Designation: F 78 – 97 (Reapproved 2002)

Standard Test Method for Calibration of Helium Leak Detectors by Use of Secondary Standards[1]

This standard is issued under the fixed designation F 78; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for calibrating a mass spectrometer-type helium leak detector with a series of commercially available calibrated leaks without need for recourse to a primary standard.

1.2 Leak detector parameters determined by this test method include:

1.2.1 Minimum detectable signal, drift noise (8.5, with recorder; 8.6, without recorder),

1.2.2 Response time,

1.2.3 Minimum detectable leak rate, and

1.2.4 Sensitivity.

1.3 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 1 Specification for ASTM Thermometers [2]

E 425 Definitions of Terms Relating to Leak Testing [3]

3. Terminology

3.1 *Definitions:*

3.1.1 *calibrated leak—in leak detection*, a device that permits leakage through it at a specified rate, of a specific gas, under specific conditions, with the downstream side of the device exposed to a pressure sufficiently low to have negligible effect on the leak rate.

3.1.2 *minimum detectable signal—in leak detection*, the smallest unambiguous output signal that can be derived from a given particular leak detector. Units are detector scale divisions.

3.1.2.1 *Discussion*—The minimum detectable signal is determined by the noise present in, and drift of, the output signal.

3.1.3 *standard leak rate—in leak detection*, the rate of flow of atmospheric air of dewpoint less than − 25°C through a leak under standard conditions specified as follows: (*1*) the inlet pressure shall be 1 standard atmosphere ±5 % (101± 5 kPa), (*2*) the outlet pressure shall be less than 1 kPa (0.01 atm), and (*3*) the temperature shall be 23 ± 3°C.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *response time—of a leak detector, for the purposes of this test method*, a measure of the speed of response of the detector to an incoming helium sample.

3.2.1.1 *Discussion*—In this test method the cleanup time and response time are assumed to be equal.

3.2.2 *sensitivity—of a leak detector, for the purposes of this test method*, the ratio of the change in the output signal to the applied helium leak rate.

3.3 Other terms used in this test method are defined in Definitions E 425.

4. Summary of Test Method

4.1 At least three calibrated leaks are tested on a helium leak detector, and a correlation is obtained between the output indication of the leak detector and the leak rate of the calibrated leaks. These readings are used to plot a calibration line from which intermediate values, within specified limits, may be read.

5. Interferences

5.1 Certain materials, particularly organic compounds, will entrap or hold helium tracer gas. Use of such materials in connections between the calibrated leak and the leak should be minimized to avoid erroneous results. (If the net output readings from any calibrated leak consistently lie outside the established limits, the leak should be returned to the supplier for a recalibration check.)

5.2 The background reading, *B*, should be at most one quarter of the output reading, *A*. If the value of *B* approaches that of *A*, the accuracy of the determination of *N* will suffer (see 9.3.1).

6. Apparatus

6.1 *Calibrated Leaks*—At least three commercial devices incorporating leaks, one having a leak rate of approximately 10^{-9} atm·cm^3·s^{-1}(10^{-10} Pa·m^3·s^{-1}), a second having a leak

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[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.03 on Metallic Materials.

Current edition approved Dec. 10, 2002. Published May 2003. Originally approved in 1967. Last previous edition approved in 1997 as F 78 – 97.

[2] *Annual Book of ASTM Standards*, Vol 14.03.

[3] Discontinued. See 1991 *Annual Book of ASTM Standards*, Vol 03.03.

value in the nominal range from 10^{-8} to 10^{-7}atm·cm^3·s^{-1}(10^{-9} to 10^{-8} Pa·m^3·s^{-1}), and the third having a leak rate of approximately 10^{-6} atm·cm^3·s^{-1}(10^{-7} Pa·m^3·s^{-1}).

6.1.1 The calibrated leaks shall be obtained from at least two independent suppliers.

6.1.2 The calibrated leaks shall have been calibrated with helium gas at a pressure of approximately 1 standard atmosphere ±5 % (101 ± 5 kPa).

6.1.3 The following information shall be provided with each calibrated leak:

6.1.3.1 Calibrated leak rate, atm·cm^3·s^{-1}(or Pa·m^3·s^{-1}),

6.1.3.2 Date of calibration,

6.1.3.3 Temperature of calibration, °C,

6.1.3.4 Temperature coefficient, atm·cm^3·s^{-1}·°C^{-1}(or Pa·m^3·s^{-1}·°C^{-1}), and

6.1.3.5 If a reservoir is an integral part of the calibrated leak, the internal pressure in the reservoir, atm (or Pa) and an aging correction.

NOTE 1—It is preferable that five, rather than three, calibrated leaks be used for initial calibrations of helium leak detectors by this method. At least two leaks shall be obtained from each of two independent suppliers when more than three leaks are used.

NOTE 2—Although the data on which this specification has been based were obtained largely from permeation-type leaks, the calibrated leaks may be of various types such as capillary, pinched tubing, tapered plug, etc. However, it is recommended that, with all types of leaks, the manufacturer's recommendations be followed to avoid erroneous test results.

6.2 *Thermometer*, accurate to ±1°C or better in the range from 18 to 28°C inclusive. A thermometer conforming to Thermometer 63C as prescribed in Specification E 1 is suitable.

6.3 *Chart Recorder*, for determining Minimum Detectable Leak, Method A; an instrument suitable for recording the output of the leak detector under test as a function of time.

6.3.1 The chart recorder shall incorporate a gain control to permit the deflection of the recorder stylus to be adjusted to full scale when the leak detector meter is reading full scale with the leak detector at its most sensitive detection setting.

6.3.2 The time constant of the chart recorder shall not be greater than that of the leak-detector output meter.

6.3.3 The chart recorder shall be capable of continuous recording for at least 1 h.

6.4 *Stopwatch*, calibrated to read in tenths of a second to 60 min over an interval of at least 1 h.

6.5 *Leak Auxiliary Manifold*—If not incorporated in the leak detector, evacuable means for connecting the calibrated leak to the leak detector, incorporating a roughing pump, leak valve, and pump valve (see Fig. 1).

6.5.1 The roughing pump shall have sufficient pumping capacity to evacuate the leak auxiliary manifold to an absolute pressure of less than 50 millitorr (or 7 Pa).

6.5.2 The leak valve shall not act as a source of helium.

6.5.3 Valves and connections shall contain a minimum of rubber or other polymeric surfaces that can serve as virtual leaks.

NOTE 3—It is preferable that the only exposed polymeric surfaces be those of O-rings.

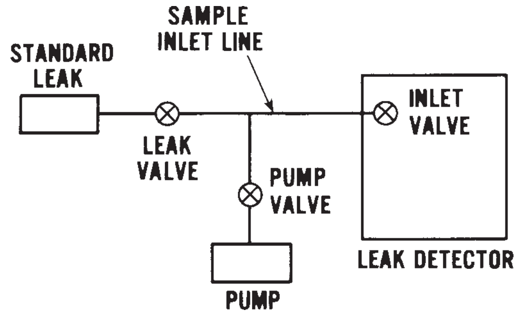

FIG. 1 Schematic Diagram of Apparatus for the Calibration of the Helium Mass Spectrometer Leak Detector

7. Material

7.1 *Helium Gas*— for use with calibrated leaks not having an integral reservoir. The helium gas shall have a purity of at least 99.9 % and a supply pressure of nominally 1 atm (101 kPa). [4]

8. Procedure

8.1 Connect the helium leak detector to be tested to a source of electric power conforming in voltage, frequency, and degree of regulation to the manufacturer's specifications.

8.2 Turn on the detector for the warm-up period specified by the manufacturer.

8.3 Adjust the detector in accordance with the manufacturer's instructions for maximum sensitivity and for maximum output for a given helium input.

8.4 Close the inlet valve of the detector.

8.5 If the electron-producing filament is not on, turn it on and adjust the detector zero position control to obtain an output signal of at least 10 % of the most sensitive scale.

8.6 If the detector has no recorder output or if a suitable chart recorder is not available, continue with 8.8; otherwise, continue with 8.7.

8.7 *Minimum Detectable Signal, Test Method A, with Recorder*:

8.7.1 Connect the detector output to the recorder.

8.7.2 Record the detector output for 60 min or until the output indication has reached full scale. Do not readjust any controls during the recording period.

8.8 *Minimum Detectable Signal, Test Method B, without Recorder*:

8.8.1 Observe the detector meter and record its indications as follows. Do not readjust the controls for the 60-min period of this test.

8.8.1.1 Record the pointer deflection, in scale divisions, at time $T = 0$ min.

8.8.1.2 Record the minimum and maximum pointer deflections occurring in the interval from time $T = 0$ to $T = 1$ min.

8.8.1.3 Record the minimum and maximum pointer deflections occurring in the interval from time $T = 9$ to $T = 10$ min; record the deflection occurring at $T = 10$ min.

[4] Commercially available compressed helium of the specified minimum purity, supplied in suitable cylinders with appropriate regulators, has been found suitable for this test method.

8.8.1.4 In like manner, record the minimum, maximum, and terminal deflections occurring during every tenth minute for the 60-min period, that is, from $T = 19$ to $T = 20$ min, $T = 29$ to $T = 30$ min, etc.

8.9 *Minimum Detectable Leak Rate and Sensitivity*:

8.9.1 Connect the apparatus as shown in Fig. 1, including one of the calibrated leaks.

8.9.2 With the filament on, zero the deflector meter reading.

8.9.3 Open the leak valve and then the pump valve.

8.9.4 If the calibrated leak has an integral reservoir, continue with 8.9.6; otherwise continue with 8.9.5.

8.9.5 Connect a source of helium at a pressure of 1 atm (101 kPa) to the calibrated leak.

8.9.6 Evacuate the atmospheric air present in the connections between the leak and the leak detector (to protect the leak detector).

NOTE 4—It may be desirable to turn off the filament of the mass spectrometer tube before continuing with 8.9.7.

8.9.7 Open the inlet valve slowly and maintain the leak detector pressure within the operational pressure range specified by the manufacturer.

8.9.8 Close the pump valve.

8.9.9 With the inlet valve fully open, observe the pressure indicator of the leak detector. Do not continue until this reading shows no observable change over 1 min.

8.9.10 Turn on the filament of the mass-spectrometer tube if it is not on.

8.9.11 Adjust the range multiplier to bring the detector meter reading on scale. When the meter pointer shows a steady deflection, with no observable change over 1 min, record the reading, *A*, in scale divisions. If required, adjust the gain control, but do not readjust any controls thereafter for the duration of this test.

8.9.12 Calculate and record a value equal to 37 % of *A*.

8.9.13 Using the thermometer, measure the ambient temperature near the leak to the nearest 1°C. Record this value.

8.9.14 Start the stopwatch and simultaneously close the leak valve *as rapidly as possible*.

8.9.15 Observe the detector meter continuously. Stop the stopwatch when the reading has decreased to 37 % of *A* (this value was recorded in 8.9.12). Record the reading of the stopwatch to the nearest 1 s as *T*, the response time.

NOTE 5—The actual value recorded is the cleanup time, which for the purposes of this method is taken as the reponse time.

8.9.16 Continue to observe the detector meter. When the pointer shows a steady deflection, with no observable change over 1 min, record the reading in scale divisions as the background reading, *B*.

8.9.17 Close the inlet valve, vent the sample inlet line to atmosphere, and replace the calibrated leak with another.

8.9.18 Repeat steps 8.9.1 through 8.9.11, 8.9.13, and 8.9.14 through 8.9.17 until each of the remaining calibrated leaks has been run.

9. Calculations

9.1 *Minimum Detectable Signal, Test Method A*:

9.1.1 If spikes appear in the chart recorder trace, construct a smooth curve that represents the average values of the detector output.

9.1.2 From the smoothed curve, determine the detector output at the beginning and at the end of each minute, in chart scale divisions. Record these values.

9.1.3 Calculate and record the change in output for each 1-min period. Compare each of these values to a reference value of ½ % of the full-scale chart reading.

9.1.3.1 If the change in output per minute is always greater than or equal to this reference value, identify by inspection the largest of these changes and record this value, in chart scale divisions per minute, as the drift.

9.1.3.2 If the change in output per minute is always less than this reference value, calculate the total change in the 60-min observation period and divide this value by 60. Record the quotient in chart scale divisions per minute as the drift.

9.1.4 Examine the recorded output curve and determine if spikes appear on both sides of the smoothed curve.

9.1.4.1 If they do, identify the two spikes, one on each side of the smoothed curve, that extend furthest from the curve. Measure the departures, in chart scale divisions, and add the two figures. Record this sum as the noise.

9.1.4.2 If spikes appear on only one side of the smoothed curve, record *twice* the largest departure from the curve, in chart scale divisions, as the noise.

9.1.5 Calculate and record the sum of the drift and noise. Compare this value to a reference value of 2 % of the full-scale chart reading.

9.1.5.1 If the sum of the drift and noise is greater than or equal to this reference value, record it, in chart scale divisions, as the minimum detectable signal.

9.1.5.2 If the sum is less than this reference value, record the reference value as the minimum detectable signal.

9.1.6 Convert the values for drift, noise, and minimum detectable signal, recorded in chart scale divisions, into equivalent meter scale divisions as follows:

9.1.6.1 Determine and record the ratio of full-scale meter divisions to the number of full-scale chart divisions.

9.1.6.2 Multiply by this ratio the values recorded in 9.1.3 (drift), 9.1.4 (noise), and 9.1.5 (minimum detectable signal). Record these values as the drift, noise, and minimum detectable signal, respectively, expressed in meter scale divisions.

NOTE 6—For the purposes of this test method, it is acceptable to sum drift and noise and to express the result in scale divisions, even though drift has units of scale divisions per unit time.

9.2 *Minimum Detectable Signal, Test Method B*:

9.2.1 Calculate and record the change in output between the initial and final meter readings for each of the 10-min intervals, that is, between $T = 0$ and $T = 10$ min, $T = 10$ and $T = 20$ min, $T = 20$ and $T = 30$ min, etc.

9.2.2 Divide each of these changes in output signal by 10. Record these values and compare them to a reference value of ½ % of the full-scale meter reading.

9.2.2.1 If the change in meter readings per 10 min is always greater than or equal to this reference value, identify by inspection the largest of these changes and record this value, in meter scale divisions per minute, as the drift.

9.2.2.2 If the change in meter readings per 10 min is always less than this reference value, calculate the total change in the 60-min period. Divide the total change value by 60 and record the result as the drift.

9.2.3 Calculate and record the differences in meter readings between the maximum and minimum signals for each of the 10th minute intervals including the first minute. By inspection, identify the largest of these differences and record this value in meter scale divisions as the noise.

9.2.4 Carry out 9.1.5-9.1.5.2 (Note Section 9).

9.3 *Minimum Detectable Leak Rate*:

9.3.1 For each calibrated leak, calculate and record the net output reading, $N = A - B$, in meter scale divisions.

9.3.1.1 If the leak detector has been set at reduced sensitivity (higher range), correct N accordingly and record the result in equivalent meter scale divisions at full sensitivity setting.

9.3.2 If the temperature recorded in 8.9.13 differs from that at which the calibrated leak was calibrated, calculate and record an adjusted leak rate using the temperature coefficient supplied by the manufacturer.

9.3.3 Calculate and record the minimum detectable leak rate (always express the minimum detectable leak rate for a response time T) in accordance with the relation:

$$\text{minimum detectable leak, for response time } T = \frac{(\text{calibrated leak rating})\ (\text{minimum detectable signal})}{N}$$

where calibrated leak rating = value recorded in 9.3.2, unless the calibration and test temperatures are the same, and N = signal due to the calibrated leak as recorded in 9.3.1.

9.4 *Sensitivity*:

9.4.1 Plot net meter readings against corrected leak rate in $atm \cdot cm^3/s$ (or in $Pa \cdot m^3/s$) on log-log paper for all leaks.

9.4.2 Calculate the coordinates of two convenient points on the line of best fit to the plotted points by the least squares method in accordance with Annex A1 and draw the best-fit line on the plot as a solid line. Record the slope of the best-fit line as the sensitivity.

9.4.3 On either side of the solid line, construct dashed lines to represent a 3-sigma limit of ±0.33 in accordance with Annex A2.

9.4.4 Examine the plot. Reject all detector calibration data from leaks for which the plotted points lie outside the dashed lines.

NOTE 7—In some applications it may be desirable to perform a test at a given leak value. In such cases, at least two calibrated leaks of approximately the desired value should be used for calibration. A calibration based on a single leak value is not recommended.

10. Report

10.1 Report the following information:

10.1.1 Date of tests;

10.1.2 Identification of operator;

10.1.3 For each calibration leak used,

10.1.3.1 Serial number of leak;

10.1.3.2 Manufacturer;

10.1.3.3 Type of leak (diffusion or capillary, with or without integral reservoir);

10.1.3.4 Supplier's labeled leak rate, $atm \cdot cm^3 \cdot s^{-1}$(or $Pa \cdot m^3 \cdot s^{-1}$); and

10.1.3.5 Temperature coefficient;

10.1.4 Test temperature, °C;

10.1.5 Identification of leak detector;

10.1.6 Drift, meter scale divisions per minute;

10.1.7 Noise, meter scale divisions;

10.1.8 Minimum detectable signal, meter scale divisions;

10.1.9 Response time, T, s;

10.1.10 Background reading, B, meter scale divisions;

10.1.11 Net output reading, N, meter scale divisions;

10.1.12 Minimum detectable leak for response time T; and

10.1.13 Sensitivity.

11. Precision

11.1 A round-robin evaluation of the precision of this test method indicates that a 3-sigma limit of ±0.33 is typical of the results that can be expected when commercially available calibrated leaks are used to calibrate a helium leak detector (see Annex A2).

11.2 The upper and lower 3-sigma limits are separated by a factor of 4.5.

NOTE 8—If more precise calibration of a leak detector is desired, the parties to the test should agree to select leaks for which the data points when plotted in a manner similar to that of Annex A2 fall within either 2-sigma or 1-sigma limits.

12. Keywords

12.1 leak detection; minimu leak rate; vacuum devices

ANNEXES

(Mandatory Information)

A1. CONSTRUCTION OF LINE OF BEST FIT

A1.1 The equation for the line of best fit can be determined by the least squares method. The equation may be expressed as:

$$y = a + bx$$

where:
y = net output in divisions,
a = intercept,
b = slope, and
x = leak rate of standard, atmos cm^3/sec.

A1.2 Since the data are expressed in logarithms, the line equation [5] will be:

$$\log y = a + b \log x$$

then

$$b = \frac{n\Sigma (\log x \times \log y) - \Sigma \log x \times \Sigma \log y}{n\Sigma (\log x)^2 - (\Sigma \log x)^2}$$

and

$$a = 1/n\,(\Sigma \log y - b\Sigma \log x)$$

where n = number of standard leaks being tested.

A1.3 The summation of x and y is mathematically expressed as:

$$\Sigma \log x = \Sigma_{i=1}^{n} \log x_i$$

$$\Sigma \log y = \Sigma_{i=1}^{n} \log y_i$$

For example, assume three leaks, 2.5×10^{-9}, 4.1×10^{-8}, and 2.3×10^{-7} atm·cm^3/s, have been tested and net output readings of 12, 170, and 1000 divisions have been obtained from the respective leaks (see Fig. A1.1). Expressed as logarithms [6] we have:

x (leak rate)

$$\log 2.5 \times 10^{-9} = 1.39794 - 10 = -8.60206$$

$$\log 4.1 \times 10^{-8} = 2.61278 - 10 = -7.38722$$

$$\log 2.3 \times 10^{-7} = 3.36173 - 10 = -6.63827$$

y(net output)

$$\log 12 = 1.07918$$

$$\log 170 = 2.23045$$

$$\log 1000 = 3$$

$$\Sigma \log x = -22.62755$$

$$\Sigma \log y = 6.30963$$

$$\Sigma (\log x)^2 = 172.63308, \Sigma (\log x \times \log y) = -45.67481$$

A1.4 Since the number of standard leaks tested in our example is 3, (n = 3), then

$$b = \frac{3(-45.67481) - (-22.62755)(6.30963)}{3(172.63308) - (-22.62755)^2}$$

b = 0.97519
a = ⅓[6.30963 − 0.97519 (−22.62755)]
a = 9.45859

Hence, in our example the line of best fit will be

$$\log y = 9.45859 + 0.97519 \log x$$

A1.5 To construct this line select two points such as 10^{-9} and 10^{-6} atm·cm^3/s.

Substituting these values into the equation we have

$$\log 10^{-9} = 1.00000 - 10 = -9$$

and

$$\log 10^{-6} = 4.00000 - 10 = -6$$

Case I (10^{-9} atm·cm^3/s):

$$\log y = 9.45859 + 0.97519\,(-9)$$

$$\log y = 0.68188$$

$$y = 4.8$$

Case II (10^{-6} atm·cm^3/s):

$$\log y = 9.45859 + 0.97519\,(-6)$$

$$\log y = 3.60745$$

$$y = 4.1 \times 10^{3}$$

This line is shown in Fig. A1.1.

[5] Crow, Davis, and Maxfield, *Statistics Manual*, Dover Publications, Inc., New York, N. Y.

[6] For ease of computation the logarithmic values have been expressed as negative numbers.

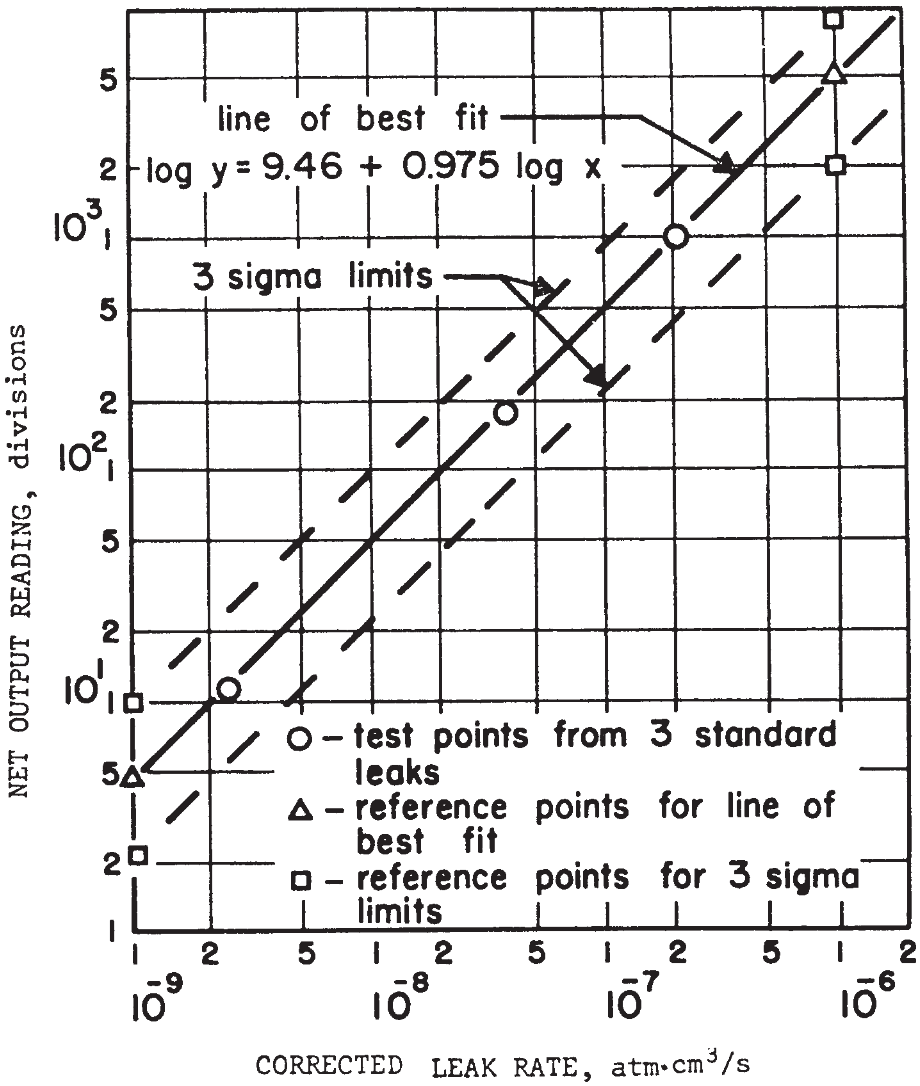

FIG. A1.1 Net Output Reading versus Corrected Leak Rate for Calibration of Helium Leak Detectors

A2. CONSTRUCTION OF SIGMA LIMITS

A2.1 A round-robin test has determined that a 3-sigma limit of ± 0.33 is the best that could be expected from the usual run of purchased leaks. The sigma value has been determined from the equation:

$$\sigma = \sqrt{\Sigma_i (n_i - 2)s_i^2 / \Sigma_i (n_i - 2)}$$

where:
n_i = number of tests by each participant, and
s_i = standard deviation from each participant's test data.

In general, s_i can be obtained from:

$$s_i = \sqrt{\Sigma(y - y_1)^2 / (n - 2)}$$

where:
y = output reading of the leak detector, and
y_1 = calculated value by least squares method.

In our example the 3-sigma limits would be log y ± 0.33 in Case I and Case II.

Case I:

$$\log y = 0.68188 \pm 0.33$$
$$\log y = 1.01188 \text{ or } 0.35188$$
$$y = 1.0 \times 10^1 \text{ or } 2.3$$

Case II:

$$\log y = 3.60745 \pm 0.33$$
$$\log y = 3.93745 \text{ or } 3.27745$$
$$y = 8.7 \times 10^3 \text{ or } 1.9 \times 10^3$$

These values are shown in Fig. A1.1 as the 3-sigma limits.