National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1626

Sulfur Dioxide Permeation Tube - 5 cm

This Standard Reference Material (SRM) is intended primarily for use in the calibration of apparatus and standardization procedures used in air pollution and related chemical analyses. SRM 1626 is a 5 cm sulfur dioxide permeation tube that individually certified according to NIST protocols and procedures.

Certification: The permeation rate was determined using the method described on page 2 of this certificate. From the measurements NIST has assigned an individual certified sulfur dioxide permeation rate to each tube in this lot. The certif rate for this SRM 1626 tube at 29.96 °C is given below:

SRM 1626 Tube No.: 93-

Permeation Rate: ± 0.02 µg/min at (29.96 ± 0.01) °C

NIST permeation tubes are well behaved, allowing the permeation rate at other temperatures to be predicted from the certif rate at 29.96 °C. However, the permeation tube temperature must be controlled to within ± 0.05 °C for the followi equation to accurately predict the new rate. This SRM's permeation rate has been verified to follow this equation temperatures between 25 °C and 35 °C.

$$\text{Log}\,[R_T] = 0.0331422\,(T) - 0.99294 + \log\,[R_c]$$

where R_T is the new permeation rate, T is the temperature, and R_c is the certified permeation rate given above.

The stated total uncertainty was determined from the uncertainty in the gravimetric permeation rate of the standard tubes a the additional uncertainty resulting from the NIST analysis of the lot. The uncertainty is expressed as an expanded uncertai $U = ku_c$ with u_c estimated from the experimental uncertainties, and the coverage factor k being equal to 2. Since errors determining the permeation rate are assumed to be normally distributed, the certified permeation rate for this SRM is asser to lie within the interval defined by the uncertainty with a level of confidence of approximately 95 %.

Stability and Storage: This tube can be expected to last for approximately 9 months when used at 30 °C. When not in u tube life can be extended by storage at freezer temperatures (4 °C). During storage, the SRM should always be protected fr moisture and kept at a relative humidity of 10 % or less. On removal from low temperature storage, the tube should equilibrated at the operating temperature for at least 48 hours (h) prior to use. Experiments have shown that the certificati of this tube remains valid as long as a visible amount of liquid sulfur dioxide remains in the tube, proper storage conditio are employed, and proper handling procedures are taken to prevent contamination of the tube's outer surface.

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

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

The calibration measurements were made by G.D. Mitchell and P.A. Johnson of the NIST Analytical Chemistry Division

The overall direction and coordination of the technical work required for the certification of this SRM was performed b
F.R. Guenther and W.J. Thorn III of the NIST Analytical Chemistry Division.

CAUTION: This SRM contains liquid sulfur dioxide at a pressure of about 0.4053 MPa (4 atm) at 25 °C. While no failure
have occurred during use, there is the possibility of rupture due to internal pressure. However, it is believed that norma
handling of the tube at temperatures up to 35 °C does not constitute a hazard.

Material Preparation: This permeation tube is one from a lot of 100 tubes that was commercially prepared according tc
NIST specifications. Each tube within the lot is individually analyzed at NIST for conformity to NIST specifications and i
certified according to NIST protocols and procedures.

Certification Method: NIST's primary method for determining SRM 1626 permeation rates is the measurement of sulfu
dioxide weight loss with time at a constant temperature. Two standard tubes were randomly selected from the lot anc
analyzed by this method. An automated method was then used to analyze the rest of the tubes comprising this lot of SRM
1626. This method compares the tubes analyzed by the primary method with the remaining tubes, using an instrument tha
responds to sulfur dioxide fluorescence. The permeation rate for each tube is then calculated from its response ratio to th
analyzed permeation rate of the standard tubes.

Use: This tube can be used to produce known concentrations of sulfur dioxide in a gas stream when both the temperatur
and flow rate of the gas stream are known. Because of the large temperature coefficient of the permeation rate, approximatel
8 % per °C, the temperature must be maintained constant and measured accurately to within 0.1 °C during use to provid
concentrations consistent with the certification uncertainty.

A desired concentration can be achieved by adjusting the flow rate. If the concentration must be varied by changing the tub
temperature, a suitable time interval must be allowed for equilibrium of the permeation rate to be re-established. For change
of 1 °C or 2 °C, a minimum period of 3 h is recommended. For changes greater than this, or when the tube is removed fron
low temperature storage, a minimum period of 48 h is recommended.

Additional information concerning the performance and use of permeation tubes can be found in references [1-3].

REFERENCES

[1] Williams, D.L., "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).

[2] Mitchell, G.D., "Trace Gas Calibration Systems Using Permeation Devices," Sampling and Calibration for Atmospheri
Measurements, ASTM STP 957, J.K. Taylor, Ed., American Society for Testing and Materials, Philadelphia, pp. 110
120, (1987).

[3] Mitchell, G.D., Dorko, W.D., and Johnson, P.A., "Long-Term Stability of Sulfur Dioxide Permeation Tube Standar
Reference Materials," Fresenius J., Anal. Chem., 344, 229-233, (1992).