

SAP #: 10013710 Measurements of Particle Size Distribution by Wet & Dry Laser Diffraction

For: USGS-NASA DFC BLDG 20 MS 964 Denver, CO 80225

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Dates of analysis: 5th July 2007

Summary

The techniques of wet and dry laser diffraction have been used to characterize the particle size distribution of Lunar Highlands Type simulant powder samples supplied by USGS-NASA. The methodologies, assumptions and derived particle size distribution are discussed within this report.

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1. Objectives

The objective of this study was to provide size distribution information on Lunar Highlands Type simulant samples from USGS-NASA. Shape information is included in another report which shows measurements of the two samples with a dry imaging technique.

2. Background

Two samples were submitted by USGS-NASA for measurement with laser diffraction techniques provided by Malvern Instruments Inc. The samples were:

- 1 Lunar Highlands Type simulant, medium grain size, NU-LHT-1M, 121
- 2 Lunar Highlands Type simulant, medium grain size, NU-LHT-1M, 026

3. Materials (and form) and instrumentation

The two samples were both grey powders, with larger black specs. The samples were analyzed on laser diffraction (Malvern Mastersizer MS2000, s/n 34355-181, 5.4 version software) with wet analysis using the Hydro S dispersion unit (capacity 100-150 ml). Dry analysis was also performed using the Scirroco dry dispersion unit.

Instruments in the laboratory are performance verified with NIST-traceable standards on a monthly basis. The MS2000 has a particle size distribution range of $0.02 - 2000 \mu m$. See:

http://www.malvern.com/common/downloads/MRK501-02_LR.pdf

4. Method

4.1 Dry Laser Diffraction

The methodology followed the broad recommendations outlined in ISO13320-1, the international standard for laser diffraction. In particular, Section 6.2.3 (Dispersion) was followed closely. In both wet and dry method development the objective is to understand the effect of energy on the particulate system (distinguishing agglomerates from dispersed particles) in line with the needs of the measurement outcome (bulk or primary particle size).

In a dry measurement, to assess both dispersion and the extent of attrition (if applicable), the material is subjected to what is called a "pressure-size titration" where the effect of increasing controlled energy input is examined (See Section 6.2.3.2 ISO 13320-1).

Controlling the ΔP across the venturi regulates this energy input. Shear processes by acceleration will (in theory) separate any agglomerates into primary particles. Increased energy can cause attrition of friable samples especially those that are organic or of high aspect ratio. For large samples then another consideration is that of carrying the particles through the measurement zone in the air stream and high flow rates are normally used for this.

Bulk samples of the material were scoop sampled and placed into the vibratory hopper of the Scirocco dry dispersion unit and consecutive repeat measurements undertaken in order to assess the reproducibility of measurement which is a function of the homogeneity of the material (or otherwise). The mass flow was adjusted until a stable and correct particle concentration was achieved at 4-bar and then left constant for the remainder of the experiments. Measurements were taken at 4-bar, 3-bar, 2-bar,1-bar and 0.5-bar disperser pressure. For setting of the correct and accurate working pressure, a wet or other reference measurement should be undertaken and the results of the dispersed material compared in both cases. The particle size distributions results will be the same if the material is in the same state of dispersion (likely to be full) and the correct optical properties used (especially in the wet measurement where the RRI is lower and can tend to unity depending on the sample).

4.2 Wet Laser Diffraction

In the case of wet dispersion, energy is imparted to the system using sonication. Similar to disperser pressure in a dry measurement, sonication can disperse any agglomerates to the primary particle size of the material. Aggregates are generally 'hard' and not dispersible under any standard measurement conditions. Excessive energy for fragile and/or friable samples may cause attrition, but this is unusual and is easily seen in the size-energy plot.

Water was used as the dispersant. Each sample was prewetted with a small amount of water and then added to the Hydro S dispersion unit. The samples were measured before, during and after a brief period of sonication.

More information on wet and dry dispersion is given in the attached application notes.

5. Assumptions

For a light scattering experiment the optical properties of the material are essential for accuracy (ISO13320-1 Appendix A) for material less than 40λ (~ 25μ m for a He-Ne laser at 0.6328 μ m) in size.

Laser diffraction instruments collect (stable) raw data in the form of an Intensity-Angle plot and it is this information (with the corresponding assumptions) that is converted to particle size distribution data. Thus the only objective of the measurement is to collect stable data suitable for deconvolution. Particle size is measured indirectly and the nature of the assumptions made is vital. Hence ancillary evidence is usually vital in confirming one result with respect to another.

The relative refractive index (RRI) is always higher in a dry measurement than in the corresponding wet one ($RI_{air} \sim 1$, $RI_{H20} \sim 1.33$, $RI_{particle} \sim 1.5$, for example) and thus the optical properties are somewhat more robust in a dry measurement especially if the material is reasonably large. For a wet measurement, especially for small material (d < 10µm or so) then the RI values need determining either by measurement or via a reference text (e.g. CRC Handbook).

In this case, an RI value was estimated from the provided composition of the samples. This estimated value was 1.75. An absorption index of 0.1 was used for all samples since it gave the best fit to the data. It also was reasonable given the colored nature of the samples.

It is important to explore the robustness of the derived measurement to small, systematic and sensible (the "3S's") changes to the RI parameters. Changes to these parameters can only be explored for a fully dispersed system, as it is quite obvious that agglomerate

optical properties (especially with respect to the imaginary component of the RI) will be different to those of a single primary particle.

The RI used for air was 1.000 and for water was 1.33 (CRC Handbook & Malvern Dispersant information within the software).

6. Results

6.1 Dry Laser Diffraction

The pressure-titration plot for Lunar Highlands Type Simulant sample 121 is shown below.

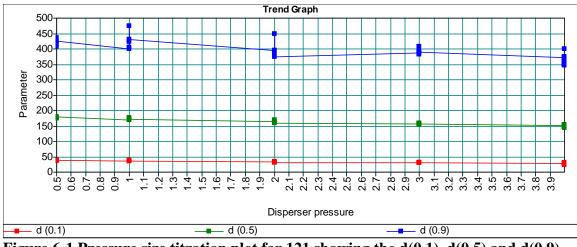


Figure 6-1 Pressure size titration plot for 121 showing the d(0.1), d(0.5) and d(0.9).

There is some change in the particle size distribution with varying disperser pressure. The variability in each set of measurements is a result of the variability within the sample itself. Given the broad range of particle sizes in this samples, some variation, such as that shown above, is expected.

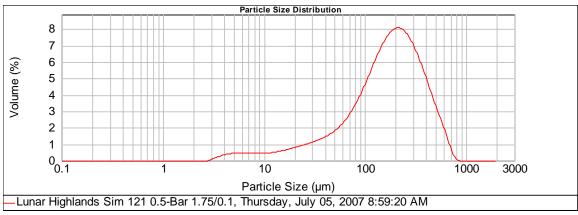
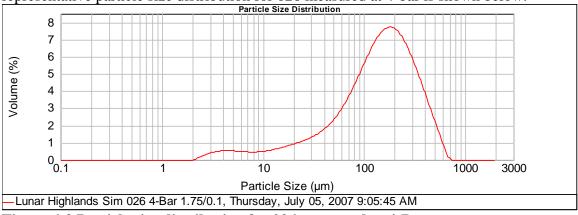


Figure 6-2 shows a particle size distribution for 121 measured at 4-Bar.

Figure 6-2 Size distribution plot for 121 measured at 4-Bar.

The 026 sample showed similar behavior when subjected to a pressure-size titration. A



representative particle size distribution for 026 measured at 4-bar is shown below.

Figure 6-3 Particle size distribution for 026 measured at 4-Bar.

Reproducibility of measurements can be assessed by exporting data to Excel. Tables 6-1 and 6-2 show reproducibility for measurements of both samples at 4-Bar disperser pressure.

Record number	Sample Name	d (0.1)	d (0.5)	d (0.9)
63	Lunar Highlands Sim 121 4-Bar 1.75/0.1	29.147	150.811	360.15
64	Lunar Highlands Sim 121 4-Bar 1.75/0.1	26.423	147.801	348.761
65	Lunar Highlands Sim 121 4-Bar 1.75/0.1	28.288	148.629	351.391
66	Lunar Highlands Sim 121 4-Bar 1.75/0.1	27.579	146.383	349.211
67	Lunar Highlands Sim 121 4-Bar 1.75/0.1	27.394	146.231	355.267
	Mean	27.77	147.97	352.96
	SD	1.02	1.88	4.77
	RSD	3.67	1.27	1.35

Table 6-1 Reproducibility for 121 at 4-Bar.

Record number	Sample Name	d (0.1)	d (0.5)	d (0.9)
101	Lunar Highlands Sim 026 4-Bar 1.75/0.1	29.153	149.46	353.415
102	Lunar Highlands Sim 026 4-Bar 1.75/0.1	28.507	146.63	357.525
103	Lunar Highlands Sim 026 4-Bar 1.75/0.1	28.502	148.277	375.18
104	Lunar Highlands Sim 026 4-Bar 1.75/0.1	28.566	148.174	370.407
	Mean	28.68	148.14	364.13
	SD	0.32	1.16	10.33
	RSD	1.10	0.78	2.84

Table 6-2 Reproducibility for 026 at 4-Bar.

All reproducibility of results is acceptable to ISO 13320-1 and is a reflection of the homogeneity of the sample, rather than of instrument variability.

6.2 Wet Laser Diffraction

The D10, D50 and D90 before, during and after sonication for 121 appear in Figure 6-4.

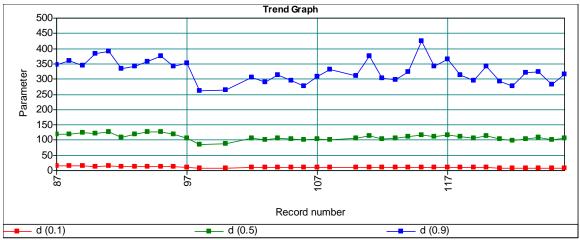


Figure 6-4 Results before (records 87-96), during (97-102) and after (98-126) sonication for 121.

Fluctuation in the d(0.9) occur both before and after sonication. These fluctuations are likely a result number fluctuations in the system. Sample 026 shows a similar response to sonication.

The particle size distributions for both samples before and after sonication are shown in Figure 6-5 and 6-6.

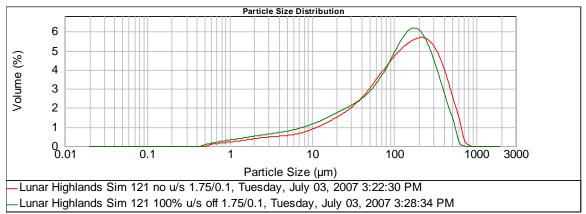


Figure 6-5 Particle size distribution plots for 121 before (red) and after (green) sonication.

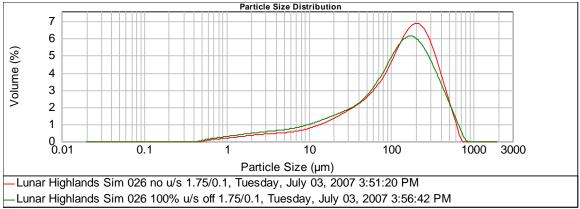


Figure 6-7 Particle size distribution for 026 before (red) and after (green) sonication.

Repeat measurements can be exported to Excel for easy statistical calculations. Tables 6-3 and 6-4 show the repeatability for each sample after sonication.

Record number	Sample Name	d (0.1)	d (0.5)	d (0.9)
87	Lunar Highlands Sim 121 no u/s 1.75/0.1	14.491	119.782	346.061
88	Lunar Highlands Sim 121 no u/s 1.75/0.1	14.336	120.048	359.425
89	Lunar Highlands Sim 121 no u/s 1.75/0.1	14.771	124.625	344.839
90	Lunar Highlands Sim 121 no u/s 1.75/0.1	13.9	122.77	383.655
	Mean	14.37	121.81	358.50
	SD	0.36	2.31	18.03
	RSD	2.53	1.90	5.03

 Table 6-2 Repeatability of measurements for 121 before sonication.

Record number	Sample Name	d (0.1)	d (0.5)	d (0.9)
130	Lunar Highlands Sim 026 no u/s 1.75/0.1	16.631	139.607	396.655
131	Lunar Highlands Sim 026 no u/s 1.75/0.1	15.297	137.633	385.234
132	Lunar Highlands Sim 026 no u/s 1.75/0.1	16.238	138.715	366.565
133	Lunar Highlands Sim 026 no u/s 1.75/0.1	16.429	136.383	387.025
	Mean	16.15	138.08	383.87
	SD	0.59	1.39	12.58
	RSD	3.65	1.01	3.28

 Table 6-3 Repeatability of measurements for 026 before sonication.

It should be noted that, in a wet measurement, the same particles are being analyzed over multiple measurements because they repeatedly circulate past the detector. This means that the statistics over repeat measurements indicate the repeatability of the measurement and how the same aliquot of material behaves over time. In a dry measurement, a different set of particles is analyzed each time, meaning that statistics over measurements are an indicator of reproducibility instead of repeatability and give an indication of the homogeneity (or otherwise) of the material.

7. Discussion

An overplot of sample 121 measured dry at 4-bar and wet before sonication is shown in Figures 7-1.

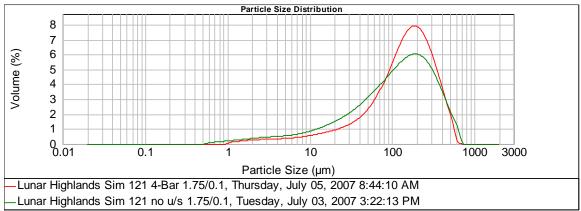


Figure 7-1 Overplot of measurements for 121 measured dry (red) at 4-bar and wet (green) before sonication.

The overall shape and range of the particle size results are similar when the wet and dry are compared. Wet measurement gives a somewhat broader distribution than dry measurement. Given the broad size range of this sample and possible reaction of the sample with water, the difference in results for the two measurement conditions is within expectations. 026 shows a similar wet/dry comparison.

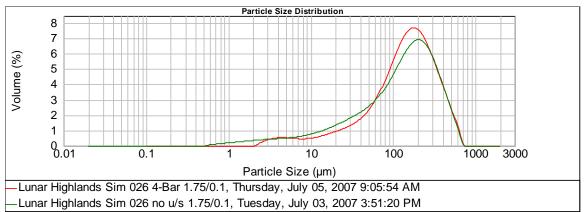


Figure 7-2 Overplot of measurements for 026 measured dry (red) at 4-bar and wet (green) before sonication.

Figure 7-3 shows an overlay of 121 and 026 measured dry at 4-bar disperser pressure.

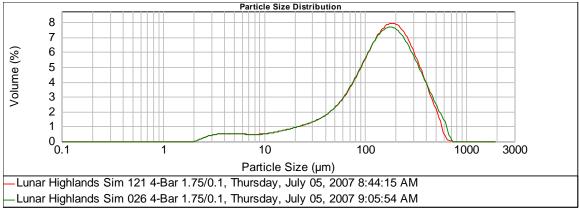


Figure 7-3 Particle size distributions for 121 (red) and 026 (green) measured dry at 4-bar.

The size distributions for the two samples are quite close as can be seen above and in the table below.

Sample Name	d(0.1)	d(0.5)	d(0.9)
Lunar Highlands Sim 121 Avg	27.7662	147.971	352.956
Lunar Highlands Sim 026 Avg	28.682	148.1353	364.1318

Table 7-1 Average d(0.1), d(0.5) and d(0.9) values for 121 and 026 measured dry at 4-bar.

8. Conclusions

The particle size distributions of the two samples are quickly and easily measured dry using the Mastersizer 2000.Wet measurements were also obtained successfully. Sampling of the material is important due to the broad range of particle sizes present in these samples.

- Attached: Dry method development application note Wet method development application note Basic Principles of Particle Size Analysis
- References: ISO 13320-1:1999 Particle Size Analysis Laser Diffraction. Available from http://www.iso.ch with a credit card payment.
 www.luxpop.com
 Cho J.; Park J. J. of Vacuum Science & Technology A. 2007, 18, 2, 329-333.
 MgO Refractive Index. Harrick Optical Materials. www.harricksci.com.

Mae Gackstetter Malvern Instruments Inc 5th July 2007





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	0.013	0.00	0.138	0.00	1.445	0.00	15.136	0.61 0.70	158.489 6.81 7 11	1659.58	7 0.00	
	0.015	0.00	0.158 0.182	0.00	1.660 1.905	0.00	17.378 19.953	0.79	181.970 7.14	1905.46 2187.76	0.00	
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	0.052 0.060 0.069 0.079 0.091 0.105	0.00 0.00 0.00 0.00 0.00	0.550 0.631 0.724 0.832 0.955 1.096	0.00 0.00 0.00 0.00 0.00	5.754 6.607 7.586 8.710 10.000 11.482	0.44 0.42 0.42 0.43 0.47	60.256 69.183 79.433 91.201 104.713 120.226	2.97 3.57 4.24 4.95 5.65	530.937 0.18 724.436 0.00 831.764 0.00 954.993 0.00 1096.478 0.00 1258.925 0.00	7585.770 8709.630 10000.000	6 0.00 6 0.00	





Sample Name: .unar Highlands Sim 121	no u/s 1.75/0.1	SOP Name:		Measured : Tuesday, J	uly 03, 2007 3:22:13	3 PM			
Sample Source & type:		Measured by: cmujat			Analysed: Thursday, July 05, 2007 11:51:59 AM				
Sample bulk lot ref:		Result Source: Edited							
Particle Name: .75/0.1 Particle RI: .750 Dispersant Name: Water		Accessory Name: Hydro 2000S (A) Absorption: 0.1 Dispersant RI: 1.330		Analysis n General pu Size range 0.020 Weighted 0.573	rpose :: to 2000.000 ur	Sensitivity: Normal Obscuration: im 5.28 % Result Emulation Off			
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d(0.1): 14.336	um	d(0.5): 120.048	um	d	(0.9): 359.425	um		
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Size (µm) Volume 0.010 0.011 0.013 0.015 0.017 0.020 0.020 0.023 0.030 0.035 0.040 0.040 0.046 0.045	e In % Size (µm) V 0.00 0.105 0.105 0.00 0.138 0.138 0.00 0.138 0.158 0.00 0.162 0.162 0.00 0.182 0.00 0.00 0.240 0.209 0.00 0.275 0.00 0.00 0.316 0.363 0.00 0.363 0.00 0.00 0.417 0.550 0.00 0.550 0.631 0.00 0.631 0.724	Size (µm) Vc 0.00 1.096 0.00 1.259 0.00 1.445 0.00 1.406 0.00 1.405 0.00 1.905 0.00 2.188 0.00 2.512 0.00 3.802 0.00 3.802 0.00 5.012 0.00 5.754 0.00 6.607 0.10 7.586	Size (µm 0.22 11.462 0.25 13.183 0.26 15.136 0.28 17.376 0.32 19.953 0.35 22.900 0.38 26.303 0.40 30.200 0.42 30.200 0.44 38.811 0.51 60.255 0.55 69.183 0.55 69.183 0.59 79.433	0.91 1.02 1.12 1.14 1.28 1.42 1.59 2.77 2.19 2.44 2.72 3.01 5.6 3.33 3.67	te (µm) Volume In % 20.226 5.00 38.038 5.225 58.489 5.441 81.970 5.441 81.970 5.451 908.930 5.334 908.930 5.334 908.930 65.344 908.930 65.344 908.930 90.534 90.977 775.423 4.664 90.967 90.967 90.967 90.967 90.957 90.400 93.764 90.900 90.9	Size (µm) Volume In % 1258.925 0.00 1445.440 0.00 1659.587 0.00 1905.461 0.00 2187.762 0.00 2884.032 0.00 3311.311 0.00 3801.894 0.00 505.158 0.00 501.872 0.00 6606.934 0.00 7585.776 0.00 7509.36 0.02			





::	Measured b cmujat Result Sour Edited Accessory b Hydro 20005 Absorption: 0.1 Dispersant 1.330 Span : 2.526 Surface We 25.768	rce: Name: S (A) : RI:			Analysis General Size ran 0.020 Weighte 0.343 Uniform 0.779	y, July 05, 2007 11 s model: purpose ge: to 2000.000 ed Residual: % ity:	um	A Sensitivity: Normal Obscuration 5.52 % Result Emu Off Result units Volume	n: lation:
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