Standard Practice for
Calibrating Gaseous Reference Leaks[1]

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 standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.5 *This standard does not purport to address all of 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:*[2]

E425 Definitions of Terms Relating to Leak Testing (Withdrawn 1991)[3]

E427 Practice for Testing for Leaks Using the Halogen Leak Detector Alkali-Ion Diode (Withdrawn 2013)[3]

E479 Guide for Preparation of a Leak Testing Specification (Withdrawn 2014)[3]

F134 Test Methods for Determining Hermeticity of Electron Devices with a Helium Mass Spectrometer Leak Detector (Withdrawn 1996)[3]

2.2 *Other Documents:*

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

Recommended Practices for the Calibration and Use of Leaks[5]

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:

$$Q_m = PV\,(t{\cdot}R{\cdot}T) \tag{1}$$

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.

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 4×10^{-10} mol/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. 1. 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. 1. 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.

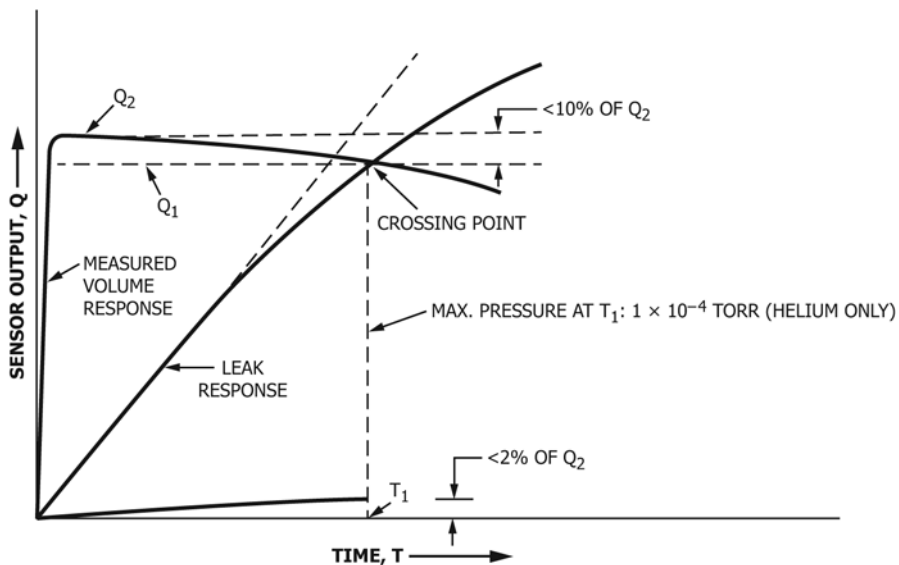4.2 *Type I Leaks,* atmosphere to vacuum, Method C:

FIG. 1 Typical Detector Curves and Deviation Limits

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 fluorocarbon 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. 2) 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. 3) (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. 4).

5.2.2 *Timer or Stop Watch.*

5.2.3 *Helium Supply.*

5.2.4 *Indicator Fluid* (dyed water).

5.2.5 *Thermometer.*

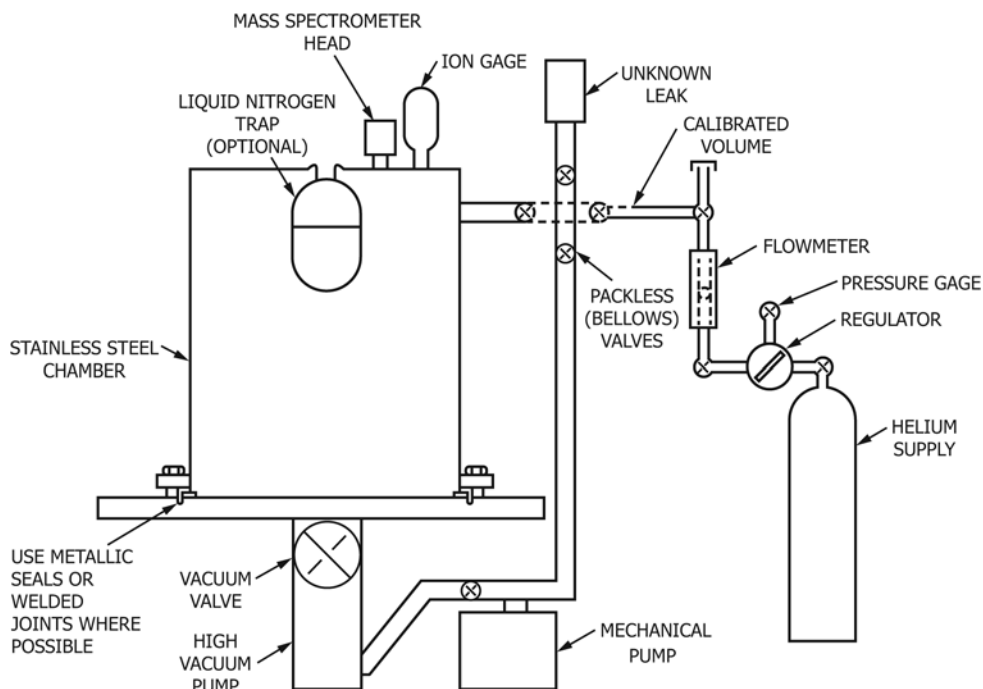**5.3** *Type II Leaks,* pressure to atmosphere, Methods A and B:

FIG. 2 Equipment for Calibrating Helium Leaks, Type I, Methods A and B