



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 973

Boric Acid

(Acidimetric Standard)

This Standard Reference Material (SRM) is certified as a chemical of known assay and is intended for use as a primary acidimetric standard. The SRM consists of highly purified boric acid (H_3BO_3) in a 100 g bottle.

Certified Value and Uncertainty: Table 1 lists the certified mass fraction, $w_{\text{H}_3\text{BO}_3}$, of total acid expressed as H_3BO_3 . A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1].

Table 1. Certified Value for SRM 973 Boric Acid

$w_{\text{H}_3\text{BO}_3}$	100.009 % $\pm$ 0.010 %
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The certified value and uncertainty given in Table 1 were obtained by fitting a Bayesian hierarchical model [2] to data from a coulometric assay of a random sample of units of SRM 973. The model was fit using non-informative prior distributions for all parameters in the model. The model accounts for random variation arising from any material heterogeneity and all sources of uncertainty inherent to the coulometric assay technique. The model was fit to the data using Markov chain Monte Carlo methods implemented in the open-source software package WinBUGS [3]. The expanded uncertainty given in Table 1 is reported at the 95 % probability level. A combined standard uncertainty of $u_c = 0.0052$ %, effective degrees of freedom of $\nu_{\text{eff}} = 60$, and a coverage factor of $k = 2$ should be used in any subsequent uncertainty calculations made using the methods described in the ISO and NIST Guides [4].

Expiration of Certificate: The certification of this SRM is valid, within the measurement uncertainty specified, until **01 April 2022**, provided the SRM is handled in accordance with instructions given in this certificate (see “Instructions for Use”). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

Coordination of the technical measurements leading to the certification of this SRM was provided by K.W. Pratt.

Coulometric analyses were performed in the NIST Analytical Chemistry Division by K.W. Pratt.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

Support aspects involved with the certification and 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: 21 April 2008

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

NOTICE AND WARNINGS TO USERS

Stability and Storage: This SRM should be stored in its original bottle at room temperature. It must be tightly re-capped after use and protected from moisture and light. The material absorbs water from the air if stored at a relative humidity above 50 %. If exposed to humid air, it should be equilibrated at room temperature for at least 24 h with air of 30 % to 40 % relative humidity to restore the composition of the material as certified. A constant relative humidity of 33 % at room temperature is conveniently obtained in a humidistat consisting of a conventional desiccator in which a saturated aqueous solution of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ containing excess solid $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ replaces the usual desiccant [5].

Homogeneity: Tests indicate that this SRM is homogeneous within the uncertainty limits for sample sizes greater than 200 mg. Samples less than 200 mg are not recommended.

Isotopic Composition: The isotopic composition ($^{10}\text{B}/^{11}\text{B}$ ratio) of this material has not been investigated. The certified acidimetric assay of SRM 973 is calculated assuming mean natural abundance (molar mass of H_3BO_3 equal to 61.8330 g/mol [6]).

Density: The density of SRM 973 was taken as 1.4844 g/cm^3 in the correction for air buoyancy associated with weighing the material. This value was obtained from pycnometric measurements of SRM 973 using hexane as the inert fluid. SRM 973 is not intended as a density standard. This density is an information value only [1].

INSTRUCTIONS FOR USE

Drying and Weighing Instructions: Use as received.

Source of Material: The boric acid used for this SRM was obtained from a commercial company. The material was examined for compliance with the specification for reagent grade H_3BO_3 as specified by the American Chemical Society [7]. The material was found to meet or exceed the minimum requirements in every respect.

Coulometric Assay: This material was assayed by automated coulometric titration [8], to the weak acid endpoint (ca. pH 8.6) in 0.375 mol/kg mannitol [9]. The titrations were performed in the absence of CO_2 in an argon atmosphere.

REFERENCES

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- [8] Pratt, K.W.; *Automated, High-Precision Coulometric Titrimetry Part I. Engineering and Implementation*; *Anal. Chim. Acta*, Vol. 289, pp. 135–142 (1994).
- [9] Marinenko, G; Champion C.E.; *J. Res. Nat. Bur. Stand.*, Vol. 75A, pp. 421–427 (1971).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; email srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.