National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3172a

Multielement Mix B-1

Lot No. 891908

This Standard Reference Material (SRM) is intended primarily for use in calibrating instruments used in atomic spectrometry, including atomic absorption spectrometry, inductively coupled plasma optical spectrometry, and inductively coupled plasma mass spectrometry. It can also be used in conjunction with any other analytical technique or procedure where an aqueous standard solution is required. One unit of SRM 3172a consists of 50 mL of a multielement solution in a high density polyethylene bottle. The solution is prepared gravimetrically to contain known amounts of 13 elements in an approximate nitric acid volume fraction of 5 %.

The certified values (Y) given in Table 1 are based 1) on gravimetric preparation and, 2) on inductively coupled plasma optical spectrometry calibrated using three independently prepared gravimetric solutions. Elemental impurities (metallic and gaseous) in the starting materials were determined by inductively coupled plasma mass spectrometry or by glow discharge mass spectrometry, oxygen and nitrogen by vacuum fusion, and hydrogen by vacuum extraction. The impurity levels found are listed in Table 2. Each certified value has been adjusted upward by 0.1 % relative based on estimated transpiration losses of solvent through the container walls of 0.2 % relative per year.

The uncertainty in each certified value is calculated as

$$U = (2u_c + 0.001\ Y + B)\ \mu g/g$$

where u_c is the "combined standard uncertainty" calculated according to the ISO Guide [1] and the quantity, B, is calculated using the procedure of Schiller and Eberhardt [2]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with the gravimetric preparation and the analytical determinations. The quantity, $0.001\ Y$, is an allowance for transpiration of the solution through the container walls, which is estimated to be ± 0.1 % of the certified value during the period of validity of the certificate. The quantity, B, is an allowance for between method differences.

Expiration of Certification: The certification of **SRM 3172a Lot No. 891908** is valid, within the measurement uncertainty specified, until **01 October 2000**, provided the SRM is handled in accordance with instructions given in this certificate (see Instructions for Use). This certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of Certification: NIST will monitor representative solutions from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Statistical consultation was provided by K.R. Eberhardt 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 N.M. Trahey.

Gaithersburg, MD 20899
Certificate Issue Date: 15 December 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

This SRM was prepared gravimetrically by T.A. Butler and analyzed using inductively coupled plasma optical spectrometry by M.L. Salit and A. Lindstrom of the NIST Analytical Chemistry Division. Inductively coupled plasma mass spectrometric analysis of several starting materials was performed by G.C. Turk of the NIST Analytical Chemistry Division. Glow discharge mass spectrometric analysis of several starting materials was performed by Shiva Technologies Inc., Cicero, NY. Gas analysis of several starting materials was performed by Luvak Inc., Boylston, MA.

Table 1. Certified Values (Y) for SRM 3172a

Element	Mass Fraction, μg/g
Arsenic	200.5 ± 2.5
Barium	9.9 ± 0.1
Calcium	10.4 ± 0.1
Cobalt	110.3 ± 0.9
Copper	102.4 ± 1.0
Lead	102.6 ± 0.9
Selenium	507.6 ± 11.1
Silver	106.4 ± 1.3
Strontium	10.5 ± 0.2
Thallium	100.4 ± 1.6
Zinc	104.9 ± 1.3

Table 2. Impurity Levels in SRM 3172a Starting Materials

Material	Metal Impurities, μg/g	Dissolved Gases, μg/g	Method
Arsenic Metal	<75	<375	GDMS, VF, VE
Barium Carbonate	<20	—	ICP-MS
Calcium Carbonate	—	—	SRM 915a
Cobalt Metal	<15	<101	ICP-MS, VF, VE
Copper Metal	<50	<920	ICP-MS, VF
Lead Metal	<5	<55	GDMS, VF, VE
Selenium Metal	—	—	SRM 726
Silver Metal	—	—	SRM 748
Strontium Carbonate	—	—	*
Thallium Metal	<125	<250	GDMS, VF, VE
Zinc Metal	—	—	SRM 740a

*Information provided by supplier.

ICP-MS - Inductively coupled plasma mass spectrometry
GDMS - Glow discharge mass spectrometry
VF - Vacuum fusion
VE - Vacuum extraction

Instructions for Use

This SRM solution should be kept tightly capped and stored under normal laboratory conditions when not in use.

Preparation of Working Standard Solutions by Mass: Each diluted working standard solution should be prepared by transferring an aliquot of the SRM to an empty, dry, preweighed polyethylene bottle, and then reweighing the bottle. An appropriate dilute acid must be added by mass to bring the solution to the desired dilution. The daily working solutions from which additional dilutions are made, should be approximately 10 μg/kg to 100 μg/kg. The dilution need not be exact since the mass of the empty bottle, mass of the bottle plus SRM aliquot, and the final diluted mass of the solution will permit calculation of the exact concentration of the working solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true concentration in vacuum. Dilute SRM solution concentration will be in μg/kg units. Volumetric dilutions are not recommended due to uncertainties in volume calibrations and variations in density. However, for user convenience, a procedure for volumetric preparation that will minimize the major sources of error, is given below.

Preparation of Working Standard Solutions by Volume: Each diluted working standard solution should be prepared by transferring an aliquot of the SRM to an empty, dry polyethylene bottle and then weighing the bottle. The solution must now be transferred to a Class A volumetric flask and the polyethylene bottle reweighed to determine the exact mass of SRM solution transferred. The solution in the flask is then diluted to 99 % + of volume using an appropriate dilute acid, mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in μg/mL) of the resulting diluted working standard solution can then be calculated by multiplying the mass (in g) of the SRM solution amount by the SRM certified value (in μg/g), and dividing the numerical product by the calibrated volume (in mL) of the flask used for dilution. If the analyst follows this procedure, no correction for density is needed and although the concentration of the resulting working standard solution may be an uneven fraction of the original SRM concentration, it will be known as accurately as a volumetric dilution permits.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, lst 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 DC, (1994).

[2] Schiller, S.B. and Eberhardt, K.R., Combining Data From Independent Chemical Analysis Methods, Spectrochimica Acta, **46B**, pp. 1607-1613, (1991).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.