National Institute of Standards & Technology

Certificate

Standard Reference Material 1627

Sulfur Dioxide Permeation Tube

2-cm
(Individually Calibrated)
Serial No.

This Standard Reference Material (SRM) is intended primarily for use in the calibration of apparatus and standardization of procedures used in air pollution and related chemical analyses. SRM 1627 is a 2-cm sulfur dioxide permeation tube that is individually certified according to NIST protocols and procedures. Certified permeation rates of sulfur dioxide at temperatures in the range of 25 to 35 °C are given in the table included in the package with the permeation tube.

Certification

The permeation rates were determined using the method described on the reverse side of this certificate.

The uncertainty of the permeation rates is the estimated upper limit of error of the certified value at the 95% confidence level. It is not expected to exceed 2.0% relative in the range of 25 and 35 °C. The uncertainty includes the estimated inaccuracy of the permeation rate of the primary standard permeation tubes that were calibrated gravimetrically and the imprecision of intercomparison of the SRM permeation tubes and the standard tubes.

Certification Period and Storage

This tube can be expected to last for approximately 9 months when used at 30 °C. When not in use, life can be extended by storage at freezer temperatures (4 °C). During storage, the SRM should always be protected from moisture and kept at a relative humidity of 10% or less (see Use). On removal from low temperature storage, the tube should be equilibrated at the operating temperature for at least 36 hours prior to use.

Experiments at NIST have shown that the certification of this tube remains valid as long as a visible amount of liquid sulfur dioxide remains in the tube and proper storage conditions are employed.

Caution

The SRM contains liquid sulfur dioxide at a pressure of about 0.4053 MPa (4 atm) at 25 °C. While no failures have occurred during use, there is the possibility of rupture due to internal pressure. However, it is believed that normal handling of the tube at temperatures up to and slightly exceeding 36 °C does not constitute a hazard.

The calibration measurements were made by G. D. Mitchell of the NIST Organic Analytical Research Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of W. D. Dorko and W. E. May of the NIST Organic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T. E. Gills.

Gaithersburg, MD 20899
August 31, 1992
(Revision of certificate dated 12-5-91)

William P. Reed, Chief
Standard Reference Materials Program

(over)

Calibration

The permeation rate of this SRM was determined at 30 °C by means of a gas analyzer equipped with a sulfur dioxide-specific detector. This detector was calibrated using three randomly selected permeation tube standards whose permeation rates were determined by gravimetry. The permeation rates of the three standards were determined at three selected temperatures, 25, 30, and 35 °C. Ten permeation tubes of the SRM lot were then selected as batch standards and their permeation rates were also determined at the selected temperatures of 25, 30, and 35 °C. The measured rates of the tubes were fitted by the method of least squares to an equation of the type, $\text{Log}(R) = M(273.16 + T\,°C) + B$, where the constants M (slope) and B (intercept) were determined from a gravimetric measurement of the permeation rate as a function of temperature for the SRM lot standards; R is the permeation rate (μg/min) at temperature T (°C). The resulting equation along with the calibrated rate were used to calculate the values of the permeation rates given in the table accompanying this tube.

Material Preparation

This tube is one of a group of permeation tubes taken from a homogenous lot that was prepared commercially according to NIST specifications. A lot consists of a minimum of 100 tubes. As stated above, each tube within the lot is individually analyzed at NIST for conformity to NIST specifications and is certified according to NIST protocols and procedures.

Use

This tube can be used to produce known concentrations of sulfur dioxide in a gas stream when both the temperature and flow rate of the gas stream are known. Apparatus and techniques for this purpose are described in references [1] and [2] which should be consulted for operational details. Because of the large temperature coefficient of the permeation rate, approximately 9% per °C, the temperature must be maintained constant and measured accurately to 0.1 °C to provide concentrations consistent with the calibration uncertainty.

The tube temperature should be held constant during use. The desired concentration can be achieved by adjusting the flow rate. If the concentration must be varied by changing the tube temperature, a suitable time interval must be allowed for equilibrium of the permeation rate to be re-established. For changes of 1 or 2 °C, a period of 3 hours is recommended. For changes greater than this or when the tube is removed from low temperature storage, a period of 36 hours is recommended.

This permeation tube is a stable and relatively rugged source of pure sulfur dioxide gas. However, it should be treated with the care necessary to assure the user that no change occurs in the character of the tube. Precautions should be exercised to prevent contamination of the tube's outer surface during handling. The tube should be protected from high concentrations of water vapor during storage and use.

Additional information concerning the performance and use of permeation tubes can be found in references [3-7].

References

[1] Health Laboratory Science, 7, No. 1, p. 4 (1970).
[2] F.P. Scarringelli, A.E. O'Keefe, E. Rosenberg, and J.P. Bell, Anal. Chem., 42, p. 871 (1970).
[3] O'Keefe and G.C. Ortman, Anal. Chem., 38, p. 760 (1966).
[4] F.P. Scarringelli, S.A. Frey, and B.E. Saltzman, Amer. Ind. Hyg. Assoc. J., 28, p. 260 (1967).
[5] J.K. Taylor, Ed., NBS Technical Note 545, December 1970.
[6] D.L. Williams, "Permeation Tube Equilibration Times and Long-Term Stability", Calibration in Air Monitoring, ASTM STP 598, American Society for Testing and Materials, pp. 183-197 (1976).
[7] G.D. Mitchell, "Trace Gas Calibration Systems Using Permeation Devices," Sampling and Calibration for Atmospheric Measurements, ASTM STP 957, J.K. Taylor, Ed., American Society for Testing and Materials, Philadelphia, pp. 110-120 (1987).