upward in a careful pattern. If probing is done above an undetected leak, tracer gas will drift down and provide a spurious signal.

The comments on signal magnitude and response time discussed in Section 1.3 apply in this technique.

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Section 2.3

PRESSURE CHANGE PROCEDURE

The pressure change procedure consists of leakage testing by detecting pressure change in a system. In this procedure the system is maintained at a pressure other than atmospheric. The test may be run with the system either evacuated or pressurized. This procedure is used only for leakage measurement.

2.3.1. PRINCIPLES OF PROCEDURE

2.3.1.1. CHARACTERISTICS

The system being tested is sealed and either pressurized or evacuated. The leakage measurement is the pressure change, multiplied by the system volume, divided by the time of the change increment.

2.3.1.2. APPLICABILITY

This procedure is used primarily for leakage measurement on large systems, but with a minor modification, it can be used to measure leakage on any size system. Although a leak may be localized by this procedure, it should not be used for leak location.

2.3.1.3. SENSITIVITY

In the pressurized system, sensitivity is dependent on the minimum detectable size of the pressure fluctuation. In the evacuated system, sensitivity is dependent not only on the pressure change which may be observed, but also the degree to which this pressure change is due to outgassing. Removal of the outgassing factor that causes pressure change increases the sensitivity of the technique. However, because smaller pressure changes can be read, the evacuation mode is a more sensitive technique. This is a static leakage measurement procedure; therefore, sensitivity is largely dependent on the duration of the test.

2.3.2. EQUIPMENT AVAILABLE

In most cases, pressure gages normally mounted on the system are used. However, a number of manufacturers are producing special differential gages specifically designed for leak testing. The characteristics of these gages are shown on Table 15.

2.3.3. PROCEDURE ADVANTAGES AND LIMITATIONS

The major advantages of this procedure are:

- The system may be tested using gages present on the system.
- No special tracer gas is required.

Table 15

COMMERCIAL PRESSURE CHANGE LEAK DETECTORS*

General Characteristics	Average Values
Sensitivity	1×10^{-6} torr
Pressure Range	3×10^{-4} torr to 10^{3} torr
Tracer Gas	None
Output Signal	Visual Observation
Power Source	115/230 volts, AC; 28 volts DC
Size	Sensor: 4" x 4" x 3" Electronic Unit: 8" x 8" x 12"
Weight	4 to 40 pounds
Price	Up to $2500

Manufacturers

Accessory Controls and Equipment Corporation
Datametrics, Incorporated
MKS Instruments, Inc.
Trans-Sonics Incorporated

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* More specific information on this equipment may be obtained from General Electric Company Report S-67-1013 (Ref. 186)

The accuracy of this procedure is high in the pressurizing mode; it is dependent of how precisely the volume is calculated and the pressure changes can be measured. In the pressurizing mode, the accuracy of this leakage measurement is easily traceable to the United States Bureau of Standards.

Two major disadvantages of this procedure are:

- The time required for the test is often rather long.
- This procedure does not permit leak location.

In the evacuation mode, a major limitation is outgassing of the system which masks the pressure rise due to leakage.

2.3.4. METHODS OF TESTING

2.3.4.1. PRESSURIZING MODE

The pressurizing mode of leakage measurement consists of filling the system with gas and observing any pressure decrease. The fundamental equation for this test is:

$$Q = \frac{V \Delta P}{t} \qquad \text{Equation 2.3-1}$$

where Q = leakage
V = system volume
ΔP = pressure decrease during test
t = duration of test

There are two large sources of error in this technique. System volume is difficult to calculate in a large or complex system. However, it can be measured by an "additional leakage" technique (Ref. 100). An additional known leak is added to the system. The system volume is then calculated from the effect of the additional leakage on the pressure decrease.

The second error inherent in this test method is that temperature variations during the test cycle tend to vary the pressure in the system (Ref. 101). This error may be corrected by measuring system temperature during the test and incorporating this information into the calculations by using the ideal gas law. Alternatively, a reference volume may be placed in the system and pressure variations in this volume observed.

2.3.4.1.1. Testing Specifications

This technique has been used to test extremely large systems (Ref. 102). For example, it has been used with a 190-foot diameter sphere of 3.6 million cubic feet volume containing a nuclear reactor (Ref. 103). It has been standardized into a specification (Ref. 4) by the Standards Committee of the American Nuclear Society for Leakage Rate Testing of Containment Structures of Nuclear Reactors. Because this technique may be generally applicable for testing many other types of systems, the entire specification is included as Appendix III to this handbook.

2.3.4.1.2. Automated System

Picatinny Arsenal (Ref. 104) uses a differential pressure drop method to measure leakage in sealed devices. A schematic of the system is shown in Figure 60. The unit to be tested is placed in the test chamber. The gross leak chamber is pressurized to test pressure and sealed. After the pressure measurement, the valves leading to the test and reference chamber are opened. If the pressure drops below a predetermined value, gas has entered the unit being tested and the test is stopped. If the unit passes this gross leak test, the valves are closed and the pressure difference between the test chamber and reference chamber measured. The leakage is measured as the pressure drop in the test chamber.

This technique gives rapid (30 second) measurements of leakage in the full range from gross leaks to 1×10^{-4} atm cc/sec. It can be fully automated so skilled test personnel are not required. Any test gas may be used, including air or nitrogen.

2.3.4.1.3. Leak Localizer

Gas companies use a modification of the pressurizing mode of leakage measurement to localize leaks in gas mains (Ref. 105). The main is tested, section by section, from the inside with the leak locator. The leak locator consists of the following parts:

1. A flexible frame on which are spaced two rubber gas bags joined with a rubber tube.

2. A duo-rubber tubing of sufficient length; this tubing reaches from the bags to the control panel. One side of the duo tubing extends through the adjacent bag and is open to space between the two bags. This tube transmits the gas pressure to the control panel.

3. A control panel connected to the duo tubing with an inclined water gage is used to measure any variation in gas pressure; a spring gage is used to indicate the air pressure in the bags; connections are included for inflating and deflating the bags.

4. Two pumps are needed -- a pressure pump to inflate the bags and a suction pump to deflate the bags.

5. A steel rod is required to propel the bag frame and tubing along inside the main. The rod is connected mechanically to the bag frame and is of sufficient flexibility to pass through a tap in the main, and of sufficient stiffness to avoid buckling when pushing the apparatus along inside the main.

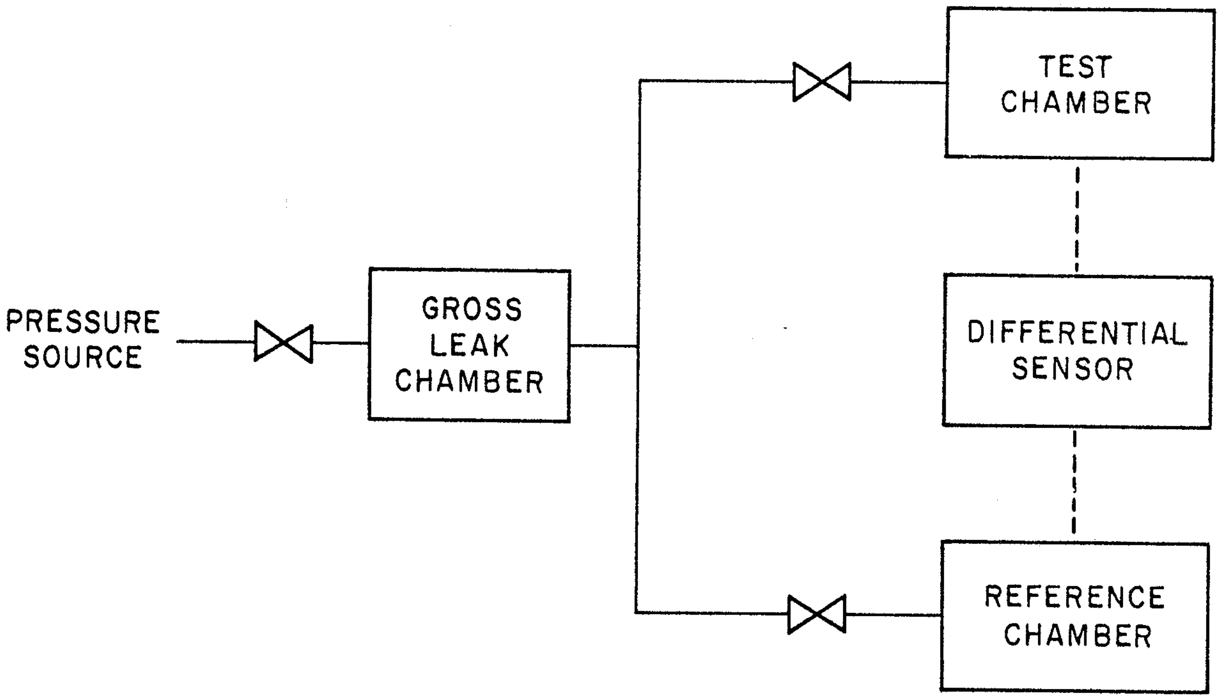

Figure 60. Differential Pressure Change Method of Leakage Measurement. (Courtesy of Picatinny Arsenal, Department of the Army. (Ref. 104))

In testing for a leak, the bags are inserted in a main containing gas under pressure. They are spaced a set distance apart on the frame, so they seal off a portion of main when inflated. The bags are inflated with a pressure tire pump and make a seal with the inside of the pipe, when under from three to six pounds of pressure. The gas pressure between the bags is registered on the water gauge as soon as the bags are in the main. When the main is sealed off completely by both bags, and there is no leak in the main between the bags, the pressure between the bags will remain constant.

After a section has been tested, the bags are deflated with the suction pump and moved forward a distance to permit an overlapping of test lengths. This is important because otherwise a leak could be overlooked if a bag were to rest on the leak while making a test.

If a leak is isolated between the bags, the gage pressure drops as soon as the bags are inflated. To locate the leak more precisely, bags are deflated and moved half the distance between the bags. If they still straddle the leak they are moved forward or backward by smaller increments until the rear bag just passes beyond the leak. The steel rod is marked and the measurement can be transferred to the pavement above the main.

2.3.4.2. EVACUATION MODE

The evacuation mode of leakage measurement consists of evacuating the system being tested, sealing from the pumps, and monitoring the change of pressure with time. Equation 2.3-1 is used to calculate the leakage, (this is the same equation used in the pressurizing mode). Since smaller pressure differences may be observed at the lower pressure, this mode should be more sensitive than the pressurizing mode.

The sensitivity and accuracy of the vacuum gage should be appropriate to the rate of pressure rise to be expected. For measuring small pressure changes (low leakage rates), a gage of high sensitivity is required, but the upper pressure limit of the gages has to be allowed for. Certain vacuum gages have a non-linear pressure scale showing a higher sensitivity at lower pressures than at higher pressures. Consequently, it may happen that the pressure rise has reached a level at which the gage is relatively insensitive to small pressure changes due to leakage, or the pressure level has fallen outside the range covered by the gage.

2.3.4.2.1. Outgassing Problems

Under certain (or it seems, most) test conditions, there is a rapid rise of pressure due to outgassing once the system has been isolated from the pumps. Outgassing is an evolution of adsorbed gases from the system surfaces. It is an equilibrium process, the pressure eventually reaching a steady-state value, as illustrated in Figure 61, Curve a. On the other hand, leakage

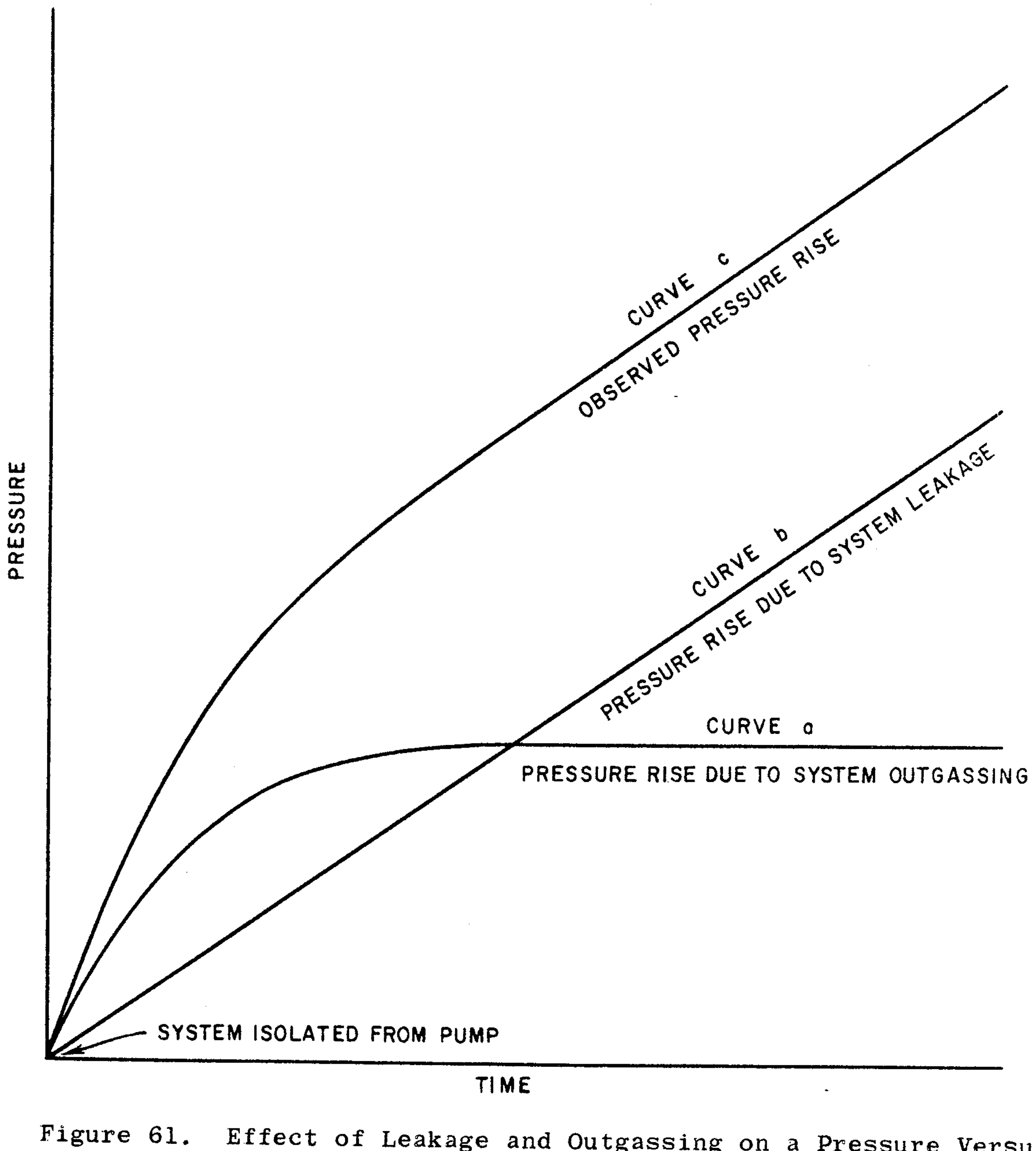

Figure 61. Effect of Leakage and Outgassing on a Pressure Versus Time Curve.

(without outgassing) produces a straight line on a plot of pressure versus time. This is illustrated in Figure 61, Curve b.

During leakage measurement on an actual system, the curve obtained is a summation of curves a and b and is illustrated in Figure 61, Curve c. Obviously, if the outgassing is too great, the pressure rise due to leakage will not be observed.

There are four ways to discriminate between the pressure rise due to outgassing and that due to leakage:

1. Outgassing minimization
2. Straight line extrapolation
3. Gage trapping
4. Vacuum hooding

2.3.4.2.1.1. Outgassing Minimization

Outgassing of the system may be minimized if certain precautions are taken. The system should be kept clean and dry. Any oil or grease on the inner surfaces of the system should be removed before testing, because it absorbs contaminants.

The system should not, if possible, be exposed to water or water vapor. No liquids having a measurable vapor pressure should be present in the system, because the system pressure will rise to their equilibrium value. The system should be operated in such a way that no virtual leaks (see Section 1.5.3.) are present.

Two ways to reduce outgassing in a system are by heating the system under vacuum and by use of a dry gas bleed. The latter procedure is described in Section 1.5.3.

2.3.4.2.1.2. Straight Line Extrapolation

If the system being tested is isolated from the pumps, the pressure rise due to outgassing will cease when an equilibrium is established between the gases in the vacuum and on the system surfaces. This has been illustrated in Figure 61. It is therefore necessary to wait until this equilibrium is established, before leakage measurements are taken. The pressure rise may be considered as due only to leakage when the rise in pressure during successive time increments is equal.

2.3.4.2.1.3. Gage Trapping

Most of the gases evolved during outgassing of a system are condensable. Therefore, if a liquid nitrogen trap is placed between the gage and the system wherein pressure rise is being measured, the pressure rise observed will be due only to leakage (air is not a condensable gas under these conditions). Care must be taken that an equilibrium value of pressure rise is obtained when such a technique is employed. When the liquid nitrogen trap is first cooled, system pressure will decrease because of the pumping effect on condensable gases by the trap.

This pressure decrease must be terminated before leakage measurement is performed.

2.3.4.2.1.4. Vacuum Hood

It is possible to discriminate between outgassing and leakage by means of an auxiliary vacuum hood. The test setup is shown in Figure 62.

The technique involves taking two sets of readings, the first with the system under test evacuated in the normal manner and measurements made of pressure rise over a selected time interval. The hood is then lowered over the component, and sealed on a suitable base. The hood is evacuated to about the same pressure as the system. A second pressure rise measurement is then made. The rise is now due to outgassing only, since there is no pressure difference between the inside and outside of the system. The arithmetical difference between the two rates gives the true leak rate. It will be appreciated from the brief description of the method that it is most suitable for repetition testing of standard components, where the provision of a suitable hood and an additional pump station is justified by the saving in time. A scheme in which the same pumping system is used to evacuate both the hood and the system under test is sometimes employed.

2.3.4.2.2. Volume Sharing Technique

The volume sharing technique is applicable to systems or parts of systems which do not contain their own pressure gage. The use of this technique is illustrated in Figure 63.

The tested volume, V_1 is evacuated to base pressure, P. The volume of the total system is V and the time which the system is isolated is t. At the end of this time, the volume, V is isolated from the pump, the volume, V_1 is opened, and the pressure in the system P_2 is measured. The leakage in the system is now calculated using the equation:

$$Q = \frac{V}{t} (P_2 - P_1) \qquad \text{Equation 2.3-2}$$

where Q = leakage
V = volume of vacuum and test system
t = isolation time
P_2 = final pressure in vacuum system
P_1 = starting or base pressure in system

This method can be quite sensitive since it can be made as large as desired. Vacuum components can be isolated for days to test for leaks.

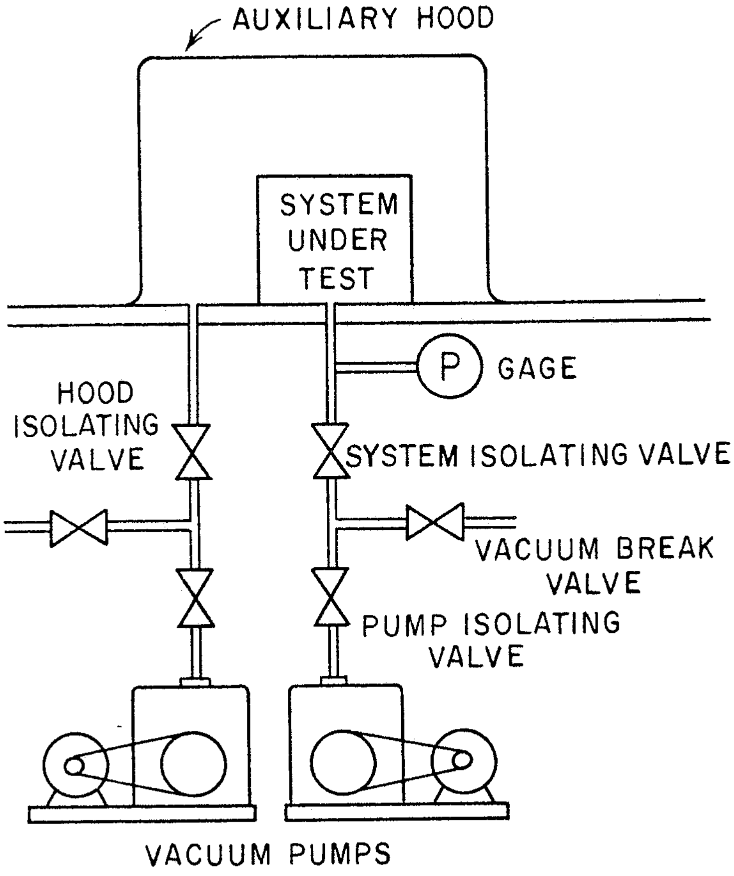

Figure 62. Test Setup for Pressure Change Testing, Using a Vacuum Hood.

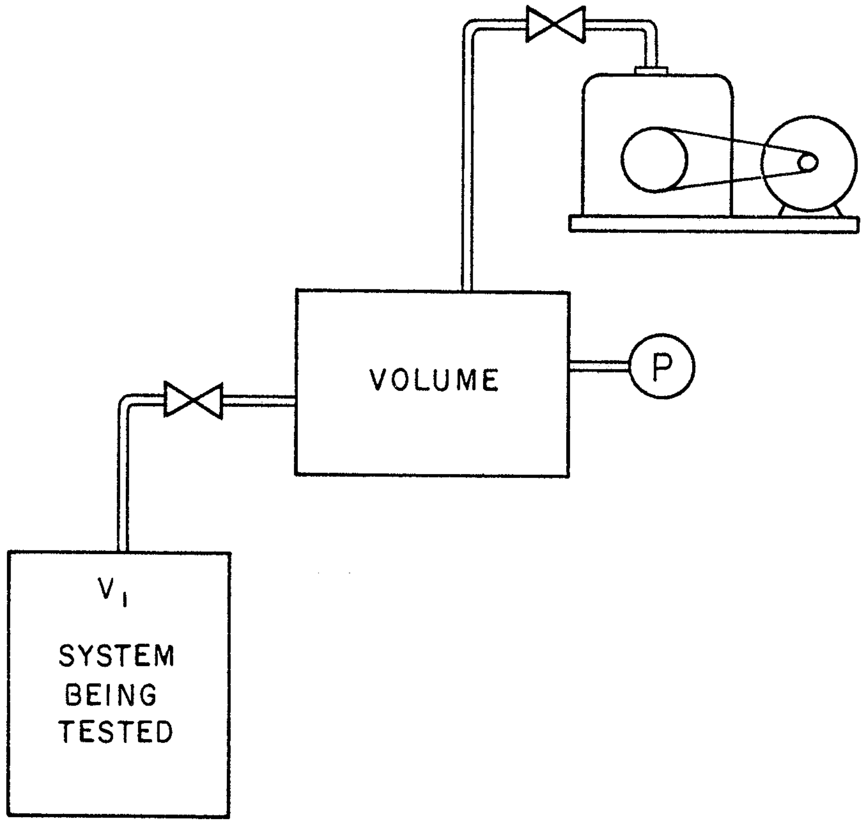

Figure 63. Volume Sharing Technique of Leakage Measurement.

2.3.4.2.3. Automatic Leak Testing

The pressure rise technique was used in an automated test setup (Ref. 106). In order to apply this principle in series tests, a carrousel type vacuum lock was developed for locking the specimens in and out of the test chamber expeditiously (Figure 64).

The carrousel lock consists of an upper stationary steel plate and a lower rotating steel disc. The rotating steel disc contains eight pouches, connected with a small auxiliary pump which keeps the pressure below 0.7 torr. Thus, any possible leaking path to the high vacuum chamber has only 1/1000 of the atmospheric pressure at its inlet. This reduces spurious leakage. With a test rate of 60 units per hour, all leakages of 2×10^{-8} torr liter/sec and higher can be detected. The manometer is an ionization gage in series with a liquid nitrogen trap.

The samples are cleaned by washing in trichlorethylene. They are then warmed on a hot plate to 80-110 C in order to remove moisture. The cleaned units are put into the pouches through a filling hole in the upper stationary plate. Following the direction of turning in Figure 64, a connection with the auxiliary pump is first made, later with the forepump of two small oil diffusion pumps, then with the first of the oil diffusion pumps which brings the pressure in the pouches down to about 1×10^{-5} torr. Then the pouch reaches the "Testing" position, and is connected with the second of the oil diffusion pumps and the manometer.

If, in the "Testing" position, a pressure of 2 to 5×10^{-6} torr is reached, usually after 15 to 20 seconds, a test valve is pressed down interrupting the pumping. The manometer and pouch with the sample form one closed chamber and the manometer is watched for a pressure increase.

Usually, a small amount of gas is given off by the walls of the chamber and specimen, causing a pressure rise of 0.5 to 2×10^{-6} torr within the first few seconds, depending upon how long the pouch was cleaned by pumping at 10^{-6} torr. After 15 seconds it can usually be decided whether a small leak is present or not. Larger leaks cause an immediate pressure increase to 10^{-5} or 10^{-4} torr; as soon as such a rapid rise is seen, the test valve is released immediately, so that the test chamber is not contaminated.

Before the test valve is released, the pressure in the test chamber will be determined by the equilibrium between leak rate and pumping rate. For large leaks, the equilibrium pressure is in the order of 10^{-4} to 10^{-3} torr. If a leaking unit is left in the test chamber long enough it will be emptied by the pumping. Calculations show, however, that more than an hour will be required to empty it so completely that it will no longer be seen in the test. Thus, the apparatus can be used to test for large leaks as well.

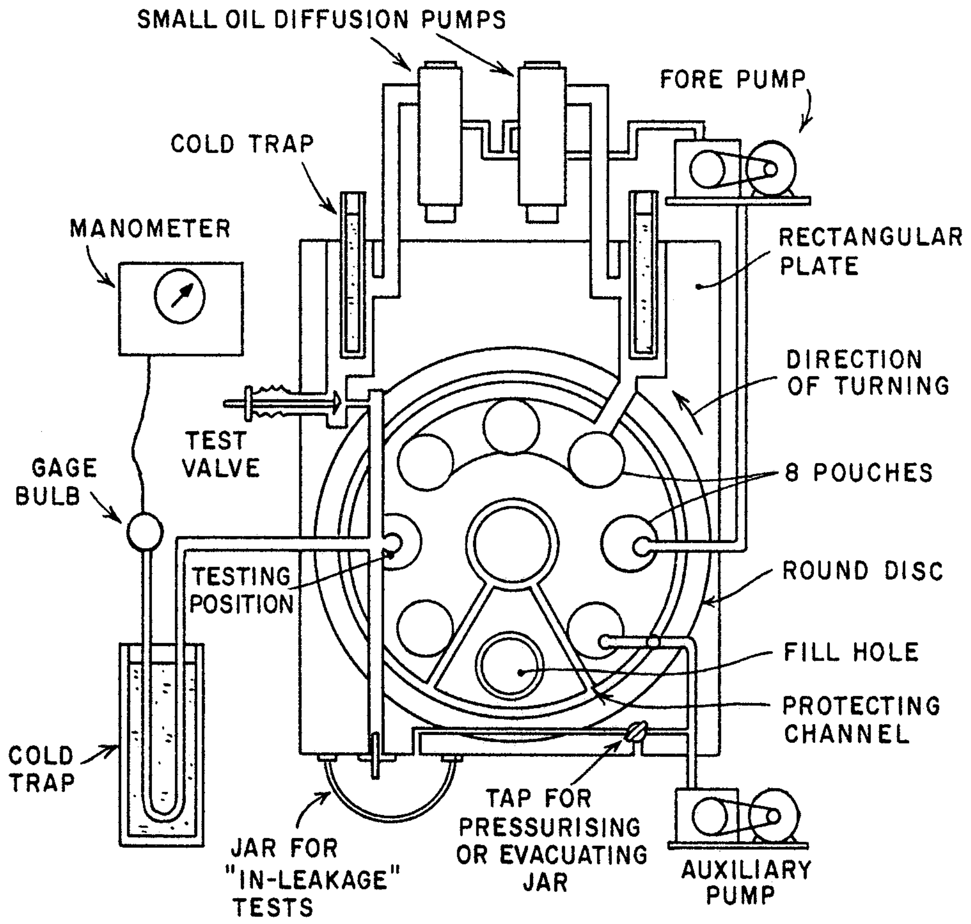

Figure 64. Pressure Change Procedure, Using a Carrousel Vacuum Lock (Reprinted with permission from B.W. Schumacher, Transactions, 1956 National Symposium on Vacuum Technology, pp. 110-113, Copyright 1956, Symposium Publications Division, Pergamon Press.)

The limit of sensitivity is determined by outgassing. After pumping for a time, the outgassing background will be around 1.5×10^{-10} torr liter/sec.

Because all but one of the test chambers are under vacuum, not much gas is given off by the walls of the chamber. For a pouch left under atmospheric pressure overnight, the outgassing would be higher if the pouch is turned to the testing position whithout leaving some time (e.g., 10 minutes) for outgassing. It is easy, however, to check the state of the pouches by a blank test.

2.3.5. GENERAL REFERENCES

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6. Schumacher, B. W., "A Nondestructive Leak Detector for Production Testing of Hermetic Enclosures for Electronic Components," Vacuum Symposium Transactions, 1956, pp. 110-113.

Section 2.4

FLOW MEASUREMENT PROCEDURE

2.4.1. PRINCIPLES OF PROCEDURE

The flow measurement procedure consists of determining the extent of leakage by measurement of flow of gas moving into or out of the system. This flow measurement may be by means of a flowmeter, or by means of the effect of pressure at a constant pumping speed.

2.4.1.1. CHARACTERISTICS

The flow measurement procedure can be roughly separated into two techniques: flow observation and pumping-pressure analysis.

In the flow observation technique the amount of leakage is measured directly. The system is pressurized (or evacuated), placed in a sealed volume, and the gas transfer between the sealed volume and the system is measured. Alternatively, the sealed system is evacuated, allowed to reach equilibrium, and the amount of gas being pumped at equilibrium measured.

In the pumping technique, the system is evacuated; the system pressure decrease is then compared with pressure expected during pumpdown of a leak-tight system. Alternatively, the system is pressurized and the compressor operated only to keep the system pressure constant. The leak rate may then be calculated from the length of time the compressor is operative.

2.4.1.2. APPLICABILITY

Flow measurement procedures are applicable on a large variety of systems. These procedures are useful only for leakage measurement. The procedures are used to measure total leakage in small sealed parts, in large sealed systems, and in systems which may be evacuated or pressurized. However, they are limited to those systems where leakage measurement need not be highly sensitive. One major advantage is that no particular tracer gas is necessary.

2.4.1.3. SENSITIVITY

The sensitivity of these procedures is relatively low. In most cases, the sensitivity is dependent on the instrument measuring flow and independent of system volume.

In a flow observation technique, a leakage of between 10^{-2} and 10^{-4} atm cc/sec may be detected, depending on the instrument used. If a sealed system is being evacuated, a flow of only about one atm cc/sec may be detected.

In the pumping-pressure analysis technique, the sensitivity is dependent on the size of the pumps; in the case of evacuation, it is dependent on the outgassing of the system being measured. These limitations are discussed more thoroughly in Sections 2.4.4.1. and 2.4.4.2. which describe the individual techniques.

2.4.2. EQUIPMENT AVAILABLE

The only commercial leak testing equipment for this procedure is that for use in the flow observation technique and for flow measurement. It is described in Table 16.

2.4.3. PROCEDURE ADVANTAGES AND LIMITATIONS

The major advantages of the flow measurement procedure are:

1. No special tracer gas is necessary. The procedure is applicable to whatever fluid is present in the system. The system need not be placed under any special environment, but may be tested in its normal operating mode.

2. The cost of the equipment is low.

3. The sensitivity is independent of system volume.

4. Although the accuracy is not very high, the leak rate may be measured without extensive calibration. Wherever calibration is required, it may be readily obtained with standard flow or volume measurement equipment.

The major disadvantages of this procedure are:

1. Sensitivity is quite low.

2. It consists of not one, but a series of techniques developed to solve individual problems. For this reason, this procedure has not gained wide recognition.

2.4.4. TESTING

Since the flow measurement procedure consists of a series of techniques with little similarity of equipment, the individual techniques are discussed separately.

2.4.4.1. FLOW OBSERVATION TECHNIQUE

2.4.4.1.1. Sealed Volume

In the sealed volume technique, the most common method of flow measurement is the observation of movement of fluid in a glass capillary. Figure 65 illustrates this method. The system is sealed into the testing volume. A capillary containing an indicating fluid is attached to the testing volume. This technique is a modification of the isobaric leak calibration method

Table 16

COMMERCIAL FLOW MEASUREMENT DETECTORS*

General Characteristics	Average Values	
Principle	Volume displacement	Flow observation
Sensitivity	1 x 10^{-5} atm cc/sec	1 x 10^{-2} atm cc/sec
Tracer Gas	Non-specific	Compressed air
Output Signal	Visual observation	Visual observation
Power Requirement	None	None
Size	Width= 3 inches, Height= 10 inches Depth= 6 inches	10 inch diameter; height= 30 inches
Weight	5 pounds	20 pounds
Price	$ 350	$ 99
Manufacturer	Volumetrics	Universal Control Corporation

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* More specific information on this equipment may be obtained from General Electric Company Report S-67-1013 (Ref. 186)

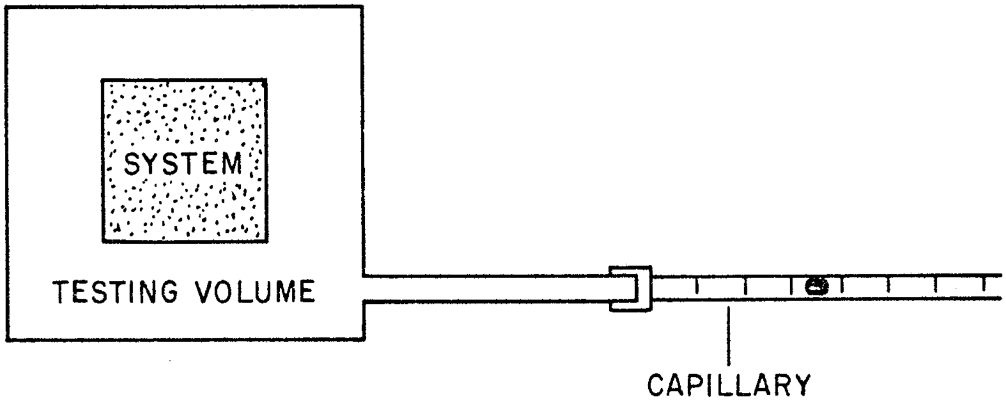

Figure 65. Flow Observation Sealed Volume Leakage Measurement.

described in Section 1.5.

For accurate readings and rapid response, the test volume should be as small as practical.

Glass capillary tubes provide a direct quantitative measure of the leakage rate if the time required for a small liquid plug to move a given distance is recorded. Since the cross-sectional area of the capillary bore is known, the volume swept out by the liquid plug during this time can be computed. The 1.5-mm glass capillary is used to measure leakage rates from 10^{-2} to 1 atm cc/sec. The 0.5-mm glass capillary can be used for measuring smaller leakage rates from 10^{-4} to 10^{-2}. The capillaries are scaled off in convenient units for computing leakage rates and a stop watch is used for timing the plug. Pipettes used for liquid measurement are convenient calibrated capillaries.

The upper limit on leakage rate measurement occurs when the liquid plug moves so fast that timing is difficult. The lower limit on leakage rate measurement is determined by the accuracy desired and errors introduced by the resistive and inertial forces affecting the movement of the plug. As the rate of liquid plug movement decreases, these errors increase, causing the leakage rate measurements to become more inaccurate.

The errors due to starting inertia are minimized by using a plug that wets the capillary surface, using a water plug about 1 mm long, and timing the liquid plug movement only after it reaches a constant velocity. If water is used, the error due to the resistive forces of surface tension can be minimized by coating the inside of the clean capillary tubing with an organosilicon compound. This effectively prevents the water from wetting the glass.

This technique is described in detail in literature (Ref. 107). The system being tested may be either evacuated or pressurized, and may be either sealed or connected to a pressure (vacuum) source. Of course care must be taken that the leakage being tested is not in the pressure connection.

This type of testing may be performed against a standard to compensate for temperature variations. In such a case, a second testing volume is placed on the other side of the capillary.

One advantage in the construction of this type of equipment is that there are no critical, leak-tight connections in the test volume. This is because the system is operating at atmospheric pressure. Therefore, although it is possible that a leak might exist between the testing volume and the atmosphere, leakage does not occur because there is no pressure differential, due to compensation by the liquid slug.

The basic principle described above can be employed in numerous ways. For large leaks, a flow measuring device such as a wet type gas meter or a rotameter may be used. These instruments produce accurate results but are useful only on very large leaks. For measurement over a wide range of leaks, a "Delta-Vee Meter" (Ref. 108) can be employed. This instrument is schematically illustrated in Figure 66. Columns A, B, and C form a U-Tube capable of withstanding extremely high pressures. Tubes B and C are of different diameter so that various leakage rates may be measured by selecting the proper tube. When all of the valves are open and the test component is pressurized, the separation level remains stationary. By closing the valve in the main line between Columns A and B, leakage is indicated by the fluid moving up in Columns B or C.

The "Delta-Vee Meter" was primarily designed for determining leakage rates in hydraulic power systems. The principle is to displace the leaking fluid with the indicating fluid. This can be done because there is a pressure loss in the leaking component. When installed in the power line to the component being tested, leakage can be measured by the displacement of the separation level.

A meter developed by Convair Instruments (Ref. 109) gives automatic and accurate leakage measurement. The meter is installed in the line between a test item and a source of high pressure fluid, such as an accumulator which replenishes make-up fluid to the component. Flow in the line between meter and component represents leak flow; it is measured by the displacement of a bellows whose deflection is sensed by a potentiometer, and recorded. This system replaces a liquid slug and visual observation. Leakage is read directly in cubic centimeters. A similar meter is now manufactured by Volumetrics.

It should be mentioned that the above instruments will work with liquids as well as gases, providing that the indicating slug is immiscible in the fluid. This makes the above techniques extremely useful for testing under operational conditions.

This technique can be used to leak test pipelines and other large volume units (Ref. 110). The line to be checked is connected to a pressure regulator and a bubbler. The line is plugged off at its end, and air pressure is passed through the bubbler to fill the line. If the line is tight, bubbling will cease; if it is not, there will be no cessation of bubbling.

Cincinnati Gas and Electric Company has developed an indicator for localizing gas leaks in gas-filled cables (Ref. 111). The instrument is essentially a U-tube manometer with appropriate valving. With the manometer installed in a segment of the line and the line pressurized, oil will rise in the glass tube toward the leak.

2.4.4.1.2. Pumping-Pressure Analysis Technique

If the system can be evacuated, leakage may be measured directly by means of a flow meter. The system is evacuated

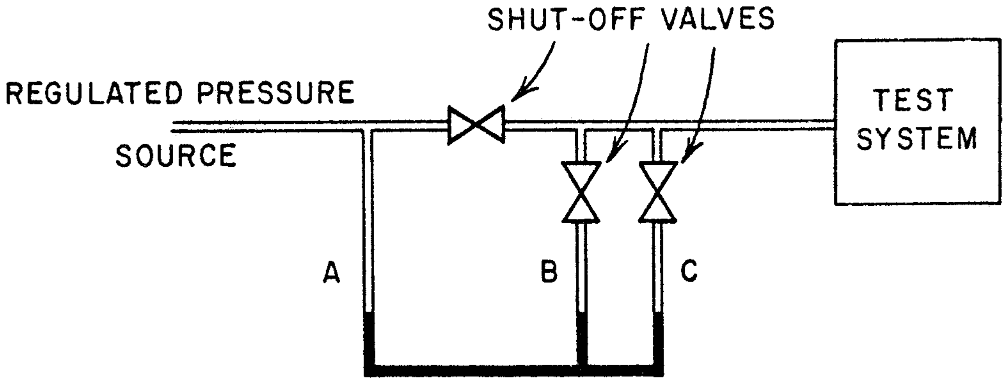

Figure 66. Delta-Vee Meter for Leakage Measurement. (Reprinted with permission from George C. Marshall Space Flight Center Conference on Leak-Tight Separable Fluid Connectors, Huntsville, Alabama, March 1964; L.E. Grimes, "Laboratory Leakage Measurement Techniques On Separable Connectors".)

through an isolation valve, the exhaust of the pump going through a surge tank to a flow meter. There is a bypass valve (bypassing the pump) between the isolation valve and the surge tank (Figure 67).

In performing the test, the isolation valve is first shut and the pumping system tightness is determined by measurement of flow through the flow meter. The isolation valve is then opened and the flow readings taken after an equilibrium has been achieved. The pressure in the system during test is adjusted by means of the bypass valve. The lower limit of pressure for which the pumping technique is useful is roughly 25 Torr (Ref. 6). The lower limit of sensitivity is approximately 1 atm cc/sec, and is mainly dependent on the availability of suitable flow meters.

2.4.4.2. PUMPING TECHNIQUES

2.4.4.2.1. Pressurizing Technique

Leakage measurements are described in literature (Ref. 100) by evaluating the intermittent-operation factor of the load cycle according to which a compressor must be operated, in order to maintain a specified pressure in the system. If it is desired to measure the absolute value of leakage by this means, the capacity of the compressor used should be accurately known or determined in a separate calibration test.

For the pressurizing technique, two stopwatches and a good pressure gage with a rapid response and a clear scale graduation are required. For the measurement, the compressor is allowed to charge the system up to its normal delivery pressure. Then, the compressor control valve is shut off and the compressor is left under no load, until the pressure in the vessel falls off to a suitable value below the initial delivery pressure. When this limit is reached, one stopwatch is started, and the compressor is put under load by manual operation of the control valve. The first watch is stopped when the pressure has risen to a predetermined value. The second watch is then started and the compressor output is cut off. When the pressure falls off by the original value, the compressor is again put under load, the first stopwatch is started again, and the other is stopped. In this way, a new cycle is initiated and alternate readings are taken. The times are noted for each partial cycle related to the load and no-load condition, respectively.

Four or five cycles are carried out in succession so as to be sure that the compressor is operating under constant running conditions and to obtain accuracy.

The method assumes that no gas is supplied to the system during the no-load period. When the load is removed from a reciprocating compressor by so-called "dead-space regulation," it is not uncommon for the compressor to continue to deliver some gas during the no-load period. To avoid this, therefore, it is

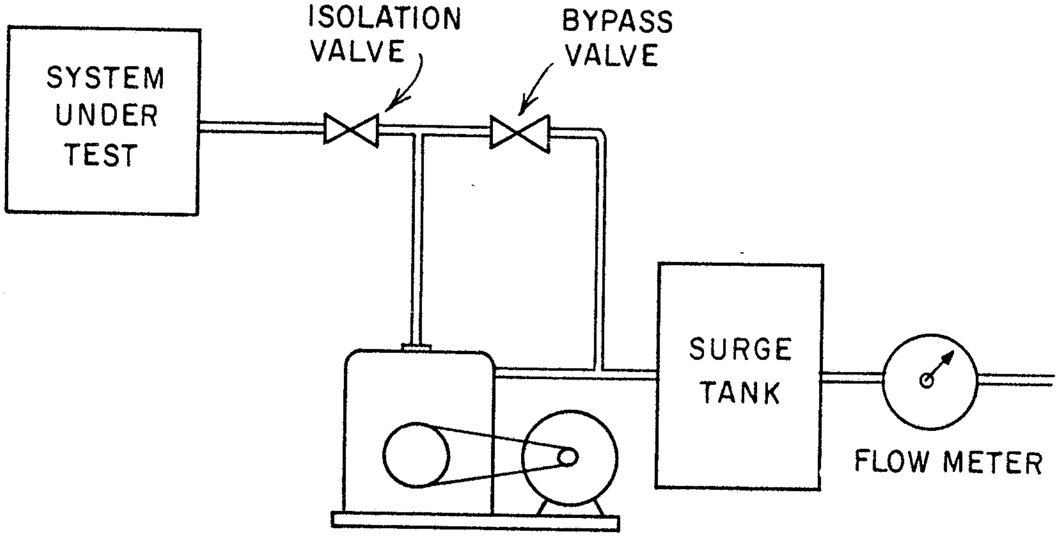

Figure 67. Pumping Technique of Leakage Measurement.

necessary to ensure that the compressor is provided with a delivery system which enables all the gas in the intercooler to be discharged into the atmosphere, during the no-load period.

This method has the advantage of requiring only very simple equipment. Its accuracy is less than that of more direct methods and it is subject to random errors, caused, for instance, by defective compressor valves. The valves should therefore be checked for satisfactory operation before starting each test. The method does not indicate the volume of the system, nor does it provide leak location.

If several compressors are available, the compressor selected for the tests should be one which, if possible, gives charging times at least as long as the leakage times. It is not advisable to operate the compressor under part-load conditions, since its delivery capacity is rarely determined with the same accuracy for lower loads as for full load. Compressors with dead-space regulation have a part-load capacity which may differ in a compressor calibration test, and in a system leakage test, if the quantity and temperature of the cooling water are different in the two cases.

2.4.4.2.2. Evacuation Technique

The evacuation technique consists of evaluation of a pressure-time response of a vacuum vessel in which the system is being tested.

To fully understand this technique, a discussion of equations of pumping is necessary. The fundamental response of a system to pumping is:

$$\frac{dP}{dt} = -\frac{SP}{V} + \frac{Q}{V} \qquad \text{Equation 2.4-1}$$

where P = pressure in the system
t = time
S = pumping speed of the pump
V = system volume
Q = gas load

The gas load may be due to leakage, evolution of gas from the walls of the system, or both. In the region where the gas load is negligible, the equation reduces to

$$\frac{dP}{dt} \doteq \frac{-SP}{V} \qquad \text{Equation 2.4-2}$$

which is integrated to

$$t_2 - t_1 = -\frac{V}{S} \ln\frac{P_2}{P_1} \qquad \text{Equation 2.4-3}$$

If the logarithm of the pressure is plotted against time, a straight line is obtained with a slope of $-\frac{S}{V}$. As outgassing (or leakage) begins to contribute to the pressure, Equation 2.4-3 is no longer valid and the integration of Equation 2.4-1 takes the form

$$t_2 - t_1 = -\frac{V}{S} \ln \frac{1 - \frac{P_2 S}{Q}}{1 - \frac{P_1 S}{Q}} \qquad \text{Equation 2.4-4}$$

This is no longer a straight line when plotted in the above manner, but a curve asymptotic to a pressure which is obtained by the relation

$$P = \frac{Q}{S} \qquad \text{Equation 2.4-5}$$

This pressure, P, is approached when the time is approximately five times the pumping speed, divided by the volume. (A more complete discussion of the above equations is presented in Section 1.3-4.)

Leakage measurement may be performed in one of two ways:

1. Pump the system down to equilibrium pressure (Equation 2.4-5), measure outgassing and leakage, and subtract from it the value of outgassing measured in a leak-free system.

2. Derive an allowable pressure-time curve for the pump-down of a system. Systems deviating from this relationship will be considered leakers.

With either type of system, it is possible to set up an automated leak test station. Such a station is described in literature (Ref. 106). A carrousel moves the samples into position, pumps them down, and measures the pressure. The biggest difficulty with this type of test is the false reading produced by outgassing of dirty samples.

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Section 2.5

BUBBLE TESTING

2.5.1. PRINCIPLES OF PROCEDURE

In the bubble testing procedure, a liquid is placed in contact with the outside of the system. A pressure differential is created between the inside of the system and its surroundings. Leakage can then be detected by observation of bubbles formed at the point of leakage.

2.5.1.1. CHARACTERISTICS

Bubble testing can be subdivided into two types: the liquid immersion technique and the liquid application technique. Bubble testing can be used for both leakage measurement and leakage location. The liquid immersion technique can serve both functions; the liquid application technique serves only to locate leaks.

2.5.1.1.1. Liquid Immersion Technique

For leakage measurement the immersion technique should be used. This technique consists of pressurizing the system or component with a gas while the component is immersed in a liquid. The source of the leak is indicated by the bubbles of gas that are formed when the gas under pressure emerges from the leak into the surrounding liquid. Provided that the apparatus has been designed so as to avoid concealed or trapped leaks, the appearance of a bubble gives an immediate indication of the opening through which the gas passes, and thus locates the source of leakage. Therefore, it can be seen that this procedure also serves to locate the leak as well as to determine that one exists.

A number of different methods may be employed to establish pressure differential across the suspected leak. Whenever convenient, the system should be pressurized with gas. However, should this prove impossible, an alternative procedure is to draw a vacuum over the surface of the liquid. A third alternative is to heat the liquid, creating a pressure differential by thermal expansion.

2.5.1.1.2. Liquid Application Technique

This technique is only useful for leak location. It consists of applying a liquid which has a tendency to form bubbles on the pressurized area suspected of leakage. The major advantage of this procedure is that it can be applied rapidly and cheaply with a minimum of cost and effort.

2.5.1.2. APPLICABILITY

In general, this procedure is useful for measuring and locating relatively large ($> 10^{-4}$ atm cc/sec) leaks. It is often used on large, pressurized systems. It is of singular importance as the only method readily available for the measurement of large leaks from small, sealed components, It is therefore used as a final check for gross leakage in the manufacture of transistors, relays, and small ordnance devices.

2.5.1.3. SENSITIVITY

The maximum sensitivity of bubble testing is in the range of 10^{-4} to 10^{-5} atm cc/sec. The sensitivity is hard to define because it is dependent on the observation and alertness of the operator.

There is no question that a 10^{-5} atm cc/sec leak can be observed. However, it is a different matter when the operator does not know that a leak exists, and has to examine a long, welded seam for a possible bubble. Conceivable, he will not wait long enough or look under every seam.

Secondly, there is a minimum pressure required to form a bubble in a liquid. Therefore, bubble testing sensitivity is dependent on the pressure across the leak. This is the only method which is an exception to the statement in Section 1.3.3. In bubble testing, the sensitivity increases with an increase of pressure across a leak.

Finally, it can be estimated that a certain size leak is being seen because a bubble of particular volume is being observed. However, this can be inaccurate on very small leaks, because of the finite solubility of the tracer gas in the test liquid. It is theoretically possible for a small leak to exist where the tracer gas from the capillary dissolves in the test liquid, and yet no indication of leakage will be visible.

2.5.2. EQUIPMENT AVAILABLE

The equipment currently available for bubble testing consists of fluids (Table 17) made by various manufacturers for the liquid application procedure and surface tension modifiers for the liquid immersion procedure.

2.5.3. PROCEDURE ADVANTAGES AND LIMITATIONS

Some of the advantages of bubble testing are:

1. Bubble methods will locate a leak very accurately.
2. Little or no skill is required to apply the technique.
3. The cost of equipment and effort is minimal.
4. The technique is safe in a combustible atmosphere.
5. The technique is satisfactory for gross leakage.

Table 17

COMMERCIAL LIQUID APPLICATION FLUIDS FOR BUBBLE TESTING*

General Characteristics	Average Values
Sensitivity	1 x 10^{-4} atm cc/sec
Tracer Gas	Non-specific, any compressed gas
Container Size	4 ounces to 55 gallon drum
Price	70 cents/4 ounces to $5/gallon

Manufacturers

American Gas and Chemical, Incorporated (Leak-Tec Division)
Cargille Scientific, Incorporated
Excelsior Varnish, Incorporated
Flamort Chemical Company
Highside Chemicals, Incorporated
Kelite Corporation
Nuclear Products Company (NUPRO Company)
Watsco, Incorporated
Winton Products Company, Incorporated

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* More specific information on these fluids can be obtained from General Electric Company Report S-67-1013 (Ref. 186)

6. Bubble methods are very rapid even for small leaks, whereas other, more sensitive techniques have a response so slow that the leak may be missed while probing. Even when bubble methods are slow, it is not necessary to move on to another point - the whole unit is examined simultaneously and patience only is needed to observe the bubbles.

7. All leaks are independent. Large leaks do not have to be found on coarse apparatus and sealed before refined apparatus can be used. The bubble testing method distinguishes real from virtual leaks, the first, but often difficult task on dynamic vacuum systems. Moreover, connection pipes and valves do not have to be leak-free.

Some of the more significant limitations of bubble testing include:

1. Small ($< 10^{-5}$ atm cc/sec) leaks can be clogged by the liquid. Most testing solutions have a low surface tension. These solutions promote surface wetting; the test liquid is thereby encouraged to enter and block very small leaks. This tendency may be reduced, however, if the vessel is always pressurized with gas before covering the area under test with any liquid.

2. Porosity leaks cannot be detected if the pores are very small.

3. Some leaks are alleged to pass gas in one direction only, and if this is inwards, the bubble method will not detect them.

4. Cleaning after use is usually required.

5. The system being tested must be dried.

6. Some leaks are intermittent; consequently, close surveillance of the surface is often necessary to detect bubbles when they do occur.

Specific disadvantages of the liquid immersion procedure using heat or evacuation are essentially three:

1. When the bubble tests are conducted on metallic vessels, some bubbles evolve on outgassing from patches of corrosion.

2. Leak location can be hindered by spurious bubble formation from air dissolved in the water. Furthermore, if enough air is evacuated, the water will boil, and vapor bubbles will form on

the surface of the unit being tested.

3. If there is a leak, the test liquid will enter the unit after the vacuum is released, causing difficulties if this leaking unit should slip past the inspection.

2.5.4. PRINCIPLES OF OPERATION

The process of forming bubbles that result from gas flow through a given leak into a liquid is dependent not only on the pressure conditions but also on the physical properties of the liquid and of the gas. Thus, by a suitable combination of liquid and gas, the sizes of the bubbles and their rate of formation can be modified. For purposes of leak detection and location it is desirable that the bubbles should be readily apparent. The sensitivity of the procedure is determined by the ability to observe bubbles formed at the outlet end of small holes and passages which may set up a high resistance to the formation of bubbles and to the passage of gas into them. The more readily the bubbles are evolved, the more easily they are observed. This condition is an important requirement to keep in mind when choosing the particular combination of gas and liquid to be used.

It is possible to change the sensitivity of the test by changing the test gas. The rated leakage of the test gas may thereby be increased; this does not imply a change of the conductance of the leak. This has been discussed in Section 1.3.2.

When the liquid does not readily wet the solid surface around the orifice, the bubble rim tends to spread away from the orifice resulting in larger bubbles. Both methyl and ethylalcohol tend to wet most solids more readily than water. Consequently, actual bubble sizes in the former liquids are in better conformity with the theory developed here which assumes complete wetting. Larger bubbles are also formed in the presence of any conditions which tend to promote partial wetting, such as traces of grease. For a given gas flow rate, the production of larger bubbles reduces the bubble frequency, the frequency being inversely proportional to the cube of the bubble radius. As a result, for a given leak, the bubble frequency in organic liquids may be 100 times the frequency of bubble formation in water.

The mathematical theory of bubble testing has been discussed in literature (Ref. 112 and 113). Bubbles are emitted from a leak immersed in a liquid when the internal gas pressure exceeds the sum of the hydrostatic head and the maximum surface tension restraint. For the case of a cylindrical hole:

$$P = P_a + \rho g h + \frac{2\sigma}{R} \qquad \text{Equation 2.5-1}$$

where P = bubble pressure
P_a = pressure above the liquid
ρ = liquid density
R = bubble radius

g = gravity
h = depth of liquid immersion
r = radius of capillary
σ = surface tension of liquid

As the gas flows through the leak, each bubble forms and expands, ultimately being attached to the rim of the leak by a neck. This is illustrated in Figure 68.

Assuming that the bubble formed at the end of a tube is part of a sphere, then as the bubble is being generated, its radius r_R first decreases, as indicated by Figure 68 (a), until it reaches its minimum value r corresponding to the radius of the capillary as shown in Figure 68 (b). Thereafter the radius R_B increases to form the expanding bubble shown in Figure 68 (c). This variation implies that the term $2\sigma/R$ in Equation 2.5-1 has a maximum value $2\sigma/r$, this corresponding to a maximum value of excess pressure. When the bubble buoyancy exceeds the surface tension restraint at the neck, the bubble is detached and rises to the surface.

In the case of a cylindrical hole the condition for bubble detachment is given by

$$\frac{4}{3} R^3 \rho g = 2r\sigma \qquad \text{Equation 2.5-2}$$

where R is the bubble radius at this stage.

The above two equations present almost the total picture of bubble formation and growth. In more rigorous equations, viscosity affects the bubble size; however, this is considered negligible (Ref. 113) in most leaks. With an increase in viscosity there will be some increase (small) in bubble size (Ref.113).

The external pressure may be regarded as constant; therefore, there is a maximum value of the pressure inside the incipient bubble which must be reached if the bubble is to expand beyond the hemispherical stage. As this condition imposes a limit on the application of bubble testing, its effect will now be briefly examined. If, for example, the end of the capillary is submerged in water at atmospheric pressure and the capillary is connected to a gas at a pressure of 14.7 psig*, (2.02×10^6 dyne/cm^2) and σ = 73 dyne/cm

then $r = \frac{2 \times 73}{1.01 \times 10^6} = 1.45 \times 10^{-4}$ cm, which represents the smallest capillary radius that it would be possible to detect by means of a bubble with the given pressure difference.

If, however, a liquid of lower surface tension were used, such as methyl or ethyl alcohol, then the same excess pressure would allow a bubble to be formed at the end of a capillary of

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*1 atm = 1.01×10^6 dyne/cm^2.

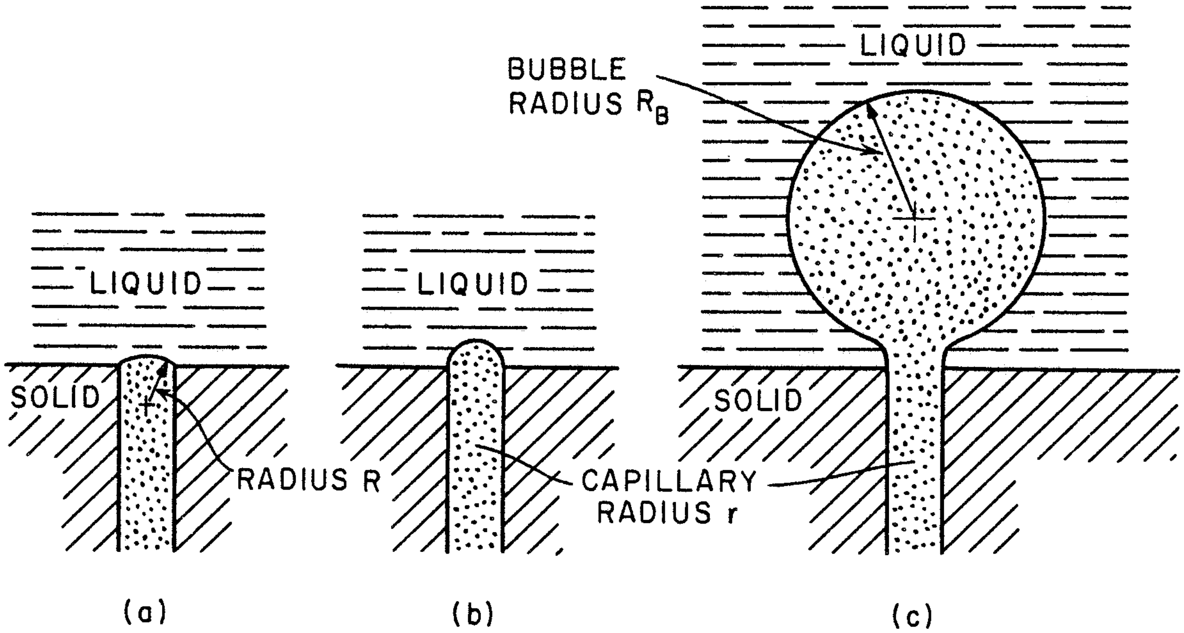

Figure 68. Bubble Formation at a Leak Site.

radius $\frac{2 \times 23}{1.01 \times 10^6} = 4.5 \times 10^{-5}$cm. This radius is less than 1/3 of the least radius of a capillary detectable when using water. Moreover, Biram and Burrows (Ref. 112) state that gas flow through the capillary is proportional to the capillary radius raised to a power of between 3 and 4, dependent on the kind of flow. The use of a liquid having lower surface tension should therefore reveal leak rates having values in the range 1/50 to 1/100 of the lowest rate detectable when the bubbles are formed in water. The foregoing theory indicates that the sensitivity of the method may be appreciably increased by using liquids of low surface tension.

Another factor which affects the sensitivity of bubble testing is the size of the bubbles evolved. It may be seen by an examination of Equation 2.5-2 that the size of the bubble increases with increase of surface tension. Therefore, for generation of a large number of bubbles, liquids of small surface tension are desired.

Bubble size can also depend on degree of vibration. If the test object is vibrated, the bubble breaks off before it would when the test piece is stationary. This can be useful, as it increases the bubble rate for a given leak rate.

If the pressure over the liquid is reduced below atmospheric until bubbles just emerge from the end of the leakage path, limitations are imposed by the tendency of a liquid to degas and boil under conditions of reduced pressure. A comparatively high boiling-point enables reasonably low pressures to be realized without boiling. To enable the smaller leaks to be detected, it is desirable to use liquids having low values of surface tension; but such liquids also have correspondingly lower boiling points. These liquids may boil spontaneously before the pressure over the liquid could be sufficiently reduced. Therefore, the choice of liquid for these tests must be very carefully made.

In summary, it is desirable to use a liquid of low surface tension and low viscosity. Pressure has to be higher with capillaries of small diameter. Finally, the use of a low viscosity, low molecular weight gas will increase the flow rate through the capillary.

2.5.5. METHODS OF TESTING

It is necessary to create a pressure differential between the inside of a component and its surroundings if a bubble test is to be used. One method used in bubble testing is a high-pressure line connected to the component. This method has one advantage, in that it is possible to create as large a pressure differential as desired. Gases discussed in the previous section may be used for pressurizing. Compressed air can be substituted, provided it is obtained from a cylinder; compressed air lines are not recommended because dirt and oil can block small leaks temporarily.

Pressure should be applied to the test unit before liquid application or immersion so that fluid will not enter small leaks; much higher pressure is needed to detect a leak once it has been clogged.

2.5.5.1. LIQUID IMMERSION TECHNIQUE

Pressure differential may be created in various ways (Ref. 115). One suitable method is to place the component in a container and evacuate the container. This method can give pressure differentials up to one atmosphere if it is assumed that the internal pressure is atmospheric. A second method is to preheat the bath in which the component is to be placed. As the temperature within the component rises, the air inside will tend to expand and the pressure will rise. The pressure differential created in this manner will be in the range of a few pounds per square inch, as shown in Figure 69. Another method of creating the pressure differential is to connect a high-pressure line to a tube that enters the component. Although there are other ways of creating a pressure differential, the three mentioned are those most commonly used in bubble testing.

With small components, from the standpoint of economy and rapidity of testing, the best method of creating the pressure differential necessary to perform a bubble test is by preheating the bath and immersing the component to be tested into it. Once the bath reaches the desired temperature, no further adjustments are necessary, except those minor ones that may be required to maintain a constant temperature. If the bath is large enough, samples to be tested may be mounted on a rack and all checked at the same time. This method is conducive to the testing of mass produced items such as resistors.

Although a test for which a vacuum must be created is more expensive to run from the standpoint of time and necessary equipment, the results are superior to those obtained with a heated bath. Caution must be exercised in the performance of a vacuum-type test to insure that the pressure above the bath does not fall too low, causing the bath to boil. When this happens, streams of bubbles rise to the surface and give false indications that the component is leaking. Most electric component manufacturers use the vacuum method or the heated bath when conducting their bubble leak test.

The third method (i.e., high-pressure line connection) is useful only for larger pieces of equipment.

The evacuated chamber test is more sensitive than the heated oil type because a pressure differential of almost 1 atmosphere exists in the former test while a pressure differential of approximately 6.2 psi exists in the heated-oil test (Figure 69). The principal advantage that the heated-oil test has over the oil under vacuum is the fact that the heated-bath type of test is simpler to perform than the vacuum-type test.

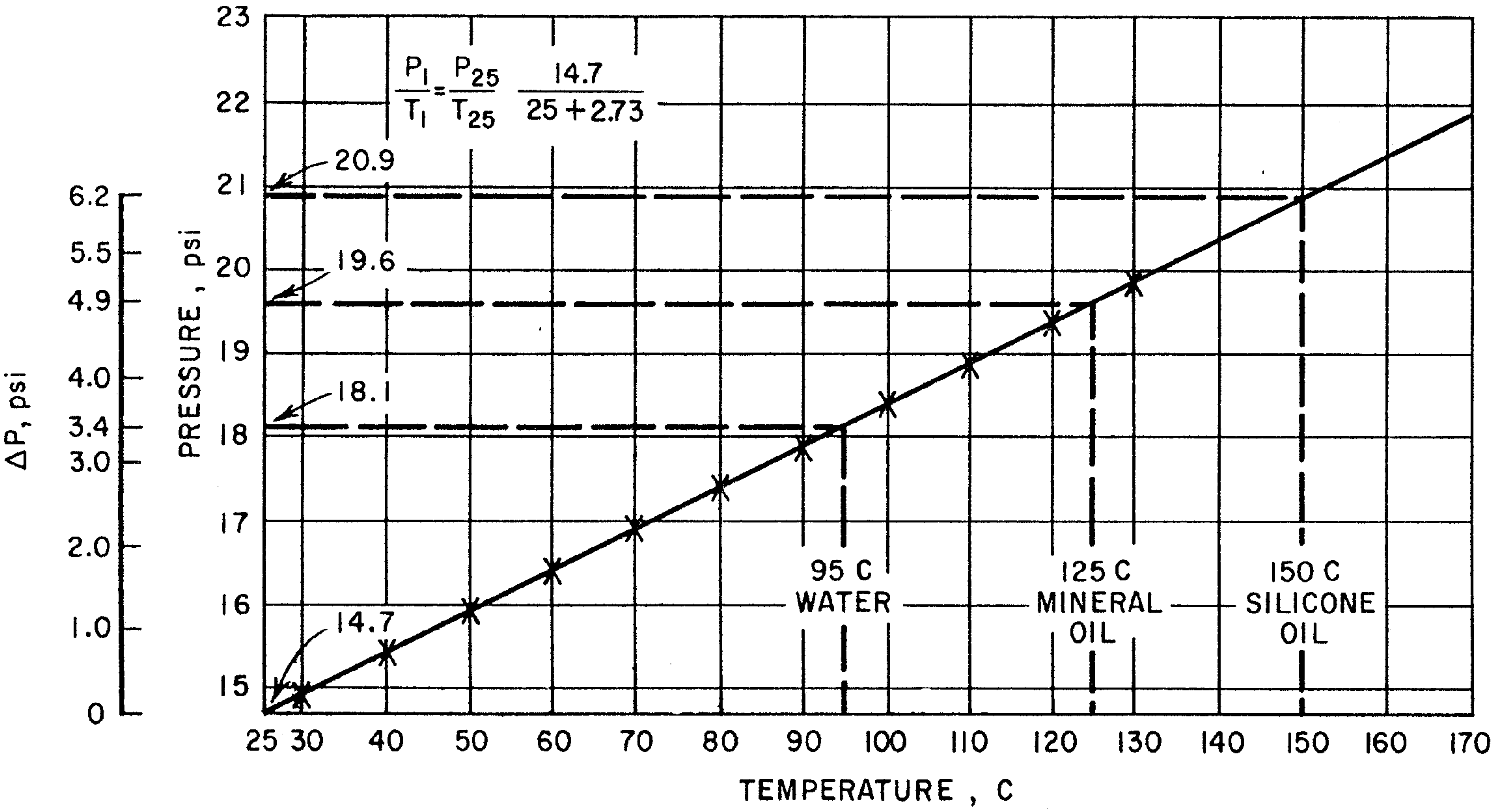

Figure 69. Pressure Versus Temperature for a Fixed Volume. (Reprinted with permission of U. S. Army Signal Equipment Support Agency, from Final Report on Task 511--Leak Detection by H. F. Scheffler, Battelle Memorial Institute; Contract DA-36-039-SC-73212, February 28, 1959.)

Water, mineral oil, or a silicone oil may be used in these tests. If water is used, it must be treated to reduce the surface tension. This reduces the bubble size and the tendency of bubbles to cling to the surface. A steady stream of extremely fine bubbles appears in an oil bath. Bubbles in a water bath cling to the surface of the component and build up to a relatively large size before breaking loose and rising to the surface. This means that in water small leaks would require a long period of time to produce a bubble that would be visible on the surface; a component would most probably be passed as acceptably sealed before time was allowed for a conclusive test.

A disadvantage common to tests using oil is the fact that the components, after being tested, must be degreased to remove the oil that adheres to the surfaces. Silicone oil bath is particularly expensive to use, because the oil clings to the component after immersion and cannot be completely recovered. Although the degreasing is an added expense in the test procedure, it is worthwhile because of the better leak sensitivity obtainable with an oil bath.

The side of the object being examined must be positioned in the test fluid so that the bubbles may rise to the surface. Obviously, the bottom side of any object being tested (i.e. the side facing the bottom of the test container) will tend to trap the bubbles, decreasing the reliability of the test, unless it is felt that the bottom surface cannot leak (e.g., it has no seams).

If the leak is small, the bubbles may be difficult to see until the eye is adapted. A reading glass will be found of great assistance (e.g. 3 inch diameter giving a magnification of 2 or 3 times at 4 to 5 inches from the object). There is a minimum size of bubble, so the glass does not introduce a new strain by revealing smaller bubbles. Good lighting is, however, essential and a dark background may be helpful. A small stream of bubbles may be more easily detectable from above than from the side.

If large vessels have to be tested, immersion may be impossible but channels can often be built round suspected areas to contain the test fluid.

Methyl, ethyl, or isopropyl alcohol may be used as a test fluid. The one advantage of using alcohol as a bath that is not found with any of the other fluids is its cleaning properties. Not only is the degreasing process eliminated, but also the alcohol cleans foreign matter introduced by production processes from the body of the component. After the component is removed from the bath, no alcohol remains on the body, because of the rapid evaporation rate of the alcohol.

Alcohol cannot be used in the heated bath technique, because of its low boiling point. In addition, the rapid evaporation of alcohol can also be a detriment in the evacuated chamber method.

The amount of fluid in the bath tends to decrease rather rapidly, and repeated replacement is necessary. A bath that can accomodate a tray with numerous components on it would have a large area exposed to the atmosphere, and the evaporation loss would be great.

Aside from being expensive to use because of loss by evaporation, there is a safety factor that must be considered in the use of the alcohols. Methyl alcohol could be extremely harmful to operating personnel if it were to get into the body or eyes. Severe poisoning or damage to vision can result to the operator or anyone in the vicinity unless adequate ventilation is provided to remove harmful vapors. These fluids are flammable so a fire hazard exists in their use.

The liquid immersion technique can be applied to a continuous manufacturing process. For example, as the component parts move along the production line toward final assembly, they can be pressurized with dry air and then immersed in clean, hot water baths. The water should be treated with a suitable softener to reduce surface tension and promote bubble growth, thus increasing the sensitivity of the test. Operators in front of large glass windows can detect leaks. These leaks can be tagged and the unit returned for repairs and recycling through the dip tank. One of the outstanding limitations of this test is the amount of entrained air carried into the tank on the surface of these units.

2.5.5.2. LIQUID APPLICATION TECHNIQUE

This relatively simple procedure consists of pressurizing the system, applying a liquid, and observing bubble formation which indicates the presence of a leak. A bubble-free solution should be applied gently to preclude bubble formation in application. The solution should be flowed rather than sprayed or brushed onto the surface. The sensitivity of this technique is dependent on the time and care taken to observe bubble formation.

Two cautions are in order. When testing flanges, threads, or any joint which has a large exposure area, it is absolutely necessary that the soap solution bridge the entire joint. Gas will invariably slip out through the smallest pinhole which is not covered. The second caution is in the choice of a soap solution. For high sensitivity it is necessary that the film does not break away from the joint and that the bubbles formed do not break due to air drying or weak surface tension.

The system or section being tested may be pressurized in numerous ways. Considerable ingenuity may have to be exercised in making up special clamps and fittings for sealing the component and attaching the air (or gas) hose. The use of rubber sheets and C-clamps should not be ignored.

Numerous commercial leak testing solutions listed in Table 17 can be used. A suitable soap solution for leak indication consists of equal parts of corn syrup, liquid detergent, and glycerin (Ref. 4). The solution should not be prepared more than 24 hours preceding the test and bubble formation properties should be checked with a sample leak every half hour during the test.

In another approach, Guthrie and Wakerling (Ref. 116) consider the rather intriguing idea, applicable only to very large chambers. A volunteer, equipped with an oxygen mask is requested to enter the chamber, which is pumped out to about 3/4 atmosphere. The man can then apply soap solution to the inside surface of the system and look for the formation of soap bubbles. Needless to say, the most careful precautions must be taken during this procedure. The man inside the vacuum system should be under observation at all times so that any signs of exhaustion or other difficulties will immediately be apparent. A continuous and uninterrupted source of oxygen must always be available. Also every precaution must be taken so that there is no chance for the valve between the vacuum system and the pumps to be opened wide by mistake. The real value of the method lies in finding large leaks in parts of the surface of a large vacuum system that are not accessible from the outside. The method obviously has quite limited possibilities, being applicable only in large vacuum systems.

In estimating leakage rate, the size and appearance of the bubbles are critical. For example, DeCastra and Wells (Ref. 117) establish three classes of bubble dimension and appearance. Class 1 appears as small, uniform, long persisting bubbles. This class implies a leakage range of 10^{-4} to 10^{-1} atm cc/sec. Class 2 appears as a mixture of random size bubbles that are moderately persistent. This class implies a leakage range of 10^{-1} to 1 atm cc/sec. Large, fast forming bubbles of short persistence constitute Class 3. The implied leakage range is between 1 and 10 atm cc/sec.

2.5.5.3. MILITARY SPECIFICATIONS

Leak testing by the bubble method is mentioned in several military specifications. The leak test compound for oxygen systems is described in MIL-L-25567A(ASG). Instructions for testing components are given in MIL-STD-202B, METHOD 112, and in the Proposed Standard for Leakage Rate Testing of Containment Structures of Nuclear Reactors (Ref. 4).

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Section 2.6

RADIOISOTOPE PROCEDURES

2.6.1. RADIFLO TECHNIQUE

2.6.1.1. PRINCIPLES OF TECHNIQUE

The radioisotope procedure consists of diverting a fluid stream containing radioisotopes through a leak and then using radioactive measurement techniques to determine the presence and location of the leak.

The radioisotope procedure may be used to measure leakage in small sealed containers (transistors, relays, etc.). This method is known as the Radiflo technique. In addition, the radioisotope procedure can be used to locate leaks in very large systems (cables, pipelines, etc.).

The radioisotope procedure is subdivided into one standardized method of leakage measurement (Radiflo) and a variety of unsystemitized leak location techniques. For that reason, the two methods are discussed separately.

2.6.1.1.1. Characteristics

In making use of this technique the components to be tested are placed in a tank, which is sealed and then evacuated to about 2 torr. Diluted krypton-85 is then pumped into the tank under a pressure. The radioactive gas diffuses into existing leaks in the components. After a prescribed "soaking" period (a few minutes to a few hundred hours), the krypton is pumped out of the tank and stored for reuse. A suitable combination of krypton pressure and "soaking" time may be selected to give the desired sensitivity. After this, an air wash is circulated over the components to remove any residual krypton from the external surfaces. The components are then removed from the tank. Those with leaks will retain some radioactive atoms, which emit gamma radiation. This radiation is detected by a suitable radiation counter, with the measured radiation being a function of the leak rate.

2.6.1.1.2. Applicability

Radiflo is useful for manufacturers that produce large numbers of small, sealed components. This technique is applicable where the required measurements are repetitive.

2.6.1.1.3. Sensitivity

The sensitivity of this technique may be as high as 10^{-13} atm cc/sec, under the right operating conditions. To attain these conditions, the soaking and counting time must be preset for the high sensitivity, and the units being tested must be of a "clean" construction with no possibility of adsorption of radioactive material on the outer surfaces.

2.6.1.2. EQUIPMENT AVAILABLE

Equipment for this leak detection system is manufactured by Consolidated Electrodynamics Corporation, Pasadena, California, under the name Radiflo. The characteristics of this equipment are itemized in Table 18.

2.6.1.3. TECHNIQUE ADVANTAGES AND LIMITATIONS

Although initial cost of the detector is high, the maintenance and operation costs are low, because almost all gas is recovered. This detector pays off when components to be tested range in the thousands per day. It gives 100 percent testing and can be calibrated for a reflection leak rate. The manufacturer gives the following labor and gas cost data for testing 5/16 inch transistors:

- Radioactive gas costs $250 per 10,000 or 2 1/2 cents per transistor.
- Activation labor is 15 minutes for 10,000 units
- Radiation check takes 0.16 minute per transistor

The Radiflo equipment is automated, so that its operation is extremely simple. However, since the use of radioactive material presents a potential danger, the operators and supervisory personnel must take a 60 hour course in instrument operation and health physics. These precautions are to an extent only necessary to establish proper personnel attitudes.

Radioactive krypton-85, due to its chemical inertness, does not participate in any metabolic processes in the body, if inhaled or ingested in any way. If accidentally inhaled for a short time, normal breathing of noncontaminated air will rapidly remove any krypton from the lungs and body tissue into which it might have diffused. With an adequate ventilating system, proper gamma-ray shielding of storage tanks and reasonable care, krypton-85 can be handled with negligible risk to the operators.

A significant disadvantage of this technique is the fact that many organic materials will take up krypton-85 under pressure and will hold it for various lengths of time after the pressure is released.

Some items to be leak tested have adsorptive surfaces, such as organic coatings, or small areas of organic insulation or gasketing. The gamma radiation emitted by this absorbed gas could make the item appear to be a leaker when, in fact, it was not.

Table 18

CHARACTERISTICS OF RADIFLO LEAK DETECTOR*

General Characteristics	Average Values
Sensitivity	1×10^{-11} atm cc/sec
Tracer Gas	Krypton 85
Output Signal	Visual and Millivolt Meter
Power Requirement	115 volts, 60 cycle AC
Size	56 inches x 55 inches x 44 inches
Weight	4000 pounds
Price	$35,000

Manufacturer

Consolidated Electrodynamics Corporation (subsidiary of Bell and Howell Company)

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* More specific information of this equipment may be obtained from General Electric Company Report S-67-1013 (Ref. 186)

Occasionally components with very large leaks (10^{-5} atm cc/sec) tend to slip through the leak test as acceptable. This is due to rapid escape of the radioactive gas through the large opening during the final pump down phase of the leak test cycle. This does not occur in practice, if the component contains any organic or other adsorptive material. In the absence of such internal adsorptive material, it is sometimes necessary to retain a slight partial pressure of krypton in the pressurizing tank before its removal for counting.

Alternatively, the components should be bubble tested after the Radiflo technique. The bubble tests will detect these gross leaks. However, since the fluid used in bubble testing will clog very small leaks, bubble testing should be performed only after the Radiflo technique has determined that no small leaks exist.

Two additional disadvantages of the Radiflo technique are a limitation of sample size to approximately 1 ft^3 and the fact that leaks cannot be localized by this technique.

2.6.1.4. PRINCIPLES OF OPERATION

Krypton-85 has a half-life of 10.3 years so that it can be used over and over again if it does not become too diluted with air or is lost into the atmosphere. Over 99 percent of the disintegrations give no gamma rays, but emit beta rays with a maximum energy of 0.67 MeV. Only 0.7 percent of the disintegrations give 0.54 MeV gamma rays. The primary usefulness of krypton-85 for leak detection is dependent on this small proportion of gamma emitting disintegrations.

The half-life of 10.3 years is equivalent to 3.25×10^8 sec. Therefore, 1 cm^3 of pure krypton-85 at normal pressure and temperature containing 2.7×10^{16} atoms will produce total disintegrations at the rate of 5.78×10^{10}/sec. Only 0.7 percent of these 5.78×10^{10} disintegrations/sec or 4×10^8/sec yield gamma quanta. Krypton-85 received from Oak Ridge contains 5 percent of the radioactive isotope. This gas is further diluted with air or by another gas for two reasons. One is to give the gas the effective viscosity of air. The other is to get sufficient volume for filling dead spaces in pressurized containers.

The gamma radiation is measured by a scintillation detector. Scintillation detectors convert a radiation pulse to a light pulse, which in turn is changed into an electrical pulse by a photo-multiplier tube. Solid or plastic crystal phosphors are selected on the basis of their particular suitability for detecting gamma radiation.

Both beta and gamma rays are detected by means of a thin wall Geiger-Muller counter. Krypton-85 adsorbed on surfaces will give readings of both beta and gamma rays. However, the walls of the device will adsorb any beta rays being emitted inside it. Therefore, it is possible to distinguish by the beta to gamma radiation ratio whether a radiation indication is due to adsorption or leakage. It goes without saying that if radiation due to adsorption is large, small amounts of leakage will go undetected. Precautions must be taken to assure that all surfaces are clean.

Radiflo is a classic use of back-pressure testing described in Section 1.3.1.4. As explained in that section, very large leaks may be overlooked because of rapid leakage out of the system during pumpdown. However, if adsorbents, such as unreconstructed Vycor* are present inside the device, krypton-85 will remain inside the device.

Absolute leakage standards are not available because tests are performed on sealed systems. Standards are available to ascertain the activity of the test gas and the accuracy of the test instruments, but test leaks to qualify the procedure do not exist. There is even some question as to validity of the correlation between the measured flow of krypton-85 and the amount of air flow it represents. Clark, Jones and Karoly (Ref. 118) found that because of difficulty in predicting the flow mode involved, leakage as measured by the Radiflo technique correlates to only one decade with flow measured by the mass spectrometer leak detector.

2.6.1.5. METHODS OF LEAKAGE MEASUREMENT

The Radiflo technique was developed by Cassen and Burnham (Ref. 111). Figure 70 shows an industrial testing unit. The activation unit includes a storage tank for radioactive gas, an activation or pressurization tank, and the vacuum pumps, compressor and other components necessary to accomplish the testing cycles. The activation unit also contains automatically programmed electronic circuitry to assure the protection of personnel from radiation hazards resulting from malfunctioning or maloperation of the equipment.

The test procedure has been described in literature (Ref. 119). Parts to be tested are placed into the activation tank which is sealed by means of a locking ring. To prevent dilution of the radioactive krypton-85, the activation tank is evacuated to approximately 2 torr. The air being evacuated is directed into an exhaust duct and vented to the atmosphere by means of a continuously operating blower unit. The activation tank is then pressurized to the desired level with krypton-85.

The activation tank remains pressurized for a preset length of time determined by the desired sensitivity of the test. At the conclusion of the activation cycle, the testing gas is returned to the storage tank thereby reducing pressure in the activating tank to 2 torr or less. The unit is then flushed with air to remove any residual krypton-85.

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*Unreconstructed Vycor, often referred to as "thirsty glass," is obtained by chemically leaching out certain constituents to obtain an almost pure silica with a porous mass of a surface area of approximately 200 m^2/g.

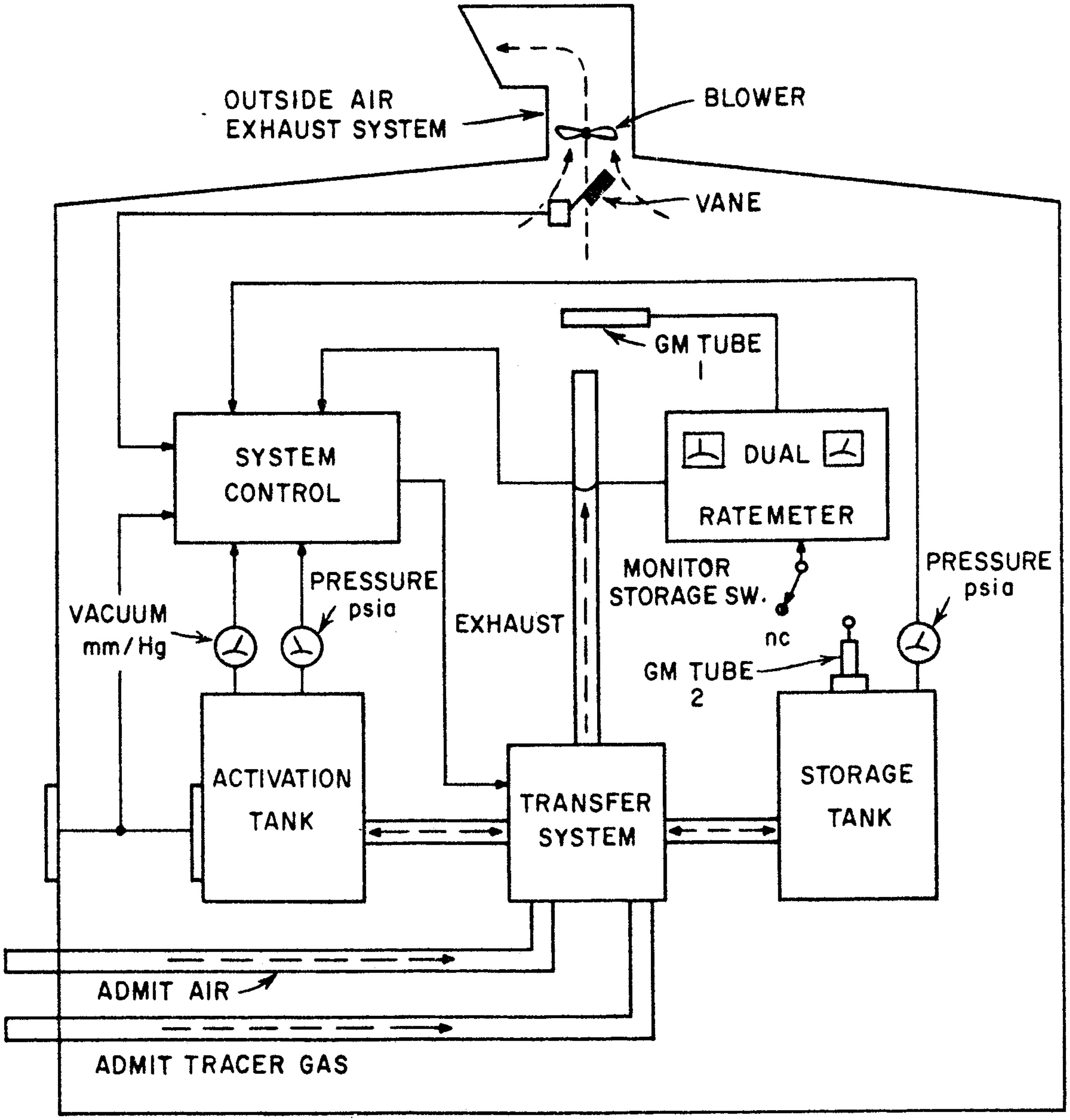

Figure 70. Unit for Radiflo Testing. (Reprinted, with permission, W. Inglis, Materials Evaluation, Vol. 24, No. 3 150 (1966), Copyright 1966, Society for Nondestructive Testing, Inc., Evanston, Ill.)

Because a radioactive isotope is used in this process, certain precautions must be taken to maintain safe operation of the equipment. Various components of the activation unit are lead shielded to ensure that the level of gamma radiation from the equipment is always well within the maximum permissible exposure level established by the AEC. The entire cycle is automatically programmed. A system of interlocks is incorporated into the equipment which prevents an accidental discharge of radioactive gas.

For fixed testing pressures and fixed exposure times, the sensitivity bottleneck is the efficiency of radiation detection, assuming no external surface adsorption of the gas on paints, grease, dirt or organic materials. Small parts can be counted in a well or tunnel-like thallium activated sodium iodide crystal scintillation counter.

Under favorable conditions, about half of the emitted gamma quanta will yield counts. The counts are random in time and are subject to Poisson statistics in the distribution of counts in a given time interval or time for a given number of counts. If the background counting rate is small compared with the part counting rate, the standard deviation of a distribution of repeated counts per unit time with an average over the distribution of N counts per unit time will be N. Thus, the total number of counts must be increased four times to double the counting accuracy, and 100 times to increase the accuracy by a factor of 10. The scintillation crystal must be well shielded from low-level ambient radiation to reduce the background count.

Fortunately, high precision counting is not necessary for most leak measurements. Leak rate distribution can cover a range of so many powers of 10 that even a factor of 2 or 3 makes very little practical difference in their classification.

Routine checking of rejected parts with a thin window Geiger-Muller tube consistently reveals surface contaminated parts. Comparison of such rejected parts with acceptable parts of the same surface composition can be used to determine the significance, if any, of the surface contamination. Those parts with significant contamination can often be decontaminated with a brief exposure to heat. Frequently, such a heating cycle is routinely incorporated into the testing procedure of certain parts having organic coatings.

For mass production testing, an automatic counting station will perform GO-NOGO testing functions at a rate from 600 to as high as 2000 units per hour, depending upon the size of the part being tested. Activated parts are placed in a shaker unit, automatically fed into cups on a continuously moving conveyor, and carried to a scintillation detector for analysis. A scaler measures and compares the activity against a preset rejection threshhold. The tested parts are then deposited in an appropriate "accept" or reject" bin.

2.6.2. METHODS OF LEAK LOCATION

Leak location techniques using radioisotopes are not systemized. These techniques are used to locate leaks in very large systems, where other methods would be extremely difficult. The advantage of this method is its high sensitivity, compatibility with any gas or liquid being used in the system, and the fact that the tracer does not have to be removed from the leakage site to be detected.

However, these techniques require experienced personnel to handle radioisotopes, measure radioactivity, and interpret pertinent data. Moreover, the use of radioisotopes represents a potential hazard, and disposal produces more handling problems than are usually encountered with other tracers.

The most complete review of these techniques is by Courtois and Gasnier (Ref. 120). The techniques may be subdivided into:

- Radioactive liquid testing
- Radioactive gas testing
- Sealed radioactive capsule testing

2.6.2.1. LIQUID AND/OR GAS TESTING

This principle is used most frequently because of universal application for pipe testing. A radioactive tracer is introduced into the system with the filling fluid. The fluid escapes through the leak, producing contamination in the vicinity of the leak. The tracer can be detected at the leak after flushing the system.

These three stages--filling, contamination, and detection--can be accomplished in various ways. The pipe may be filled with a homogeneous tracer or by a slug (or puff) of radioactivity. With a homogeneous solution, the search can be carried out in a pipe under constant pressure and with no flow. On the other hand, it is necessary to check the homogeneity of the product introduced and to evacuate a considerable volume of tracer-contaminated fluid.

Alternatively, a "puff" of a radioactive tracer is introduced between two columns of the inactive fluid separated by two scraper pistons or "godevils". This method is applicable only for the liquid phase, because in a gaseous phase the two scraper pistons would meet. Only a relatively limited quantity of fluid is contaminated in this kind of tracing.

Detection may be at ground level, from the interior of the pipe, or by probing. At ground level, which is, of course, the most simple method, the detection team is equipped with portable radiation detectors and moves along the pipeline. This method requires heavy activity of the radioelement emitting gamma rays.

because radiation must pass through a ground layer of considerable thickness, which is often compounded by a layer of paving, cement, or other covering.

This method of detection requires the use of very sensitive detectors, i.e., NaI(Tl) crystal scintillation counters.

In the case of gas leaks, considerable diffusion of gas takes place towards the surface. This diffusion is largely determined by the nature of the ground as well as of the tracer gas. This reduces the accuracy of localization, but permits the use of a radioelement of lower activity, by reducing the distance between the tracer and the detector.

It is possible to probe for the leak from inside the pipe, as described by Putman and Jefferson (Ref. 121 and 122). This procedure includes passing through the pipe a scraper piston or a "godevil" which contains the electronic detection system and a "memory." This detecting scraper is moved along inside the pipe by the pumping action and records any increase of activity in passing along the contaminated ground.

The great advantage of this method is that it requires only a low tracer activity at the leak because the radiation emitted need only pass through the pipe wall to reach the detector. The activities required are usually some 1,000 times less than those necessary for detection from ground level.

Some of the practical parameters which limit the use of this method are:

1. The pipe diameter must be sufficient to permit passage of the detection electronic device.

2. The bends of the pipe must be such that the scraper piston, the length of which is generally in the order of one and a half times the diameter of the pipe, may pass through.

3. After the passage of the tracer fluid, the pipe must be rinsed so as to be free from traces of activity.

4. The pipe must, insofar as possible, be free from elements likely to adsorb the tracer.

5. The suspension of the electronic detection equipment and its memory must be able to absorb the considerable shocks to which the scraper is subjected.

6. The detecting scraper must be absolutely impervious.

7. A number of small radioactive sources (e.g., Co^{60}) be placed at certain distances along the pipeline. The presence of these sources is recorded to make

localization of the leak easier.

If detection from the inside of the pipe is impossible, the alternative method consists of digging holes for lowering the detection probe, in order to reduce the thickness of the ground layer screen between the source and the detector.

For systematic examination, the holes must be very close to one another. This kind of exploration would be possible only on very short pipelines. It is often sufficient to probe only suspicious points (connections, valves, etc.).

The tracer must fulfill a certain set of conditions, the main ones being:

1. It must emit gamma rays of sufficient penetrating power.

2. It must be prepared at a sufficiently high activity and low cost, which means a cheap irradiated target and an adequate neutron cross-section.

3. Its half-life must be short. If possible, it should be equal the time necessary for the search, to permit rapid elimination of the contaminated products.

4. It must have as high as possible a tolerance level in air and in water.

5. It must be easily absorbed by the ground, contaminating the spot of the leak without being adsorbed on the walls of the pipe.

Some of the elements used are listed in Table 19.

The nature of the ground's effects on the leak-generated radioactivity are discussed by Gemant (Ref. 123, 124, and 125). The two main problems are diffusion away from the leak area and shielding by the soil.

Modifications of the techniques discussed above are occasionally used (Ref. 126). In one, a slug of radioactivity was introduced suddenly, and its rate of movement along the pipe followed. At a leak there was a discontinuity in the rate that spotted the trouble. In the second method both ends of the main were closed, and the isotope was introduced at the center. The operator observed which way the tracer flowed into the pipe, to the right or to the left. Then he bisected the offending section and tried again.

The isotope used was 15-hr Na^{24} in sodium bicarbonate at a specific activity less than 1/10 tolerance for potable water. Where higher levels were required, overchlorination was used to make the water distasteful. Holding the solution in an unused portion of the main for a week and then dumping into a convenient

Table 19

ELEMENTS USED FOR RADIOACTIVE TRACING

Element	Compound	Half-life	Energy, MeV
		Gas Leaks	
Br^{82}	CH_3Br	36 hours	1.35, 0.8
Xe^{133}		5.3 days	0.08
Kr^{85}		10 years	0.670
A^{41}		2 hours	1.33
Rn^{222}		3.64 days	0.51
C^{14}	CO_2	5600 years	0.160
		Liquid Leaks	
Na^{24}	NaCl, $NaHCO_3$	15 hours	1.4, 2.8
As^{76}	$(NH_4)_3AsO_4$	27 hours	1.2, 0.6
I^{131}	NaI	8 days	0.74, 0.66, 0.53
Sb^{122}	$(C_6H_5)_3$ Sb	2.8 days	0.56

stream took care of disposal.

2.6.2.2. SEALED FLOAT TESTING

Sealed, isotope-tagged floats for leak location are advantageous since smaller amounts of activity are required, and the active material does not mix with the pipe contents or contaminate surroundings. Leaks are found by placing a properly weighted radioactive float in the fluid of a sealed pipe and allowing the current to carry it (Ref. 127). With normal exits closed, the only current is that of fluid escaping through the leak. The float moves with the current and stops at the leak. If the course of the piping is marked beforehand, a man with a detector can easily follow the source.

Once the leak has been located, it is possible to recover the source by draining the line through a large gate valve (it may have to be installed for the purpose) and catching the source in a minnow seine.

Experiments with a mockup of a leaking pipe have shown that the best float for this kind of tracing is a rigid sphere with projections at the equator. The float must have the density of the fluid in the pipe. Since it is rigid and incompressible, pressure changes do not cause it to settle or float out of the current. Projections enable the current to grip the float so that it stays in the current when the direction of flow changes, as at an elbow.

Material to be activated goes into a quartz capsule; the capsule is sealed in an outer container; the whole assembly is properly weighted, and then sent to a reactor of irradiation. Na^{24} and Co^{60} have been used as float-marking radionuclides. Na^{24} is ideal: its energetic (2.76-MeV) gamma radiation makes it usable even in deeply buried lines; its short (15-hr) half-life ensures that if a source cannot be recovered, its activity will soon decay to a safe value. Co^{60} is ordinarily useful only in shallow lines because its 1.17- and 1.33-MeV gammas are readily attenuated. Furthermore its 5.2-year half-life makes it necessary to recover a source if it gets stuck.

Several leaks in one system can be found, since current flows from each. If all are of the same size, the source usually travels as far as the first one and stops. To sweep the source to the next leak, current has to be created larger than the leakage. This is done by increasing pressure or opening an exit.

When there are leaks of different sizes, the float often goes to a large leak and stops. It may slow down, however, as it passes a small leak, and an experienced observer can often recognize such a change of speed. If no speed changes are observed, one can locate and repair the largest leaks, then reexamine the line for smaller ones.

Sometimes a leak is so large that one cannot make the float pass it. Giving the float a new starting point will usually reveal other leaks even as it flows once more to the large leak. Choosing new starting points and changing the direction of flow will distinguish a leak from a mechanical block. A leak stops or slows the source regardless of direction; the source may go past a mechanical block from one direction, although it is stopped from the other.

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Section 2.7

HALIDE TORCH

2.7.1. PRINCIPLES OF PROCEDURE

The halide torch is used to locate leaks in systems filled with gas containing halogens. The color of the flame changes upon introduction of halogen gas.

2.7.1.1. CHARACTERISTICS

The halide torch consists of a burner connected to a tank of gas or alcohol (Figure 71). Some of the air for combustion is drawn into the flame (chimney fashion) through a tube near the bottom of the burner. A flexible extension of this tube is used as a probe to locate leaks. When the open end of this tube passes near a gas leak, the gas is drawn into the flame.

The detector is a small burner arranged to pull primary combustion air through a tube and into the burner. The burner heats a copper plate. The flame is a pale blue if only air is pulled into the burner through the suction hose. If small amounts of vapor containing halogen compounds enter the suction tube, the flame turns green, characteristic of copper.

2.7.1.2. APPLICABILITY

This procedure is used to locate leaks in pressurized systems. It is a desirable technique because of its low cost and portability.

2.7.1.3. SENSITIVITY

The torch permits locating leaks down to about 8 or 10 ounces of refrigerant gas per year. The sensitivity to refrigerant gas is approximately 100 ppm. This makes the general sensitivity approximately 10^{-4} atm cc/sec.

2.7.2 EQUIPMENT AVAILABLE

The torch is sold both as an individual unit and as an attachment to portable gas cylinders. A more complex instrument using the same principle is also sold. The properties of these instruments are shown on Tables 20 and 21.

2.7.3. PROCEDURE ADVANTAGES AND LIMITATIONS

In general, it can be said that this test is as sensitive and rapid as the soap-bubble test. In addition, the torch method permits location of leaks in places where soap bubbles could not be seen. Moreover, no residue of water and soap must be removed before the tested apparatus can be used. The technique is, of course, safer than any test involving hydrogen. Freon is non-

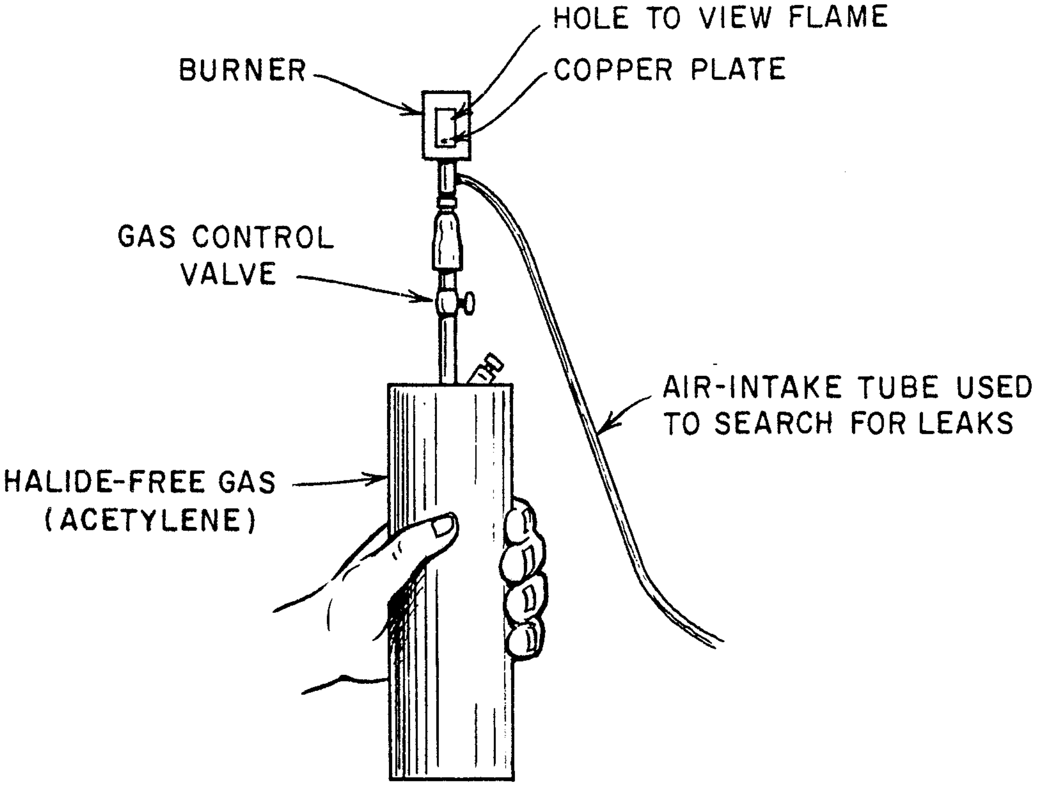

Figure 71. Halide Torch for Leak Location.

Table 20

CHARACTERISTICS OF HALIDE TORCH DETECTORS*

General Characteristics	Average Values
Sensitivity	10^{-4} atm cc/sec
Tracer Gas	Halogen Compounds
Output	Visual Observation
Power Requirement	Compressed Gas Source
Size	15 inches; 4 inch diameter
Weight	1 pound
Price	$10

Manufacturers

Bernzomatic Corporation
Justrite Manufacturing Company
Lenk Manufacturing Company

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* More specific information on this equipment may be obtained from General Electric Company Report S-67-1013 (Ref.186)

Table 21

CHARACTERISTICS OF FLAME COLOR CHANGE DETECTOR*

General Characteristics	Average Values
Sensitivity	50 parts per million; 5 x 10^{-5} atm cc/sec
Tracer Gas	Halogen containing compounds
Output	Meter
Power Requirement	120 volt, 60 cycle AC
Size	15 inches x 16 inches x 9 1/2 inches
Weight	35 pounds
Price	$675

Manufacturers:

Davis Emergency Equipment Company, Incorporated
(Instrument Division)

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* More specific information in this equipment may be obtained from General Electric Company Report S-67-1013 (Ref.186)

flammable, and, once a leak is found, it may be soldered without fear of explosion.

Other advantages are low cost, portability, simplicity, and ease of operation.

The torch has no means of accurate calibration and few users realize that it is difficult to locate leaks smaller than 8 or 10 ounces per year when the unit to be tested is filled with 100 percent refrigerant gas. When the atmosphere is contaminated, and when a leak is located, toxic gas is generated. Therefore, the torch should not be used in confined areas.

Carbon tetrachloride vapors are toxic. They should never be used unless there is adequate ventilation. Continued exposure to vapors is dangerous; persons using this material should be carefully instructed beforehand so testing will be safe.

Another major drawback is that the procedure consumes oxygen and may give off enough toxic gas to make it unsafe in confined areas that aren't adequately ventilated. The open flame may be a serious hazard in certain atmospheres.

A single large leak may mask other adjacent smaller leaks, necessitating prior location (and correction) of such larger leaks by separate test.

This procedure uses halogenated hydrocarbons and therefore has the same diffusion and stratification problems as described in Sections 1.5.1.5. and 2.2.2.

2.7.4. METHODS OF TESTING

To hunt for leaks, the torch is lighted and checked for proper operation by sucking in a trace of halogen gas from the supply tank. Then the air-intake tube is used to search the surface of the system being tested at the rate of about a fraction of an inch per second. Since the tracer gas has four times the density of air, it is advisable to start work on the lower side of a possible leak. A small trace of halogen gas will show up as a green flame, a large quantity as a violet flame. Servicemen usually move the probe about 1/4-inch per second to locate leaks down to 8 ounces per year. Although any halide gas could be used in this test, Freon (i.e., CCl_2F_2) is the best with respect to sensitivity, vapor pressure, inertness, and safety.

Another test involving filling the system or some part of the system that can be isolated, with tracer gas and then probing the surface of the system to detect traces of gas that issue from the leaks. Depending on the size of the vessel and the sensitivity desired, the air may or may not be evacuated before the tracer gas is introduced. Evacuation takes longer and is not practicable for very small pipes; but if accomplished, it makes possible a pure tracer gas atmosphere that can be pumped back into the storage tank afterward.

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Section 2.8

SONIC DETECTORS

2.8.1. PRINCIPLES OF TECHNIQUE

Sonic energy produced by turbulence that occurs in the flow of a fluid provides a detectable and measurable quantity useful for leak location. The technique is applicable to both pressure and vacuum systems. The turbulence that occurs in the region of a leak in a vacuum system as the gas flows in creates sonic energy similar to that created by pressure leaks. It has been shown that the technique is applicable, so long as there is sufficient pressure differential to produce the turbulent conditions associated with generation of sonic energy. This leak is located by location of the source of the sound.

2.8.1.1. CHARACTERISTICS

Sonic detection techniques may be divided into both active and passive types. In the active techniques, sound is injected into the system. The leak is located by the effect of the leak on sound transmission. The passive technique of leak location involves two methods: (1) detection of the sonic signal from outside the pipe, and (2) detection of the signals inside the pipe. The frequencies of the signals are entirely different in these two instances, and two different types of techniques must be employed.

It is possible to locate very large leaks unaided by any equipment, by simply locating the source of sound. Alternatively, a simple medical stethoscope may be used to aid in location. Obviously, with such simple equipment the sensitivity is lower and the effect of noise background is greater, since little discrimination between noises is possible.

2.8.1.2. APPLICABILITY

This technique is applicable for leak location in systems containing a vacuum, liquids, or gases. The only requirement for applicability is that the leakage be large enough (10^{-2} atm cc/sec) to generate noise (of some frequency, which may be ultrasonic) during flow. There are no restrictions as to the fluid generating the sound.

2.8.1.3. SENSITIVITY

This technique has a maximum sensitivity of approximately 10^{-2} atm cc/sec. But this sensitivity will not always be achieved, since a laminar flow leak does not generate sound. This technique is intended for large leaks only.

2.8.2. EQUIPMENT AVAILABLE

The primary equipment commercially available is of the ultrasonic type described in literature (Ref. 128), and in this section. These instruments convert the ultrasonic sound to an audible range.

Other detectors are simply amplifiers which increase the level of the sound without conversion to another frequency. The third type of instrument is essentially a stethoscope, which mechanically locates the source of the audible leak. The characteristics and manufacturers of these instruments are listed in Table 22.

Of the various types, commercial equipment is only sold for passive detection outside the system.

2.8.3. ADVANTAGES AND LIMITATIONS

As long as the conditions for generating the sound are met, leaks can be detected at distances up to and beyond 100 feet. In this detection scheme, no physical contact between the detector and the instrument is necessary if the fluid is escaping into air. If it is flowing into another chamber, contact has to be made to that chamber. However, flow through porous material or labyrinth type leaks does not possess the required turbulence conditions and, consequently, is not readily detected ultrasonically.

Perhaps the biggest advantage of this technique is that it may be used with any fluid in the system, providing the physical conditions for sound generation are met. This eliminates the need for a special tracer gas.

Occasionally, the noise level in the area in which the detector is being used drowns out the sound of the leak. Sometimes normal flow of fluids through a pipe will give an input to the instrument, making it useless except under static conditions. The ease with which ultrasonic energy is reflected from hard surfaces sometimes poses a problem in establishing the exact location of a source, if the operator cannot get close to it. This requires some experience on the part of the operator to quickly recognize whether he is intercepting a direct wave or a reflection. The ability to recognize the direct and reflected waves can be readily adquired, so it is not a serious problem.

2.8.4. PRINCIPLES OF OPERATION

Whenever gas escapes through a leak, it does so by means of turbulent flow. If the energy spectra generated by this turbulent flow are examined, it is found that all leaks generate sound at frequencies between 30 and 50 KHz (Ref. 97).

At lower pressures, there is a distinct maximum around 40 KHz. At higher pressures, (ca. 2000 psi), there is a broad energy distribution with the prominent 40 KHz peak showing at the lower flow rate.

Ultrasonic energy is converted to electrical energy by an appropriate transducer. By restricting instrument response to

Table 22

CHARACTERISTICS OF SONIC LEAK DETECTORS*

General Characteristics	Average Values		
	Ultrasonic	Sonic	Stethoscope
Output	Audible Signal and Meter	Audible Signal and Meter	Audible Signal
Power Requirement	Battery	Battery	None
Size	5 inches x 5 inches x 10 inches	5 inches x 3 inches x 3 inches	---
Weight	8 pounds	5 pounds	1 pound
Price	$300	$100	$20

Manufacturers

Ultrasonic	-	Burnett Electronics Laboratory, Incorporated Fisher Research Laboratory, Incorporated General Dynamics - Liquid Carbonic Division Hewlett-Packard Company (Delcon Division) Techsonics (formerly Eldevco) Tel Air Products Corporation
Sonic	-	Fisher Research Laboratory, Incorporated Goldak Company, Incorporated Marcol, S.A. (M. Paquet and Company, distributors) Tinker and Rasor (Detection Division) Minear Scientific Instruments, Inc.
Stethoscope	-	Schneider Manufacturing Company (Grisby Company, distributor)

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* More specific information on the equipment may be obtained from General Electric Company Report S-67-1013 (Ref. 186)

to the region of maximum ultrasonic energy, it is possible to enhance the devices discriminatory capabilities. Noise in the audible range which would mask the weak audible sounds of small leaks are not a problem. The same conditions are valid for both airborne and conducted sounds.

McElwee and Scott (Ref. 129) describe the use of a stethoscope and a simple electrical amplifier to detect leaks by the sound they generate in the audible range. Their system involved detection from inside of pipes. Another system is described in literature (Ref. 130) using the sound of leaking water for detection.

The active method of sonic leak detection is discussed by Long (Ref. 131) and by the experimenters at Ohio University (Ref. 97). These techniques have not been fully developed at this time.

2.8.5. METHODS OF TESTING

The ultrasonic energy produced by gas or liquid flow turbulence in the leak is converted by an appropriate transducer into electrical energy. The process is illustrated in the block diagram of an ultrasonic detector shown in Figure 72. The minute electrical output produced by the ultrasonic transducer is amplified and then translated into the audible range in the modulator by the conventional mixing process, commonly used in superheterodyne radio receivers. These converted ultrasonic signals exhibit audible sound characteristics that are very similar to their audible counterparts for the same physical phenomena (e.g., a leak has the hissing sound that is characteristic of air escaping from a pressure vessel; friction has a scratching sound that is characteristic of two surfaces rubbing together).

Two types of transducers find wide application for the location of gas and internal hydraulic leaks. The directional transducer has a directional horn which enables the operator to scan an area and locate a leak in much the same way that a radio direction finder is used to obtain radio bearings. This particular unit has a directional pattern that is 22° wide at the -3 db points. The short wavelengths of the ultrasonic frequencies make it possible to design highly directional horns that are small in size and convenient to use. Use of a parabolic dish permits a directivity pattern of a fraction of a degree.

The contact probe is used in the location of internal leaks in hydraulic systems and requires direct contact with the components in the system. The ultrasonic energy produced by the fluid flow is conducted through the walls of the valves, tubing, and other components; it makes possible the detection of leak flows and changes in the flow conditions that are usually difficult to locate without disconnecting lines and partially disassembling the system.

The detector is portable and can be used as a leak locator with either the directional or contact probe. Leak location is performed by scanning the suspected areas with the probe.

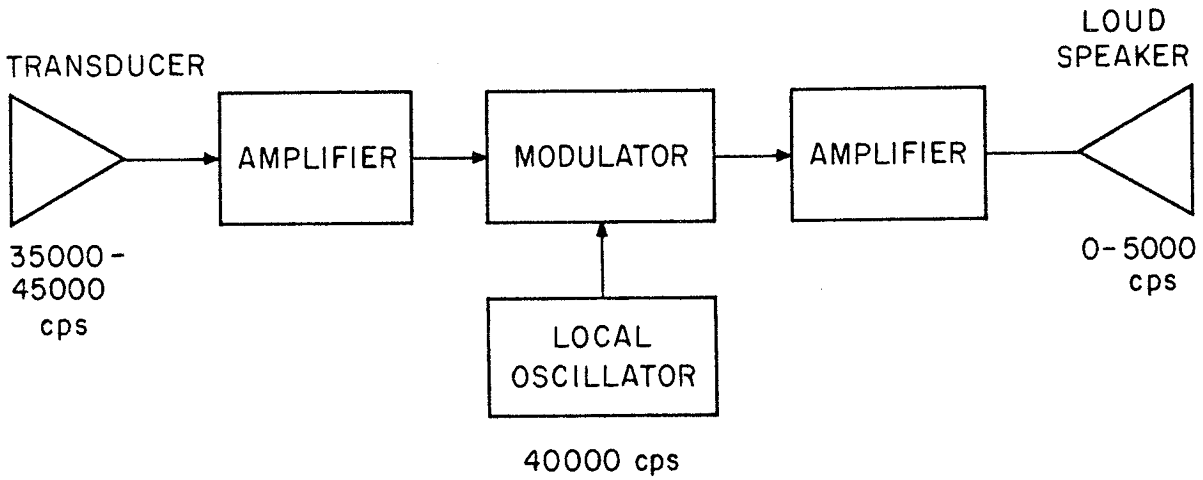

Figure 72. Block Diagram of an Ultrasonic Leak Detector. (Courtesy of Hewlett-Packard Company, Delcon Division.)

A calibrator for the sonic leak detector is a small plastic squeeze bottle. It is said that a brisk squeeze on this bottle may be heard by a sonic detector at a distance of fifty feet. Although this device will not provide a quantitative calibration, it enables the operator to determine if the instrument is qualified for leak location.

2.8.6. GENERAL REFERENCES

1. Anonymous, "Demonstration of the Aqua-visual Apparatus for Leak Detection at Sheffield," Water and Water Engineering, May 1953, pp. 197-199.
2. Goddard, E.G., "Ultrasonic Leak Detection." Paper presented at 56th Air Force/Industry Conference on Missile Systems Leaks and Spills, February 24-26, 1965, Los Angeles, California.
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5. Quisenberry, R.C., Leak Detection Technique Improvement Study for Space Vehicles, Second Formal Report by Ohio University, Athens Ohio, on National Aeronautics and Space Administration Contract NAS 8-2563.

Section 2.9

LIGHT ABSORPTION

2.9.1. PRINCIPLES OF PROCEDURE

A tracer gas which absorbs radiation of a particular wavelength is used in the light absorption procedure. The detector analyzes for the presence of this tracer gas by checking if radiation of that particular wavelength is being transmitted. Although ultraviolet or visible radiation may be used, available commercial instruments are built specifically for infrared radiation. Instruments for detection in the ultraviolet range are usually sold for leakage monitoring.

2.9.1.1. CHARACTERISTICS

The detection system consists of a radiation source, sample and reference cells, and a radiation detector. The radiation passing through the sample cell is compared with the amount passing through the reference. Tracer gas in the sample cell absorbs the radiation, and this absorption is electrically measured by the detector.

The above method measures the concentration of tracer gas in the system. By attaching a probe and a pumping station on the sample cell, it is possible to make such a system into a detector probe leak locator.

2.9.1.2. APPLICABILITY

The light absorption method may be used for both static and dynamic leakage measurement.

It is especially suitable for leak location using a detector probe. This instrument is designed for operation in an ambient atmosphere. However, it may be used at a higher or reduced pressure.

2.9.1.3. SENSITIVITY

The instruments are capable of a full scale reading at 100 parts per million nitrous oxide in air. This implies a detectable limit of several parts per million. With the internal pumping system in the cc per second range, these instruments can detect leaks at the range of 10^{-6} atm cc/sec.

2.9.2 EQUIPMENT AVAILABLE

The equipment available for infrared leak detection is manufactured by three companies, two in the United States and one in Great Britain. The companies and the characteristics of their products are listed in Table 23.

Table 23

CHARACTERISTICS OF LIGHT ABSORPTION LEAK DETECTORS*

General Characteristics	Average Values	
	Infrared	Ultraviolet
Sensitivity	1×10^{-6} atm cc/sec 10 parts per million	5 parts per million
Tracer Gas	nitrous oxide, carbon dioxide	chloronated hydrocarbons aromatic hydrocarbons
Output	Milliammeter	Milliammeter
Power Requirement	115 volts 60 cycle or 190/260 volt 50 cycle	115 volt 60 cycle
Size	12 inches x 12 inches x 20 inches	amplifier 8 1/2 inches x 11 inches x 5 inches detector 6 inches x 6 inches x 19 inches
Weight	60 pounds	30 pounds
Price	$2000	$400

Manufacturers

Honeywell, Incorporated
(Apparatus Controls Division)
Teledyne, Incorporated
(Analytic Systems Company, Division)

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* More specific information of this equipment may be obtained from General Electric Company Report S-67-1013 (Ref. 186)

It is of historic interest that the first detectors were developed by the Perkin-Elmer Company, Norwalk, Connecticut. More recently the manufacture and distribution of these units were taken over by the Mine Safety Appliances Company, Pittsburgh, Pennsylvania.

2.9.3. ADVANTAGES AND LIMITATIONS

The infrared detector is in the same general class of applicability and sensitivity as the heated anode halogen detector. It is therefore pertinent to compare its advantages and limitations in that light.

The detection instrument can be made specific for one gas and will generally not be affected by contaminating impurities in the atmosphere. The detector will not be worn out by an exceptionally high concentration of tracer gas. Nitrous oxide is not soluble to any large extent in rubber or plastics, so that systems using these materials may be leak checked. Because its molecular weight is only slightly higher than air, nitrous oxide does not stratify as much as do the halogenated hydrocarbons.

However, the infrared system is at least one decade less sensitive than the heated anode halogen leak detectors. Its price is at least twice the equivalent halogen system. In general, the response time of the infrared detectors is twice that of the equivalent halogen system.

2.9.4. PRINCIPLES OF OPERATION

Infrared is the wavelength usually used in the detection instruments. One exception is the description of a detector (Ref. 132) using ultraviolet radiation. However, this instrument has never been developed commercially.

The infrared active tracer gas must have an absorption peak between 2 and 10 microns. Such a peak implies a polyatomic molecule of dissimilar atoms. Other requirements of the tracer gas are that it

- Be noncorrosive to the system material
- Present no fire or explosion hazard
- Be nontoxic
- Be reasonably priced and readily available
- Produce good discrimination in normal atmospheric contaminants

Nitrous oxide, N_2O, is usually used as the tracer gas because it meets these requirements. Also, its major absorption band at 4.5μ affords excellent discrimination ratios against atmospheric contaminants normally found in manufacturing plants.

Nitrous oxide is completely nontoxic. With oxygen, it is a simple asphyxiant in the same class as nitrogen, helium, and hydrocarbons (Ref. 133). Nitrous oxide is an anesthetic, but a very weak one, since a concentration of more than 80 percent is necessary in order to produce deep anesthesia (Ref. 134). There is no evidence in literature of any toxic effects due to ingestion or inhalation of nitrous oxide. The compound is noncorrosive and nonreactive with most materials (Ref. 135).

Nitrous oxide, N_2O should not be confused with nitrogen oxide, NO , nitric oxide $(NO)_2$, nitrogen dioxide, NO_2 , nitrogen tetroxide (N_2O_4) or nitrous fumes (a mixture of NO and NO_2). With the exception of nitrous oxide, N_2O , all the other oxides of nitrogen listed above are corrosive and poisonous. Unless the equipment is designed for their use, no attempt should be made to use any of these other nitrogen oxides for infrared leak detection.

Other gases besides nitrous oxide can be used as leak tracers. They must of course, be infrared active, and their sensitivity will depend mostly on the discrimination ratios of the background components. For instance, almost all hydrocarbons can be used in a background of air and give results in the low ppm range of detection. Other gases which may be used are CO, SO_2, and the halogenated hydrocarbons.

The infrared analyzer is usually a nondispersive type of optical nulling system. This system is designed for ruggedness and long term stability.

A schematic diagram of the detector as described in literature (Ref. 136) is shown in Figure 73. A hot wire source provides the radiation energy. The energy diverges and takes two paths, one through the reference cell and one through the sample cell. The two radiation paths are converged by the filter cell and admitted alternately into the detector by a radiation chopper rotating at 6.5 cycles per second. This frequency is used because it is not divisible into 60. Therefore, the signal can be distinguished from 60 Hertz ac line frequency.

The radiation detector is a pneumatic device consisting of two chambers separated by a very thin membrane. These two chambers are filled with an infrared active gas, which usually is the same gas for which sensitivity is required. Hence, the name "selective detector." There is a very small leak connecting the two chambers which provides pressure equilibrium across the membrane during slow ambient-temperature changes. Its impedance however, is very high to the relatively-fast pressure changes caused by the chopped radiation flux. The membrane is close to a fixed plate and the two form a small electric capacitor. If chopped radiation is allowed to enter the inner chamber, the gas will be alternately heated and cooled. The resulting pressure changes flex the membrane and this changes the electrical capacitance. These capacitance changes are detected by a sensitive radio-frequency oscillator.

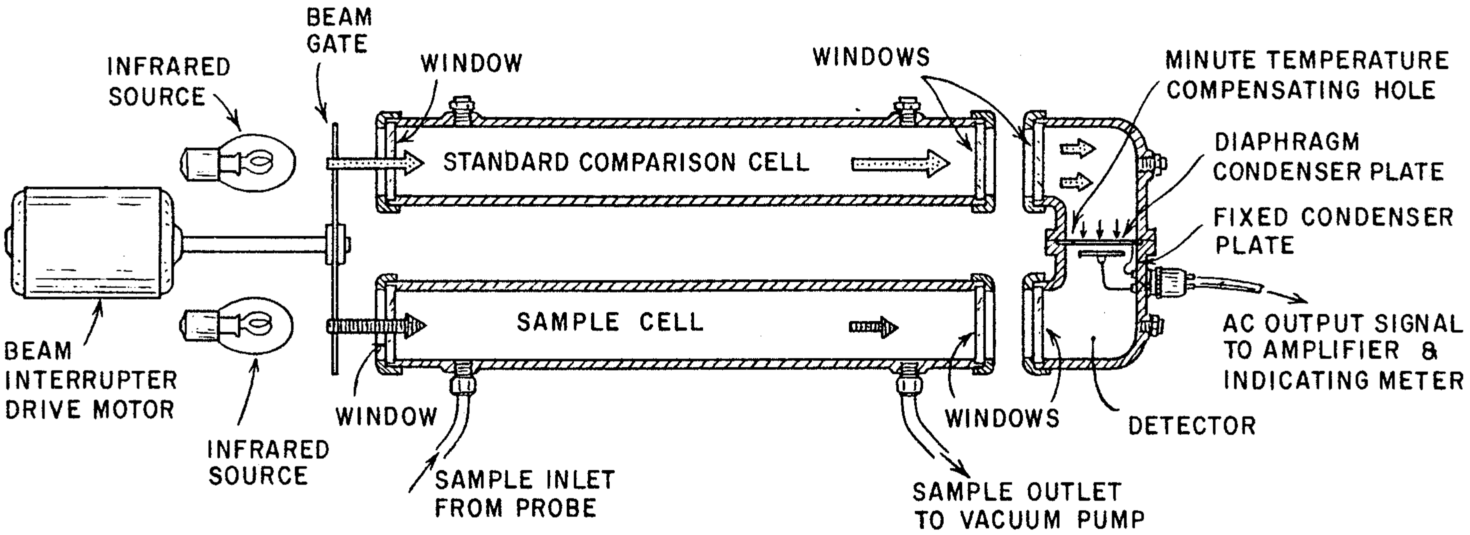

Figure 73. Infrared Leak Detector. (Reprinted, with permission, from Neeley, A.H., Nitrous Oxide for Leak Detection, A: Chemical, Physical and Pharmacological Properties, with Results of Corrosion Tests (Ohio Chemical Technical Information Series: IA); Division of Air Reduction Company, Inc., Madison, Wisconsin, May 1957.)

Initially, the two beams are balanced and, since the chopped radiation coming from the two beams is 180° out of phase, there will be no output from the detector. When the intensity of the sample beam becomes different from that of the reference beam (due to the presence of a gas which absorbs radiation at the same wave lengths as the detector), the radiation flux produces pressure changes in the detector. The membrane moves accordingly, the capacitance changes, and an "error" signal develops.

This error signal is detected in the proper phase and amplified sufficiently to drive the two-phase servomotor. In turn, the servomotor drives the attenuator into (or out of) the reference beam until the error signal is reduced to zero and beam balance is restored. Simultaneously, the servomotor also changes the setting of the transmitting potentiometer. This change in resistance eventually is measured by an indicating meter or recorder.

Because the optics operate in the infrared, CaF_2, windows are used throughout the optical system. To further isolate the 4.5μ region, mica discs are installed in series with the sample cell to cut off interfering wavelengths beyond 6.0μ. This isolation increases the discrimination for nitrous oxide.

The system also contains a built-in test circuit which enables the operator to make a rapid check of the various subassemblies, and the radiation chopper system without removing dust- or explosion-proof covers.

The usual instrument package consists of a detector probe system.

The leaking gas enters the probe, is filtered, analyzed in the sample cell, passes through a flow meter and a metering valve, and exits through the pump. The valve adjusts the flow to the rate required for adequate sensitivity and response time. A neon light is provided at the end of the probe to indicate leak conditions when the operator is in a noisy area and out of visual range of the indicator. The other indicators are an alarm horn and a meter. The sampling system operates at slightly sub-atmospheric pressure; the pump can provide flow rates up to 2 liters/min.

2.9.5. METHODS OF TESTING

The system as sold is designed for leak location using a detector probe. Alternatively, it may be used for leakage measurement using a static or dynamic testing method. However, the pumping speed of the system is only about 2 liters per minute. Of course, internal pumping is not necessary, except to place the tracer gas in the system. Auxiliary pumps may be used to increase the pumping speed. Alternatively, the detector may be used without pumps as a concentration monitor in static leakage measurement.

The system may be automated and used for leakage measurement on assembly lines. The structure of the system is rugged and the operation is simple. The manufacturers claim a 2 percent of full-scale sensitivity drift in twenty-four hours of operation.

When used for leak location, the system has a response time of approximately 3 seconds. This of course can be changed by altering pumping speed or probe length.

The response of the instrument is linear over the full scale. The instrument is calibrated by introducing tracer gas of a known concentration from a compressed gas cylinder into the sample chamber of the detector. In this way, the instrument response is calibrated in parts per million of tracer gas. The leakage rate response is obtained by multiplying this response by the flow rate of gas through the sample chamber.

The detector may be used either evacuated or at atmospheric pressure. If evacuated, the system pressure should not be allowed to rise higher than fifty times the pressure caused by the tracer gas. This is because outgassing products usually found in an evacuated system (water, carbon dioxide and hydrocarbons) also absorb infrared radiation.

Kaufman (Ref. 137) has shown that the infrared detector may be used in locating leaks in buried water lines. Water lines were injected with nitrous oxide to a ratio of 0.37 volumes of N_2O to 1 volume H_2O. Leakage out of the lines was located by a detector probe as a result of the diffusion of gas to the surface of the ground.

2.9.6. GENERAL REFERENCES

1. Anonymous, "PE Tube Gas Detection," Electronic Industries, March 1944, pp. 108-109.

2. Balko, P.G., Bresky, D.R., and Coates, V.J., "IR Analyzer Solves Leak Detection Problem," ISA Journal, Vol. 5, No. 12, December 1958, pp. 44-47.

3. Kaufman, J.E., "Laughing Gas Shows Value in Spotting Pipeline Leaks," Oil and Gas Journal, Vol. 58, No. 8, February 22, 1960, pp. 100-102.

4. Neeley, A.H., "Nitrous Oxide for Leak Detection," Ohio Chemical Technical Information Series 1A, Ohio Chemical and Surgical Equipment Company, Madison, Wisconsin, May 1957.

Section 2.10

CHEMICAL INDICATOR

2.10.1. PRINCIPLES OF PROCEDURE

The chemical indicator procedure entails location of leaks by means of a color indication. This color may be due to chemical reaction of a gas or a liquid, or the inherent color of the indicator liquid as seen after it has permeated through a leak.

2.10.1.1. CHARACTERISTICS

The chemical indicator procedure may be subdivided into two distinct techniques:

- The chemical reaction technique, where the color indication is due to reaction between the leaking tracer and a developer.
- The penetrant technique, where the color is inherent to the penetrant and is evident at the leak site because of migration to that point.

2.10.1.2. APPLICABILITY

The chemical indicator procedure is fundamentally a leak location procedure. However, it may be used for leakage measurement, providing the entire surface of the object can be examined. The penetrant technique not only locates leaks, but also reveals cracks, surface imperfections, and bad welds. Consequently, it may be used to examine precision and high reliability equipment.

The chemical reaction method usually is not used on large systems because the cost of equipment and tracer gas is too high.

2.10.1.3. SENSITIVITY

The chemical indicator procedure is a static testing technique. Therefore, the longer the test is run, the more sensitive it becomes. Unfortunately, the tests are not quantitative, because the response is usually the size and color intensity of one indicated leak. However, the estimated sensitivity of the chemical reaction indicators is in the range of 10^{-3} atm cc/sec. The sensitivity of the penetrants is thought to be several decades greater. Sensitivity of the penetrants is determined by learning the time required for enough penetrants to flow through to be observed. When the physical properties of the penetrants are known, it is possible to calculate the sensitivity, using equations stated in literature (Ref. 34).

2.10.2. EQUIPMENT AVAILABLE

Most of the commercial equipment consists of chemical for use as penetrants and developers, and dyes to use in solution in the operating fluids. Major suppliers of these materials are listed in Table 24.

2.10.3. PROCEDURE ADVANTAGES AND LIMITATIONS

The major advantage of the precedure is relatively low cost, since no expensive equipment is required. The major disadvantage is that the procedure is qualitative, and the size of the leak cannot usually be determined. Each of the two techniques (i.e., penetrants and chemical reaction) contain advantages and limitations peculiar to the technique.

For example, the major limitation of the chemical reaction technique is the possibility that reactive chemicals might damage system parts. The penetrants technique, on the other hand, depending on the exact modification, will locate both leaks and surface imperfections which are not holes. Moreover, since the penetrants are liquids, clogging of leaks can occur (see Section 1.4.5.). This fact limits the utility of penetrants; they should not be used on systems which must be tested for very small ($< 10^{-6}$ atm cc/sec atm) leaks.

2.10.4. METHODS OF TESTING

2.10.4.1. PENETRANTS TECHNIQUE

The dye penetrant technique is the only leak testing method thoroughly described in literature (Ref. 138). The dye penetrant process is described briefly below.

Basically, the process consists of applying a liquid penetrant to the surface of a part. The penetrant is left on the part for a period of time and penetrates into any defects open to the surface.

After the penetrating period, the excess penetrant on the surface is removed and a developer is placed on the surface. The developer is a light-colored, powdered material (possible in suspension), which adsorbs the penetrant that had previously seeped into surface openings. The adsorbed coating forms indications which are much larger than the surface openings with which they are associated. These indications are evident against the background of the developer.

Penetrants are usually of two types: a dye which fluoresces under ultraviolet light and a colored dye which is highly contrasting against the metal or developer. The penetrant may be applied on the same surface as the developer, showing both leaks and surface imperfections, or the penetrant may be placed on one side of a surface and the developer on the other side. In this manner, only leaks through the part will be seen.

Most tests now use commercial penetrants which have the desired properties of high wetting power, low surface tension, low viscosity, and high indicating ability. However, it should be

Table 24

MANUFACTURERS OF CHEMICAL INDICATORS FOR LEAK DETECTION*

Manufacturer	Product	Type
George W. Gates and Company	Raymaster	soluble dye
Highside Chemicals, Incorporated	Trace	soluble dye
Magnaflux Corporation	Zyglo and Zyglo Penetrex	penetrant
Met-L-Chek Company	Met-L-Chek	penetrant
Shannon Luminous Materials Company Tracer-Technical Division	Tracer-Tech	penetrant
Sherwin Incorporated Belmont Chemicals Division	DUBL-Check	penetrant
Spectronics Corporation (Black Light Eastern Division)		soluble dye
Sperry Products (Division of Automation Industries Incorporated, Ritter Chemical Company, Incorporated, manufacturer)	Chex-All	penetrant
Testing Systems Incorporated	FLUORO-Finder	penetrant
Turco Products Incorporated Division of Purex Corporation	VISI-Chek DY-Chek	penetrant

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* More specific information on this equipment may be obtained from General Electric Company Report S-67-1013 (Ref. 186)

pointed out that the original penetrants were simply oils with whitewash as the developer. An oil stain on the whitewash indicated the presence of penetrant. Indication with such a penetrant was not nearly as good as the current processes.

Other fluids which will show an indication of the presence of a leak can be used, especially if they are inherent to the system. For example, many oils are fluorescent under ultraviolet light and their presence can be used as an indication of leaks.

Large systems are occasionally tested under stress by filling the systems with operating fluid under pressure and examining for leaks. Polito (Ref. 139) describes a water solution for such tests; it consists of 0.5 percent by weight monoethanolamine, 0.05 percent polyethylene glycol tert-dodecylthioether and 50 parts per million fluorescein. The amine is used in this case as an antioxidant and a corrosion inhibitor. The thioether is used as a surfactant to reduce the surface tension of the solution and to better wet the leak sides. The indicator is fluorescein, which glows brightly in ultraviolet light. After pressurization with the test liquid, the suspected leak areas on the outside of the system are examined with a portable ultraviolet light.

2.10.4.2. CHEMICAL INDICATOR TECHNIQUE

The chemical indicator technique depends on a chemical reaction producing a smoke or change in color at the leak site. This technique is not standardized, but consists of a variety of tests as described below.

2.10.4.2.1. Ammonia Gas Technique

Ammonia gas makes an excellent tracer because it is chemically basic, is not very toxic, and is only mildly corrosive. Its corrosive nature is strongly exhibited only on brass parts. Delafosse, Noe and Troadec (Ref. 140) describe studies to develop a variety of possible tests using the gas.

The ammonia tracer can be introduced as an anhydrous gas, or by placing a cloth saturated with ammonia solution within the pressurized space. The sensitivity of the tests will be dependent on the concentration of the gas.

2.10.4.2.1.1. Fumes Technique

Where leaks are present, the leakage of ammonia may be revealed by a white chemical fog on probing the atmosphere with a swab wetted with 0.1 N hydrochloric acid. Sulphur dioxide, such as from a sulphur candle, can also be used as the revealing reactant. A third gas which may be used for ammonia is carbon dioxide. It is not as sensitive as the other two, but has the advantage of being noncorrosive.

2.10.4.2.1.2. Indicator Solutions

There is a variety of solutions which may be used to give a color change with ammonia. These all depend on a change of pH of the indicator. Therefore, the pH of the solutions should be carefully adjusted so it is at the threshold of color change prior to use. The indicators must be damp for a color change to take place.

2.10.4.2.1.2.1. Phenolphthalein. A cloth dampened with the phenolphthalein solution and placed over the test area shows the location of leaks by a pink discoloration. The indicator consists of 1.0 percent phenolphthalein in a solution of equal amounts of water and ethyl alcohol.

A more elegant indicator is made of 5 parts 2 percent phenolphthalein in alcohol, 2 parts distilled water, 10 parts glycerin, and sufficient titanium oxide powder to thicken the solution to thin paint consistency. The titanium oxide powder serves the additional purpose of providing a white background against which the color change is contrasted.

2.10.4.2.1.2.2. Bromocresol Purple (Ref. 141). Forty mg. of bromocresol purple is dissolved in 4 ml. of 0.02 N sodium hydroxide. This is diluted to 100 ml. In a separate container, 10 grams of starch is added to 500 ml. of boiling water and stirred to form a slurry. The dye solution is added to the starch slurry. To this mixture is added 5 ml. of Tween 40 wetting agent and pH is adjusted to 5.0 with dilute hydrochloric acid. To test, a small portion of the indicating agent is put on a small piece of paper and the paper is moved rapidly through the air, horizontally about 12 inches away from an open top of a bottle of standard ammonia reagent. Immediate appearance of purplish color on paper shows the agent is ready for trial application. Otherwise, slight additional pH adjustment may be necessary.

2.10.4.2.1.2.3. Mixed Indicators (Ref. 140). It is possible to use alizarin between a pH of 5.5 and 6.8 or methyl red between a pH of 4.4 and 6.0 as an indicator. These show a color change better if a trace of methelene blue is added to the solution. Studies were made of the possibility of using calcium chloride in the indicator solution to complex the ammonia for increased retention. This should increase the sensitivity of the technique.

2.10.4.2.2. Carbon Dioxide

It is possible to fill the system with carbon dioxide and spray the outside with an agar-agar solution of indicator (Ref. 142). Agar-agar solutions can be "loaded" with sodium carbonate and phenolphthalein, yielding a bright cherry red spray solution, which turns white upon contact with carbon dioxide. This red solution can be sprayed onto the surface of a test piece, to yield a stable red film. Carbon dioxide causes a white spot to be produced in the agar-agar film at points of leakage. Studies indicate that the volume of agar-agar film which is discolored is directly proportional to the amount of carbon dioxide that enters the film. Therefore, the area of the discoloration is directly proportional to the amount of carbon dioxide which has escaped.

The spray solution consists of (parts by weight): agar-agar, 1.0; distilled water, 40.0; anhydrous sodium carbonate, 0.10; and phenolphthalein, 0.15. It is important to obtain the proper viscosity of agar-agar solution, which should be between 8 and 10 millipoises at the spraying temperature.

When the spray solution is prepared, the dry powders (agar-agar, sodium carbonate, and phenolphthalein) should be blended thoroughly in proper proportions. To the dried powders, the cooling distilled water should be added, with stirring to disperse the solid constituents. The resulting mixture should be heated with constant stirring, to between 96 and 98 C on either a hot plate or a steam bath.

When the solid is completely dissolved and a clear solution is obtained, it should be allowed to cool to between 65 and 70 C. At this temperature it may be stored in a closed glass solution, provided the container is sealed to exclude air and small quantities of carbon dioxide.

To perform the test, the hot agar-agar solution from the storage container (at 65C to 70C) is poured into the preheated sprayer bottle. The compressed air supply should be preheated in a heat exchanger to the spraying temperature. The spray nozzle should be held approximately 2 feet from the test piece. A single coating should be applied in one pass. Multiple coatings should be avoided. Spraying should always be done in a horizontal direction, never vertically.

After testing, the agar-agar film can be removed completely, leaving the surface of the instrument clean and dry. A jet of high velocity air from an air nozzle is applied to the surface. The air jet will lift the film from the surface, leaving it in good condition.

2.10.4.2.3. Hydrogen Sulfide

Hydrogen sulfide has been used to locate leaks in containers (Ref. 143). The indicator was a 5 percent solution of stannous chloride. The location was by means of a brown stain of stannous sulfide. Because of the poisonous nature of hydrogen sulfide, this is not a very popular method.

2.10.4.2.4. Miscellaneous Methods

A variety of other reactions may be used for leak location. For example, precipitates may be formed by hydrogen sulfide with silver or lead salts (Ref. 141). Leaks in gas pipelines were located by the reaction of silver salt solutions with acetylene gas (Ref. 144). Water has been used as a penetrant (Ref. 145) using anhydrous copper sulfate as a developer. Anhydrous copper sulfate turns blue in the presence of water.

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Section 2.11

HIGH VOLTAGE DISCHARGE

2.11.1. PRINCIPLES OF PROCEDURE

High-voltage discharge is used in two techniques for leak testing:

White-spark which is observing the spark jumping to the leak when the probe tip is 1 cm or so from it

Color-differentiation which is observing a change in color of the glow discharge produced by probe gases or vapors entering the leak

2.11.2. WHITE SPARK TECHNIQUE

2.11.2.1. CHARACTERISTICS

Leaks in glass vacuum systems are readily located by passing the high-potential electrode of a spark coil, such as the Tesla coil, over the surface of the glass. When the probe comes near a pinhole, or crack, a high-frequency spark jumps from its tip to the flaw. The flaw then glows white against the reddish-purple glow discharge produced by the Tesla coil in the residual air within the system. This discharge occurs when the air pressure lies between 10 and 10^{-2} torr. The spark jumps to the leak when the probe is about one centimeter from the leak.

2.11.2.2. APPLICABILITY

High-voltage discharge is primarily a method of leak location and is applicable to evacuated systems. It is employed in the detector probe mode.

The white-spark technique is only applicable where no metal exists since the spark from such a coil will ground through metal parts. The spark tip cannot be brought closer than several centimeters from metal parts, since the spark will jump to the metal.

2.11.2.3. SENSITIVITY

The white-spark technique is qualitative, but will probably detect leaks as small as 10^{-4} atm cc/sec. The ultimate size of detectability is dependent on leak geometry.

2.11.2.4. TECHNIQUE LIMITATIONS

Spark coils cannot be used on all-metal systems. However, they can be quite useful on all-glass systems, or even on metal systems containing glass parts.

Upon continual exposure, the high-voltage spark may puncture thin glass walls. The probe should, therefore, be slowly moved rather than held in one place. In the same manner, a spark might score the barrel of a Teflon stopcock and rupture plastic or rubber gaskets.

2.11.2.5. METHOD OF TESTING

The technique consists of evacuating the system to a pressure between 10^{-2} and 10 torr and the probing of the suspected areas with a high-voltage coil. The rate of passage of the probe may be as high as several yards per minute. Little skill is required for this technique. The reliability in testing depends on operator skill in probing the complete system.

2.11.2.6. READING INTERPRETATION

If the probe passes within 1/4-inch of the leak, a spark will jump to the leak and a very bright spark may be observed passing into the system through the leak. The system will at that time show a glow with a purple discharge, indicating the presence of nitrogen.

This technique is not quantitative, but the physical size of a leak may be compared with other leaks located in the system.

2.11.3. COLOR-DIFFERENTIATION TECHNIQUE

2.11.3.1. CHARACTERISTICS

The color-differentiation technique involves observing change in color of a high-voltage glow discharge produced by probe gases or vapors entering the leak. A spark coil can be used to excite a visible discharge (if the pressure in the system lies within the range 10^{-2} to 10 torr) while a tracer gas, such as carbon dioxide, or a volatile liquid, such as benzene, acetone or methyl alcohol, is applied to the surface of the vacuum system. When the tracer gas or vapor enters the system through a leak, the color of the discharge changes from the reddish-purple of air to a color characteristic of the tracer material. For the liquids mentioned above, the color would be grayish-blue while carbon dioxide gives a bluish-green glow.

2.11.3.2. APPLICABILITY

The color-differentiation technique is primarily a method of leak location and is applicable to evacuated systems. It is always employed in the tracer probe mode.

This technique can be used for leakage measurement, if a leak of a sufficient size exists. It can also be used as a static leakage measurement technique. The color differentiation technique can be used on metal systems, providing the system contains an area (such as a glass tube) in which a visible discharge can be initiated. The technique can also be used to measure leakage in a sealed,evacuated glass component.

2.11.3.3. SENSITIVITY

The color differentiation technique will detect a gas composition change of approximately 10^{-2} torr. The sensitivity of the technique is dependent on the pumping speed of the system (in the discharge area), using the formula:

$$Q = PS \qquad \text{Equation 2.11-1}$$

where Q = detectable leakage
P = detectable gas composition change, expressed as a partial pressure
S = pumping speed of the system

2.11.3.4. TECHNIQUE LIMITATIONS

Part of the envelope has to be transparent so that the color of the discharge may be seen. Since the procedure depends on detecting total pressure build-up, the time that the sample has to be left standing prior to testing increases with an increase in desired sensitivity. Any gas or liquid whose glow discharge color is different from the background discharge color may be used as a tracer. However, gasoline, benezene, pyridine and solutions containing nitrogen compounds should not be used because they adhere to glass.

The glass may be punctured either by holding the spark coil tip too long at one spot or by using too high a voltage. A spark gap (1/4 to 1/2 inch) in parallel with the high-voltage tip and ground will eliminate the latter difficulty.

2.11.3.5. METHODS OF TESTING

The spark coil tip is kept on one glass section of the system. Preferably this section will be between the diffusion pump and the fore pump in order to have a high enough pressure to maintain a glow discharge. The nature of this discharge will depend on the pressure and on the gases in the system. The color is characteristic of the gases present. For air, this color is reddish or purplish. The exact color (as for other gases) depends to some extent on the glass used in the system. Pyrex or soda glass will show a yellow-greenfluoresence while lead glass shows a blue fluoresence. The probe material used can be a gas or a liquid. Some materials that are commonly used are: illuminating gas, ether, and carbon dioxide. With the first two materials the discharge takes on a grayish-blue appearance. This is similar to the characteristic color of carbon dioxide (see Table 25) but, possibly due to fluorescence of the glass, the color is often reported as bluish-green.

Instead of a spark coil, a discharge tube (like a Geissler tube) can be used. In order that the pressure in the discharge tube be sufficiently high to obtain a glow discharge, the discharge tube is usually connected to the vacuum system between the diffusion and mechanical pumps. Furthermore, by locating the tube here, advantage is taken of the magnified pressure change on the

Table 25

DISCHARGE COLORS IN GASES AND VAPORS AT LOW PRESSURES (REF. 54)*

Gas	Negative Glow	Positive Column
Air	Blue	(Reddish)
Nitrogen	Blue	Yellow (red gold)
Oxygen	Yellowish white	Lemon
Hydrogen	Bluish pink (bright blue)	Pink (rose)
Helium	Pale green	Violet-red
Argon	Bluish	Deep red (violet)
Neon	Red-orange	Red-orange (blood red)
Krypton	Green	--
Xenon	Bluish white	--
Carbon monoxide	Greenish white	(White)
Carbon dioxide	Blue	(White)
Methane	Reddish violet	--
Ammonia	Yellow-green	--
Chlorine	Greenish	Light green
Bromine	Yellowish green	Reddish
Iodine	Orange-yellow	Peach blossom colored
Lithium	Bright red	--
Sodium	Yellowish green (whitish)	Yellow
Potassium	Green	Green
Mercury	Green (goldish white)	Greenish blue (greenish)

-- indicates no distinctive color given.

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* Reprinted with permission from Vacuum Technology, A. Guthrie, John Wiley and Sons, Inc., New York, 1963, p. 456 .

backing side produced by a pressure change on the high vacuum side of a pumping system. The pressure range required for a discharge is about the same as for a spark coil, i.e., 10^{-2} torr to a few torr. Design of the tube is not critical -- a simple glass tube with two sealed-in electrodes and a source of high voltage (d-c or high frequency). A discharge tube of this type is manufactured by Edwards High Vacuum, Inc., Grand Island, New York.

Once voltage has been applied to the tube and a glow discharge is evident, leak hunting can proceed as when employing a spark coil. The probe materials indicated for a spark coil can be used. Carbon dioxide appears to be one of the most useful probe gases. Molecules that are heavy and have a high viscosity are not very suitable for leak location.

The problem of two leaks of more or less equal size, which occurs rarely but must be considered with complicated apparatus, is fortunately solved by the fact that in a mixture of more or less equal parts of carbon dioxide and air, the color of the positive column tends toward that of carbon dioxide. So if there is carbon dioxide on either of the leaks, that leak is located and can be stopped, and the other can be found later.

Although the sensitivity and reliability are dependent on operator skill, recognition of a leak is relatively easy. The operator has to be knowledgeable in probing all possible leakage areas and be able to recognize the indication of the leak. Probing of the system with tracer fluid may proceed at a rate of approximately one foot per minute. Because of the high molecular weights of some of the tracers, considerable delay may occur between the time the tracer enters the vacuum system and passes into the discharge area.

The entire system may be checked for leakage by hooding with tracer gas. The hooding techniques are described in Section 1.5.

Pirani and Yarwood (Ref. 53) describe a method of leak-checking a sealed system. The system is evacuated to a pressure of at least 10^{-4} torr, outgassed, and filled with neon gas to a pressure of 1 torr. After waiting for the required time, a glow discharge is induced in the envelope with a Tesla coil and the color compared with a standard which is known not to leak. The checking method is rapid, approximately 15 seconds per sample. The operator is required to have a skill in distinguishing small changes in color.

2.11.3.6. READING INTERPRETATION

Interpretation of leakage is made by analysis of any color change. Guthrie (Ref. 54) observes that the applicability and sensitivity may be increased by observation of the glow discharge with a spectroscope for specific spectral lines of the leaking gas. Lloyd (Ref. 146) describes a circuit for converting a discharge tube to an audible detector. As shown in Figure 74, the circuit consists of a relaxation oscillator. The oscillation frequency is made audible by a loud-speaker. The frequency changes with pressure or gas composition charge.

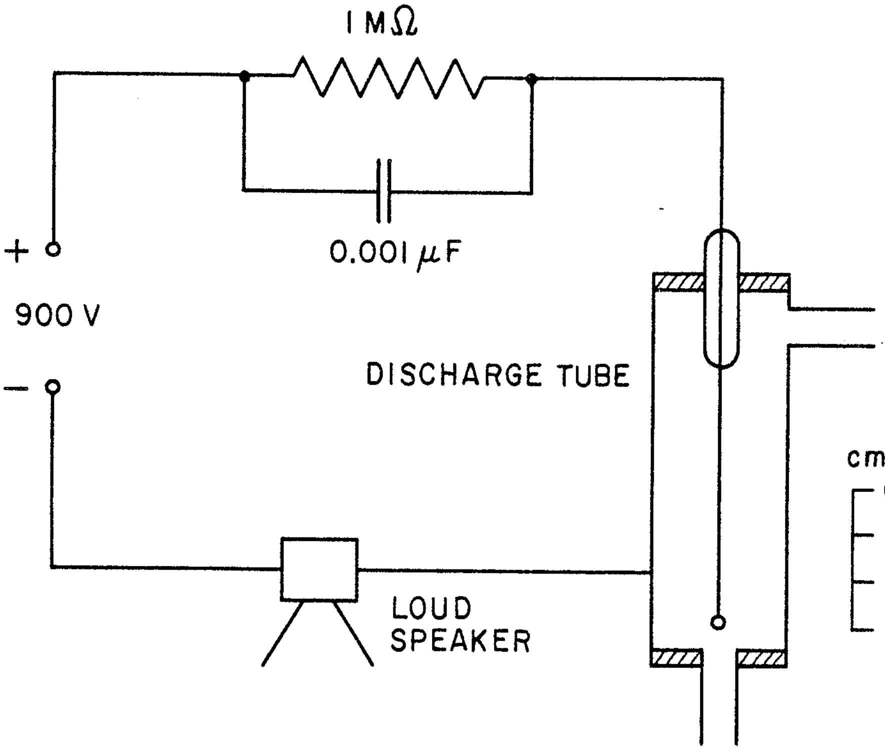

Figure 74. Discharge Tube Audible Leak Locator. (Reprinted, with permission, from J.T. Lloyd, Journal of Scientific Instruments, Vol. 27, 1950, p. 76; The Institute of Physics and The Physical Society, London.)

This technique does not readily lend itself to measuring the leak-rate. An estimate within two or three decades may be made by estimating the degree of color change against various standard leaks. The sensitivity of this technique may be increased by optimizing the pumping speed, the trade off being an increase in response time. Some increase may be gained by proper choice of the tracer gas used. Webster (Ref. 147) states that the discharge must be at high enough pressure to show a positive column, because the difference in color between air and any other gas shows so much better in a positive column than in a cathode-ray discharge.

For the smallest leaks, a discharge tube and a stopcock should be connected to the line between the forepump and the diffusion pump. When the stopcock is closed, gas coming through the diffusion pump accumulates in the discharge tube. If the discharge tube is clean, it is possible to distinguish between tracer gas and air in the accumulated gas. Traces of gas on the walls of the discharge tube will interfere seriously with the test if they are freed. So, if it takes longer than a few minutes to make a test, the discharge should not be run at all until it is time to test it. With this precaution, however, one may make conclusive tests and locate a leak definitely, even when it takes hours to accumulate enough gas.

2.11.4 EQUIPMENT AVAILABLE

The source of high voltage is usually a Tesla coil. The Tesla coil, producing high-frequency high-voltage (about 40 kilovolts, a-c), is usually packaged so that it may be handled by its insulated body while the metal tip is at the high potential. The specifications of such a coil are listed in Table 26. Other sources of high voltage may be used in place of the Tesla coil. Dowling (Ref. 148) has pointed out that the high-frequency transformer used with television tubes can be used as a spark coil. He describes a suitable circuit for this device.

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Section 2.12

VACUUM GAGE RESPONSE

This procedure is based on the principle that most vacuum gages have a pressure response dependent on gas composition. If the composition of gas in a system changes, the reading on the detector reflects this change. Leak location therefore consists of spraying a tracer gas on the suspected leak and observing any response of the gage to the tracer gas that enters the system through the leak.

2.12.1. PRINCIPLES OF PROCEDURE

2.12.1.1. CHARACTERISTICS

Most pressure gages do not actually measure pressure; instead, they measure a property of the gas, such as thermal conductivity or ionization efficiency. These properties are proportional to the gas concentration in a system. The tracer gas has a particular, known value for these properties. Consequently, a pressure gage can detect a difference in gas composition brought about by the introduction of tracer gas to a system.

In the evacuation mode, the system is evacuated and the suspected leak is sprayed with tracer gas. Pressure gage response to the tracer gas indicates that a leak has been located. In the pressurized mode, the system is pressurized with tracer gas and a thermal conductivity detector probe is used for leak location.

2.12.1.2. APPLICABILITY

This procedure is extremely popular for leak location on vacuum systems, because a gage is usually present on the system. The only other requirement for the test is tracer gas. This procedure was once widely used for leak testing of components, but with the advent of more specific and more sensitive leak detectors, it has fallen into disuse.

It is possible to use this procedure for leakage measurement by stabilizing the system, hooding it, and introducing tracer gas into the hood. However, the response is not generally quantitative and is too nonspecific to be of any value. It is always questionable whether the pressure gage response is due to increased concentration of the tracer gas or some other factor.

2.12.1.3. SENSITIVITY

Sensitivity of the procedure is dependent on the sensitivity of the gages being used, and on the pumping system on which they are mounted. The sensitivity is ordinarily in the range of 10^{-4} - 10^{-6} atm cc/sec. This can be increased by modifications that increase specificity of the gages.

Table 26

CHARACTERISTICS OF HIGH VOLTAGE DISCHARGE COILS

General Characteristics	Average Values
Tracer Gas	None for glass systems with tracer fluid-carbon dioxide, alcohol, ether or methane
Power Requirement	115 volts, a-c, 0.1 ampere
Output	50 kilovolts, 4 microvolts
Size	One foot long, 2 inches diameter
Weight	2 pounds
Manufacturers:	Chemical supply houses; listed under Vacuum Tester, High-frequency Tester, Tesla Coil, Spark Coil

In the tracer probe technique, the size of the leak which can be detected by this procedure is dependent on the pumping speed of the system. As a first approximation, this procedure can detect a pressure change of one-fiftieth the pressure in the system. Smaller leaks, i.e. leaks that do not contribute more to system pressure or composition, will not be detected by this procedure.

The thermal conductivity detector in the pressurizing mode has a sensitivity of approximately 10^{-4} atm cc/sec.

2.12.2. EQUIPMENT AVAILABLE

Many gages which use the thermal conductivity principle (e.g. Pirani and thermocouple gages) have a leak checking position on their meter scale. In this position, the indicator needle is in the center of the meter scale and operates at high sensitivity. Any movement of the needle indicates a leak.

Instruments are sold by several companies (Table 27) which amplify the change of pressure indication of gages. These instruments are used to simplify the leak location procedure.

Table 28 lists the characteristics of ionization gages and manufacturers currently making them. These gages are specifically modified for leak testing.

Table 29 lists the characteristics and manufacturers of detectors using the thermal conductivity principle. These instruments are used for leak location in the pressurizing mode.

2.12.3. PROCEDURE ADVANTAGES AND LIMITATIONS

In the evacuation mode, the major advantage of this procedure is that no additional equipment is necessary. Leak location may be performed using gages already on the system. The procedure is inexpensive and no highly trained personnel are necessary.

The major limitation of this procedure is that it is most directly applicable to the major leaks in the system. It would be very difficult (if not impossible) to locate a leak one-hundreth the size of the total system leakage.

This procedure is dependent on a constant pressure in the system. If the system pressure varies for reasons unrelated to testing, leak location using this procedure is impossible.

In the pressurizing mode, this procedure is also an inexpensive method of leak location. The equipment is portable and may be used on a variety of gases in the system.

The sensitivity of this procedure is relatively low (10^{-4} atm cc/sec) and the necessary instruments cannot be used in a contaminated atmosphere because they will respond to other gases present in the air. Therefore, these instruments are not widely

Table 27

CHARACTERISTICS OF PRESSURE INDICATION LEAK DETECTORS
(Ref. 186)

General Characteristics	Average Values
Sensitivity	1 x 10^{-6} mm Hg
Tracer Gas	None
Output	Meter
Power Requirement	115/230 volts, 60 cycles 28 volts DC
Size	
Sensor	4 inches x 4 inches x 3 inches
Electronic Unit	8 inches x 8 inches x 12 inches
Weight	4 to 40 pounds
Price	Up to $2500

Manufacturers

Accessory Controls and Equipment Corporation
Datametrics, Inc.
MKS Instruments, Inc.
Trans-Sonics, Inc.

Table 28

CHARACTERISTICS OF IONIZATION GAGE LEAK DETECTORS

(Ref. 186)

General Characteristics	Average Values
Sensitivity	3×10^{-8} std cc/sec at 5 mm Hg
Tracer Gas	Specific Detectors for either Hydrogen, Helium or Halogenated Hydrocarbon
Output	Meter, Tone, or Light
Power Requirement	120 Volts AC or Battery
Size	From 1.75 inches x 3 inches 4 inches to 19 inches x 10 1/2 inches x 16 inches
Weight	1 to 18 pounds
Price	$250 to $1240

Manufacturers

Canal Industrial Corporation
Edwards High Vacuum, Inc.
Mine Safety Appliances Company

Table 29

CHARACTERISTICS OF THERMAL CONDUCTIVITY LEAK DETECTORS

(Ref. 186)

General Characteristics	Average Values
Sensitivity	1 x 10^{-3} std cc/sec
Tracer Gas	Bi-gaseous Mixtures
Output	Meter, Tone, or Light
Power Requirement	115/230 volt, 60 cycle
Size	About 3 inches x 7 inches x 9 inches
Weight	4 to 8 pounds
Price	$165 to $2840

Manufacturers

Bacharach Industrial Instrument Company
Consolidated Electrodynamics Corporation
Davis Emergency Equipment Company, Inc.
Devco Engineering, Inc.
Edwards High Vacuum, Inc.
Gas Analysis Systems, Inc.
Gas Purifying Materials Company, Inc.
Matheson Company, Inc.
Teledyne, Inc.
Uson Corporation

used where welding (inert gases), cleaning (solvent fumes), brazing (combustion products), or painting (paint solvents) operations are performed.

2.12.4 PRINCIPLES OF OPERATION

This discussion covers the vacuum gage procedure of leak location in the evacuation mode. In general terms, the procedure is to expose the surfaces of an evacuated system to a tracer gas. If a leak is present, this gas enters the system and displaces any residual gas in the neighborhood of the gage. There are several variations of this procedure, depending on the detector used and the method of increasing specificity, but the various techniques have a number of features in common. Such a theory was developed by Blears and Leck (Ref. 14) and is discussed here.

Consider a system being tested as illustrated in Figure 75. If a leak is present in the system, system pressure will be:

$$Q = P_2 C \qquad \text{Equation 2.12-1}$$

where Q = leakage into the system
P_2 = pressure in the system
C = conductance of the tubing leading to the pumps

The conductance of the tubing usually limits the pumping speed. In this equation, conductance is based on the effective pumping speed at the site of the gage.

If the leak is laminar (See Section 1.4.1.1.1.), leakage will be inversely proportional to the viscosity of the gas:

$$Q = \frac{C_L\,(P_1^2 - P_2^2)}{\eta_a} \qquad \text{Equation 2.12-2}$$

where C_L = Conductance of the leak
P_1 = Pressure outside the system (one atmosphere)
η_a = Viscosity of air

Equation 2.12-2 can be simplified to:

$$Q_A = \frac{C_L\,P_1^2}{\eta_a} \qquad \text{Equation 2.12-3}$$

since P_1 is much greater than P_2.

If concentration of the tracer gas is x atmospheres ($x < 1$),

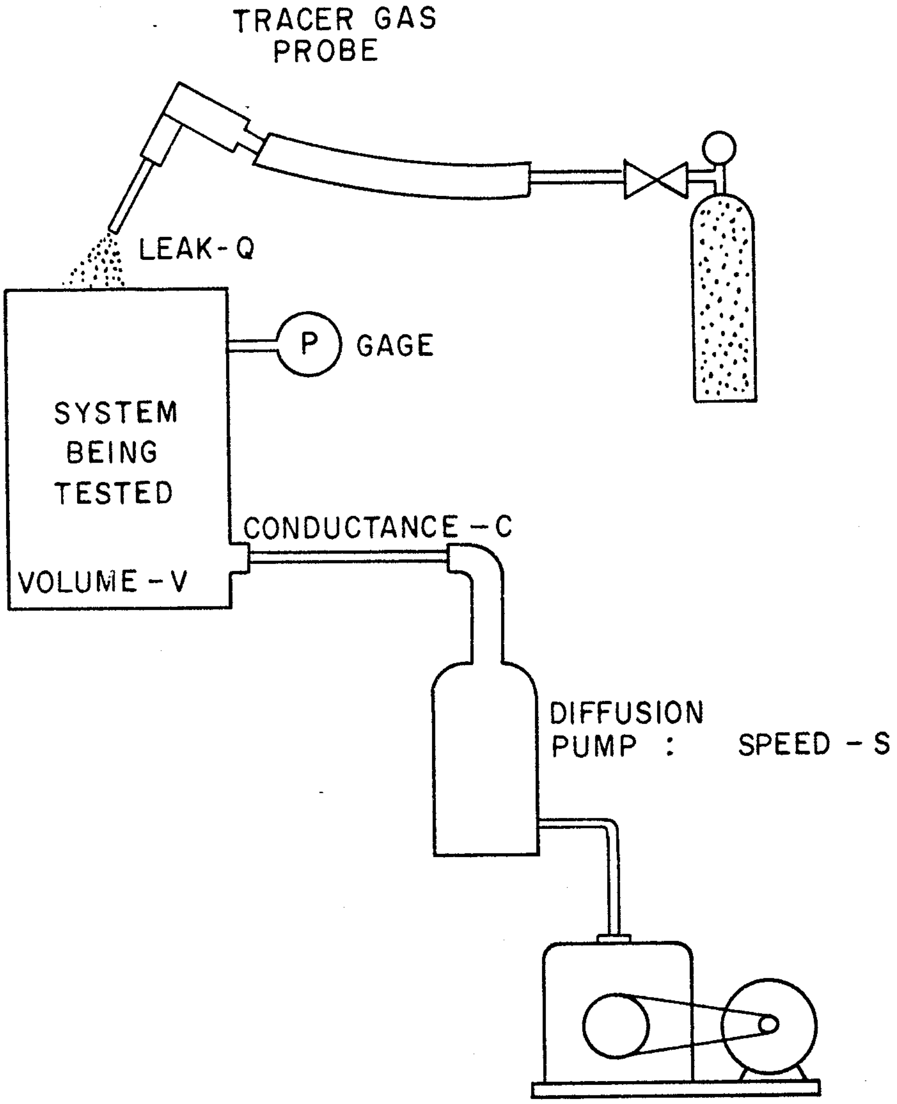

Figure 75. Idealized System for Vacuum Gage Response Testing.

the concentration of air is 1-x. If viscosity of the tracer gas is the same as air, then tracer gas leakage will be the same as air leakage.

It then follows from Equation 2.12-1 that:

$$Q = C_a P_{2a} + C_x P_{2x} \qquad \text{Equation 2.12-4}$$

where subscript a denotes air and subscript x denotes tracer gas.

In laminar flow, a concentration of gas just outside a leak means the same concentration will enter the system (see Section 1.5.1.5.1.). It then follows that:

$$\frac{C_L P_1^2}{\eta_x} (1-x) = C_a P_{2a} \qquad \text{Equation 2.12-5}$$

and

$$\frac{C_L P_1^2}{\eta_x} x = C_x P_{2x} \qquad \text{Equation 2.12-6}$$

Total pressure in the system can be obtained by an addition of Equations 2.12-5 and 2.12-6:

$$P_{2a} + P_{2x} = \frac{C_L P_1^2}{\eta_x} \left(\frac{1-x}{C_a} + \frac{x}{C_x} \right) \qquad \text{Equation 2.12-7}$$

The change in pressure due to the presence of tracer gas may be obtained by subtracting Equation 2.12-5 from 2.12-7

$$\Delta P_2 = C_L P_1^2 \left[\frac{1}{\eta_x} \left(\frac{1-x}{C_x} + \frac{x}{C_x} \right) - \frac{1}{\eta_x C_a} \right] \qquad \text{Equation 2.12-8}$$

When pure tracer gas surrounds the leak, x = 1 and Equation 2.12-8 becomes:

$$\Delta P_2 = C_L P_1^2 \left(\frac{1}{\eta_x C_x} - \frac{1}{\eta_a C_a} \right) \qquad \text{Equation 2.12-9}$$

If the sensitivity of a gage to air is given K_A and K_x for the tracer gas, the change of gage reading ΔG on applying the tracer gas is:

$$\Delta G = C_L P_1^2 \left(\frac{K_x}{\eta_x C_x} - \frac{K_a}{\eta_a C_a} \right) \qquad \text{Equation 2.12-10}$$

From the above discussion, it follows that the maximum sensitivity will be obtained when the test includes:

1. Complete coverage of the leak by the tracer gas.
2. High sensitivity of the gage to the tracer gas i.e. large K_x.
3. Low value of viscosity (η_x) of the tracer gas.
4. A small value of C_x, the effective pumping speed of the tracer gas.

Since C_x depends on the conductance of the tubing, and that is inversely proportional to the square root of the molecular weight of the gas involved (see Section 1.4.1.1.2.), the tracer gas should have a high molecular weight.

It is possible to use a small pump to evacuate the system being tested. However, pressure fluctuation will be created by a small pump. The pumping speed is more effectively reduced by using a large pump and a small conductance. In practice, a diffusion pump is preferable to a mechanical pump, because a diffusion pump produces less pressure fluctuation. Of course on a system with built-in pumps, the pumping speed cannot be altered for leak location, so the sensitivity is fixed by system design.

The above derivation of gage response assumes that flow through the leak is laminar. In small leaks (10^{-6} atm cc/sec), the flow will be molecular. In molecular flow, the leakage is inversely proportional to the square root of the molecular weight of the leaking gas. The same relationship applies to the conductance which determines the pumping speed of tublation. It can therefore be shown in a manner similar to the derivation of Equation 2.12-9, that if the leakage into the system is molecular, and the pumping speed determined by the tubulation leading to the pump, the pressure in the system is independent of the property of the leaking gas. The gage response is then dependent only on the relative sensitivity of the gage to the tracer gas as compared to air.

Since there are a variety of factors involved in choosing a proper gas and gage combination, it is often easier to experimentally determine the sensitivity factor. Blears and Leck (Ref. 14) define this factor as:

$$\varphi = \frac{\text{pressure caused by tracer gas on the leak}}{\text{pressure on system with air on leak}}$$

The experimental values of this factor are shown on Table 30.

The minimum detectable leak can be determined by:

$$Q_{min} = \frac{\Delta P_{2a} C_a}{\varphi} \qquad \text{Equation 2.12-11}$$

where ΔP_{2a} = smallest measurable air pressure variation
Q_{min} = smallest measurable leakage
C_a = pumping speed for air at the gage

Table 30

LEAK TESTING SUBSTITUTION FACTORS φ (REF. 14)*

Tracer Gas	Hot Cathode Ionization Gage	Pirani Gage
Butane	10	1
Diethyl ether	5	0.7
Carbon dioxide	1	0.3
Carbon tetrachloride	1	0.05
Benzine	0.3	0.1'
Hydrogen	0.4	0.4
Coal gas	0.25	0.25

(*Reprinted, with permission, from J. Blears and J. H. Leck, Journal of Scientific Instruments, Vol. 28, 1951, Supp. No. 1, p.20; Institute of Physics and Physical Society.)

It is apparent from the above discussion that the minimum measurable leakage will be within a decade of the minimum measurable pressure change, multiplied by the pumping speed at the pressure measurement site. In designing this type of leakage measurement, the response time of the system must also be taken into account (Section 1.3.4.2.2.). The pumping speed used in this section is the pumping speed at the site of the gage. Thus the location of the gage affects the sensitivity. If the gage is connected by way of a restriction, it will be difficult to detect small leaks anywhere except near the gage itself.

2.12.5 TESTING

The testing techniques can be divided into three categories: sealing techniques, thermal conductivity techniques, and ionization efficiency techniques. These in turn have various subdivisions which are discussed individually on the following pages.

2.12.5.1. SEALING TECHNIQUE

The sealing technique involves gradually covering outside parts of a system being evacuated with some material that will seal the leak. Once the leak has been covered, the pressure will drop. In this way leaks can be located and permanent repairs made. The procedure is to paint, brush, or spray the sealing substance over various parts of the system until a change in pressure is noted. Either a thermal conductivity or an ionization gage may be used, the choice being dictated by the pressure.

The sealing substance may temporarily or permanently seal the leaks. Some permanent sealants are glyptal lacquers, shellac in alcohol, and vacuum cements that are liquid at room temperature, such as Eastman Kodak Resin 910 or cellulose acetate. Some temporary sealants are water, acetone, and alcohol.

Two effects result from the use of a liquid. First, after the initial closing of the leak, the pressure will drop. Second, as the vapor enters the system, the gage will show a change in pressure. The change in pressure will depend on the nature of the vapor and on the type of gage. The vapors from the above mentioned solvents are readily condensable. Consequently, all gages used with a cold trap will show a pressure drop when a leak is covered by a liquid. The particular liquid used (no cold trap) will determine whether the gage shows an increase or decrease in pressure. Alcohol, acetone, and ether, which are commonly used probe liquids, all show an increased pressure reading with an ionization gage.

The temporary sealing substances are quite effective for all sizes of leaks except the very smallest. If a very small leak is sealed with a temporary sealant, it will open again at some inopportune time. Therefore, this technique is not recommended if the small leaks have to be located and permanently repaired.

For very small leaks, a permanent sealing material works satisfactorily. However, in repairing large leaks the material is drawn into the vacuum system and a seal cannot be obtained. Although the permanent sealing substances give fairly satisfactory results with leaks in metal plates, in soldered, brazed, and welded joints, and in glass systems, they are not as satisfactory as a final repair obtained by reworking the material of the vacuum system by soldering or welding. The permanent sealing substances make further reworking of the glass or metal very difficult.

2.12.5.2. IONIZATION EFFICIENCY TECHNIQUE

2.12.5.2.1. General Techniques

The ionization efficiency technique consists of spraying the suspected leak area with tracer gas and observing any pressure change indicated on an ionization gage. Any gage which measures ionization of the gas may be used. This implies either a hot cathode gage, a cold cathode gage, or even an ion pump.

In ionization gages, ionization current depends on the probability of ionizing collisions. With all other variables held constant this varies from one gas to another. When the tracer gas is applied to the leak, some of the gas in the gage is replaced by tracer gas giving a lower or higher ionization current than the steady ionization current due to the prevailing pressure in the system.

It is desirable that the tracer gas have as different an ionization efficiency from the background gas (air) as possible. In general, gage sensitivity increases with the number of electrons in the molecule (Ref. 149). Examination of ion gage sensitivities suggests that the best gases for this technique are either the low molecular ones (hydrogen, helium, neon), or the high molecular vapors (acetone, ether and alcohol).

In using the vapors, care must be taken that they do not plug the leak. In some cases, response may be delayed because of adsorption on the leak surface (Section 1.4.4.4.)

As long as the leaks being located are the ones that limit the system pressure, this technique may be applicable to very low pressures and/or very low leak rates. Alpert (Ref. 150), for example, used it for location of leaks in the first measured ultra high vacuum systems. He found this technique to be more sensitive than the mass spectrometer leak detector.

In order to obtain adequate sensitivity with this method, the background ionization current may be nulled using a sensitive difference amplifier or a galvanometer with backing-off voltage control, so that very small changes in ionization current are detected. An example of a circuit for such testing is shown in Figure 76 (Ref. 96). The indicating instrument has been replaced with a potentiometer. The null balance instrument can be a galvanometer or a sensitive microammeter, either of which is provided with a suitable shunting circuit. In a stable vacuum, a constant current flows through the gage tube and the potentiometer, creating a steady voltage drop across the potentiometer. The battery provides a reference voltage and the potentiometer can be adjusted to give a null indication on the galvanometer. The shunting switch is left closed until this adjustment is made.

Care must be taken that the tracer gas does not permanently react and change the gage sensitivity. For example, Varicak (Ref. 151) found that after carbon dioxide had been applied for some time, sensitivity of a Penning Gage changed. The discharge current decreased about 30-40 percent, probably because of a film of carbonates on the electrodes.

This general method can be modified in several ways. Instead of an ionization gage, an ion pump may be used. Selectivity of the gage to the tracer gas may be increased by use of a double gage setup, where a gage is positioned so that it is selective only to the tracer gas. Another modification of this technique is to use the poisoning effect of oxygen on the generation of electrons on a tungsten filament.

2.12.5.2.2. Ion Pump Technique

The cold cathode, gas discharge ion pumps are convenient instruments for leak location. An ion pump acts not only as a pump, but also as an effective pressure gage, since the pump current is proportional to the number of molecules being pumped. The pump current is also dependent on the ionization efficiency of the gas molecules being pumped. The pumping speed is dependent on their chemical reactivity rather than their molecular weight. Therefore, the response of an ion pump to a tracer gas will be different than an ionization gage.

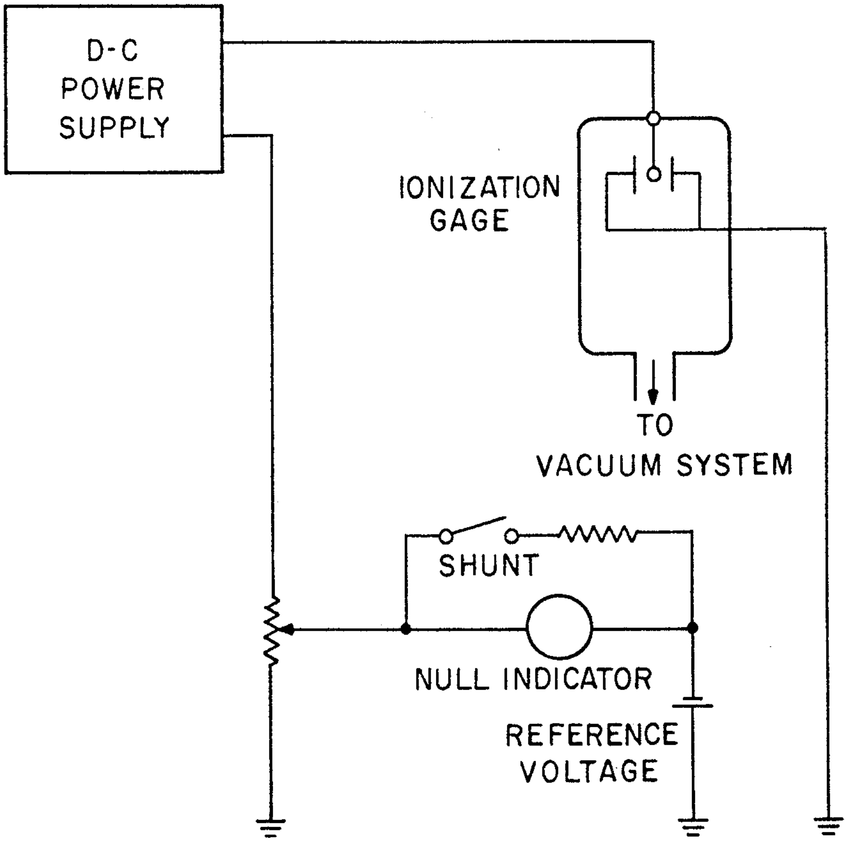

Figure 76. Null Balance Circuit for Leak Location. (Reprinted with permission, from W.E. Briggs, A.C. Jones, and J.A. Roberts, "Leak Detection Techniques", 1958 Fifth National Symposium on Vacuum Technology Transactions, Symposium Publications Division, Pergamon Press, New York)

Ackley and his associates (Ref. 152) made a theoretical study of the response of an ion pump to various probe gases. They derived response curves at various times. Some of these responses are shown in Figure 77. As may be seen from those curves, the response differs with time, not only in magnitude, but also occasionally in size. This has been observed by others (Ref. 153). The best gases for leak location using an ion pump seem to be argon, oxygen, and carbon dioxide.

Barrington (Ref. 154) estimates that leaks in the 10^{-11} atm cc/sec range may be located with an ion pump. This is a conservative estimate of the sensitivity; the current changes being measured are several orders of magnitude greater than the corresponding mass-spectrometer ion currents.

A novel leak detector has been described by J. R. Young (Ref. 155). With the system shown in Figure 78, the procedure was to evacuate an ion pump and keep it operating at low pressure with the valve V_1 closed. The system to be leak-tested was first evacuated by a mechanical pump to a pressure of 1-10 x 10^{-3} torr. Valve V_1 was then opened and V_2 closed and an equilibrium pressure was reached in a few minutes. When the leak was probed with argon, the ion pump current increased rapidly, presumably due to the low speed of the pump for argon. Probing with hydrogen and oxygen caused a reduction in pressure, since these gases are pumped more rapidly than air. Helium was also used as the search gas but the sensitivity was lower than for argon.

Leaks as small at 10^{-10} torr l/sec were located using this technique. Leaks between 10^{-3} and 10^{-5} torr l/sec could be located by partially opening V_1 and by having V_2 opened sufficiently to avoid a pressure increase in the system during the leak testing procedure. Leaks of 10^{-5} to 10^{-8} torr l/sec could be determined a few minutes after opening V_1 and closing V_2. Leaks smaller than 10^{-8} torr l/sec required a longer time depending on the volume and outgassing properties of the item under test.

2.12.5.2.3. Selective Transmission Technique

The sensitivity of an ion gage to tracer gas can be increased if the gas is selectively brought to the ion gage. If this is done, the gage will not respond to extraneous pressure changes. Selectivity can be increased by use of a selective membrane or a cryogenic trap in front of the gage.

2.12.5.2.3.1. Palladium Barrier Ionization Gage

When heated to a temperature of 700 C or higher, palladium metal is permeable to hydrogen and to hydrogen only. If an evacuated ionization gage is sealed off from the rest of a vacuum system by a heated palladium barrier, it will detect the presence of hydrogen in the system and will be unaffected by other gases.

The gage as originally designed by Nelson (Ref. 156) is

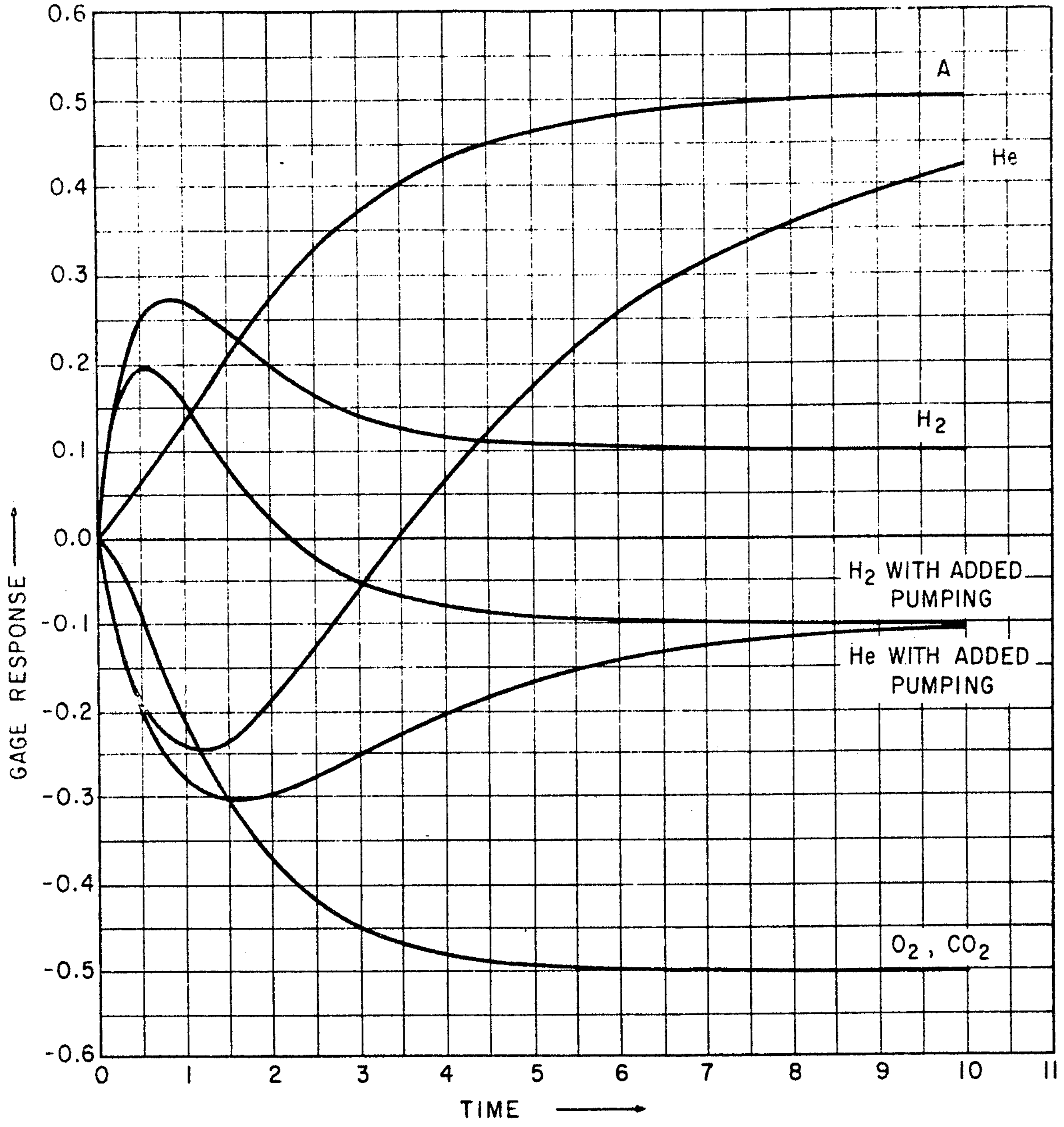

Figure 77. Response of an Ion Gage to Leaks of Various Gases. (Reprinted with permission from 1962 transactions, Ninth National Vacuum Symposium of the American Vacuum Society, J. W. Ackley and others, pp. 380-383, Copyright 1962, American Vacuum Society (Ref. 14))

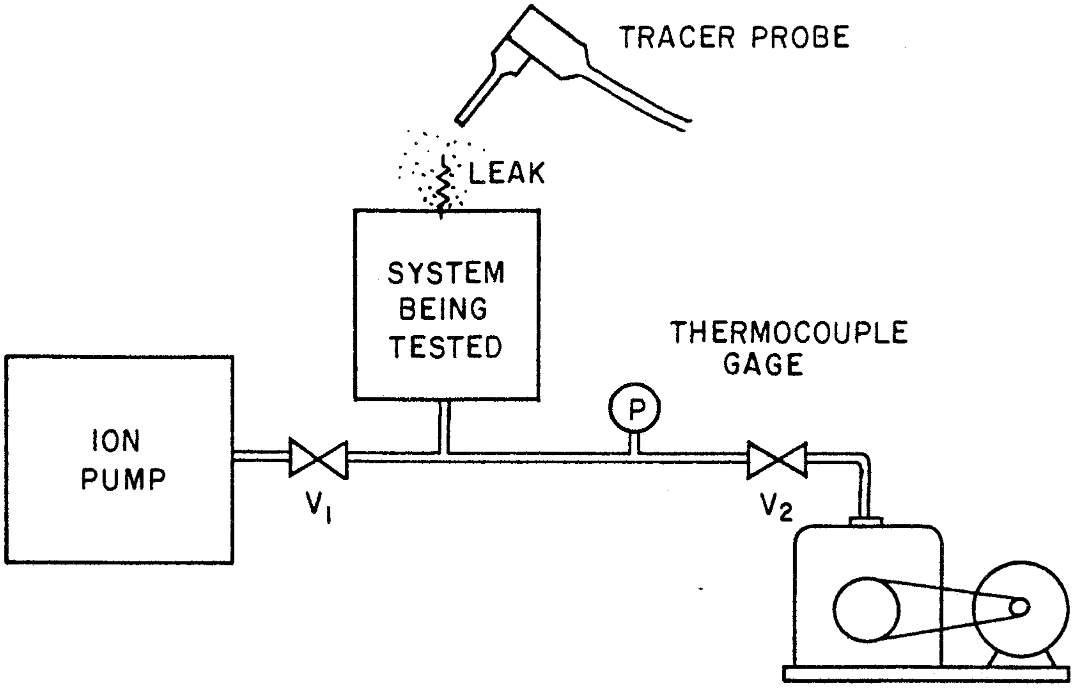

Figure 78. Ion Pump Leak Detector. (Reprinted with permission, from J.R. Young, Review of Scientific Instruments, Vol. 32, 1961, p. 85; Copyright 1961, American Institute of Physics.)

shown in Figure 79. Part A is an evacuated and sealed-off ionization gage tube with a piece of palladium tubing P forming part of its envelope. Part B is made from Nonex glass tubing, ring-sealed onto the glass envelope of the ionization-gage tube. C is a coil of platinum-clad molbdenum wire which serves to heat the palladium tubing.

This original design has been modified. The palladium tube is now the anode is some cases and is heated by the electron beam. The principle of the gage, however, remains the same.

The rate at which hydrogen passes through the barrier depends on the hydrogen pressure differential across the barrier. Thus, hydrogen in the gage can be removed by reducing the external hydrogen pressure below the internal value.

This gage can detect a pressure change of about 2 x 10^{-8} torr, but must be operated under fairly carefully controlled conditions in order to achieve this sensitivity. It is necessary to place a liquid nitrogen trap between the gage and the rest of the system in order to exclude hydrocarbons and water vapor from the gage. These vapors dissociate at the hot palladium surface to give hydrogen, which produces a spurious response. In addition, the cracked hydrocarbons build up a carbide layer on the palladium which reduces its permeability. It is also desirable to use a mercury rather than an oil diffusion pump in the vacuum system, since the hydrogen which results from the decompositon of diffusion pump oil gives rise to an unstable background ion current in the gage. In a system containing multiple leaks, oxygen in the air entering the undectected leaks combines at the hot palladium surface with the hydrogen entering through a leak which is being probed. If there is an excess of oxygen, all hydrogen will react with the oxygen before it can pass through the barrier and will therefore be undetected. Under these circumstances, Ochert and Steckelmacher (Ref. 72) have suggested that a controlled leak of hydrogen should be admitted to the system to take up the oxygen.

Nelson used a hydrogen generator consisting of a tungsten filament which decomposes oil vapors present in the vacuum system. To obtain maximum leak-detection sensitivity, it was sometimes found necessary to maintain a hydrogen partial pressure in the system of about 3 x 10^{-7} torr, by glowing the tungsten filament at a temperature of about 800 C.

If air is admitted to the ion gage, the palladium becomes oxidized even if it is cold. Whenever this occurs, two to three hours of run-in time is required to obtain reproducible results on duplicate runs. Therefore, even if the gage is not is use, the forepumps should be operated continuously to prevent air contact with the palladium. If the gas is left exposed to the atmosphere, several warm-up runs should be made, allowing hydrogen to pass through the calibrated leaks and pumping down between successive runs.

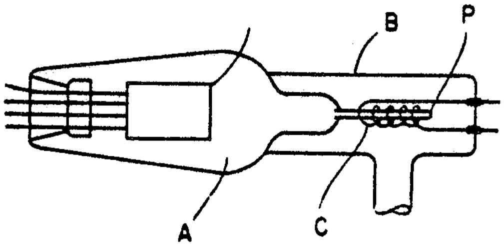

Figure 79. Palladium Barrier Ionization Gage Leak Detector. (Reprinted, with permission, from H. Nelson, The Review of Scientific Instruments, Vol. 16, No. 10, 1945, pp. 273-275. Copyright 1945 American Institute of Physics.)

2.12.5.2.3.2. Cryogenically Trapped Gage

Instead of palladium, it is possible to use an absorbent to pass the tracer gas and block air. Silica gel, outgassed at 300 C and then cooled to liquid nitrogen temperatures, is commonly used for this purpose. Under these circumstances, silica gel readily passes hydrogen and the noble gases (helium, neon, argon), but not air. A system using silica gel with a cold cathode gage and hydrogen has been described by van Leeuwen and Oskam (Ref. 157). Further refinements of this gage are described by Botden (Ref. 158). The gage was separated from the system by a liquid nitrogen cold trap filled with silica gel.

Van Leeuwen and Oskam claimed this system to be about a hundred times more sensitive than the palladium-hydrogen system. However, several hours were required to measure leaks of the order of 10^{-12} atm cc/sec, and careful degassing of the leak detector and the tube to be tested was necessary. One advantage claimed for silica gel is a long usage time before it has to be degassed. The increased sensitivity of silica gel is claimed to be due to less gas evolution from the gel than from heated palladium, which results in lower pressures. This detector, although very sensitive, is limited by long pumpdown times.

Beck and King (Ref. 159) constructed a leak detector using two magnetron ionization gages. The instrument was enclosed as a unit of the same general dimensions as the mass spectrometer leak detector.

The detector consisted of two ionization gages in series, the second of which was cryogenically trapped. The two gages were balanced on a bridge. Tracer gas changed the current of the first gage, but was condensed and therefore did not affect the second gage. With two gages, background pressure variations did not effect the detector. The sensitivity of this detector is reported to be 10^{-10} atm cc/sec. Lundberg described another detector using the same principle (Ref. 160).

2.12.5.2.4. Reduction of Electron Emission

A very sensitive means of locating leaks in vacuum systems is to observe the temperature-limited emission of a tungsten filament in a vacuum. Lawton (Ref. 161) found that when a stream of oxygen is blown over the outside of a leak, the resulting increase in oxygen pressure in the vacuum system causes the filament's emission to drop.

Although the principle has been known for a long time, and various circuits developed for its use (Ref. 162), this technique has not been extensively used.

Leger (Ref. 163) developed an instrument using this technique. Barton (Ref. 164) describes experiments in which the grid of a triode ionization gage was connected externally to the collector to form diode, which was used to detect oxygen admitted to the appartus under controlled conditions. He reports that a leak which gave rise to a partial air pressure of 10^{-7} torr was readily detected when probed with oxygen.

The detection circuit used was a modified ionization gage control unit. The filament was heated by a regulated power supply, but was not emission regulated. The emission current was read on a 10μa meter, which was backed by a 4.5-volt battery and a resistor in series. In this way, it was possible to read a change of a microampere at an emission of more than $10\,\mu$a.

Bloomer and Brooks (Ref. 165) examined the requirements for stable operation of this type of detector, using thoria coated tungsten filaments. They found it is best to reduce the thoria to thorium at the beginning of the test by heating for a few seconds to a temperature of 2400 K.

The greatest sensitivity is at an operation-temperature just below 1900 K, when the tungsten surface is partly covered with thorium. This can be obtained only when leaks of 10^{-9} torr l/sec or less are remaining in a well-baked system pumped at a speed of 0.1 l/sec.

The filament can become desensitized when it becomes carburized. It is because of the danger of this happening in the presence of hydrocarbon vapors, and because of the influence of residual water vapor upon the emission of electrons from the thoriated tungsten, that the detector is not very suitable for use in unbaked apparatus. If a filament becomes carburized accidentally it must be replaced; no thermal treatment cycle will bring it to a sensitive state again. But in a well-baked system thoriated filaments can, if necessary, always be restored to a desired state of sensitivity again by a short period of running at a temperature of about 2400 K.

2.12.5.3. THERMAL CONDUCTIVITY TECHNIQUE

The thermal conductivity technique may be used in either the system pressurized (detector probe technique) or the system evacuated mode (tracer probe technique). In the system evacuated mode, gages normally found on the system are used. In the system pressurized mode, special leak detectors are necessary.

2.12.5.3.1. Tracer Probe Technique

The principle of operation for the tracer probe technique is that described in Section 2.3.2.5. Either thermocouple or Pirani gages normally mounted on the system are used. Since these gages best respond to a pressure between 1 and 10^{-4} torr, they are used on systems with low pumping speed. Alternatively, these gages can be placed between the diffusion pump and the fore pump on a vacuum system.

The technique is very old (Ref. 166 and 167). Because it is continually used in leak location on vacuum systems, technical papers are frequently written on uses of new fluids which enhance the technique (Ref. 168), and on modifications of the pumping equipment which increase the sensitivity. For example, if probing is performed with hydrogen gas, an increase of tracer gas partial pressure may be obtained by reducing the diffusion pump heater voltage (Ref. 169). This decrease of hydrogen gas pumping speed is obtained without materially reducing the pumping speed for other gases.

Modifications of the simple leak location technique are similar to those described in Section 2.3.2.5.2. for ionization gages. For example, Kent (Ref. 170) describes a Pirani leak detector using hydrogen gas. In this detector, the gage is isolated from the system by a cooled charcoal trap. With this device it is possible to locate leaks as small as 10^{-6} atm cc/sec. Attempts have been made to increase stability of the detector by using two gages in a differential technique (Ref. 171 and 172).

Ishic (Ref. 173) developed a differential leak detector using butane gas. It has two gages in a Wheatstone bridge circuit. One of the gages is in series with a charcoal trap. This arrangement has stability because any random pressure changes will be detected by both gages, while the butane tracer gas will be absorbed by the charcoal. In this technique, the charcoal does not have to be heated during detection. The sensitivity of this system is reported to be 10^{-6} atm cc/sec.

Minter (Ref. 174) has been working on the use of thermal conductivity bridges for leak detection. He has attempted to use these four-element devices as extremely stable and sensitive detectors.

2.12.5.3.2. Detector Probe Technique

Whereas the detectors for the tracer probe technique all evolved from thermal conductivity gages present on the system, the detectors were specifically designed for the detector probe technique.

An example of such a detector is one designed by Stekelmacher (Ref. 175). This leak detector is based on a hot-wire bridge in which two resistance elements form two arms of the bridge network. One element is exposed to air containing tracer gas, while the the other is exposed only to air and serves as a reference to compensate for changes in ambient conditions.

As shown in Figure 80, the sensing elements are mounted in a metal block inside a hand held probe unit. Gas samples are drawn up through a narrow bore tube.

The tracer gas emerging from the leak is drawn into the sampling probe by the action of a small pump. The pump can be run at two speeds: a fast speed for maximum response, and a slower speed to give an increased detection sensitivity at some sacrifice in response time.

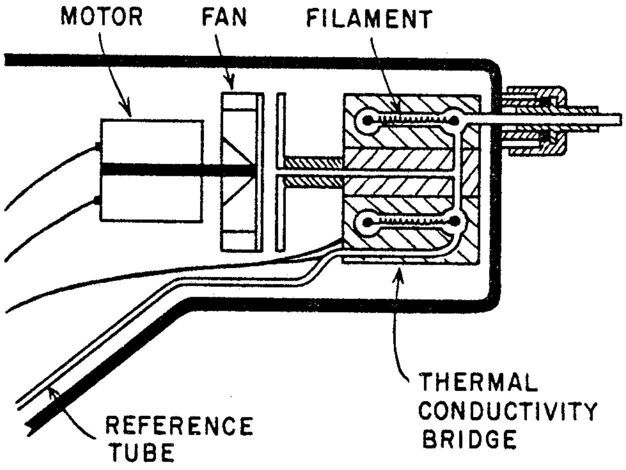

Figure 80. Thermal Conductivity Leak Detector. (Reprinted, with permission, From W. Steckelmacher and D.M. Tinsley, Vacuum, Vol. 12, No. 3, 1962, pp. 153-159, Copyright 1962, Pergamon Press, Inc., New York.)

To obtain a good response, the sensing elements must be small enough to fit in chambers of small volume. Since it is intended to detect changes in gas concentration rather than rates of flow, the gas should be made to flow past the entrance of the element chambers, rather than through them.

The sensing elements consist of coils of thin tungsten wire mounted on glass-metal seals in a compact assembly. The pump connects into this assembly. The sensing probe is also fitted with a small meter to repeat the leak indication of the amplifier unit. This assembly was found to be convenient to operators, particularly when testing awkwardly shaped equipment.

The electronic circuitry can be transistorized and thereby made compact enough so the unit can be hand held. The electronic components consist mainly of a stabilized power supply for the thermal conductivity bridge and an amplifier to increase and measure the amount of bridge unbalance.

The electrical power source can be either batteries or line current. A four step attenuator makes it possible to vary the sensitivity of the meter response by two decades.

The minimum detectable leak, in terms of quantity of tracer gas per unit time, depends on the rate of flow of the gas through the leak detector, and the minimum concentration to which the detector will respond. By reducing the rate of flow, smaller leaks can be detected. However, there is a practical limit, since it is important in leak location that the detector should respond quickly when the probe traverses the position of the leak. Reducing the rate of flow lengthens the response time, and beyond a certain point, the indications from the leak detector become meaningless.

The detector described by Steckelmacher (Ref. 175) can detect a 60 ppm concentration of hydrogen gas. This is a 1/10 full-scale response with a pumping speed for the probe of 8 cc/min. The instrument will detect a 6×10^{-6} torr l/sec hydrogen leak. With argon, which has a much lower thermal conductivity difference from air, only a 1×10^{-4} torr l/sec leak can be detected.

When testing with the thermal conductivity detector, the atmosphere must be free from tracer gas. If a system with very large leak is being tested, the local atmosphere may become concentrated with tracer gas. While this will be inherently balanced out by the reference circuit ultimate sensitivity is bound to decline.

The relatively low operating temperature of the filaments makes the detector quite safe to use under most industrial conditions. The functional life and long term stability of the sensing elements are good. The only effect which has been noted after long periods of operation under industrial conditions was the accumulation of a dust deposit in the intake line which was easily removed.

In principle, any tracer gas having a thermal conductivity different from that of air could be used. The sensitivity depends on relative differences of the thermal conductivities of the gases which are compared in Table 31. It is apparent that both hydrogen and helium show large relative differences, and are therefore the most sensitive tracer gases with this method. For special applications, it is sometimes desirable to employ one of the other tracer gases. The Table gives some indication of results expected. It is clear that either gases with a thermal conductivity greater than air (such as hydrogen, helium, neon, methane, etc.) or those with thermal conductivities less than air (such as halogenated hydrocarbons, argon, carbon dioxide etc.) would be suitable.

Unfortunately, this versatility is also a disadvantage. Because of a lack of selectivity, this instrument cannot be operated at high sensitivity in atmospheres contaminated with other gases.

The thermal conductivity bridges used in these detectors do not acutally measure thermal conductivity. Because of their structure, the readings obtained with these detectors is dependent on thermal conductivity combined with density, accomodation coefficient, and viscosity. Therefore, the values of sensitivity, derived from Table 31, are not absolute, but merely an indication of the expected general trend in the results.

A thermal conductivity detector, similar to Steckelmacher's, was developed by Minter (Ref. 176) using a four-element wire bridge. This bridge was also found useful for vacuum leak detection (Ref. 174). The sensitivity of this type of leak detector was improved by use of thermistors, with their higher thermal coefficient of resistance, instead of wire elements (Ref. 177). These detectors were tested in submarine service, where they were found useful in detecting leaks of a variety of gases (Ref. 178).

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Table 31

THERMAL CONDUCTIVITIES OF TRACER GASES

Gas	Chemical Formula	Molecular Weight	Source of Data	Thermal Conductivity
Air	(Mixture)	29.9	I	0.01478
Acetylene	C_2H_2	26.	I	0.01128
Ammonia	NH_3	17.	I	0.01333
Argon	A	39.9	I	0.01016
Benzene	C_6H_6	78	I	0.00538
Butane	C_4H_{10}	58	I	0.00822
Carbon dioxide	CO_2	44	I	0.00873
Carbon disulfide	CS_2	76	I	0.00410
Carbon monoxide	CO	28	I	0.01360
Ethane	C_2H_6	30	I	0.01102
Ethylene	C_2H_4	28	I	0.01025
Halogenated Hydrocarbon F-11	CCl_3F	137.4	III	0.00470
Halogenated Hydrocarbon F-12	CCl_2F_2	120.9	III	0.00542
Halogenated Hydrocarbon F-21	$CHCl_2F$	102.9	III	0.00554
Halogenated Hydrocarbon F-22	$CHClF_2$	86.5	III	0.00660
Halogenated Hydrocarbon F-113	$CCl_2F\text{-}CClF_2$	187.4	III	0.00438
Halogenated Hydrocarbon F-114	$CClF_2\text{-}CClF_2$	170.9	III	0.00629
Helium	He	4	I	0.08740
Hydrogen	H_2	2	I	0.10770
Hydrogen sulfide	H_2S	34	I	0.00770
Krypton	Kr	83.8	I	0.00540
Methane	CH_4	16	I	0.01872
Neon	Ne	20.2	II	0.02660
Nitric oxide	NO	30	I	0.01180
Nitrogen	N_2	28	I	0.01462
Nitrous oxide	N_2O	44	I	0.00925
Oxygen	O_2	32	I	0.01490
Propane	C_3H_8	44	I	0.00925
Sulfur dioxide	SO_2	64	I	0.00514
Water vapor	H_2O	18	I	0.01087
Xenon	Xe	131.3	II	0.03000

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Section 2.13

MISCELLANEOUS PROCEDURES

There are a number of procedures which do not precisely fit in any of the more widely used categories. These procedures are not standarized, and in many cases the equipment is in the developmental stage. This final section of the Leak Detection Handbook is not a comprehensive analysis of new and/or unusual methods of location and measurement; rather, it is intended to give the reader a familiarity with several such methods, as they exist today.

The miscellaneous procedures can be divided into three categories:

- Procedures intended for leak testing
- Procedures intended for other uses, but modified for leak testing
- Biological procedures adapted to leak testing

2.13.1. PROCEDURES INTENDED FOR LEAK TESTING

2.13.1.1. COMBUSTIBLE GAS DETECTORS

A combustible gas detector is manufactured by several companies (Table 32). This detector is designed for leak location by the detector probe technique on systems filled with combustible gases. The design of this detector is similar to that of the thermal conductivity detectors described in Section 2.12.5.3.2. The detector is a hand-held unit, usually with its own pump and power supply. As in the thermal conductivity detector, the tracer gas is passed over a thermocouple or other temperature sensor. However, in the combustible gas detector, the heat sensing element is in contact with a combustion catalyst. When a combustible tracer gas comes in contact with the catalyst, the heat of combustion raises the catalyst temperature and produces a response on the heat sensing element.

2.13.1.2. CONDENSATION NUCLEI DETECTORS

It is possible to detect small particles in the air by using them as condensation nuclei; their concentration can be measured by the number of condensation sites they form on an adiabatic expansion of a saturated atmosphere (Ref. 179). Such a system has been used for leak location (Ref. 180). In the leak location technique, the particles are formed by the tracer gas reacting in the detector. For example, if the tracer gas is ammonia, the detector might contain a dilute atmosphere of hydrogen chloride. The particle forming reaction would then be:

$$NH_3 + HCl \rightarrow NH_4Cl \qquad \text{Equation 2.13-1}$$

Table 32

CHARACTERISTICS OF COMBUSTABLE GAS DETECTORS
(Ref. 186)

General Characteristics	Average Values
Sensitivity	1 cu. ft/hr. 10 ppm Hydrogen, 1% L.E.L.
Tracer Gas	Combustible Mixtures
Output	Meter, Tone, and Light
Power Requirement	115 volts, 60 cycle, Battery
Size	From 4 inches x 8 inches x 10 inches to 15 inches x 17 inches x 10 inches
Weight	2 to 60 pounds
Price	$75 to $600

Manufacturers

Chemical Rubber Company
Davis Emergency Equipment Company, Inc.
Erdco Engineering Corporation
Gas Purifying Materials Company, Inc.
General Monitors, Inc.
International Telephone and Telegraph Corporation
Johnson-Williams Division of Bacharach Industrial Instrument Company
Salen and Wieander, AB
Teledyne, Inc.

This extremely sensitive leak location equipment is manufactured by two companies (Table 33). Two major advantages possessed by the equipment are that it is designed for operation at atmospheric pressure, and that it may be modified for operation with a variety of tracer gases.

2.13.1.3. BOUYANCY CHANGE DETECTOR

TRW Laboratories have developed a leakage measurement instrument which employs a principle of bouyancy to measure the leakage of a system (Ref. 181). The system being tested is pressurized with tracer gas and placed in a sealed chamber. A bouyancy balance measures the change of density in the chamber. This change of density is directly related to the amount and type of leaking gas. The major advantage of this technique is that it can give accurate quantitative leakage values for any gas.

2.13.2 PROCEDURES ORIGINALLY NOT DESIGNED FOR LEAK TESTING

Almost any detector which will show a change of gas concentration or composition may be used for leakage measurement. The sensitivity and practicality will depend on the inenuity with which the detector is employed. Two examples are described. However, the list of possibilities is almost limitless.

2.13.2.1. MASS SPECTROMETER

Occasionally, a mass spectrometer is part of the analytical equipment present on a vacuum system. This instrument is more versatile than the mass spectrometer leak detector described in Section 2.1; it is a device which can measure the concentration of a variety of gases. Such an instrument can be used to diagnose problems with the vacuum system, and occasionally locate the source of a leak. This technique has been described in literature (Ref. 182 and 183). Briefly some of the responses and their diagnoses are:

Gas Present in System	Probable Cause
Oxygen, nitrogen and argon	Air leak
Nitrogen	Leak in liquid nitrogen baffle
Water	Virtual leak, leak in coolant lines
Hydrocarbons	Inoperative baffling of pumps

Once the problem has been diagnosed, leaks may be located by a tracer probe technique, using the mass spectrometer as the detector.

2.13.2.2. GAS CHROMATOGRAPH

A gas chromatagraph separates a mixture of gases and measures their individual concentration. The separation is by means of the difference of their residence time in a column of sorbent. This instrument has been used to measure the permeability of polymers (Ref. 184). The same basic equipment design may be

Table 33

CHARACTERISTICS OF CONDENSATION NUCLEI LEAK DETECTORS

(Ref. 186)

General Characteristics	Average Values
Sensitivity	10^{-3} to 2 ppm
Tracer Gas	Varies with Application
Output	Meter Reading
Power Requirement	115 volts, 60 cycle
Size	22 inches x 19 inches x 20 inches
Weight	90 to 130 pounds
Price	$2000 to $4500

Manufacturers

General Electric Company (Research and Development Center)
Phoenix Precision Instrument Company

used for leakage measurement. The system is pressurized with tracer gas and placed in a chamber which is at atmospheric pressure. An inert carrier gas is passed through the chamber, and sweeps across the system. The concentration of tracer gas in the carrier is measured with the gas chromatograph. There are several advantages to this system:

- The tracer gas may be one of a variety which can be detected with a gas chromatograph.
- The system measures leakage to atmospheric pressure.
- The leakage measurement can be calibrated by measurements traceable to the Bureau of Standards.

2.13.3 BIOLOGICAL TECHNIQUES ADAPTED FOR LEAK TESTING

It has often been suggested that one simple method of leak location is to fill a system with honey, release ants, and watch where they enter the system. Although this dubious technique is related in jest, attempts were seriously made to develop a leak location technique using a sex hormone of a butterfly (Ref. 185). This hormone was injected into pipelines; the location of the leaks was clearly established by the congregation of a large number of butterflies. Presumably, it was felt that the sensitivity of the technique could be increased by using a drive greater than hunger.

Biological techniques are not always used in such a sophisticated fashion. Gas companies routinely place an odor-producing compound in natural gas. This odor-producing compound serves as a leakage monitor, indicating the existence of a leak. To an extent, a leak may be localized using such olfactory techniques.

The sensitivity of the nose as a leak testing instrument should not be underrated. It has been estimated that a concentration of propyl or butyl mercaptan as low as one part per billion in the atmosphere can be detected. This fact is regarded with envy by mass spectrometer leak detector manufacturers whose instruments can detect helium at a concentration of only one part per ten million (Section 2.1.2). Fortunately for these manufacturers, olfactory response is neither quantitative nor very directional.

Vegetation response can be used for leak location. The growth of many plants is affected by the presence of natural gas. Gas companies frequently locate leaks in pipelines by traveling the route of the line and observing plant growth adjacent to the pipeline. (Ref. 105) Leaks in the line will produce stunted plant growth in the vicinity of the leak. This will be exibited by a pre-seasonal color change, or a slowdown of the growth rate. Such changes can readily be detected by trained personnel.

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Appendix A

PROPERTIES OF TRACER GASES

PRECAUTIONARY MEASURES FOR USING VARIOUS TRACER GASES

PROPERTIES OF TRACER GASES

Name	Chemical Formula	Molecular Weight	Thermal Conductivity BTU/hr ft^2 F/ft at 20C	Viscosity poise x10^6 at 0 C	Diffusion Coefficient ft^2/hr at 1 atm and 0 C	Heat Capacity Ratio Cp/Cv	Molecular Diameter cm x 10^8
Acetylene	C_2H_2	26	0.01128	93.5	0.55	1.26. 15C	
Air	mixture	29.9	0.01478	170.8		1.403, 0C	
Ammonia	NH_3	17	0.01333	91.8	0.66	1.31 25C	2.97
Argon	A	39.9	0.01016	209.6	0.61	1.67 25C	3.67
Benzene	C_6H_6	78	0.00538	70.9	0.30	1.10. 90C	7.65
Butane	C_4H_{10}	58	0.00822	84.04, 14.7C	0.33	1.11, 15C	7.06
Carbon Dioxide	CO_2	44	0.00873	139.0	0.52	1.29. 25C	4.65
Carbon Disulfide	CS_2	76	0.00410	91.1	0.36	1.63, 99.7C	
Carbon Monoxide	CO	28	0.01360	166	0.67	1.404 15C	3.19
Ethane	C_2H_6	30	0.01102	84.8	0.49	1.19	5.37
Ethylene	C_2H_4	28	0.01025	90.66, 0.05C	0.52	1.255. 15C	
Halogenated Hydrocarbon F 11	CCl_3F	137.4	0.00470	96.0	0.30	1.124 160F	
Halogenated Hydrocarbon F 12	CCl_2F_2	120.9	0.00542	328, 5F	0.32		
Halogenated Hydrocarbon F 21	$CHCl_2F$	102.9	0.00554	101.0	0.33	1.157, 160F	
Halogenated Hydrocarbon F22	$CHClF_2$	86.5	0.00660	286, 5F	0.37		
Halogenated Hydrocarbon F 113	CCl_2F-CCl_2F	187.4	0.00438	93.0	0.25	1.077. 160 F	
Halogenated Hydrocarbon F 114	$CClF_2-CClF_2$	170.9	0.00629	102.0	0.28	1.080. 160F	
Helium	He	4	0.08740	186	2.7	1.67. 25C	2.18
Hydrogen	H_2	2	0.10770	83.5	2.6	1.41	2.75
Hydrogen Sulfide	H_2S	34	0.00770	116.6	0.53	1.32. 15C	
Krypton^{o}	Kr	83.8	0.00540	232.7	0.51	1.68 19C	4.15
Methane	CH_4	16	0.01872	102.6	0.72	1.31, 15C	4.19
Neon	Ne	20.2	0.02660	297.3	1.1	1.64 19C	2.60
Nitric Oxide	NO	30	0.01180	178	0.70	1.4. 15C	
Nitrogen	N_2	28	0.01462	166	0.68	1.40	3.15
Nitrous Oxide	N_2O	44	0.00925	136.2	0.52	1.303. 15C	
Oxygen	O_2	32	0.01490	189	0.68	1.40	2.92
Propane	C_3H_8	44	0.00925	79.5, 17.9C	0.39	1.13	6.32
Sulfur Dioxide	SO_2	64	0.00514	117	0.42	1.29, 15C	
Water Vapor	H_2O	18	0.01087	90.4	0.85	1.324. 100C	
Xenon	Xe	131.3	0.03000	210.1	0.42	1.66, 19C	4.91

PRECAUTIONARY MEASURES FOR USING VARIOUS TRACER GASES*

AMMONIA

At room temperature and atmospheric pressure, ammonia is a colorless, alkaline gas having a pungent odor. Ammonia dissolves readily in water. It is shipped as a liquefied gas under its own vapor pressure of 114 psig at 70°F.

The pungent odor of ammonia provides ample warning of its presence. The American Conference of Governmental Industrial Hygienists has recommended 100 ppm as the maximum allowable concentration for an 8-hour daily exposure. However, concentrations in the range of 50 to 100 ppm, although not harmful, will be of considerable nuisance. It is therefore unlikely that an individual would unknowingly become overexposed.

The physiological effects of various concentrations of ammonia are:

Atmospheric Concentration	Physiological Effects
20 ppm	First perceptible odor
40 ppm	A few individuals may suffer slight eye irritation
100 ppm	Noticeable irritation of eyes and nasal passages after few minutes exposure
400 ppm	Severe irritation of the throat, nasal passages, and upper respiratory tract
700 ppm	Severe eye irritation. No permanent effect if the exposure is limited to less than one-half hour
1700 ppm	Serious coughing, bronchial spasms, less than a half hour of exposure may be fatal.
5000 ppm	Serious edema, strangulation, asphyxia; fatal almost immediately.

The corrosive action of high concentration of ammonia (above 700 ppm) can cause extensive injuries to the eyes, causing severe

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*Courtesy of Mr. R.J. Roehrs, Nooter Corporation, St. Louis, Missouri irritation, hemorrhages, and swollen lids. If not treated immediately, partial or total loss of sight may result. The mucous lining of the mouth, throat, nose and lungs is particularly sensitive to ammonia attack.

One percent ammonia in the atmosphere is mildly irritant to moist skin, two percent has a more pronounced effect, and three percent produces a stinging sensation and may produce chemical burns with blisters after a few minutes. Liquid ammonia in contact with the skin will produce severe burns, and the freezing effect due to rapid evaporation from the skin surface can cause frostbite.

Anyone working with ammonia should wear rubber gloves, chemical goggles, and a rubber or plastic apron. Cylinders should never be dropped or be permitted to strike each other violently. No part of a cylinder should be subjected to a temperature higher than 125 F. Ammonia cylinders should never be directly heated by steam or flames. Uncontrolled heating of a cylinder can cause the liquid to expand to a point where dangerous hydrostatic pressure will be developed. Any heating should be done in a thermostated water or oil bath. The temperature should not be allowed to exceed 125 F. Cylinders containing less than 165 pounds of ammonia are not equipped with safety devices.

Although ammonia does not represent a serious flammibility hazard, mixture of air and ammonia containing from fifteen percent to twenty-eight percent ammonia by volume will ignite when sparked or exposed to temperatures exceeding 1200 F: therefore, flames and sparks should not be allowed in the area where ammonia is being used.

Iron and steel are recommended for all equipment coming in contact with ammonia. Copper, tin, zinc, and their alloys are attacked by moist ammonia and should not be used. Piping should be rigid steel, except where short connections are needed such as between cylinders and manifold or pipe lines. For these applications reinforced flexible, neoprene line is recommended.

Ammonia can combine with mercury to form explosive compounds; therefore, instruments containing mercury that will be exposed to ammonia should not be used.

ARGON

Argon is a colorless, odorless, and tasteless gas somewhat soluble in water (4 volumes in 100). It is normally supplied as a nonliquified gas compressed into cylinders at a pressure of approximately 2200 psig at 70 F.

Argon is nontoxic, but can act as a simple asphyxiant by displacing the necessary amount of air to support life.

Argon is a chemically inactive gas. It will not react with other elements or compounds. While a few compounds of argon and other rare gases are reported to have been prepared, the results obtained may be considered of scientific interest only.

CARBON DIOXIDE

Carbon dioxide is a nonflammable, colorless, odorless, slightly acid gas. It is approximately one and one-half times as heavy as air. One volume of carbon dioxide will dissolve in approximately one volume of water at atmospheric pressure and 15 C. In high concentrations it has an acidic taste. Carbon dioxide is shipped in Interstate Commerce Commission approved, high pressure steel cylinders as a liquid under its own vapor pressure of approximately 830 psig at 70 F.

In high concentrations carbon dioxide can paralyze the respiratory center. It is therefore, an industrial hazard. It is heavier than air and does not diffuse readily; consequently, it may collect in confined, unventilated areas. Where persons are working in confined spaces, the amount of carbon dioxide formed by breathing may assume dangerous proportions. Carbon dioxide is the regulator of the breathing function, and an increase in the carbon dioxide inhaled will cause an increased rate of breathing:

Carbon Dioxide in Air (Volume Percent)	Increased Lung Ventilation
0.1-1	Slight and unnoticeable increase
2	50% increase
3	100% increase
5	300% increase, breathing becomes laborious

Ten percent carbon dioxide in air can be endured for only a few minutes. Twelve to fifteen percent soon causes unconsciousness. Twenty-five percent may cause death in exposures for several hours. A generally recommended maximum allowable concentration for an 8-hour day is 5000 ppm. The normal concentration of carbon dioxide in the air is 0.03 percent, or 300 ppm.

Carbon dioxide is not a chemically active compound. High temperatures are generally required to promote its reactions. Carbon dioxide is stable under normal conditions, but at temperatures above 1500 C, carbon dioxide almost completely dissociates into oxygen and carbon monoxide according to the following formula:

$$2\ CO_2 \rightarrow 2\ CO + O_2$$

DICHLORODIFLUOROMETHANE

Dichlorodifluoromethane (Freon 12, Genetron 12, Isotron 12, Ucon 12) is a colorless, nonflammable gas at normal temperatures and pressure. In concentrations of less than 20% dichlorodifluoromethane is odorless; in higher concentrations, its odor is mild and somewhat ethereal, and similar to that of carbon tetrachloride. It is readily liquefied and is shipped in steel cylinders as a liquefied gas under its own vapor pressure of about 70 psig at 70 F.

Dischlorodifluoromethane is practically non-toxic. It shows no toxic effects in guinea pigs in concentrations up to at least 20 percent (by volume) for two hour exposures. In higher concentrations, it may produce some physiological action, caused primarily by oxygen deficiency. The generally accepted maximum allowable concentration for an 8-hour daily exposure is 1000 ppm.

No part of a cylinder should be subjected to a temperature higher than 125 F. Temperatures in excess of 125 F, may cause excessive hydrostatic pressure buildup. Never permit a flame to come in contact with any part of a compressed gas cylinder.

In general, gasket materials should not contain natural rubber. Neoprene or Isoprene rubber, pressed fibers, including abestos, with a number of insoluble binders and metallic gaskets may be used.

Dichlorodifluoromethane is thermally stable. It is nonflammable in any mixture with air and will not explode or propagate a flame. It is hydrolytically stable. It is noncorrosive under normal conditions to all common metals of construction, but it is corrosive at elevated temperatures.

HELIUM

Helium is the lightest member of the rare gas family. It is an inert, colorless, odorless, and tasteless gas, and is only slightly soluble in water (0.87 parts in 100 parts). Helium is compressed into cylinders at a pressure of approximately 2200 psig at 70 F.

Helium is nontoxic but can act as an asphyxiant by displacing the necessary amount of air to support life.

Helium is a chemically inactive gas. It will not react with other elements or compounds under ordinary conditions.

HYDROGEN CHLORIDE

Anhydrous hydrogen chloride is a colorless, pungent, corrosive gas having a suffocating odor. Hydrogen chloride is heavier than air; it fumes strongly in moist air. It is very soluble in water and alcohol, and ether. While in the cylinder under pressure, hydrogen chloride is in the form of a gas over liquid, the cylinder pressure being about 613 psig at 70 F. As long as liquid is present in the cylinder, the pressure will remain fairly constant.

Hydrogen chloride is a highly toxic gas; it is severely irritating to the upper respiratory tract and corrosive to the eyes, skin, and mucous membranes. The acid formed neutralizes the alkali of the tissues and causes death as a result of edema or spasm of the larynx and inflammation of the upper respiratory system. Concentrations of 0.13 to 0.2 percent are lethal for human beings in exposures lasting a few minutes. Concentrations of 0.1 to 2.13 percent are dangerous if breathed 30 to 60 minutes. The maximum concentration tolerated for exposures of 60 minutes is in the range 0.005 to 0.01 percent and the maximum tolerated for several hours of exposure is 0.001 to 0.005 percent. The generally accepted maximum allowable concentration for an 8-hour daily exposure is 5 ppm. However, hydrogen chloride provides adequate warning for prompt voluntary withdrawal from contaminated atmospheres.

Workers who handle hydrogen chloride should wear protective clothing such as rubber or plastic aprons, rubber gloves, and suitable gas-tight chemical safety goggles. Woolen outside clothing or other acid resistant fabrics are also recommended.

Hydrogen chloride is essentially inert to metals and does not attack the commonly used structural metals under normal conditions of use (room temperature and atmospheric pressure). In the presence of moisture, however, hydrogen chloride will corrode most metals other than silver, platinum, and tantalum. When used at higher pressures, it is necessary to use extra heavy black-iron pipe throughout. No galvanized pipe or brass or bronze fittings should be used, since these will corrode. High pressure monel or aluminum-iron-bronze valves should be used throughout. Hydrogen chloride lines should always be shut-off from the use end, backward to the cylinders.

Hydrogen chloride is thermally stable. It is stable to oxidation except at elevated temperatures. Hydrogen chloride is extremely soluble in water, its aqueous solution is a strong acid.

HYDROGEN

Hydrogen is colorless and odorless and is the lightest gas known. Hydrogen is only very slightly soluble in water (1.93 volumes in 100 volumes of water). Hydrogen is usually shipped in high pressure steel cylinders at 2000 psig at 70 F.

Hydrogen is nontoxic, but can act as an asphyxiant by displacing the necessary amount of air required to support life.

Cylinders of hydrogen should never be used in areas where flames, excessive heat, or sparks may occur. Only explosion proof equipment, and spark-proof tools should be used in areas where hydrogen is handled. All equipment and lines used with hydrogen should be grounded. A flame should never be used to detect hydrogen gas leaks. Reserve stocks of hydrogen should never be stored with cylinders containing oxygen or other highly oxidizing materials.

Hydrogen, although relatively inactive at ambient temperatures, reacts with almost all the other elements at high temperatures. The reaction between hydrogen and oxygen at high temperatures is usually very violent. Hydrogen will reduce metallic oxides at elevated temperatures.

KRYPTON

Krypton is an inert gas and one of the rarest gases known. Krypton exists in very minute traces in the atmosphere (1 ppm by volume of air). Krypton is a colorless and odorless gas, and only slightly soluble in water. Krypton is available in small cylinders at various pressures up to 800 psig and in Pyrex liter flasks.

Krypton is chemically inert under all normal conditions, and does not combine chemically with other elements or compounds.

METHANE

Methane is a flammable, colorless, odorless, and tasteless gas. It is the major constituent of natural gas. The material is normally shipped as a nonliquefied gas in high pressure cylinders at approximately 2265 psig at 70 F.

Methane is not considered a toxic gas. Physiologically, it is a simple asphyxiant; in high concentrations it can displace the oxygen required to sustain life. Miners frequently breathe air containing 9 percent of methane and do not appear to suffer. When the percent increases above this point, pressure on the forehead and eyes is noticed. However, this pressure disappears again on breathing fresh air.

The hazards due to the handling of methane stem mainly from its extreme flammability. Cylinders of methane should be stored in a well-ventilated area away from heat and all ignition sources. Flames should never be used to detect flammable gas leaks. Methane should not be used around sparking motors or other non-explosion-proof equipment. Reserve stocks of methane cylinders should never be stored with cylinders containing oxygen, chlorine, or other highly oxidizing or flammable materials. Although methane is generally inert at room temperature and atmospheric pressure, it does undergo reaction under certain conditions.

NEON

Neon is a rare inert gas that exists in the atmosphere. Neon constitutes 0.00182 percent by volume of air. Neon is a colorless and odorless gas; it is normally available in compressed cylinders at varying pressures up to 1800 psig and also in Pyrex flasks at atmospheric pressure.

Neon is nontoxic but can act as an asphyxiant by displacing the necessary amount of air to support life.

Neon is chemically inert under all normal conditions.

NITROGEN

Nitrogen comprises approximately 79 percent by volume of the air. Nitrogen will not burn and will not support combustion. Nitrogen is normally available in cylinders compressed to 2200 psig at 70 F.

Nitrogen is nontoxic but can act as an asphyxiant by displacing the necessary amount of air to sustain life.

Nitrogen is extremely inert, except when heated to very high temperatures, where it combines with metals to form nitrides.

NITROUS OXIDE

Nitrous oxide is a colorless, nonflammable, nontoxic gas with a slightly sweetish taste and odor. It is shipped as a liquefied compressed gas under its own vapor pressure of about 745 psig at 70 F. It is somewhat soluble in water and more soluble in alcohol.

Nitrous oxide is non-irritating and is extensively used as an anesthetic in medicine and dentistry. It is a rather weak anesthetic, and must be inhaled in high concentrations mixed with air or oxygen. When inhaled without oxygen, it is a simple asphyxiant. Inhalation of small amounts often produces a type of hysteria; hence its trivial name, laughing gas.

Nitrous oxide is stable and comparatively unreactive at ordinary temperatures. At elevated temperatures nitrous oxide decomposes into nitrogen and oxygen, the rate of decomposition being appreciable above 565 C. At elevated temperatures, nitrous oxide supports combustion and oxidizes certain organic compounds, the alkali metals, etc.

OXYGEN

Oxygen is a colorless, odorless, and tasteless gas. Its outstanding properties are its ability to sustain animal life and to support combustion. It is somewhat soluble in water. It is usually shipped as a nonliquefied gas at 2200 psig at 70 F.

The inhalation of 100 percent oxygen at atmospheric pressure for periods up to 16 hours per day, for many days, has caused no observed injury to man. Long periods of exposure to higher pressures can adversely affect neuromuscular coordination, and power of attention.

Oil, grease, or other readily combustible substances should never contact oxygen cylinders, valves, regulators, gauges, and fittings. Oxygen valves, regulators, gauges, or fittings should never be lubricated with oil or any other combustible substance. In welding shops and industrial plants using both oxyacetylene and electric welding apparatus care should be taken to avoid the handling of these equipments in any manner which may permit the compressed gas cylinders to come in contact with electrical circuits. Manifolds should never be used for oxygen cylinders unless constructed by a qualified engineer under the standards of a recognized safety authority. Oxygen should not be supplied by a system of shop piping without consulting the supplier for recommendations. No foreign substance should be allowed to enter the cylinder or valve. Cylinders of oxygen should not be stored near cylinders of acetylene or other combustible gases. Unless well separated, there should be a fire-resistant partition between oxygen and acetylene, or other cylinders of combustible gases.

SULFUR DIOXIDE

Sulfur dioxide is a highly irritating, nonflammable, colorless gas at room temperature and atmospheric pressure. It is soluble in water, forming a weak solution of sulfurous acid. It is readily liquefied and is shipped in steel cylinders as a liquefied gas under its own vapor pressure of about 35 psig at 70 F.

Sulfur dioxide is a highly irritating gas in the vapor form; it is readily detectable in concentrations of 3-5 ppm, providing ample warning of its presence. In higher concentrations, the severely irritating effect of the gas makes it unlikely that any person would be able to remain in such a contaminated atmosphere unless he were unconscious or trapped.

Liquid sulfur dioxide may cause skin and eye burns upon contact with these tissues, which result from the freezing effect of the liquid on the skin or eyes.

Acute exposure to sulfur dioxide has the following effects:

Atmospheric Concentration	Physiological Effects
8-12 ppm	causes throat irritation, coughing, constriction of the chest, tearing, and smarting of the eyes
150 ppm	causes extreme irritation and can be tolerated only for a few minutes
500 ppm	so acutely irritating that it causes a sense of suffocation

There are no known systemic effects of acute exposure to sulfur dioxide. The generally accepted maximum allowable concentration of sulfur dioxide for an 8-hour daily exposure is 10 ppm.

Sulfur dioxide should be handled only in a well-ventilated area, preferably a hood with forced ventilation. Personnel handling sulfur dioxide should wear chemical safety goggles and/or plastic face shields, approved safety shoes, and rubber gloves. In an emergency, gas masks, approved by the U.S. Bureau of Mines for sulfur dioxide, should be worn as required. Additional gas masks, air-line gas masks, and self-contained breathing apparatus should be conveniently located for use in emergencies. Instant-acting safety showers should be available in convenient locations.

Sulfur dioxide forms both sulfite and bisulfite salts.

Appendix B

GLOSSARY OF TERMS

Appendix B
GLOSSARY OF TERMS

This glossary is composed mainly of terms published in the American Vacuum Society, Glossary of Terms Used in Vacuum Technology, Pergamon Press, New York, 1958. An asterisk denotes those terms preferred by the Standards Committee of the American Vacuum Society. The Greek φ indicates those terms considered inappropriate by the Committee.

Wherever a term is discussed in detail in this book, the Section number is listed in parenthesis.

ABSOLUTE PRESSURE - A term used in engineering literature to indicate pressure above the absolute zero value corresponding to empty space as distinguished from gage pressure. In vacuum, technology pressure always corresponds to absolute pressure not gage pressure, and therefore the term absolute pressure is not required.

ABSOLUTE VACUUM - A space completely empty of matter. Also called a perfect vacuum.

ABSORPTION - The binding of gas in the interior of a solid (or liquid).

ACCOMMODATION COEFFICIENT (for condensation) - The ratio of the condensation rate to the impingement rate. The term condensation coefficient* is recommended for this ratio.

ACCOMMODATION COEFFICIENT (for free-molecule heat transfer)* - The ratio of the energy actually transferred between impinging gas molecules and a surface and the energy which would be theoretically transferred if the impinging molecules reached complete thermal equilibrium with the surface.

ACCUMULATION TEST - A leak test in which gas contained in the system tested collects for a period of time in a closed chamber containing the system, at the end of which period the chamber is opened to a leak detector. This is not an A.V.S. definition. In the handbook, this is referred to as static testing. (Section 1.3.4.1.1.)

ADSORPTION - The condensing of gas on the surface of a solid.

AIR-INLET VALVE - A valve used for letting atmospheric air into a vacuum system. Also called a vacuum breaker.

APERTURE IMPEDANCE - In calculating the total impedance of a pipe line constructed of short tubes of different cross section for the molecular flow of gas, the aperture impedance is the additional resistance to gas flow created by the abrupt change in cross section from an area A_1 to an area A_2. The aperture impedance is calculated by multiplying the theoretical impedance for molecular effusion through an orifice of area A_2 by the aperture correction factor $(1 - A_2/A_1)$. (Section 1.4.1.1.2.)

ATMOSPHERIC PRESSURE - The pressure of the atmosphere at a specified place and time.

a. The standard atmosphere, or normal atmosphere, is defined (independently of barometric height) as a pressure of 1,013,250 dyne/cm^2.*

b. The normal atmosphere has also been defined as the pressure exerted by a mercury column 760 mm in height at 0°C under standard acceleration of gravity of 980.665 cm/sec^2. Assuming a density of mercury at 0°C of 13.59509 g/cm^3 this is equal to 1,013,249 dyne/cm^2.

AUDIBLE LEAK INDICATOR - See Squealer

AVERAGE MOLECULAR VELOCITY - The average velocity of molecules in a gas at rest under equilibrium conditions. (Section 1.4.1.1.2.)

BACKGROUND - In the case of a leak detector, the spurious output, expressed in suitable terms, due to the response to other gases than the actual gas being used for probing. The background may be inherent in the detector, or accidental.

BACK SEAT GASKET - A gasket placed on that side of the disk holder of a valve which is opposite the disk insert, so as to seat when the valve is opened and prevent leakage through the bonnet or stem assembly.

BACKING PUMP - See fore pump

BACKING SPACE - The space between a "backing pump" (fore pump) and the associated diffusion pump (or other type of pump requiring a fore pump). Also referred to as the ballast.

BACKING SPACE TECHNIQUE - A system of leak detection in which the leak detector is connected to the backing space to take advantage of the compression of gas that occurs between the vacuum system and the backing pump, due to the action of the diffusion pump (or other type of pump of high speed relative to its backing pump). (Section 1.3.4.1.2.3.)

BACK PRESSURE Φ - See fore pressure

BACK PRESSURIZING TESTING - A method of testing sealed units in which the units are placed under a tracer gas pressure for a period, the tracer gas flushed from outside the unit, and the traces leakage from the unit measured. Also called helium bomb and pressure-evacuation testing. This is not an A.V.S. definition. (Section 1.3.4.3.2.)

BAFFLE* - a. A system of cooled walls, plates, or tubing placed near the inlet of a vapor pump to condense back-streaming vapor at a temperature below that of the room and return the fluid to the boiler.* The baffle plates may be located in the "head" of the pump or in a separate housing attached to the inlet.

b. A system of plates placed in a pipe line to deviate the flow from the axial direction.

BAFFLE VALVE - A valve of such design that the valve disk remains in line with the valve opening and so can act as a baffle.

BAKE-OUT - The degassing of a vacuum system by heating in an oven during the pumping process. (Section 1.5.3. and 2.3.4.2.1.1.)

BALLAST TANK - A vessel placed in the fore line, between the foreline valve and a diffusion pump, to serve as a gas receiver while the fore pump is temporarily isolated from the diffusion pump. (Also see backing-space technique)

BAR - A unit of pressure equal to 10^6 dynes per square centimeter.

BAYARD - ALPERT IONIZATION GAGE - See ionization vacuum gage.

BELL JAR TESTING - Sometimes referred to as "inside-out testing" and "pressure-vacuum testing". A test used for detecting leakage from an object filled with a tracer gas and placed in a vacuum chamber or bell jar. This is not an A.V.S. definition.

BELLOWS SEALED VALVE - A valve type in which the stem seal is accomplished by means of a flexible bellows, one end of which is attached to the disk end of the stem, the other end to either the bonnet or the body.

BLANK-OFF PLATE - A metal plate or flange used to seal off an open port in an enclosure which is being evacuated for leak test or other tests, or to keep the enclosure from contamination during storage or shipping. Also called cover plate.

BOMB TEST - A form of leak test in which electronic components are immersed in a liquid which is then pressurized for the purpose of driving it through possible leak passages and thus into the internal cavities where its presence will cause some form of electrical disturbance. This is not an A.V.S. definition.

BUBBLE TEST - A form of leak test suitable for detecting relatively large leaks in gas-containing enclosures in which the enclosure is fully immersed in a liquid that is either heated or over which the ambient gas pressure is reduced for the purpose of driving the enclosed gas out of the enclosure in the form of bubbles that become visible as they pass through the liquid. This is not an A.V.S. definition. (Section 2.5.1.1.1.)

BY-PASS LINE - The name applied to a roughing line when the roughing pump also serves as a backing pump, and which implies a separate fore line.

CATHODE ACTIVATION - The thermal and/or electrical treatment of a cathode which results in conditions conducive to active electron emission in normal operation. (Section 2.12.6.2.)

CATHODE AGEING - The stabilization of electron emission from a newly activated cathode by controlled operation. (Section 2.12.6.2.)

CATHODE POISONING - Inactivation of an activated cathode by contaminents. (Section 2.12.6.2.)

CHEMISORPTION - The binding of gas on the surface or in the interior of a solid (or liquid) by chemical action. (Section 1.5.3.)

CHEVRON SEAL - a. A type of seal built up of a number of nested V-ring gaskets.

b. A modified Wilson seal containing several gaskets in a pile without spacers.

CLEAN-UP - A term sometimes used to describe the process of removing gas from a vacuum system or device by sorption or ion pumping. (Section 1.3.4.1.2.2.)

CLEAN-UP TIME* - The time required for a leak detector or a leak detection system to reduce its signal output to 37% of the signal indicated at the time that tracer gas ceases to be applied to the test system.* (Section 1.3.4.1.2.2.)

CLUSECψ - A flow rate equal to .01 lusec. (Section 1.3.1.2.)

COLD CATHODE IONIZATION GAGE - See ionization vacuum gage

COLD TRAP - A vessel designed to hold a refrigerant, or cooled by coils in which a refrigerant circulates, inserted into a vacuum system so as to condense on its inner surface vapors present in the system.

COLLISION FREQUENCY - The number of collisions between molecules in a gas per cubic centimeter per second. (Section 1.4.1.1.1 and 1.4.1.1.2.)

COLLISION RATE*-The average number of collisions per second suffered by a molecule or other particle moving through a gas. Also called the collision frequency per molecule. (Section 1.4.1.1.1. and 1.4.1.1.2.)

COMPOUND MECHANICAL PUMP - A mechanical pump having two or more stages in series.

CONDENSATION RATE* - The number of molecules which condense on a surface per square centimeter per second.

CONDENSER* - A system of cooled walls, plates, or tubing placed in front and to the side of a vapor jet to condense the vapor and return the fluid to the boiler.*

CONDUCTANCE* - a. (Measured value) - The throughput under steady state conservative conditions divided by the measured difference in pressure between two specified cross sections inside a pumping system.*$C=Q/(P_1 - P_2)$ (Section 1.3.2. and 1.4.1.)

b. (Calculated from long tube formulae) - The throughput under steady-state conservative conditions at a given mean pressure per unit pressure difference across two specified sections in a theoretical pipe of length much greater than the distance between the sections and of uniform or slowly changing cross section as calculated from formulae which neglect entrance and aperture corrections.

c. (Calculated from short tube formulae) - The throughput under steady-state conservative conditions in a pipe line at a given mean pressure per unit pressure difference between two large chambers connected by the pipe line as calculated from formulae which include entrance and aperture corrections.

COVER PLATE - See blank-off plate

DEADHEAD - A port which is inoperative or which is sealed with a temporary plug rather than a tube to be processed on an exhaust or aluminizing system.

DEGASSING* - The deliberate removal of gas from a material, usually by application of heat under high vacuum.*(Section 1.5.3. and 2.3.4.2.1.)

DEMOUNTABLE HIGH-VACUUM SYSTEM - A system with demountable joints and seals usually containing materials which do not have a very low outgassing rate and in which limiting pressures of 5×10^{-5} to 5×10^{-7} torr may be obtained by the use of extremely fast pumps in spite of the outgassing load or even the presence of small leaks. Sometimes called a dynamic vacuum system or kinetic vacuum system.

DESORPTION - The process of removing physically sorbed gas. (Sections 1.5

DETECTOR PROBE - See Sampling probe

DIAPHRAGM VALVE - A valve type in which a thin flexible disk, clamped tightly between the body and bonnet, is interposed between the stem and disk, thus sealing both the bonnet and the stem, and permitting transmission of stem motion by its flexure.

DIFFERENTIAL PIRANI LEAK DETECTOR - A differential leak detector employing two similar Pirani tubes as arms of a Wheatstone bridge, both tubes being connected to the system under test. A cold trap is placed between one tube and the system. Condensable tracer gas entering the system reaches one tube but not the other, thus unbalancing the bridge and indicating a leak. (Section 2.12.6.4.)

DIFFERENTIAL LEAK DETECTOR - A leak detector employing two similar gauge tubes in a bridge circuit with a trap which is selective for the tracer gas between the system and one of the tubes. The circuit responds to the tracer gas but not to fluctuations in air pressure. (Sections 2.3.4.2 and 2.12.6.4.)

DIFFUSION COEFFICIENT - The absolute value of the ratio of the molecular flux per unit area to the concentration gradient of a gas diffusing through a gas or a porous medium where the molecular flux is evaluated across a surface perpendicular to the direction of the concentration gradient. (Section 1.5.1.5.3.)

DIFFUSION-EJECTOR PUMP* - A vapor pump having a boiler pressure of more than a few torr and having one or more ejector stages combined with one or more diffusion stages to which the vapor is supplied at reduced pressure by suitable throttling means.*

DIFFUSION PUMP* - A vapor pump having boiler pressures less than a few torr and capable of pumping gas with full efficiency at intake pressures not exceeding about 20 millitorr and discharge pressures (forepressures) not exceeding about 500 millitorr.* More precisely, the term diffusion should be applied only to pumps in which the pumping action of each vapor jet occurs by the diffusion of gas molecules through the low-density scattered vapor into the denser forward moving core of a freely expanding vapor jet. Most of the gas molecules are then driven at an acute angle towards the wall and on into the fore vacuum. The required optimum condition for this process is that the mean free path of gas molecules through vapor molecules in the nozzle clearance area be greater than the nozzle clearange. This condition can not usually be realized unless the vapor jet expands freely and condenses on a wall at temperatures low enough to make re-evaporation negligible. Sometimes called a condensation pump, vapor pump, or vapor jet pump.

DISCHARGE TUBE LEAK INDICATOR - A glass tube with electrodes attached to a source of high frequency high voltage, such as a Tesla coil or induction coil, so that changes in the color of the electrical discharge can be observed when a suitable tracer gas (methane, carbon dioxide, alcohol) flows through the leak. (Section 2.7.)

DRIFT - A shift or change in output level of the leak detector due to the electronics rather than a change in the level of the tracer gas. (Appendix III)

DUPLEX MECHANICAL PUMP - A mechanical pump having two single stage units in parallel operated by the same drive.

DYNAMIC LEAKAGE MEASUREMENT - In the handbook, leakage measured by measuring the tracer gas equilibrium partial pressure while the system is actively being pumped. This is not an A.V.S. definition. (Section 1.3.4.1.2.)

DYNAMIC VACUUM SYSTEM - See demountable high-vacuum system

EFFUSION LAW* - The formula for the theoretical volumetric flow rate per cm^2 of cross section by molecular effusion through a hole in a thin plane wall between a chamber at the given pressure and a chamber at zero pressure.*

$$f = \bar{v}/4 = 3638 \quad T/M \ cm^3 \cdot sec^{-1} \cdot cm^{-2}$$

(Section 1.4.1.1.2.)

EJECTOR PUMP* - A vapor pump having boiler pressures of more than a few torr and capable of pumping gas with full efficiency at intake pressures of more than about 20 millitorr and discharge pressures (forepressures) exceeding about 500 millitorr.* More precisely the term ejector should be applied only to pumps in which the pumping action of each vapor jet depends mainly on the entrainment of gas by viscous drag and by diffusion of gas into the vapor at the boundary of a dense vapor stream which carries the gas at supersonic velocities down a converging chamber (diffuser) and through a throat which is equal to or smaller than the cross section of the vapor stream. Turbulent mixing of gas and vapor may occur in the boundary of the high-pressure jets of steam ejectors. The condition for the formation of a distinct gas-vapor boundary is that the mean free path of gas molecules through the vapor issuing from the nozzle should be considerably less than the nozzle clearance. At peak pumping speed condensation of the vapor occurs beyond the throat of the diffuser. At lower pressures the vapor jet will diverge and strike the diffuser walls before reaching the throat, and some cooling may therefore be required on the converging section of the diffuser.

a. *Oil ejector pumps* employ a stable organic or silicone oil as the pump fluid.

b. *Mercury ejector pumps* employ mercury as the pump fluid.

c. *Steam ejector pumps* employ high pressure steam which passes through the nozzle and diffuser without condensing, and may then pass through the intake of another steam ejector stage or may be condensed in an *intercondenser* placed between the two stages.

EXHAUST TUBULATION - See pump-out tubulation.

EXTERNAL THROUGHPUT - The throughput of gas which comes from the vacuum system exclusive of the pump.

FLAP GASKET - A large annular flat gasket with outside diameter considerably larger and inside diameter considerably smaller than the end of the pipe or bell jar over which it is drawn so that the gasket clings to the pipe and bends outward to make a seal against a flange or base plate.

FLAT GASKET - A gasket made from flat stock.

FLOODED SYSTEM - A system which, while being leak tested under vacuum, becomes so filled with tracer gas as to make impracticable further leak detection by means of a probe.

FORE LINE - The line between a fore pump and the pump it backs.

FORE-LINE VALVE - A vacuum valve placed in the fore line to permit isolation of the diffusion pump from its backing pump.

FOREPRESSURE* - The total pressure on the outlet side of a pump measured near the outlet port. Sometimes called the back pressureφ or backing pressure. (The term backing pressure is commonly used in England and is preferred by the British Committee). Also known as outlet pressure, exhaust pressure, or discharge pressure. In discussing the action of a vapor jet the term forepressure may be used to designate the total pressure of the gas against which the jet impinges.

FORE PUMP* - The pump which produces the necessary fore vacuum for a pump which is incapable of discharging gases at atmospheric pressure. Sometimes called the backing pump.

FORE VACUUM* - The gas-filled space on the outlet side of a vapor jet or a pump. Sometimes called the primary vacuum.

FRACTIONATING PUMP* - A multi-stage vapor pump in which the vapor supplied to the first stage (jet nearest the high vacuum) has been purged of the more volatile impurities, resulting from decomposition or contamination, by the partial condensation and refluxing of vapor in the condenser and the circulating of the condensed pump fluid through a series of boilers feeding the various stages so that the unwanted volatile constituents will be ejected in the states closest to the fore vacuum.* Also called a self-fractionating or purifying pump.

GAGE PRESSURE - A term used in engineering literature to indicate the difference between the absolute pressure and atmospheric pressure as read from a differential manometer having atmospheric pressure on the reference side. This term is seldom used in vacuum technology.

GAS - Gas is defined as the state of matter in which the molecules are practically unrestricted by intermolecular forces so that the molecules are free to occupy any space within an enclosure.* In vacuum technology the word gas has been loosely applied to the non-condensable gas and vapor within a vacuum system.

GAS BALLAST* - The venting of the compression chamber of a mechanical pump to the atmosphere to prevent condensation of condensable vapors within the pump. Also called vented exhaust.

GAS BALLAST PUMP - A mechanical pump (usually of the rotary type) equipped with an inlet and valve through which a suitable quantity of atmospheric air or "dry" gas can be admitted into the compression chamber so as to prevent condensation of vapors within the chamber by maintaining the partial pressure of the vapors below the saturation value. Sometimes called a vented-exhaust mechanical pump.

GAS RATIO - The ratio of ion current to a negative electrode to the electron current to a positive electrode which serves as a relative measure of gas pressure in a sealed-off electronic tube. Also called vacuum factor.φ (Section 1.8.2.1.)

GASKET - A replaceable sealing device, usually of annular form, constructed from a compressible elastic or semi-elastic solid with or without stiffening means.

GASKET SEAL - A seal which is effected by compressing a gasket between the parts to be sealed.

GEAR PUMP - A pump for liquids or gases which entrains the fluid between the teeth of a pair of gears and the wall of the pump casing which fits closely around the gears except in the exhaust region where the teeth engage and the intake region where the teeth disengage.

GETTER - a. (Noun) A material which is included in a vacuum system or device for removing gas by sorption.*
    b. (Verb) To remove gas by sorption.

GETTER-ION PUMP - An ion-sorption pump in which a getter is continuously or intermittently vaporized and condensed on the trapping surface to give a fresh deposit of sorbent. (Section 2.12.6.3.)

GUARD VACUUM* - An enclosed evacuated space intermediate between the vacuum system and the atmosphere to reduce the leak rate into the vacuum system through a seal which can not be made tight.

HALOGEN LEAK DETECTOR - A leak detector which responds to tracer gases which contain one or more of the chemical elements chlorine, bromine, fluorine and iodine in their molecule. Also called halogen-sensitive leak detector or halide leak detector. The copper-flame detector or halide torch consists of a Bunsen burner with flame impinging on a copper plate or screen and a hose with sampling probe to carry tracer gas to the air intake of the burner. The hot-platinum halogen detector depends on the variation of positive ion emission from a heated platinum anode when halogen molecules enter the sensing element. Since the copper-flame halogen detector is seldom used in present practice, the hot-platinum halogen detector is commonly referred to as the halogen leak detector. (Sections 2.2. and 2.7.)

HANGUP - A type of virtual leak caused by the slow release of absorbed or entrapped tracer gas in a leak detector vacuum system. Hangup is one major cause of background in the leak detector. This is not an A.V.S. definition.

HELIUM DRIFT - In the case of leak detection with a helium probe, the drift of helium to a leak or permeable gasket located at a point sufficiently remote from the end of the probe to mislead the operator into suspecting the area near the probe.(Section 1.5.2.2.1 and 1.5.2.2.2.)

HELIUM BOMBING - A method of leak detection in which hermetically sealed units containing an internal volume are subjected to a high helium pressure prior to being bell jar tested. If leaks are present in the sealed unit, the high helium pressure will drive some helium into the internal volume and may be subsequently detected during bell jar testing. Also see back-pressurizing testing.This is not an A.V.S. definition. (Section 1.3.4.3.2.)

HIGH VACUUM*- The term high vacuum has frequently been defined as a pressure less than some upper limit, such as 1 mm Hg or 1 μHg. High vacuum, and similar vacuum terms, should not be defined as a pressure but rather as the condition or state in a gas-filled space at pressures less than some upper limit or within specified limits.

a. High vacuum is defined as the condition in a gas-filled space at pressures less than 10^{-3} Torr.*

b. The following classification of degrees of "high vacuum" has been proposed:

Condition	Pressure Range
HIGH VACUUM:	10^{-3} to 10^{-6} Torr (microtorr range)
VERY HIGH VACUUM:	10^{-6} to 10^{-9} Torr (nanotorr range)
ULTRA-HIGH VACUUM*:	10^{-9} Torr and below*

HIGH-VACUUM VALVE - A valve designed to permit system pressures in the high vacuum region. The combined inleakage rate and outgassing rate for such valves must be less than one tenth of the product of the desired ultimate pressure and the conductance of the open valve.

HOLDING PUMP*- A fore pump used to hold a vapor pump at efficient operating conditions while a roughing pump reduces the system pressure to a point at which the valve between the vapor pump and the system can be opened without stopping the flow of vapor from the nozzles.

HOOD TEST - An overall test in which an object under vacuum test is enclosed by a "hood" which is filled with tracer gas so as to subject all parts of the test object to examination at one time. (Section 1.5.2.1.1.)

HOT CATHODE IONIZATION GAGE- See ionization vacuum gage.

IDEAL GAS - A gas which obeys Boyle's law and has zero heat of free expansion (or also obeys Charles' law). Also known as a perfect gas.

IMPEDANCE* - The reciprocal of the conductance. Also called resistance (Section 1.3.2. and 1.4.1.)

IMPINGEMENT RATE* - The number of molecules which strike a plane surface per square centimeter per second in a gas at rest. Also known as rate of incidence.

IMPLOSION - The rapid inward collapsing of the walls of a vacuum system or device as the result of failure of the walls to sustain the pieces with possible danger to nearby equipment and personnel. (Section 1.5.1.4.4.)

INLEAKAGE RATE - The combined leak rate (in pressure-volume units per unit time) from all existing leaks in a specified evacuated vessel. Sometimes expressed in terms of the rate of rise of pressure in the isolated vessel.

INLET PRESSURE* - In connection with performance data on pumps, when not otherwise specified, inlet pressure refers to the "total static pressure" measured in a standard testing chamber by a vacuum gauge located near the inlet port (1955 Vacuum Symposium Transactions, Committee on Vacuum Techniques, Inc. pp. 91 - 95). Sometimes referred to as intake pressure, fine pressure or, head pressure.

INLINE EXHAUST SYSTEM - A pumping system consisting of a series of packaged pumping units connected as a train on guide rails and moved past fixed stations for performing the necessary steps involved in the exhaust and processing of a specified vacuum device.

INTRINSIC SPEED - The intrinsic throughput divided by the total intake pressure (exclusive of pump fluid vapor) measured at a specified point near the throat (or nozzle clearance area) of a vapor pump. $S_i = Q_i/P_i$

ION PUMP - An electrical device for pumping gas comprising a means for ionizing the gas and a system of electrodes at suitable potentials, and in some cases also a magnetic field, which causes the ions formed to move towards an auxiliary pump or trap. (Section 2.12.5.2.2.)

ION-SORPTION PUMP - A pump which combines the action of an ion pump and a sorption pump by driving the ions into the sorbent.

ION SOURCE - That part of a spectrometer tube in which tracer gas is ionized preliminary to being detected. (Section 2.1.3.1.8.3.)

IONIZATION VACUUM GAUGE - A vacuum gauge comprising a means of ionizing the gas molecules and a means of correlating the number and type of ions produced with the pressure of the gas. Various types of ionization gauge are distinguished according to the method of producing the ionization. The common types are as follows:

a. Hot cathode ionization gauge - The ions are produced by collisions with electrons emitted from a hot filament (or cathode) and accelerated by an electric field. Also called hot filament ionization gauge, ionization gauge, or simply ion gauge. The Bayard-Alpert ionization gauge employs a tube with an electrode structure designed to minimize X-ray induced electron emission from the ion collector.

b. Cold cathode ionization gauge - The ions are produced by a cold cahtode discharge, usually in the presence of a magnetic field which lengthens the path of the electrons between cathode and anode. The discharge tube is a transparent tube in which the color and form of a cold-cathode discharge (without the presence of a magnetic field) gives an indication of the pressure and the nature of the gas. The Philips ionization gauge,is a cold-cathode ionization gauge in which a magnetic field is directed parallel to the axis of an annular electrode (normally the anode) located between two plate electrodes perpendicular to the axis. Various modifications of the Penning gauge are named after the inventors, and certain types are referred to as magnetron vacuum gauges.

c. Radioactive ionization gauge - The ions are produced by radiations (usually alpha particles) emitted from a radioactive source. (Section 2.3. and 2.12.)

ISOLATION TEST - A method of determining whether a leak is present in a system, or of obtaining an estimate of its magnitude, by observing the rate of rise of pressure in the evacuated system when the system is isolated from the pump. Also called rate of rise test. (Section 2.3.4.2.)

JOY BOMB TEST - Used in the electronic components field, a bomb test in which a detergent (Joy) is used to enhance the passage of liquid into the component. This is not an A.V.S. definition. See Bomb Test.

KINETIC VACUUM SYSTEM - See Demountable High Vacuum System.

KNUDSEN FLOW* - The flow of gases through ducts and tubes under conditions intermediate between laminar viscous flow and molecular flow.* Also called transition flow. Knudsen's equation of flow applies to this intermediate range for the case of pipes with circular section. (Section 1.4.1.1.3.)

L-RING GASKET - A molded circular gasket of L-shaped cross section fitting over the outside and lower edge of bell jars or unflanged pipes.

LEAK - In vacuum technology a hole, or porosity, in the wall of an enclosure capable of passing gas from one side of the wall to the other under action of a pressure or concentration differential existing across the wall. (Section 1,3,1. and 1.3.2.)

LEAK DETECTOR - A device for detecting and locating leaks, and indicating the magnitude thereof.

LEAK RATE - In leak detection practice leak rate is defined as the rate of flow (in pressure-volume units per unit time) through a leak with gas at a specified high pressure (usually atmospheric pressure) on the inlet side and gas at a pressure on the exit side which is low enough to have negligible effect on the rate of flow. (Section 1.3.1.)

LEAK RATE* - The quantity of gas in pressure-volume units at room temperature flowing into the system or through the pump from an external source in unit time.* Recommended unit in Torr . liter per second at 20° C.* (Also expressed in micron-liters per second, or micron cubic feet per minute, or cc-atmos/sec at 25°C). (Section 1.3.1.)

LOAD* - The quantity of gas (not including pump fluid vapor) in mass units flowing across the inlet port of a pump in unit time.* Sometimes called capacity. Typical units are pounds per hour or grams per hour.

LOW VACUUM* - The term low vacuum applies to the condition in a gas-filled space at pressures less than 760 Torr and greater than some lower limit. It is recommended that this lower limit be chosen at 25 Torr corresponding approximately to the vapor pressure of water at 25° C and to one inch of mercury.*

a. Two self-consistent classification schemes which have been proposed for the pressure range from 760 to 10^{-3} Torr are as follows:

	Condition	Pressure Range
40.	LOW VACUUM:	760 to 25 Torr
41.	MEDIUM VACUUM:	25 to 10^{-3} Torr
42.	ROUGH VACUUM:	760 to 1 Torr (Torr range)
43.	FINE VACUUM:	1 to 10^{-3} Torr (millitorr range)

LUSEC$^{\Phi}$ - A unit of flow rate equal to 1 micron liter per second. (Section 1.3.1.2.)

MASKING - The covering of a section of a test object so as to prevent tracer gas from entering leaks that may exist in the covered section. (Section 1.5.2.2.)

MASS SPECTROMETER - An instrument which is capable of separating ionized molecules of different mass/charge ratio and measuring the respective ion currents. The mass spectrometer may be used as a vacuum gauge to measure the partial pressure of a specified gas, as a leak detector sensitive to a particular tracer gas, or as an analytical instrument to determine the percentage composition of a gas mixture. Various types are distinguished by the method of separating the ions. The principal types are as follows:

a. Dempster M.S. - The ions are first accelerated by an electric field through a slit, and are then deflected by a magnetic field through 180° so as to pass through a second slit.

b. Bainbridge-Jordan M.S. - The ions are separated by means of a radial electrostatic field and a magnetic field deflecting the ions through 60° so arranged that the dispersion of ions in the electric field is exactly compensated by the dispersion in the magnetic field for a given velocity difference.

c. Bleakney M.S. - The ions are separated by crossed electric and magnetic fields. Also called crossed-fields M.S.

d. Nier M.S. - A modification of the Dempster M.S. in which the magnetic field deflects the ions through 60°.

e. Time of Flight M.S. - The gas is ionized by a pulse-modulated electron beam and each group of ions is accelerated toward the ion collector. Ions of different mass/charge ratio traverse their paths in different times.

f. Radio-frequency M.S. - The ions are accelerated into a radio-frequency analyzer in which ions of a selected mass/charge ratio are accelerated through openings in a series of spaced plates alternately attached across a radio-frequency oscillator. The ions emerge into an electrostatic field which permits only the ions accelerated in the analyzer to reach the collector.

g. Omegatron M.S. - The ions are accelerated by the cyclotron principle. (Section 2.1. and 2.1.3.2.1.)

MASS SPECTROMETER LEAK DETECTOR - A mass spectrometer adjusted to respond only to the tracer gas. Helium is commonly used as the tracer gas, and thus the instrument is normally referred to as a helium leak detector. (Section 2.1.)

MEAN FREE PATH(of any particle)* - The average distance that a particle travels between successive collisions with the other particles of an ensemble.* In vacuum technology the ensemble of particles of interest comprises only the molecules in the gas phase. (Section 1.4.1.1.2.)

MEASURED SPEED(of pump)* - The leak rate of gas admitted from a flow measuring device divided by the total pressure measured near the mouth (or inlet port) of a pump with a specified gauge mounted in a specified testing head.* $S_m = L_m/P$

MECHANICAL PUMP - A pump which moves the gas by the cyclic motion of a system of mechanical parts such as pistons, eccentric rotors, vanes, valves, etc.

MICROBAR - The unit of pressure in the c.g.s. system and equal to one dyne per square centimeter. In British literature the term barye has been used. Abbreviated as μb.

MICRON OF MERCURY - A unit of pressure equal to 1/1000th of one millimeter of mercury pressure. Abbreviated as μ of Hg or μ Hg.

MICROTORR - A proposed new unit of pressure equal to 10^{-6} Torr.

MILLI-MICRON OF MERCURY - A unit of pressure equal to 10^{-6} mm of Hg. Abbreviated as mμ.Hg.

MILLIBAR - A unit of pressure equal to 10^{-3} dynes per square centimeter. Abbreviated as mb.

MILLIMETER OF MERCURY - A unit of pressure corresponding to a column of mercury exactly one millimeter high at 0° C under standard acceleration of gravity of 980.665 cm/sec^2 By "mercury at 0° C" is meant a hypothetical fluid having an invariable density exactly 13.5951 g/cm^3. Abbreviated as mm Hg. Also see Torr)

MILLITORR - A proposed new unit of pressure equal to 10^{-3} Torr.

MINIMUM DETECTABLE LEAD - a. The size of the smallest leak, expressed in terms of mass flow per unit time, that can be unambiguously detected by a given leak detector in the presence of noise and background.

b. The product of the minimum detectable pressure change and the pumping speed at the detector. (Appendix III)

MINIMUM DETECTABLE PRESSURE CHANGE - The pressure producing an indication of three times the noise level. (Appendix III)

MOLECULAR EFFUSION* - The passage of gas through a single opening in a plane wall of negligible thickness where the largest dimension of the hole is smaller than the mean free path.* (Section 1.4.1.1.2)

MOLECULAR FLOW* - The flow of gas through a duct under conditions such that the mean free path is greater than the largest dimension of a transverse section of the duct.* (Section 1.4.1.1.2.)

MULTI-STAGE PUMP - A pump which compresses the gases in two or more stages within a common pump casing.

NEEDLE VALVE - In vacuum technology, a throttling valve in which a needle can be moved against a long needle-shaped seat, or a valve so designed as to give equivalent throttling action.

NET SPEED* - a. (Measured value) - The throughput divided by the pressure at a specified point in the system at a distance from the inlet port of the pump.* Also called simply the speed of flow at the specified point. $S_n = Q/P_x$

b. (Calculated value) - The product of the measured pump speed and the conductance of the pipe line divided by the sum of the measured pump speed and the conductance.* $S_n = S_m C/(S_m + C)$

NOISE LEVEL - In the case of a leak detector, the spurious output, expressed in suitable terms, exhibited by the detector in the absence of an output due to tracer gas. (Appendix III)

NON-CONDENSABLE GAS* - A gas whose temperature is above its critical temperature, so that it cannot be liquified by increase of pressure alone.

O-RING GASKET - A circular cross-section, made to specified dimensional tolerances.

OCCLUSION - The trapping of undissolved gas in a solid during solidification.

OPERATIONAL SPEED - The ratio between a small change in "external" throughput and the corresponding small change in pressure measured at the throat of a vapor pump. $S_o = \Delta Q_e / \Delta p_i$

OUTGASSING* - The spontaneous evolution of gas from a material in a vacuum.* (Section 1.5.3. and 2.3.4.2.1)

PACKAGED PUMPING SYSTEM - A complete pumping system mounted on a framework or dolly requiring only to be connected to the electrical power line, water line, and in some cases also a compressed air line to be ready for operation.

PACKED VALVE - A valve type in which a packing material, placed between the stem and the bonnet, helps to prevent leakage at the point where the stem passes through the bonnet.

PACKLESS VALVE - A valve so constructed that the seal between stem and bonnet is positive, in contradistinction to a packed valve or one which depends on any other kind of sliding seal.

PALLADIUM BARRIER LEAK DETECTOR - A leak detector using hydrogen as the tracer gas and using the principle of hydrogen diffusing rapidly through a hot Palladium barrier into an evacuated vacuum gauge. Also called palladium leak detector. (Section 2.12.5.2.3.1.)

PALLADIUM LEAK DETECTOR - See Palladium Barrier Leak Detector

PARTIAL PRESSURE - The pressure of a designated component of a gaseous mixture. This may be separately measured in some cases by suitable selection of gauges, traps, or analytical trains. When the percentage composition of the mixture is known, the partial pressure may be calculated from the total pressure by Dalton's law of partial pressure. This law states that the total pressure in a mixture of perfect gases is equal to the sum of the pressures exerted by the component gases were each to occupy the same volume by itself. (Section 1.5.1.5.1.)

PARTS PER MILLION TESTING. A method of leak testing utilizing the leak detectors ability to detect the helium partial pressure at atmospheric pressure into which an object, presurized with tracer gas is leaking. This is not an A.V.S. definition. This term is predominantly used by the Consolidated Electrodynamics Corporation. (Section 2.1.5.3.1.)

PENNING GAGE - See Ionization Vacuum Gage.

PERFECT GAS - See Ideal Gas.

PERMEABILITY COEFFICIENT* - The steady-state rate of flow of gas through unit area and thickness of a solid barrier per unit pressure differential at a given temperature. Usually expressed in cubic centimeters(S.T.P.) per second, per square centimeter of cross section, per millimeter of thickness, per centimeter of mercury pressure drop across the barrier.* Also called simply permeability. (Section 1.4.1.2.)

PERMEATION - As applied to gas flow through solids permeation is the passage of gas into, through, and out of a solid barrier having no holes large enough to permit more than a small fraction of the gas to pass through any one hole. The process always involves diffusion through the solid and may involve various surface phenomena, such as sorption, dissociation, migration, and desorption of the gas molecules. (Section 1.4.1.2.)

PHILIPS IONIZATION GAGE - See Ionization Vacuum Gage.

PIRANI GAGE - See Thermal Conductivity Vacuum Gage.

POISEUILLE FLOW* - The particular case of laminar viscous flow through a long pipe of circular section.* (Section 1.4.1.1.1.)

PRESSURE(in a gas) - The net rate of transfer of momentum in the direction of the positive normal to an imaginary plane surface of specified area located in a specified position in the gas by molecules crossing the surface in both directions, momentum transmitted in the opposite direction being counted as negative, divided by the area of the surface. In general it is assumed that the area of the imaginary plane surface is small enough so that the pressure with respect to any part of the surface is equal (within narrow limits) to the pressure based on the whole surface. Different kinds of pressure (static, dynamic, partial, total, vapor, etc.) are distinguished by the orientation of the surface with respect to mass-flow velocity vectors or by the restriction to a specified set of molecular species crossing the imaginary surface.

a. Static pressure may be defined as the pressure with respect to a stationary surface tangent to the mass-flow velocity vector, or it may be defined as the pressure with respect to a surface translated with a velocity equal and parallel to the mass-flow velocity.

b. Dynamic pressure(or kinetic pressure) is defined as the pressure with respect to an imaginary stationary surface whose positive normal is parallel to the mass-flow velocity vector.

c. Impact pressure is a measured quantity obtained by placing an open-ended tube, known as an impact tube or Pitot tube, in a gas stream and noting the pressure in the tube on a suitable manometer. Since the pressure is exerted at a stagnation point, the impact pressure is sometimes referred to as the stagnation pressure or total pressure.

d. Velocity pressure or velocity head is the difference between dynamic (or total) pressure and static pressure.

e. Total pressure usually refers to the pressure determined by all of the molecular species crossing the imaginary surface.

f. Partial pressure refers to the transfer by a specified molecular species.

g. The term pressure when used alone can be assumed to refer to the total pressure in a gas at rest or else to refer to the static pressure in a gas flowing under steady state conditions.

PRESSURE(as measured in a vacuum system) - The term pressure as used in vacuum technology usually must be interpreted as the quantity measured at a specified time by a so-called "vacuum gauge", whose sensing element is located in a cavity (gauge tube) with an opening oriented in a specified direction at a specified point within the system, assuming a specified calibration factor. The sensitivity of the sensing element is in general not the same for all molecular species, but the gauge reading is frequently reported using the calibration factor for air regardless of the composition of the gas. The opening to the gauge tube is often carelessly oriented with respect to mass-flow vectors in the gas (which is seldom at rest), and errors due to variations in wall temperatures of tube and system are frequently neglected. The actual total pressure in a high-vacuum system can not usually be measured by a single gauge, but in vacuum technology the term "total pressure" is sometimes used to refer to the reading of a single untrapped gauge which responds to condensable vapors as well as permanent gases.

PRESSURE COOKER TEST - A bomb test used by the electronics industry in which a houseware pressure cooker is used to attain the stream-list water pressure cycle. Also see Bomb Test. This is not an A.V.S. definition.

PRESSURE-DYE TEST - A leak test in which the systems are immersed in a liquid dye or fluorescent oil, for the purpose of driving liquid through possible leakage paths and so make its presence visible when the excess liquid has been removed from the exterior. This is not an A.V.S. definition. (Section 2.10.4.1.)

PRESSURE-EVACUATION TEST - See Back-Pressurizing Testing.

PRESSURE PROBE - See Sampling Probe.

PRESSURE TESTING - A leak detecting procedure in which tracer gas is introduced under pressure into the enclosure under examination, and detected as it is emitted from a leak. In this handbook this is called Leak Location using a Detector Probe Technique.

PROBE - A tube having a fine opening at one end, used for directing a stream of tracer gas. (Section 1.3.4.2. and 1.5.2.2.)

PROBE GAS - A tracer gas which issues from a more-or-less fine orifice so as to impinge on a restricted test area. (Section 1.3.4.2. and 1.5.2.2.)

PROBE TEST - A leak test in which the tracer gas in applied by means of a probe so that the area covered by the tracer gas is localized. This enables the individual leaks to be located. In this handbook, this is called Leak Locating with a Tracer Probe (Section 1.3.4.2.)

PROCESS PUMP - The process pump is the pump which maintains the system at the operating pressure.(Term introduced by the British Committee).

PUMP-DOWN TIME - See Time of evacuation.

PUMP-OUT - A port of channel with entrance located between two gaskets which are in series, and through which any leakage from the outer one can be pumped away.

PUMP-OUT TUBULATION - A tube extending from a vacuum device through which gas is pumped and which is usually permanently sealed off after the device has been evacuated. Sometimes called exhaust tubulation.

QUICK CONNECT VACUUM COUPLING - See Vacuum Coupling.

RATE OF RISE - The time rate of pressure increase at a given time in a vacuum system which is suddenly isolated from the pump by a valve. The volume and temperature of the system are held constant during the rate of rise measurement. (Section 2.3.4.2.)

REVERSE PROBE - See Sampling Probe.

RESIDUAL VAPOR PRESSURE - The vapor pressure in a system which has reached the ultimate or limiting value of total pressure.

RESISTANCE - See Impedance

RESPONSE TIME* - The time for a leak detector or a leak detection system to yield a signal output equal to 63% of the maximum signal attained when tracer gas is applied indefinitely to the system under test.* (Section 1.3.4.1.2.2.)

REYNOLDS NUMBER- As applied to the flow of gas through a circular tube the Reynolds number is a dimensionless quantity equal to the product of the gas density in grams per cubic centimeter times the flow velocity in centimeters per second times the tube diameter in centimeters divided by the viscosity coefficient in poises. $R_e = \rho v\ d/\eta$ (Section 1.4.1.1.4.)

ROOTS BLOWER PUMP - A rotary blower pump having a pair of two-lobe interengaging impellers of special design.

ROTARY BLOWER PUMP - A pump without a discharge valve which moves the gas by the propelling action of one or more rapidly rotating members provided with lobes, blades, or vanes. Sometimes called a mechanical booster pump when used in series with a mechanical fore pump. Rotary blowers are sometimes classified as either axial flow or cross flow types depending on the direction of flow gas.

ROTARY EXHAUST SYSTEM - A pumping system having several intake ports arranged on the circumference of a circular table which can rotate uniformly or intermittently so that the devices to be evacuated can be moved past fixed stations for performing the necessary steps in a batch process, such as loading, evacuating, degassing, sealing-off, and unloading. (Section 2.3.4.2.3. and 2.4.4.2.)

ROTARY VACUUM SEAL - A seal designed to prevent leakage at the point where a rotating shaft enters a vacuum system.

ROUGHING LINE - A line running from a mechanical pump to a vacuum chamber through which preliminary pumping is conducted in the rough vacuum range.

ROUGHING PUMP* - The pump used to reduce the system pressure to the point at which a vapor pump (or other pump requiring a fore vacuum) can take hold and operate efficiently. The roughing pump may then also be used as the fore pump for the vapor pump, or the roughing pump may be shut off and a smaller pump used as fore pump when the gas load is relatively small.

ROUGHING TIME* - The time required to pump a given system from atmospheric pressure to the take-hold pressure for the vapor pump (or other high-vacuum pump) or to a pressure at which valves to the vapor pump can be opened without stopping the flow of vapor from the nozzles.*

ROUGHING VALVE - A vacuum valve placed in a roughing line to isolate the vacuum system from the roughing pump.

SAMPLING PROBE - A device used in pressure testing and so designed as to collect tracer gas from a restricted area of the test object and feed it to the leak detector. Also called pressure probe, sniffer, or reverse probe. In this handbook, it is called a Detector probe (Section 1.3.4.2. and 1.5.2.2.)

SATURATION VAPOR PRESSURE - The vapor pressure in an isolated system under equilibrium conditions in the presence of the condensed phase.

SEAL-OFF - The final act of sealing a vacuum device after evacuation by a pumping system.

SEARCH-GAS - See Tracer Gas.

SEAT GASKET - The gasket which seals the port when the valve is closed.

SENSITIVITY - a. In the case of a leak detector, the smallest concentration of tracer gas in air that can be detected by the device.
b. The sensitivity of a leak sensing element is the minimum partial pressure of air which, if changed to the same pressure of search gas, produces the minimum detectable indication (e.g.,an indication of three times random noise in three seconds). (Appendix III)

SNIFFER - See Sampling Probe.

SOAP BUBBLE TEST - A type of pressure testing in which the tracer gas is detected by bubbles formed in a layer of soap solution applied to the surface of the test object. (Section 2.5)

SORPTION - The taking up of gas by absorption, adsorption, chemisorption or any combination of these processes. (Sections 1.5.3. and 2.3.4.2.1.)

SORPTION PUMP - A pump with a renewable trapping surface which reduces the partial pressure of gases by adsorption, absorption, or chemisorption.

SPARK COIL LEAK DETECTOR - A high frequency discharge coil of the Tesla type which indicates pin holes in glass vacuum systems by a spark jumping between the core of the coil and the pinhole. (Section 2.11)

SPECTROMETER TUBE - The sensing element of a mass spectrometer (Section 2.1.3.1.8.)

SPEED* - The speed of a pump for a given gas in the ratio of the throughout of that gas to the partial pressure of that gas at a specified point near the mouth(or inlet port) of a pump.* Also known as the <u>admittance</u> for a given gas. $S_a = Q_a/P_a$

SPEED OF EXHAUST* - The magnitude of the rate of reduction of pressure in the system multiplied by the volume and divided by the measured pressure.* Also called <u>speed of exhaustion</u>. $S_e = -(dP/dt)(V/P)$

SPRAY PROBE - See Probe.

SQUEALER(AUDIBLE LEAK INDICATOR) - An auxiliary to a leak detector which converts the output signal to an audible note whose frequency is a function of the leak size.

STANDARD LEAK - a. A device which permits leakage through it, at a specified rate, of a specified gas, with atmospheric pressure at one end of the device and a pressure on the other side sufficiently low to have negligible effect on the leak rate.

b. A capillary or porous wall leak, usually in a glass or metal tube, whose dimensions have been adjusted to give a conductance within specified limits for a specified gas at a standard reference temperature with specified inlet and exit pressures. Standard leak for attaching to vacuum test manifolds with air at atmospheric pressure exposed to the inlet, are usually protected by filters to avoid clogging by dust particles. Standard leaks for calibrating mass spectrometers are usually fused to a glass reservoir containing the specified gas at a known high pressure.

c. A device providing a known throughput into a vacuum system. Also referred to as a calibrated leak. (Section 1.6)

THERMAL CONDUCTIVITY VACUUM GAUGE - A vacuum gauge containing two surfaces at different temperatures between which heat can be transported by the gas molecules so that changes in the temperature (or in the heating power required to maintain constant temperature) of one of the surfaces can be correlated with the gas pressure by calibration against a McLeod gauge. Various types of thermal conductivity gauge are distinguished according to the method of indicating the temperature change. The common types are listed below:

a. Pirani gauge - An increase of pressure from the zero point causes a decrease in the temperature of a heated filament of material having a large temperature coefficient of resistance thus unbalancing a Wheatstone bridge circuit (or the circuit is adjusted to maintain the filament temperature constant).

b. Thermocouple gauge - The decrease in temperature of a heated filament as the pressure rises is indicated by decreased e.m.f. in a thermocouple circuit having the junction in thermal contact with the center of the heated filament.

c. Thermistor gauge - A form of Pirani gauge employing a thermistor as the heated element.

d. Bimetallic strip gauge - Deflection of a bimetallic strip with changing temperature indicates the changes in pressure.

THERMAL TRANSPIRATION* - The passage of gas through a connection between two vessels at different temperatures resulting in a pressure gradient when equilibrium is reached.* Under conditions of molecular flow the equilibrium condition is expressed by

$$P_a/P_b = \sqrt{T_a/T_b}$$

where P_a and T_a are the pressure and absolute temperature in one vessel and P_b and T_b are the pressure and absolute temperature in the other.

THERMOCOUPLE GAGE - See Thermal Conductivity Vacuum Gage

THROTTLING - Reducing the net pumping speed of a pumping system by partially closing a valve or installing a section of pipe line with low conductance. (Section 1.3.4.1.2.1 and 2.12.4)

THROUGHPUT* - The quantity of gas in pressure-volume units at a specified temperature flowing per unit time across a specified open cross-section of a pump or pipe line.* The specified temperature may be the actual temperature of the gas or a standard reference temperature. It is recommended that throughput be referred to standard room temperature. The recommended unit of throughput is the Torr . liter per second at 20° C.* Other units of throughput in common use are micron liters per second at 25° C and micron cubic feet per minute at 68° F.

a. Under conditions of steady-state conservative flow the throughput across the entrance to a pipe is equal to the throughput at the exit. In this case throughput can be defined as the quantity of gas flowing through a pipe in pressure-volume units per unit time at room temperature.

b. Values of throughput calculated from pressure measurements depend on previous use of calibrated leaks or on theoretical conductance formulas. When untrapped gauges are used to measure pressure, the calculated throughput is influenced by the partial pressure of vapors. However, the effect of backstreaming pump fluid vapor is frequently neglected in calculating the flow of "gas" across the entrance of a vapor pump or through a pipe near the pump. An alternative definition of throughput might therefore state explicitly that pump fluid vapor is not included in the quantity of "gas" involved in the flow calculation.

TIGHT - Free from leaks according to a given specification.

TIME CONSTANT* - The ratio of the volume of a system to the pumping for a specified gas.* Term used in leak detection. (Section 1.3.4.1.2.2.)

TIME OF EVACUATION* - The time required to pump a given system from atmospheric pressure to a specified pressure. Also known as *pump-down time* or *time of exhaust.*Φ

TORCHING - The degassing of a vacuum system by application of a gas burner flame to the walls during the pumping process.

TORR* - Suggested international standard term to replace the English term millimeter of mercury and its abbreviation mm of Hg (or the French mm de Hg). Both *Tor* and *Torr* have been used in Germany, the latter spelling being more common and the one officially adopted by the German Standards Association. The Torr is defined as 1/760 of a standard atmosphere or 1.013,250/760 dynes per square centimeter. This is equivalent to defining the Torr as 1333.22 microbars and differs by only one part in seven million from the International Standard millimeter of mercury. It is recommended that Torr not be abbreviated. However, the abbreviation t has been used.

TRACER GAS* - A gas which, passing through a leak, can then be detected by a specific leak detector and thus disclose the presence of a leak. Also called *search gas.* In this handbook, Tracer Gas is the Preferred Term.

TRACER PROBE LEAK LOCATION - See Probe Test

TRANSITION FLOW - See Knudsen Flow

TRANSPIRATION - The passage of gas through a porous solid (usually under conditions of molecular flow).

TRAP(adsorption)* - A chamber containing activated charcoal, or other medium for adsorbing gases and vapors (usually at room temperature), designed to reduce the partial pressure of adsorbable gases in the system.*

TRAP(refrigerated)* - A system of cooled walls, plates, or tubing placed beyond the baffle or the condenser to reduce the partial pressure of vapor migrating from the pump, or from the vacuum system, and from which the condensed vapor can not return to the pump boiler.*

TROLLEY EXHAUST SYSTEM - A pumping system containing one or more intake ports arranged so that processing equipment can be moved up to or over the devices attached to the ports. (Section 1.5.2.1.3.)

ULTIMATE PRESSURE* - The limiting pressure approached in the vacuum system after sufficient pumping time to establish that further reductions in pressure will be negligible. Sometimes called the ultimate vacuum Φ. The terms blank-off pressure or base pressure are also sometimes used in referring to a pump under test.

ULTRA-HIGH VACUUM SYSTEM - A system constructed with materials having a very low outgassing rate and which can be thoroughly degassed so that pressures less than 10^{-9} Torr can be achieved with pumps of moderate speed. Sometimes called a static vacuum system. Φ

V-RING GASKET - A gasket of roughly V-shaped cross-section.

VACUA - Plural of vacuum.

VACUUM - In vacuum technology the term vacuum refers to a given space filled with gas at pressures below atmospheric pressure. The word "vacuum" was originally a Latin word meaning empty. Since it is not possible in practice to remove all matter from a space of ordinary magnitude, degrees of vacuum (e.g., high vacuum, ultra-high vacuum) have been arbitrarily distinguished according to the pressure ranges characteristic of various techniques, pumping equipment, or laws of gas flow.

VACUUM BREAKER - See Air-Inlet Valve

VACUUM CEMENT - A cement of low vapor pressure (usually less than 10^{-8} Torr) capable of maintaining its properties in a vacuum.

VACUUM COUPLING (quick connect) - A device for quickly making a joint between two elements of a vacuum system, the joint being easily borken and remade.

VACUUM FACTOR Φ - See Gas Ratio.

VACUUM GAUGE CONTROL CIRCUIT - The part of an electically operated vacuum gauge which comprises the electrical circuit necessary to energize the tube, control and measure currents or voltages, and in some cases to supply power for degassing the tube elements. Does not include the vacuum gauge tube or any meter or recorder which is not an integral part of the cabinet or panel assembly in which the circuit is mounted. Also referred to as the vacuum gauge power supply, the vacuum gauge power rack, the vacuum gauge control box, or simply the the vacuum gauge control. Various terms are formed by substituting in the above in place of the word "vacuum" the words Pirani, thermocouple, ionization, Philips, etc. referring to particular types of vacuum gauges.

VACUUM GAUGE TUBE - The part of a vacuum gauge which contains the elements exposed directly to the vacuum system including the pressure sensing means, the envelope or means for supporting the operating elements, and any connecting tube attached permanently to the envelope. External connecting means, such as electrical connectors, may be included in referring to a particular gauge tube. Sometimes called vacuum gauge head. Certain contractions are commonly used in referring to specific types of tubes, e.g. Pirani tube instead of Pirani gauge tube, ion tube instead of ionization gauge tube.

VACUUM GREASE - A grease of low vapor pressure (usually less than 10^{-4} Torr).

VACUUM MANIFOLD - An enclosure with several ports so that a number of vacuum devices may be attached to it at one time for evacuation and processing. (Section 1.5.2.1.2.)

VACUUM PAINT - A paint having a low vapor pressure (usually less than 10^{-8} Torr) when air dried.

VACUUM TESTING - A leak detecting procedure in which the enclosure under examination is evacuated, the tracer gas applied to the outside surface of the enclosure, and the gas detected after entering the enclosure. (this category definition is not used in this handbook)

VACUUM WAX - A wax of low vapor pressure (usually less than 10^{-4} Torr) used for joining, sealing, mending leaks, etc.

VALVE - A device for adjusting the rate of flow of a fluid or for completely stopping the flow. A conventional valve consists of three major elements, namely, the body, the bonnet, and the stem or stem assembly.

VAPOR - A gas whose temperature is below its critical temperature, so that it can be condensed to the liquid or solid state by increase of pressure alone.*

VAPOR PRESSURE - a. The sum of the partial pressures of all the vapors in a system. b. The partial pressure of a specified vapor.

VAPOR PUMP* - a. Any pump employing a vapor jet as the pumping means. Applies to ejector pumps as well as diffusion pumps.
b. A diffusion or condensation pump.Φ

VENTING TIME - The time required to raise the pressure in a given system from the operating value to atmospheric pressure by admitting air through a specified valve and intake line.

VIRTUAL LEAK - a. The evolution of vapor from a cold trap on which the vapor had previously condensed at higher pressures or lower trap temperatures.
b. The semblance of a leak in a vacuum system caused by slow release of sorbed or occluded gas.* The term is sometimes used to include volatilization or disintegration of a material under vacuum. (Sections 1.5.1.2. and 1.5.3.)

VISCOUS FLOW* - The flow of gas through a duct under conditions such that the mean free path is very small in comparison with the smallest dimension of a transverse section of the duct.* This flow may be either laminar or turbulent. (Sections 1.4.1.1.1 and 1.4.1.1.4.)

WILSON SEAL - A type of rotary shaft seal in which a flat gasket has a hole cut with a diameter smaller than the shaft diameter, so that the gasket is constrained to bend, when installed, in such a manner that a line contact is established with the shaft. In the modified Wilson seal two gaskets are used in series with a conical metal spacer.

Appendix C

LEAK TESTING SPECIFICATIONS

American Vacuum Society -- AVS 2.1-1963
American Nuclear Society -- ANS 7.60

Helium Mass-spectrometer Leak-Detector Calibration

1. INTRODUCTION

1.1 Scope. This standard prescribes procedures to be used for calibrating leak detectors of the mass-spectrometer type, that is, for determining a sensitivity figure for such leak detectors. The procedures require the use of a calibrated leak and a standard mixture; the preparation and standardization of these is outside the scope of this standard. Refer to the appropriate AVS standards.

While various leak-tracing gases have been used in conjunction with mass-spectrometer leak detectors, the present standard has reference only to the use of helium-4.

The application of this standard is restricted to leak detectors with rated sensitivity corresponding to leak rates larger than 10^{-11} atm cc/sec. Factors that are unimportant for larger leaks may become significant for gas flows that are substantially less than 10^{-11} atm cc/sec; consequently, a separate standard will cover the calibration of detectors designed for leaks smaller than this figure.

1.2 Mode of Presentation. Sections 2–4 deal with preliminary matters such as definitions.

The determination of sensitivity (Secs. 6 and 7) involves a figure referred to as the minimum detectable signal. The procedure for determining this figure is covered in Sec. 5 for the determination in Sec. 6, and in Sec. 7.2 for the determination in Sec. 7.

Section 6 covers two procedures for determining minimum detectable leak, the second procedure being followed if rubber or other polymeric substances are present in the connections to the standardizing leak. Section 7 presents the procedure for determining minimum detectable concentration ratio.

2. DESCRIPTION OF LEAK DETECTOR

The helium-leak detector is essentially a gas analyzer, employing the mass-spectrometer principle. In the mass-spectrometer tube, a mixture of gases to be analyzed is first ionized, then separated into a series of ion beams or groups, each group ideally representing a single species of gas. (Strictly speaking, the ions in each beam have the same mass-to-charge ratio.) In the helium-leak detector, means are provided for "tuning" the instrument so that only the beam due to helium hits an ion collector. (The detector can generally be tuned to respond to other gases.) The current produced by the beam is amplified, and its magnitude is a measure of the partial pressure of the helium gas in the incoming sample. It will be assumed that the gas ionization is produced by the electrons from a hot filament.

Mass-spectrometer leak detectors consist of a mass-spectrometer tube, a high-vacuum system for maintaining the tube under suitable vacuum with a flow of gas sample through or into the tube, suitable voltage supplies, and an ion-current amplifier. The output of the amplifier is displayed on an output meter, and means are provided for reducing the output so that a large range of leak sizes can be detected and measured. In other words, the leak detector may be set at one of a number of different detection levels, hereafter referred to as *sensitivity settings.*

Since the spectrometer tube must be able to receive a gas sample from the system under test and must also be kept under vacuum, a line is provided for leading gas from the outside into the spectrometer tube, and this line must have an isolation valve in it. In this standard, the line is referred to as the *sample inlet line.*

3. DEFINITIONS

3.1 General. Many of the technical expressions used in this standard are defined in *Glossary of Terms Used in Vacuum Technology,* issued by the American Vacuum Society (Pergamon Press Inc., New York, 1958). Starred definitions are not from the AVS Glossary.

3.2 Channel Leak. A hole, or pores, in the wall of an enclosure, capable of passing gas from one side of the

Approved by American Vaccum Society, Inc., October 1963

Additional copies of this Standard are available at $1 each from the secretary of AVS, P. O. Box 1282, Boston, Mass. 02014.

wall to the other under action of a pressure or concentration differential existing across the wall.

***3.3 Calibrated Leak.** A device that permits leakage through it at a specified rate, of a specified gas, under specified conditions, the downstream side of the device being exposed to a pressure sufficiently low to have negligible effect on the leak rate.

***3.4 Standard Leak Rate.** The rate of flow of atmospheric air of dewpoint less than −25°C through a leak under standard conditions specified as follows: the inlet pressure shall be 1 standard atmosphere ±5%, the outlet pressure shall be less than 0.01 of 1 atmosphere pressure, and the temperature shall be 23°C ± 3.

***3.5 Viscous Leak.** A leak, the flow through which is controlled by viscosity.

***3.6 Membrane Leak.** A leak that produces a gas flow by permeation of the gas through a nonporous wall. For helium, this wall may be of glass, quartz, or other suitable material.

3.7 Concentration Ratio. In this standard, the ratio of partial pressure to total pressure.

3.8 Sensitivity.

3.8.1 In the case of a leak detector, the smallest leak that can be detected. The leak is specified in terms of its standard leak rate (see 3.4). A synonym used in this standard is *minimum detectable leak.*

3.8.2 In the case of a leak detector, the minimum concentration ratio of helium gas in a helium–air mixture that can be detected by the device.

4. TEST CONDITIONS AND APPARATUS

4.1 Ambient Temperature. Ambient temperature shall be 23°C ± 3.

4.2 Ambient Pressure. Ambient pressure shall be 760 Torr ± 5%.

4.3 Calibrated Leaks. Two calibrated leaks may be required: one with a relatively small leak rate and the other with a relatively large leak rate. The small leak is used for determining minimum detectable leak, the large leak for minimum detectable concentration ratio. The small leak may be of the channel type or of the membrane type. The leaks are specified as follows:

4.3.1 Small Channel Leak. This shall have a leak rate such that when helium, at 1 standard atmosphere pressure, is fed to the leak and thence into the leak detector under test, a deflection is produced on the output meter of not less than 100 times the minimum detectable signal (see Sec. 5.3 below). The leak detector shall have been adjusted as in Sec. 4.8 below.

4.3.2 Small Membrane Leak. This shall leak helium at a rate to produce a deflection as specified under Sec. 4.3.1. above. Its leak rate shall be specified in terms of *equivalent air leakage rate.* The equivalent air leakage rate shall be taken as 0.37 times the helium leakage rate through the membrane under specified conditions. A temperature correction shall be specified for the leak, and the correction applied for the difference between the temperature of the leak at the time of use and the temperature specified in the calibration.

4.3.3 Large Calibrated Leak. This shall be a viscous leak of such leakage rate that, when connected to the leak detector with ambient air at the inlet side of the leak, the pressure in the leak detector rises to the optimum (high) operating pressure specified by the manufacturer.

4.4 Helium. This shall be at least 99.9% helium (available from commercial dealers in cylinder gases).

4.5 Helium Mixture. This shall be a helium and air mixture of a known concentration such that it produces a deflection of at least 10 times the minimum detectable signal (see Sec. 7.2) when fed at a pressure of 760 Torr ±5% and at ambient temperature to the large calibrated leak (Sec. 4.3 above) and thence into the leak detector under test. Where applicable, atmospheric air may be used as the helium mixture; in any case, the mixture air shall be obtained from a point at least 5 ft outside the walls of the building housing the test equipment. Helium concentration ratio shall be expressed as a fraction with numerator reduced to unity, and this ratio will be represented by the symbol C. Alternatively, the concentration ratio may be expresed in parts of helium per million parts of mixture (parts per million by volume). The concentration ratio of helium in air shall be taken arbitrarily as 1/200 000 or 5 parts per million, and this figure shall be taken into account when preparing mixtures containing more helium. [*Note:* The latest data indicate 5.24 parts per million of helium in air by volume.[1]]

4.6 Leak Detector.

4.6.1 The leak detector shall have been connected to a power source conforming in voltage, frequency, and regulation to the manufacturer's specifications.

4.6.2 The leak detector shall have been "warmed up" as specified by the manufacturer, prior to all test procedures.

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[1] E. Glueckauf, "Compendium of Meteorology," T. F. Malone, ed., (American Meteorological Society, Boston, 1951), pp. 3–10.

4.6.3 The leak detector under test shall have been adjusted for detecting helium in the manner specified by the manufacturer.

4.7 Chart Recorder. This shall be an instrument of at least 1 h recording time suitable for recording the output of the leak detector under test and adjustable so that full scale on the recorder equals full scale of the leak-detector output meter when the leak detector is set at its most sensitive detection setting. The time constant of the recorder shall not be greater than that of the leak-detector output meter. In the absence of a suitable recorder, visual observations of the output meter may be used.

4.8 Test Setups. These are illustrated diagrammatically in Figs. 1.0 and 1.1.

5. TEST PROCEDURE—MINIMUM DETECTABLE SIGNAL[2]

5.1 Drift Determination

5.1.1 The output of the leak detector is connected to the recorder, the leak detector being at its maximum sensitivity setting and the inlet valve being closed.

5.1.2 The leak detector is adjusted so that the recorder reads less than 10% of full scale, the electron-producing filament being on.

5.1.3 The output is recorded for 60 min or until the output indication has reached full scale.

5.1.4 Generally, the recorded-output curve will show spikes. If spikes do appear, the curve is to be faired in standard engineering fashion.

5.1.5 From the faired curve, the output is determined initially and at the end of each minute, in scale divisions.

5.1.6 The change in output is calculated for each 1-min period and the largest of these changes is determined. This largest change shall be called the *drift* (scale divisions). If the change of output per minute is always less than $\frac{1}{2}$ of 1% of full scale, the total change in the 60-min observation period is determined. The total change divided by 60 shall be called the *drift*.

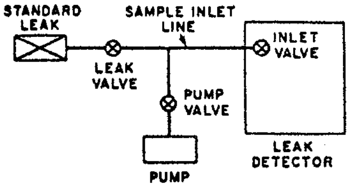

FIGURE 1.0.

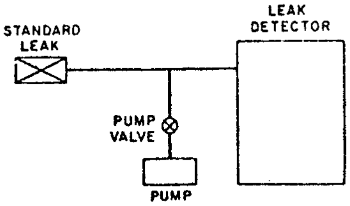

FIGURE 1.1.

5.2 Noise Determination. The recorded output curve is examined to determine the two spikes, one on each side of the faired curve, that extend furthest from the curve. The departures are measured (in scale divisions) and the two figures are added. The sum shall be called the *noise*. If the output is such that spikes appear only on one side, the noise shall be taken as *twice* the largest departure from the curve (scale divisions).

5.3 Minimum Detectable Signal. The minimum detectable signal shall be taken to be equal to the sum of the drift and the noise (scale divisions). If the sum is less than the scale divisions corresponding to 2% of full scale, then the scale divisions corresponding to 2% of full scale shall be called the minimum detectable signal.

6. TEST PROCEDURE—MINIMUM DETECTABLE LEAK

6.1 General. In the procedures below, a calibrated leak is connected to the leak detector under test. Preferably, the connections should contain no rubber or other polymeric surfaces between the leak and the leak valve (see Fig. 1.0); if such is the case, the procedure under Sec. 6.2 is followed. If rubber or polymeric surfaces are present, the procedure under Sec. 6.3 is followed.

6.2 Sensitivity Determination I. The leak detector is prepared as in Sec. 4.8 and connected to the recorder.

6.2.1 The small calibrated leak (see Sec. 4.3) is connected to the leak detector in a manner specified by the manufacturer. Refer to Fig. 1.0.

Specified connections shall include a valve (preferably all-metal) that does not act as a source of helium. located between the leak and the leak detector. This valve will be referred to as the *leak valve.*

Specified connections shall include a pumping line to permit evacuation of said connections. The pumping line shall include a valve hereafter referred to as the *pump valve,* which shall be located adjacent to the pump.

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[2] As used here, minimum detectable signal refers to the performance of a particular leak detector; it is determined by spurious outputs of the device that set a lower limit to the helium flow rate which rate will produce an unambiguous output signal.

Specified connections shall contain a minimum of rubber or other polymeric surface(s). Preferably, such surface(s) shall consist only of the exposed partial surface(s) of O ring(s).

6.2.2 The output is zeroed, with the filament on.

6.2.3 The leak valve is opened.

6.2.4 The pump valve is opened.

6.2.5 Helium at 760 Torr pressure is applied to the leak. If the leak has its own supply of helium, this step is omitted.

6.2.6 The atmospheric air present in the connections between the leak and the leak detector is evacuated to protect the leak detector.

6.2.7 The pump valve is closed.

6.2.8 The inlet valve in the leak detector sample line is opened promptly after Sec. 6.2.7. [*Note:* The filament of the mass spectrometer tube may be turned off before Sec. 6.2.7 above.] The pressure in the leak detector is allowed to reach a steady value, showing no observable change in 1 min.

6.2.9 Turn on filament of mass-spectrometer tube if it is not on.

6.2.10 At this point, it may be necessary to change the sensitivity setting. When the output signal has reached a steady value, showing no observable change in 1 min, the reading is noted (scale divisions).

6.2.11 The stop watch is started and simultaneously the leak valve is closed *as rapidly as feasible.*

6.2.12 The output is observed continuously, and the stop watch is stopped when the reading has decreased to 37% of the reading obtained in Sec. 6.2.10 above. The reading of the stop watch is noted (T sec).
T is a measure of the time required to remove the output signal initially caused by the helium input; this time figure is referred to as the *cleanup time.* It is assumed, for the purpose of this standard, that the leak detector is so constituted that the cleanup time and response time are equal, the *response time* being a measure of the speed of response of the leak detector to an incoming helium sample. Hereafter, T will be called the response time.

6.2.13 When the output signal has reached a steady reading, showing no observable change in 1 min, the reading is noted.

6.2.14 The signal due to the calibrated leak shall be taken as the difference between the reading noted in Sec. 6.2.10 above and that noted in Sec. 6.2.13 above. If the leak detector has been set at reduced sensitivity, the difference shall be converted into equivalent scale divisions at full-sensitivity setting. The minimum detectable leak shall be calculated by the formula below and shall *always* be stated together with the response time, T.

$$\text{MINIMUM DETECTABLE LEAK, WITH RESPONSE TIME } T = \text{CALIBRATED LEAK RATING} \times \frac{\text{MINIMUM DETECTABLE SIGNAL}}{\text{SIGNAL DUE TO CALIBRATED LEAK}}$$

6.3 Sensitivity Determination II.

6.3.1 The leak detector is prepared as in Sec. 4.8 and connected to the recorder.

(a) A stainless-steel plug is connected to the leak detector, in place of the calibrated leak shown in Fig. 1.0. Refer to Sec. 6.2.1.
(b) The output is zeroed, with the filament on.
(c) The leak valve is opened.
(d) The pump valve is opened.
(e) The atmospheric air present in the connections between the plug and the leak detector is evacuated to protect the leak detector.
(f) The pump valve is closed.
(g) The inlet valve is opened promptly after Sec. 6.3.1f above. [*Note:* The filament of the mass-spectrometer tube may be turned off before Sec. 6.3.1f]. The pressure in the leak detector is allowed to reach a steady value, showing no observable change in 1 min.
(h) Turn on filament of mass spectrometer tube if it is not on.
(i) When the output has reached a steady value, but in any case not longer than 3 min after Sec. 6.3.1g above, the output reading is noted. If the leak detector has been set at reduced sensitivity, the reading shall be converted to the equivalent scale divisions for full-sensitivity setting.
(j) Close the leak valve.
(k) When the output has reached a steady value, showing no observable change in 1 min, the output reading is noted. As in Sec. 6.3.1i, convert the reading if necessary.
(l) The difference between the readings in Secs. 6.3.1i and 6.3.1k, above, is calculated and will be considered a correction figure in Sec. 6.3.2m below.
(m) Close the inlet valve.

6.3.2 The plug *only* is removed from the inlet line. All connections are to remain in place.

(a) The small calibrated leak is put in place of the plug removed in Sec. 6.3.2 above, the leak being inserted the same distance into the connections as the plug had been.
(b) The output is zeroed, with the filament on.
(c) The leak valve is opened.

(d) The pump valve is opened.

(e) The atmospheric air present in the connections between the plug and the leak detector is evacuated to protect the leak detector.

(f) The pump valve is closed.

(g) The inlet valve is opened promptly after Sec. 6.3.2f [*Note:* The filament of the mass-spectrometer tube may be turned off before Sec. 6.3.2f above.] The pressure in the leak detector is allowed to reach a steady value, showing no observable change in 1 min.

(h) Turn on filament of mass-spectrometer tube if it is not on.

(i) When the output has reached a steady value, but in any case not longer than 1 min after step 6.3.2g above, the output reading is noted. If the leak detector has been set at reduced sensitivity, the reading shall be converted to the equivalent scale divisions for full-sensitivity setting.

(j) Close the leak valve.

(k) When the output has reached a steady value, showing no observable change in 1 min, the output reading is noted. As in Sec. 6.3.2i, convert the reading if necessary.

(l) The *uncorrected* signal due to calibrated leak shall be taken as the difference between the reading noted in Sec. 6.3.2i above and that noted in Sec. 6.3.2k above. If the leak detector has been set at reduced sensitivity, the difference shall be converted into equivalent scale divisions at full-sensitivity setting.

(m) The signal due to the calibrated leak shall be taken as the difference between the uncorrected signal, Sec. 6.3.2l above, and the correction in Sec. 6.3.1l above. The minimum detectable leak shall be calculated by the formula below and shall always be stated together with the response time, *T*.

$$\text{MINIMUM DETECTABLE LEAK, WITH RESPONSE TIME } T = \text{CALIBRATED LEAK RATING} \times \frac{\text{SIGNAL DUE TO CALIBRATED LEAK}}{\text{MINIMUM DETECTABLE SIGNAL}}$$

7. TEST PROCEDURE—MINIMUM DETECTABLE CONCENTRATION RATIO

7.1 General. A significant determination of minimum-detectable concentration ratio requires means within the leak detector under test for scanning the helium peak. This refers to the ability to tune or adjust the detector for detecting helium (see Sec. 2.) and thereby to attain a maximum or peak output for a given helium input, and the ability to detune either side of the peak adjustment until a minimum signal is produced. The process of adjusting a leak detector from one side of the correct adjustment for helium, to the correct adjustment, and then past the correct adjustment, is referred to as *scanning the (helium) peak.* The leak-detector adjustment referred to above is almost always an adjustment of the accelerating voltage, and it will be assumed that this is the case. When leak-detector output (scale divisions) is plotted against accelerating voltage, a curve is obtained whose general features are illustrated by the solid line in Fig. 2.0. (Note that voltage may be plotted in the reverse direction and this will give a reverse slope to the curve.) The rise in the curve to a peak at B is due to the presence of helium. The faired curve indicated by a broken line is due to a varying *background* signal contributed by other ions than helium. With helium present, and in the absence of background, the curve obtained would be symmetrical, tailing to zero on either side of the peak voltage. The curve shown in Fig. 2.0 is very nearly a direct superposition of the background curve and the symmetrical pure-helium curve.

It will be noted that, as one varies the voltage from the left side of the graph to the right, the output first decreases, then increases, and finally decreases again. This reversal in direction, indicating the presence of helium, is very easily detected when the scan is being observed visually on a meter. As the helium input signal becomes smaller, the reversal becomes smaller. At some point, a curve such as is shown by the solid line in Fig. 2.1 is obtained. At this point, the output never reverses; it "stands still" for a very short interval. Such a condition will just barely be detected by the usual visual observations. Accordingly, in the absence of noise and drift, the concentration ratio of helium which produces this condition determines the minimum-detectable concentration ratio.

Under practical conditions, it is not possible to make a rigidly correct determination of the minimum detectable concentration ratio as defined above. In the following, somewhat arbitrary determinations are used for calculating a sensitivity figure. The sensitivity so obtained is one that is reasonable in light of practical experience.

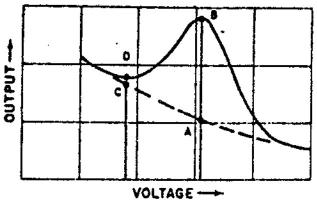

FIGURE 2.0.

Foreword

(This foreword is not a part of the proposed standard.)

This proposed standard was prepared by Evan F. Wilson of the Allis-Chalmers Manufacturing Company in his capacity as a member and, presently, Chairman of Subcommittee ANS-7, Reactor Components, of the American Nuclear Society Standards Committee. The work was initiated early in 1959, and the standard has undergone some ten or more reviews and revisions. Corrections and additions were incorporated into four formal revisions of which this is the latest. Representatives of 16 companies involved in nuclear research and development and other companies involved in the fabrication and construction of containment vessels participated in the reviews of this standard. The following are presently members of Subcommittee ANS-7:

E. F. Wilson, Chairman, Allis-Chalmers Manufacturing Company
S. F. Bacharach, Aerojet-General, Azusa
E. S. Brown, Phillips Petroleum Company, NRTS
S. Davis, United Nuclear Corporation
K. H. Dufrane, The Martin Company
A. W. Flynn, Ebasco Services, Inc.
L. W. Fromm, Argonne National Laboratory
W. R. Gall, Oak Ridge National Laboratory
R. Hobson, Westinghouse Atomic Power Division
K. C. Hoffman, Brookhaven National Laboratory
Arne B. Holt, U. S. Atomic Energy Commission
H. Hopkins, General Atomic
R. L. Koontz, Atomics International
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W. R. Smith, General Electric Company, APFD
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N. O. Strand, General Electric Company, Hanford
T. H. Thomas, Lockheed Nuclear Products

Following review and discussion in the June 15, 1964 meeting of the ANS Standards Committee, a vote was taken that was in favor of publication of this draft for comment by ANS members. The vote was not unanimous; the dissenting members were of the opinion that Section 8 should be a separate standard.

This proposed standard is issued for review and comment by ANS members and other interested persons. Comments will be accepted until February 1st and should be addressed to:

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ANS 7.60

Proposed Standard

for

Leakage Rate Testing of Containment Structures for Nuclear Reactors

Approved for Publication for Comments

June 15, 1964

ANS Standards Committee

American Nuclear Society

It is the policy and practice of the Standards Committee of the American Nuclear Society through its subcommittees to formulate and promulgate proposed standards for the nuclear industry. This standard was prepared on the consensus principle and is based on the experience and knowledge available at the time. This standard is intended as a guide to aid the manufacturer, the consumer, and the general public. The existence of a standard does not in any respect preclude any party from manufacturing, selling, or using products, processes, or procedures not conforming to the standard. This standard is subject to periodic review and reaffirmation or revision. The existence of this standard does not relieve its user from the requirement that he exercise good judgment in its application, and that he provide himself with technical competence commensurate to his activities, nor does compliance with ANS Standards assure acceptability to federal, state, or local authorities.

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Proposed Standard
for
Leakage Rate Testing of Containment Structures for Nuclear Reactors

1. Purpose and Scope

1.1 Purpose. The purpose of this standard is to specify uniform methods for determining the ability of a reactor container to retain, within the limits of permissible leakage rates, any gases, vapors, liquid, or other fluid materials which would be of a hazardous nature if not contained and which might be present in the containment structure as a result of an energy release, rupture, or leak in the nuclear reactor components or accessories. The need for restriction of leakage from the containment structure is based on the maintenance of public health and safety, the protection of operating and maintenance personnel, and the preservation of property.

1.2 Scope. The provisions of this standard specify the preferred practices and test requirements for the quantitative determination of gas leakage rates of containment structures for the housing of operating nuclear reactors. The provisions apply to containment structures for nuclear power, test, research, and training reactors, wherever a gas-tight containment structure is specified as a condition for operation.

2. Conjunctive Standards

2.1 Conditions of Applicability. This standard shall be applied in conjunction with other standards and codes as they may apply and are also specified. Acceptance of a containment structure with respect to the requirements of this standard shall be contingent upon compliance with the specified codes for design, fabrication, construction, inspection, proof testing, and maintenance, as subsequently listed in 2.2.

2.2 Conjunctive Standards. Standards or codes which are conjunctive to the present standard are the following:

2.2.1 *ASME Boiler and Pressure Vessel Code*, Section 3, Nuclear Vessels.

2.2.2 *ASME Boiler and Pressure Vessel Code*, Section 2, Material Specifications.

2.2.3 *ASME Boiler and Pressure Vessel Code*, Case Interpretations, 1962 and later revisions.

2.2.4 ASA N 6.2: Proposed *Safety Standard for Design Fabrication and Maintenance of Steel Containment Structures for Stationary Atomic Power Reactors.*

2.2.5 ASA Standard A57.1: American Institute of Steel Construction, *Specifications for the Design, Fabrication and Erection of Structural Steel for Buildings.*

2.2.6 ASA Standard A58.1: *Building Code Requirements for Minimum Design Loads in Buildings and Other Structures.*

2.2.7 ASA Standard A89.1: *Building Code Requirements for Reinforced Concrete.* (ACI-318)

2.2.8 *National Fire Codes*, National Fire Protection Association.

2.2.9 American Petroleum Institute, *Recommended Rules for the Design and Construction of Large, Welded, Low-Pressure Storage Tanks.*

3. Definitions and Descriptions of Terms

To assure common understanding of the terms employed in this specification, the following definitions shall apply.

3.1 Containment Structure. A containment structure, within the meaning of this standard, shall be an erected vessel, building, or underground location that provides an outer housing for the reactor system, including the primary vessels, components, and accessories. The function of the containment structure shall be the emergency and secondary retention of fluids and objects in the event of their accidental release from the reactor vessels or system.

3.2 Leak. A leak, in the context of this standard, shall constitute an opening, however minute, which allows the passage of a fluid and which is detectable by the means and methods specified herein for leak detection or leakage measurement.

3.3 Leakage. Leakage shall be interpreted as the measureable quantity of fluid escaping from a leak. For the purposes of this standard, air shall be used as the reference fluid.

3.4 Leakage Rate. Leakage rate is that leakage experienced during a specified period of time. For the purposes of this standard, leakage rate shall be reported as the percentage by weight of the original content of air by weight pressurized to the leakage rate test pressure which could escape during a 24-hour period. The leakage rate shall be that experienced at the outside atmosphere and containment structure air conditions prevailing during the period of leakage rate testing.

3.5 Maximum Allowable Leakage Rate. The maximum allowable leakage rate governing the acceptability of the containment structure by those responsible for its reliability shall be that stipulated in the specification for the individual containment structure.

4. Preliminaries to Leakage Rate Testing.

4.1 Sequence of Tests. Leakage rate testing should be conducted after the inspection and testing of welded joints, penetrations, and mechanical closures; completion of repair measures for the minimizing of leakage; and completion of containment structure pressure tests for strength. Where the containment structure is to be subsequently covered with concrete or will otherwise be inaccessible to direct examination, particular care should be given to inspection of these areas prior to such coverage. Integral or local leak detection should preferably precede leakage rate tests.

4.2 Pressure Tests for Strength. Hydrostatic or pneumatic pressure tests to determine whether the containment structure complies with specified strength and design requirements shall precede leakage rate testing. Also, the results of pressure tests shall meet the stipulated requirements before leakage rate tests are initiated.

4.3 Integral Pneumatic Leak Detection Tests. The detection of individual leak locations, preliminary to leakage rate testing, may be effected by local or integral pressurizing of the containment structure or both, and the use of soap solution to provide air-bubble indications on exterior surfaces. In the initial integral testing for location of leaks, the air pressure should be between 85% and 100% of the design pressure for the containment structure. Repairs or adjustments should be made as the leak detection results indicate. The use of integral pneumatic leak detection methods is particularly adapted to the examination of welded joints, gaskets, threaded connections, and mechanical closures, such as air locks, valves, vacuum breakers, and ducts, where local leak detection methods are not conveniently applicable.

4.4 Local Leak Detection Tests. Localized pressure tests may be advantageously employed in some circumstances where the part or area is especially susceptible to leakage or it is wished to employ higher pressures than in the integral-pressurizing detection test. Local leak detection methods may include the pneumatic soap-bubble test, vacuum testing, air-ammonia and halogen sniffer tests, or other tests developed for special examinations. Local tests are particularly suitable for inspection of equipment prior to installation in the container and for inspection of moderately small but complex assemblies where leaks are difficult to locate and where the leakage rate is especially slow. Descriptions of local leak detection methods are given in Appendix A. If the local leak detection test is carried out with internal pressurizing, a pressure of at least 5 psig shall be used if the design pressure of the containment structure is above 10 psig; and at least one half of the design pressure, if this pressure is 10 psig or less.

4.5 General Preparations for Test Pressurizing. Preparatory to test pressurizing for leakage rate determination, contents of the containment structure that are sensitive to damage by a pressure differential, such as some instruments, should be removed or otherwise protected. This also applies to fan and blower motors employed for air circulation where the load is a function of air density. The protection of the structure from damage, such as by underpressure, should be assured by checking the operative reliability of vacuum breakers. The vacuum-release devices should operate within 10% of their design pressures for internal or external loading.

4.6 Time Scheduling of the Leakage Rate Test. To assure favorable test conditions for leakage rate tests without large or abrupt changes in atmospheric temperatures ,or barometric pressures, the scheduling of the test should be planned insofar as feasible in accordance with advance weather predictions. Final weather checks to assure safety of the containment structure should be made just prior to and during the test. To minimize temperature fluctuations caused by solar radiation, wind effects, or appreciable changes in temperature, a relatively windless day during a period of relatively stable weather conditions is to be preferred. The anticipated weather conditions during the test should indicate little or moderate barometric pressure variations in order to improve the reproducibility of leakage rate results.

5. Leakage Rate Test Methods

5.1 Applicable Test Methods. Leakage rate test procedures applicable to this standard may be either the absolute method or the reference vessel method. The choice of either method shall be a matter of agreement between parties who are charged with responsible acceptance of the vessel and those in charge of the leakage rate test procedures.

5.2 Description of Methods. The absolute method of leakage rate testing shall constitute the determination and calculation of air losses by containment structure leakage over a stated period of time by the means of direct pressure and temperature observations during the period of test with temperature detectors properly located to provide an average air temperature. The reference vessel method shall constitute the determination and calculation of air losses by observations of the pressure differentials between the containment structure and a gas-tight reference system, with the reference vessels located so as to represent, with reasonable accuracy, the average temperature of the aggregate containment air.

5.3 Leakage Rate Pressures. Leakage rate determinations shall be conducted at the pressure at which the leakage rate was specified and after all other pressure testing required in the specification.

6. Test Equipment and Facilities

6.1 Pressurizing Facilities. Pressurizing facilities for containment structure leakage rate tests should be of sufficient capacity to bring the structure pressure to the test level within a sufficient period of time for scheduling with reference to favorable weather conditions. Valves and repressurizing facilities should be available for adjusting to subsequent atmospheric changes, as appropriate to specific test requirements.

6.2 Temperature Measurements. All thermometric equipment shall be compared over a normal range of atmospheric variations with a reference thermometer of established calibration. Corrections based on the reference thermometer shall be available before the leakage rate test is started. Thermometers, thermocouples, and thermographs employed in the leakage rate tests shall be reproducibly readable to 0.2°F, or equivalent, or to the extent specified as the tolerable error for the maximum allowable leakage rate of the structure subject to test.

6.3 Pressure Measurements. Mercurial or aneroid barometers for the observation of containment structure and outside atmospheres shall be reproducibly readable to 0.1 mm or less (0.004 in.) or to the extent specified as the tolerable error for the maximum allowable leakage rate. Barographs for the recording of the outside atmospheric changes need be only of such accuracy as will indicate gross barometric changes pertinent to the scheduling of tests. All barometric equipment shall be compared with a single precision mercurial barometer equipped with vernier and shall be correctable for temperature and readable to 0.1 mm. Manometers for the reading of pressure differentials shall be of precision bore and plainly readable to 1 mm (0.04 in.) or less of water by marked graduations.

6.4 Atmospheric Humidity. Hygrometers or psychrometers shall be available to determine relative humidities during the period of test within and outside the containment structure when required.

7. Test Procedures

7.1 The Absolute Method. The absolute method of leakage rate determination depends on the measurement of the temperature and pressure of a constant volume of containment structure air with suitable correction for changes in temperature and humidity control under a nearly constant pressure difference with respect to the atmosphere outside the structure. It is assumed that the temperature variations during the test will be insufficient to effect significant changes in the internal volume of the structure or the partial pressure of water vapor in the contained air.

7.2 The Reference Vessel Method. The reference vessel method of leakage rate determination depends on the changes in pressure of a constant volume of contained air compared with that of a hermetically closed reference vessel which may be at the same pressure as the contained air at the start of the test or may have a small differential. The reference vessels shall be so placed and of such a geometry that they will assume the temperatures of the contained air within a reasonable time lag. The reference vessels shall be subject to leakage rate determination in accordance with the absolute method prior to their use for containment structure testing according to the applicable procedures of this standard or may be checked by the halogen sniffer test or by retention of vacuum.

7.3 Leak Minimization. Before pressurizing for leakage rate testing, all discernible leaks in the container shall be revealed by local and integral leak detection tests and shall be repaired or corrected. All openable closures and vacuum breakers shall be checked for leak tightness.

7.4 Pressurizing. Pressurizing for the leakage rate test shall be carried out under atmospheric conditions which provide relatively low air humidity in order to avoid moisture condensation within the containment structure. Any moisture that condenses out of the pressurized air and collects at the bottom of the structure shall be drained off prior to the start of the test to prevent re-evaporation. Reference vessels should be similarly drained. To provide low humidity and to improve pumping efficiency, cool night air is usually preferred for pressurization. The structure shall be pressurized to as near the design pressure as is possible under prevailing conditions or to pressures stipulated as a condition for test acceptance.

7.5 Temperature Measurements. Area surveys within the structure shall be made in advance of leakage rate testing to establish any tendencies to regional variations in temperature. Additionally, thermometers and thermocouples shall be located at different parts of the structure wherever local variations may be expected in the course of the test. Fans or other means for air circulation may be used to equalize temperatures in any region where representative temperature measurements are taken and appreciable temperature variations exist.

The temperature pattern revealed by the survey shall be employed in connection with the mean representative temperature determination for the absolute method of leakage rate testing. Location of reference vessels shall be made with consideration of the temperature pattern in order to reflect representative temperatures. Where testing experience with vessels of various configurations has established appropriate locations for reference vessels, temperature surveys may be eliminated for those vessels having similar proportions.

7.6 Personnel Access to Pressurized Containment Structures. Exposure of personnel to pressurized air and return to normal atmospheric pressures during the course of containment structure leakage rate testing shall be governed by approved decompression procedures involving a controlled depressurizing rate and waiting periods at intermediate pressures. For exposures of no longer than 200 min at pressures not greater than 14.3 psig, no intermediate holding periods or decompression stops are required provided that the time rate of pressure reduction in the air lock to atmospheric level is not less than 30 sec. For exposures to pressurization in excess of 14.3 psig, and for exposure periods including repetitive exposure within 12 hr, the practices should conform to those stipulated in Section 1.5, Diving Tables of the U. S. Navy Diving Manual, NAVSHIP 250-538, January 1959.

7.7 Period of Test. The leakage rate test period shall extend to not less than 24 hr of retained internal pressure. Completion of the test should be scheduled to coincide with atmospheric temperatures and pressures close to those at the start of the test, as far as is possible. Check tests or repetition of tests shall be a matter of agreement between those responsible for the acceptance of the containment structure and those in charge of the leakage rate testing.

7.8 Humidity. The relative humidity of the containment structure shall be monitored during the course of the leakage rate test to assure that the dew point is not reached and that there is no condensation of moisture in any part of the structure. Concrete structures within the containment structure should be properly cured prior to testing to minimize high humidity from moisture release; however, where appreciable evaporation may occur from exposed surfaces of incompletely cured concrete, such surfaces should be covered with plastic sheeting, or other suitable precautions should be taken. Open pools of water may be similarly covered. To minimize the effect of variation in the partial pressure of water vapor, it is desirable to maintain the containment structure air at a reasonably constant temperature level, particularly near the completion of the test. Air conditioning, prior to testing, may be employed to approach this condition. Any moisture condensation occurring during the course of the test will result in an apparent leakage rate in excess of actual.

7.9 Recording of Data. Pressure, temperature, and humidity observations shall be made within the containment structure and recorded during the course of the leakage rate test at hourly or more frequent intervals. Pressure and temperature measurements of the outside atmosphere shall also be made and recorded at corresponding intervals and times. The times of observations shall be denoted in hours and minutes. A dated log of events and pertinent observations shall also be maintained during the test, and the correctness of data shall be attested to by those responsible for the test and, where specified, by a competent witness. Records of the leakage rate tests shall be maintained in accordance with the terms of agreements with those responsible for the acceptance of the containment structure.

7.10 Computation of Leakage Rate, The Absolute Method. For the absolute method of leakage rate testing, the calculation of the per cent leakage of air from the containment structure in terms of the original amount contained and that which escaped during a 24-hr period shall be made in accordance with the following formula:

$$\text{Per cent leakage in 24 hr} = \frac{24}{H}\left(1 - \frac{T_1 P_2}{T_2 P_1}\right) 100,$$

where

T_1 = mean absolute temperature of the containment structure air at the start of the test,

T_2 = mean absolute temperature of the containment structure air at the completion of the test,

P_1 = absolute pressure of the containment structure air at the start of the test,

P_2 = absolute pressure of the containment structure air at the completion of the test,

H = time period of the leakage rate test, in hours.

The derivation of this formula is given in Appendix B.

7.11 Computation of Leakage Rate, The Reference Vessel Method. For the reference vessel method of leakage rate testing, the calculation of the per cent leakage of air from the containment structure in terms of the original amount contained and that which escapes during a 24-hr period, shall be made in accordance with the following formula:

Per cent leakage in 24 hr =

$$= \frac{24}{H}\left[\frac{T_1 (P_2' - P_2)}{T_2 P_1} - \frac{(P_1' - P_1)}{P_1}\right] 100,$$

where P_1' and P_2' are, respectively, the absolute pressures of the reference vessel at the start and completion of the test.

8. Reinspection and Recheck of the Containment Structure

8.1 Reinspection. Annual reinspection is recommended to determine whether visual evidence of deterioration of the structure has occurred and whether this might affect its tightness with respect to the leakage rate. Such inspection should include evidence of unequal settlement of the foundations, significant corrosion, significant weathering of sealing compounds or other nonmetallic materials, cracking at weld areas or other regions of stress concentration, and damage resulting from operations or accidents. Penetrations and closures should be examined and their functional reliability determined.

8.2 Local Leak Detection Retests. Localized pressure tests, such as those described in 4.4, should be made whenever annual inspection tests or other circumstances show deterioration or otherwise indicate the desirability of such retests. Localized pressure tests shall be made whenever repairs or new construction are involved. A record of local leak tests results should be maintained for reference.

Appendix A. Local Leak Testing Procedures.

(This material is informative only and is not a part of the Standard for Leakage Rate Testing of Containment Structures for Nuclear Reactors.)

A1. Applicability of Local Leak Tests. Local leak tests may be selected for the qualitative inspection of specific materials or components where methods other than air pressurizing are not objectionable and provide a more searching and convenient method. Such tests are particularly applicable to parts of or accessories to the containment structure.

A2. Water Submersion Test. The water submersion test consists of covering an area which may contain a leak with clean water on the low pressure side of a differential pressure. The water should be such as to provide full submergence with convenient observation of bubble formation. Repeated bubble formation occurring within 5 min after a previous bubble has been wiped away will indicate a leak.

A3. Vacuum Test. The vacuum test employs a vacuum box which can be placed over an area to be tested and evacuated to at least a 5-psi pressure differential with the atmospheric pressure where the edge seals provide a tight seating closure. Air leakage through the area tested may be revealed by changes in a manometer level after the absence of seating leakage is determined by soap-suds indicators. If a soap solution is applied to the test area before covering with the vacuum box, leaks may be revealed by bubble formation visible through a glass-covered opening in the box within a 5-min examination period.

A suitable soap solution for air leak indication is one consisting of equal parts of corn syrup, liquid detergent, and glycerin. The solution should not be prepared more than 24 hr preceding the test and bubble formation properties should be checked with a sample leak every half hour during the test.

A4. Air-Ammonia Test. The air-ammonia test is an air-pressurizing method employing anhydrous ammonia as an indicator. Where leaks are present, the leakage permeation of ammonia is revealed by a white chemical fog on probing the atmosphere with a swab wetted with 0.1 N hydrochloric acid. Sulphur dioxide, such as from a sulphur candle, can also be used as the revealing reactant. Other methods employing ammonia use 1.0% phenolphthalein in a solution of equal amounts of water and ethyl alcohol. A cloth dampened with the phenolphthalein solution and placed over the test area shows the location of leaks by a pink discoloration. The ammonia indicator can be introduced as an anhydrous gas or by placing a cloth saturated with ammonia solution within the pressurized space.

A5. Halogen Sniffer Test. The halogen sniffer test employs a halogen compound leak indicator, such as freon gas, in the pressurized air. About 0.3 ounces per cubic foot of air is commonly used. Leakage is revealed by traversing the test area with a detector that senses the effects of the halogen compound on ion emission from a heated metal surface. Locating the leak is best accomplished by holding the sniffer at about 1/2 in. from the surface to be examined and traversing this at a rate of 1/2 in./sec. A leak is indicated by a milliammeter pointer movement or audible signal. Detection is also made by flame coloration from halogen indicator additions to the contained air. It should be realized that halogen detectors are sensitive to cigarette smoke or vapor from dry cleaning fluids in recently cleaned clothing. Also, if halogen compounds are used with stress-corrosion sensitive materials, chloride attack is possible unless thorough-cleaning follows this test.

Appendix B. Derivation of Formulas for Containment Structure Leakage Rates.

(This material is informative only and is not a part of the Standard for Leakage Rate Testing of Containment Structures for Nuclear Reactors.)

B1. Definition of Symbols.

P_1 = absolute pressure of containment structure air at the start of the leakage rate test,

P_2 = absolute pressure of containment structure air at the end of the leakage rate test,

T_1 = mean absolute temperature at the start of the leakage rate test, °F + 459.7° or °C + 273°,

T_2 = mean absolute temperature at completion of test,

w_1 = original weight of contained air at the start of the test,

w_2 = final weight of contained air at the end of the test,

V = internal volume of containment structure, assumed to remain constant,

R = gas constant for a perfect gas, applicable to air for the test conditions employed,

H = time length of test, in hours,

T', P', V' = reference vessel conditions.

B2. Determination of Leakage Rate, The Absolute Method

$$P_1V = w_1RT_1 \quad \text{and} \quad P_2V = w_2RT_2$$

$$\frac{w_1T_1}{P_1} = \frac{V}{R} \quad \text{and} \quad \frac{w_2T_2}{P_2} = \frac{V}{R} .$$

Therefore

$$\frac{w_1T_1}{P_1} = \frac{w_2T_2}{P_2} .$$

Whereby

$$w_1 = \frac{w_2T_2P_1}{T_1P_2} \quad \text{and} \quad w_2 = \frac{w_1T_1P_2}{T_2P_1} .$$

$$\text{Leakage} = \frac{w_1 - w_2}{w_1} = \frac{w_2\left(\frac{T_2P_1}{T_1P_2} - 1\right)}{w_2\left(\frac{T_2P_1}{T_1P_2}\right)} = 1 - \frac{T_1P_2}{T_2P_1} .$$

$$\text{Per cent leakage rate per 24 hr} = \frac{24}{H}\left(1 - \frac{T_1P_2}{T_2P_1}\right)100.$$

B3. Determination of Leakage Rate, The Reference Vessel Method

In the reference vessel

$$P_1'V' = w'RT_1' \quad \text{and} \quad P_2'V' = w'RT_2'$$

$$P_1' = \frac{w'RT_1'}{V'} \quad \text{and} \quad P_2' = \frac{w'RT_2'}{V'}$$

$$w' = \frac{P_1'V'}{RT_1'}$$

where the prime mark denotes the reference vessel conditions.

In the containment structure

$$P_1V = w_1RT_1 \quad \text{and} \quad P_2V = w_2RT_2$$

$$P_1 = \frac{w_1RT_1}{V} \quad \text{and} \quad P_2 = \frac{w_2RT_2}{V}$$

$$w_1 = \frac{P_1V}{RT_1} .$$

In the system of reference vessel and containment structure, the pressure difference between the two structures is expressed by

$$\Delta P_1 = P_1' - P_1 = R\left(\frac{w'T_1'}{V'} - \frac{w_1T_1}{V}\right)$$

$$\Delta P_2 = P_2' - P_2 = R\left(\frac{w'T_2'}{V'} - \frac{w_2T_2}{V}\right) .$$

By transposition

$$w_1 = \frac{V}{T_1}\left(\frac{w'T_1'}{V'} - \frac{\Delta P_1}{R}\right)$$

$$w_2 = \frac{V}{T_2}\left(\frac{w'T_2'}{V'} - \frac{\Delta P_2}{R}\right)$$

$$w_1 - w_2 = \frac{Vw'}{V'}\left(\frac{T_1'}{T_1} - \frac{T_2'}{T_2}\right) + \frac{V}{R}\left(\frac{\Delta P_2}{T_2} - \frac{\Delta P_1}{T_1}\right) .$$

Substituting for w' the terms $(P_1'V'/RT_1')$ and dividing the expression for $(w_1 - w_2)$ by the equivalent of w_1 or (P_1V/RT_1) gives

$$\text{Leakage} = \frac{w_1 - w_2}{w_1} = \frac{T_1P_1'}{T_1'P_1}\left(\frac{T_1'}{T_1} - \frac{T_2'}{T_2}\right) + \frac{T_1}{P_1}\left(\frac{\Delta P_2}{T_2} - \frac{\Delta P_1}{T_1}\right) .$$

The principle of the leakage rate test using the reference vessel method assumes temperature equalization between the reference vessel and the containment structure air, so that in the equation above

$$T_1 = T_1' \quad \text{and} \quad T_2 = T_2' .$$

This reduces the equation to a general expression for leakage:

$$\text{Leakage} = \frac{w_1 - w_2}{w_1} = \frac{T_1}{P_1}\left(\frac{\Delta P_2}{T_2} - \frac{\Delta P_1}{T_1}\right) =$$

$$= \frac{1}{P_1}\left(\frac{T_1\Delta P_2}{T_2} - \Delta P_1\right) .$$

Under the conditions in which the test is started with the pressure in the reference vessel equal to that in the containment structure, then $P_1 = P_1$, and $\Delta P_1 = 0$, whereby

$$\text{Leakage} = \frac{T_1\Delta P_2}{T_2P_1} .$$

Under the conditions in which the test is ended with the containment structure air temperature the same as that at the start, $T_1 = T_2$, and

$$\text{Leakage} = \frac{1}{P_1}(\Delta P_2 - \Delta P_1) .$$

The leakage rate is expressed in percentage values for a 24-hr period. The general expression for leakage rate becomes

Per cent leakage rate in 24 hr =

$$\frac{24}{H}\left[\frac{T_1(P_2' - P_2)}{T_2P_1} - \frac{(P_1' - P_1)}{P_1}\right]100 .$$