**Designation: ~~D4298 – 04 (Reapproved 2014)$^{\varepsilon 1}$~~ D4298 – 22**

# Standard Guide for Intercomparing Permeation Tubes to Establish Traceability[1]

This standard is issued under the fixed designation D4298; 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 ($\varepsilon$) indicates an editorial change since the last revision or reapproval.

~~$\varepsilon^1$ NOTE—Editorial corrections were made throughout in December 2014.~~

## 1. Scope

1.1 This guide covers two procedures for establishing the permeation rate of a permeation tube and defining the uncertainty of the rate by comparison to National Institute of Standards and Technology's Standard Reference Materials (SRM).

1.2 Procedure A consists of a direct comparison of the permeation rate of the device undergoing calibration with that of an SRM.

1.3 Procedure B consists of a gravimetric calibration process in which a certified permeation tube is used as a quality control for the measurements.

1.4 Both procedures are limited to the case where a suitable certified permeation device is available.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* (See 8.2 on Safety Precautions.)

1.7 *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]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres
D3249 Practice for General Ambient Air Analyzer Procedures
D3609 Practice for Calibration Techniques Using Permeation Tubes
D3631 Test Methods for Measuring Surface Atmospheric Pressure
E1 Specification for ASTM Liquid-in-Glass Thermometers
E319 Practice for the Evaluation of Single-Pan Mechanical Balances (Withdrawn 2021)[3]
E617 Specification for Laboratory Weights and Precision Mass Standards

[1] This guide is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.01 on Quality Control.
Current edition approved ~~Dec. 1, 2014~~Sept. 1, 2022. Published ~~January 2015~~September 2022. Originally approved in 1983. Last previous edition approved in ~~2010~~2014 as D4298 – 04 ~~(2010).~~(2014)$^{\varepsilon 1}$. DOI: ~~10.1520/D4298-04R14E01.~~10.1520/D4298-22.

[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.

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this guide, refer to Terminology D1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *working standard*—a standard used in the laboratory or field for periodic standardization of a measuring instrument.

## 4. Summary of Guide

4.1 *Procedure A*—A certified SRM permeation source, obtained from the National Institute of Standards and Technology is used to calibrate a continuous analyzer. The analyzer is then used to measure the concentration of a gaseous mixture generated from the permeation tube under calibration. Equations are provided that permit calibration of the permeation rate of the latter from the test data.

4.2 *Procedure B*—The permeation source is calibrated, gravimetrically, using temperature and mass standards traceable to ~~NIST standards.~~ the United States National Institute of Standards and Technology (NIST), or other national or international standard reference. The validity of the calibration is confirmed by concurrently measuring the permeation rate of a ~~Certified Reference Material~~certified reference material (CRM).

## 5. Significance and Use



5.1 The accuracy of air pollution measurements is directly dependent upon accurate calibrations.

5.2 Such measurements gain accuracy and can be intercompared when the measurement procedures are traceable to national measurement standards.

5.3 This guide describes procedures for enhancing the accuracy of air pollution measurements which may be specified by those organizations requiring traceability to national standards.



## 6. Apparatus

6.1 For apparatus used in the calibration of permeation devices, refer to Practice D3609.

6.1.1 The thermometers used shall conform to Specification E1 or Specification E2251 and shall have calibration certificates traceable to the NIST. Measurement uncertainty should be ~~0.1°C~~0.1 °C or less.

6.1.2 *Barometer*—~~The mercury barometer shall conform to~~ An aneroid or other barometer capable of measuring atmospheric pressure to within ±300 Pa (25 mm Hg) shall be used. Calibrate the barometer as described in Test Methods D3631.

6.2 *Apparatus for Procedure A:*

6.2.1 *Analytical Instruments*—An analytical instrument responsive to the permeant with the following minimum performance specifications:

| | |
|---|---|
| Noise | 1 % of full-scale |
| Zero drift | ±4 % of full-scale per day |
| Span drift | ±3 % of full-scale per day |
| ~~Range~~ | ~~0 to 0.5 ppm (or appropriate for source strength)~~ |
| Range | 0 ppm to 0.5 ppm (or appropriate for source strength) |

6.2.2 Continuous strip chart recorder with the following minimum performance specifications:

| | |
|---|---|
| Uncertainty component | 0.33 × (0.25 % full-scale deflection) |
| Chart width | no less than 6 in. |
| Time for full-scale travel | 1 s |

NOTE 1—ISO ~~GUM~~JCGM 100:2008[4] points out that with approximately normal plotted points, the above maximum 3 × standard deviations is equivalent to >99 % of plotted points lying within ± (0.25 % full-scale deflection) of true values.

6.3 *Apparatus for Procedure B:*

6.3.1 Analytical balance, meeting the requirements of Practices E319 and D3609.

6.3.2 Analytical weights meeting the requirements of Specification E617 and having a calibration certificate traceable to ~~the NIST.~~NIST or other national or international standard reference weights.

## 7. Materials

7.1 Refer to Practice D3609.

7.2 CRM Permeation Device.[5]

## 8. Precautions

8.1 *Procedural Precautions:*

8.1.1 The procedural precautions described in Practice D3609 are applicable to ~~the present~~ this guide.

8.1.2 When possible, the permeation device should be compared to the CRM using the same system with identical flow and temperature conditions. Unpredictable errors may be introduced if permeation devices are compared at widely different temperatures and flow rates (pertains to Procedure A). Intercomparisons are valid only at temperatures for which the CRM tube is calibrated.

8.1.3 Equilibration of the permeation device, the calibration equipment, and the analytical system must be quality assured, prior to use. During storage, avoid exposing tubes to high ~~humidities~~humidity or wide variations in ~~temperature,~~temperature that may permanently alter the permeation rate.

8.2 *Safety Precautions:*

8.2.1 For precautions concerning the use of analytical instruments and of cylinders containing pressurized gases, see Practice D3249.

## 9. Calibration and Standardization

9.1 *Procedure A:*

9.1.1 Set up a gas generation system using a ~~Certified Reference Material~~certified reference material (CRM) permeation tube and apparatus and procedure such as described in Practice D3609. Equilibrate at the desired temperature of calibration.

9.1.2 Optimize the performance of the analytical instrument according to the manufacturer's instructions.

9.1.3 Using dry air or nitrogen, set the zero point of the instrument.

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[4] ~~ISO GUM,~~JCGM 100:2008, ~~Guide to the Expression of Uncertainty in Measurement,~~GUM 1995 with minor corrections, Evaluation of measurement data—Guide to the expression of uncertainty in measurement; ~~International Organization for Standardization (ISO), available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.~~www.bipm.org/documents/20126/2071204/JCGM_100_2008_E.pdf/cb0ef43f-baa5-11cf-3f85-4dcd86f77bd6.

[5] Certified permeation tubes may be obtained from NIST currently only by special order from the Office of Standard Reference Materials, National Institute of Standards and Technology, Washington, DC 20234.

9.1.4 Use the gas generation system to provide gas concentrations corresponding to ~~20, 40, 60,~~20 %, 40 %, 60 %, and 80 % of full-scale readings. Record the concentration and respective readings. Repeat the measurements in random order.

9.1.5 Plot concentration versus instrument readings and draw the line of best fit, or alternatively fit by linear least squares regression. Calculate the slope (ppm(v)/scale reading) and standard deviation estimate $s_{cal}$, expressed as µg/min.

9.1.5.1 If any point deviates by more than ±1 % from the line of best fit, repeat the calibration.

9.2 *Procedure B:*

9.2.1 The standard masses and the thermometer used must have a valid calibration certificate or be calibrated prior to use.

## 10. Procedure

10.1 *Procedure A:*

10.1.1 Place test permeation device in the system, equilibrate at the temperature of calibration, and generate gas mixtures corresponding approximately to ~~20, 40, 60,~~20 %, 40 %, 60 %, and 80 % of full scale readings, respectively.

10.1.2 Record the instrument readings for each gas mixture.

10.1.3 Using calibration curves described in 9.1.5, calculate the concentrations of the gas mixtures.

10.1.4 Calculate the permeation rates as described in 11.1.

10.1.5 Repeat the measurements of 10.1.1 in random order and record as in 10.1.2.



10.2 *Procedure B:*

10.2.1 Maintain the permeation device at constant temperature, T, during the sequence of measurements described as follows:

10.2.2 Weigh permeation device, periodically recording the mass and time of weighing, as described in Practice D3609.



10.2.3 Calculate the mass loss per unit of time in the units of µg/min at temperature, T.

10.2.4 Calibrate a CRM permeation tube using the same procedure and concurrently with the test permeation device.

## 11. Calculations

11.1 *Procedure A:*

11.1.1 Calculate the permeation rate for each of the eight measurements, using the following equation:

$$R = C_{ppm(v)} \frac{F \times MW}{MV} \quad (1)$$

where:

$R$ = permeation rate, µg/min,
$C_{ppm(v)}$ = measured concentration, ppm(v) (by volume),
$F$ = total flow rate of gas (L/min),
$MW$ = molecular weight of permeant, and
~~$MV$ = molecular volume (24.47 L at 25°C and 101.3 kPa).~~
$MV$ = molecular volume (24.47 L at 25 °C and 101.3 kPa).

11.1.2 Calculate the mean $\bar{R}$ of measured rates and the standard deviation, $s_R$. The temperature corresponding to ~~$\bar{R}$~~ this determination must be stated.