National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 113b

Zinc Concentrate

This Standard Reference Material (SRM) is intended primarily for use in the analysis of zinc sulfide ores and concentrates. SRM 113b is in the form of a fine powder, and is packaged in an inert argon atmosphere to prevent air oxidation of the material, which would lead to changes in the certified values over time [1,2]. A unit consists of 100 g of the powdered concentrate.

The certified values for SRM 113b are given in Table 1. The values are based on measurements using isotope dilution mass spectrometry (IDMS) and titrimetry for the analysis of zinc, and on IDMS only for the remaining elements. All values are based on measurements using a sample weight of 100 mg, with correction to the dry weight basis (see Instructions for Drying).

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification is valid for 5 years from the date of shipment from NIST, provided that the samples are maintained in an inert atmosphere. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Return the attached registration card to facilitate notification.

Handling and Storage: Exposure to air must be limited to the minimum time required for weighing of the analytical subsample. After each weighing, the sample bottle should be flushed with argon or nitrogen and capped immediately. The capped bottle should be placed in a desiccator, which is then flushed with the same gas and maintained in the inert gas atmosphere; see instructions on label.

Use: A minimum sample weight of 100 mg should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

Instructions for Drying: Drying should be done at 105 °C for 1 h on a separate subsample, and the analytical result should be corrected to the dry weight basis, using the measured weight loss. Typically this weight loss as measured during certification was approximately 0.05%.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the direction of J.D. Fassett of the NIST Analytical Chemistry Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.S. Kane.

Gaithersburg, MD 20899
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Thomas E. Gills, Chief
Standard Reference Materials Program

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Source and Preparation of Material: SRM 113b is a re-issue of SRM 113a, which had air-oxidized non-uniformly among bottles since its original bottling. After blending all stock to homogenize the material, it was rebottled in an argon -filled glove box. The open bottles stood overnight in the argon atmosphere before being capped in the glove box. All bottles were then placed in aluminized barrier bags, this process also taking place in the argon-filled glove box. Each bag was heat-sealed immediately after being removed from the glove box.

SRM 113a was initially supplied by Cominco American Inc., Spokane, WA, and prepared at the Magmont Mines, Bixby, MO.

Analysis: The homogeneity of the material was assessed for all elements certified by comparing the total analytical uncertainty of analysis by isotope dilution mass spectrometry (IDMS) with the mass ratio measurement uncertainty as determined from spike calibration solutions measured in the same analytical sequence as the SRM samples. Heterogeneity was clearly detected for Cu and was borderline for Mg; for both elements it is included in the uncertainty of the certified value (see below).

Certification analyses were performed at NIST using an anion exchange separation with an EDTA titration finish and ID- thermal ionization mass spectrometry (ID-TIMS) for Zinc; ID-TIMS for S, Fe, Ca, and Mg; and using ID-inductively coupled plasma mass spectrometry for Cd, Cu, Ag, and Pb. Mercury was determined using flow injection atomic absorption spectrometry; the Hg concentration is not certified, and is provided for information only. In addition to certification analyses performed at NIST, corroborating analytical data were also provided by USGS for Zn by titrimetry, and by CANMET for Zn, Fe, and Cu.

Certified Values and Uncertainties: The certified and information only values given in Table 1 are based on NIST data only. The uncertainties were calculated according to the ISO Guide [3]. Each uncertainty includes both measurement varability and material variability, and has an approximate level of confidence of 95%. The uncertainties for Cu and Mg also include estimates of material variability.

Table 1. Certified Values

Element	Mass Fraction as Percent (g /100 g)
Zinc	56.49 ± 0.10
Sulfur	30.032 ± 0.057
Cadmium	0.7804 ± 0.0013
Calcium	0.8196 ± 0.0039
Copper	0.2953 ± 0.0094
Iron	2.077 ± 0.028
Lead	2.731 ± 0.021
Magnesium	0.4460 ± 0.0065
Silver	0.04607 ± 0.00006
Mercury*	(0.55 μg/g)

*The mercury concentration is not certified, and is provided for information only.

Analysts, National Institute of Standards and Technology:

1. E.S. Beary
2. C.M. Beck II
3. M.S. Epstein
4. J.D. Fassett
5. W.R. Kelly
6. J.R. Moody
7. P.J. Paulsen
8. M. Rearick
9. R. Saraswati
10. R.D. Vocke, Jr.

Participating Collaborating Laboratories:

U. S. Geological Survey, Reston, VA; A.F. Dorrzapf, Jr. and J.M. Marinenko
CANMET, Ottawa, Canada; M. Leaver and R. McCorkell

REFERENCES

[1] Faye, G.H. and Steger, H.F., Talanta, **26**:309-315, (1979).
[2] Steger, H.G. and Sutarno, R., Geostand. Newsl. **10**:93-98, (1986).
[3] *"Guide to Expression of Uncertainty in Measurement"*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993).