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MARTIN MARIETTA

MARTIN COMPANY

Contract AF04(647)-576

CR-64-80
ME #540

IMPROVED LEAK DETECTION, CORRELATION OF ACTUAL LEAKAGE WITH INSTRUMENT INDICATIONS, EFFECTS OF HUMIDITY ON LEAKS AND CATEGORIZATION OF LEAK INFORMATION - PROGRESS REPORT #2

DSR S 10441
31 March 1964

Authors

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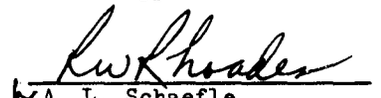
Approved



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Approval Number 21-R138.1

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FOREWORD

This document is submitted under paragraph 3.14.2 of Contractor's Data Specification BMS-TII-CD-71,000 (Rev. 2), as incorporated in Contract AF04(647)-576.

This is Progress Report #2 to DSR S 10441.

SUMMARY

The following reports on "Propellant Ullage Decay" (ME #495), and "Use of a Flow Conversion Nomograph for Leaks and Capillaries" (ME #484) are submitted as Progress Report #2 to DSR 10441.

The primary purpose of the ullage decay study was to determine a relationship between the helium leak rates and the actual ullage gas leak rates. The results of this study showed that the propellant gases flow at a rate equal to, or slower than, helium. In many instances the escaping ullage gas (particularly in the case of nitrogen tetroxide) actually condensed in the leak path and eliminated any further gas leakage.

The nomograph (ME #484) is submitted for information on flow rate conversions. By use of the nomograph the flow rate of any gas, through any given path, can be converted to the flow rate of any liquid at any pressure through the same path. The nomograph was developed theoretically; however, the gas portion has been proven experimentally.

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MATERIALS ENGINEERING SECTION

TEST REPORT

ME NO: 495 31 January 1964

TITLE: Improved Leak Detection, Correlation of Actual Leakage with Instrument Indications, Effects of Humidity on Leaks and Categorization of Leak Information.

Status Report #2 - DSR 11041

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INTRODUCTION

The main objective of this study was to experimentally establish a relationship between helium leakage and propellant vapor leakage in the ullage area of Titan II tanks. The contractual requirement is to lose no more than 2 psi per month (which would result in not being in a state of readiness). Therefore, it was necessary to verify that the ullage gas would leak at a slower rate than the helium leak rates as predicted by theoretical calculations.

The conductance of the leak paths were selected to study the flow rates of the various gases. The conductance combines two important variables (hole geometry and fluid properties) and therefore monitors the flow change. Since the mass spectrometer is used only for helium flow rates, both the helium leak rates and the propellant gas leakage rates were measured by the pressure decay method. The decay tests were performed on weld type leaks, conoseal leaks, and leaks in bolted flanges using fuel ullage gas or oxidizer ullage gas.

SUMMARY

The pressure decay tests performed on leaking welds showed that the paths partially plug in very short times when either A-50 or N_2O_4 vapor is present. In the case of N_2O_4 the conductance through leak paths is reduced by 70 - 85 percent within eleven hours. For fuel there was a gradual decrease in conductance for 60 hours until the leak path geometry stabilized at twenty-five percent closed. The closing of the leak path represents a decrease in the hole diameter when the holes (leaks) are assumed to be cylindrical smooth bare capillaries.

Two conoseals were tested, one with A-50, the other with N_2O_4. With N_2O_4 vapor in the test chamber the leak path geometry was stable, with conductance reduced by a factor of ten. The conoseal tested with A-50 vapor partially plugged, and again the conductance was reduced to one tenth of its initial value.

One bolted flange was tested to determine a relationship between the sampling probe reading and the actual leak rate.

TEST PROCEDURE

1. A known leak (conoseal, weld or bolted flange) was installed in the test pot and pressurized to two atmospheres with the test gas mixture (10% He + 90% N_2) for a determination of the rate using the helium mass spectrometer.

2. The specimen was then pressurized to 50 psig to determine the leak rate by pressure decay. The decay was monitored using a pressure transducer installed in the pot (see Figure 1).

3. Pressure readings were taken until sufficient information was obtained to calculate the path conductance.

4. A small amount of propellant was introduced in the chamber (insuring that the liquid did not physically contact the defect). The chamber was then repressurized and the pressure decay was monitored.

The CEC probe leak rate values for weld type leaks were determined with a vinyl tube on the end of the probe. (These measurements were taken before the flexible rubber tip was being used). The vacuum conductance values were not always obtainable due to the size of the leak. The vacuum leak measured leak rates were determined as described in Status Report #1.

To determine the leak rates for the conoseals, the conoseal was "bagged" with polyethylene and then the probe was inserted into the bag and and monitored until a constant reading was obtained. The probe leak rate for the bolted flange was obtained by summing the probe readings around the diameter and taking the average. The total leakage predicted by the probe was obtained as follows:

$$Q_{actual} = \frac{Q_{probe} (\pi \times D_{flange})}{D_{probe}}$$

Since the pressure transducer measures total gas loss and the mass spectrometer measures only helium loss, the helium concentration was corrected from 10% to 100%,

RESULTS

The results are based on the assumption that the pressure decay follows the equation:

$$P = P_o e^{-Kt} \quad (1)$$

Where:
- P = Pressure at time (t) psig
- P_o = Pressure at time (t = o) psig
- t = Time
- K = F/V = hole conductance/pot volume, (time^{-1})

This equation implies that the conductance (F) is independent of pressure. There are limits beyond which this assumption is no longer valid; however, equation (1) is valid for leaks with conductance values $< 10^{-3}$ cc/sec., and can be applied for conductance values of 10^{-2} cc/sec. For very low conductance values, $< 10^{-5}$ cc/sec. test times become prohibitively large, and temperature variations in the laboratory make test results difficult to interpret.

The results of the pressure-time readings are plotted as $\ln (P_o/P) = Kt$ (see Figure 2). The conductance (f) can be readily calculated by multiplying the slope of the curve (K) by the pot volume (V). The leak rate (Q) can also be calculated from the following relationship:

$$Q = F(\Delta P) \quad (2)$$

Where:
- Q = Leak rate (atm-cc/sec)
- F = Leak conductance (cc/sec)
- P = Pressure drop across leak (atm)

Leak conductance values calculated from the decay tests can be used to calculate a reduction in "hole diameter" by using the following equation:

$$F = \frac{65D^3}{L\sqrt{M}} \quad (3)$$

Where:
- F = Leak conductance (cc/sec)
- D = Hole diameter (cm)
- L = Hole length (cm)
- M = Molecular weight of gas present

The reduction in "hole diameter" is possibly caused by the reaction of the N_2O_4 or A-50 with the walls of the "capillary". Also the "hole" size is reduced due to the pressure drop, which caused a condensation of vapors on the low pressure side of the leak path (as was visually observed in the weld defect specimens).

Equation (3) was derived for circular holes, and therefore the results are not exact since the actual leak path is not a smooth bore cylindrical capillary. In addition to the non-circular cross-section, the average molecular weight of the gas will be altered by the addition of the propellants. This effect is slight because of the small amounts of propellants added.

By taking a ratio of conductances before and after hole reduction occurs, the diameter change can be calculated:

$$\left(\frac{F_2}{F_1}\right)^{1/3} = \frac{D_{(2)}}{D_{(1)}} \qquad (4)$$

$$100\left[1 - \left(\frac{F_2}{F_1}\right)^{1/3}\right] = \left[1 - \frac{D_2}{D_1}\right] 100 \qquad (5)$$

The percent change is given by equation (5), where D_2, F_2 are reduced hole diameter and conductance, respectively; D_1 and F_2 are original hole diameter and conductance respectively. The calculated change does give a quantitative value for the effect of propellant vapor on the hole geometry.

Table 1 summarizes the results of the pressure decay tests. Probe leak rate values are shown for reference only. Figures (2) through (5) show pressure decay curves and the effect of N_2O_4 and A-50. Figure 7 represents supplementary data for the probe-vacuum relationship presented in Status Report #1. This plot was created from two separate sets of data taken from the same weld type leak path. Leak rates were calculated from the decay test using equation (2) for pot pressures up to 50 psig. Also leak rates were determined from CEC probe readings (using the flexible rubber tip) at pot pressures from 10 psig to 50 psig.

For a perfect correlation between the actual leak rate (pressure decay) and that determined by the probe method, the slope should be equal to unity. This does not occur and the scatter in probe leak rate values increase proportionally with the pot pressure.

To determine a more accurate relationship between the actual leak rate and that determined from the probe readings many different leaks must be tested as was the one for Figure 9. A statistical approach must then be used to determine the relationship and corresponding level of confidence.

CONCLUSIONS

1. The ullage gases leak at a lower rate than the test gas for the weld type leaks.

   a. The fuel ullage gas leaked at a lower rate than the test gas by a factor of 2.5, due to the changes in hole geometry.

   b. The oxidizer ullage gases leaked at the same rate as the test gas until the hole closed, due to condensation of the N_2O_4 vapors.

2. The ullage gases leaked less than the test gas mixture, approximately by a factor of ten for leaks created in conoseals.

3. The bolted flange decayed only a factor of ten more than predicted. This factor would not be applicable to a field inspection because the probe tip cannot always be "on top" of the leak

4. The sniffing probe is only applicable as a qualitative check.

TABLE 1

PRESSURE DECAY TEST RESULTS

Leak Type	Test Gas	Decay Conductance	Probe Leak Rate	Vacuum Conductance	Percent Leak Dia. Change
Welds	10% He - 90% N_2	3.34×10^{-3} cc/sec	8×10^{-8} atm cc/sec	Off scale	---
	10% He - 90% N_2 + A-50	1.40×10^{-3} cc/sec	---	---	-25
	10% He - 90% N_2	5.67×10^{-3} cc/sec	2.7×10^{-10} atm cc/sec	Off scale	
	10% He - 90% N_2 + N_2O_4	1.28×10^{-4} cc/sec	---	---	-72
	10% He - 90% N_2	1.01×10^{-3} cc/sec	1.13×10^{-6} atm cc/sec	Off scale	
	10% He - 90% N_2 + N_2O_4	4.48×10^{-6} cc/sec	---	---	-84
Conoseals	10% He - 90% N_2	2.76×10^{-3} cc/sec	2.38×10^{-6} atm cc/sec	---	---
	10% He - 90% N_2 + A-50	2.57×10^{-4} cc/sec	---	---	---
	10% He - 90% N_2	3.18×10^{-3} cc/sec	2.74×10^{-6} atm cc/sec	---	---
	10% He - 90% N_2 + N_2O_4	3.72×10^{-4} cc/sec	---	---	---
Bolted Flange	10% He - 90% N_2	0.02 cc/sec	3×10^{-3} atm cc/sec	---	---

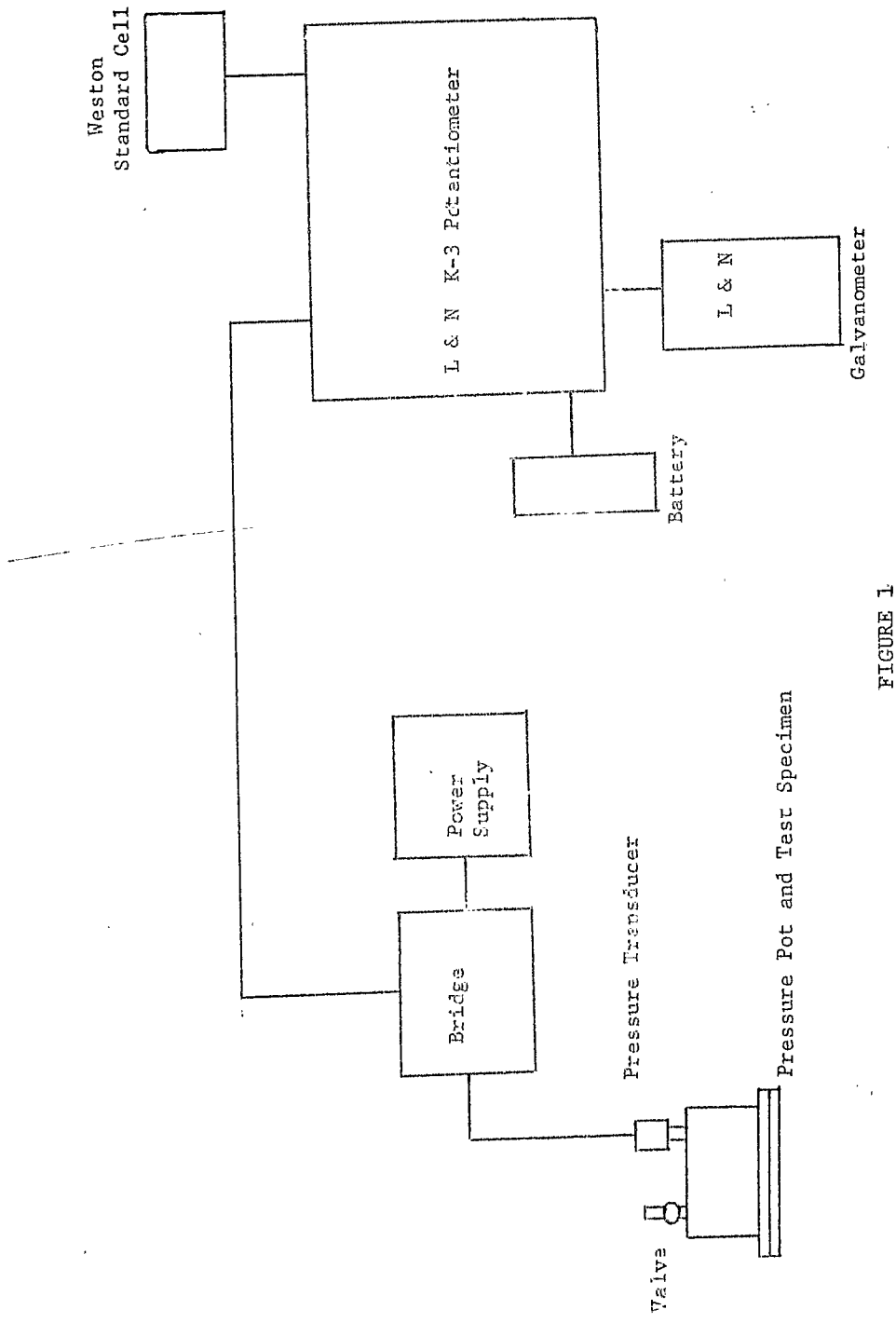

FIGURE 1

PRESSURE DECAY TEST EQUIPMENT

Schematic

K&E 10 X 10 TO THE CM. KEUFFEL & ESSER CO. ALBANENE 359T-14 MADE IN U.S.A.

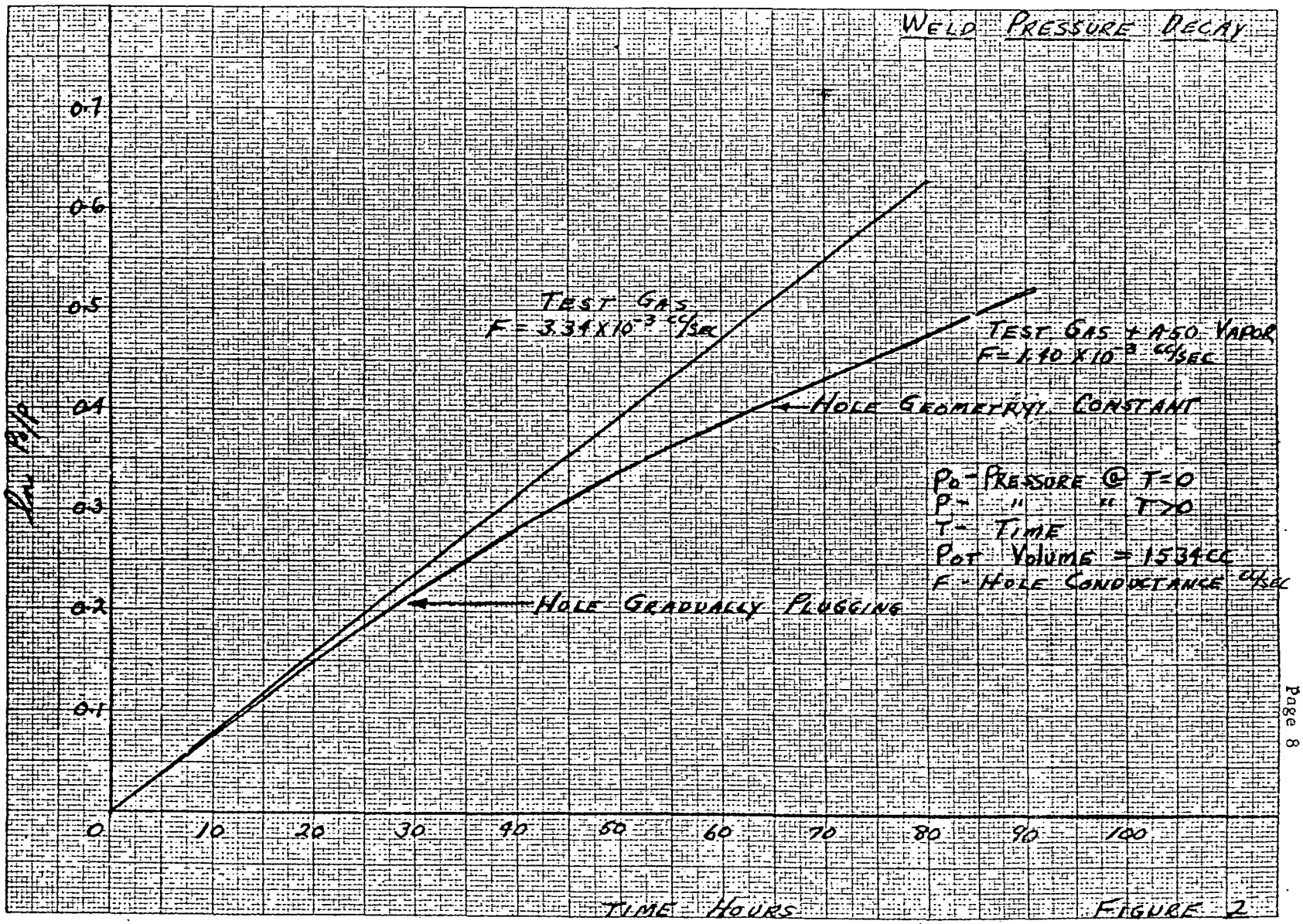

FIGURE 2

K&E 10 X 10 TO THE CM. KEUFFEL & ESSER CO. ALBANENE 359T-14 MADE IN U.S.A.

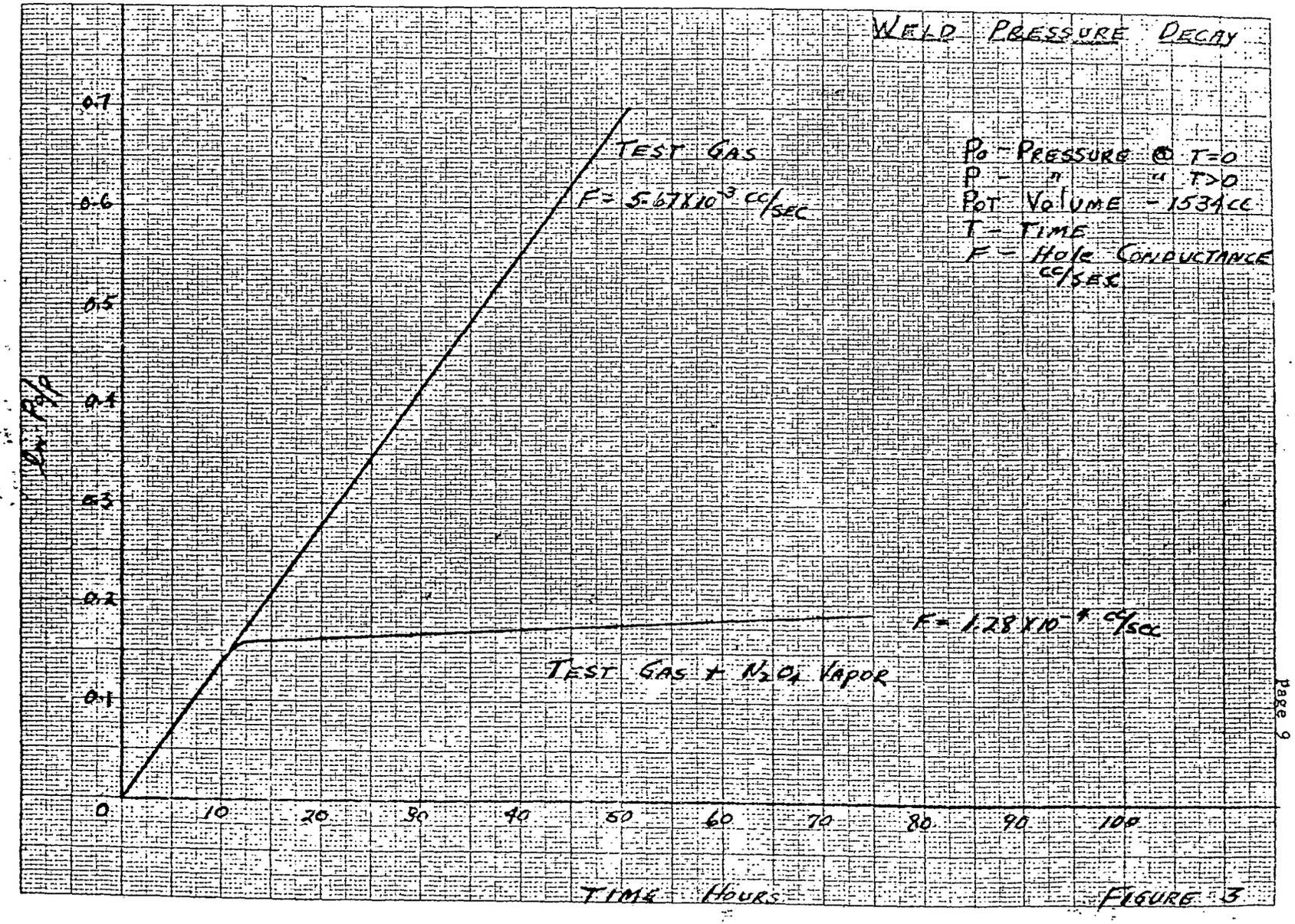

FIGURE 3

K·E 10 X 10 TO THE CM. KEUFFEL & ESSER CO. ALBANENE 359T-14 MADE IN U.S.A.

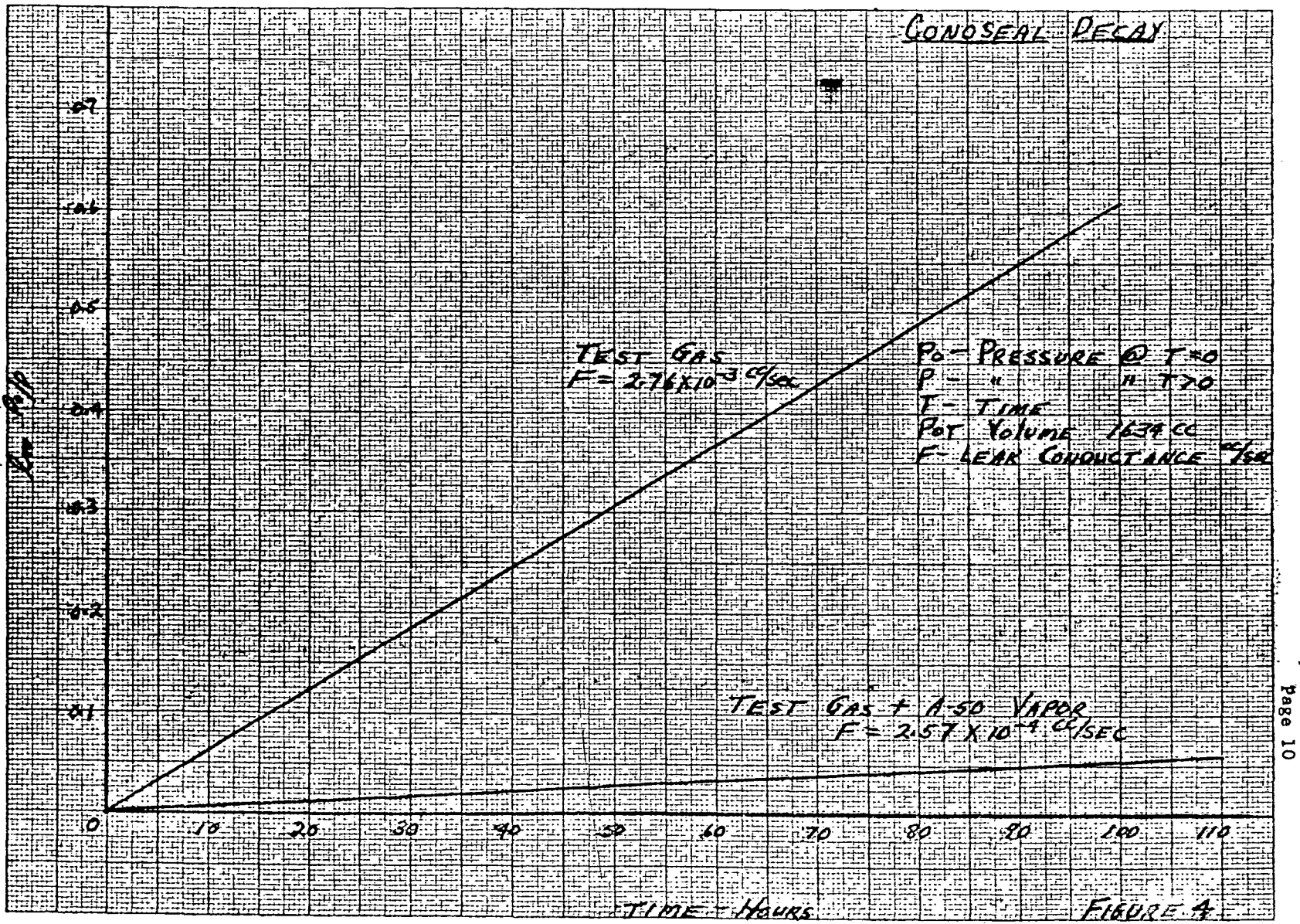

FIGURE 4

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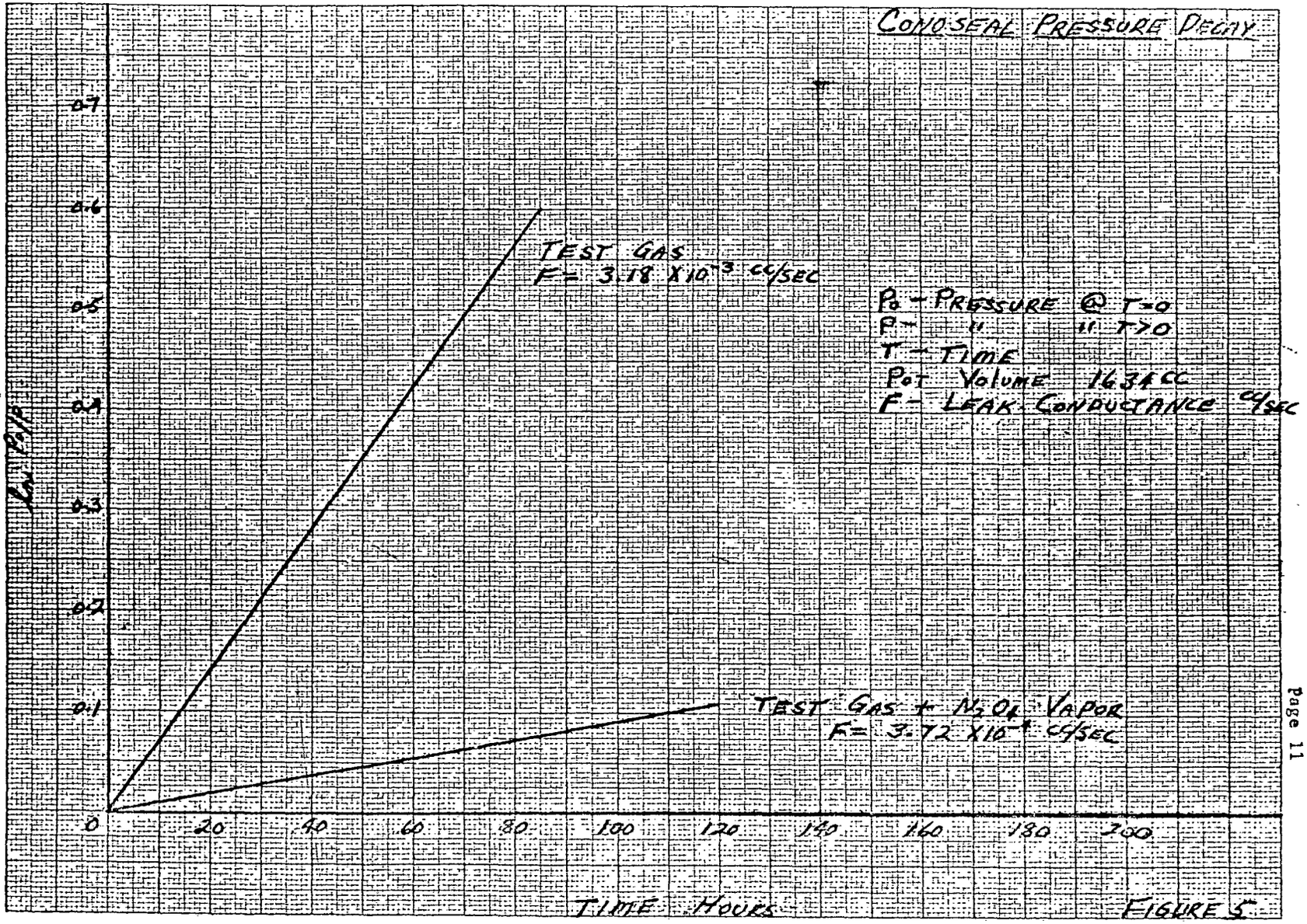

FIGURE 5

K·E 10 X 10 TO THE CM. KEUFFEL & ESSER CO. ALBANENE 359T-14 MADE IN U.S.A.

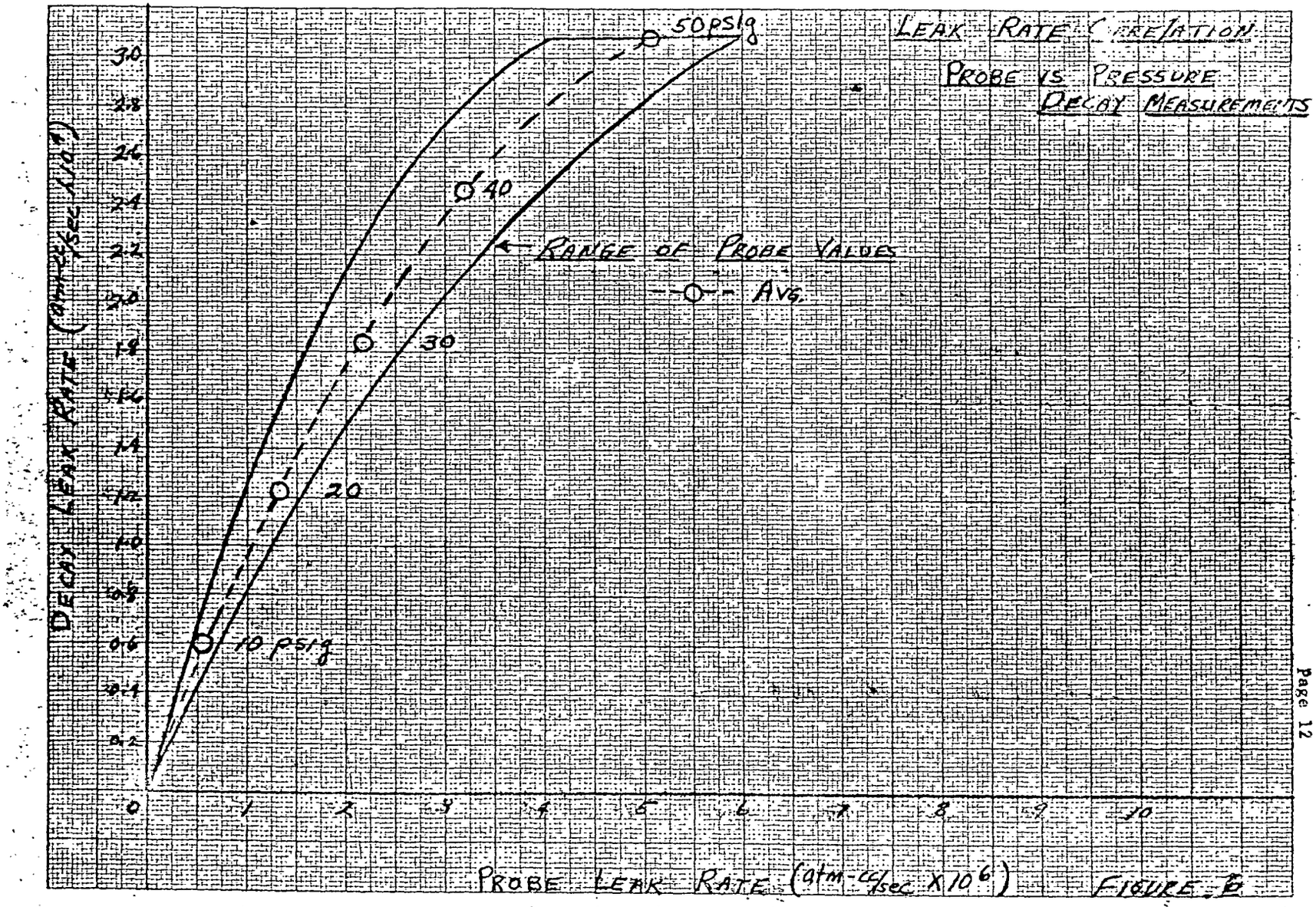

FIGURE 8

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MATERIALS ENGINEERING SECTION

TEST REPORT

ME. NO: 484 23 January 1964

TITLE: Use of a Flow Conversion Nomograph for Leaks and Capillaries

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L. Wilkens	C. Maze
C. Russell	R. Foll
D. Schwartz	C. Brown
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REFERENCES:

(a) DSR #10441

(b) Handbook of Chemistry and Physics, 33rd Edition

(c) "Computation of Leak Rate of Fluids and Gases Through Capillaries", Robert L. Douglas, Parker Pen Company, 20 November 1963

(d) National Symposium on Vacuum Technology, 1956 Transactions, D. J. Santeler and T. W. Moller, General Electric Company

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SUMMARY

The nomograph included in this report can be utilized to theoretically determine the flow rate of any liquid or gas through a leak or capillary with any pressure differential impressed upon it provided a pressure differential and corresponding flow rate for any other gas or liquid are known. A correction for flow in the molecular regime is included.

DESCRIPTION AND EXAMPLES

In the nomograph, ΔP is plotted as the abscissa, and the gas and liquid flow rates are plotted as the right and left hand ordinates respectively. Liquid and gaseous viscosity are also plotted to permit the use of any liquid or gaseous fluid. The horizontal guide lines are positioned so as to be characteristic of the particular flow equation from which the constant factor has been taken. The curved lines and lines with slopes of one and two are characteristic of the pressure dependency in each of three flow equations.

The various manipulations necessary for the proper use of this graph can best be shown by some sample solutions.

Example 1. Use of the graph without taking molecular flow correction into account. (Gas to Gas)

Suppose a given leak permits a flow rate of 1×10^{-6} atm. cc/sec. of helium when tested from 5 to 1 atmospheres. It is desired to determine the flow rate of nitrogen gas when tested from 1/10 atmosphere (gauge) to one atmosphere (absolute).

Solution: The nomograph is entered on the ordinate labeled "gas flow" at the value 1×10^{-6} atm. cc/sec. Move horizontally to the left to the point corresponding to the viscosity of helium (μ = 0.0194 centipoises at 20° C). Upon reaching this point, move upward and to the left on the sloped pressure guideline (interpolate if necessary) to $\Delta P = 4.0$. Move horizontally to $\Delta P = 0.1$ and back down and to the right on a new pressure guideline to the viscosity of N_2 (μ = 0.0178 centipoises at 27.4° C). Read the corresponding flow rate (approximately 2.0×10^{-8} atm. cc/sec.) on the gas flow ordinate.

Example 2 Use of the graph without taking molecular flow correction into account (Gas to Liquid).

Using the same "known" flow rate as in Example 1, it is desired to find the flow rate of liquid Nitrogen Tetroxide (N_2O_4) at a pressure differential as in Example 1 (from 1/10 atmosphere gauge to one atmosphere absolute).

Solution: The same procedure as in Example 1 is followed except that instead of moving down along the pressure guideline, move up and to the left on a 45° slope to the point at which the viscosity equals that of N_2O_4 (μ = 0.442 centipoises at 60° F). The flow rate is then read on the liquid flow rate ordinate (approximately 4.0×10^{-5} cc/day or 4.65×10^{-10} cc/sec).

Note: A straight line with a slope of two rather than the curve is followed for gases whenever the pressure differential is from a positive value to a vacuum.

Example 3 Use of the graph while taking molecular flow into account (Gas to Gas)

The family of curved lines on the lower right margin has as a parameter the expression $1P^2$ where 1 is the length of the capillary in centimeters and P is the inlet pressure for the test condition in atmospheres. This particular family of lines has been drawn for helium. Families of lines for other gases have been omitted for the sake of clarity.

The correction for molecular flow only becomes necessary for relatively small leaks and small pressure differentials.

For a case in which a molecular flow correction is necessary, the solution is accomplished in the same manner as in Examples 1 and 2 except that an assumed capillary length must be multiplied by the square of the inlet pressure. This factor ($1P^2$) is then used as the new location of the viscosity stopping point after moving horizontally from the given flow rate. The correction factor is also used for the reverse process, i. e. used as a stopping point when moving down the pressure guide line toward the unknown flow rate.

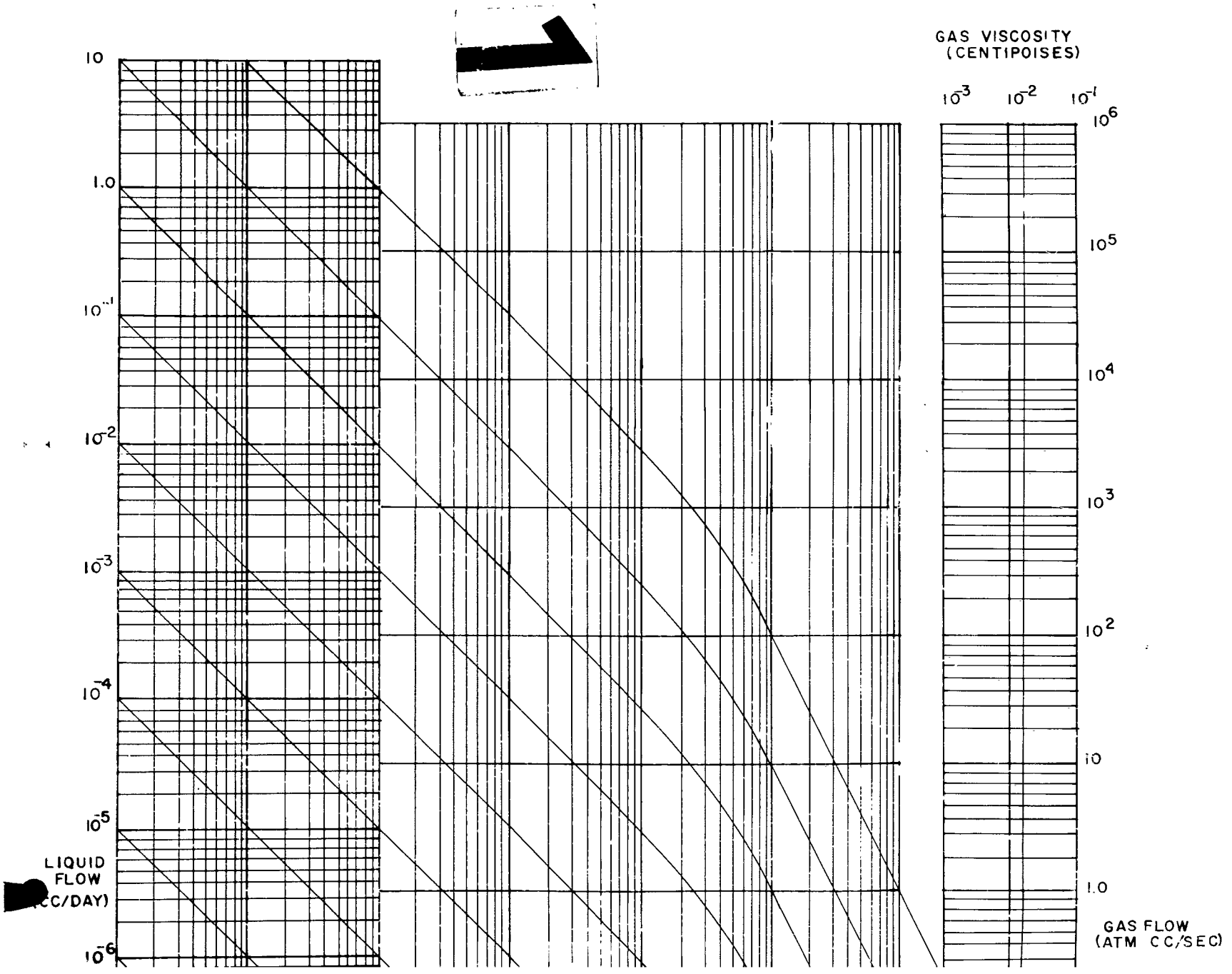
Note: A slight layout error has been found to exist in the nomograph. This, in turn, creates a slight error in calculations. Depending upon the type of calculation desired, this error can be as great as a factor of ~ 2 and as small as ~ 1.2.

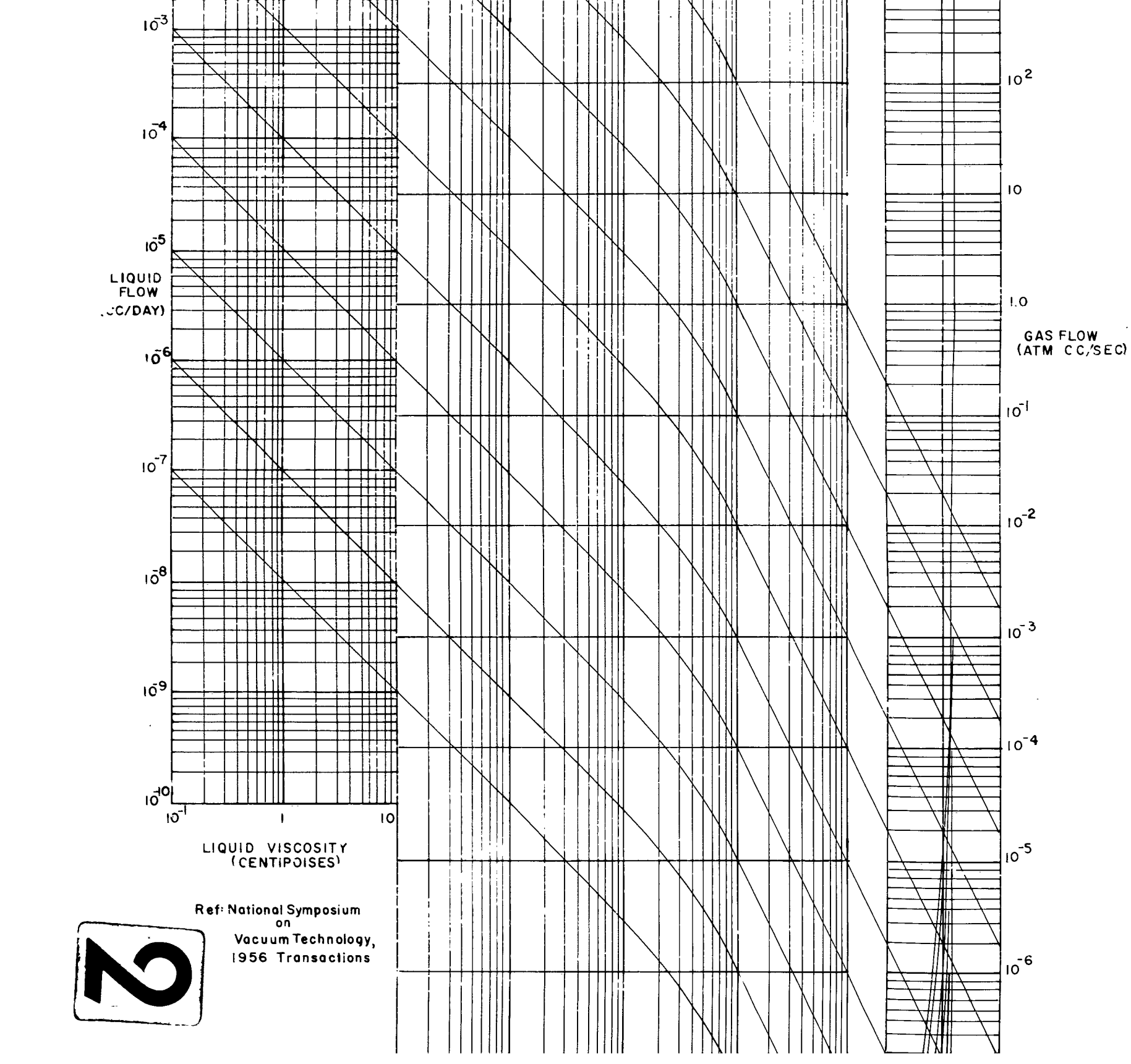
Some useful constants are given below:

Gas	Temperature	Viscosity (Centipoises)
Air	18° C	0.01827
Air	40° C	0.01958
Air	74° C	0.02102
Helium	20° C	0.01941
Helium	100° C	0.02281
Hydrogen	20.7° C	0.00876
Hydrogen	129.4° C	0.01086
Nitrogen	27.4° C	0.01781
Nitrogen	127.4° C	0.02191
Oxygen	19.1° C	0.02018
Oxygen	127.7° C	0.02568
Water Vapor	100° C	0.01255
Oxidizer Ullage Gas	60° F	0.01460
Fuel Ullage Gas	60° F	0.01710
Liquid		
Nitrogen Tetroxide	60° F	0.442
50% N_2H_4 - 50% UDMH	60° F	0.943
Water	60° F	1.120
Nitric Acid	10° C	0.776

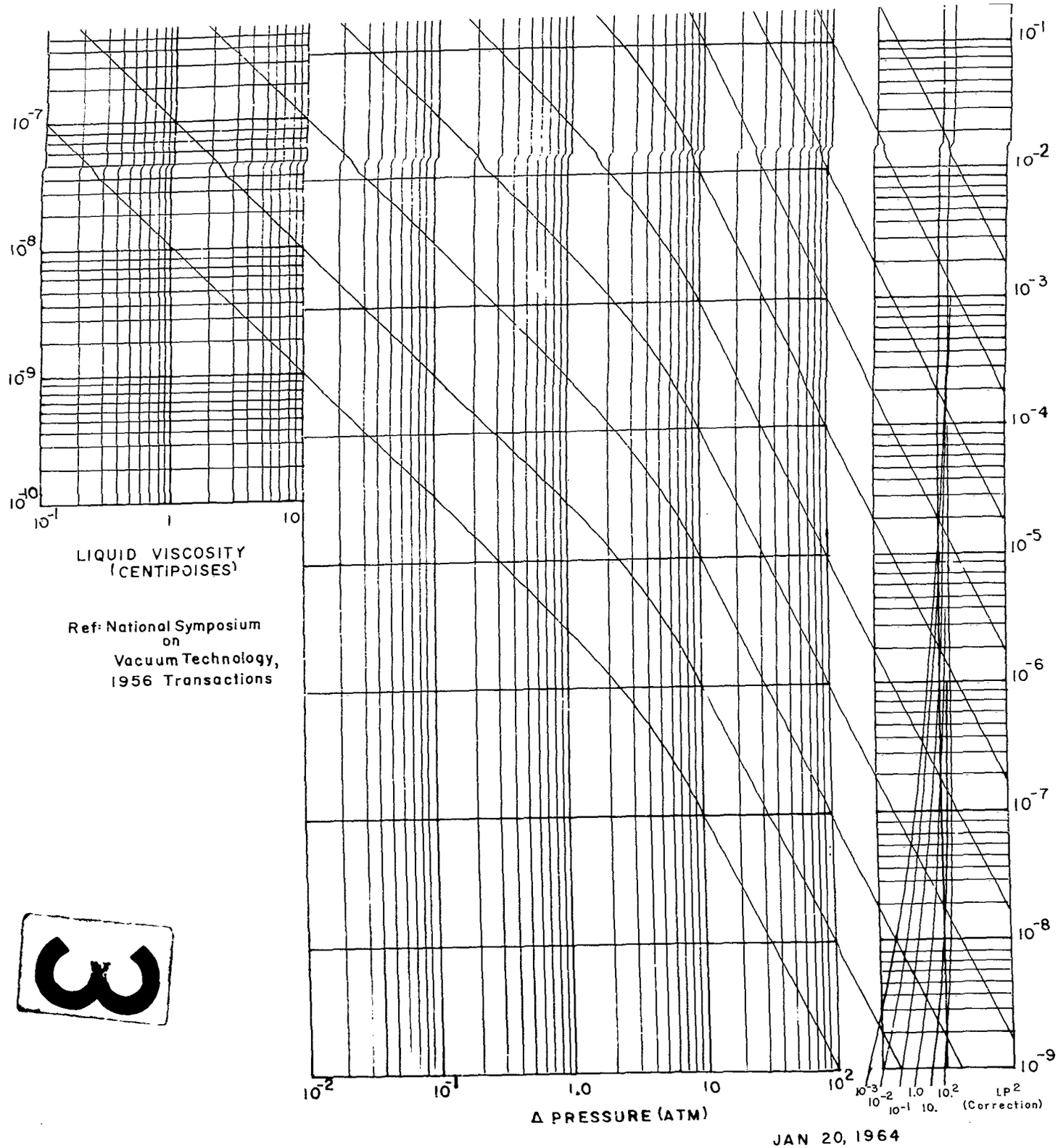
1 cc/day = 1.08×10^{-5} cc/sec.

FLUID FLOW CONVERSION NOMOGRAPH





Ref: National Symposium on Vacuum Technology, 1956 Transactions



Ref: National Symposium on Vacuum Technology, 1956 Transactions

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