National Institute of Standards & Technology

Report of Investigation

Reference Material 8510

Moisture in Methanol

Reference Material (RM) 8510 is intended primarily for use in the calibration of instruments and the evaluation of methods used for moisture determination. A unit of RM 8510 consists of five ampoules, each containing approximately 5 mL of methanol solution with a trace concentration level of water.

Analytical Results

The reference value is based on the results of an interlaboratory comparison exercise conducted among 13 laboratories and NIST using the coulometric Karl Fischer method. In addition, NIST measurements using the volumetric Karl Fischer method were performed to provide an independent assessment of both the moisture content as well as the homogeneity of the RM lot. It is important to note that this material is **not** a Standard Reference Material" and the reference value is method dependent.

Reference Concentration of Value Moisture: 325 mg/kg ± 14 mg/kg"

"The reference value and uncertainty describe an interval which is at least a 95 % confidence interval for the moisture concentration in each ampoule of RM 8510. The uncertainty includes the uncertainty due to the interlaboratory comparison exercise measurements as well as a trend observed within the ampoule fill sequence.

Expiration of Reference Value: The reference value of RM 8510 is valid, within the measurement uncertainty specified, until December 31, 2027, provided the RM is handled in accordance with instructions given in this Report of Investigation (see Instructions for Use). This reference value is nullified if the RM is damaged, contaminated, or modified.

The coordination of the technical measurements was under the direction of S.A. Margolis and S.A. Wise of the NIST Analytical Chemistry Division.

Preparation and analytical measurements were performed by S.A. Margolis, J.C. Williams, and L.C. Sander of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.B. Schiller and L.M. Gill of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, analysis, and issuance of this RM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899
Certificate Issue Date: June 12, 1997*
8/21/95 (original certificate date)
*This revision reports an expiration date and editorially changed "recommended" to "reference" value.

Thomas E. Gills, Chief
Standard Reference Materials Program

INSTRUCTIONS FOR USE

Handling: The ampoules are sealed under dry argon. This material is hygroscopic; therefore, after opening the ampoules, extreme caution should be exercised to avoid contamination with moisture (e.g., dry measuring devices and containers must be used).

Storage and Use: Ampoules should be stored at room temperature. For the reference value to be valid within the stated uncertainty, samples of the material for analysis should be withdrawn from the ampoules immediately after opening, transferred into oven-dried measuring devices and used without delay. The reference value is not applicable to ampoules that have been stored after opening, even if the ampoules are resealed.

PREPARATION AND ANALYSIS

Preparation of RM 8510: Methanol, as supplied from the manufacturer, typically contains trace levels of water. The nominal moisture level was determined in several lots of HPLC-grade methanol and the appropriate lot was selected for ampouling. Therefore, the reference moisture value reflects the content after processing at NIST and not necessarily the moisture content when obtained from the manufacturer. Approximately 16 L (four bottles) of the selected lot were poured into a 18.9 L (5-gal) bottle containing dry argon, and the solution stirred for 30 min. The argon headspace above the liquid was maintained with argon from an attached Teflon[a] bag containing dry argon. As the level of the solution decreased in the bottle over the course of the ampouling, argon replaced the fluid. In this way a sealed system was maintained to minimize moisture contamination from the air. Aliquots of 5 mL were dispensed automatically into argon-filled, dried ampoules and then sealed.

Analytical Methods: An interlaboratory comparison exercise among 13 laboratories and NIST was conducted to determine the moisture content of RM 8510. All analyses in the interlaboratory comparison exercise were performed using coulometric Karl Fischer instrumentation and reagents routinely used in the participating laboratories. Each cooperating laboratory analyzed a single sample from each of two ampoules of RM 8510. The participating laboratories are listed below.

Homogeneity Study: Samples from 40 ampoules, selected throughout the fill sequence, were measured at NIST in a random order over four days using the volumetric Karl Fischer method. A statistically significant increase in moisture content with fill sequence was detected in the homogeneity study. A 13 mg/kg change in concentration was predicted by the homogeneity study over the entire population of ampoules of RM 8510 and was included in the total uncertainty of the reference value. The results from the volumetric Karl Fisher method were also used as an independent method to confirm the round robin results obtained by the use of the coulometric Karl Fisher method.

Participants in the RM 8510 Interlaboratory Comparison Exercise:

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J., Greer, United Power Services Inc., Nashville, TN
C., Smith, Cosa Instrument Co., Norwood, NJ
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G., Gauger, McGraw-Edison Power System, Franksville, WI
P., Griffin, Doble Engineering Co., Watertown, MA
R., Deering, Bonneville Power Administration, Vancouver, WA
D., Meyers, S.D. Meyers Inc., Tallmadge, OH
G., Thomas, Big River Electric Corp., Henderson, KY
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[1] Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for this purpose.