NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Technical Report No. 32-926

Basic Criteria and Definitions for Zero Fluid Leakage

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ABSTRACT

This Report defines both liquid and gaseous zero leakage over a pressure range from 1.0 and 0.1 psi, respectively, to 10,000 psi. Graphs are included for clarity and use. The zero liquid leakage graph contains an equivalent gaseous helium test leakage curve. A fluid flow conversion nomograph was constructed from certain applications of the Poiseuille equation and is offered to help simplify efforts of specification writers and test personnel in obtaining equivalent flow rates between fluids, over a wide range of pressures and temperatures. Discussions of leak test methods and test techniques complete the Report. This Report is important to such current programs as *Mariner* and *Surveyor*.

I. INTRODUCTION

As spacecraft missions become more sophisticated, and operate for increasingly greater periods of time, the requirements for improved performance by the systems and components become tighter. One measure of performance is the ability of the fluid flow system, or individual component, to perform satisfactorily under all predicted conditions without failure, which includes without excessive leakage.

Excessive gas or propellant leaks could: (1) affect the performance of an onboard scientific experiment located near the leak exit; (2) require additional attitude control system compensation to create abnormal increase of operating fluid consumption; (3) create a hazardous condition resulting from accumulation in the spacecraft; and 4) decrease the operating fluid's reserve to cause a system's decreased performance, or even a malfunction.

The word *excessive* is relative to the size of the system and to the quantity of the reserve operating fluids. The need for maximizing the payloads is a constant system factor. The resulting propulsion system requirements demand economy of propellants and pressurizing gases. The requirement for extremely small, or no leakage, is fast becoming a prime requisite.

The development of techniques to detect leaking fluids has been painfully slow. The problem has been complicated by the fact that many less-than-satisfactory means of defining the leakage requirements have been attempted for the sake of simplifying design, procurement, and testing specifications. Early definitions of the term *zero leakage* were vague, and did not suggest any leak test procedure as the basis for any definition which could be used by specification writers and test personnel.

II. LEAK DEFINITIONS AND UNITS OF MEASURE

A. Definitions

The confusion that exists over definitions in the field of leak detection demands that certain terms be defined before continuing further.

1. Leak

As noted in Ref. 1: "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."

For the purpose of this Report the preceding should be amended to:

> a crack, hole, or porosity in the wall of an enclosure capable (contrary to intent) of passing fluid from one side of the wall to the other under action of a pressure or concentration differential existing across the wall.

As a rule, although a leak may be detected during testing, its geometry will remain unknown. Leak testing is mandatory for inspection and rate measurement, because analysis is impossible.

2. Leak Rate and Leakage

In defining the rate of flow of fluids past a leak, the words *leak rate* and *leakage* are used synonymously.

B. Units of Measure

1. Gas Flow

As observed in Ref. 1: "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 pressure on the exit side, which is low enough to have negligible effect on the rate of flow."

In this Report, the unit of measure for gas flow, only, shall be standard cubic centimeters per second (std cm³/sec). The word *standard* is used here to indicate that the flow measurements were converted to standard temperature and pressure (STP) conditions and to show the relationship to mass flow. The following equations are presented for illustration purposes:

$$\dot{W} = \frac{PVM}{RT} \tag{1}$$

as derived from

$$PV = \frac{W}{M} RT$$

2. Liquid Flow

In this Report the liquid flow, *liquid leakage*, is a volumetric unit of measure, cm³/sec.

III. ZERO LIQUID LEAKAGE

A. Definition

Zero liquid leakage is that value of liquid leak or flow rate at which the surface tension of the liquid has just overcome the pressure acting on the liquid, Fig. 1, and *no* flow occurs – assuming a given pressure and leak diameter. Experiments conducted (Ref. 2) have indicated that generally for liquid there is a range of values at each pressure where the flow rate becomes zero. It is in this range that each liquid has its own zero point at any given pressure.

B. Discussion

Figure 2 shows the two major liquid flow modes: turbulent and laminar. The equation for liquid turbulent flow is (Ref. 2)

$$Q_v = \frac{\pi}{4} d^{5/2} \left(\frac{\Delta P}{2\rho f \zeta} \right)^{1/2} \qquad (2)$$

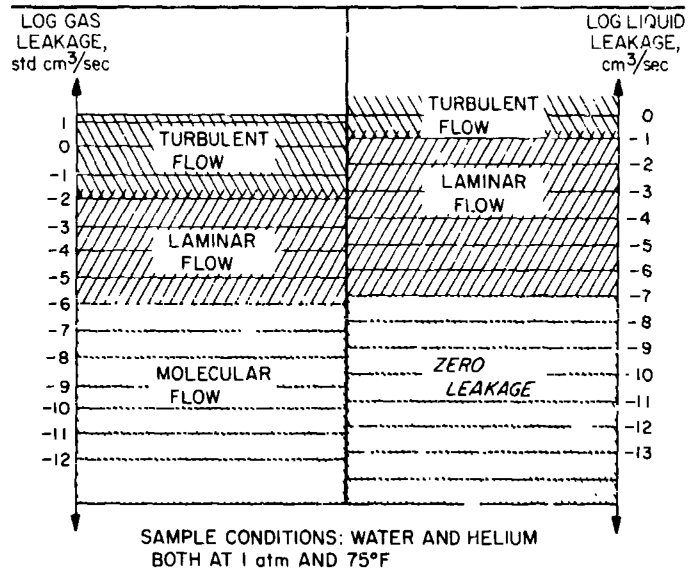


Fig. 2. Correlated Liquid and Gas Flow Modes

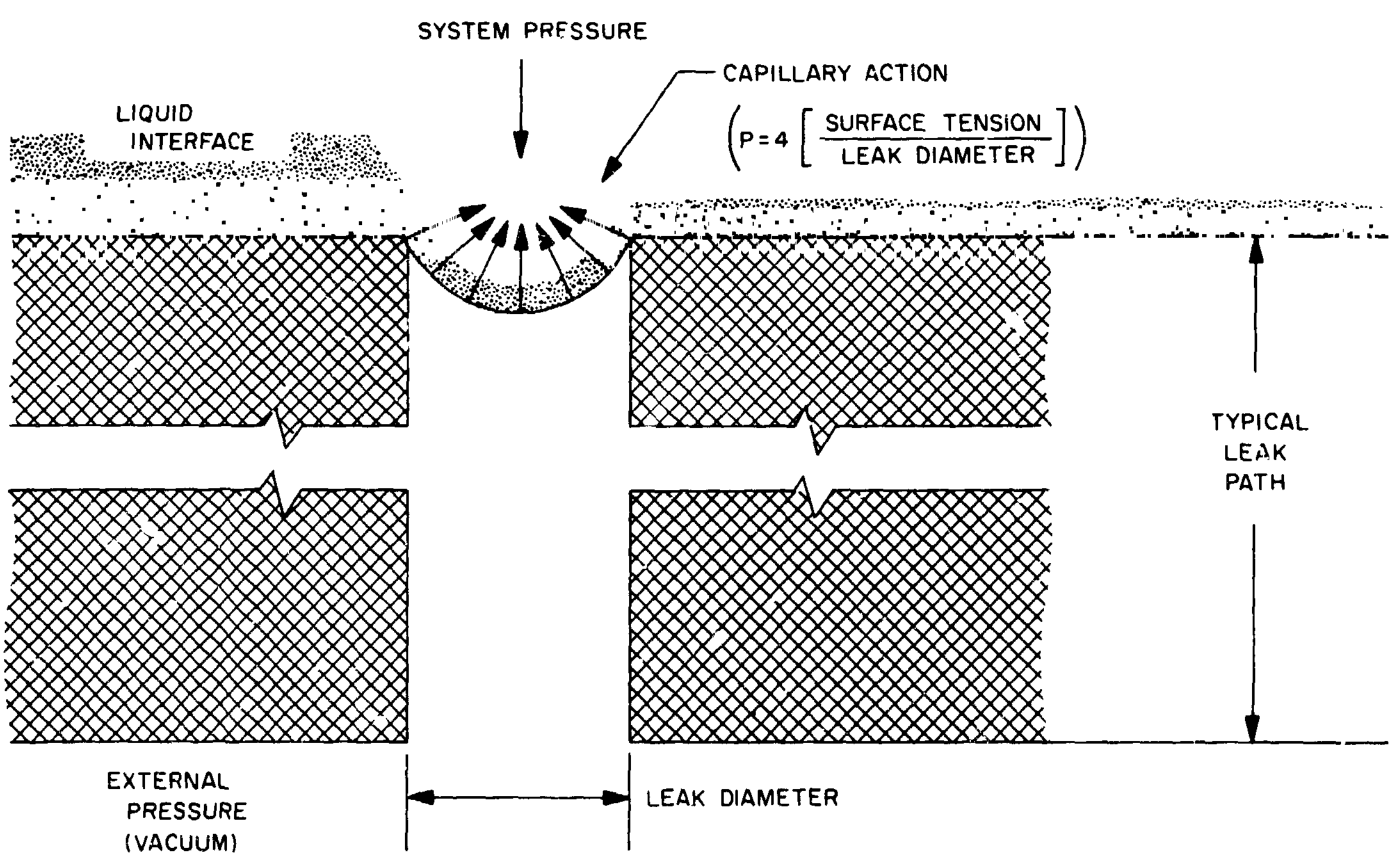


Fig. 1. Mechanism of Clogging

The equation for laminar flow is (Ref. 4)

$$Q = \frac{\pi}{4}\left(\frac{d}{2}\right)^4 \frac{P_2 - P_1}{\eta \zeta} \qquad (3)$$

Flow rate Q is in pressure-volume/time units.

The preceding equations are presented as illustrations only, and are not to be used for calculations.

As the Reynolds Number for laminar flow decreases as a result of increasing viscosity, surface tension becomes the dominant force causing cessation of flow. At this point, zero leakage occurs. However, there is a peculiar set of conditions in which a liquid *wets* the walls of the leak path, thus eliminating the surface tension effects, in turn permitting liquid flow to continue even farther. More work is required to increase knowledge about this effect. It will be shown later that any increased pressure serves to lower the zero leak point for a liquid.

The rate of propellant evaporation through the leak path to a vacuum was examined and found negligible compared with the original liquid leak rate.

Figure 2 shows gaseous helium flow rates directly correlated with liquid flow rates for the same pressure and temperature (75°F, 14.7 psia). This simplified method for correlating liquid and gaseous flow rates was developed at the General Electric Company, Ref. 8, and presented in the form of a nomograph. The nomograph was constructed from the following equation, and is presented here for the reader's interest only.

For gas flow from a high pressure to atmospheric pressure, the Poiseuille equation for viscous flow is

$$Q = \frac{K}{\eta}(P^2 - 1)$$

where pressure units are in atmospheres.

For gas flow from a pressure, generally, but not necessarily atmospheric, to vacuum, the Poiseuille equation becomes

$$Q = \frac{KP^2}{\eta}$$

For essentially incompressible liquid flow with a known pressure differential, the Poiseuille equation becomes

$$Q = \frac{K' \Delta P}{\eta}$$

To construct the nomograph, the preceding three equations, using a common parameter (Δ P), were reduced to the following forms:

$$Q = \frac{K}{\eta} (\Delta P) (\Delta P + 2) \qquad (4)$$

$$Q = \frac{K}{\eta} \Delta P^2 \qquad (5)$$

$$\text{and } Q = \frac{K'}{\eta} \Delta P \qquad (6)$$

Figure 3 shows a simplified version of the nomograph with a sample problem to illustrate its use. The guidelines with a slope of 2:1 and located on the right-hand side represent the gas flow equations; while guidelines having 1:1 slope, and located on the left-hand side, represent the liquid flow equation. Correlating one fluid to the other requires drawing lines parallel to the nomograph guidelines. The discontinuity found between the gas flow and liquid flow guidelines is only the result of the original drawing style. However, a transition between gas and liquid flows is illustrated in the nomograph. The gradual bend represents exhaustion to atmospheric conditions. A sharp, or sudden change from liquid to gas, or vice versa, represents exhaustion to vacuum.

Working the sample problem shown in Fig. 3 will illustrate the application of the nomograph to predict the equivalent liquid propellant leak rate from measured gaseous test helium leakage at a seal.

Assume the following conditions:

1. Gaseous helium leakage past a seal at 8×10^{-6} std cm^3/sec.
2. Helium test pressure of 1 atmosphere (atm) or 14.7 psia with vacuum on downstream side of the leak, the ΔP across the leak being 14.7 psia.
3. Liquid system flow pressure of 10 atm or 147 psia with vacuum externally, or 147 psia ΔP across the leak.
4. Liquid viscosity of 8×10^{-1} centipoises (cp).

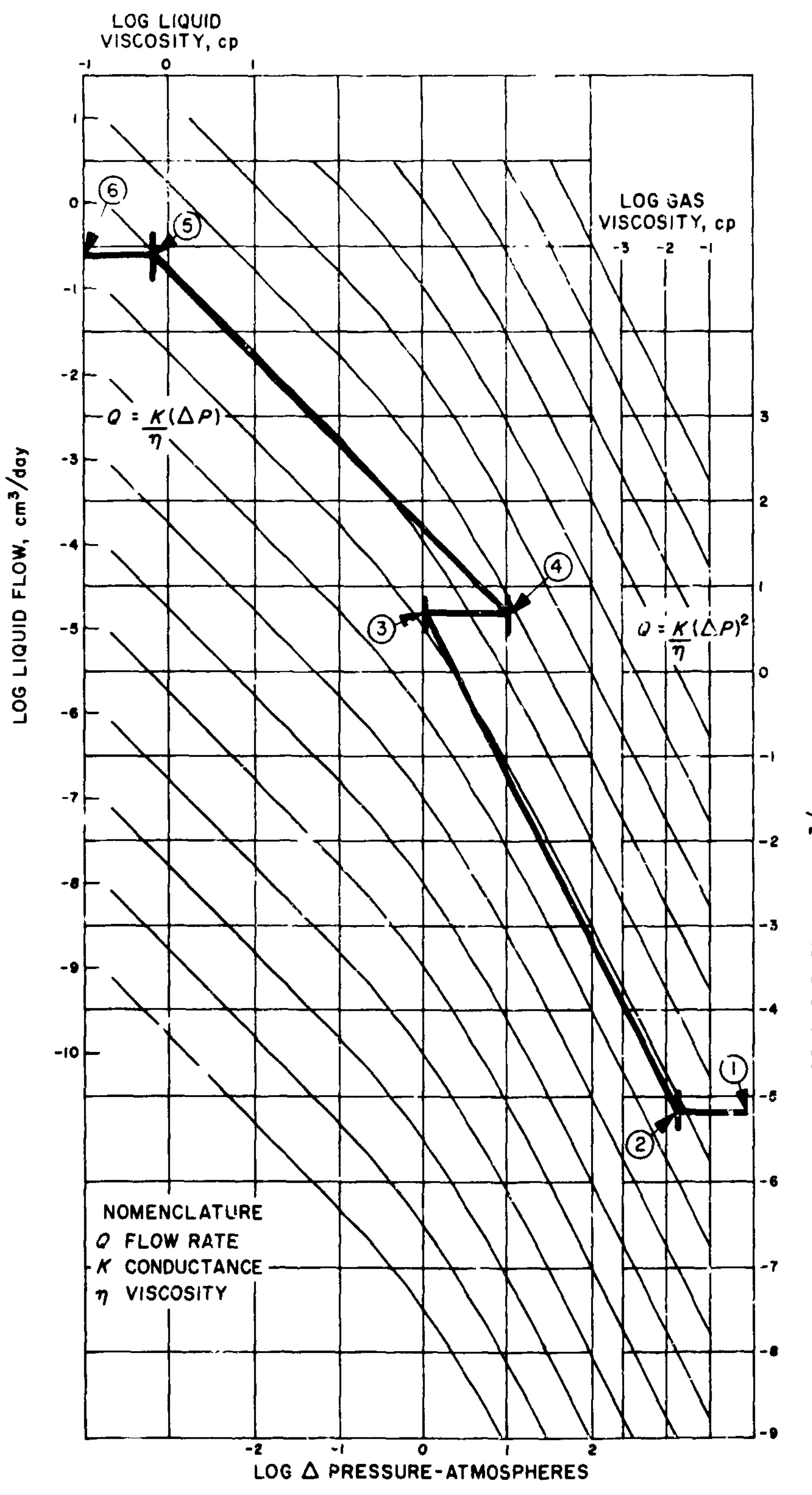


Fig. 3. Simplified Fluid Flow Conversion Graph

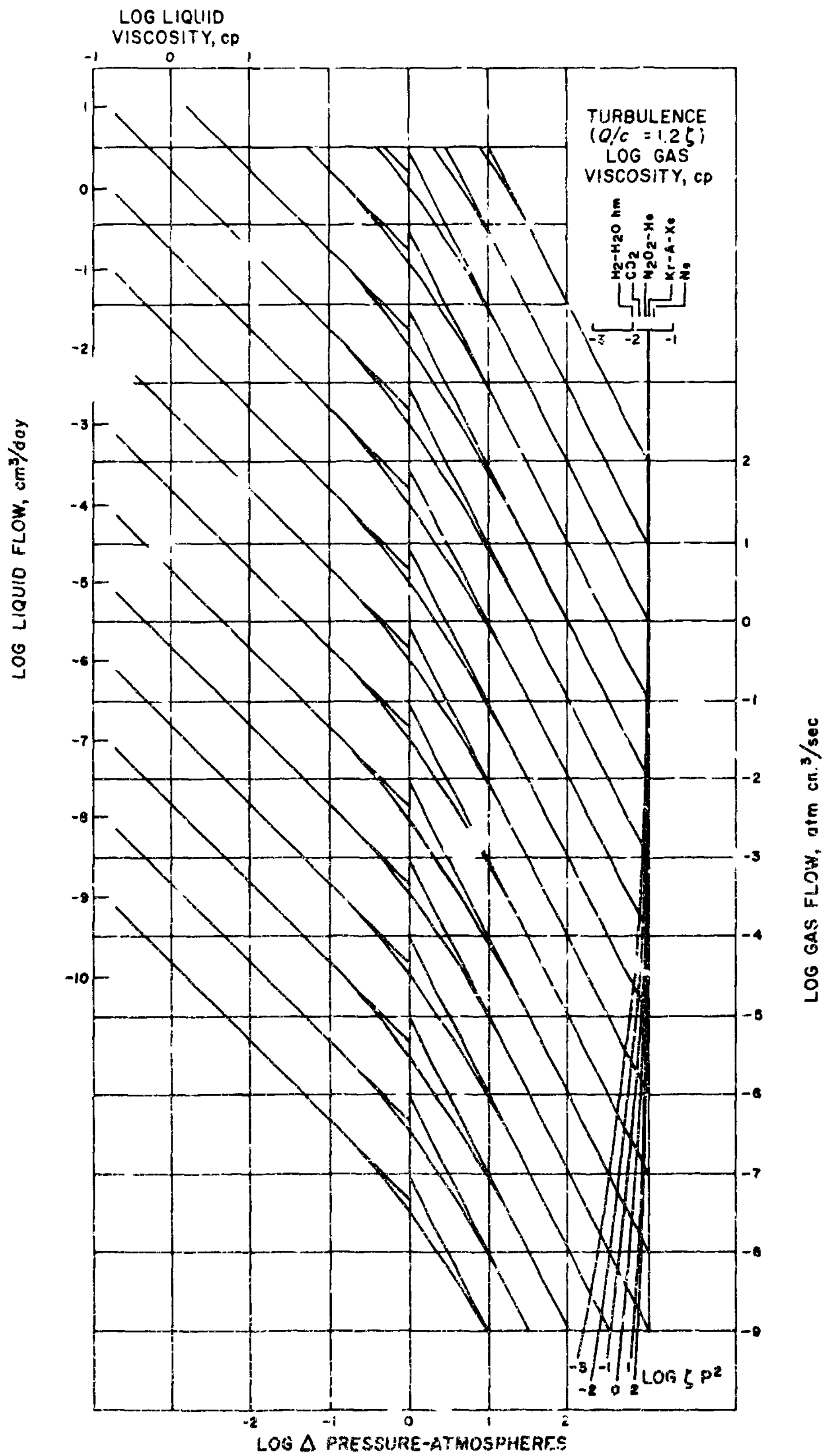
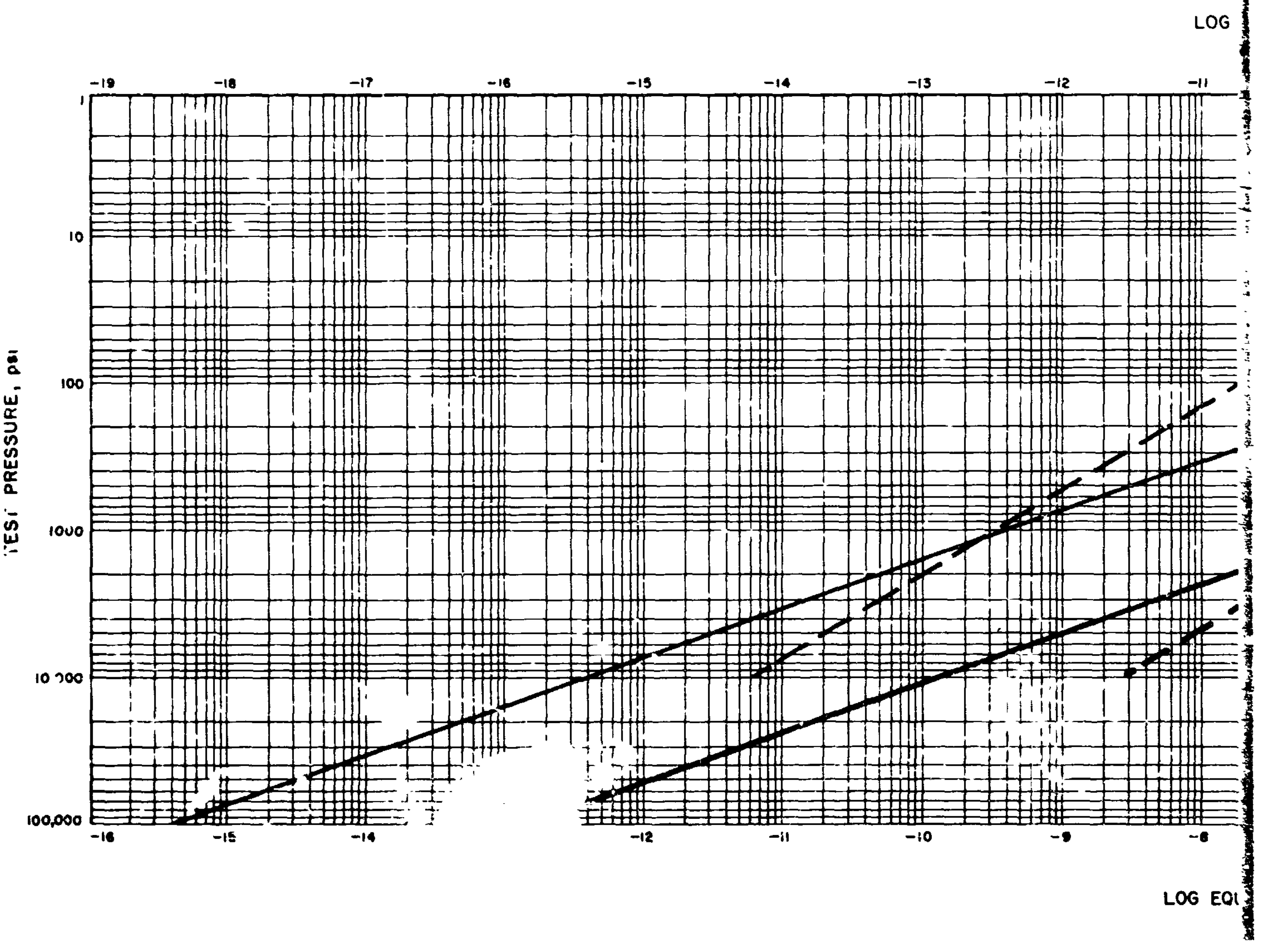


Fig. 4. Fluid Flow Conversion Graph



LOG
-19
-18
-17
-16
-15
-14
-13
-12
-11
TEST PRESSURE, psi
1
10
100
1000
10,000
100,000
-16
-15
-14
-12
-11
-10
-9
-8
LOG EQU

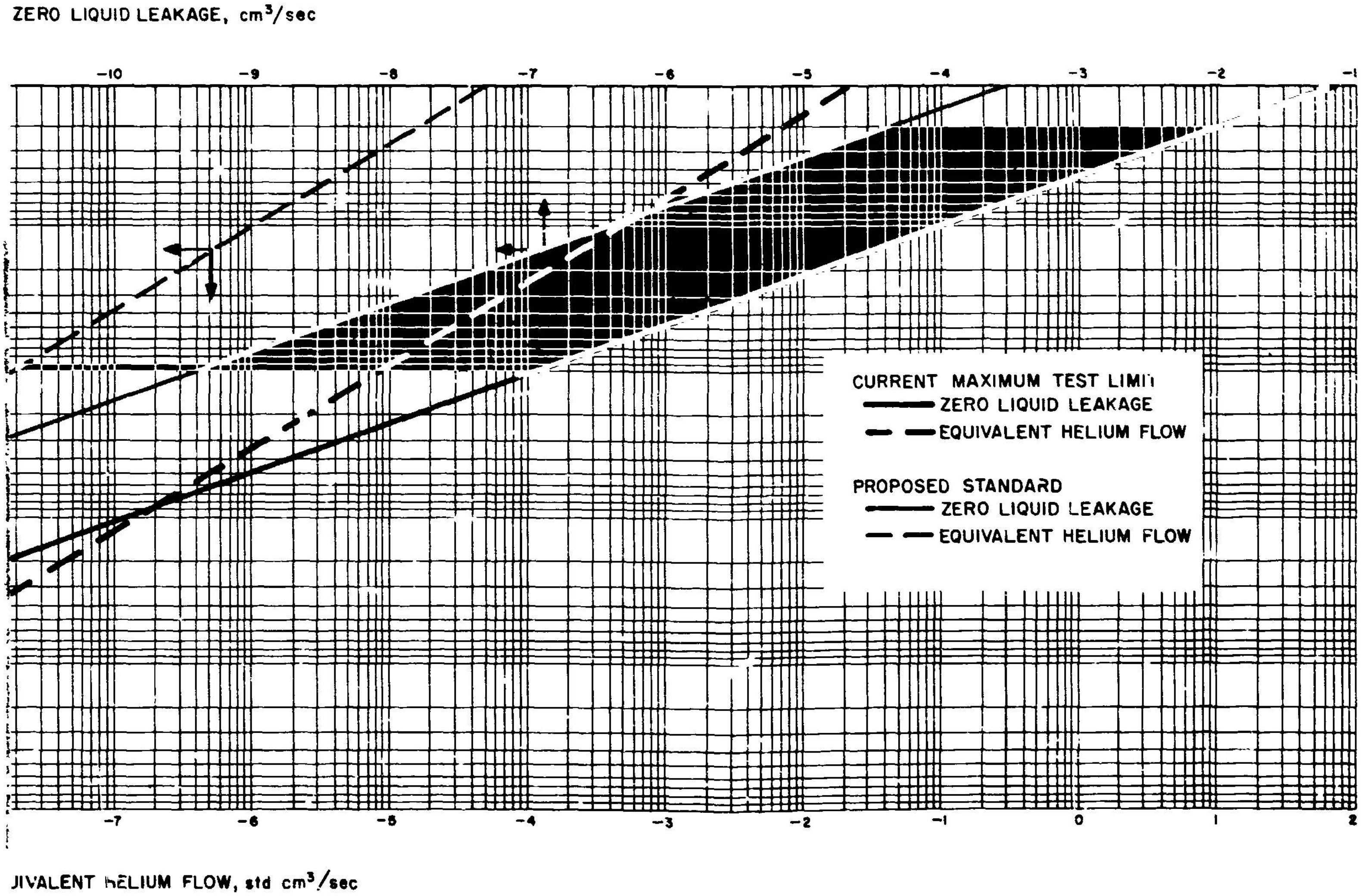


Fig. 5. Zero Liquid Leakage With Equivalent Test Helium Flow

To solve the problem of predicting the equivalent liquid *leak rate* for the known gaseous leakage at the seal, the following plots are made on the nomograph:

1. 8×10^{-6} std cm³/sec helium leakage is located on the right-hand ordinate (log gas flow).
2. A horizontal line is drawn intersecting the helium viscosity value along the abscissa (log gas viscosity).
3. A line parallel with the 2:1 slope is extended from the helium viscosity value until 1 atm pressure is intersected along the Δ pressure abscissa.
4. This pressure point is connected with the liquid system pressure (10 atm) by a horizontal line.
5. A line is drawn parallel with the 1:1 slope graph lines from the previously found liquid pressure value until the intersection is made with the value for the liquid viscosity along the log liquid viscosity abscissa.
6. A horizontal line is drawn from the intersection of liquid flow with liquid viscosity to find the predicted liquid leakage along the log liquid flow ordinate: 3×10^{-1} cm³/day.

While Fig. 2 shows a liquid-gas correlation at 1 atm pressure and 75°F, Fig. 3 provides a graphical solution for correlation at any pressure. Any temperature may be considered merely by selecting that corresponding value of viscosity.

Figure 4 is a complete fluid flow conversion graph. Added to it is a transitional flow component, which includes molecular flow, represented by the ζP^2 factor.

Figure 5 presents two zero liquid leakage models for most liquid propellants and their respective equivalent helium test gas flow rate curves. The upper zero leakage curve indicates the current standard, or maximum limit. It is based on one experimental test point developed earlier by the General Electric Company, whereby, at 1 atm of pressure, the clogging, or zero liquid leakage point for water was in the range of 10^{-6} to 10^{-7} cm³/sec. Assuming the value of 5×10^{-7} cm³/sec as one point on the curve, the following mathematical analysis provides that the required curve pass through the point, for the purpose of extrapolation through the desired pressure range. The equivalent helium flow rate is obtained with the application of the graph, given in Fig. 4, at the same point.

The second, or proposed, standard curve is based on later experimental data provided by Dr. Mann of the General Electric Company. It was shown that at 1 atm of pressure, the clogging, or zero leakage point for water is about 10^{-7} cm³/sec of equivalent helium flow. With this new point available, the new zero liquid leakage curve was obtained by employing the following mathematical analysis. However, as of this writing, the lower leakage curve remains as the proposed standard for further study and consideration. The desired curves are straight lines as shown by Eq. (10) and (13). The slopes of the curves drawn through the selected points are obtained from the following analysis.

For liquid flow to start, the pressure must just exceed the liquid surface tension, Ref. 7, or

$$\Delta P = \frac{4\gamma}{d} \tag{7}$$

which gives,

$$d = \frac{4\gamma}{\Delta P} \tag{8}$$

where γ is surface tension and d is leak diameter. Since from Eq. (3), $Q_{liquid} \propto d^4 (\Delta P)$, then substituting in d

$$Q_{liquid} \propto \frac{(4\gamma)^4}{(\Delta P)^4} (\Delta P) \tag{9}$$

or,

$$Q_{liquid} \propto \frac{(4\gamma)^4}{(\Delta P)^3} \tag{10}$$

Hence, the slope of the *zero liquid leakage* curve with respect to pressure is −3:1.

The equivalent gas flow based on surface tension of the liquid, or rather based on its start of flow because of just overcoming the surface tension γ effect, is found in a like manner.

From Eq. (5),

$$Q_{gas} \propto d^4 (\Delta P)^2 \tag{11}$$

and with the substitution of d, is

$$Q_{gas} \propto \frac{(4\gamma)^4}{(\Delta P)^4} (\Delta P)^2 \tag{12}$$

or finally

$$Q_{gas} \propto \frac{(\Delta\gamma)^4}{(\Delta P)^2} \tag{13}$$

Hence, the slope of the equivalent gas flow curve with respect to pressure is −2:1.

Therefore, from the preceding discussion, it is now possible to specify a *zero liquid leakage* value for liquid propellant systems and components. The curves in Fig. 5 define *zero liquid leakage* in terms of liquid flow rate and also suggest the means of precision testing, an objective stated in the Introduction. Until instruments capable of measuring extremely finite liquid flow rates are developed, the use of helium gas as a detecting medium will provide a precision technique. The helium spectrometer type of leak detector affords the means of detecting the low flows of helium which are equivalent to the required zero leakage, or rather that amount of equivalent gas flow, at any given pressure, where liquid flow ceases. In this manner, the specification writers and the testing personnel now have a practical definition for *zero liquid leakage*.

IV. ZERO GAS LEAKAGE

A. Definition

Zero gas leakage as such does not exist as far as laboratory measurements have thus far been able to determine because of the limitations of laboratory instruments. Therefore an arbitrary curve was constructed as shown in Fig. 6 for use as a specification standard.

B. Discussion

Figure 6 is a straight, sloped curve with a discontinuity at the leakage value of 1×10^{-7} std cm³/sec at which point the line is translated but maintains its original slope.

The lower portion of the curve is based on the basic point of 1 std cm³/yr at 1 atm differential pressure. Other points making up that portion of the curve were obtained from the correlation of the 1 std cm³/yr with equivalent flow rates at the other pressures using the fluid conversion graph. However, the knowledge of future propulsion system requirements dictated that the maximum acceptable equivalent leakage, as originally constructed, was too great at the higher pressures. Hence the arbitrary decision was made to shift a part of the curve upward at 1×10^{-7} std cm³/sec.

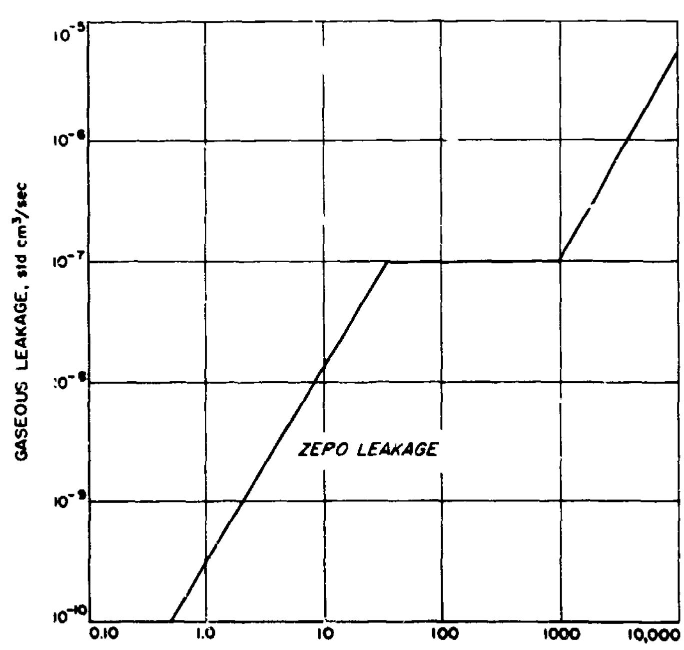


Fig. 6. Zero Gas Leakage

V. LEAK TEST TECHNIQUES

Two types of external leaks exist in most flow systems: (1) permeation through plastic and elastic materials and, (2) viscous flow as through a pinhole. The latter form of leak is the type to permit an equivalent liquid flow. However, the first form (permeation) is detectable during the bell jar type of helium mass spectrometer leak testing and can lead to a false picture when converting the total helium leakage to equivalent propellant leakage. Permeation leakage is the passage or migration rate of atomic helium (by various mechanisms) through the materials, being first adsorbed into the surfaces of the materials under a pressure-type or concentration-gradient driving mechanism and finally desorbed out of the external surface. The basic identifying characteristic of this kind of leak is a gradually increasing rate of change with respect to time. A strip-chart recorder wired to the mass spectrometer target output voltage displays the characteristic whenever it occurs, and also simplifies leak test monitoring. A second identifying characteristic is that at any given pressure, every plastic and elastomer has a helium saturation or leveling-out point past which the leak rate will not increase. This salient feature can be used as a final verification for permeation leakage. If repeated testing is required, and the testing cycle is a matter of only a few hours, the original starting test pressure must be low enough to permit saturation to occur. A convenient rule for increasing test pressure levels during repetitive testing within hours of each other is 2X over the previous value. However, with the removal of helium pressure, the helium concentration will eventually decay from within the plastic or elastomer. The time for helium concentration decay can be calculated. If one can tolerate the long period of time between tests, which may range from an hour to a day, then the test can be repeated at the same pressure as in the original test.

The identifying characteristic of the viscous leak is the relatively constant rate of fluid flow. However, because the leak path can be very tortuous to the flow of a test gas, the time required to traverse the path may take hours, even at pressures greater than 3000 psi. Therefore, precise leak tests should consume at least 3–4 hr of continuous monitoring. When recording the leak characteristic, the sudden change of *leak rate* to a higher but fairly constant value is indicative of additional helium from another viscous leak path.

It is this last type of leak that will produce the liquid propellant leak in a propellant subsystem. Therefore, it is now possible to correlate the leak from gaseous helium in terms of equivalent units of propellant in accordance with the procedure previously described. It is important that initial leak testing should first employ a gas, rather than a liquid, in order not to clog the leak paths.

Experiments have been conducted which show that the *zero leak* condition for liquids is between 10^{-6} and 10^{-7} cm^3/sec of liquids with surface tension being the criterion for the exact number. Experiments are currently under way to find the *zero leak* point for liquids, under NASA Contracts No. NAS 7-396 and -434.

Discrepancies appear at times between analytical and empirical leakage data. These may be caused: (1) by the lack of sufficient experimental data to support fully certain analytical techniques; (2) by the actual leak-test methods and techniques; and (3) by the fact that flow systems themselves are not designed to include plans concerning future leak checks. While experimental evidence is currently building up in support of the analytical methods used herein, the comparable work in the field is not keeping pace. Not enough work is being accomplished at the system level to improve the accuracy of actual test data; methods that are not amenable to precision continue to be employed because of poor planning and lack of patience in the test execution.

A number of leak-testing techniques, summarized in Fig. 7, have been used for some time. The more common ones are:

1. Soap solution (maximum test sensitivity to 10^{-4} std cm^3/sec).
2. Helium mass spectrometer probe or "sniffer." (Maximum test sensitivity quite variable from 10^{-4} to 10^{-6} std cm^3/sec.)
3. Bell jar or other such container with a helium mass spectrometer (maximum test sensitivity better than 10^{-9} std cm^3/sec).
4. Methods 2 and 3 employ a mixture of nitrogen and helium gases. The helium detection sensitivity is degraded by the volume ratio of the gases in order to use the preceding techniques for detection of gross leaks.

Use of the first two techniques does not assure the operator that he is measuring all of the escaping helium.

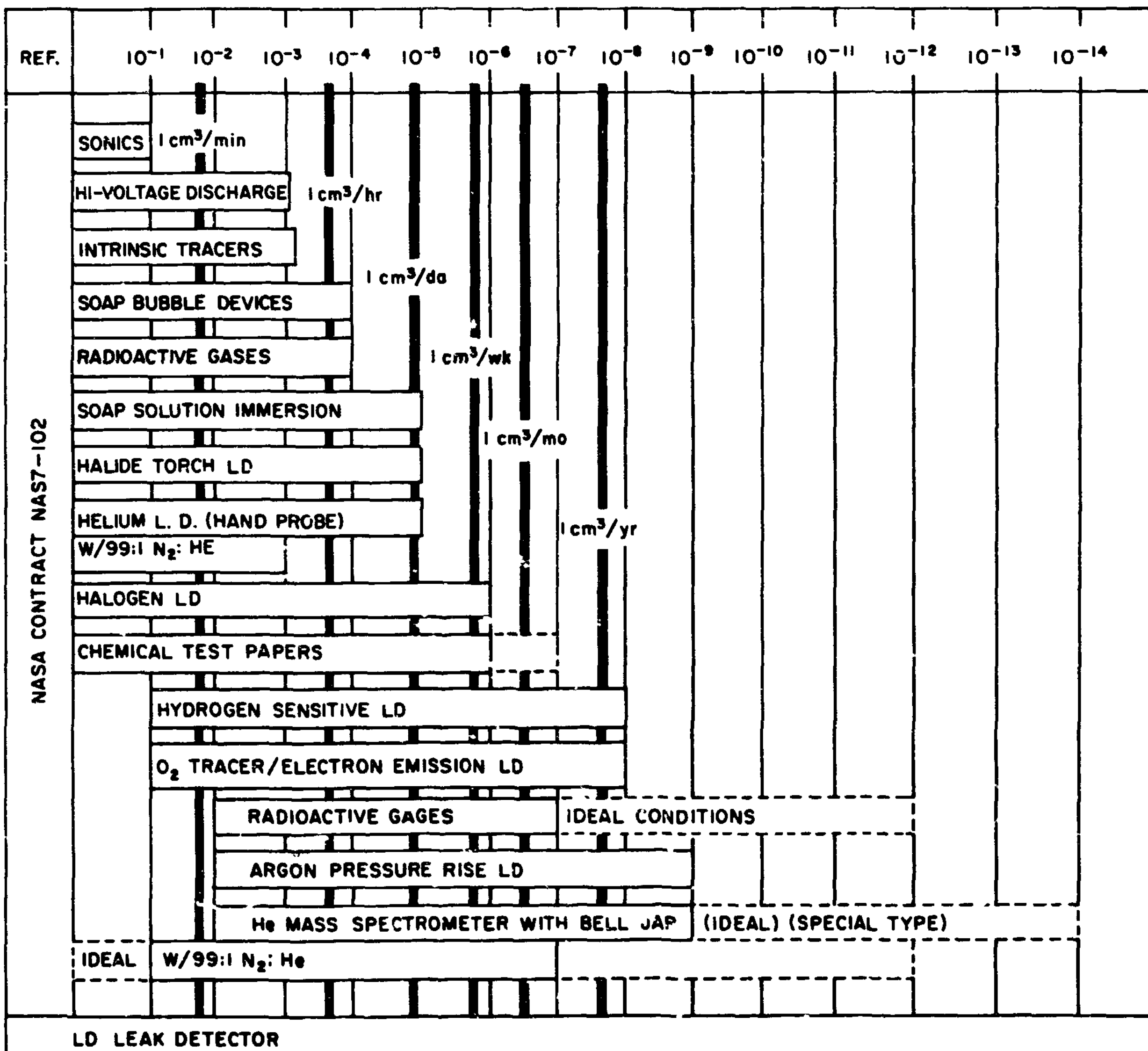


Fig. 7. Test Methods and Their Test Sensitivities

Although the third technique makes the most accurate determination, it is not easily adapted to field use.

Chemical test papers have been the most widely used indication of propellant leakage at the system level in the field. While the test papers may be as sensitive as 10^{-6} to 10^{-7} cm^3/sec, the titration of the paper proves somewhat tricky and the results are not always consistent. To date, there is no simple method to measure total liquid propellant leakage; however, some promising techniques have been observed experimentally, Ref. 8.

The importance of temperature effects should always be considered. The leak flow rate changes 1) directly with such geometry alterations as expansion and contraction of cross sections, and 2) inversely with viscosity. The change in viscosity produces the greater effect. Because it can be shown that laminar flow varies inversely with viscosity, a lower temperature requires a greater equivalent helium leak for a given liquid propellant leak. Also, for higher temperature operation, a smaller equivalent helium leak is required for a given propellant leak.

Gaseous nitrogen may be used in lieu of helium for tests where gross leaks are expected. It is possible to test for propellant leakage in terms of equivalent nitrogen leakage using the fluid flow conversion graph, e.g., Fig. 4. As with helium testing, it is still necessary to capture all of the leaking nitrogen gas, but the vacuum systems of the mass spectrometer are not necessary if the N_2 is passed up a precision-bore glass tube against a low-mass piston, where the rate of ascent equals the leak rate (less the gravitational effects). If leakage is measured in this manner, N_2 is more desirable than helium because it will not permeate through the piston material, e.g., a soap solution film, and because the laminar flow condition is viscosity dependent; therefore this may result in a 10% sensitivity increase.

In the final analysis, some form of total gas leakage measurement technique must be used for correlation with liquid leakage. In light of this, the work previously accomplished at JPL undertakes to fill the lack of such information. The numbers described in this analysis resulted from use of the fluids conversion chart as originated and copyrighted by the General Electric Company.

NOMENCLATURE

- d diameter of leak path
- f friction factor, flow passage
- K,K′ conductivity
- M molecular weight
- P pressure
- Q flow rate
- R gas constant
- T absolute temperature
- V volume
- W mass of the gas
- γ surface tension
- ζ length of leak path
- η viscosity
- ρ density
- $\propto$ proportionality

Subscripts

- 1 down stream conditions
- 2 up stream conditions
- v volume conditions

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