

MOUNT ISA MINES LIMITED

**LABORATORY AND TECHNICAL INFORMATION
SERVICES**

METHODS FOR THE SIZING ANALYSIS

OF

ORES AND METALLURGICAL SAMPLES

Master Sizer Operation

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Amendment Record

DATE	Version	Reason for Change	Auth.
30.08.1997	1.0	Document Issued	MDR
14/04/04	1.1	Revised to include calibration steps and method updated.	JWST

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1 SCOPE

This method sets out the steps to be followed in order to perform a sizing analysis, using the Malvern Mastersizer Microplus, as requested and required by the Metallurgical Services Laboratory. The computer section of the instrument is configured to enable presentation of the results of the analysis in the required manner and transfer of data to LIMS for storage.

2 APPLICATION

The Malvern Mastersizer Microplus is capable of treating samples with particles which are in the range 0.05 - 556 µm in diameter. As such it is suitable for use with samples from both the Copper and Lead/Zinc Concentrator.

3 PRINCIPLE

The Malvern Mastersizer Microplus optical unit captures the scattering pattern which results when light (**from a laser**) is passed through a field of particles. The software of the system analyses the raw data coming from the detectors that record a succession of scattering patterns and presents an evaluation of the size of the particles. Once completed the averaged results can be graphed and/or printed out.

4 REFERENCED DOCUMENTS

The following document was referred to in writing this method:

Getting Started, MAN 0106, Issue 1.0, January 1996 (Malvern Instruments Ltd. 1995).

Before using this method personnel should ensure that they are familiar with (and understand) – PRP.012 Rotary Splitting and Bagging of Drill Core.

5 EQUIPMENT

- 5.1 Malvern Mastersizer Microplus.
- 5.2 Sample
- 5.3 Rotary Sample divider.
- 5.4 Pipettes – plastic disposable (1-2 mL).
- 5.5 Sample beakers – 1L plastic and 80-100 mL glass.

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- 5.6 Water – deionised quality or better.
- 5.7 Lens cleaner tissues.
- 5.8 Clean plastic vial scoop.
- 5.9 Magnet
- 5.10 Magnetic Stirrer
- 5.11 Calgon - Water Conditioner
- 5.12 10% Soda Ash Solution
- 5.13 Clean labelled plastic sample vials.

6 CLEANING THE MASTERSIZER CELL AND WINDOWS

The Malvern Mastersizer Microplus is a precision optical device and cleanliness of the optics is fundamental to producing good measurements. In the main there is only one part which the operator is expected to keep clean and this is the cell window assembly. Removal and cleaning of the diode protection window is the responsibility of the Analytical Instrument Maintenance section.

- 6.1 Malvern Mastersizer Microplus pump must be switched off and the pump assembly in the fully raised position.
- 6.2 Pull the cell drawer open until the catch holds it open.
- 6.3 When the system has drained pull up and rotate the cell top lever 90o anticlockwise.
- 6.4 Pull the cell top free using the raised rim of the window retainer.
- 6.5 Place the cell top on a piece of clean tissue paper on the bench, with the “O” ring facing up

Note 1: Ensure that no finger contact is made with the glass of the window.

- 6.6 Using a clean Kimberley Clark tissue dab dry the metal faces cell drawer.
- 6.7 Using a clean Kimberley Clark tissue dab clean cell top.
- 6.8 Flush out the groove around the window in the cell drawer using a fine stream of deionised water to remove sample which may be caught there then repeat step 6.7.

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Note 2: Never clean the windows dry as this will produce scratches (if necessary breathe gently on the window or squirt with clean water from a wash bottle to wet it).

- 6.9 Carefully replace the cell top into the cell drawer (if necessary apply a few drops of deionised water to glass on the cell drawer, then with one hand supporting the drawer rotate and press down the cell top lever.
- 6.10 With one hand hold the cell drawer and with the other hand push down the drawer catch.
- 6.11 Slowly ease in the drawer, taking care to remove the hand holding the catch once the drawer starts to move.

Note 3: When the Malvern Mastersizer Microplus is in normal use this cleaning routine should be performed each time the system is used.

- 6.12 Completion of the cleaning should be verified by entry on the appropriate form.

6.2 Checking for Leaks

- 6.2.1 If the cell top or sample tubes are not replaced properly the system may leak.
- 6.2.2 The appearance of dispersant on the workbench is a tell-tale sign that the cell is leaking.
- 6.2.3 If a leak is suspected notify the Laboratory Coordinator.

7 ALIGNMENT OF THE INSTRUMENT

Prior to commencement of operations on any one day or following any cleaning procedures, the instrument must first be aligned. This is accomplished in the following manner:

- 7.1 Check: (a) the computer and optics are connected and switched on
(b) the cell top is correctly fitted and locked in position.
(c) there are no leaks from the cell and cell ports.
- 7.2 Replace the 1 L plastic beaker of deionised water that is housing the pump assembly with a beaker of clean deionised water and lower the pump into position.
- 7.3 Turn the pump on and adjust if necessary the pump speed to 2400 rpm.
- 7.4 Turn on the Ultrasonic Displacement up to 6.5
- 7.5 Allow unit to run for 1 minute.

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- 7.6 Turn on computer and enter into the Mastersizer program on the desktop.
- 7.7 Click on **FILE**, then **LOAD CONFIGURATION**.
- 7.8 Choose which file you want the results to be kept in and double click on it.
- 7.9 Right hand side of screen (graph area) should now be clear, if **not** erase graph by clicking on delete key in tool bar.
- 7.10 Click on **MEASURE MENU** (this screen will display a dialogue box).
- 7.11 Click on **ALIGN**.
- 7.12 Follow prompts to align, (check the bottom right hand side of the screen to ensure all sweeps are completed before you proceed to the next prompt) record the laser power on the daily check list.
- 7.13 If aligned properly the laser power should show a value >75. If not repeat Cleaning Process from 6.1 - 6.11, then **ALIGN** lasersizer again, steps 7.2 - 7.12.

8 MIXING OF SAMPLE USING MAGNET STIRRER

With the Lasersizer aligned and ready (to proceed with the sizing of a sample) we have to transform the solid sample into a slurry for the Lasersizer to be able to analyse its size particles. Prior to commencement of lasersizing a sample we must mix the sample thoroughly.

- 8.1 Turn on the Magnetic Stirrer at the power point.
- 8.2 Fill an 80 mL glass beaker with deionised water and add a full squirt of Calgon.
- 8.3 Add a magnet to the beaker and place beaker onto the plate of the magnetic stirrer.
- 8.4 Fill the plastic vial (which already has the solid sample in it) 2/3 full of deionised water and shake vigorously to mix.
- 8.5 Pour sample from the vial to the glass beaker on the magnetic stirrer and allow to stir.

Note 4: Method for the Preparation of a Sample before Lasersizing, refer to later in this Procedure. Now that the sample is mixed and the Lasersizer is aligned we will proceed with the sizing of a sample.

9 LASERSIZING A SAMPLE

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- 9.1 Now that instrument is successfully aligned, remove any graphs that may appear on the screen by the delete key in the tool bar.
- 9.2 Press the F7 key to set your date.
- 9.3 Type in the date as ddmmyy.SAM (You will see "DEFAULT.SAM" where you have to put the date. Delete the word "DEFAULT" and type in the date as shown).
- 9.4 Click OK
- 9.5 Click Yes. (Screen will display a message "File 120404 does not exist would you like to create a new one).
- 9.6 Type in the Sample ID and click OK.
- 9.7 Follow the Prompt -
"Please place a container of clean water into position and lower the probe. Please wait a few seconds".
- 9.8 Now place the container of clean water into position and lower probe.
- 9.9 Click OK -
"Background to follow"
- 9.10 Click OK -
"If background is greater than 100 click cancel. Please re-flush and/or clean cell. Press Enter if OK"
- Note 5: Average reading for background is between 20 & 50. If the reading is higher, please re-flush the system.**
- 9.11 Click OK -
"Please add sample into the beaker after you press enter"
- 9.12 Click OK
- 9.13 Slowly using a pipette add the sample (from the glass beaker whilst still stirring) to the plastic beaker, up to 10-15% Obscuration.
- 9.14 Press OK and the lasersizer will now analyse the sample and print out the result.
- 9.15 When sizing of sample is complete lift the probe and press OK. Wait for all the water to run from the probe.
- 9.16 Rinse the system and remove beaker from the machine and place a clean 1Ltr plastic beaker with deionised water in position. Lower the probe for 1 minute.

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- 9.17 Replace this beaker with another beaker of deionised water and a squirt of Calgon.
- 9.18 Lower the probe, allow it to run to dispense of air bubbles.
- 9.19 If you have more than one sample to be Lasersized repeat steps 9.1 - 9.18
- 9.20 When you have completed your lasersizings, place a clean plastic beaker of deionised water with a squirt of Calgon. Lower the probe and let it run for 1 minute to disperse of air bubbles.
- 9.21 Turn off the pump and ultrasonic displacement.
- 9.22 Exit window and SAVE the configuration.
- 9.23 Shut down computer and turn off the magnetic stirrer.
- 9.24 Rinse and clean all equipment.

10 PREPARATION OF SRM SAMPLE FOR LASERSIZING

- 10.1 Collect approximately 5kg of a suitable material Pb/Zn Concentrator when operation is at optimum level.
- 10.2 Dry the sample as normal.
- 10.3 Pour the sample into a clean Rotary sample divider.
- 10.4 Make continuous passes through the Rotary sample divider to provide a final sample weight of 20g.
- 10.5 Store these 20g portions in sealed alfoil/poly bags which are suitably marked and dated.
- 10.6 When required open the packet obtained at step 10.3 and spread the contents onto a sheet of clean paper, forming a pipe.
- 10.7 Divide the sample into 5 equal portions of 3-5g and using a spatula place each portion into a plastic vial marked with the SRM number and the week dates it is to be used.

Note 6: A new alfoil/poly bag should be opened at the start of each week and the five portions used during that week.

- 10.8 Proceed to Steps 8.1 - 8.5 (Mixing a sample to be Lasersized)
- 10.9 Then proceed to Steps 9.1 - 9.24 (Lasersizing a Sample)

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11 PREPARATION OF DRY SAMPLES

- 11.1 Clean and prepare Rotary Sample Divider.
- 11.2 Clean sample preparation area and vinyl mat.
- 11.3 Pour the sample into the Divider.
- 11.4 Start the Rotary Sample Divider.
- 11.5 Pass entire sample through Rotary Sample Divider.
- 11.6 Split sample down till you have the required amount.
- 11.7 Remove sample cups with divided sample from Rotary Sample Divider.
- 11.8 Choose sample cup/s with the desired amount of sample and place any reserve in a labeled alfoil/poly bag.
- 11.9 Place sample onto vinyl mixing mat in the sample preparation area.
- 11.10 Cut desired amount (average 3-5gr) from sample with spatula and place cut into a plastic vial with a lid.
- 11.11 Fill vial 2/3 full with deionised water and 3 drops of Calgon and shake sample vigorously to mix.
- 11.12 Then proceed to Steps 9.1 - 9.24 of this procedure (Lasersizing a Sample)

12 PREPARATION OF SLURRY SAMPLES

- 12.1 Thoroughly mix slurry sample, ensuring all solids are in suspension.
- 12.2 Label and date a plastic vial.
- 12.3 Cut out an amount of slurry sample with plastic vial scoop inserted into mixing sample.
- 12.4 Transfer cut slurry sample into appropriately labelled vial.
- 12.5 Then proceed to Steps 9.1 - 9.24 of this procedure (Lasersizing a Sample)

13 CHECKING INSTRUMENT PERFORMANCE

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A instrument performance check using a Malvern Quality Audit Standard for Micro and Microplus Particle Size Analysers is run using the Polydispersed Glass Bead Transfer Standard (purchased from ATA Scientific Pty.Ltd) to determine if the instrument and operator are performing within specification. These glass beads are certified to be a certain size.

- 13.1 Start up computer.
- 13.2 Check that cell window is clean and free from scratches.
- 13.3 Flush and drain the system at least twice, with clean water (de-ionised or better) before the measure to eliminate any contamination from previous samples.
- 13.4 With the beaker filled, turn the pump/stirrer speed down to zero and turn on the ultrasonics for 30 seconds (to allow air bubbles to disperse). Turn off the ultrasonics and run the pump/stirrer up to full, then back down to zero to clear any air bubbles.
- 13.5 Set the pump/stirrer speed to 3500rpm
- 13.6 Add 4 drops of 5 % Nonidet P40 surfactant to the beaker (to avoid bubbles, don not increase the dosage or concentration).
- 13.7 **ALIGN**
- 13.8 Use presentation code, 50AD (Microplus) analysis model: Polydisperse.

Note 7: To do this go into SETUP on the Menu - Click on PRESENTATION, on LHS of the screen change STANDARD to GLASS (this will automatically change the presentation code from 50HD to 50AD). LOAD. Go back into SETUP again and check the presentation code has changed to 50AD and GLASS (just a way to double check).

- 13.9 Set MEASUREMENT SWEEPS and BACKGROUND SWEEPS to 12,000

Note 8: To do this go into SETUP – click on EXPERIMENT - change SAMPLE NUMBER to 12,000 and BACKGROUND NUMBER to 12,000. OK. Go back into SETUP again and check the sweeps have changed to 12000 (just a way to double check).

- 13.10 Perform one measurement of the Glass Microsphere standard – then perform two more repeats – save the data each time.
- 13.11 Flush the system.
- 13.12 Stop the Mastersizer.

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13.13 Read the results generated – the expected results should lie within the limits set out below

	Dv10	Dv50	Dv90
Lower Limit	26.72	45.40	72.38
Median Value	28.43	46.80	77
Upper Limit	30.14	48.20	81.62

13.14 Change the Presentation Code back .

Note 9: Go into SETUP – select PRESENTATION and on the LHS of the screen, change GLASS to STANDARD (this will automatically change the PRESENTATION code from 50AD to 50HD, which is where it should be when running samples). LOAD. Check SETUP to ensure PRESENTATION has changed back to 50HD (just a way to double check).

13.15 Change back the Sweeps Measurement and Background Sweeps.

Note 10: Go into SETUP select EXPERIMENT and change SAMPLE NUMBER and BACKGROUND NUMBER to 2,000 instead of 12,000.

14 QUALITY CONTROL REQUIREMENTS

14.1 Quality Control Sample Frequencies

14.2 The frequency of quality control samples used during batch analysis is as follows

Quality Control Sample type	SRM Number	Frequency	Position
Internally Certified Standard	126	Once per batch	At start of batch

15 REPORTING REQUIREMENTS

Detection Limit:

Maximum number of decimal places to report 2

Maximum number of significant figures to report 4

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16 METHOD VERIFICATION DATA

To be gathered???

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APPENDIX – Form MET03.01FA

CELL CLEANING CHECKLIST		
DATE	CLEANED	SIGN. & COMMENTS

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