IMPORTANT NOTICE: The current official version of this document is available via the Sandia National Laboratories WIPP Online Documents website. A printed copy of this document may not be the version currently in effect.

ACTIVITY/PROJECT SPECIFIC PROCEDURE

SP 12-12
MALVERN MASTERSIZER PARTICLE ANALYZER 3000 AND THE SAMPLE DISPERSION UNITS
Revision 3

Effective Date: March 25, 2019

Author:	Cassie Marrs	*Original signed by Cassie Marrs*	*3/25/19*
	(printed name)	(signature)	(date)

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 3000 (Mastersizer). The Mastersizer can measure particle diameters between 0.01-3500 µm.

The Mastersizer is comprised of the main optical unit, two dispersion units and a measurement cell. The purpose of the main optical unit is to transmit red laser light and blue light through a sample and then use its detectors to generate data about the light scattering pattern caused by particles in the sample. There are two dispersion units, a wet and dry unit. The wet unit, Hydro MV controls the dispersion of a sample suspended within a liquid dispersant. The dry unit, Aero S, ensures that a dry sample is dispersed and evenly fed to the measurement cell within a continuous stream of air. The cell is the interface between the dispersion unit and the optical unit. The sample is routed between measurement windows in the cell so that the laser can pass through it in order to make a measurement. The mastersizer application software controls the optical and dispersion units and also processes the raw data, providing flexible data analysis and reporting features.

This document is a supplement to manufacturer's manuals for the operation of the Malvern Mastersizer 3000 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 sample dispersion units (Hydro MV and Aero S). 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.

© 2019 NTES of Sandia, LLC.

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 3000 and sample dispersion units for an understanding of assembly parts and nomenclature, the controls, operation, and measurement methods. The Mastersizer and sample dispersion units are 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 3000 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. Removal of these covers may result in the emission of laser radiation or exposure to dangerous voltages.

2.3 Powering On and Starting the Software

1. Switch on the optical unit. The blue light on the top of the instrument indicates that it is on. Check also that the blue light on the dispersion unit is pulsating, showing that the dispersion unit has power. **Note:** Leave the instrument powered on for 30 minutes before making a measurement to allow the laser to stabilize.
2. When 30 minutes has elapsed, switch on the computer, log in to Windows on the computer and start the software by double-clicking the **Mastersizer** icon on the desktop.
3. The main mastersizer application software window is now displayed. A correct connection to the instrument shows both the instrument and accessory name in the status bar.

2.3.1 Creating and Editing a Standard Operating Procedure (SOP)

1. Select **New-SOP** from the **Home** ribbon. **Note:** To edit an existing SOP, choose **Open-SOP** instead.
2. Select an SOP template from the list in the **New SOP** window or click the **From an existing SOP...** button to locate an SOP in the file system that fulfils (or is close to) your required criteria.
3. The **SOP editor** window is now displayed
4. Step through the **SOP Editor** by clicking the section titles on the left (**Sample-Identification, Sample-Material** etc.) or clicking the Forward/Back arrows on its toolbar.
5. When all the required settings have been specified, you can either choose to save the SOP as a file or as a template. Choose **File-Save** and then give the SOP a logical name.

2.3.2 Make a Measurement with the Hydro MV

1. Select **Run SOP** from the **Measurements** section of the ribbon.
2. The **SOP Selector** window is shown, which initially presents all SOPS filtered for the connected accessory.
3. Select SOP and then click OK
4. The **Measurement Display** window is shown – the progress status bar at the top of the window reports what is happening and shows what to do next.
5. Click the **Start** button to **Initialize the instrument**. If specified in the SOP settings, the system prompts the user to enter/confirm sample documentation details. Following this stage, the system automatically moves to the **Measure Background** stage and measures both the red and blue light values of the background.
6. When this is complete, the SOP pauses. The system now requests that you add sample – do this until the **Obscuration bar** (in the laser panel) indicates about 10-20%. This is a rough guide to a suitable value for a wet dispersion unit – refer to the Help system for more guidance.
7. Click the **Start** button to disperse the sample into the system and then commence the measurement. The system measures first the red and then the blue light values. The SOP may make several measurements before completion, depending on the **Number of measurements** specified in the SOP settings. When complete, the **Trend view** is updated with new measurement figures.
8. Complete the measurement by closing the **SOP Measurement** window.
9. The measurement is complete, the results are now presented in the **Record View.**

2.3.3 Make a Measurement with the Aero SS

1. Open the Aero S sample area lid and set the hopper flow rate to an appropriate gap level using the dial.
2. Add your sample into the hopper.
3. The mesh basket and ball bearings may be used to smooth the flow of sample, breaking up any loose agglomerates before they fall into the hopper.
4. Close the lid of the Aero.
5. Select **Run SOP** from the **Measurements** section of the ribbon.
6. The **SOP selector** window lists all available SOPs.
7. Choose an appropriate SOP for the Aero dispersion unit and venture type and then click OK. The **Measurement Display** is shown.
8. Click **Start-** the SOP automatically, turns on the air, aligns the optical system and then measures the background using red light.
9. After this has completed, the sample tray starts vibrating and the air pressure specified in the SOP is applied. The overall rate at which the sample is fed into the cell is a function of the combination of air pressure, vibration rate and hopper flow-rate setting.
10. This SOP is optimized for the dry dispersion unit and will only make a measurement once the obscuration is within the specified range (1-10% obscuration is the rough guide for a dry dispersion unit - refer to the Help system for further advice on obscuration setting).
11. The results are shown immediately in the **Trend view** as they are gathered. Once all of the measurements have been made, the system pauses - optionally, **click Clean System** on the top progress bar to initiate a cleaning cycle.
12. Complete the measurement by closing the SOP Measurement window. The results will now be presented in the **Record View.**

2.3.4 Measuring Samples in Manual Mode

1. From the **Home** ribbon click **Manual measurement** from the **Measurements** group. The **Manual Measurement** window is displayed, together with the **Manual Measurement Settings** window.
2. Choose the **Sample-Identification** settings to record both the **Sample Name** details and define any further information fields (for example, Notes) that will help users to repeat the measurement at a later date.
3. Use the **Sample-Material** section to define the optical properties (the **Refractive index** and **Absorption index**) of the new material. The material may already be stored in the system's **Materials database**. Check this first by clicking **Browse database** – select the material if it is present to automatically add the RI and Absorption values to the measurement.
4. Use the **Measurement-Duration** section to setup measurement times for the red and blue measurements.
5. In the **Measurement-Obscuration** section define the lower and upper obscuration limits – as a very rough guide, about 10-20% for a wet dispersion unit and 1-10% for a dry dispersion unit.
6. Click OK to close the **Manual measurement settings** window.
7. From the **Initialize Instrument** drop down menu, select **Auto Alignment**.
8. Click **Fill** on the Accessory panel to add dispersant to the tank. The beaker in the software indicates when the unit is full.
9. Drag the **Stirrer** slider bar to a rate (RPM) that is appropriate for your sample and dispersant and then click the **Start** button next to the **Stirrer** slider bar. The dispersant is now circulated through the cell.
10. Click the large **Start** button on the measurement sequence bar to initialize the instrument – when complete, the system progresses to the **Measure Background** stage.
11. Click **Start** again – both the red and blue light values for the background are measured.
12. The system now requests the user to **Add Samples** – add small quantities of sample to the hydro unit until the obscuration is within range (in the case of a wet dispersion). With a dry dispersion, the system will skip this step.
13. Click **Start** again twice to begin the measurement. First the red and then the blue light values are measured. When this is complete, the **Trend view** is updated with new measurement figures.
14. Continue this final **Measure Sample** process as many times as required by clicking **Start** again. To complete the measurement, just close the **Manual Measurement** window. The new records are now added to the **Record View.**

2.3.5 Record Calibration and Sample Information

Follow the instructions from Section 2.3.2 to perform a calibration. As a minimum, the following should be entered into this dialogue:

1. Quality Audit Standard batch number or sample ID
2. Quality Audit Standard bottle number or sample description
3. 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.

Results will meet the certified values within the D(v, 0.5), 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.

2.3.7 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.

2.4 System Cleaning

1. In the **Active accessory** control window (**Tools-Accessories)** pressing clean will start the cleaning process.
2. You can select the cleaning sequence required: Quick (1 cleaning cycle), Normal (3 cycles), Extensive (5 cycles). The dispersion unit will perform a clean sequence, for the selected duration, that will remove any remaining sample from the unit.

2.5 Maintenance

Refer to the Basic Guide in the owner's 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 the Basic Guide in the owner's 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)
- Certificate of Analysis using the QAS3001 polydisperse glass beads or equivalent
- Mastersizer Measurement and Analysis Data Report

4.0 Appendices

Not Applicable. There are no appendices in this SP.

This work of authorship was prepared as an account of work sponsored by an agency of the United States Government. Accordingly, the United States Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so for United States Government purposes. Neither the National Technology and Engineering Solutions of Sandia, LLC., the United States Government, nor any agency thereof, nor any of their employees makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately-owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the National Technology and Engineering Solutions of Sandia, LLC., the United States Government, or any agency thereof. The views and opinions expressed herein do not necessarily state or reflect those of the National Technology and Engineering Solutions of Sandia, LLC., the United States Government or any agency thereof.

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy’s National Nuclear Security Administration under contract DE-NA-0003525.

Parties are allowed to download copies at no cost for internal use within your organization only provided that any copies made are true and accurate. Copies must include a statement acknowledging Sandia's authorship of the subject matter.