

*Procedure for the Assaying of*

# MOLYBDENITE CONCENTRATES



GUIDELINES FROM



# INTRODUCTION

This Guideline on good practice in relation to the Assaying of Molybdenite Concentrates is one of a six part series on Weighing, Sampling and Assaying which has been drawn up and published by the International Molybdenum Association for the benefit of its members and the industry at large.

The aim of the IMOA Sampling and Assaying Committee was to prepare worldwide industry guidelines to improve consistency and quality in Weighing, Sampling and Assaying procedures for Molybdenite Concentrates, Technical Grade Molybdenum Oxide and Ferromolybdenum.

The full series includes:

**Procedure for the Weighing and Sampling of Molybdenite Concentrates.**

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**Procedure for the Weighing and Sampling of Technical Grade Molybdenum Oxide.**

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**Procedure for the Weighing and Sampling of Ferromolybdenum.**

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**Procedure for the Assaying of Molybdenite Concentrates.**

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**Procedure for the Assaying of Technical Grade Molybdenum Oxide.**

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**Procedure for the Assaying of Ferromolybdenum.**

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# Procedure for the Chemical Analysis of **MOLYBDENITE CONCENTRATES**

## 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.

***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.***

Use only reagents of recognised 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

## SAMPLE

The sample is to be obtained as per the "IMOA 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.**

## 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. Reboil 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 colour, 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:

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

Where:

0.2613 = gravimetric factor (Mo/PbMoO<sub>4</sub>).

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 PbMoO<sub>4</sub> → Mo = 0.2613

*Assays should agree with 0.3% absolute.*

Chemical Name	CAS No.	Chemical Formula	Synonyms
Molybdenum Disulphide	1317-33-5	MoS <sub>2</sub>	<ul style="list-style-type: none"><li>■ Molybdenite Concentrate</li><li>■ Mo (IV) disulphide</li><li>■ Mo disulphide</li><li>■ Moly sulphide</li></ul>

# NOTES



INTERNATIONAL MOLYBDENUM ASSOCIATION

The International Molybdenum Association (IMOA) was registered in 1989 as a legal entity in Belgium and has become the focal point of promotional, statistical and technical activities for the worldwide molybdenum industry. Membership is broad based and includes producers, consumers, converters, traders and assayers. IMOA's secretariat is based in London.

## **IMOA's main activities currently include:**

- Promoting molybdenum as a material with superior properties and performance in a wide variety of metallurgical, chemical and other product applications;
- Promoting the applications in which molybdenum is used via market development programmes which identify key areas offering potential for increasing molybdenum consumption. With the co-operation of consumers, end-users and allied organisations worldwide, technical brochures are published and training seminars organised which explain the advantages of using molybdenum-containing products in various industries;
- Monitoring molybdenum in relation to health, safety and environmental issues, including the conduct of a Life Cycle Inventory of certain molybdenum products. IMOA's HSE Database may be accessed via the website: [www.imoa.info](http://www.imoa.info)
- Collecting the industry's most comprehensive historical statistics on world supply and demand of molybdenum products which are distributed to all IMOA members on a regular basis;
- Organising meetings and promotional conferences beneficial to the molybdenum industry;
- Preparing worldwide industry guidelines to improve consistency and quality in sampling and assaying procedures for molybdenum compounds.

These guidelines relating to assaying procedures for Molybdenite Concentrates are provided for reference purposes only. They are designed to promote the standardisation 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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