National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1629a

Nitrogen Dioxide Permeation Device

Serial No.

This Standard Reference Material (SRM) is a 1 cm nitrogen dioxide permeation device that is individually certified according to NIST protocols and procedures. SRM 1629a is intended primarily for use in the calibration of apparatus and standardization of procedures used in air pollution and related chemical analyses.

Certification: This SRM has been certified for the permeation rate of nitrogen dioxide. The certified permeation rate given below applies to the NIST serial number identified above.

The certified permeation rate is ( ) μg/min @ 25.00 °C as of

The uncertainty of the certified value includes the estimated uncertainty of the NIST temperature and weight measurements. The uncertainty is expressed as an expanded uncertainty $U = ku_c$ with u_c being determined from experimental standard deviations and the coverage factor k being equal to 2. The true value for the nitrogen dioxide permeation rate is asserted to lie in the interval defined by the certified value $\pm$ U with a level of confidence of approximately 95 % [1].

Expiration of Certification: The certified value on this certificate is valid for six (6) months from the date of shipment from NIST. A validation sticker is supplied with each unit to validate its certification period.

Handling Information: The device consists of a stainless steel reservoir and a short length of TFE-fluorocarbon tubing. The device contains less than 1 g of liquid nitrogen dioxide at a pressure of 100 kPa (one atmosphere) at 25 °C. While no documented failures have occurred during use, there is the slight possibility of rupture due to internal pressure. However, it is believed that handling of the device at temperatures up to 39 °C does not constitute a hazard.

Stability: No losses of nitrogen dioxide by any process other than permeation have been observed for retained samples of this SRM for periods of time greater than two years.

The analytical measurements leading to the certification of this current SRM lot were performed by G.D Mitchell and P.A. Johnson of the NIST Analytical Chemistry Division.

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

Statistical consultation and data analysis was performed by S.B. Schiller of the NIST Statistical Engineering Division.

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

Gaithersburg, MD 20899
Certificate Issue Date: June 6, 1996

Thomas E. Gills, Chief
Standard Reference Materials Program

Material Preparation: The device is one of a group of permeation devices taken from a lot that was prepared commercially according to NIST specifications. Each device in the lot is individually calibrated at NIST for conformity to NIST specifications and is certified according to NIST protocols and procedures.

Analytical Method: This device was individually calibrated by gravimetric determination of the mass lost of nitrogen dioxide at (25.08 ± 0.01) °C. The device was held at this temperature for a period of not less than three (3) months during which time weekly mass determinations were made. The permeation rate is determined from a linear regression plot of mass loss of nitrogen dioxide in μg versus time in minutes.

Instructions for Use: The device can be used to produce mixtures of nitrogen dioxide in air or other gases by placing the device in a thermostatic chamber through which a stream of dry gas flowing at a known rate is passed. The accuracy of the concentration produced is dependent not only on the accuracy with which the device is calibrated, but also on the accuracy with which the purity of the diluent gas, the rate of flow, and the temperature are known. Systems for generating known concentrations of nitrogen dioxide with the device are described elsewhere [2-3].

When the device is first placed in service, a period of at least 48 h should be allowed for equilibrium at the temperature at which the device is to be used.

The permeation rate of this device is certified at 25.00 °C. If the user wishes to vary the temperature of the device to within ± 5.0 °C from the certified temperature, the following equation can be used to calculate the permeation rate. If the temperature is within ± 1.0 °C the uncertainty must be increased by 0.4 %, and if the temperature is within ± 5.0 °C the uncertainty must be increased by 2 %.

It is also recommended that the device be used at the calibration temperature of 25.0 °C, but if the temperature does not vary more than 5 °C, an adjustment to the rate may be made according to the equation:

$$\text{Log } R_t = \log R_{25\,^\circ C} + 0.034512\,(t-25.0)$$

where R_t is the permeation rate of the device at the temperature of use t(°C), and $R_{25\,^\circ C}$ is the rate certified by NIST at 25.00 °C. The constant, 0.034512, was empirically determined using observations of the temperature-permeation rate relationship for identical devices at temperatures between 20.00 °C and 30.00 °C.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, D.C. (1994).

[2] D.L. Williams, "Permeation Tube Equilibration Time and Long-Term Stability," Calibration in Air Monitoring, ASTM STP 598, American Society for Testing and Materials, pp. 183-197, (1976).

[3] 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, PA, pp. 110-120, (1987).