National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 423

Molybdenum Oxide Concentrate
(Powder Form)

This Standard Reference Material (SRM) is a molybdenum oxide (MoO_3) concentrate from a commercial mining and refining process. SRM 423 is intended for use in the evaluation of chemical and instrumental methods of analysis. It can be used to validate value assignment of in-house reference materials. A unit of SRM 423 consists of one pouch containing approximately 60 g of powder.

Certified Mass Fraction Values: Certified values for constituents of SRM 423 are reported in Table 1 as mass fractions [1] of the elements in molybdenum oxide on the as-received basis. 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 taken into account [2]. The certified values are metrologically traceable to the SI derived unit of mass fraction (expressed as percent). The expanded uncertainty estimates are expressed at a confidence level of approximately 95 %.

Table 1. Certified Mass Fraction Values for SRM 423 Molybdenum Oxide Concentrate (Powder Form)

Constituent	Mass Fraction (%)
Copper (Cu)	0.0640 ± 0.0028
Molybdenum (Mo)	58.61 ± 0.13

Expiration of Certification: The certification of **SRM 423** is valid, within the measurement uncertainty specified, until **01 July 2026**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Handling, Storage and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

Coordination of the technical measurements for certification of SRM 423 was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Statistical consultation for the value assignment of SRM 423 was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 10 January 2018
Certificate Revision History on Last Page

Stephen J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE AND USE

The pouch of powder should be kept sealed, as delivered, until it is needed for the first time. It is recommended that the pouch be opened by cutting straight across at the heat-sealed end. When not in use, the material should be stored in its original pouch placed inside a tightly sealed container. It is recommended that the open end be folded once or twice and clipped shut. The user is cautioned to use care when deciding whether to transfer the contents of a pouch to a new container as this may contaminate the SRM. The material is certified on the as-received basis. No preparation is needed prior to analysis. The minimum recommended sample mass for a single determination is 0.9 g.

To use the uncertainty estimates given in this certificate, divide the expanded uncertainty by $k = 2$ to obtain the combined standard uncertainty. The effective degrees of freedom of the combined standard uncertainty are ≥60.

PREPARATION AND ANALYSIS[(1)]

All material was dried at 105 ºC to a constant mass, then de-oiled with repeated washes of acetone to achieve a constant mass. At Highland Valley Copper, all material was screen classified to particle sizes <53 μm (100 % passing 270 mesh) with all oversize material removed. Dry, volatiles-free material was blended and sealed in foil-covered plastic pouches to maintain future integrity of the materials. Measurements for homogeneity testing of SRM 423 were performed at NIST using X-ray fluorescence spectrometry.

Measurements for value assignment of SRM 423 were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Chemical Sciences Division. Additional analytical determinations for value assignments of the SRM 423 were performed by D. Lincoln, P. Martin, D. Enders, T. Havers, D. Howard, K. Heaton, N. Woods, J. Mihalech, D. Arbuckle, D. Leavitt, M. Desjardine, and D. Comte, Highland Valley Copper (Logan Lake, BC, Canada); R. Fraser and K. Alexander, Roca Mines, Inc. (Trout Lake, BC, Canada); N. Hampson, N. Golborne, P. Ritson, G. Smith, T. Sutcliffe, S. Williams, Alfred H. Knight International Ltd. (St. Helens, United Kingdom); D. Court, Alex Stewart Assayers, Ltd. (Liverpool, United Kingdom); L. Longacre, P. Schubert, M. Souder, N. Eppley, R. Eakin, J. Davies, Andrew S. McCreath and Son, Inc. (Harrisburg, PA); B. Minor, K. Campo, and J. Lynch, Endako Mines (Fraser Lake, BC, Canada); A. Iniestra Ramírez, S. Rodríguez Salas, G. Morales Martínez, F. Hernández Martínez, Ersa Global Mex, S.A. de C.V. (Torreón, Coahuila, Mexico); O. Baca, Freeport McMoRan Process Tech. Center, (Safford, AZ); F. Liberato and D. Porchiran, Langeloth Metallurgical, Co. (Langeloth, PA); V. Gilman, J. Gress, R. Ball, B. McGee, Montana Resources, LLP (Butte, MT); and C. Whipple, W. Barragan, J. Margues, P. Robinson, T. Young, D. McGhee, R. Lemos, Robinson Nevada Mining Co. (Ruth, NV). Seventeen additional laboratories provided data for value assignment but declined to be identified.

The following test methods were employed at NIST and the collaborating laboratories.

Gravimetry by the $PbMoO_4$ method:	Mo
Gravimetry by the α-benzoin oxime method:	Mo
Titrimetry by the $KMnO_4$ method after reduction to Mo^{+2}:	Mo
X-ray fluorescence spectrometry:	Fe, Cu, Mo, Re, Pb
Inductively coupled plasma optical emission spectrometry:	Fe, Cu, Re, Pb
Combustion with infrared detection	S
Acid-insoluble residue after HCl dissolution	

For molybdenum and acid insoluble residue, the assigned value is the weighted mean of a set of results obtained using the test methods listed above [3]. The uncertainty of the value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide [4] as $U = ku_c$, where u_c is calculated, at the level of one standard deviation, by combining a between-method variance and a pooled, within-method variance. The coverage factor, $k = 2$, was chosen to approximate a 95 % confidence level [4].

For copper, iron, and lead, the assigned value is the median of the set of results obtained by the NIST and collaborating laboratories using the test methods listed above. The uncertainty of the value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide [4] as $U = ku_c$, where u_c is calculated, at the level of one standard deviation, by combining a between-method variance, estimated from the median absolute deviation of the laboratory mean results from the median, with the pooled standard deviations of the laboratory means.

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

The median absolute deviation was expanded by a factor of 1.483 because it underestimates the true standard uncertainty of a data set. The coverage factor, $k = 2$, was chosen to approximate a 95 % confidence level [5].

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 423.

Reference Mass Fraction Values: Reference values for constituents in SRM 423 are reported in Table 2. Reference values are non-certified values that are the present best estimates of the true values based on available data; however, the values do not meet the NIST criteria for certification and are 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 statistical agreement among multiple analytical methods [2].

Table 2. Reference Mass Fraction Values for SRM 423 Molybdenum Oxide Concentrate (Powder Form)

Constituent	Mass Fraction (%)		
Iron (Fe)	1.708	±	0.055
Lead (Pb)	0.0433	±	0.0030
Acid-Insoluble Residue	7.69	±	0.33

Information Mass Fraction Values: Information values for constituents in SRM 423 are reported as mass fractions in Table 3. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Information values cannot be used to establish metrological traceability. The information values were determined using either flame atomic absorption spectrometry or inductively coupled plasma optical emission spectrometry.

Table 3. Information Mass Fraction Values for SRM 423 Molybdenum Oxide Concentrate (Powder Form)

Constituent	Mass Fraction (%)
Ag	0.0029
Bi	0.006
C	0.025
Ca	0.10
Cr	0.0034
Mg	0.10
Mn	0.009
Na	0.2
Re	0.004
S	0.063
Sb	0.0024
V	0.0023
Zn	0.017

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.

REFERENCES

[1] Thompson A., Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*, NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at http://www.nist.gov/pml/pubs/index.cfm/ (accessed Jan 2018).

[2] May, W.; Parris, R.; Beck, C.M.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurement*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Jan 2018).

[3] Vangel, M.G.; Rukhin, A.L.; *Maximum likelihood Analysis for Heteroscedastic One-Way Random Effects ANOVA in Interlaboratory Studies*; Biometrics, Vol. 55, No. 1, pp. 129-136 (1999).

[4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2018); see also Taylor, B.N.; 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); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Jan 2018).

[5] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).

Certificate Revision History: 10 January 2018 (Change of expiration date; editorial changes); 17 February 2010 (Original certificate).

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.