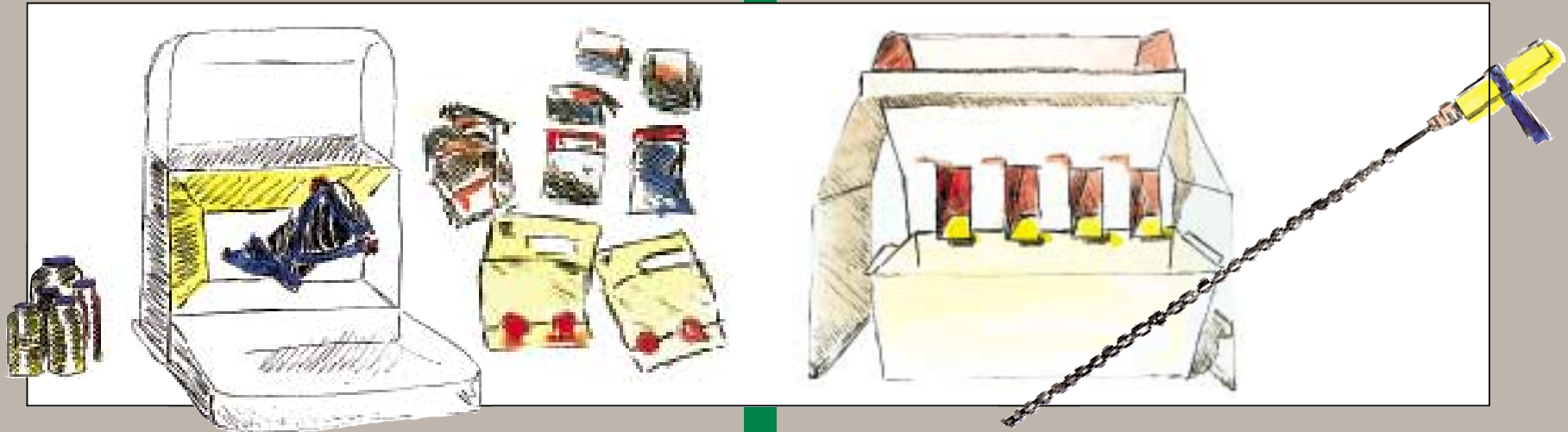


*Procedure for the Weighing
and Sampling of*

**MOLYBDENITE
CONCENTRATES**



Guidelines from the
**INTERNATIONAL MOLYBDENUM
ASSOCIATION**

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INTRODUCTION

This Guideline on good practice in relation to the Weighing and Sampling 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 Sub-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.

Other current titles in this series are:

- Procedure for the Weighing and Sampling of Technical Grade Molybdenum Oxide
- Procedure for the Weighing and Sampling of Ferromolybdenum

Future titles:

- Procedures for Assaying Molybdenite Concentrates
- Procedures for Assaying Technical Grade Molybdenum Oxide
- Procedures for Assaying Ferromolybdenum



The International Molybdenum Association (IMOA) was established in 1989 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;
- monitoring molybdenum in relation to health, safety and environmental issues; with the increasing amount of legislation in many countries relating to the use and disposal of metals and metal bearing materials, IMOA provides a centralised service including research studies on those issues that may affect the molybdenum industry;
- 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; and
- preparing worldwide industry guidelines to improve consistency and quality in sampling and assaying procedures for molybdenum compounds.

* These guidelines relating to weighing and sampling procedures for Molybdenite Concentrates are provided for reference purposes only. They are designed to promote the standardisation of weighing and sampling 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 in any Molybdenite Concentrates that has been weighed and / or sampled using these guidelines *.

5. DISTRIBUTION/COST ALLOCATION

- Documents to be distributed to:
- Samples to be distributed to:
- Reserve samples stored at:.....

Address for invoice:

NOTES

Procedure for the Weighing and Sampling of MOLYBDENITE CONCENTRATES

1.

SCOPE AND FIELD OF APPLICATION

This best practice guideline specifies a method for weighing and sampling Molybdenite Concentrates for their chemical quality.

The procedure shall be applied to each individual Lot. Lot size is defined between the contract parties involved, and shall be clearly stated in the Weighing and Sampling reports. These reports shall also specify the control procedure used, e.g. the "IMOA Procedure for the Weighing and Sampling of Molybdenite Concentrates."

If other procedures or standards are used, they are to be specified in the W/S report.

2.

DEFINITION OF TERMS

Auger

Means a coarse spiral with a diameter and flight spacing of at least 10 mm, and an operational length at least the depth of the material in the container. It is capable of being rotated mechanically through the full depth of material and of withdrawing a complete plug of material from the bulk.

Calibration

Means the process of comparing and adjusting measurement systems or procedures against Certified Weights, or against Certified Reference Materials. This process is carried out strictly in accordance with International Standards. The International Standard used in the Calibration process is to be specified in the Calibration report.

Certificate of Weighing and/or Sampling

Means a report of the activities, observations and readings executed during the control procedure. This report can be issued by the producer of the product, by the receiver of the product or by an organisation independent of the two parties. In the latter case, the Certificate becomes an independent Certificate, the minimum content of which is defined in the Guidelines for Instructions to the Supervisor for Inspection, Weighing, Sampling and Reporting.

Every Certificate must be duly signed by an authorised person.

Clean Certificate

Means a Certificate issued without qualification when both of the following conditions have been met:

1. The equipment available for carrying out the procedure conforms to the IMOA guidelines.
2. The procedure followed conforms to the IMOA guidelines.

Claused Certificate

Means a Certificate issued containing a statement that one or both of the following conditions prevail:

1. The equipment available for carrying out the procedure does not conform to the IMOA guidelines.
2. The procedure followed does not conform to the IMOA guidelines.

Consignment

Means Molibdenite Concentrates delivered within a specific period agreed between the contract parties concerned. A Consignment may consist of one or more Lots, or part of a Lot.

Increment

Means the quantity of Molybdenite Concentrate collected in a single operation of the sampling device.

Lot

Means the smallest unit to be weighed and for which a Sample and an assay have to be produced, as agreed between the contract parties.

Report of Findings

Means a report to be issued when it is considered that facilities and/or conditions are proven to be unsatisfactory to perform weighing and/or sampling in accordance with the IMOA guidelines, specifying the reasons.

Sample

- **Gross Sample**
Means the quantity of Molybdenite Concentrate in its received state, which results from the combination of all Increments of a Lot.
- **Preparation of Gross Sample**
Means the process of division, milling, mixing, sieving, etc, of the Gross Sample in the prescribed manner, resulting in the Fine Sample.
- **Fine Sample**
Means the prepared Sample after the prescribed division, milling, mixing and sieving steps, to be divided into Assay Samples.
- **Assay Sample**
Means the Sample drawn from the Fine Sample for the purpose of determining the required chemical quality parameter(s) of the sampled Lot, and which can be described as "Volatile Free at 105°C - may contain heavier oil fractions".

Top Size (for determining sampling equipment, division, milling, sieving in relation to sampling)

Means the particle size expressed by the aperture size of a square aperture sieve on which about 5% of the sample remains.

4. THE REPORT/CERTIFICATE OF INSPECTION/WEIGHING/SAMPLING SHALL AT LEAST INCLUDE THE FOLLOWING:

Identification of the Lot	<input type="checkbox"/>
Condition of the material at arrival	<input type="checkbox"/>
Observation of markings, identification, labels	<input type="checkbox"/>
Number of units in the Lot and Sub-lots	<input type="checkbox"/>
Weighing and Sampling Procedure used	<input type="checkbox"/>
Weight:	<input type="checkbox"/>
• Declared	<input type="checkbox"/>
• Observed	<input type="checkbox"/>
Inspection/Weighing/Sampling:	<input type="checkbox"/>
• Place	<input type="checkbox"/>
• Date	<input type="checkbox"/>
• Conditions	<input type="checkbox"/>
Equipment used	<input type="checkbox"/>
Calibration status of Weighing Scale	<input type="checkbox"/>
Quantities of Samples at different stages	<input type="checkbox"/>
Destination of Samples	<input type="checkbox"/>
Determination of Volatiles Content :	<input type="checkbox"/>
• Procedure used, including time, temperature	<input type="checkbox"/>
• Result	<input type="checkbox"/>
• Net Volatile Free Weight	<input type="checkbox"/>
Moisture content Determination:	<input type="checkbox"/>
Procedure used, including time, temperature	<input type="checkbox"/>
Result	<input type="checkbox"/>
Net dry weight	<input type="checkbox"/>
Weight of the final sample(s)	<input type="checkbox"/>
Final state of the sample(s) to be distributed	<input type="checkbox"/>
Seals on the sample(s)	<input type="checkbox"/>
Distribution of sample(s)	<input type="checkbox"/>
Distribution and number of original reports/ certificates	<input type="checkbox"/>
Total number of pages of the report/ certificate	<input type="checkbox"/>
Status of the certificate: Clean or Cloused	<input type="checkbox"/>
Full reference on each page	<input type="checkbox"/>
Signature of authorised person	<input type="checkbox"/>
Indication/marking of end of certificate	<input type="checkbox"/>

Certificates must be produced in such a manner that no changes can be made after distribution of the certificate.

2. DEFINITION OF THE OPERATION:

2.1 OPERATION TO BE PERFORMED IN ACCORDANCE WITH:

- IMO A Guidelines for the W/S of Technical Grade MoO₃
- IMO A Guidelines for the W/S of Ferromolybdenum
- IMO A Guidelines for W/S of Molybdenite Concentrates
- Other (specify)

2.2 ACTIVITIES TO BE PERFORMED ARE:

- Inspection
- Weighing
- Sampling
- Moisture Determination
- Determination of Volatiles
- Calculation of Net Volatile Free Weight
- Calculation of Net Dry Weight
- Grain Size Determination
- Preparation of Samples
- Sealing of Samples

- Compile Report of:
 - Inspection
 - Weighing
 - Sampling

- Compile Certificate of:
 - Inspection
 - Weighing
 - Sampling

3. SPECIAL INSTRUCTIONS:

- Precision required in calculations/results:
 - 2 decimal places for Volatiles Content
 - 2 decimal places for final Dry or final Volatile Free Weight
- Other (specify):

Volatiles Content

Means the (percentage) weight loss during heating for 18 hours at 105°C, and subsequent cooling to room temperature in the air.

Net Wet Weight

Means the weight after deduction of the tare weight from the gross weight.

Volatiles Weight

Means the weight of volatiles included in the Net Wet Weight, calculated by multiplying the Net Wet Weight by the percentage Volatiles Content.

Net Volatiles Free Weight

Means the weight after deduction of the Volatiles Weight from the Net Wet Weight.

Note: Because volatiles are determined at 105°C, heavier oil fractions may remain in the sample.

3.

WEIGHT DETERMINATION

The equipment used in all weighing operations shall have at least Class III accuracy in accordance with the recommendations of the Bureau International de Métrologie Légale.

The Standard applicable in Europe is EN 45501:1993.

Weighing operations shall conform to this Standard or to other International Standards in other countries. They shall be specified in the W/S report.

ACCURACY CLASS III (MEDIUM) MEANS: (Ref: EN 45501:1993)	
Verification scale interval	e > 5g
Minimum number of verification scale intervals	500 e
Maximum number of verification scale intervals	10000 e
Minimum capacity (lower limit)	20 e
MAXIMUM PERMISSIBLE ERROR LEVEL IN SERVICE: ACCURACY CLASS III (MEDIUM): (REF: EN 45501:1993)	
Load	Max. Perm. Error
0 < load < 500 e	± e
500 e < load < 2000 e	± 2e
2000 e < load < 10000 e	± 3e

EXAMPLE:

weighing scale with verification scale interval	=	1 kg
minimum number of verification scale intervals	500 e	= 500 kg
maximum number of verification scale intervals	10000 e	= 10000 kg
minimum capacity (lower limit)	20 e	= 20 kg

This means the scale has at least a capacity of 500 kg and a maximum of 10000 kg. It is not to be used for weights below 20 kg and it has the following permissible error level:

Load	Max. Perm. Error
0 kg < load < 500 kg	± 1 kg
500 kg < load < 2000 kg	± 2 kg
2000 kg < load < 10000 kg	± 3 kg

PROCEDURE

WEIGHT DETERMINATION

GROSS WEIGHT

a. Standard Procedure

The total gross weight shall be determined on a small interval scale which has been previously checked for accuracy and correct functioning. This scale must have been calibrated during the prescribed periods by an authorised calibrator. The scale capacity should not exceed 3000 kg and the maximum interval should be 1 kg. The minimum scale load should be preferably 10%, but not less than 2% of the scale capacity. All containers must be clean outside before weighing. Any extraneous matter must be removed prior to the commencement of weight determination.

Should these specifications not be met, either a Claused Certificate or a Report of Findings should be issued.

b. Optional Procedure

If agreed by the contract parties, the total gross weight may be determined on a scale with a capacity of max. 60000 kg, with intervals of max. 20 kg. If the weight determined at the weighbridge has a difference of less than 0.5% from the declared weight, the declared weight shall be accepted. If the difference is more than 0.5% from the declared weight, the contract parties are to decide the procedure to be followed, taking into account the subsequent Volatiles Content Determination. The Calibration data of the weighbridge must be available to each party.

GUIDELINES FOR INSTRUCTIONS TO THE SUPERVISOR FOR INSPECTION, WEIGHING, SAMPLING AND REPORTING (Referred to as 'the Operation')

Proper definition of the Operation is essential to ensure that all parties involved are conversant with the full requirements of the intended Operation, from methodology to reporting.

These Guidelines are presented in the form of a Checklist. As far as possible they contain all the essential elements needed to define the Operation. They may be used to prepare the instructions to the surveying company, and likewise enable the potential surveying company to quote for the

specified activities.

The Guidelines should be tailored to meet individual needs. The information contained in the following checklist is designed to assist technical representatives to service accurately the client's requirements, and to deliver a report consistent in format and quality.

The report should contain sufficient detail to allow the client to ascertain whether the IMO guidelines have been adhered to, and/or any deviation therefrom.

EASY REFERENCE CHECKLIST

Operation to be performed on behalf of:

1. IDENTIFICATION OF THE LOT:

- 1.1 Product name:
- 1.2 Shipment by:
- 1.3 Lot Reference:
- 1.4 Sub-lot references:
- 1.5 Declared weight:
- 1.6 Number of units:
- 1.7 Transportation by:
- 1.8 Loading port:
- 1.9 B/L date:
- 1.10 Discharge port:
- 1.11 E.T.A.:
- 1.12 Buyer/Seller:
- 1.13 Operation location:
- 1.14 Operation commissioned by:
- 1.15 Other party present at operation:
- 1.16 Operation supervised by:
- 1.17 Operation performed by:

Chemical Name	CAS No.	Chemical Formula	Synonyms
Molybdenum	1317-33-5	MoS ₂	• Molybdenite
Disulphide			Concentrate
			• Mo (IV) disulphide
			• Mo disulphide
			• Moly sulphide

4.1.2 **TARE WEIGHT - PALLETS**

If palletised, a significant percentage of each type of pallet, and any strapping used to secure their loads, shall be weighed to determine their tare weight after the removal of bags or drums, and such weight deducted from the initially established total gross weight in order to define the total gross weight of the material-filled containers. The number of pallets and the strapping used to determine such weights should not be less than 10% of the total within each type. Predetermined weights of pallets established prior to the actual commencement of current weighing operations should never be accepted, unless by prior mutual agreement.

A smaller scale should preferably be used to guarantee accurate measurement of the tare weight.

4.1.3 **TARE WEIGHT - BAGS OR DRUMS**

Unless by prior mutual agreement, a number of bags or drums in their entirety, i.e. complete with any attachments such as seals, bands, lids, bolts, etc, are to be completely emptied of their contents and fully cleaned inside. The number of containers for tare weighing will be the square root (rounded upwards) of the total number of containers in order to obtain a sufficient basis for calculating the total tare weight. The actual number of emptied containers weighed and their combined weight are to be indicated in the resultant Report/Certificate.

A smaller scale should preferably be used to guarantee accurate measurement of the tare weight.

4.1.4 **NET WET WEIGHT**

After the total tare weight of the bags or drums has been determined, it shall be deducted from their previously established total gross weight, in order to define the total Net Wet Weight of the material. Where the weight of the Gross Sample is disadvantageous to any of the interested parties, such weight will be included in the tare weight.

4.1.5 **NET VOLATILE FREE WEIGHT**

After the percentage Volatiles Content has been determined (see point 4.3) the calculated Volatiles Weight shall be deducted from the previously established total Net Wet Weight in order to define the Net Volatile Free Weight of the material.

4.2 **SAMPLING**

4.2.1 **AMOUNT OF PACKAGING UNITS TO BE SAMPLED**

Sampling shall be carried out on 100% of the bags or drums to ensure that the Gross Sample drawn is truly representative of the whole Lot. The percentage or exact number of drums/bags is to be indicated on the resultant Report/Certificate.

4.2.2 **BULK-BAG OR DRUM SAMPLING**

The drawing of a sample from each bag/drum shall be carried out by the use of a sampling spear to obtain an Increment of mass 500-1000 g per position. The diameter shall be at least four times the Top Size of the material and the length shall be sufficient to allow a core sample to be drawn completely from top to bottom of the material. Spear samples are to be drawn from five positions in each bag, i.e. the four corners and the central position. For drums, only one position is required.

At each position, the spear shall be passed from top to bottom of the material, sampling a quarter of the depth of the bag/drum in successive stages down the same hole.

Alternative methods of sampling may be used, provided they have proven efficiency, such as mechanical screw threaded Auger sampling, which may be used one stage per hole.

4.3 **DETERMINATION OF VOLATILES**

4.3.1 **PREPARATION OF THE SAMPLE**

The collected Gross Sample shall be thoroughly mixed (taking care to break up any lumps or agglomerates), divided by means of a reliable splitting method (riffle splitter, slab cake splitting, cone and quartering ...) to at least 2 x 500 g sub-samples. Reduction of the Gross Sample is to be performed as quickly as possible with a minimum of handling and in conditions which will not affect the Volatiles Content.

4.3.2 **PROCEDURE**

The two sub-samples of at least 500 g each are to be dried in an Electronically Controlled Oven at a temperature of $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 18 hours and cooled to room temperature in the air.

4.3.3 **CALCULATION**

The resultant loss in weight shall be expressed as a percentage of wet weight to be used as the basis for the calculation of the Volatiles Content of the Lot. If the results obtained from the two dried samples differ by less than 0.3% absolute, then the average of both Volatiles Contents shall be calculated. If the two percentages differ by more than 0.3% absolute, the procedure is to be repeated using fresh samples.

4.4 **SAMPLE PREPARATION**

4.4.1 **SIEVING AND MILLING**

It is essential that all equipment used is clean in order to prevent contamination of the Sample.

After determination of Volatiles Content the sub-samples drawn shall be combined, thoroughly mixed, preferably mechanically, sieved through a 100 Mesh (ASTM) sieve (0.15 mm aperture). The particles remaining on the 100 Mesh (ASTM) sieve are to be milled to pass through the sieve, returned to the bulk, and mixed thoroughly.

In particular cases, a high oil content may prevent efficient preparation of the sample as described above. If this is so, de-oiling the dried sample may be necessary. Such actions should be reported in the Weighing & Sampling Certificate/Report, and the reasons stated. The report should contain the following minimum information; reagents used, temperature, filtration medium and contact time.

4.4.2 **PORTIONING, PACKING & MARKING OF ASSAY SAMPLES**

The Fine Sample shall be thoroughly mixed, preferably mechanically, and divided into as many portions of minimum 50 g for assaying as required.

The sample containers, which must be suitable for hermetic sealing and withstand loss of oil, are then to be marked with the designation of the material or the origin of the material, Lot reference, number and marks of drums or bags sampled, etc. The sample container shall also mention the Net Wet Weight, the Net Volatile Free Weight, and the Volatiles Content.

The hermetically sealed sample container shall also be clearly marked "TO BE ASSAYED IN ITS PACKED STATE". It shall mention "Volatiles Free at 105°C - may contain heavier oil fractions", and shall bear the necessary seals.

4.4.3 **SAMPLES FOR THE EXCHANGE OF ASSAYS FOR SETTLEMENT**

When two sets of assay results, produced independently of each other, are required to be exchanged for settlement purposes, it is essential that the final samples used for this purpose are part of the same carefully prepared, milled and mixed portion.