

National Bureau of Standards

Certificate

Standard Reference Material 1626

Sulfur Dioxide Permeation Tube

(Individually Calibrated)

E. E. Hughes and W. P. Schmidt

This Standard Reference Material consists of a 5 cm sulfur dioxide permeation tube, individually calibrated, for use in the preparation of gases of known sulfur dioxide content. It is intended for standardization of apparatus and procedures used in air pollution and related chemical analyses. Permeation rates for temperatures in the range of 20 to 30 °C are given in the table accompanying each tube.

The tabulated values result from determinations of the permeation rates for the specified tube, using the method described on the reverse of this certificate. The uncertainty of the certified permeation rates, based on the results of the calibration of approximately 25 tubes, is less than ± 0.5 percent at 25 °C and does not exceed ± 1.0 percent at 20 and 30 °C, respectively.

Experiments in this laboratory have shown that the calibration remains valid as long as visible amounts of liquid sulfur dioxide remain in the tube.

The calibration measurements were made by E. E. Hughes and W. P. Schmidt, Analytical Chemistry Division, NBS Institute for Materials Research.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. K. Taylor.

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

Washington, D. C. 20234
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J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

CALIBRATION

This tube was individually calibrated by gravimetric determination of weight losses at 20, 25, and 30 °C, respectively. The tube was held at constant temperature for several days at each level, and the permeation rate was determined by weighing the tubes at 24-hour intervals, using a micro-balance. The measured rates were fitted by the method of least squares to an equation of the type $\log R = mt + b$. The resulting equation, given on the table accompanying the tube was used to calculate the values of the permeation rates.

The precision of calibration was estimated from measurements on approximately 25 tubes in this lot at each calibration temperature. The uncertainties indicated are the approximate half width of the 95 percent confidence interval. It is believed that the systematic errors concerned with the calibration are negligible.

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 [3] and [4] and should be consulted for operational details. Because of the large temperature coefficient of the permeation rate, approximately 9 percent per degree celsius, the temperature must be maintained constant and measured accurately to 0.1 °C to provide concentrations consistent with the calibration uncertainty.

It is recommended that the tube temperature be held constant during use and that desired concentration levels be achieved by adjustment of the flow rate. If it is necessary to vary the concentration 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 degrees celsius, a period of 3 hours should suffice. For changes of 10 degrees or when removed from low temperature storage, a period of 24 hours is advisable.

This permeation tube is a stable and relatively rugged source of sulfur dioxide and no extreme measures are necessary to ensure that the calibration of the tube will be maintained during its useful life. 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 outer surface during handling. The tube should be protected from high concentrations of water vapor during storage and use. A relative humidity of 10 percent should have no effect on the permeation rate within the calibration uncertainty.

STORAGE

The useful life of this certified sulfur dioxide permeation tube is about 9 months. Storage at lower temperatures will prolong the life. However, it should be protected from moisture during storage. On removal from low temperature storage, the tube should be equilibrated at the operating temperature for at least 24 hours, before use as an analytical standard.

PRECAUTION

This permeation tube contains liquid sulfur dioxide at a pressure of about 4 atmospheres at room temperature. 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 tubes at temperatures up to and slightly exceeding 35 °C does not constitute a hazard.

SELECTED REFERENCES

- [1] A. E. O'Keeffe and G. C. Ortman, *Anal. Chem.* **38**, 760 (1966).
- [2] F. P. Scaringelli, S. A. Frey, and B. E. Saltzman, *Amer. Ind. Hyg. Assoc. J.* **28**, 260 (1967).
- [3] *Health Laboratory Science* **7**, No. 1, 4 (1970).
- [4] F. P. Scaringelli, A. E. O'Keeffe, E. Rosenberg, and J. P. Bell, *Anal. Chem.* **42**, 871 (1970).
- [5] J. K. Taylor, Ed., *NBS Technical Note* 545, December 1970.