National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2430

Scheelite Ore

This Standard Reference Material (SRM) is primarily intended for use in the validation of chemical and instrumental methods of analysis of tungsten-bearing ores and other materials of similar matrix for elemental content. It can be used to validate value assignment of in-house reference materials. A unit of SRM 2430 consists of a bottle containing approximately 100 g of powder ground to pass a 150 mesh sieve (< 0.1 mm).

Certified Mass Fraction Values: The certified mass fraction values [1] for constituents in SRM 2430 are listed in Table 1. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. 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 taken into account. A certified value is the present best estimate of the true value.

Reference Mass Fraction Values: Reference mass fraction values for SRM 2430 are listed in Table 2. A reference value is a non-certified value that is the present best estimates of the true values; however, the value does not meet the NIST criteria for certification [2] and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods.

Information Mass Fraction Values: Information mass fraction values for SRM 2430 are listed in Table 3. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 2430** is valid indefinitely, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. 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, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements for the original certification of SRM 2430 was performed by J.I. Shultz formerly of the National Bureau of Standards (NBS). Review and revision of value assignments were performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 2430 were performed by P.A. Pella and Z. Wang of the NIST Chemical Sciences Division. Additional measurements were performed by collaborating laboratories: J. Madera and R. Stouder, Amax Molybdenum Division (Ft. Madison, IA); F.F. Pitard Amax Extractive Research and Development, Inc. (Golden, CO); J.W. Fulton, General Electric Company (Cleveland, OH); C. Terry, Kennametal Inc. (Fallon, NE); S. Kallmann, C.L. Maul, and E.W. Hobart, Ledoux & Co. (Teaneck, NJ); R. Dyck, M. Saffer, and J. Mras, Sylvania GTE Products Corp. (Towanda, PA).

Statistical consultation for this SRM was provided by D.D. Leber of the NIST Statistical Engineering Division.

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Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
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Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR STORAGE AND USE

To relate analytical determinations to the values in this Certificate of Analysis, a minimum sample quantity of 100 mg is recommended. The mass fraction values are based on samples dried for 2 h at 105 °C. The material should be stored in its original container in a cool, dry location.

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

PREPARATION AND ANALYSIS[(1)]

The material for the preparation of this SRM was provided by Amax Tungsten, and represents a mixture of two tungsten concentrates. Preparation and preliminary homogeneity testing were performed at Ledoux & Co., and the material was found to be acceptable. The material was blended and bottled at NIST.

Certified Mass Fraction Values: The measurands are the mass fractions of the elements in tungsten concentrate. Tungsten and calcium, they are expressed as the oxides by industry consensus. The certified values are metrologically traceable to the derived SI unit for mass fraction, expressed as percent (%). The values in Table 1 are the weighted means of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [3–5] and the DerSimonian-Laird procedure [6,7]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [8–11]. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effects of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. The expansion factor, k, corresponds to an approximately 95 % confidence level.

Table 1. Certified Mass Fraction Values for SRM 2430 Scheelite Ore

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor (k)
Tungsten Oxide (WO_3)	70.30	0.16	2.16
Calcium Oxide (CaO)	19.44	0.38	2.18
Silicon (Si)	1.74	0.22	2.20
Potassium (K)	0.179	0.023	2.22
Manganese (Mn)	0.1178	0.0049	2.20

Reference Mass Fraction Values: The measurands are the mass fractions of the elements in tungsten concentrate, as determined by the methods indicated in Table 4. The reference values are metrologically traceable to the derived SI unit for mass fraction, expressed as percent (%). The values in Table 2 are the weighted means of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [3–5] and the DerSimonian-Laird procedure [6,7]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [8–11]. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effects of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. The expansion factor, k, corresponds to an approximately 95 % confidence level.

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

Table 2. Reference Mass Fraction Values for SRM 2430 Scheelite Ore

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor (*k*)
Arsenic (As)	0.0022	0.0007	2.32
Bismuth (Bi)	0.080	0.009	6.29
Copper (Cu)	0.0086	0.0014	2.18
Iron (Fe)	1.13	0.13	2.21
Molybdenum (Mo)	0.22	0.03	2.39
Sodium (Na)	0.018	0.005	2.62
Sulfur (S)	0.25	0.04	3.70

Information Mass Fraction Values: In Table 3, the values for the listed elements represent the result of one method or the estimated limits of detection of the applied test methods.

Table 3. Information Mass Fraction Values for SRM 2430 Scheelite Ore

Constituent	Mass Fraction (%)
Aluminum (Al)	0.4
Antimony (Sb)	< 0.005
Fluorine (F)	1.3
Magnesium (Mg)	0.5
Niobium (Nb)	< 0.02
Phosphorus (P)	0.02
Tantalum (Ta)	< 0.06

Table 4. Analytical Methods Used for SRM 2430 Scheelite Ore

Constituent	Methods[(a)]	Constituent	Methods[(a)]
Aluminum	16	Molybdenum	3, 12, 15
Antimony	3, 17	Niobium	16
Arsenic	3, 17	Phosphorus	4
Bismuth	3, 7, 16	Potassium	1, 3
Calcium	1, 14	Silicon	1, 3, 10
Copper	3, 12	Sodium	3, 16, 17
Fluorine	6	Sulfur	5, 11
Iron	1, 3, 8	Tantalum	13, 17
Magnesium	16	Tungsten	1, 2, 7
Manganese	1, 3, 9		

[(a)] Key to Methods in Table 4:

1. X-ray fluorescence spectrometry (XRF)
2. Gravimetry by cinchonine precipitation
3. Atomic absorption spectrometry with standard additions
4. Phosphomolybdate colorimetric method
5. Combustion with infrared detection
6. Ion selective electrode titration after steam distillation
7. Direct current plasma optical emission spectrometry
8. Titration with $K_2Cr_2O_7$
9. Titration with $FeSO_4 \cdot 7H_2O$
10. Precipitation in acid solution and volatilization with HF
11. Oxidation to sulfate and precipitation with $BaCl_2$
12. KOH fusion and spectrometric determination
13. Extraction with methyl-isobutyl ketone and spectrometric determination
14. Titration method
15. Photometric method
16. Spark source optical emission spectrometry (SS-OES)
17. Spark source mass spectrometry (SSMS)

REFERENCES

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Certificate Revision History: 13 July 2016 (Information values revised to reference values for Cu, Fe, and Na, and to certified values for Mn and K; certified values revised to reference values for As, Bi, Mo, and S, and to an information value for P; added certified values for CaO and Si; removed certified value for SiO_2; editorial changes); 02 January 1987 (Original certificate 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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.