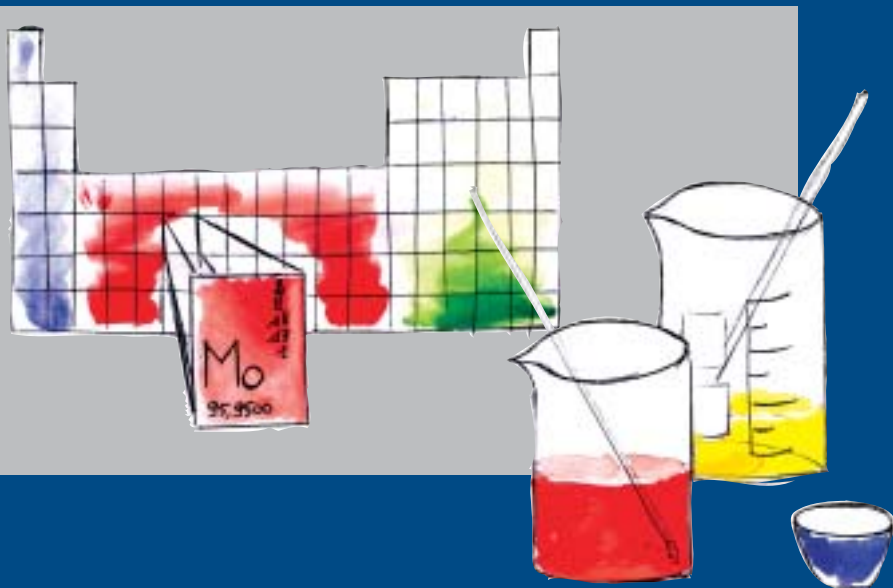


Procedure for the Assaying of **FERROMOLYBDENUM**



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.

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

Procedure for the Weighing and Sampling of Ferromolybdenum.

Procedure for the Assaying of Molybdenite Concentrates.

Procedure for the Assaying of Technical Grade Molybdenum Oxide.

Procedure for the Assaying of Ferromolybdenum.

Procedure for the Chemical Analysis of **FERROMOLYBDENUM**

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 ferromolybdenum within the normal ranges of this material.

2. **PRINCIPLE**

Dissolution of sample using acids, 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 to 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 Sodium hydroxide pellets

3.5 Glacial acetic acid (1.05 S.G.)

3.6 Acetic acid solution (0.5%):

Dilute 5 ml of acetic acid (3.5) to 1000 ml with water.

3.7 Lead acetate solution:

Dissolve 40 g of lead acetate in 500 ml water. Add 5 ml glacial acetic acid (3.5) and dilute to 1000 ml with water.

3.8 Ammonium acetate solution (20%):

Dissolve 200 g of ammonium acetate in 500 ml water. Add 10 ml of glacial acetic acid (3.5) and dilute to 1000 ml with water.

3.9 Ammonium acetate wash solution:

Dilute 200 ml of ammonium acetate solution (3.8) to 1000 ml with water.

3.10 Hydrochloric acid wash solution:

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

3.11 Methyl orange:

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

3.12 Ammonia solution (0.88 S.G.)

3.13 Ammonia solution (1 + 1):

Dilute 500 ml of ammonia solution (3.12) to 1000 ml with water

3.14 Litmus paper

4.

APPARATUS

a) Grade 'A' glassware: 500 ml volumetric flask, 250 ml volumetric flask.

b) Usual laboratory equipment, including an analytical four decimal place balance. The filtrations at steps 6.13 and 6.15 may be modified to accept either filtration by pulp pad or vacuum filtration. In the latter case a hardened paper is required.

c) Filter papers: 150 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 "IMO A Guideline Procedure for the Weighing and Sampling of Ferromolybdenum", ensuring that the sample passes through a 100 mesh (ASTM) sieve (0.15 mm aperture). The sample is to be redried at 105°C to constant weight, and mixed well prior to removal of the test portions to be assayed.

Analyses to be performed at least in duplicate.

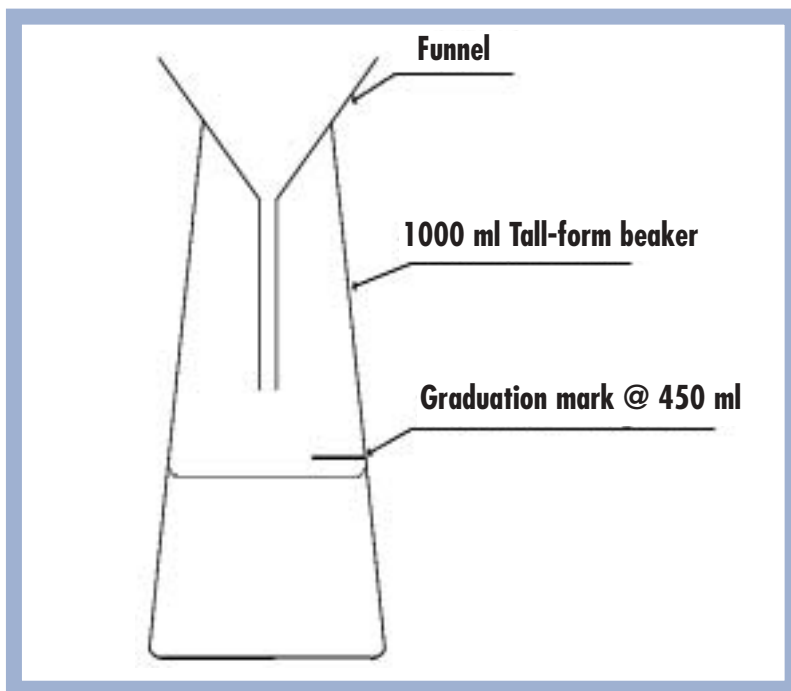
PROCEDURE

6.1 Accurately weigh to four decimal places approximately 0.5 g of sample (W1) and transfer to a 400 ml tall form glass beaker.

6.2 Carefully add 10 ml sulphuric acid (3.3) and 5 ml nitric acid (3.2), swirl to disperse the sample, and cover with a watch glass. Place on a hotplate until reaction commences. Remove from hotplate and when reaction has subsided add 5 ml hydrochloric acid (3.1). Swirl, replace on hotplate and evaporate gently to fumes of sulphuric and then fume strongly. This is evidenced by dense white fumes filling the beaker. Ensure that no losses occur due to spitting.

6.3 Remove from the hotplate, cool, then cautiously wash down the sides of the beaker and watchglass with 10 ml cold water. Dilute to approximately 100 ml using hot water.

6.4 Transfer 30 g sodium hydroxide pellets (3.4) into a tall form 1000 ml beaker, previously marked at approximately 450 ml. Place a funnel containing an 150 mm No. 54 Whatman filter paper or equivalent, such that it rests on the lip of the beaker (see sketch) forming a seal to prevent losses from spattering. The stem of the funnel should be of a length that it is above the final level of the solution.



The steps described in 6.5 below necessarily produce extremely vigorous reactions, and great care must be taken. It is also important that they should be performed in a continuous manner.

6.5 Place the solution from step 6.3 onto the hotplate to boil. On boiling lift funnel from the 1000 ml beaker, add 150 ml boiling water to the sodium hydroxide pellets and replace funnel. Remove the boiling acid solution from the hotplate and wash off watch-glass into funnel with boiling water.

Carefully pour the acid solution into the funnel containing the filter paper at a speed sufficient to maintain a steady boiling reaction within the beaker.

Using boiling water, wash the beaker into the funnel using a fine jet, then clean the beaker using a rubber-tipped glass rod. Wash again with boiling water then transfer the washings into the filter funnel. Swirl the 1000 ml beaker to ensure thorough mixing.

6.6 Wash the filter paper with boiling hydrochloric acid wash solution (3.10) using a fine jet. Wash the filter paper with boiling water. Once drained, remove the filter paper and wash the funnel into the beaker, replacing the funnel with a watch-glass. The final volume should be a maximum of 450 ml.

Note 1: *At this stage the filter paper may be tested for occluded molybdenum.*

6.7 Swirl beaker and place on hotplate. Continue swirling to avoid localised overheating, and boil for 3 minutes. Remove beaker from hotplate and cool to ambient temperature.

6.8 Quantitatively transfer to a 500 ml volumetric flask, mix and dilute to volume with water, then re-mix.

6.9 The 500 ml caustic solution requires filtering to remove iron. Filter approximately 25 ml into the 250 ml volumetric flask to provide a wash solution (although this is to be discarded, the filtration is necessary to avoid iron entering the final solution). Wash the flask thoroughly, discard and repeat. Filter the remaining solution up to the final 250 ml, to give exactly half the initial sample weight.

6.10 Using a filter paper, wipe clean the neck and stem of the flask.

6.11 Transfer the solution from the volumetric flask into a clean tall-form 1000 ml beaker. Thoroughly wash the flask into the beaker with water, to ensure that the beaker contains all the measured solution, and therefore exactly half the sample weight.

Note 2: *If you proceed after step 6.11, steps 6.12 – 6.16 must be completed within the same day.*

6.12 Using litmus paper (3.14) dropped into the solution as the indicator, acidify the solution by adding acetic acid (3.5). Add 50 ml of ammonium

acetate solution (3.8) and bring to the boil. Whilst boiling, add 25 ml of lead acetate solution (3.7) 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.

6.13 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 three times by decantation using approximately 50 ml boiling ammonium acetate wash solution (3.9) for each washing. Finally, wash the filter papers with the same solution (3.9) (See Note 3). Discard the filtrate. (See Note 4). Transfer the funnel with the filter paper to the original beaker, open the papers and carefully wash off the precipitate with hot water. Wash papers with hot hydrochloric acid wash solution (3.10) 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*

6.14 Add 0.5 ml of lead acetate solution (3.7) and heat to completely dissolve. Following complete dissolution, add 2 – 3 drops of the methyl orange indicator (3.11). Remove from the hotplate and dropwise carefully add ammonia solution (3.12), whilst swirling, until a slight turbidity persists. Bring to the boil, add hydrochloric acid wash solution (3.10) dropwise to clear the turbidity. Maintaining a boiling solution, add ammonium acetate solution (3.8) until the indicator changes colour, then add a further 20 ml of ammonium acetate solution (3.8). 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.

6.15 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 (3.9). See Note 4.

6.16 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.

6.17 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).

6.18 Calculation:

$$\text{Molybdenum, \%} = \frac{0.2613 \times \mathbf{W\ 2} \times 100}{\mathbf{W1} \div 2}$$

Where:

0.2613 = gravimetric factor Mo (Mo/PbMo₄)

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

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

Therefore the factor PbMo₄ → Mo = 0.2613

Assays should agree within 0.3% absolute.

Chemical Name	CAS No.	Chemical Formula	Synonyms
Ferromolybdenum	94277-04-0		FeMo



INTERNATIONAL MOLYBDENUM ASSOCIATION

The International Molybdenum Association (IMO A) 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. IMO A's secretariat is based in London.

IMO A'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. IMO A's HSE Database may be accessed via the website: www.imoa.info
- Collecting the industry's most comprehensive historical statistics on world supply and demand of molybdenum products which are distributed to all IMO A 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 Ferromolybdenum 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 IMO A does not create an obligation on anyone to adhere to these guidelines. IMO A makes no warranty of any kind, whether of merchantability, fitness for a particular use or purpose, or otherwise for any Ferromolybdenum that has been assayed using these guidelines.



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