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ACTIVITY/PROJECT SPECIFIC PROCEDURE

SP 12-12
MALVERN MASTERSIZER PARTICLE ANALYZER AND THE QS SMALL VOLUME SAMPLE DISPERSION UNIT
Revision 2

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1.0 Purpose and Scope

The purpose of this procedure is to establish a method for determining mean particle size and particle size distributions by use of the Malvern Mastersizer (Mastersizer). The Mastersizer can measure particle diameters between 0.05 and 900 μm with the 300RF lens installed or between 0.5 and 900 μm with the 300mm lens installed. This procedure applies to the use of the Mastersizer with the 300RF lens installed.

The Mastersizer captures scattering patterns from a field of particles with its optical unit. The optical unit is made up of many detectors. Each detector collects the light scattering from a particular range of angles – this is called a sweep. To obtain a representative measurement the Mastersizer will take many (normally over 2000) sweeps for each measurement. The raw data collected is then analyzed by comparing the measured data to either the Fraunhofer or the Mie theories for predicting particle scattering patterns for particles of known characteristics. The Mastersizer fits the measured data to a predicted scattering pattern from theoretical particles and reports a “Residual” value once the analysis is completed. The residual is an indication of how closely the calculated data has been fitted to the measured data. If the residual (expressed as a percentage) is less than 1%, the fit is considered to be good.

This document is a supplement to manufacturer’s manuals for the operation of the Malvern Mastersizer operating manuals. This supplement is not intended to be formal instrument documentation, nor does it cover all features or use of the instrument, but rather a hands-on user’s guide to the typical operation of both the Mastersizer and the QS Small Volume Sample Dispersion Unit. Details of Mastersizer operation can be found in the manufacturer’s manuals kept in the lab.

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the Sandia National Laboratories (SNL) WIPP Online Documents web site.

2.0 Implementation Actions

2.1 Responsibility

The Principal Investigator (PI) or designee is responsible for implementing the requirements of this procedure.

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The Technical Staff is responsible for performing the measurements following the requirements of this procedure, and documenting all required information as described in this SP. Technical staff should familiarize themselves with the manufacturer's manuals for the Malvern Mastersizer for an understanding of assembly parts and nomenclature, the controls, operation, and measurement methods. The Mastersizer is controlled by software via the host computer.

If the procedure cannot be implemented as written, the user has the responsibility to stop work and resolve all concerns with the PI/designee or safety, as appropriate, prior to proceeding with the work.

2.2 Safety

The activities described in this SP shall conform with the SNL Environmental Safety and Health programs (ES&H) as described in the current version of the laboratory TWD/SOP.

All users shall read the Malvern Health and Safety Manual before using the Mastersizer.

The Malvern Mastersizer uses a Class 3A laser to analyze particle sizes. Laser radiation can be harmful to the body and can cause permanent eye damage. The system does contain safety interlocks to prevent instrument operation if any of the optical unit safety interlocks are defeated.

Note: No one, except a qualified Malvern representative, should remove the transmitter or receiver covers of the instrument (see diagram in the *"Getting Started"* manual page 2.2). Removal of these covers may result in the emission of laser radiation or exposure to dangerous voltages.

2.3 Instrument Startup

- Turn on the host computer
- Turn on the Mastersizer (red button will light up) and turn the Laser key to the 'on' position (green LED will light up).
- Make sure that the dispersion unit is connected, and powered up following the instructions in chapter 3 of the QS Small Volume Sample Dispersion Unit manual.
- Start up the Mastersizer software.
- Fill the dispersion unit tank with distilled water. Following the instructions in chapter 6 of the QS Small Volume Sample Dispersion Unit manual clean the dispersion unit tank, and flush the system. Flush the system at least three to six times to ensure a clean start. Once the tank is clean fill the dispersion unit tank a final time, turn the pump speed down to zero for 3 seconds to allow air to dissipate.
- Set the dispersion unit pump speed (recommend starting with ~1600 rpm).
- Wait for the system temperature to stabilize, and then proceed with the steps below.

2.3.1 Setup Hardware/Software

Before taking measurements:

- Verify the hardware settings are correct in the Mastersizer software. From the Setup-Hardware dialogue verify the 300RF lens is selected from the "Range" drop down menu, and that the QS small volume sample dispersion unit is selected from the "Sample Unit" drop down menu. Refer to the "Getting Started" manual page 4.6 for instructions on instrument preparation/hardware setup.
- Select the analysis mode (Monomodal is recommended to start per the QAS3001 Certificate of Analysis, however other modes can be used) in the Mastersizer software. Refer to chapter 5 of the "Getting Started" manual for information on how to choose the correct analysis mode.

- Select the 3OAD Malvern Presentation Grid in the Mastersizer software. Refer to chapter 5 of the "Getting Started" manual for information on how to choose the correct presentation.
- From the Setup-Experiment dialogue set the measurement sweeps and background sweeps to 12000, and set the number of experiments to be taken to three (3), one measurement, followed by two repeats.

2.3.1.1 Select the Proper Lens

The Malvern Mastersizer has two lenses available for use; the 300RF with a particle size range of 0.05 – 900 μm or the 300mm with a particle size range of 0.5 - 900 μm. Ensure that the 300RF lens is installed. Following the instructions in the *"Getting Started"* manual chapter 4 (page 4.3) select the proper lens.

2.3.1.2 Installation of Lens

Following the instructions in chapter G1, page G.31, of the *"Installation"* manual to install the lens selected above.

2.3.2 Align the System

The laser must be aligned centrally on the detector. The software requires an alignment of the system once per session. Other dialogues will be grayed out until the alignment is performed. Refer to the *"Getting Started"* manual page 4.10 for instructions on aligning the system.

- From the Measure-Align dialogue, select Options. Make sure Auto is checked for automatic alignment of the optics.
- Select Start. The instrument will automatically align.
- When correctly aligned the laser power reading should show a value greater than 75.

2.3.3 Measure Background

A background measurement is used to subtract the ambient light signals from the total scattering received from a sample. Refer to chapter 4 of the *"Getting Started"* manual, page 4.11 for instructions on taking a background measurement.

- From the Measure-Align dialogue, select Next. The Measure-Background dialogue will appear.
- Select Start. The instrument will automatically take the background measurement.

2.3.4 Add the Sample to the Dispersion Unit

The system measures the percentage of light lost from the analyzer beam when the sample is introduced to the system. This is called obscuration. Ideally the obscuration should be between 10% and 30%. The obscuration value is displayed on bottom left of the dialogue box, and an obscuration bar is located in the center of the dialogue. When the obscuration bar is green the sample is in the "ideal" range. Refer to chapter 4 of the *"Getting Started"* manual, page 4.12 for instructions on adding the sample to the dispersion unit.

- From the Measure-Background dialogue, select Next. The Measure-Inspect dialogue will appear.
- Select Start. The instrument will automatically measure the obscuration.

- Add the Quality Audit Standard or sample to the dispersion unit. Watch the obscuration bar or value displayed in the dialogue box. Ideally the obscuration should be between 10-30%. The obscuration bar will be green when this is achieved.
- Allow the obscuration to stabilize (indicating the sample has properly dispersed) before proceeding. The time for stabilization may vary depending on the sample.

2.3.5 Record Calibration and Sample Information

From the Measure-Document dialogue enter information about the calibration check or sample to be executed. Refer to the *"Getting Started"* manual page 4.8 for instructions on documenting the measurement. As a minimum, the following should be entered into this dialogue:

- Quality Audit Standard batch number or sample ID
- Quality Audit Standard bottle number or sample description
- Dispersant material

2.3.6 Calibration

The Mastersizer is not an instrument that is calibrated using known standards. Instead the Mastersizer measures scattering patterns of a sample and then fits the measured data to a predicted scattering pattern from theoretical particles of known characteristics. The Mastersizer performance can be checked using a sizing standard. Malvern Instruments supplies polydisperse glass beads Quality Audit Standards (QAS) for checking the performance of the Mastersizer.

A calibration check of the instrument should be performed every six months. Follow the steps below to carry out a Calibration Check of the Mastersizer, using the Measurement Protocols provided by Malvern using the QAS3001 polydisperse glass beads or equivalent.

- Compare results from the Calibration Check to the table below. Results should be within 2% on the D(v, 0.5) and 3% on the D(v, 0.1) and D(v, 0.9) median values. Record the results of the comparison of the median value to the measured value in the Scientific notebook to be used for sample analysis.

	D(v, 0.1)	**D(v, 0.5)**	**D(v, 0.9)**
Median Values	**37.94**	**61.41**	**89.00**

2.3.7 Analyze the Data

- From the Measure-Sample dialogue, select Next. The Measure-Calculate Result dialogue will appear.
- The instrument will automatically analyze the sample measurement data.
- Once the analysis is complete the data table and graph will appear on the screen.

2.3.8 Reporting

- Once the sample has been measured and analyzed the data report should be printed. The report should be recorded in a Scientific Notebook or a Scientific Notebook Supplement..
- Save after each measurement is taken. If the software crashes there is no way to recover the data if it has not been saved.

2.4 System Shutdown

- Following the instructions in chapter 6 of the QS Small Volume Sample Dispersion Unit manual clean the dispersion unit tank and flush the system three to six times with distilled water. Materials that can not be disposed of in the sewage or garbage will be treated as hazardous materials.
- Fill the dispersion tank a final time with distilled water. Tank should be filled to the top of the rim.
- Turn off the dispersion unit.
- Shutdown the Mastersizer software and turn off the host computer.
- Turn the Laser key to the 'off' position and turn off the Mastersizer.

2.5 Maintenance

Refer to chapter 11 of the *"Getting Started"* manual for instructions on how to maintain the Mastersizer.

Cleaning the Optics

Cleanliness of the Mastersizer optics is necessary to ensure good measurements. It is not expected that the range lenses and cell windows will require cleaning before each use of the Mastersizer, however when it is necessary, follow the steps outlined in Chapter 11 of the *"Getting Started"* manual. Always turn off the laser and unplug the unit before cleaning.

3.0 Records

The following records, generated through implementation of this procedure, shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records):

QA Record

- SN (Scientific Notebook)
- SNS (Scientific Notebook Supplement)
- Calibration Check using the QAS3001 polydisperse glass beads or equivalent
- Mastersizer Measurement and Analysis Data Report

4.0 Appendices

There are no appendices in this SP.

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