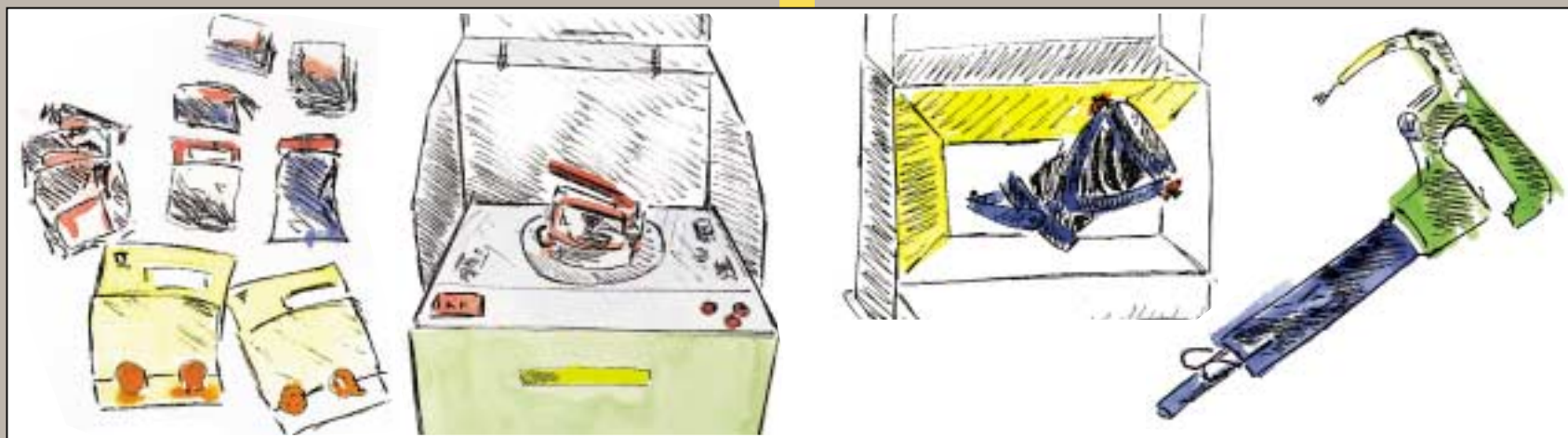


*Procedure for the Weighing  
and Sampling of*

**TECHNICAL GRADE  
MOLYBDENUM OXIDE**



*Guidelines from the*  
**INTERNATIONAL MOLYBDENUM  
ASSOCIATION**

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Unit 7 Hackford Walk, 119 - 123 Hackford Road, London SW9 0QT, England  
Tel: + 44 171 582 2777 Fax: + 44 171 582 0556  
E-mail: ITIA\_IMOA@compuserve.com

# INTRODUCTION

This Guideline on good practice in relation to the Weighing and Sampling of Technical Grade Molybdenum Oxide 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 Molybdenite Concentrates
- 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 (IMOIA) 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. IMOIA's secretariat is based in London.

## **IMOIA'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, IMOIA 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 IMOIA 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 technical grade molybdenum oxide 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 IMOIA does not create an obligation on anyone to adhere to these guidelines. IMOIA makes no warranty of any kind, whether of merchantability, fitness for a particular use or purpose, or otherwise in any technical grade molybdenum oxide 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 TECHNICAL GRADE MOLYBDENUM OXIDE

## 1.

### SCOPE AND FIELD OF APPLICATION

This best practice guideline specifies a method for weighing and sampling Technical Grade Molybdenum Oxide for its 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 Technical Grade Molybdenum Oxide".

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

## 2.

### DEFINITION OF TERMS

#### *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 will be as 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 used 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 used does not conform to the IMOA guidelines.

### Consignment

Means Technical Grade Molybdenum Oxide 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 Technical Grade Molybdenum Oxide collected in a single operation of the sampling device.

### Lot

Means the smallest unit to be weighed and against 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 IMO guidelines, specifying the reasons.

### Sample

- **Gross Sample**  
Means the quantity of Technical Grade Molybdenum Oxide 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, sieving steps, etc., 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.
- **Sizing Sample**  
Means the Sample drawn from the Gross Sample for the purpose of determining the required physical quality parameter(s) of the sampled Lot.

### 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.

### Net Wet Weight

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

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

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

*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 MoO<sub>3</sub>
- 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): .....

## 3.

### Net Dry Weight

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

### Moisture Weight

Means the weight of moisture included in the Net Wet Weight, calculated by multiplying the Net Wet Weight by the percentage moisture content.

## WEIGHT DETERMINATION

The equipment used in all weighing operations is to 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. The Calibration data of the weighbridge must be available to each party.

### 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.

# 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 A 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: .....

## NOTES

Chemical Name	CAS No.	Chemical Formula	Synonyms
Molybdenum Trioxide (Technical Grade)	86089-09-0	MoO <sub>3</sub>	<ul style="list-style-type: none"><li>• Roasted Molybdenite Concentrate</li><li>• Mo (VI) oxide</li><li>• Molybdic anhydride</li><li>• Molybdic oxide</li></ul>

### 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 is to 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 DRY WEIGHT

If there is suspicion that the Technical Grade Molybdenum Oxide is contaminated with moisture, then the contract parties should agree whether determination of the moisture content is required. After the moisture content has been determined (see point 4.3), the calculated Moisture Weight should be deducted from the previously established total Net Wet Weight, in order to define the Net Dry Weight of the material.

## 4.2 SAMPLING

### 4.2.1 AMOUNT OF PACKAGING UNITS TO BE SAMPLED

#### *a. Percentage of Bags or Drums*

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.

#### *b. Percentage of Cans*

Sampling shall be carried out on one can out of each layer per pallet. The number of cans and pallets sampled is to be indicated on the resultant Report/Certificate.

4.2.2 **'BULK-BAG' SAMPLING**

**a. Oxide Powder**

The drawing of a sample from each bag shall be carried out by the use of a sampling spear to obtain an Increment of mass 200-300 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 the five positions in each bag, i.e. the four corners and the central position. Alternative methods of sampling may be used, provided they have proven efficiency.

**b. Oxide Briquettes**

The drawing of a Sample shall be carried out by the use of a scoop to obtain an Increment of about 200 g. Five scoops of about 200 g each per tonne should be taken out at the top and from the corners, together with a Sample from as deep as possible.

4.2.3 **DRUM SAMPLING**

**a. Oxide Powder**

The drawing of a Sample from each drum shall be carried out by the use of a sampling spear to obtain an Increment of mass 200-300 g. The diameter shall be at least four times the Top Size of the material and the length should be sufficient to allow a core sample to be drawn completely from top to bottom of the material. Alternative mechanical methods of sampling may be used, provided they have proven efficiency.

**b. Oxide Briquettes**

The drawing of a Sample shall be carried out by the use of a scoop to obtain an Increment of about 200 g. Five random scoops of about 200 g each per tonne should be taken out of the drums.

4.2.4 **CAN SAMPLING**

The drawing of a Sample from each can shall be carried out by the use of a sampling spear to obtain an Increment of about 100 g.

4.3 **MOISTURE DETERMINATION (OPTIONAL)**

4.3.1 **DRYING**

The collected Gross Sample is to be crushed to less than 4 mm if necessary, thoroughly mixed, then reduced by coning and quartering or by other recognised methods as quickly as possible and in a manner that avoids any loss of moisture (for example by evaporation) during this stage of preparation. Two portions of minimum 2 kg each are to be drawn and dried in an Electronically Controlled Oven at a temperature of 105° C +/- 5° C until constant weight is achieved.

4.3.2 **MOISTURE CONTENT CALCULATION**

The resultant loss in weight is to be expressed as a percentage of Wet Weight to be used as the basis for the calculation of the total moisture 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 moisture contents is to be calculated. If the two moisture percentages differ by more than 0.3% absolute, the drying procedure is to be repeated.

4.4 **SAMPLE PREPARATION**

4.4.1 **DIVISION, MILLING, MIXING AND SIEVING**

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

After drying, the 2 partial samples drawn for Moisture Determination shall be combined, thoroughly mixed, preferably mechanically, and divided into a quantity dependent upon the top size (see table below).

If no drying is required, the Gross Sample should be thoroughly mixed and divided into a quantity dependent upon the Top Size (see table below).

This quantity is then milled to pass through a 100 Mesh (ASTM) sieve (0.15 mm aperture).

**Table: Relation between Top Size & Division of Gross Sample**

Top Size	Minimum Quantity after Division
< 2 mm	0.5 kg
2 - 4 mm	1.0 kg
> 4 mm	2.0 kg

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 each for assaying as required.

The sample containers, which must be suitable for hermetic sealing, 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 and, if applicable, the Net Dry Weight and the moisture content.

The hermetically sealed sample container shall also be clearly marked "TO BE DRIED AT 105°C BEFORE ASSAYING", 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.