National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 83d

Arsenic Trioxide (As_2O_3) Reductometric Standard

This Standard Reference Material (SRM) consists of highly purified arsenic trioxide and is intended for use in reductometric standardization. It conforms to the American Chemical Society specifications for analytical reagent grade material, and meets the primary standard criteria of the Analytical Chemistry Section of the International Union of Pure and Applied Chemistry [1]. SRM 83d is supplied as a crystalline material in a unit of 60 g.

Reductometric Assay (mass fraction): 99.9926 ± 0.0030 Wt %

Uncertainty: The above uncertainty is a 95 % confidence interval for the mean, based on 11 degrees of freedom, and includes a contribution of 0.0008 Wt % from all known sources of possible systematic error.

Homogeneity: The material in this lot of arsenic trioxide is homogeneous within the bounds of the random error uncertainty of the measurement process.

Microscopic examination of the original material revealed the presence of particulate contamination. Therefore, the material was reprocessed by sieving through a 125 μm screen. Subsequent microscopic examination of the reprocessed material found no significant particulate contamination.

The concentration of antimony in this material, as determined spectrophotometrically, is 0.0015 Wt % ± 0.0004 Wt %.

Expiration of Certification: The certification of **SRM 83d** is valid, within the measurement uncertainty specified, until **26 December 2017**. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The experimental design and the coulometric analysis of the material were performed by G. Marinenko. The antimony content of the material was determined spectrophotometrically by B.I. Diamondstone of the NIST Analytical Chemistry Division. The microscopic examination of the material was made by E.B. Steel of the NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 08 September 2010

Robert L. Watters, Jr., Chief
Measurement Services Division

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NOTICE AND WARNINGS TO USER

Arsenic is classified as a poison, and should be handled with extreme care. Refer to the Material Safety Data Sheet for precautions for handling and use approved methods of disposal.

SOURCE, PREPARATION, AND ANALYSIS[1]

The material was obtained from the J.T. Baker Chemical Co., Phillipsburg, NJ, and was reprocessed by G. Marinenko and W.F. Koch of the NIST Analytical Chemistry Division.

Coulometric Assay: The certified reductometric assay of SRM 83d, Arsenic Trioxide, is based on the absolute coulometric oxidation of trivalent arsenic to pentavalent arsenic in 500 mg samples via electrogenerated iodine.

The relative molecular mass of As_2O_3 used in all calculations is 197.8412, the density used for calculating the air-buoyancy correction is 3.71 $g \cdot cm^{-3}$, and the value of the Faraday constant is 96486.5 A s mol^{-1}.

The electrochemical equivalent of As_2O_3, SRM 83d, was determined to be $0.512\,652 \pm 0.000\,017$ $mg \cdot C^{-1}$, where the uncertainty represents the 95 % confidence interval for the mean, based on 11 degrees of freedom.

The coulometric procedure for these titrations has been described by G. Marinenko and J.K. Taylor in reference 2.

Drying Instructions: The assay is based on material dried at 110 °C for 12 h, and stored in a desiccator over anhydrous magnesium perchlorate.

REFERENCES

[1] AMC; *Sodium Carbonate as a Primary Standard in Acid-Base Titrimetry*; Analytical Methods Committee of the Society for Analytical Chemistry; Analyst, Vol. 90, p. 251, (1965).

[2] Marinenko, G.; Taylor, J.K.; *High Precision Coulometric Iodimetry*; Anal. Chem., Vol. 39, No. 13, p. 1568 (1967).

Certificate Revision History: 08 September 2010 (Extension of certification period; editorial changes); 01 April 1995 (Editorial revisions to reflect program and organizational changes at NIST and at the Department of Commerce); 01 March 1982 (Original certificate issue date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.