Designation: E 908 – 91

Standard Practice for
Calibrating Gaseous Reference Leaks[1]

This standard is issued under the fixed designation E 908; 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 practice covers procedures for calibrating leak artifacts of a specified gas, that may be used for determining the response of leak detectors, or in other situations where a known small flow of gas is required. The purpose of this practice is to establish calibration without reference to other calibrated leaks in as straightforward a manner as possible using the likeliest available equipment. While the uncertainties associated with these procedures will most likely be greater than those obtained via traceable calibration chains (on the order of 10 %), these procedures allow independent means of establishing or verifying the leakage rate from leak artifacts of questionable history, or when traceable leak artifacts are not available.

1.2 Two types of leaks are considered:

1.2.1 *Type I*—Pressure to vacuum.

1.2.2 *Type II*—Pressure to atmosphere.

1.3 Three calibration methods are described under each type of reference leak:

1.3.1 *Method A*—Accumulation comparison, using a known volume of gas at specified conditions of temperature and pressure as a reference.

1.3.2 *Method B*—Accumulation comparison, using a leak artifact calibrated using Method A.

1.3.3 *Method C*—Displacement of a liquid slug, by the leak, in capillary tube of known dimensions.

1.4 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be appropriate.

1.5 *This standard does not purport to address the safety problems 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 425 Definitions of Terms Relating to Leak Testing[2]

E 427 Practice for Testing for Leaks Using the Halogen Leak Detector (Alkali-Ion Diode)[2]

E 479 Guide for Preparation of a Leak Testing Specification[2]

F 134 Test Methods for Determining Hermeticity of Electron Devices With a Helium Mass Spectrometer Leak Detector[3]

2.2 *Other Standard:*

AVS 2.2-1968 Method for Vacuum Leak Calibration[4]

3. Summary of Practice

3.1 *Method A*—Accumulation comparison, using a known volume of tracer gas:

3.1.1 This method uses a closed chamber of nonreactive material having a means of removing all tracer gas and a connection to the tracer sensor.

3.1.2 A small, known quantity of tracer gas is discharged into the chamber and the response recorded for a period of time in which it is anticipated the unknown leak will require to reach the same concentration.

3.1.3 The tracer gas is removed from the chamber, and the unknown leak is allowed to discharge into it until the sensor response equals that of 3.1.2.

3.1.4 The leakage rate in mol/s can be calculated as:

$$Q_m = PV(t \cdot R \cdot T)$$

where:

P = pressure in known volume in atmospheres (1 atm = 101 325 Pa),

V = the volume of gas in cm^3 introduced in 3.1.2,

t = the time in seconds required for the concentration in 3.1.3 to equal that in 3.1.2,

R = gas constant = 82.06 = 1 atm cm^3/mol/K, and

T = absolute temperature, K.

3.1.5 It will be observed that chamber volume and sensor linearity are not factors in this equation. However, the chamber volume must be selected to give a concentration within the sensor range. Also, this concentration must also be achieved by the unknown leak discharging into the chamber in a reasonable length of time and must be appropriate so as not to significantly affect the equilibrium flow rate from the leak. This is particularly true of permeation leaks.

3.2 *Method B*—Accumulation comparison using a reference leak as calibrated in Method A, 3.1:

3.2.1 This method is a means of extending the primary calibration by a factor of up to 10, by comparing with previously-calibrated leak artifacts for longer periods of time. For example, a 5 × 10^{-12} mol/s leak that calibrated in Method A at 300 s can be used for 30 s to calibrate a 5 × 10^{-13} mol/s leak.

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[1] This practice is under the jurisdiction of ASTM Committee E-7 on Nondestructive Testing and is the direct responsibility of Subcommittee E07.08 on Leak Testing.

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[2] *Annual Book of ASTM Standards*, Vol 03.03.

[3] *Annual Book of ASTM Standards*, Vol 10.04.

[4] Available from AVS, American Vacuum Society, 335 E. 45th Street, New York, N.Y., 10017.

3.2.2 When this method is used, it should be realized that the total possible error will be at least doubled.

3.3 *Method C*—Direct measurement of leak rate by timing the movement of a liquid slug in a capillary tube of known dimensions:

3.3.1 The tube is closely coupled to the leak, and has a vent/fill valve to allow gas filling or positioning of the slug, or both, which is then driven by the leakage of the gas.

3.3.2 Due to capillary "friction," this method is limited to a minimum leak size of about 1×10^{-5} atm·cm^3/s (1 μPa·m^3/s).

4. Interferences

4.1 *Type I Leaks*, atmosphere to vacuum, Methods A and B:

4.1.1 For the purposes of this section, it will be assumed that the gas is helium and the detector is the mass spectrometer tuned for helium.

NOTE 1—Other gases or detectors, or both, can be used with little difference in procedures or interferences.

4.1.2 *Pressure Rise*—There will inevitably be some pressure rise in a closed evacuated chamber, due to outgassing and small leaks. This may cause a decrease in ionization efficiency in the spectrometer tube and thus a steadily declining signal as indicated in Fig. 5. However, this effect should be quite constant from run to run, and so largely cancel out in final result.

4.1.3 *Helium Signal Rise*—There will usually be a noticeable increase in helium signal when the chamber is closed, due to outgassing and in-leakage from the atmosphere as indicated in Fig. 5. Again, this will be a constant which mostly cancels out.

4.1.4 *Spectrometer Sensitivity Drift*—This will be noticed as variations in zero and in reading levels with the same helium input. With properly tuned and maintained systems operating at least one decade below maximum sensitivity, this should be a minor effect.

4.1.5 *Leaks*—All detectable valve leaks and leaks from the atmosphere should be repaired.

4.1.6 *Barometric Variations*—(Not applicable to sealed reservoir units.) If the gage used to measure the pressure in the known volume is of the gage type, then account must be made of the local barometric pressure when calculating the absolute pressure. This is probably true for falling pressures of the known volume near 1 atmosphere or less.

4.1.7 *Temperature Drift*—Changes in temperature between measurements may result in slight variations in indicated pressures. These should be recorded and compensated for accordingly.

4.2 *Type I Leaks*, atmosphere to vacuum, Method C:

4.2.1 *Liquid Slug Friction*—This can be appreciable in small capillaries. It should be measured and a correction made for it.

4.2.2 *Vapor Pressure of Liquid*—Water is the recommended liquid, and has a vapor pressure of about 20 mm Hg (3 kPa) at room temperature. This gives a theoretical increase in leak indication of 20/760 ($3 \times 10^3/1 \times 10^{-5}$) or approximately 3 %. This correction should be added to the final result.

4.2.3 *Excess Volume Between Leak and Capillary*—This will cause delayed and jerky movement of the slug, and

should be kept to an absolute minimum.

4.2.4 *Dirty Capillary*—Symptoms similar to 4.2.3. The slug should move smoothly when capillary tube is held at an angle.

4.3 *Type II Leaks*, pressure to standard atmosphere, Methods A and B:

4.3.1 For the purposes of this section, it will be assumed that the gas is R-12 (dichlorodifluoromethane) and the detector is the alkali-ion halogen detector diode. Other gases or detectors, or both, can be used with little difference in procedures or interferences.

4.3.2 *Halogen Signal Rise*—There will usually be a small increase in halogen signal due to outgassing, particularly from elastomers or plastics. With minimum use of these materials in the chamber, no correction for this will ordinarily be needed.

4.3.3 *Sensor Sensitivity Drift*—This will be noticed as variations in zero and reading levels with the same halogen input. With properly maintained systems operating at least one decade below maximum sensitivity, this should be a minor effect.

4.3.4 *Barometric Variations*—Substantial variations from standard atmosphere pressure should be corrected.

4.4 *Type II Leaks*, pressure to atmosphere, Method C—Same as Type I, Method C, in 4.2.

5. Apparatus

5.1 *Type I Leaks*, pressure to vacuum, Methods A and B:

5.1.1 *Mass Spectrometer with Remote Tube Tuned for Helium*—Minimum resolution (5×10^{-15} mol/s) helium, when operated as a leak detector.

5.1.2 *Helium Supply with Pressure Regulator and Flowmeter* (approximately 10 cm^3/s).

5.1.3 *Stainless-Steel Chamber* (see Fig. 1) with provisions for:

5.1.3.1 Attachment of spectrometer tube,

5.1.3.2 Liquid nitrogen trap,

5.1.3.3 Vacuum pumping to at least 1×10^{-6} torr (130 μPa) with isolating valve,

5.1.3.4 Ionization vacuum gage,

5.1.3.5 Attachment of helium leak with isolating valve and separate rough pumping means,

5.1.3.6 Measured helium volume device (see Fig. 2) (see Note 2), and

5.1.3.7 Strip chart or flat-bed recorder.

NOTE 2—Other types of calibrated volumes in this range may be substituted.

5.1.3.8 Thermometer.

5.2 *Type I Leaks*, pressure to vacuum, Method C:

5.2.1 *Glass Capillary Tube with Vent Valve* (see Fig. 3).

5.2.2 *Timer or Stop Watch*.

5.2.3 *Helium Supply*.

5.2.4 *Indicator Fluid* (dyed water).

5.2.5 *Thermometer*.

5.3 *Type II Leaks*, pressure to atmosphere, Methods A and B:

5.3.1 *Halogen Detector*—Minimum sensitivity 1×10^{-8} atm·cm^3/s (1 nPa·m^3/s).

5.3.2 *R-12 Supply with Flowmeter*.

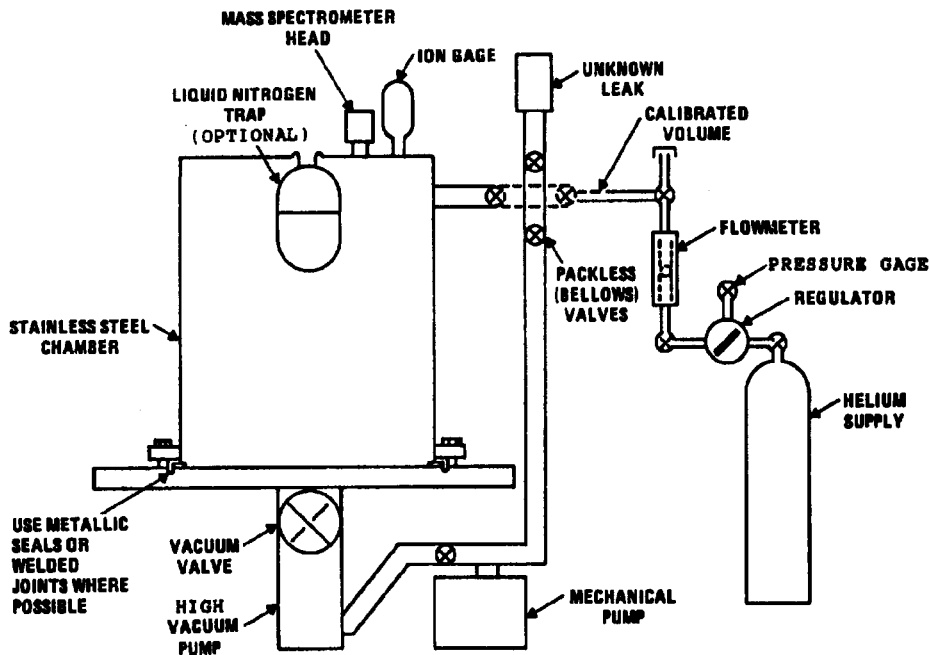5.3.3 *Stainless-Steel Chamber* (see Fig. 4) with provisions for:

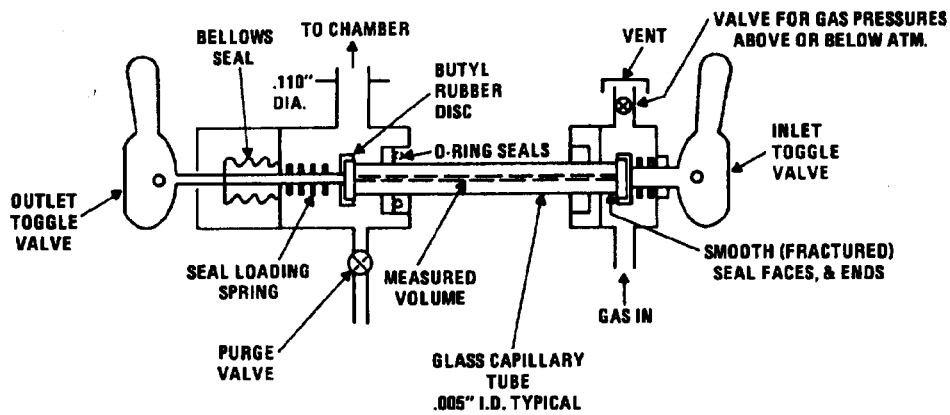FIG. 1 Equipment for Calibrating Helium Leaks, Type I, Methods A and B
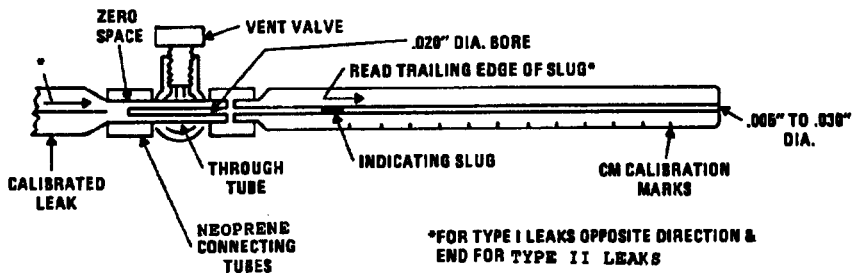

FIG. 2 Measured Volume Device

FIG. 3 Calibration Capillary and Vent Valve Assembly

5.3.3.1 Attachment of sensor sampling tube,

5.3.3.2 Pure air supply,

5.3.3.3 Attachment of halogen leak,

5.3.3.4 Measured halogen volume device (see Fig. 2), and

5.3.3.5 Strip chart or flat-bed recorder.

5.3.3.6 *Thermometer.*

5.4 *Type II Leaks,* pressure to atmosphere, Method C:

5.4.1 *Glass Capillary with Vent Valve* (see Fig. 3).

5.4.2 *Timer or Stop Watch.*

5.4.3 *Halogen Supply.*

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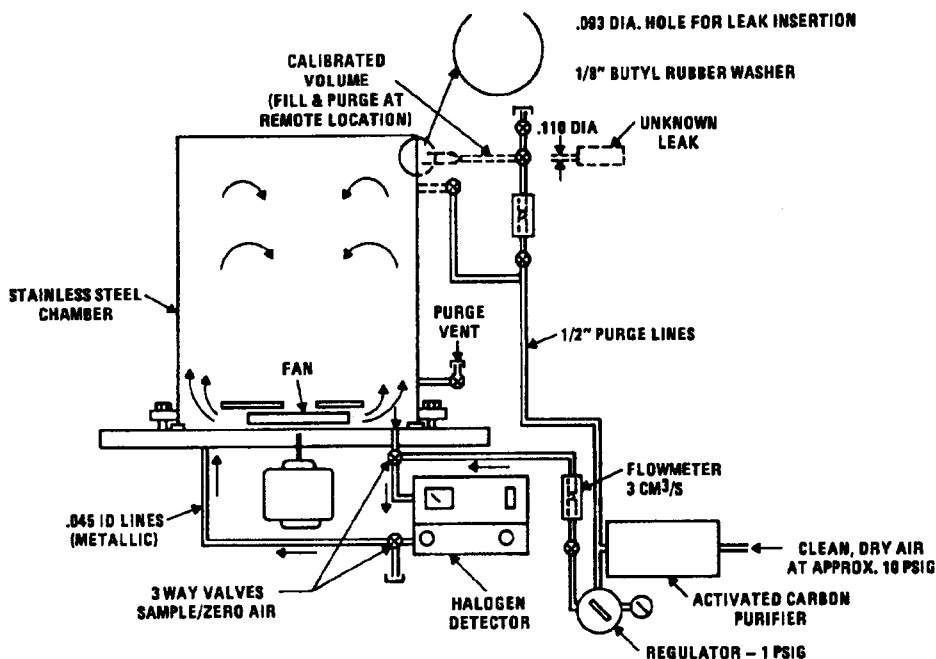
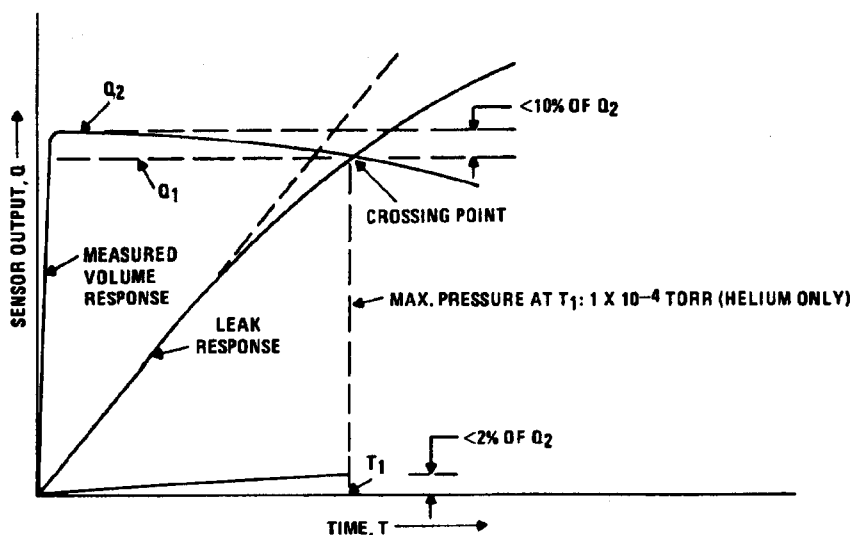FIG. 4 Equipment for Calibrating Halogen Leaks, Type II, Methods A and B

FIG. 5 Typical Detector Curves and Deviation Limits

5.4.4 *Indicator Fluid* (dyed water).

5.4.5 *Thermometer.*

6. Procedures

6.1 *Type I Leaks*, atmosphere (or sealed reservoir) to vacuum, Method A:

6.1.1 Start vacuum pumps, and pump chamber (see Fig. 1) down to 10^{-6} torr (130 μPa) or lower, as measured by the ion gage. Fill liquid nitrogen trap.

6.1.2 Attach measured helium volume device (see Fig. 2), and evacuate to the helium inlet valve.

6.1.3 Start mass spectrometer and determine that it is properly tuned to required sensitivity, and is stable.

6.1.4 With the helium outlet valve open, pass helium by helium inlet valve. No leakage should be observed.

6.1.5 Close helium outlet valve and open inlet valve for 5 s. No leakage should be observed.

6.1.6 Attach and evacuate leak to be calibrated. Apply helium if not a sealed reservoir type, record the helium pressure. Allow the system, including the leak itself, sufficient time to equilibrate.

6.1.7 Close vacuum valve and record rate of helium signal rise on the strip chart recorder for several minutes, selecting a range that will stay on scale for this length of time.

6.1.8 Isolate helium leak and pump down the chamber until chart reads zero. When isolating the helium leak from
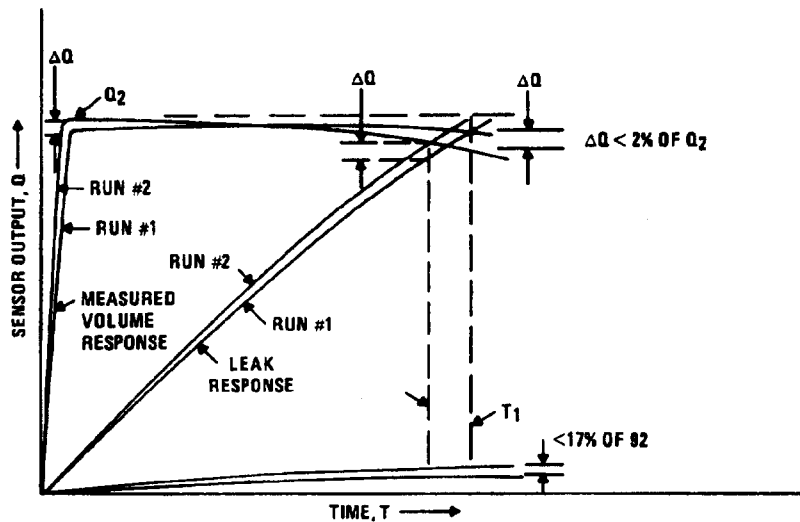
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FIG. 6 Typical Run-to-Run Variations and Limits

the chamber, alternate pumping on the leak should be provided, or sufficient time for reequilibration must be left, to attain lowest uncertainties. Reset chart to time zero and close vacuum valve. Record rise for the same period. If the rise exceeds 2 % of the helium signal, locate the source (such as air in leakage, outgassing of elastomers, or leaky valves) and repair.

6.1.9 Note the pressure rise in the system. If it exceeds 1 × 10⁻⁴ torr (13 mPa) at thé end of the time period, repair the leaks responsible.

6.1.10 Reevacuate the chamber, close the vacuum valve, and admit the measured helium volume, and note whether or not the signal is on scale and at least 30 % of full scale.

6.1.11 It will be necessary to have the traces of the unknown leak and the known volume of helium cross in no less than 30 s and no longer than it takes: (See Fig. 5.)

6.1.11.1 The background helium signal to rise 2 %.

6.1.11.2 The helium leak signal to depart 10 % from linearity.

6.1.11.3 The known helium volume signal to change 10 %.

6.1.11.4 The pressure to rise to more than 1 × 10⁻⁴ torr (13 mPa). (With a proper system this time should be in excess of 5 min.)

6.1.12 If necessary, adjust:

6.1.12.1 The accumulation time (within the above limits).

6.1.12.2 The helium pressure in the measured volume.

6.1.12.3 The size of the measured volume.

6.1.12.4 The sensitivity of the mass spectrometer.

6.1.13 Retrace all the above curves. No variation in excess of 2 % should occur.

6.1.14 Calculate the unknown leakage rate by:

$$Q_m = PV/(t \cdot R \cdot T)$$

where:

Q_m = leakage rate, mol/s,
P = pressure at known volume in atmospheres (1 atm = 101 325 Pa),
V = volume of helium, cm³,
t = time at which the leak and standard traces cross, s,
R = gas constant = 82.06 atm cm³/mol/K, and

T = absolute temperature, K.

See Figs. 5 and 6.

6.2 *Type I Leaks*, atmosphere (or sealed chamber) to vacuum, Method B:

6.2.1 The purpose of this procedure is to extend the range of calibration of a given chamber-measured volume system by a factor of about 10, by comparing with previously calibrated leak artifacts for longer periods of time.

6.2.2 The procedure is the same as Method A except that a leak that has been calibrated by Method A is used as a transfer standard (with some loss in accuracy) to calibrate an unknown leak.

6.2.3 Allow the known leak (leak rate Q_{m_1}) to leak into the chamber for a measured time T_1 until a desired level of helium is achieved, at which time it is shut off and the level recorded for an additional period (roughly 10 times t_1).

6.2.4 Reevacuate the chamber, close the vacuum valve, reset the recorder to time zero, and allow the unknown leak to leak into the chamber until the two traces cross at t_2.

6.2.5 Calculate the unknown leak rate Q_{m_2} by:

$$Q_{m_2} = Q_{m_1} (t_1)/(t_2)$$

6.3 *Type I Leaks*, pressure to vacuum (applicable only to capillary-type leaks 5 × 10⁻¹⁰ mol/s and larger), Method C.

6.3.1 Attach the unknown leak to a vacuum of 1 torr (130 Pa) or better.

6.3.2 Attach capillary-vent valve assembly (Fig. 3) to the atmospheric end of the leak.

6.3.3 Purge helium through the vent valve, leak, and capillary for 30 s minimum.

6.3.4 Start a slug of indicating fluid into the end of the capillary about 13 mm long.

6.3.5 Close the vent valve and note the time t_1 in seconds for the slug to transverse a number of centimetres, N. Open the vent valve before the slug is drawn into the leak!

6.3.6 Reposition the slug to the end of the capillary again, using helium pressure.

6.3.7 Repeat the test. The times for several runs should repeat within ±5 %.

6.3.8 Calculate the unknown leakage rate by:

$$Q_m = (\text{PNK}/T_{average} \, RT) \times 0.97$$

where:

K = the capillary constant in cm^3/cm, and

0.97 is the correction for water vapor pressure (see 4.2.2).

6.4 *Type II Leaks*, pressure to atmosphere, Method A:

6.4.1 Start halogen detector and allow to stabilize.

6.4.2 Purge the chamber (Fig. 4) until chamber air and zero air read the same.

6.4.3 Attach the measured volume device (Fig. 2) that has been filled within the last 5 min with R-12 and the chambers purged of R-12 with zero air, as follows:

6.4.3.1 Remove measured volume device (Fig. 2) from chamber.

6.4.3.2 Attach to a source of R-12 (dichlorodifluoromethane) gas in a ventilated hood or in a remote location, and purge the gas in chamber with about 100 mL of gas, (both toggle valves closed).

6.4.3.3 Close vent valve, open toggle valves, and apply 1 psig R-12 gas pressure to inlet chamber.

6.4.3.4 After 10 s, close both toggle valves, remove R-12 source, and open vent and purge valves.

6.4.3.5 Purge zero air through both chambers for at least 1 min at a rate of at least 50 cm^3/s, and close purge valve.

6.4.4 Start the circulating fan and discharge the R-12 into the chamber, using zero air at 1 psi (7 kPa) above atmospheric pressure for 15 s.

6.4.5 Sample the chamber for 5 s every 30 s, and record maximum readings, for as long as it is anticipated it will take the unknown leak to reach this concentration.

6.4.6 Purge the chamber as in 6.4.2.

6.4.7 Repeat 6.4.5 and record zero drift for the same period.

6.4.8 Repeat 6.4.2.

6.4.9 Insert unknown leak into the chamber, start fan, and record halogen concentration rise as in 6.4.5.

6.4.10 Adjust times, etc., and find leak rate as in 6.1.11 through 6.1.14. (Omit 6.1.11.4 as pressure rise is not a problem in a system at atmospheric pressure.) See Fig. 5.

6.5 *Type II Leaks*, pressure to atmosphere, Method B:

6.5.1 This procedure is the same as Method A except that a leak that has been calibrated by Method A is used as a transfer standard (with some loss in accuracy) to calibrate an unknown leak.

6.5.2 Insert the known leak into the purged chamber until the desired concentration is reached, then proceed as in 6.4.5 through 6.4.10.

6.6 *Type II Leaks*, pressure to atmosphere—(Applicable only to capillary-type leaks 5×10^{-10} mol/s and larger), Method C:

6.6.1 Attach capillary-vent valve assembly (Fig. 3) to the atmospheric end of the leak.

6.6.2 Start a slug of indicating fluid about ½ in. (13 mm) long in the capillary, and draw it slowly up the tube close to the other end.

6.6.3 Close the vent valve and time the movement of the slug for a convenient number of centimetres, N.

6.6.4 Reposition the slug and repeat 6.6.2. Do this several times. The times t_i should repeat within ±5 %.

6.6.5 Calculate the unknown leak rate by:

$$Q_m = (\Delta PNK)/(T_{average} \, RT)$$

where:

K = the capillary constant in cm^3/cm, and

ΔP = the pressure difference between the applied pressure and local atmospheric pressure.