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QUANTITATIVE LEAK TEST DESIGN GUIDE

by

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QUANTITATIVE LEAK TEST DESIGN GUIDE

August 1967

Prepared for

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PASADENA, CALIFORNIA 91103

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JPL Technical Representative: Edgar Koch

FOREWORD

This final report, "Quantitative Leak Test Design Guide," was prepared for the Jet Propulsion Laboratory, California Institute of Technology, Pasadena, California. The work was performed by J. L. Manganaro of the Electrochemical Systems Branch of the Chemical Systems and Processes Laboratory of the General Electric Research and Development Center, Schenectady, New York and D. L. Hollinger of the Plastics Process Development Operation of the Manufacturing Engineering Service of the General Electric Company, Schenectady, New York. It was done between 8 December 1966 and 7 August 1967 under Contract No. 951763.

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Section 1

SUMMARY

The object of this report is to document the performance of equipment for estimating leak rates by a calibrated enclosure technique, and to report the results of experimental tests run with this equipment. An operating procedure is also outlined. The equipment was designed and fabricated for and delivered to the Jet Propulsion Laboratory under Contract No. 951763. The major items of hardware delivered under this contract were:

- Steady leak flow calibrating system
- Set of three leaks with filters to span the range from 10^{-4} to 10^{-2} cubic centimeters atmosphere per second
- Enclosure including a rigid LEXAN frame, a polyvinyl chloride bag, and a lifting harness
- Syringe for the injection testing method
- Calibrating pipet

A "Quantitative Test Design Guide," including a suggested operating procedure and a technical discussion of the equipment was also written.

In this report the results are given of an investigation of the following system characteristics: circulation and diffusion of helium, system linearity, system stability, the effect of leak and proper position, and system sensitivity. Three experimental procedures for leak testing with this equipment were investigated; comparison method, superposition method, and an injection method. The methods are compared and data are given for each.

The Injection Method proved to be the easiest and most rapid. Leaks ranging from 14.4 to 99.2 cubic centimeters (standard temperature and pressure) per hour were detected with an average accuracy of 10 percent in the delivered enclosure which had a volume of 231 cubic feet. Testing times for the runs ranged from 2 to 4 hours. A small office fan was found adequate for mixing in the enclosure. The detector system exhibited a linear response over the helium concentration range investigated. Stable operation was obtained over a period of several hours. No effect of changing the probe and leak positions was observed. At a manifold pressure of 15μ and a tube pressure of 4×10^{-5} torr, the leak detector sensitivity was $15\mu a/\mu$atm He.

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Section 2

INTRODUCTION

In the leak testing of large systems, speed and accuracy are frequently of major concern. For example, satellite and spacecraft pressure systems are now tested at Cape Kennedy by a differential pressure method which, although yielding highly accurate results, requires two or three days to perform. In such situations, the test system is in high demand and time is a critical factor. The same problem arises in the leak testing of a large quantity of items, or of large industrial components.

Under Contract No. 951763 a system was designed and fabricated to perform rapid, accurate leak testing. The object of this final report is to document the performance of the system and to report the results of experimental tests run with this equipment. An operating procedure is also outlined.

With this system the determination of leak rates is accomplished by a calibrated enclosure technique. The object to be tested is pressurized with helium and the helium leak rate determined. Once the helium leak rate is known, the leak rates of other gases, such as nitrogen, can be estimated. The major steps of this procedure are:

1. Pressuring the test object with pure helium or a helium-nitrogen mixture (preferably the former).
2. Enclosing the object in a bag or tent.
3. Sensing the helium content in the enclosure and determining the rate of current increase in a mass spectrometer tube.
4. Calibrating the current increase in the mass spectrometer in terms of a helium rate by either 1) comparing to known helium leaks or 2) injecting a known quantity of helium.

Three different methods for estimating leak rates with this sytem are outlined and the results of experimental tests run by these methods are reported. The methods are:

1. Comparison Method, in which the test leak is directly compared with a calibrated leak.
2. Superposition Method, in which the ratio of the rate of increase for the test leak and the combined calibrated and test leaks is an indication of the test leak rate.
3. Injection Method, in which the rate of helium increase in the enclosure is determined, and then the enclosure calibrated by injecting a known amount of helium.

The major items of hardware delivered under this contract were:

Steady leak flow calibrating system

Set of three leaks with filters to span the range from 10^{-4} to 10^{-2} cubic centimeters atmosphere per second

Enclosure including a rigid LEXAN frame, a polyvinyl chloride bag, and a lifting harness

Syringe for the Injection Testing Method

Calibrating pipet

A "Quantitative Leak Test Design Guide" was written. In addition to a description and theoretical discussion of the operating and testing procedures, the "Design Guide" has:

Design Criteria for the Enclosure and Calibrator

Discussion of Various Test Procedures with Actual Data

Discussion of Error Analysis

Discussion of Leak Rate as a Function of Gas Composition and Pressure, and Leak Structure

Section 3

SYMBOLS

m	Slope rate ratio used in Superposition Method
P_m	Pressure in manifold, microns
P_t	Pressure in mass spectrometer detector tube, torr
Q	Leak rate, cubic centimeters(standard temperature and pressure) per hour, or cubic centimeters(standard temperature and pressure) per second
Q_c	Calibrated leak rate, cubic centimeters(standard temperature and pressure) per hour
Q_u	Unknown leak rate, cubic centimeters(standard temperature and pressure) per hour

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Section 4

TEST EQUIPMENT

This section describes the equipment delivered under Contract No. 951763 and gives instructions for its use. The major items of hardware delivered were:

- Steady leak flow calibrating system
- Set of three leaks with filters to span the range from 10^{-4} to 10^{-2} cubic centimeters atmosphere per second
- Enclosure including a rigid LEXAN frame, a polyvinyl chloride bag, and a lifting harness
- Syringe for the Injection Testing Method
- Calibrating pipet

A "Quantitative Test Design Guide," including a suggested operating procedure and a technical discussion of the equipment was also written.

STEADY LEAK FLOW CALIBRATOR SYSTEM

The calibrator system is designed to supply a steady leak flow of trace gas over a given period of time through the use of a ballast tank. For a discussion of the sizing of the ballast tank the reader is referred to the "Design Guide." A schematic of the calibrator system is shown in Figure 1. The major components of the calibrator are:

- Hoke Stainless-steel Cylinder, 8HD1-G
- Wallace and Tiernan Differential Fine Pressure Gage, 0 to 300 pounds per square inch FA-234160
- Coarse Pressure Gage
- Hoke Bellows Brass Seal Valves, No. 284
- Bud Cabinet, 14-3/4 by 22 by 19 Inches

The calibrator must also have a high pressure leak gas cylinder of helium to charge the calibrator ballast tank, and a vacuum pump. These two items are not supplied by General Electric. The vacuum pump is connected to the port labeled "vacuum" and the leak gas cylinder to the port labeled "leak gas". To the port labeled "leak" is attached the tubing for the calibrated leak.

Instructions for Start-up From Total Shutdown

The following steps are taken to start the calibrator system from total shutdown:

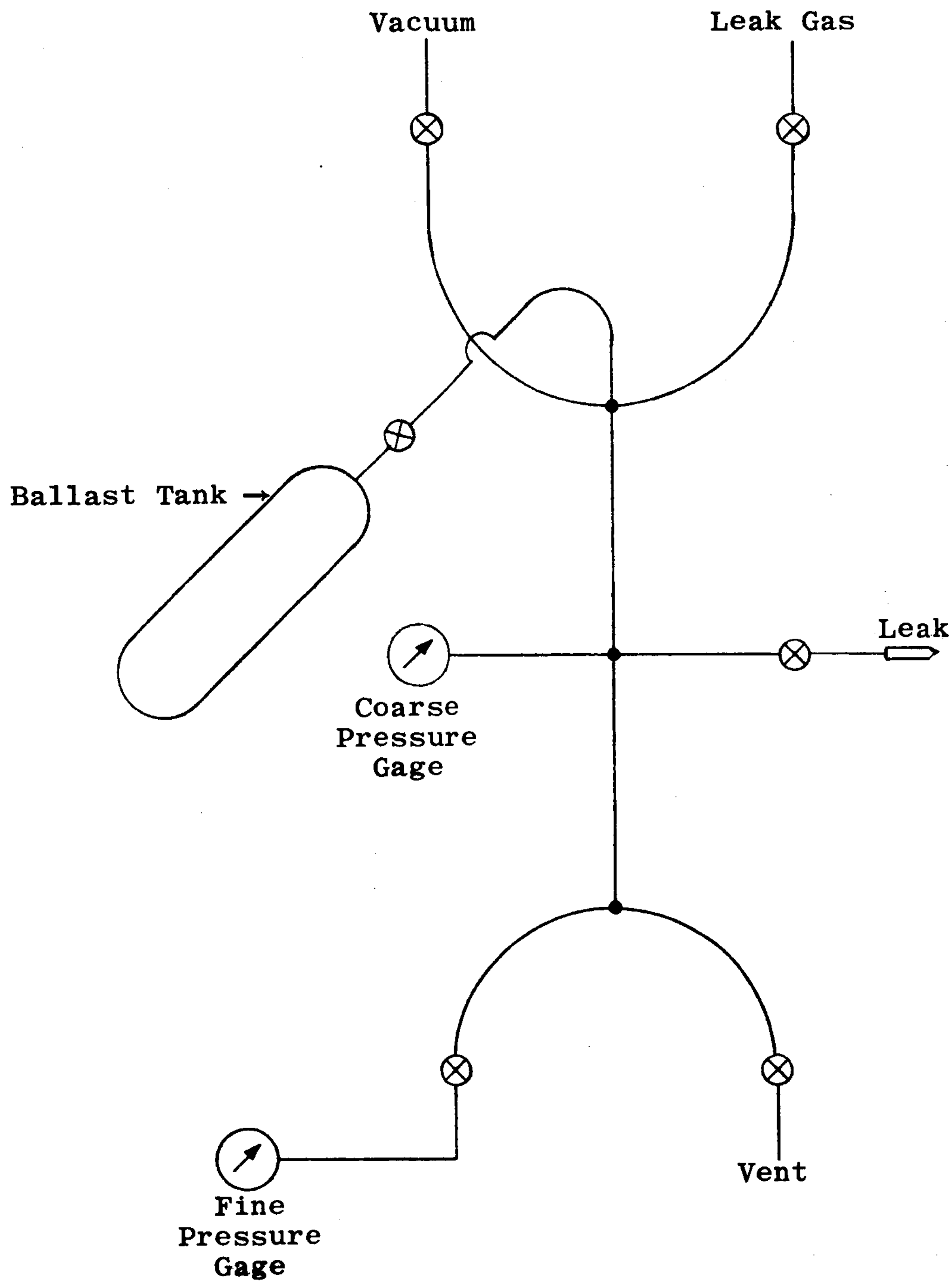

Figure 1. Schematic of Ballast Tank Calibrator.

1. The vacuum pump, leak gas cylinder, and leak are attached to the cylinder. The vacuum, ballast tank, leak gas, and fine gage valves are then opened. Opening of the fine gage valve permits evacuation of the Bourdon tube in the fine gage. Vacuum causes the fine pressure gage to read approximately -14.7 pounds per square inch. This is not, however, harmful. The coarse pressure gage may also be used in this step as a means of indicating when a sufficiently low pressure has been achieved in the system. When the calibrator was designed it was not believed that the fine gage could be subjected to vacuum, hence the use of the coarse, combination gage. The system should be evacuated for five minutes after the fine gage indicates -14.7 pounds per square inch.

   The leak gas cylinder is isolated from the calibrator for this step by attaching a valve and regulator to the leak gas cylinder. The regulator is set to deliver 250 pounds per square inch to the calibrator. Thus, during the evacuation, the air in the tube leading from the leak gas valve to the valve of the leak gas cylinder is also evacuated.

2. The vacuum and leak gas valves are closed, and the leak gas cylinder valve is opened. The leak gas valve of the calibrator is opened, admitting helium to a pressure of about 100 pounds per square inch, slowly, so that the precision gage is not surged. Next the valve leading to the leak, which is a copper tubing extension, is opened. The Swagelok fitting holding the appropriate calibrated leak is loosened, in order to purge the leak and its surroundings of any occluded air, and then tightened. In starting from total shutdown steps 1 and 2 may be repeated to completely remove all air.

3. The leak gas pressure, as read on the fine pressure gage, is adjusted to the level which will produce the desired flow rate by means of the vent and/or leak gas valves. About one minute should be allowed for the system to achieve pressure equilibrium. The pressure will tend to overshoot and gradually decay to a steady value. The system should not be pressurized above 200 pounds per square inch. With the leaks supplied this will be sufficient to span the leak range of interest. Pressures greater than 300 pounds per square inch will ruin the fine pressure gage if the protection valve is open.

4. Once the pressure has been adjusted and the valve to the leak opened, the calibrator is ready to deliver a given flow rate.

Instructions for Partial Shutdown

Less time is taken to start the calibrator system from a state of partial shutdown than from one of total shutdown. In a state of partial shutdown the ballast tank remains charged with leak gas. The only parts of the system which see air are the copper tubes running between the ports and valves for the vacuum, the leak gas, and the leak. Partial shutdown is readily achieved by:

1. Maintaining a positive leak gas pressure while closing all valves
2. Disconnecting the leads to the ports

It may be convenient to ship the calibrator in this state.

Instructions for Start-up from Partial Shutdown

The following steps are taken to start the calibrator system from partial shutdown:

1. The vacuum pump, leak gas cylinder, and leak are attached to calibrator.
2. With all valves originally closed, the vacuum and leak gas valves are opened. This evacuates the tubing up to the leak gas cylinder and ballast tank, whose valves remain closed.
3. The vacuum and leak gas valves are closed. The leak gas cylinder valve is opened.
4. The ballast tank and pressure gage valves are opened and the pressure is adjusted to desired level in the usual manner.

Instructions for Total Shutdown

All contained leak gas is released and the valves are closed.

ENCLOSURE

General Requirements

The requirements for an enclosure to confine and allow measurement of the gas escaping from leaks in a space vehicle are simple. A constant, but not necessarily known, free volume and a low, but not necessarily zero, permeability to helium are the principal process-defined needs. The volume is fixed by the dimensions of the various space vehicles to be tested, and the upper limit on bag permeability and porosity is a function of the free volume, anticipated leak rate, and testing time available. The values for these parameters which were supplied by Jet Propulsion Laboratory are: free volume of between 10 and 1000 cubic feet, for measurement of leak rates in the range 1/2 to 100 cubic centimeters per hour, with testing times less than 24 hours. Penetrations into the bag for connection to measuring instruments, calibrated leaks, and air circulating devices were anticipated.

The shape of the enclosure was not specified. For practical engineering reasons a cylindrical design is desirable. This shape is compatible with the shapes of the space vehicles to be tested. To make the developmental bag usable for testing at least one current, specific space vehicle, a cylindrically shaped bag, 66 inches in diameter and 117 inches high was decided upon. The enclosure is to be completely assembled in an area adjacent to the space vehicle test site and lowered over the vehicle by crane facilities. Hence, a reasonable degree of form stability is necessary.

Design Considerations

In addition to the general requirements mentioned above, these factors were also considered in arriving at a suitable design for the enclosure:

1. The enclosure should be easily adaptable to existing test facilities in a number of different locations. It should be essentially self-contained, or require almost no on-site parts acquisition, except such materials as ladders and standard hand tools.

2. Assembly and disassembly of the complete enclosure should be simple. It should require not more than a couple of hours to set up or take down.

3. All parts should be reducible to relatively small packaging size for shipping. The number of easily lost small parts should be kept to a minimum.

4. The enclosure should be durable enough to withstand repeated assembly, disassembly, repackaging, and shipping without developing porosity. Procedures should be available to repair damage.

5. Transparency of the enclosure was not considered a primary requirement.

Design and Fabrication Procedures

The volume within the bag, it was decided, would be controlled almost entirely by means of a rigid frame. A conforming, flexible plastic bag drawn over this frame would provide the impermeable gas barrier. The complete enclosure would be comprised of four major components:

Semirigid, plastic, cylindrical frame

Rigid reinforcing rings for the cylindrical frame ends

Flexible plastic bag

Lifting harness

Semirigid Plastic Cylindrical Frame

The enclosure frame is a cylinder of LEXAN plastic sheet, 66 inches in diameter and 117 inches high, with a wall thickness of 0.090 inch.

LEXAN was chosen as the frame material because it is tough enough to resist damage from repeated handling, because it has dimensional stability including lack of creep at service temperatures, and because it is self-extinguishing.

The cylinder is assembled from five smaller, flat sheets of LEXAN, four measuring 48 inches by 117 inches and a fifth measuring 31 inches by 117 inches. Since the largest size sheet available in small quantities was 48 by 96 inches, the 117-inch long panels were made up by bonding on extensions with a polyurethane adhesive, Adiprene L-100 by DuPont, cured with 11 parts by weight of methylenebis(orthochloroaniline). Each sheet, or panel, has a series of Quick-Lock fasteners attached permanently along the two long edges. These bind the panels together to form the complete cylinder and are operated by a one-quarter turn with a common screwdriver.

Rigid Reinforcing Rings

To maintain the cylinder in an accurately round shape, reinforcing rings of fiberglass laminate are bolted inside to its top and bottom. These rings are composed of six overlapping segments, bolted together, and have an essentially L-shaped cross section. Fiberglass is used primarily because the desired shape could be obtained with low tooling cost. The reinforcing rings could also be aluminum.

Polyvinyl Chloride Bag

A tailored plastic bag made of polyvinyl chloride film, 0.020 inch thick, is placed over the frame. The bag has reasonably low gas permeability, has good strength and toughness, can be readily obtained in heavy gages and large sheet widths, is self-extenguishing, lends itself well to fabrication by heat sealing, and has a useful degree of elasticity and flexibility.

A separate sheet of the same polyvinyl chloride material is supplied for use as a floor covering. It is to be placed beneath the test vehicle in locations where the existing floor does not lend itself to forming a seal with the bag. In the test work done at General Electric, the bag was taped directly to the asphalt tile floor, using black vinyl plastic tape.

Lifting Harness

A nylon rope lifting harness is supplied to facilitate the positioning of the enclosure over a space vehicle. The harness is placed over the enclosure and the three hooks of the harness are inserted into the holes in the LEXAN frame and the bottom reinforcing ring. The steel ring at the top of this harness may then be picked up by a crane and the complete enclosure lifted into position. The three hooks are released from the structure when the bottom seal is made, but the harness remains in position on the outside.

The only hardware which is not permanently attached to the major pieces of this equipment are some 5/16 - 18 x 3/4 inch long pan head machine screws and matching nuts. They are used to fasten the reinforcing ring segments together and to fasten the rings to the LEXAN panels.

Since the polyvinyl chloride bag fits rather tightly over the LEXAN frame, it is not feasible to align the same small holes made through these two hardwares for instrument penetrations in subsequent runs. It is recommended that, when the instrumentation is withdrawn at the end of a run, the holes in the polyvinyl chloride bag be sealed with either polyvinyl chloride pressure sensitive tape or small patches of the floor piece material applied with vinyl plastic cement, for example, VC-2 by Schwartz Chemical Company, Inc., Long Island City, New York. When the next run is to be made, new holes can easily be made in the polyvinyl chloride bag to coincide with the existing holes in the LEXAN frame.

Enclosure Ventilation

The contents of the enclosure may be exhausted by lifting the structure from the floor or plastic sheet and ventilating by means of fans or blowers. Ventilation may also be accomplished by raising the polyvinyl chloride bag material to a height of about four feet from the floor. This enables the smallest LEXAN panel to be opened at the bottom by releasing about four disconnect screws along a side. Leaving the panel partially open while the fans inside the enclosure are operating will ventilate the system in 15 to 30 minutes. Blowers may be used to speed ventilation.

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Section 5

SYSTEM CHARACTERISTICS

The following system characteristics were investigated with the equipment designed and fabricated under this contract:

- Circulation and Diffusion of Helium In Enclosure
- System Linearity
- System Stability
- Effect of Leak and Probe Position
- System Sensitivity

CIRCULATION AND DIFFUSION OF HELIUM IN ENCLOSURE

In order to gain a better understanding of the mixing effectiveness within the enclosure, a fixed quantity of helium (5 cubic centimeters) was injected into the bag for two cases:

1. Fan operating
2. Fan not operating

The results of this test are shown in Figure 2.

The sniffer probe was placed about 7 feet from the point of injection. The response of the leak detector was almost instantaneous with the fan on (Figure 2A). The overshoot may have been due to a "pocket" of helium being carried along the enclosure walls to the probe and then mixed, since both the probe and injection were located near the wall. Mixing appeared complete, when determined by a steady-state value, within two minutes. Therefore, the single household fan employed appeared to be adequate for the enclosure volume of 231 cubic feet.

Figure 2B shows the response obtained for injecting 5 cubic centimeters of helium with the fan off. Within 45 seconds a "pocket" of helium was detected. The profile of the response indicated that the helium in fact traveled as a "pocket" and was gradually dispersed by diffusion and convection. Steady-state or complete mixing was achieved for this situation within five minutes.

Rapid mixing has also been confirmed in a run where 120 cubic centimeters per hour were leaked into the enclosure (Figure 3). Since this is a large flow rate, it is the worst condition. The slope with the fan operating was 14.5 and without was 13.7, a difference of about six percent. The curve for the fan-off condition was more choppy, as is to be expected.

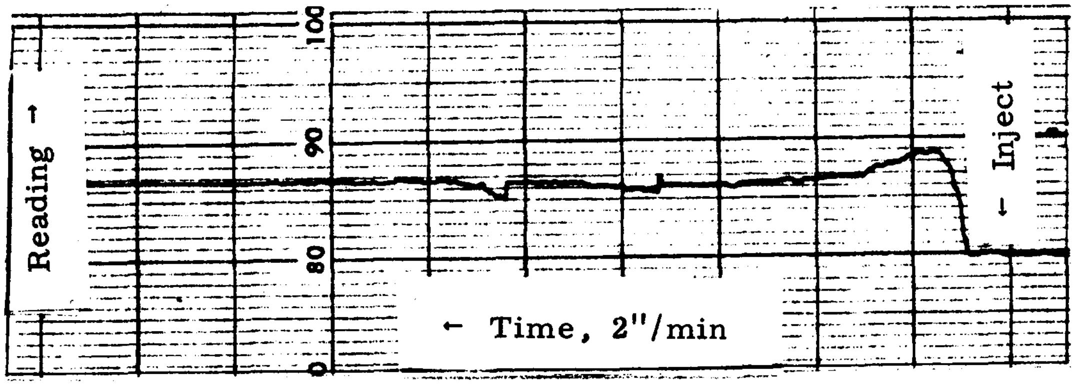

Figure 2A. Fan On (time increase to left)

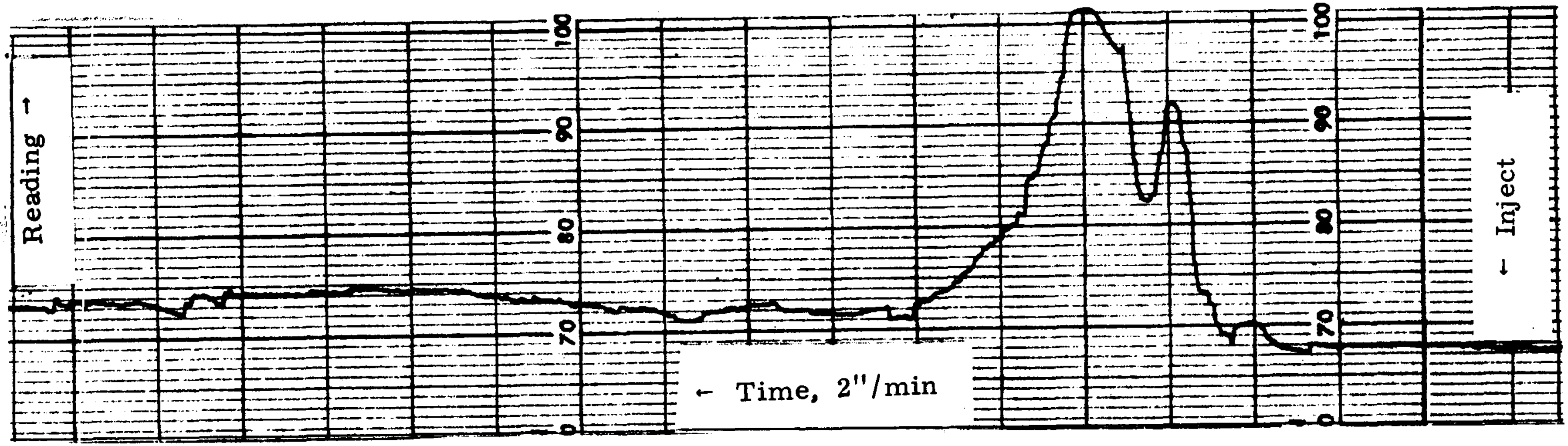

Figure 2B. Fan Off (time increase to left)

Figure 2. Comparison of Mixing With and Without Fan

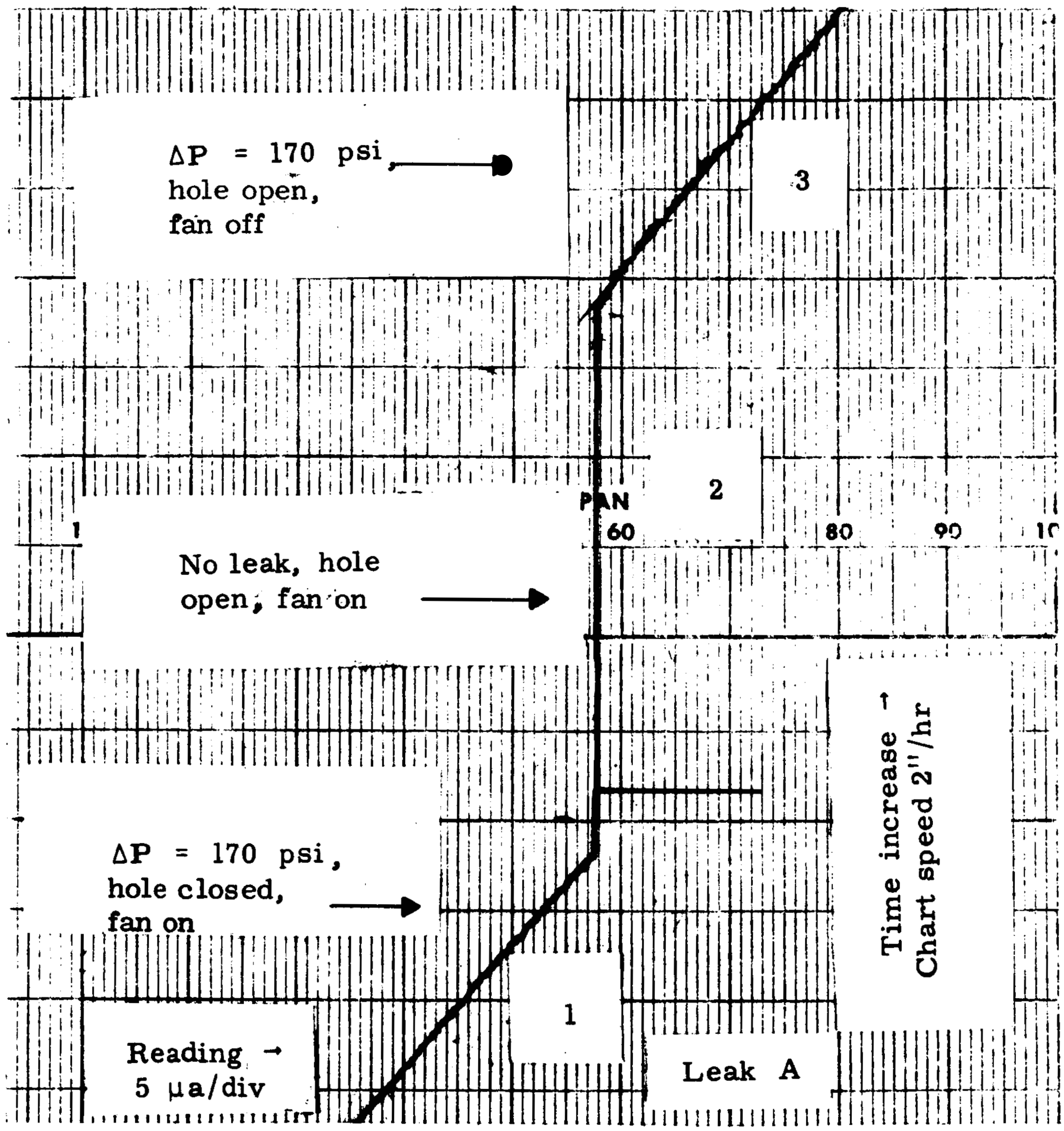

Figure 3. Experimental Run Showing the Effect of a Fan on Mixing and Also the Effect of a Hole in the Enclosure. Line Segment 1 - Leak A, 170 psi, Q = 120 cc(STP)/hr, Hole Closed, Fan On. Line Segment 2 - No Leak, Hole Open, Fan On. Line Segment 3 - Leak A, 170 psi, Q = 120 cc(STP)/hr., Hole Open, Fan Off.

SYSTEM LINEARITY

System linearity facilitates the interpretation of results. This characteristic was investigated by injecting fixed portions of helium into the enclosure with the fan on, and noting the incremental changes in the mass spectrometer reading throughout the span of the scale. Table 1 shows successive increments for the X1 and X10 scales on the General Electric LC-20 mass spectrometer.

Table 1

SCALE LINEARITY DATA INCREMENTS

X1 Scale Injection of 10 cc (divisions)	X10 Scale Injection of 50 cc (divisions)
5.8	11.0
5.4	11.6
5.9	11.2
5.8	11.5
5.6	11.1
5.5	

Mass Spectrometer: General Electric LC-20.
Settings: $P_m = 5\mu$, $P_t = 4 \times 10^{-5}$ torr; Emission = 1.

The difference between the greatest and least increment reading of the X1 scale was 5.32 percent and of the X10 scale was 8.83 percent. These values indicate that there is fairly reasonable linearity over the entire detection range. Although the results suggest the X1 scale has better linearity, it was found that there tended to be greater drift problems on this scale than on the X10 scale. Hence, use of the X10 scale is recommended whenever possible. Figure 4 is a reproduction of the data of the X10 scale.

SYSTEM STABILITY

The stability of the system is important in obtaining accurate results. In general, adequate stability over a period of several hours was obtained with this system. Line segment of Figure 3 illustrates the system stability obtained over a 1.5 hour period. This segment was obtained by removing the leak from the enclosure, without plugging the port for the leak. No significant change in reading is apparent.

The mass spectrometer is the key to achieving system stability. Until the operating characteristics of the mass spectrometer are well known, close attention should be paid to manifold and tube pressure settings. As pointed out in "Quantitative Leak Test Design Guide," sensitivity is a strong function of tube pressure, and hence a reliable means of controlling this pressure should be provided. Tube pressure control by means of the "inlet valve" in the original

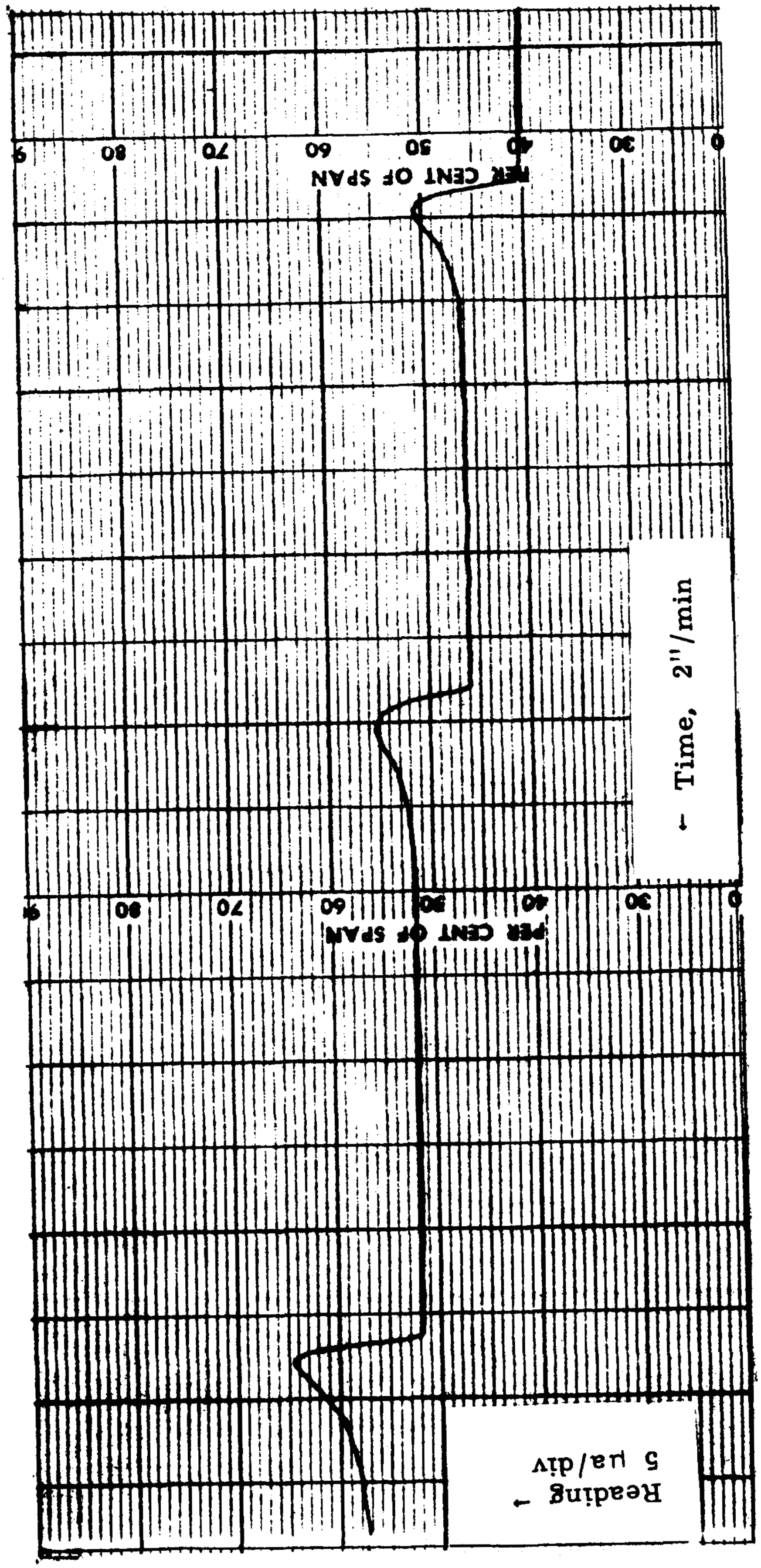

Figure 4. Scale Linearity Test

General Electric LC-20 mass spectrometer was rather touchy. The slightest jar to the inlet valve would result in large pressure excursions. However, with the use of a deep seated valve base, constant tube pressure and, therefore, stable operation were obtained. The latest General Electric models use a different valve.

A mass spectrometer with a large volume cold trap is recommended, since the quantity of liquid nitrogen remaining in the trap will also affect the tube pressure. Furthermore, it is recommended that the mass spectrometer be operated in a standby state for at least 12 hours before a run.

The calibrator also plays a part in system stability. It is recommended that, after adjusting the calibrator to the selected pressure, the proper valves be carefully closed so that there is neither pressure gain or undue loss. As a precautionary measure, the calibrator should be examined for pressure changes at least every 30 minutes and adjusted if necessary.

EFFECT OF LEAK AND PROBE POSITION

The concentration of helium, it might be anticipated, would be somewhat greater in the upper portion of the enclosure because of the relative densities of helium and air. For this reason the majority of tests were conducted with the probe relatively high (88 inches from the floor) and the calibrating leak relatively low (39 inches from the floor). However, a series of tests were performed in which the probe and the calibrating leak positions were interchanged. Figure 5 is typical of the results for the interchanged position. There is no evidence of any difference between the interchanged and normal positions; the spikes were caused by the electronics. A listing of the flow rates and errors for the interchanged configuration are given in Table 2.

Table 2

FLOW RATES AND PERCENT DIFFERENCES

Q(STP) (cc/hr.)	% Difference
15.8	- 2.3 †
15.8	- 7.5
15.8	+12.0
17.3	-12.5
36.0	+10.9
45.0	-16.3
68.5	+ 1.6
68.5	-12.6

Data are based on the Injection Method. The mass spectrometer was set at $P_m = 5\mu$, $P_t = 4 \times 10^{-5}$ torr in all the runs except the one marked with † where $P_m = 10\mu$

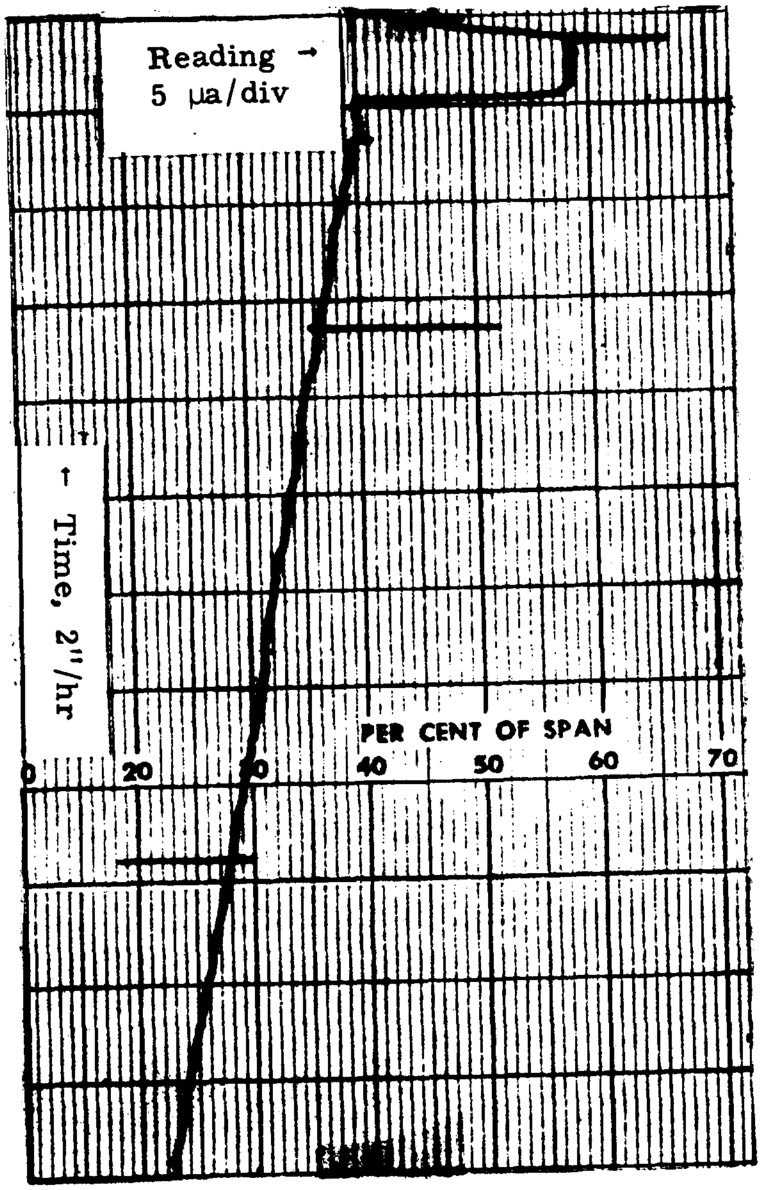

Figure 5. Injection Method With Probe And Leak Interchanged.

The average error in this setup was 9.5 percent, not significantly different from the average error for the original positioning. Therefore, provided the enclosure is adequately equipped with fans or blowers there is no effect of probe or leak position.

SYSTEM SENSITIVITY

A plot of the sensitivity of the system versus tube pressure at a manifold pressure of 15μ is given in Figure 6. The sensitivity is in units of μa/μatm where μatm equals 10^{-6} atm.

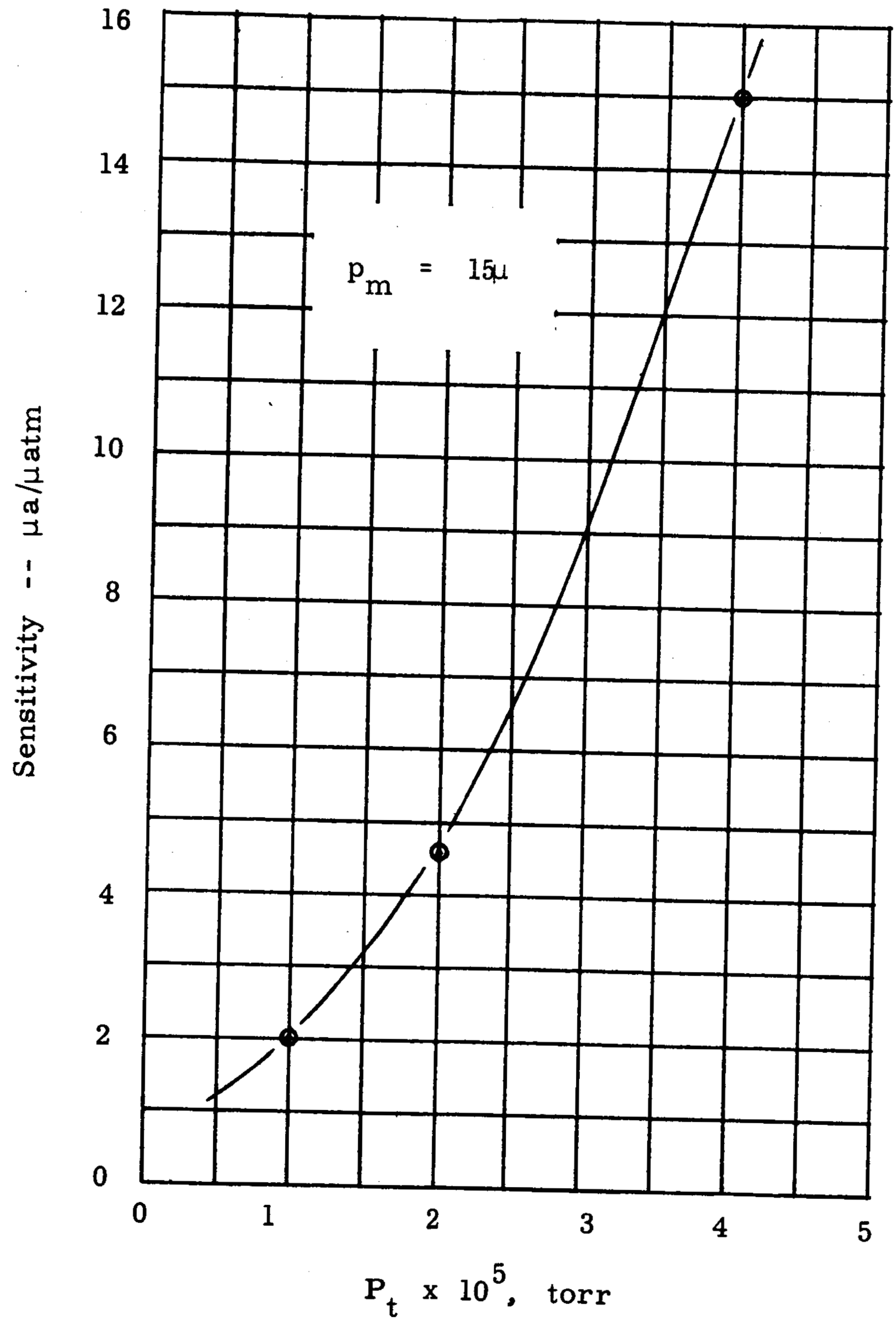

Figure 6. Detector Sensitivity versus Tube Pressure

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Section 6

SUMMARY OF EXPERIMENTAL TEST RESULTS

Three different methods for estimating leak rates with this system are described in the "Quantitative Leak Test Design Guide." These methods are outlined in this section and the results of experimental tests run by the methods are reported. The methods are:

1. The Comparison Method, in which the test leak is directly compared with a calibrated leak.
2. The Superposition Method, in which the ratio of the rate of increase for the test leak alone and the combined calibrated and test leaks is an indication of the test leak rate.
3. The Injection Method, in which the rate of helium increase in the enclosure is determined, and then the enclosure calibrated by injecting a known amount of helium.

Procedures common to all the methods are:

1. Pressurizing the test object with pure helium or a helium-nitrogen mixture (preferably the former).
2. Enclosing the object in a bag or tent.
3. Sensing the helium content in the enclosure and determining the rate of current increase in a mass spectrometer tube.
4. Calibrating the current increase in the mass spectrometer in terms of a helium rate by either 1) comparing to known helium leaks or 2) injecting a known quantity of helium

COMPARISON METHOD

In the Comparison Method the leak rate data of an unknown leak are compared directly with the data obtained from a known leak. In the experimental test run helium was allowed to leak into the enclosure (Run A). The enclosure was ventilated and the leak allowed to continue at the same rate (Run B). Conditions of permeability and detector sensitivity are assumed identical for both runs.

Data from an experimental test run of the comparison method are given in Figure 7. In this figure, as in all the others, time increases to the left. Both Figures 7A and 7B show data from a test where the leak rate was 15.8 cubic centimeters per hour. A tracing made of the data from Run A superimposed on the data from Run B would show an almost exact agreement between the data from the two runs. Some very interesting observations may be made from these figures. The following data for the slope was taken directly from the curves:

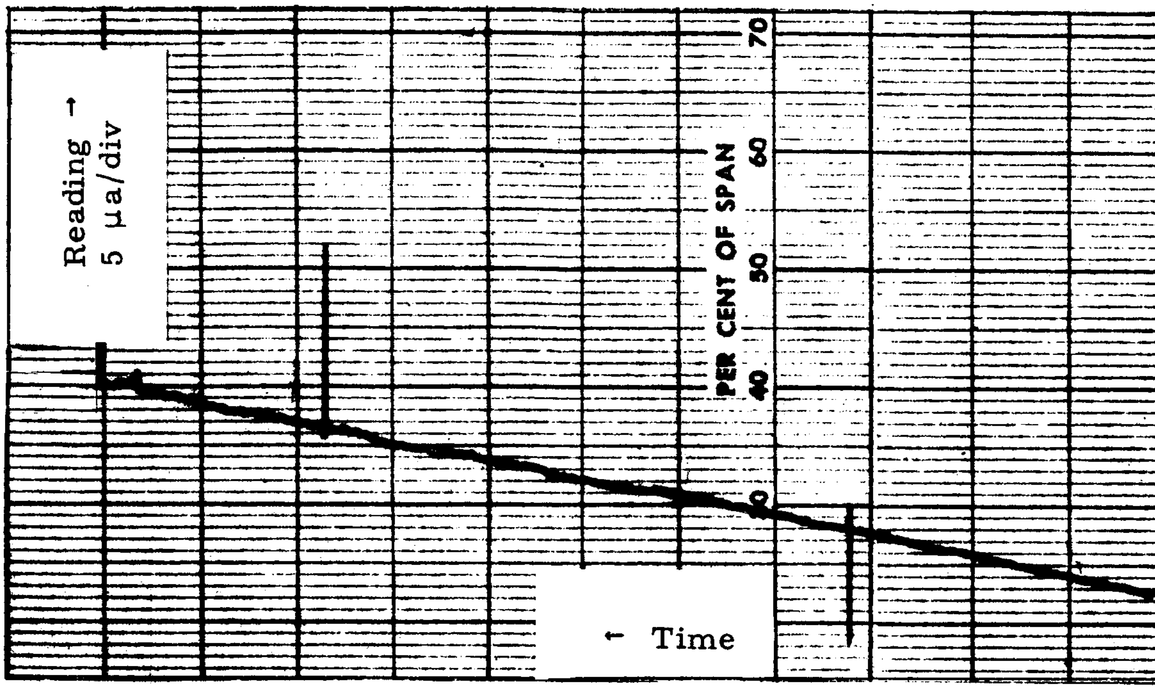

Figure 7A. (Q = 15.8 cc/hr, 2"/hr)

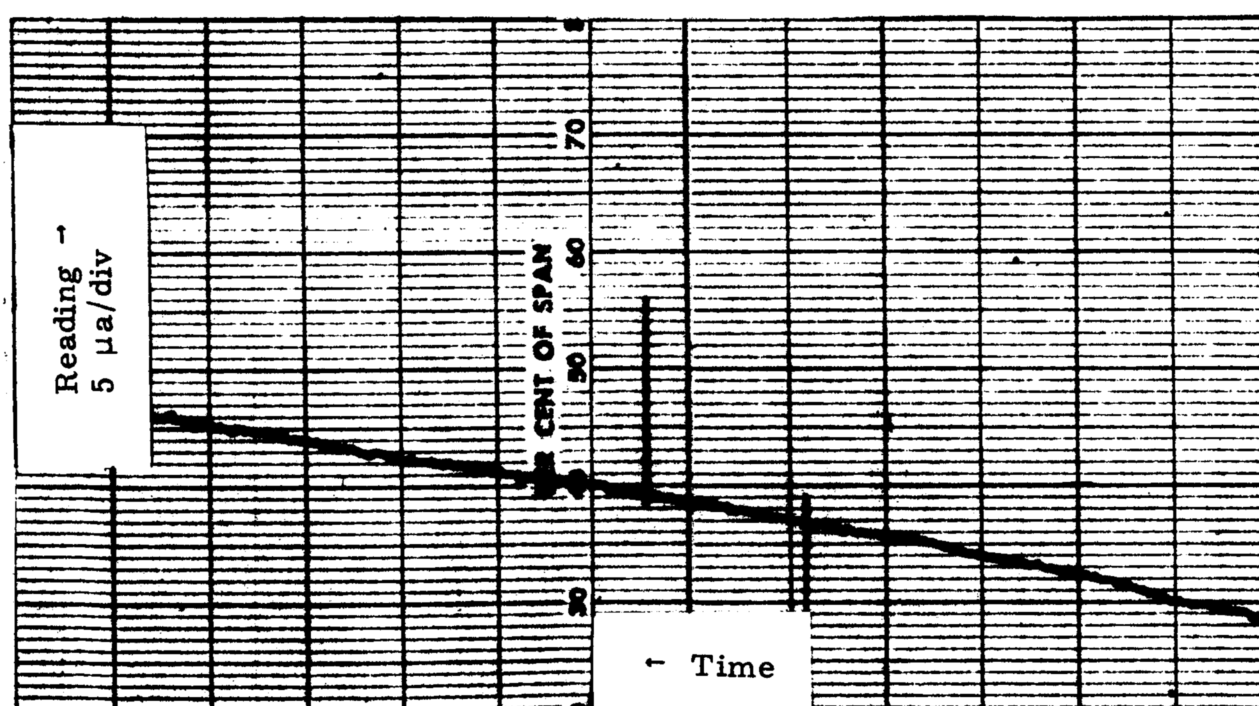

Figure 7B. (Q = 15.8 cc/hr, 2"/hr)

Figure 7. Comparison Method Data

Run	Range	Slope
A	30 to 40 divisions	6.14 divisions per hour
B	30 to 40 divisions	6.00 divisions per hour
B	40 to 50 divisions	5.34 divisions per hour

The slopes of the curves from Runs A and B agree within 2.3 percent in the same range, that is, the 30 to 40 divisions range. However, a comparison of slope A in the 30 to 40 range with slope B in the 40 to 50 range reveals a discrepancy of 11 percent. This might be attributable to permeation at the higher helium concentrations. Permeation here does not necessarily mean permeation through the bag but through seals, say, to the floor. This might appear to contradict the statements regarding system stability which were made in Section 5, "System Characteristics". However, the hole caused by opening the port for the leak in the system stability study was protected by a fitting, and therefore may not have been as susceptible to helium loss as an unprotected hole or one in another position. Care should be exercised in forming the enclosure seals.

Of the three procedures, the Comparison Method requires the most time. It is the most general and will be the most likely to yield accurate results in cases of high permeation. Several calibrator runs may be necessary to obtain a good estimate of the unknown leak.

SUPERPOSITION METHOD

The data shown in Figure 8 were obtained by the Superposition Method. The first leak of 15.8 cubic centimeters (STP) per hour was run into the enclosure for a period of time and then another leak of 45.0 cubic centimeters (STP) per hour was superimposed. In an actual test the first leak would have been the unknown and the second would have been the standard or calibrated leak. The slopes of segments 1 and 2 are, respectively:

$$S_1 = 6.4 \text{ divisions per hour}$$

$$S_2 = 22.6 \text{ divisions per hour}$$

The slope ratio is $m = 22.6 / 6.4 = 3.53$. In the discussion of the Superposition Method in the "Quantitative Leak Test Design Guide", it was shown that the unknown leak is related to the calibrated leak rate by the expression:

$$Q_u = \frac{Q_c}{m - 1} \qquad (1)$$

where Q_u is the unknown leak rate and Q_c is the calibrated leak rate. Solving for Q_u:

$$Q_u = \frac{45.0}{3.53 - 1} = 17.8 \text{ cubic centimeters per hour}$$

which differs from the rate of 15.8 cubic centimeters per hour as measured by a calibrating pipet by 12.6 percent.

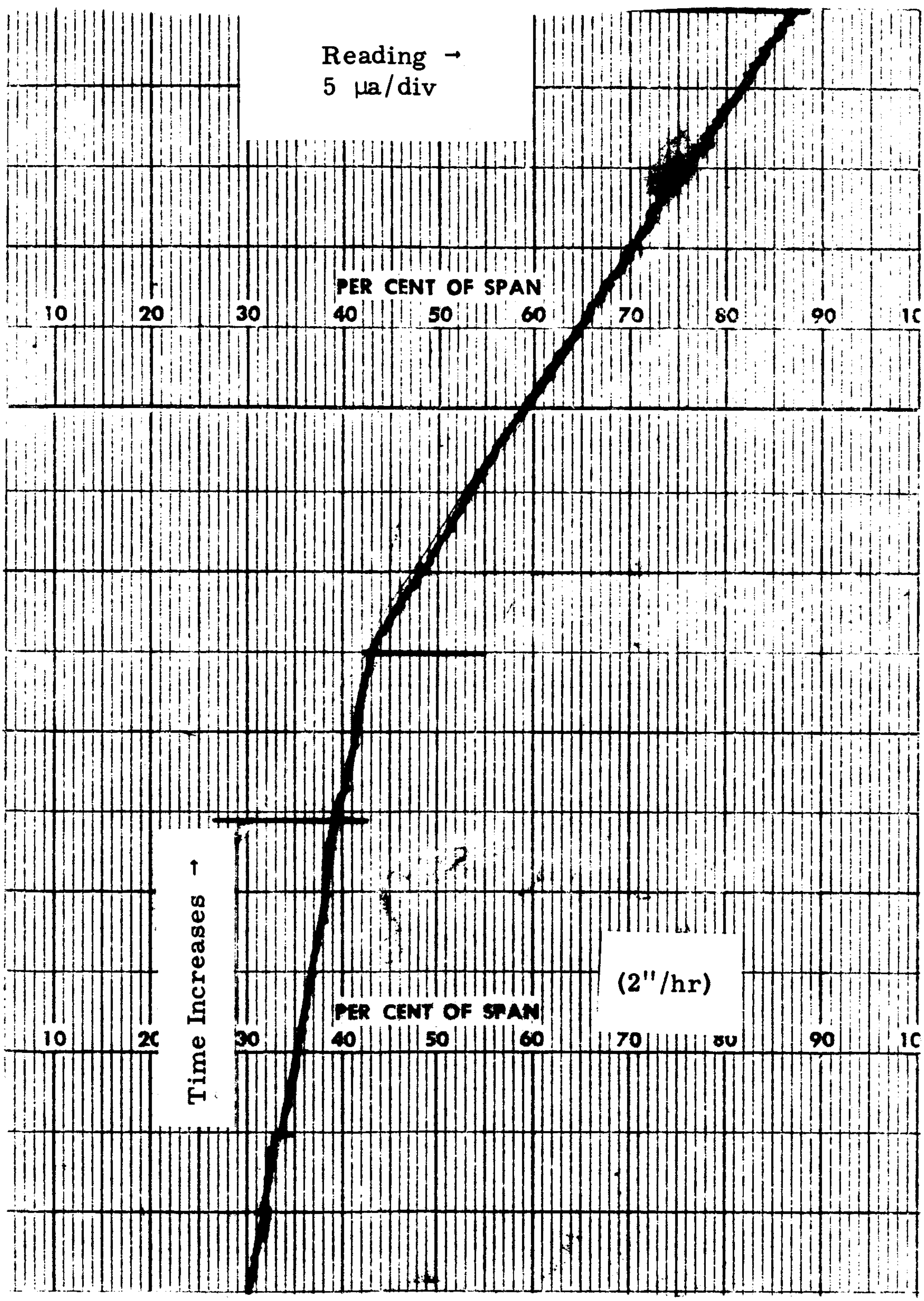

Figure 8. Superposition Method Data

The Superposition Method presented the most problems with regard to accuracy. Even if the slopes are accurate within a few percent, the estimation of the unknown leak rate may not be as accurate as with the other methods. Most of the superposition runs evidence little or no permeation. Hence, the reason for the relatively low accuracy obtained by this method is not obvious. Further study of the method is needed before positive conclusions about its accuracy can be drawn.

INJECTION METHOD

In the Injection Method the unknown leak is allowed to run for a period of time sufficient to establish a firm slope. Then helium is injected into the enclosure, calibrating the system. A detailed description of the method is given in the "Quantitative Leak Test Design Guide." A sample plot is presented in Figure 9. From this graph, the leak rate was estimated to be 16.2 cubic centimeters (STP) per hour, whereas in actuality it was 15.8 cubic centimeters (STP) per hour, a difference of 2.27 percent.

Some practical hints for performing injection runs are:

1. The rate of increase in the mass spectrometer reading is large at the beginning of the run. After about ten minutes this rate decays to a smaller, steady rate. This does not appear to be due to permeation, but rather seems to be a characteristic of the mass spectrometer. However, if "tailing-off" is observed, an inspection should be made of the seals.

2. After the injection is made, the run should be allowed to progress undisturbed until a second slope, displaced in proportion to the amount of injection, is firmly established. This second line is useful in ascertaining the slope. It was found convenient to use two 30-60 triangles aligned to form two parallel lines. The parallel lines of the triangles then are adjusted to match the lines of the data from which the slope may be determined.

3, With the exception of two or three points, all the data were gathered with leak detector settings of $P_m = 5\mu$, $P_t = 4 \times 10$ torr. There is some further evidence, which was not fully explained due to lack of time, that greater accuracy can be obtained with $P_m = 10$, 15, or 20μ.

The data for several injection runs are summarized in Table 3. A positive error means that the actual leak (as measured by a calibrating pipet) is greater than the calculated leak rate and conversely for negative errors. The average error for 20 runs, neglecting the -51.5 percent point was 10 percent. There were 12 positive and 8 negative deviations with average errors of 10.8 percent and 9.8 percent respectively. There does not seem to be a systematic error. The average length of time for a run was two to four hours.

The leak rate plotted in Figure 9 is only 15.8 cubic centimeters (STP) per hour into an enclosure of 231 cubic feet. However, about 2.5 hours were required to complete the run and estimate the rate to within 2.3 percent.

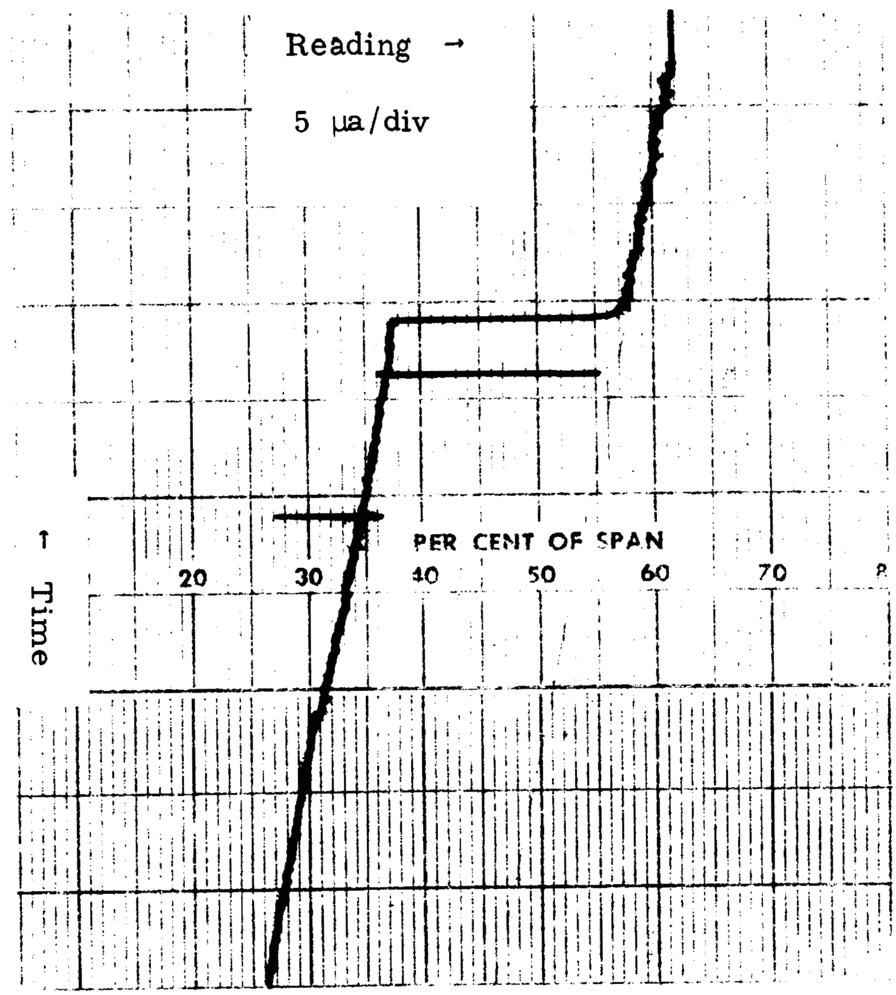

Figure 9. Injection Method Data (2"/hr, 50 cc injected).

For this enclosure volume, probably 5 cubic centimeters per hour is the lowest leak rate which can be accurately estimated. Since this leak rate is about one-third that of Figure 9, we would expect the testing time to be about 10 hours. Possibly rates lower than 5 cubic centimeters per hour might be accurately estimated with a higher sensitivity scale. However, stability problems then generally become serious.

Table 3

INJECTION DATA

Leak Rate (cc(STP)/hr)	Percent Error
6.125	+ 28.8
14.4	- 11.5
15.8	- 1.4
15.8	- 7.5*
15.8	+ 12.0*
16.2	+ 11.0
17.3	- 12.5*
32.7	+ 3.2
32.7	+ 0.5
33.5	+ 8.38
36.0	+ 10.9*
37.8	- 51.5
45.0	- 16.3*
66.7	+ 20.0
66.7	+ 16.75
67.4	- 13.9
68.5	+ 1.6*
68.5	- 12.6*
99.2	+ 10.2
99.2	+ 5.45

*These data were taken with the probe and leak interchanged, i.e. probe low and leak high. The other data were taken with the normal positioning.

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Section 7

CONCLUSIONS AND RECOMMENDATIONS

TEST EQUIPMENT

1. The calibrator performs as designed. During the course of a run it should be checked every 30 minutes and adjusted if necessary.
2. There is negligible permeation through the polyvinyl choride bag. However, the seals should be carefully made.
3. The enclosure may be readily vented after a run either by lifting or by opening one of the sides.
4. For added convenience, a door could be put in the enclosure which would facilitate venting.

SYSTEM CHARACTERISTICS

1. One household fan in the enclosure is an effective mixer for most systems. However, large complex systems may require two fans.
2. The system exhibits a linear response over the entire range of helium levels investigated.
3. Attention should be given the stability of leak detector settings and particularly to the mass spectrometer tube pressure.
4. The position of the probe or leak is not of great importance.

EXPERIMENTAL RESULTS

1. The Comparison Method has the widest range of applications but requires the most time.
2. The Injection Method gives the most rapid results, taking three to four hours with an accuracy of 10 percent. It is recommended for general use. There is some indication that better accuracies can be obtained by using manifold pressures of 10, 15, or 20μ. This should be further investigated.
3. In view of the speed of the methods it would be possible to perform three to four runs on the test object in a 24-hour period. An average value would enhance the accuracy of all the methods.

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Appendix I

LEAK DATA

Three leaks spanning the range from 10^{-4} to 10^{-2} cubic centimeters atmosphere per second, equipped with Millipore 0.22μ filters and filter holders were shipped along with the calibrator. The revised data for each of the leaks are given in Figure 10. It is this data which should be used for calibrations.

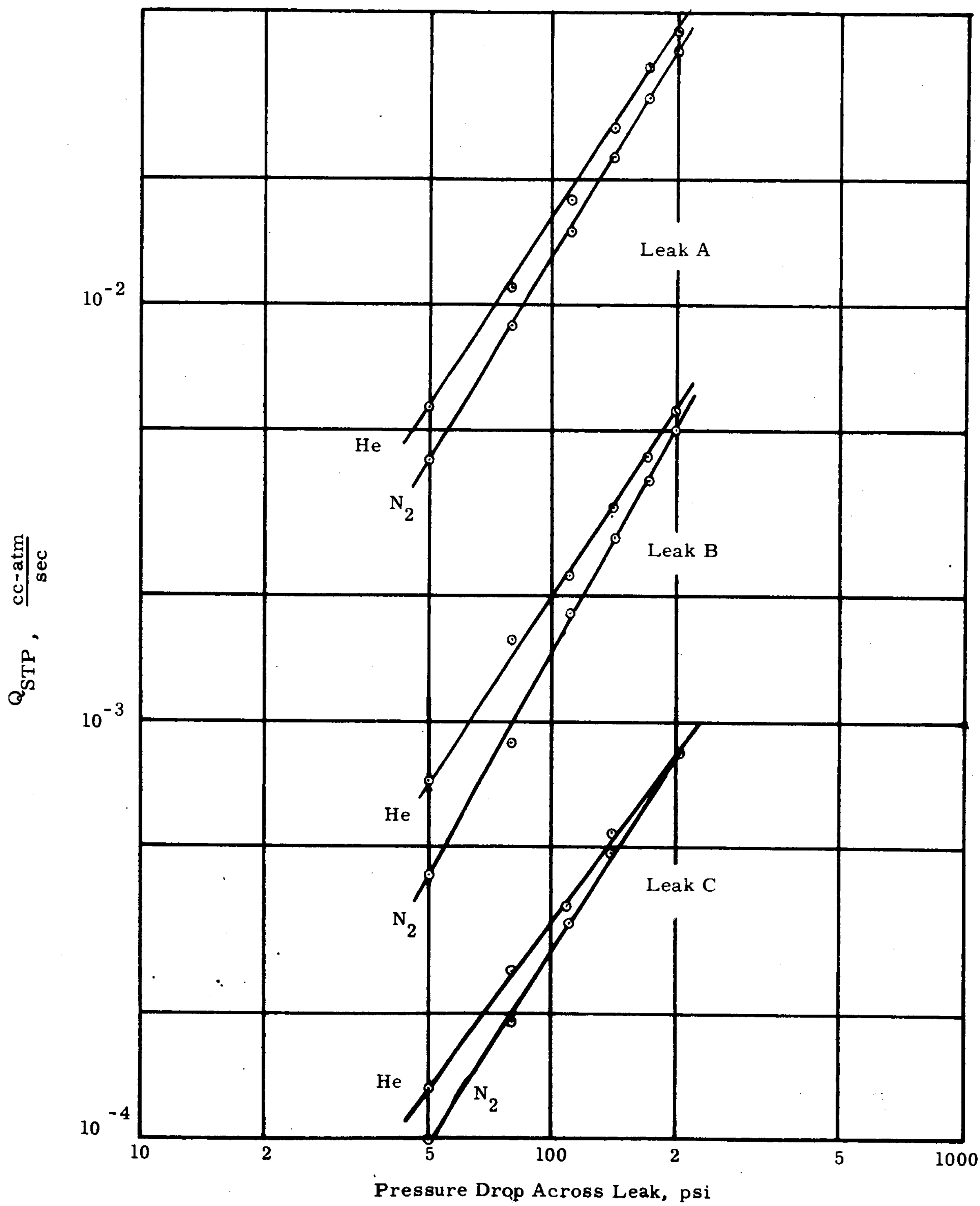

Figure 10. Data for Supplied Leaks