

Laser Diffraction Testing Guide: MS3000

RACING SURFACES TESTING LABORATORY

PREPARED BY MACKENZIE ROCKEFELLER



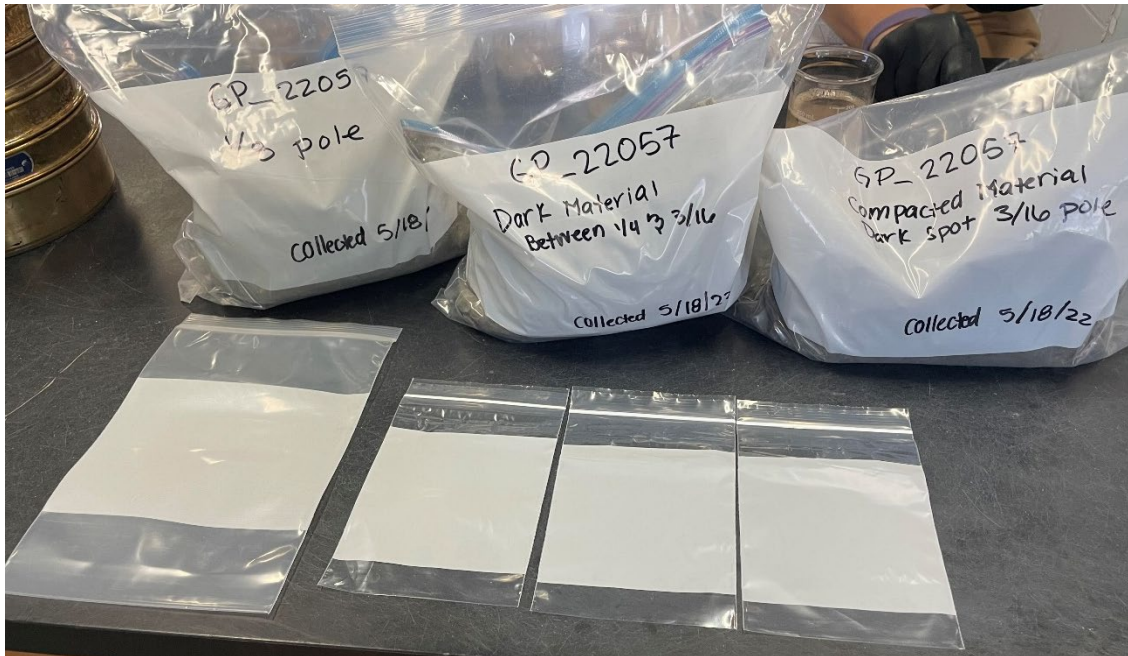
Table of Contents

I. Procedure: Screening and bagging samples	2
II. MS3000 Operation:.....	6
III. Procedure: Preparing samples for testing	12
IV. Exporting Data	18
V. Hydrosight Operation	20

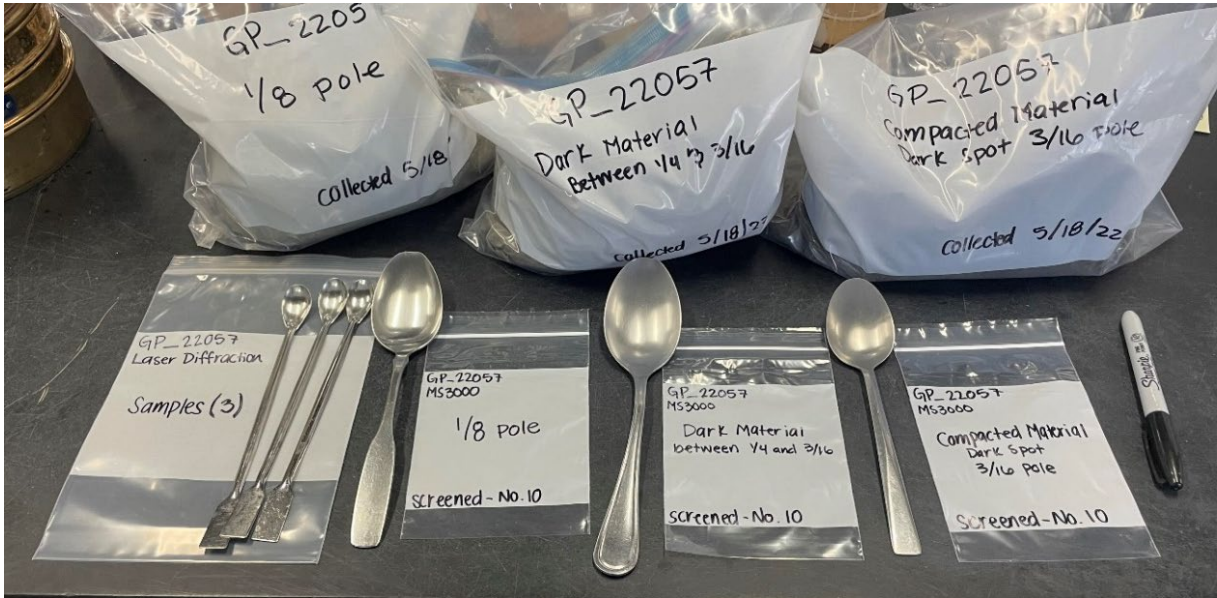


I. Procedure: Screening and bagging samples

1. Upon collection or receipt of samples, thoroughly mix the material in the sample bag to promote sample homogeneity. If the bag is damaged or compromised, transferring the sample to a new container is recommended.
2. Obtain **one medium-sized sample bag** per project, and **one small-sized sample bag per sample**. These will be used to store the samples together as a project after each has been screened through the No. 10 sieve.



3. Label the main project bag with the project ID at the top left (i.e., "GP_22057") and "Laser Diffraction" directly underneath. In the middle of the bag, write how many samples are included with the project (i.e., "Samples (7)").
4. Label the smaller sample bags with the project ID at the top left, "MS3000" directly underneath the project ID, the sample name in the middle of the bag, and "Screened- No.10" at the bottom of the bag.
5. Set all labeled bags aside. Grab **one long metal spoon per sample** and set aside with the bags.



6. Place a **No. 10 sieve** on top of a **Sieve Catch Pan**. With a **clean metal tablespoon**, mix the sample one more time in its original bag and place a spoonful of the sample (approximately 150.0 +/- 50.0 g) on top of the No. 10 Sieve screen mesh.



7. With gloves on, gently massage and sieve the material placed onto the No. 10 sieve by hand.
*If any aggregates/clumps are found, break these by pinching and rolling the material by hand.

Continue working the material through the No. 10 sieve until at least 100.0 +/- 25.0g or more of the sample has passed through the No. 10 sieve.

*If material is getting stuck in the sieve, gently tap the sieve on the table to encourage material to move through (shown in photos below). All material passing is not required



as the sample sizes required for laser diffraction is typically 0.7-5.0 grams per test. This will allow for future retesting as required.



8. Discard any material remaining in the No. 10 sieve. These particles are too large to go through the MS3000 without clogging/damaging it.
9. Collect the screened material in the Sieve Catch pan and place in the appropriately labeled sample bag along with a clean long metal spoon. Zip the bag closed and set aside until all samples have been screened and bagged.





10. Repeat steps 6-10 for each sample to be tested. **Be sure to use a clean No. 10 sieve, Sieve Catch pan, metal tablespoon, and wiped/cleaned off gloves for each sample** – leftover silt and clay may contaminate subsequent samples!





II. MS3000 Operation:

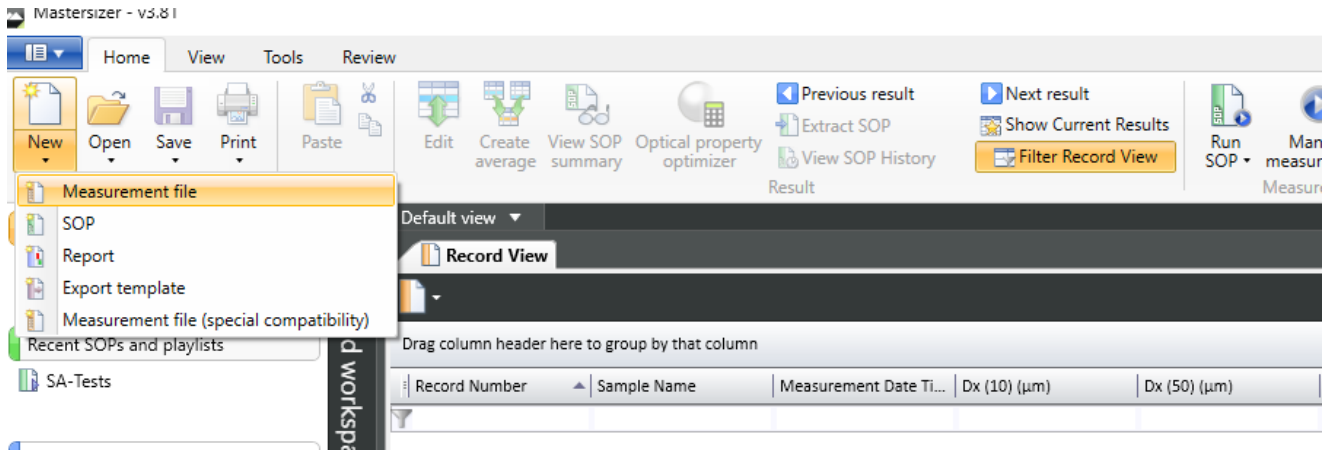
1. Before operation, on the left-hand side of the Mastersizer3000 (MS3000) press the power button. The machine will have a blue light turn on to indicate power is being received.



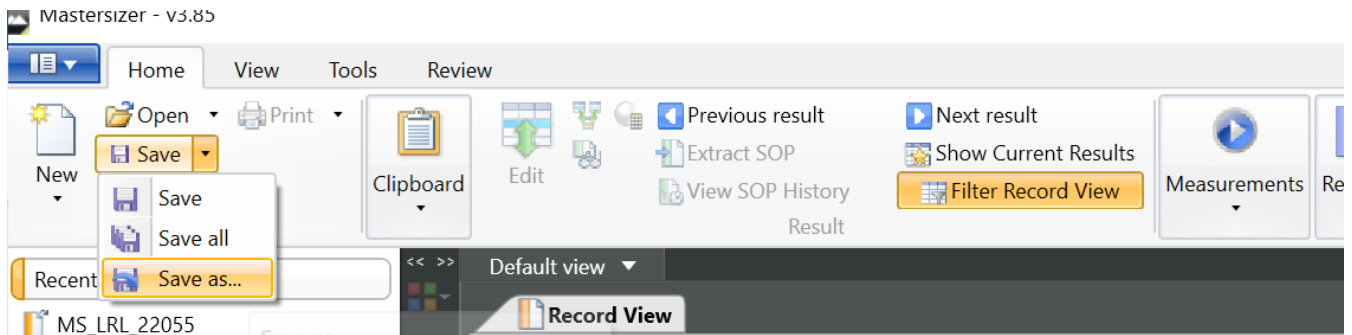
2. On the Desktop, open the MS3000 program.
***Note: If these are the first samples being run since the MS3000 has been turned on, please allow the MS3000 and Malvern software to be on for *at least 30 minutes* prior to testing for the laser to stabilize!**



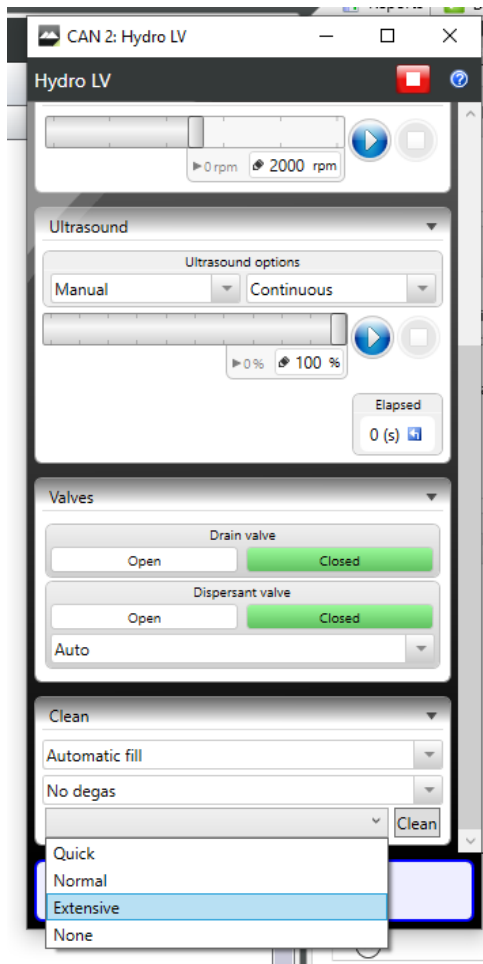
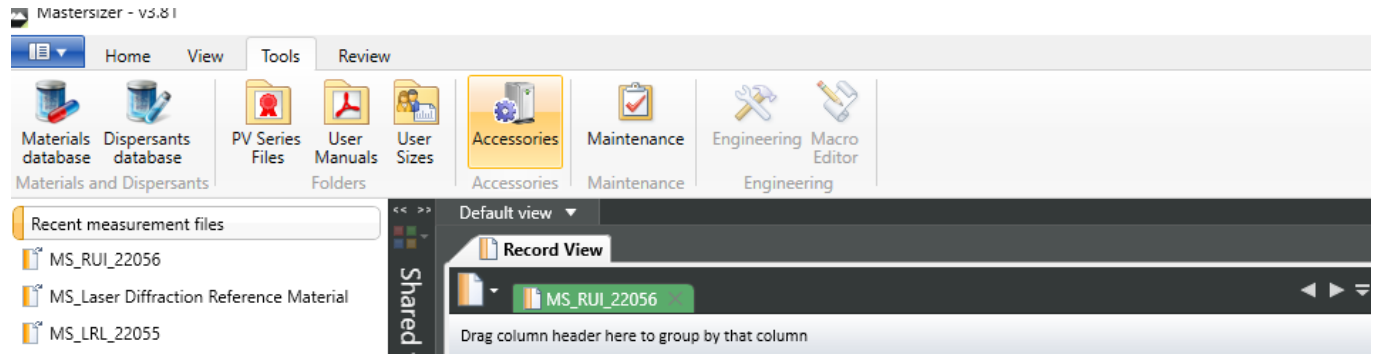
3. In the sample project folder on Dropbox, create a new folder called: 'MS'.
4. In the upper left-hand corner of the screen, select 'New' and then 'Measurement file'.



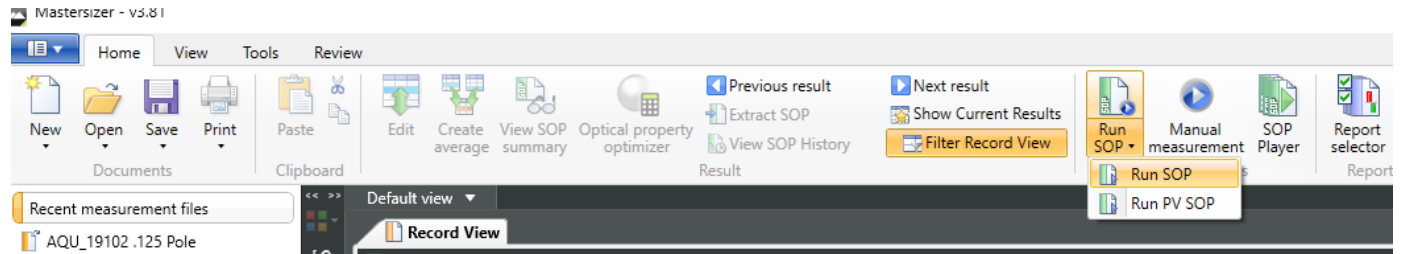
5. Once a new measurement file has been created, click 'Save as...' and store the measurement file in the 'MS' folder you just created in Dropbox. Make sure to save the measurement file with the appropriate project ID.
 - a. MS_Track_ProjectNumber
 - b. Example: MS_GP_22057.mmes



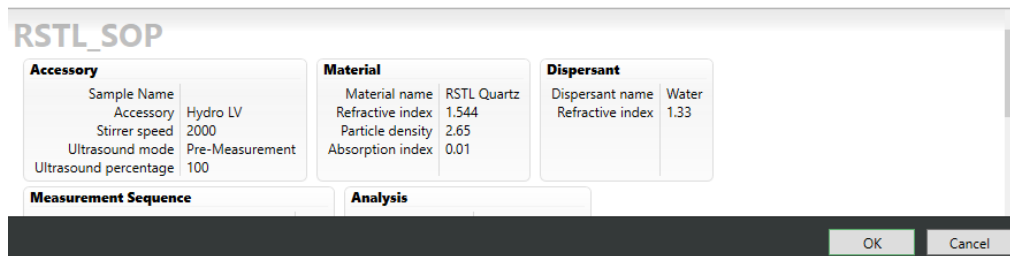
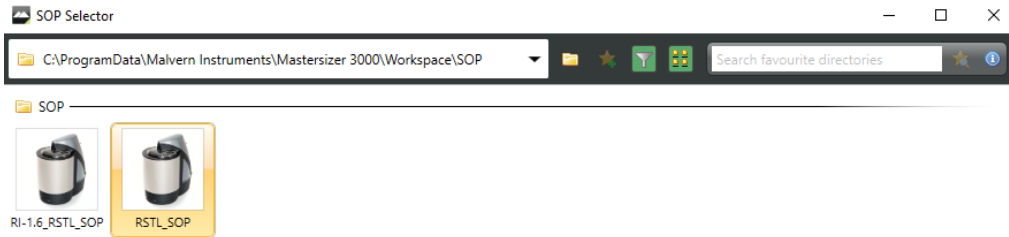
6. Before starting any new project, run an 'Extensive Clean' cycle. On the top left of the screen, click on the 'Tools' tab, then click on 'Accessories'. On the pop-up, scroll down to the blank drop-down box under 'Clean' and select 'Extensive', then click 'Clean'. Rinse down the sides of the wet dispersion unit with Distilled Water every time the tank empties. The cycle will be completed once the tank is filled and the light turns off on the wet dispersion unit.



7. Once the Extensive clean cycle is completed, go to the upper-middle of the program and select 'Run SOP' as shown.

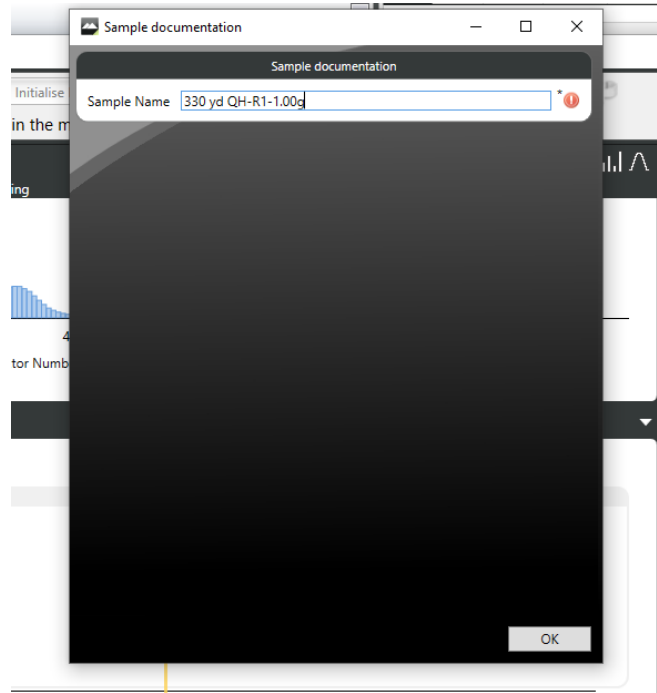


8. A pop-up window should open the option to select the SOP. Select 'RSTL_SOP' and click 'OK'.



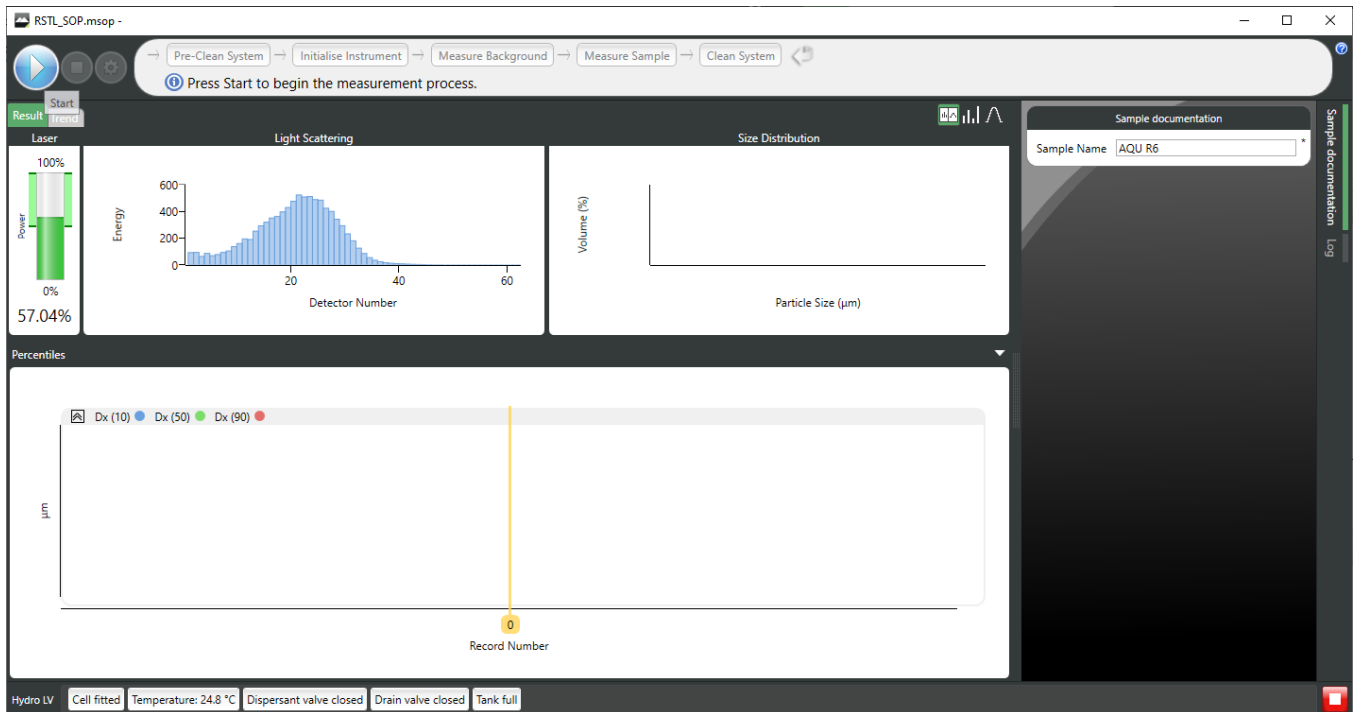
9. After selecting 'OK' the window will close and a new window will open with a prompt to input the sample name. Input the first **sample's name, which run** (R1, R2, R3...), and the **sample size** (i.e., 1.00g) and select 'OK'.

a. Example: 330 yd QH-R1-1.00g



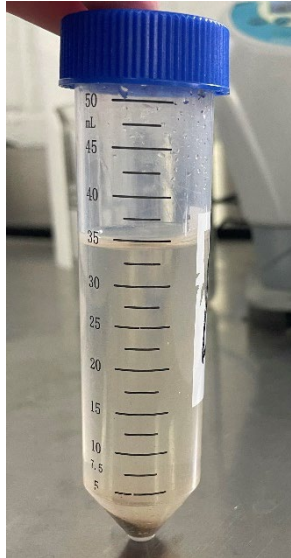
10. Again, if this is the first sample being run with the MS3000 on, then it MUST be left on for no less than 30 minutes to allow the laser to stabilize. Failure to do so may result in poor data.

You should see the screen below once the MS3000 is ready to start taking measurements.





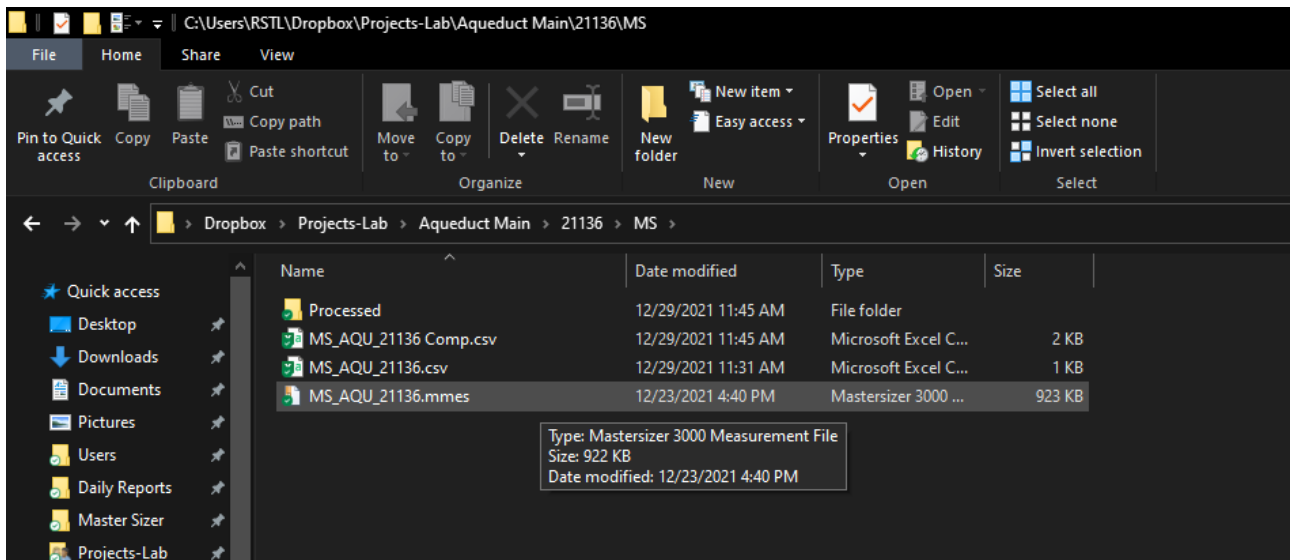
11. At this time, a sub sample of the surface material should be prepared as outlined in the Section III.





III. Procedure: Preparing samples for testing

1. Determine the appropriate sample size needed for this particular location. Sample sizes can range from 0.5g at the lowest to 10g at the highest. Check DropBox to see if the location you are testing has been tested through the MS3000 before – the project folder should include a folder named “MS” and have a .mmes file. Open this .mmes file in the Malvern software to see what previous samples sizes used were.



2. If the location you are testing has NOT been tested before, a safe place to typically start is 1.00g. Prepare the sample using the procedure outlined below and run it through the MS3000 once to see if the desired values for Weighted Residual and Laser Obscuration are met. If so, you may prep the next sample at the same sample size.

***Note:** This amount must be determined experimentally and only serves as reference for future sampling. *Typical* values expected for a sample may range from 0.7-3.5g of material. Cleaner sands – sands used in synthetic surfaces or top-dressings – may require up to 10g to reach the specified Laser Obscuration. Maintaining a consistent Laser Obscuration is important for the repeatability of the testing. The Laser Obscuration range recommended by the manufacturers is generally **10-20%**. By keeping the required Laser Obscuration as high as possible within this range, the more amount of material is allowed to be put into the wet disperser and optical bench without effecting the accuracy and precision of the machine.



Use discretion to either increase or decrease sample size; **if you are unsure of how to proceed, please ask someone in the lab for help!**

General Rules of Thumb:

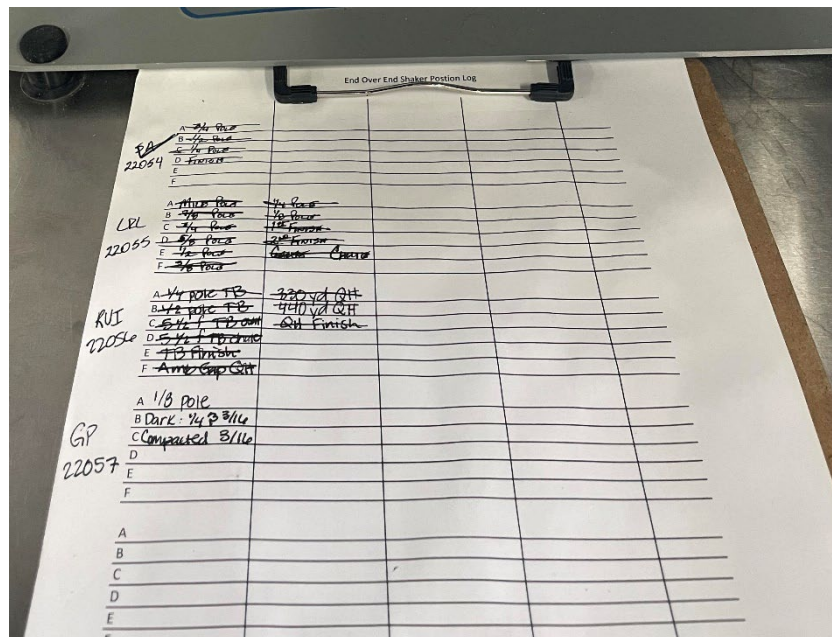
- If Laser Obscuration is $> 20\%$ \rightarrow decrease sample size
 - If Laser Obscuration is $< 10\%$ \rightarrow increase sample size
 - If Weighted Residual is $> 1\%$ but Laser Obscuration is in range, run a clean cycle and test again at same sample size. If still $> 1\%$, decrease sample size.
3. Once an adequate sample size has been determined, take the sample bags to the scale. Place a **clean sample boat** on the scale and **tare**.



4. Using the long metal spoon already placed in the sample bag, scoop out the desired amount of material onto the sample boat. Grab a **clean test tube with a blue lid** and pour the weighed material into the test tube. Each sample will need two runs, so repeat this step once more. **You should have two identical test tubes with material for each sample.**



- Before soaking the screened material, fill out the project ID and sample names on the “**End Over End Shaker Position Log**” in order to keep track of which samples are in which shaker positions. Write the project ID to the left, and start with the ‘A’ position for the first sample. In the shaker, there will be two spots (A1 and A2) that are designated for the two test tubes per one sample. **Continue to label the samples in chronological order (A->F) corresponding to the order the samples will be tested.**





6. Add about **35ml of the 5 g/L SHMP solution** to each test tube containing the screened material. Place the test tubes into the End Over End Shaker in the correct location according to the position log.

***Make sure both sides of the shaker are equal in weight;** if at any time there is one less tube on one side than the other, use one of the “counter balance” test tubes that are filled with distilled water.

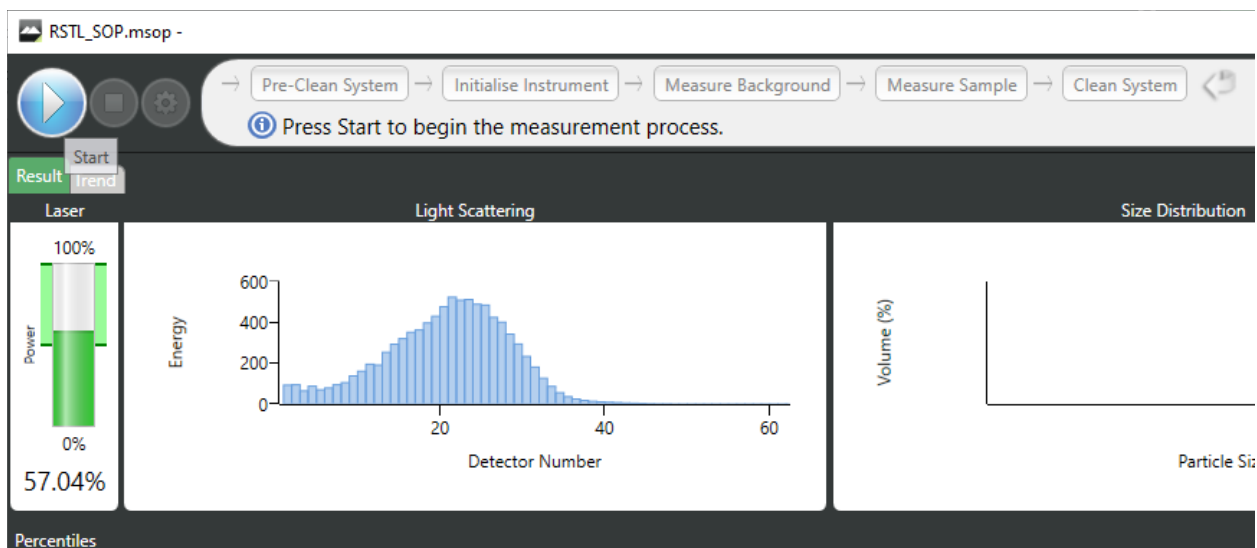


7. Turn on the shaker by holding down the power button. Then, press the green “Start/Stop” button. To switch sides, press the blue button on the far right with the diagram of two test tubes. **Allow samples to mix for at least 2 minutes prior to beginning testing.** It will not hurt the sample to remain shaking for longer than that.





- Once the MS3000 has been properly warmed up to allow laser stabilization, go back to the **Malvern Software** and **select the 'Play'/'Start'** button on the pre-measurement screen in the upper left-hand corner.



- At this time, the machine will begin taking background measurements. After the blue-light background scan is completed, **you will be prompted to add sample until the material has reached the specified 'Obscuration Range'** shown on the left-hand side of the program.
- Transfer all contents of the test tube using a distilled water filled squirt bottle** into the wet dispersion unit until the Laser Obscuration is in range. **Make sure to rinse the test tube AND the cap well into the unit so that no particles remain.** Place the plastic covering onto the wet dispersion unit and wait for MS3000 to take 5 measurement scans.
 - *No more than 25 mL of distilled water should be added into the wet dispersion unit as this will cause water overflow.
- Upon completion of the 5 measurement scans, **thoroughly clean the system by flushing the sides of the tank with distilled water every time the tank drains.** Look to see if any sand particles are stuck to the sides and/or metal tubes and rinse well.
 - *Failure to thoroughly clean the system may result in poor background scans between samples and/or result in cross contamination. This will affect the accuracy and precision of results.



12. If the Weighted Residual of the testing is over 1.0%, the sample and or replicate must be reformed.
13. If the Laser Obscuration does not fall in range of 10-20% the sample and/or replicate must be reformed.
14. Repeat steps 14-23 for each sample in the project.

Things to remember:

- a. There must be two completed runs per sample – with the same sample size – that meet the above requirements;
- b. Be sure to record accurate sample sizes for each run, especially when trying to determine the correct amount;
- c. Rinse the water dispersion unit every time it drains; and
- d. Save the file after every run.



IV. Exporting Data

1. After testing has been completed on a sample set/project, using the control key (CTRL), click on the average of each run such that they are the only selected data sets. Then click on 'Export Selected Records' in the upper middle screen.

The screenshot displays the Mastersizer software interface. The main window shows a 'Record View' table with columns for Record Number, Sample Name, Measurement Date, and particle size distributions (Dx). The table contains 66 records, with several rows selected. A 'Reports' window is open on the right, showing a detailed report for 'Silica SiO2'. The report includes parameters such as Particle Refractive Index, Particle Absorption Index, Dispersant Name, and Specific Surface Area. A graph titled 'Frequency (compatible)' shows Volume Density (%) versus Size Classes (µm) on a logarithmic scale. Below the graph is a 'Result' table with columns for Size (µm) and % Volume In.

Record Number	Sample Name	Measurement Date	Dx (10) (µm)	Dx (50) (µm)	Dx (90) (µm)	Operator
41	AQU R4	8/17/2020 6:21:39 PM	12.0	276	650	RSTL
42	Average of 'AQU R4'	8/17/2020 6:17:21 PM	11.7	279	652	RSTL
43	AQU R5	8/17/2020 6:29:21 PM	11.2	273	615	RSTL
44	AQU R5	8/17/2020 6:30:25 PM	11.3	274	662	RSTL
45	AQU R5	8/17/2020 6:31:30 PM	11.3	273	628	RSTL
46	AQU R5	8/17/2020 6:32:34 PM	11.6	285	674	RSTL
47	AQU R5	8/17/2020 6:33:39 PM	11.3	266	607	RSTL
48	Average of 'AQU R5'	8/17/2020 6:29:21 PM	11.7	274	637	RSTL
49	AQU R6	8/18/2020 10:55:10 AM	13.6	290	713	RSTL
50	AQU R6	8/18/2020 10:56:15 AM	15.3	298	675	RSTL
51	AQU R6	8/18/2020 10:57:19 AM	11.1	271	592	RSTL
52	AQU R6	8/18/2020 10:58:23 AM	12.2	275	607	RSTL
53	AQU R6	8/18/2020 10:59:28 AM	11.5	273	594	RSTL
54	Average of 'AQU R6'	8/18/2020 10:55:10 AM	12.6	281	634	RSTL
55	AQU R7	8/18/2020 11:08:19 AM	10.0	268	626	RSTL
56	AQU R7	8/18/2020 11:09:23 AM	11.7	283	669	RSTL
57	AQU R7	8/18/2020 11:10:28 AM	12.2	288	713	RSTL
58	AQU R7	8/18/2020 11:11:32 AM	11.0	271	640	RSTL
59	AQU R7	8/18/2020 11:12:37 AM	11.9	280	646	RSTL
60	Average of 'AQU R7'	8/18/2020 11:08:19 AM	11.3	278	657	RSTL
61	AQU R8	8/18/2020 11:20:49 AM	10.8	275	615	RSTL
62	AQU R8	8/18/2020 11:21:54 AM	13.3	282	659	RSTL
63	AQU R8	8/18/2020 11:22:58 AM	12.5	284	658	RSTL
64	AQU R8	8/18/2020 11:24:02 AM	12.0	277	640	RSTL
65	AQU R8	8/18/2020 11:25:07 AM	12.4	277	675	RSTL
66	Average of 'AQU R8'	8/18/2020 11:20:49 AM	12.1	279	649	RSTL

2. A pop-up window will open. In the dropdown 'Template' menu, select 'RSTL Export' to create the correct output file. Then select 'Export to file...'.



Export Data

Export data

Default template
This default template uses the record view column configuration.

Default template
This default template uses the record view column configuration.

RSTL Export
General Exporting format.

42	Average of 'AQU R4'	8/17/2020 6:17:21 PM	11.7	279	652	RSTL	MAL1236250
48	Average of 'AQU R5'	8/17/2020 6:29:21 PM	11.7	274	637	RSTL	MAL1236250
54	Average of 'AQU R6'	8/18/2020 10:55:10 AM	12.6	281	634	RSTL	MAL1236250
60	Average of 'AQU R7'	8/18/2020 11:08:19 AM	11.3	278	657	RSTL	MAL1236250
66	Average of 'AQU R8'	8/18/2020 11:20:49 AM	12.1	279	649	RSTL	MAL1236250

Export to file... Export to clipboard Cancel

Export Data

Export data

RSTL Export
General Exporting format.

File Name,Sample Name,Measurement Date Time,Laser Obscuration,Dx (10),Dx (50),Dx (60),Dx (90),1.00,2.00,53.0,74.0,105,149,250,420,500,1000,1410,2000,2380,2830,3360
 AQU_19102_125 Pole,Average of 'AQU R1'-1.5g,8/17/2020 5:40:04 PM,18.74,11.3,277.335,634.3,0.0,13.01,1.53,2.86,6.10,18.57,26.89,8.55,18.58,0.85,0.00,0.00,0.00,0.00
 AQU_19102_125 Pole,Average of 'AQU R2',8/17/2020 5:52:14 PM,17.82,12.6,277.334,637.2,87,12.48,1.48,2.79,6.12,19.14,27.31,8.45,18.21,1.10,0.00,0.00,0.00,0.00
 AQU_19102_125 Pole,Average of 'AQU R3',8/17/2020 6:04:39 PM,17.45,12.5,279.337,644.2,89,12.55,1.49,2.82,6.07,18.77,26.98,8.48,18.77,1.12,0.00,0.00,0.00,0.00
 AQU_19102_125 Pole,Average of 'AQU R4',8/17/2020 6:17:21 PM,17.84,11.7,279.337,652.2,94,12.65,1.45,2.72,6.02,18.85,26.85,8.35,18.75,1.38,0.00,0.00,0.00,0.00
 AQU_19102_125 Pole,Average of 'AQU R5',8/17/2020 6:29:21 PM,17.23,11.7,274.331,637.2,94,12.67,1.47,2.84,6.24,19.24,27.12,8.31,17.84,1.24,0.04,0.00,0.00,0.00
 AQU_19102_125 Pole,Average of 'AQU R6',8/18/2020 10:55:10 AM,17.15,12.6,281.339,634.2,86,12.55,1.59,2.86,5.92,18.44,27.41,8.76,18.52,0.88,0.15,0.01,0.00,0.00,0.00
 AQU_19102_125 Pole,Average of 'AQU R7',8/18/2020 11:08:19 AM,17.46,11.3,278.338,657.2,94,12.91,1.50,2.86,6.15,18.57,26.26,8.29,18.94,1.49,0.04,0.00,0.00,0.00,0.00
 AQU_19102_125 Pole,Average of 'AQU R8',8/18/2020 11:20:49 AM,17.65,12.1,279.338,649.2,88,12.59,1.49,2.84,6.09,18.68,26.84,8.44,18.78,1.29,0.03,0.00,0.00,0.00,0.00

Export to file... Export to clipboard Cancel

3. Save the exported data file to the 'MS' folder created in the project folder using the MS_Track_ProjectNumber format.
 - a. Example. MS_GP_22057.csv

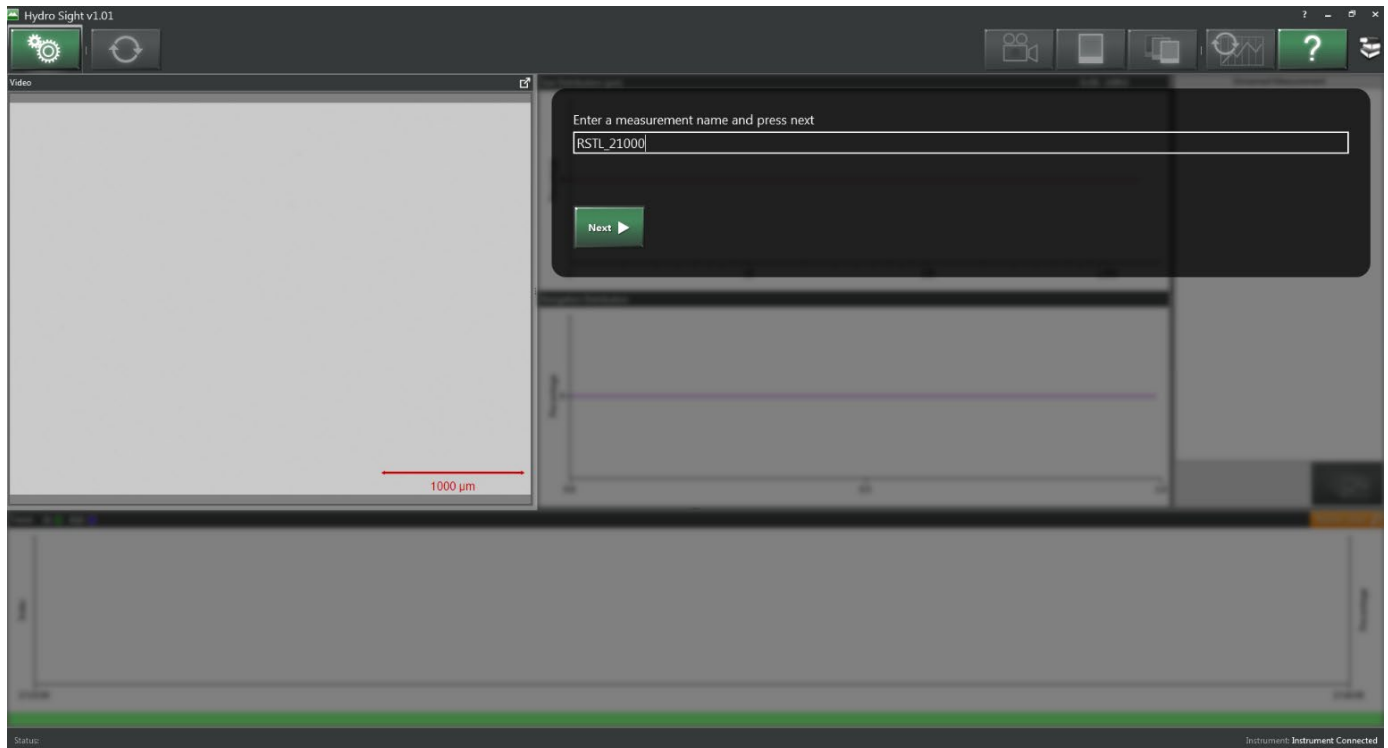


V. Hydrosight Operation

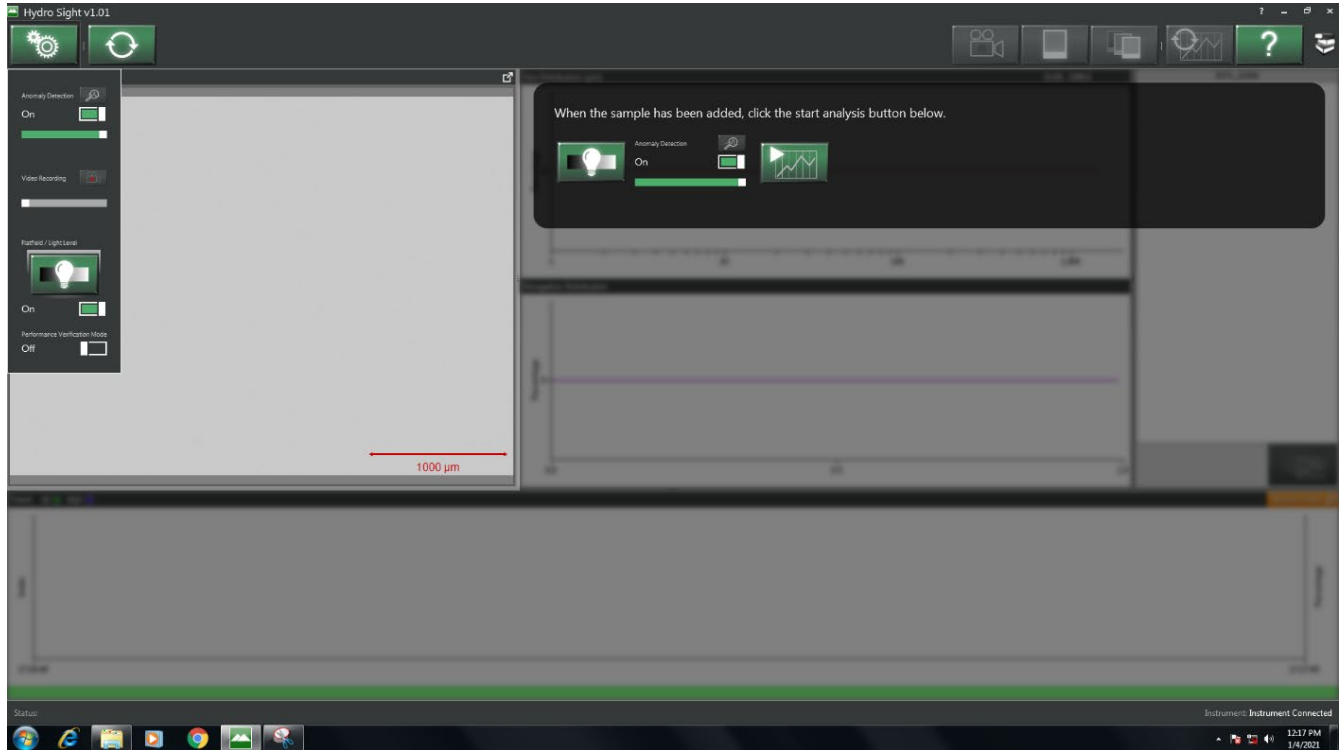
1. Prior to testing a sample in the MS3000, open the Hydrosight software found on the **Window's 7 computer** adjacent to the MS3000 computer.



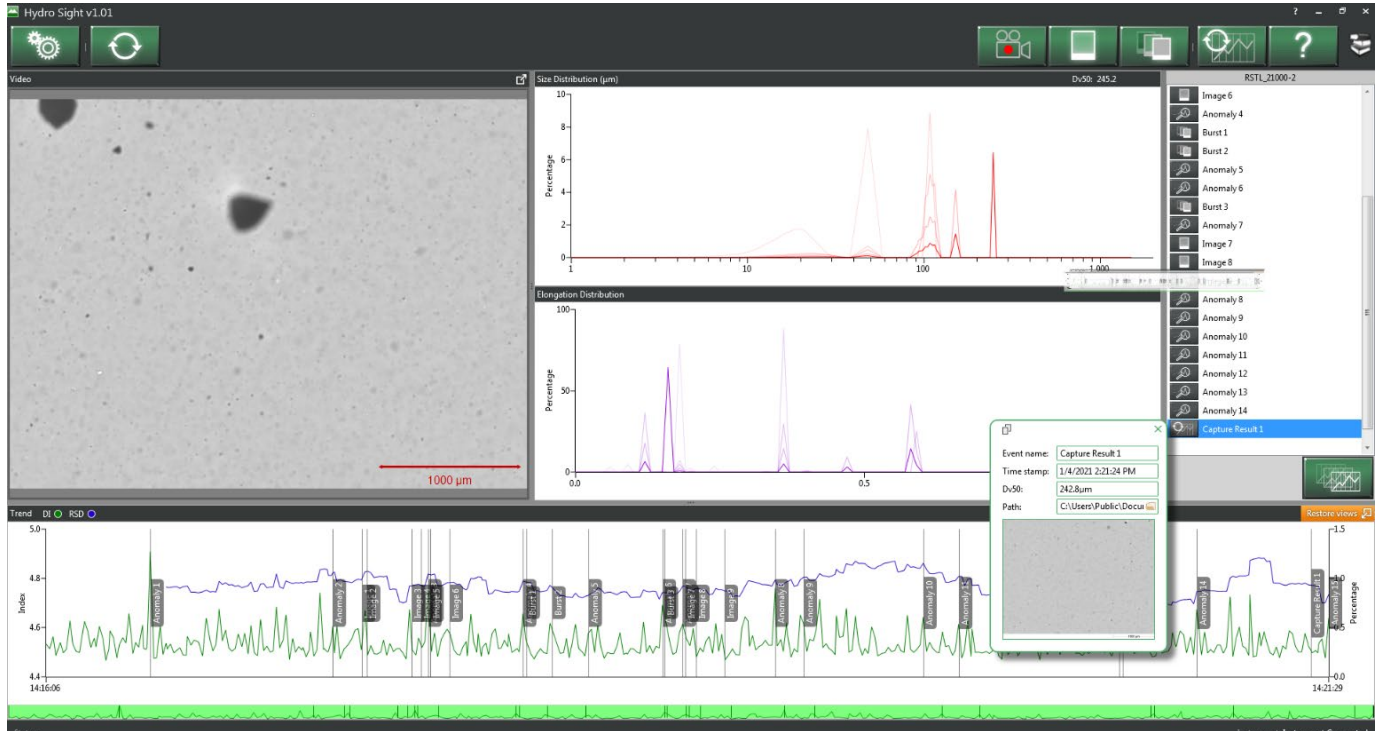
2. Allow time for the video feed of the Hydrosight to initialize. Afterwards, you will be promoted to input a sample name. Input the Project number or sample name into the input field.



3. Verify the **anomaly detection is on and on the high setting**. Click the **lightbulb icon** to perform a **background scan**. Afterwards wait to continue.



4. Once the sample has been added to the LV Hydro and has begun the measure process (post ultrasound and system degas), **click the icon with the play button and chart** to begin particle detection and image capturing.



5. After the MS3000 has completed the 5th measurement, ***immediately click on the chart icon with the refresh icon in the upper right corner to capture the data.*** Failure to do so will result in post measurement cleaning being included in the charts provided by the software.
6. As shown in the figure above, **click on the Captured result and relabel** as the project number or sample name. Then **click on the chart icon at the bottom right corner of the column and export the data.**
7. Afterwards, **click the refresh icon in the upper left corner of the screen** for the next sample.
 - *Note: previous data CANNOT be accessed again. Failure to export the data will result in data loss and will need to be retested.
8. On the desktop, access the libraries folder, select the Hydrosight folder and copy the contents of the tested project to a USB flash drive. Place the copied contents into Dropbox into the correct folder as required.
9. Close the software to de-initialize the camera after test completion.