Designation: E908 – 98 (Reapproved 2018)

Standard Practice for
Calibrating Gaseous Reference Leaks[1]

This standard is issued under the fixed designation E908; 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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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.

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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

[5] C.D. Ehrlich and J.A. Basford, Journal of Vac. Sci, Technology, A(10), 1992, pp. 1–17.

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:

$$Q_m = PV(t \cdot R \cdot T) \qquad (1)$$

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
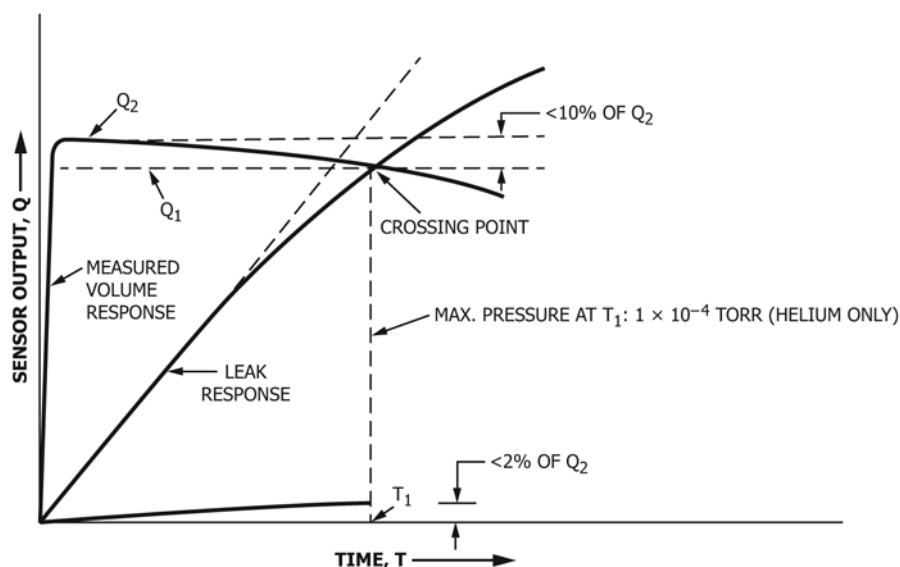

FIG. 1 Typical Detector Curves and Deviation Limits