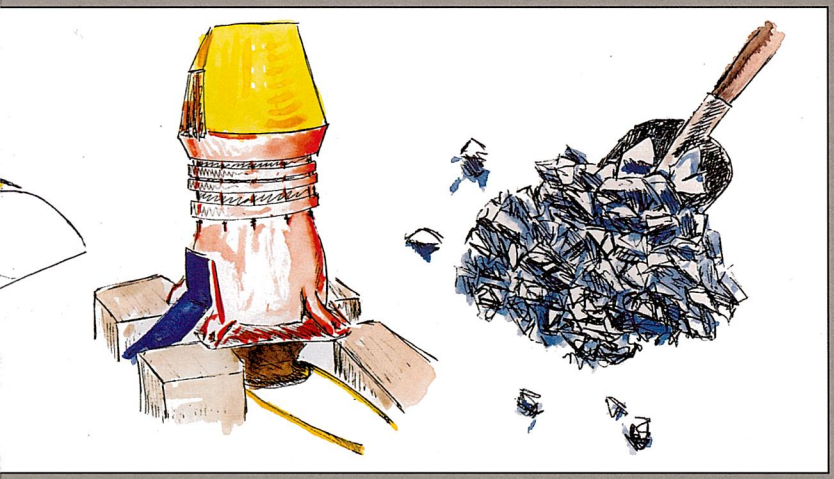
Procedure for the Weighing and Sampling of

FERROMOLYBDENUM



Guidelines from the

INTERNATIONAL MOLYBDENUM ASSOCIATION

INTRODUCTION

This Guideline on good practice in relation to the Weighing and Sampling of Ferromolybdenum 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 Technical Grade Molybdenum Oxide

Future titles:

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

Procedure for the Weighing and Sampling of FERROMOLYBDENUM

1. SCOPE AND FIELD OF APPLICATION

This best practice guideline specifies a method for weighing and sampling Ferromolybdenum for its chemical and/or physical 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 Ferromolybdenum".

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 is defined in the Guidelines for Instructions to the Supervisor for Inspection, Weighing, Sampling and Reporting.

Every Certificate must be 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 with 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 Ferromolybdenum delivered within a specific period agreed between the contract parties. A Consignment may consist of one or more Lots, or part of a Lot.

Increment

Means the quantity of Ferromolybdenum 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 Ferromolybdenum 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, 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.

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.

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

4. PROCEDURE

4.1 *WEIGHT DETERMINATION*

4.1.1 *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.

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 DRY WEIGHT*

If it is suspected that the Ferromolybdenum is contaminated with moisture, the moisture content has to be determined. After the moisture content has been determined (see point 4.4), 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 OF A LOT DURING THE PACKING OF UNITS*

4.2.1 *AMOUNT OF PACKAGING UNITS TO BE SAMPLED*

a. Percentage of Bags or Drums

Sampling shall be carried out in such a manner that Increments drawn from the material stream represent 100% of the bags or drums, thus ensuring that the Gross Sample drawn is truly representative of the whole Lot.

b. Percentage of Cans

Sampling shall be carried out during the packing of Ferromolybdenum in cans in such a manner that the number of Increments is at least equal to one increment per 250 kg.

4.2.2 *'BULK-BAG' SAMPLING*

A minimum of three increments shall be drawn from each bag, representing at least the bottom, middle and top of the bag. Drawing shall be carried out either by diverting the falling stream or by inserting a suitable tray into the stream. The dimension of the tray should be adapted to the Increment Weight, i.e. in accordance with ISO 3713:1987.

The weight of each Increment shall be defined by the Top Size of the material, and shall be in accordance with the tables annexed to this procedure.

4.2.3 *DRUM SAMPLING*

The drawing of an Increment shall be carried out either by diverting the falling stream or by inserting a suitable tray into the stream. The dimension of the tray should be adapted to the Increment Weight, i.e. in accordance with ISO 3713:1987.

The weight of each Increment shall be defined by the Top Size of the material, and shall be in accordance with the tables annexed to this procedure. At least one Increment shall be drawn per drum during the packing of the drums. Since the drums are packed in sequence, the timing of drawing the Increment is unimportant provided the size distribution at the moment of sampling is representative of the filling operation.

4.2.4 *CAN SAMPLING*

The drawing of the Increment shall be carried out in the same manner as described for bag and drum sampling. The number of Increments drawn shall be at least equal to one increment per 250 kg.

4.3 *SAMPLING A LOT SUPPLIED IN PACKED UNITS*

4.3.1 *AMOUNT OF PACKAGING UNITS TO BE SAMPLED*

a. Percentage of Bags or Drums

Sampling should 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 bags/drums sampled shall be indicated on the resultant Report/Certificate. If for practical reasons 100% sampling of drums is not possible, then for a drum size of 250-400 kg at least one drum chosen at random per 1000 kg shall be sampled. For smaller drums of, for example 100 kg, at least 5 drums randomly selected from different pallets per 1000 kg shall be sampled.

Where a Lot is composed of individually identified small sub-lots, preference shall be given to sampling all drums of the whole Lot. If this is not possible, all drums shall be inspected for the presence of sub-lot identification, and an inventory be made of all sub-lot data, i.e. the exact number of drums of each sub-lot and the weight of each sub-lot.

Using this inventory, the number of drums to be opened shall be determined in such a manner to ensure that the quantity of each sub-lot sample within the Gross Sample is proportional to the sub-lot size within the whole Lot.

The sampling report/certificate shall clearly specify the data regarding lot/ sub-lots/number of drums sampled per sub-lot/quantity of Sample per sub-lot and total quantity of Gross Sample for the whole Lot.

b. Percentage of Cans

Sampling shall be carried out on one can per layer of each pallet. The number of cans and pallets sampled shall be specified in the resulting report/certificate.

4.3.2 *BULK-BAG SAMPLING*

Due to the size of the pieces and the potential assay differences between individual pieces, the drawing of a Sample from each bag must be carried out during the transfer of the contents to another suitable container. A minimum of three Increments shall be drawn from each bag, representing at least the bottom, middle and top of the bag. Drawing shall be carried out either by diverting the falling stream or by inserting a suitable tray into the stream. The dimensions of the tray should be adapted to the Increment Weight, i.e. in accordance with ISO 3713:1987.

The weight of each Increment shall be defined by the Top Size of the material, and shall be in accordance with the tables annexed to this procedure.

4.3.3 *DRUM SAMPLING*

The drawing of a Sample from each drum (as determined in 4.3.1.a.) shall be preferably carried out during the transfer of the contents of a drum into another container, e.g. the transfer from one drum to another via an intermediate hopper. At least two Increments shall be drawn from each drum, one from the bottom and another from the top.

The drawing of the Sample shall be carried out using a suitable tray, i.e. in accordance with ISO 3713:1987, to obtain an Increment of mass defined by the Top Size as indicated in the tables annexed to this procedure.

4.3.4 *CAN SAMPLING*

Sampling of cans shall be carried out using the full contents of a can as the Increment.

4.4 MOISTURE DETERMINATION (OPTIONAL)

4.4.1 DRYING

The collected Gross Sample is to be crushed to less than 10 mm if necessary, thoroughly mixed, and then reduced by coning and quartering or by other recognised methods throughout each stage of the sample reduction process. Reduction must be performed 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 10 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.

REMARK ABOUT HIGH VOLUME GROSS SAMPLES:

The collected Gross Sample could amount to over two tons, on a 10% sample. This should be reduced to about 250 kg before crushing, and then crushed to less than 19 mm to avoid producing large quantities of fines. After crushing, the sample should be well mixed and reduced to 60 kg. The 60 kg sample should be further crushed to 5mm and, after mixing well, reduced to 20 kg. Two portions of minimum 10 kg each should be drawn and dried in an Electronically Controlled Oven at 105°C +/- 5°C until constant weight is achieved.

4.4.2 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 shall be calculated. If the two moisture percentages differ by more than 0.3% absolute, the drying procedure is to be repeated.

4.5 SAMPLE PREPARATION

4.5.1 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 two partial Samples drawn for Moisture Determination shall be combined and thoroughly mixed.

If no drying is required, the Gross Sample shall be crushed to less than 10 mm, thoroughly mixed and divided up to at least 20 kg. (See also the Remark under 4.4.1 about high volume Gross Samples).

The 20 kg sample of less than 10 mm, as defined in 4.4.1 or 4.5.1, or the 20 kg sample of less than 5 mm as defined in the Remark of 4.4.1, should be crushed to less than 3 mm. After crushing, the Sample shall be thoroughly mixed and reduced to at least 0.5 kg.

This quantity is then sieved and milled to pass through a 100 mesh (ASTM) sieve (0.15 mm aperture). Milling shall be carried out in short time intervals, preferably 30-40 seconds each, followed by sieving through 100 mesh. Only the particles which do not pass through the sieve should be milled, using the short milling time intervals.

4.5.2 *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.

4.5.3 *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.

Table 1:
Relation between Top Size & the Minimum Weight of the Increment Sample for Chemical Quality Determination

Top Size	Minimum Weight of Increment
> 50 mm	5 kg
50 mm	3.5 kg
25 mm	1.5 kg
< 10 mm	0.5 kg

Table 2:
Relation between Top Size & the Minimum Weight of the Increment Sample for Physical Quality Determination

Top Size	Minimum Weight of Increment
> 100 mm	100 kg
100 mm	80 kg
75 mm	50 kg
50 mm	25 kg
25 mm	8 kg
10 mm	1.5 kg
5 mm	0.5 kg
3mm	0.5 kg

ADDENDUM 1

The following extracts from ISO 3713: 1987(E) are reproduced with permission. Complete editions of the standard can be obtained by post from BSI Customer Services, 389 Chiswick High Road, London, W4 4AL, UK (Tel: + 44 181 996 9000), or through national standards bodies.

Table 1:

Recommended dimensions of scoops for increment sampling

Number of scoop	Aprox. volume cm^3	Dimensions mm						Material thickness mm	a/c b/c
		a	b	c	d	e*	f g		
1	15	30	15	30	25	12		0,5	1,0 0,50
3	40	40	25	40	30	15		0,5	1,0 0,62
5	75	50	30	50	40	20		1	1,0 0,60
10	125	60	35	60	50	25	Select	1	1,0 0,58
15	200	70	40	70	60	30	according to	2	1,0 0,57
20	300	80	45	80	70	35	circumstances	2	1,0 0,56
30	400	90	50	90	80	40		2	1,0 0,58
40	790	110	55	110	95	50		2	1,0 0,59
50	1700	150	75	150	130	65		2	1,0 0,50
75	4000	200	100	200	170	80		2	1,0 0,50
100	7000	250	110	250	220	100		2	1,0 0,44

* If a scoop is also used for sample division, e = 0, i.e. the front of the scoop is cut off.

7.2.2 A scoop for increment sampling shall be of dimensions corresponding to the increment volumes and shall be sufficiently durable (see figure 2 and table 1).

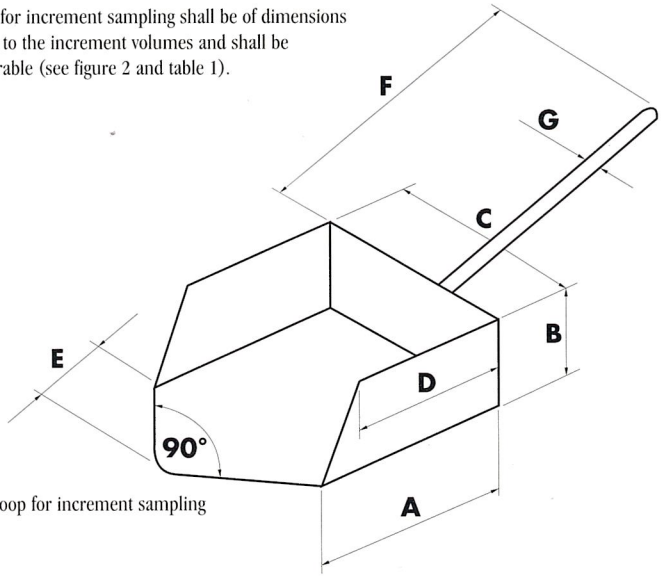


Figure 2 - A scoop for increment sampling

Table 2:
Recommended dimensions of containers for increment sampling

Approximate volume cm^3	Dimensions mm		
	a	b	c
75	50	50	30
125	60	60	35
200	70	70	40
300	80	80	45
400	90	90	50
790	110	110	65
1700	150	150	75
4000	200	200	100
7000	250	250	120

7.2.3 A container for increment sampling shall be selected on the basis of the volume of a sample (see figure 3 and table 2).

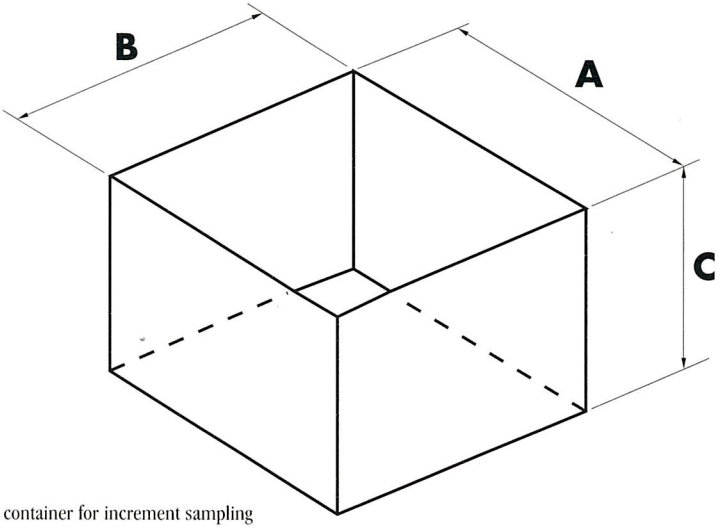


Figure 3 - A container for increment sampling

NOTES

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

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 IMOA 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: .....

2. DEFINITION OF THE OPERATION:

2.1 OPERATION TO BE PERFORMED IN ACCORDANCE WITH:

- IMOA Guidelines for the W/S of Technical Grade MoO_3 ❑
- IMOA Guidelines for the W/S of Ferromolybdenum ❑
- IMOA 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): .....

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

Identification of the Lot ❑
Condition of the material at 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/marking of end of certificate ❑

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

5. DISTRIBUTION/COST ALLOCATION

- Documents to be distributed to: .....
.....
.....
.....
.....
- Samples to be distributed to: .....
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- Reserve samples stored at: .....
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Address for invoice: .....
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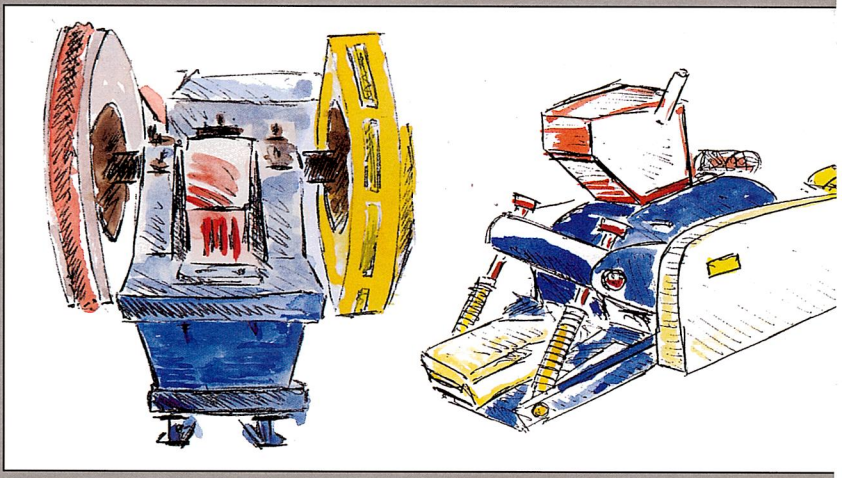
NOTES



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