National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3113

Standard Solution

Cobalt

Lot No. 891102

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 3113 consists of 50 mL of a single element solution in a high density polyethylene bottle. The solution is prepared gravimetrically to contain a known amount of cobalt in an approximate nitric acid volume fraction of 10 %.

Certified Value (Y) of Cobalt: 10.00 mg/g $\pm$ 0.03 mg/g

The certified value (Y) is based 1) on gravimetric preparation and, 2) inductively coupled plasma optical emission spectrometry using three independently prepared gravimetric solutions. Metallic elemental impurities in the cobalt metal starting material were determined by glow discharge mass spectrometry, oxygen and nitrogen by inert gas fusion, hydrogen by vacuum extraction. The material was found to contain less than 710 mg/kg total metallic impurities and 105 mg/kg dissolved gases. The 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 the certified value is calculated as

$$U = (2u_c + 0.001Y + B) \text{ mg/g}$$

where u_c is the "combined" standard uncertainty calculated according to the ISO Guide [1] and the procedure of Schiller and Eberhardt for combining independent analytical methods [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, $0.001Y$, is an allowance for transpiration of the solution through the container walls, which is estimated to be $\pm$ 0.1 % of the certified value during the one-year period of validity of the certification. The quantity, B, is an allowance for between method differences.

Expiration of Certification: The certification of **SRM 3113 Lot No. 891102** is valid, within the measurement uncertainty specified, until **01 April 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.

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: 28 May 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 emission spectrometry by M.L. Salit and A. Lindstrom of the NIST Analytical Chemistry Division. Glow discharge mass spectrometric analysis of the starting material was performed by Shiva Technologies, Inc., Cicero, NY. Gas analysis was performed at Luvak, Inc., Boylston, MA.

Statistical consultation was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

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 mg/kg to 100 mg/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 mg/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 mg/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 mg/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., Spectrochimica Acta, **46B,** pp. 1607-1613, (1991).

It is the responsibility of users of this SRM to assure 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.