

Procedure for Assaying

Molybdenite Concentrates

2023 update of 2003 original

Introductory remarks

Developed and published by the International Molybdenum Association (IMOA), this good practice Guideline for Assaying Molybdenite Concentrates is one of a six-part series on weighing, sampling and assaying.

This updated second edition continues to detail a gravimetric analysis guideline procedure specifically designed for the determination of total molybdenum in molybdenite concentrates. However, since the first edition of this guideline in 2003, X-Ray Fluorescence spectroscopy (XRF) is now a well-established, equally acceptable analytical technique used to identify and quantify the chemical composition of a wide range of materials, including mineral concentrates. Therefore a brief overview of XRF is now included on page 11.

Downloadable from the IMOA website, the full series includes:

Original 2003 version:

- [Procedure for the Weighing and Sampling of Molybdenite Concentrates](#)
- [Procedure for the Weighing and Sampling of Technical Grade Molybdenum Oxide](#)
- [Procedure for the Weighing and Sampling of Ferromolybdenum](#)

Updated 2023 version:

- [Procedure for Assaying Molybdenite Concentrates](#)
- [Procedure for Assaying Technical Grade Molybdenum Oxide](#)
- [Procedure for Assaying Ferromolybdenum](#)

Procedure for the Chemical Analysis of Molybdenite Concentrates



WARNING: This Guideline Procedure involves hazardous materials, operations and equipment. It is the responsibility of the user of this Guideline Procedure to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope and Field of Application

This guideline procedure specifies a chemical method which is specifically designed to be applicable for the determination of total molybdenum in molybdenite concentrates, within the normal ranges of this material.

2 Principle

Oxidation of sample, dissolution of molybdenum using ammonia, separation of iron and silica by filtration, prior to precipitation, and gravimetric determination as lead molybdate.

Tungsten and vanadium interfere and the procedure may require correction for these to the final figure, as they co-precipitate with the lead molybdate, causing enhancement of the final molybdenum assay.

3 Reagents

Use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

3.1 Hydrochloric acid (1.18 S.G.)

3.2 Nitric acid (1.46 S.G.)

3.3 Sulphuric Acid (1 + 1):

Mix one volume of sulphuric acid (1.84 S.G.) with one volume of water, always adding the acid **slowly** to the water, with continuous mixing and cooling. The reaction is exothermic, giving off great heat, so it is important that the addition is done slowly and with care.

3.4 Ammonia solution (0.88 S.G.)

3.5 Ammonia wash solution (20%):

Dilute 200 ml ammonia solution (3.4) to 1000 ml with water.

3.6 Hydrogen peroxide (20 vol %)

3.7 Glacial acetic acid (1.05 S.G.)

3.8 Lead acetate solution:

Dissolve 40 g of lead acetate in 500 ml water. Add 5 ml glacial acetic acid (3.7), and dilute to 1000 ml with water.

3.9 Ammonium acetate solution (20%):

Dissolve 200 g of ammonium acetate in 500 ml water. Add 10 ml of glacial acetic acid (3.7) and dilute to 1000 ml with water.

3.10 Ammonium acetate wash solution:

Dilute 200 ml of ammonium acetate solution (3.9) to 1000 ml with water.

3.11 Hydrochloric acid wash solution:

Dilute 500 ml of hydrochloric acid (3.1) to 1000 ml with water.

3.12 Methyl orange:

Dissolve 1 g of methyl orange in 100 ml of water.

3.13 Ammonia solution (1 + 1):

Dilute 500 ml of ammonia solution (3.4) to 1000 ml with water.

3.14 Litmus Paper

4 Apparatus

- a** Usual laboratory equipment, including an analytical four decimal place balance. The filtrations at steps 6.8 and 6.10 may be modified to accept either filtration by pulp pad or vacuum filtration. In the latter case a hardened paper is required.
- b** Filter papers: 110 mm No. 54 Whatman filter paper or equivalent
150 mm No. 40 Whatman filter paper or equivalent

5 Sample

The sample is to be obtained as per the “IMO A Guideline Procedure for the Weighing and Sampling of Molybdenite Concentrates”, ensuring the sample passes through a 100 mesh (ASTM) sieve (0.15 mm aperture), and mixed well prior to removal of the test portions to be assayed. The afore-mentioned procedure produces a hermetically sealed sample, the contents of which are to be assayed in their packed state (point 4.4.2 refers).

Analyses to be performed at least in duplicate.

6 Procedure

- 6.1** Accurately weigh (**W1**) to four decimal places approximately 0.5 g of sample and transfer to a 400 ml squat form glass beaker.
- 6.2** Carefully add 15 ml nitric acid (3.2), swirl to disperse the sample, cover beaker with a watch glass and place it on a hot plate. When the reaction has ceased, remove from hotplate, add 10 ml sulphuric acid (3.3), return to hotplate and take to fumes of sulphuric, cool, add an additional 3–4 ml nitric acid (3.2), dropwise, and re-heat to strong fumes. If any dark particles remain on the meniscus, repeat the procedure with nitric acid (3.2). Take to dryness.

- 6.3** Remove from the hotplate, cool, add 15 ml hydrochloric acid (3.1), heat to boiling for two minutes, rinse down the sides with 40 ml of hot water, and with care add 1 ml hydrogen peroxide (3.6) and a small amount of paper pulp. Re-boil and whilst stirring, carefully add ammonia solution (3.4) until ammoniacal, testing for this with litmus paper (3.14), and boil for 2–3 minutes.
- 6.4** Filter through a 110 mm No. 54 Whatman filter paper or equivalent, into a 1000 ml tall-form beaker. Wash the precipitate on the filter paper twice with hot ammonia wash solution (3.5) followed by 5–6 washings with hot water. Reserve the filtrate.
- 6.5** Without opening the filter paper, using a stream of hot water, wash the iron precipitate off the paper and back into the original beaker. Repeat stages 6.3 through to 6.5, but without the addition of further paper pulp.
- 6.6** Finally, filter the solution through the original paper into the 1000 ml beaker containing the original filtrate, washing with hot ammonia wash solution (3.5), then with hot water as before. Reserve the precipitate and paper (**See Note 1**). Wash the funnel thoroughly with hot water. The volume should now be approximately 450–500 ml.

Note 1: *This precipitate may be tested for occluded molybdenum.*

Note 2: *If you proceed after step 6.6, steps 6.7 to 6.11 must be completed within the same day.*

- 6.7** Acidify this solution by adding acetic acid (3.7), using litmus paper (3.14) dropped into the solution as the indicator. Add 50 ml of ammonium acetate solution (3.9) and bring to the boil. Whilst boiling, add 35 ml of lead acetate solution (3.8) slowly (typically 90 seconds) from a 50 ml burette. To aid coagulation, continue to boil the solution for 2–3 minutes, then allow to stand for a minimum of 30 minutes at approximately 60°C. At this stage the precipitate should be white.
- 6.8** Filter the solution over double thickness 150 mm No. 40 Whatman filter paper, or equivalent, retaining the bulk of the precipitate in the original beaker. Wash the precipitate in the original beaker 3 times by decantation using approximately 50 ml boiling ammonium acetate wash solution (3.10) for each washing. Finally, wash the filter papers with the same solution (3.10) (**See Note 3**). Discard the filtrate. (**See Note 4**). Transfer the funnel with the filter papers to the original beaker, open the papers and carefully wash off the precipitate with hot water. Wash papers with hot hydrochloric acid wash solution (3.11) and then hot water. Discard filter papers. At this stage the volume should be approximately 200–250 ml.

Note 3: *Care should be taken when washing the filter paper as the finer precipitate may creep up the sides of the funnel.*

Note 4: *This filtrate may be tested for molybdenum.*

- 6.9** Add 0.5 ml of lead acetate solution (3.8) and heat to completely dissolve. Following complete dissolution, add 2–3 drops of the methyl orange indicator (3.12). Remove from the hotplate and, dropwise, carefully add ammonia solution (3.13), whilst swirling, until a slight turbidity persists. Bring to the boil, add hydrochloric acid wash solution (3.11) dropwise to clear the turbidity. Maintaining a boiling solution, add ammonium acetate solution (3.9) until the indicator changes color, then add a further 20 ml of ammonium acetate solution (3.9). To aid coagulation, continue to boil the solution for 2 – 3 minutes, then allow to stand for a minimum of 30 minutes / maximum 60 minutes at approximately 60°C.
- 6.10** Filter the solution over double thickness 150 mm No. 40 Whatman filter paper (or equivalent) and, using a rubber-tipped glass rod, ensure all precipitate is transferred from the beaker to the paper. Wash well with hot ammonium acetate wash solution (3.10).
(See Note 4).
- 6.11** Transfer the paper with precipitate to a glazed crucible. To ensure that no precipitate is retained on either the funnel or the beaker, wipe them both clean using a piece of moistened filter paper and add this paper to the crucible.
- 6.12** Dry on a warm hotplate. Then place the crucible in a furnace and reduce to ash at 550°C. Cool in a desiccator, and weigh the lead molybdate **(W2)**.

6.13 Calculation:

$$\text{Molybdenum, \%} = \frac{0.2613 \times W2 \times 100}{W1}$$

Where:

0.2613 = gravimetric factor (Mo/PbMoO₄).

Atomic Weights:	Oxygen:	15.999
	Molybdenum:	95.95
	Lead:	207.19

$$\frac{\text{Mo}}{\text{PbMoO}_4} = \frac{95.95}{367.14} = 0.2613$$

Therefore the factor PbMoO₄ → Mo = 0.2613

Assays should agree with 0.3% absolute.

Chemical Name	Identifiers	Chemical Formula	Synonyms
Molybdenum Disulfide	For roasting: CAS No. 1309-56-4 & EC No. 215-172-4 For lubricant grade: CAS No. 1317-33-5 & EC No. 215-263-9	MoS ₂	Molybdenite concentrate Unroasted molybdenite concentrate (UMC) Moly concs Mo (IV) disulfide Mo disulfide Moly sulfide

X-Ray Fluorescence Spectroscopy (XRF) Short Overview

XRF is a well-established analytical technique to identify and quantify the chemical composition of a wide range of materials. Wavelength dispersive X-Ray Fluorescence (WDXRF) is widely used in the mining, metals and minerals analysis sectors, and is capable of elemental analysis of a wide range of materials including solids, powders, and liquids. Multiple manufacturers and suppliers offer a wide range of spectrometers and applications.

As is the case for any analysis, the quality of the analytical data produced is reliant on the method, suitable equipment and the skills, expertise and experience of the analytical specialists performing the analysis.

XRF is a relative method, calibrated to use samples with known element concentrations. During measurement each element in the sample emits characteristic fluorescence x-ray lines which are read against the calibration.

XRF is characterized by having very high long-term reproducibility and stability, however small changes can occur over the long term due to degradation of the detector or tube. Re-calibration can overcome this by analyzing all calibration beads anew or using a drift correction monitor bead.

Further XRF overview information is available on the [IMOA website](#).

About IMOA

IMOA is a non profit trade association, registered in Belgium. Representing the majority of the molybdenum industry worldwide, it works to raise awareness about the unique properties of molybdenum, its beneficial effects on materials performance, its safety in use and its contribution to sustainable development.

IMOA's HSE Committee works tirelessly to secure continued access to markets for members, using rigorous scientific evidence to support appropriate regulation setting and chemical management. Its main objectives are to:

- monitor scientific literature for studies on health and environmental effects of molybdenum
- act pre-emptively by developing data and providing input to regulatory authorities
- monitor regulatory developments and proposals that may impact the molybdenum industry

Disclaimer:

These guidelines relating to assaying procedures for Molybdenite Concentrates are provided for reference purposes only. They are designed to promote the standardization of assaying methodology, with a view to improving quality and reliability for molybdenum producers, consumers, converters, assayers and others in the industry. Use of the guidelines is purely voluntary on the part of the user, and participation in IMOA does not create an obligation on anyone to adhere to these guidelines. IMOA makes no warranty of any kind, whether of merchantability, fitness for a particular use or purpose, or otherwise for any Molybdenite Concentrates that have been assayed using these guidelines.



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