National Institute of Standards and Technology
U.S. Department of Commerce

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Standard Reference Material® 423
Molybdenum Oxide Concentrate
(Powder Form)
CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are 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.

Description: This SRM is a molybdenum oxide (MoO_3) concentrate from a commercial mining and refining process. A unit of SRM 423 consists of one pouch containing approximately 50 g of powder.

Certified 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

Non-Certified Values: Non-certified values are provided in the Appendix A.

Additional Information: Values of potential interest to users and additional information are provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 423** are valid within the measurement uncertainty specified until **01 July 2026**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

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Safety: Consult the Safety Data Sheet (SDS), enclosed with the SRM shipment, for chemical hazard information.

Storage: The pouch of powder should be kept sealed, as delivered, until it is needed for the first time. Store the SRM in a dry location (relative humidity ≤50 %) under normal laboratory conditions. 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.

Use: 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.

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 https://www.nist.gov/pml/pubs/index.cfm/ (accessed May 2023).

[2] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology: Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed May 2023).

[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* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed May 2023); 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 https://www.nist.gov/pml/pubs/index.cfm/ (accessed May 2023).

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

Certificate Revision History: 08 May 2023 (Updated format; editorial changes); 12 February 2019 (Updated unit size; editorial changes); 10 January 2018 (Change of expiration date; editorial changes); 17 February 2010 (Original certificate).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher-order reference system. Non-certified mass fraction values are provided below in Table A1.

Table A1. Non-Certified 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

Period of Validity: The non-certified values delivered by **SRM 423** are valid within the measurement uncertainty specified until **01 July 2026**. The values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate of Analysis and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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APPENDIX B

Values of potential interest to users: Values for constituents in SRM 423 are reported as mass fractions in Table B1. The values were determined using either flame atomic absorption spectrometry or inductively coupled plasma optical emission spectrometry.

Table B1. Mass Fraction Values for SRM 423 Molybdenum Oxide Concentrate (Powder Form)

Constituent	Mass Fraction (%)
Silver (Ag)	0.0029
Bismuth (Bi)	0.006
Carbon (C)	0.025
Calcium (Ca)	0.10
Chromium (Cr)	0.0034
Magnesium (Mg)	0.10
Manganese (Mn)	0.009
Sodium (Na)	0.2
Rhenium (Re)	0.004
Sulfur (S)	0.063
Antimony (Sb)	0.0024
Vanadium (V)	0.0023
Zinc (Zn)	0.017

Preparation: 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.

Analysis: 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/JCGTM 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/JCGTM 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. 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].

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