

NEW MEASUREMENTS OF THE PARTICLE SIZE DISTRIBUTION OF APOLLO 11 LUNAR SOIL 10084. D. S. McKay<sup>1</sup> B. L. Cooper<sup>2</sup>, and L. M. Riofrio<sup>2</sup> <sup>1</sup>NASA Johnson Space Center ([david.s.mckay@nasa.gov](mailto:david.s.mckay@nasa.gov)), <sup>2</sup>Oceanering Space Systems.

**Introduction:** We have initiated a major new program to determine the grain size distribution of nearly all lunar soils collected in the Apollo program. Following the return of Apollo soil and core samples, a number of investigators including our own group performed grain size distribution studies and published the results [1-11]. Nearly all of these studies were done by sieving the samples, usually with a working fluid such as Freon™ or water. We have measured the particle size distribution of lunar soil 10084,2005 in water, using a Microtrac™ laser diffraction instrument. Details of our own sieving technique and protocol (also used in [11]), are given in [4].

While sieving usually produces accurate and reproducible results, it has disadvantages. It is very labor intensive and requires hours to days to perform properly. Even using automated sieve shaking devices, four or five days may be needed to sieve each sample, although multiple sieve stacks increases productivity. Second, sieving is subject to loss of grains through handling and weighing operations, and these losses are concentrated in the finest grain sizes. Loss from handling becomes a more acute problem when smaller amounts of material are used. While we were able to quantitatively sieve into 6 or 8 size fractions using starting soil masses as low as 50mg, attrition and handling problems limit the practicality of sieving smaller amounts. Third, sieving below 10 or 20µm is not practical because of the problems of grain loss, and smaller grains sticking to coarser grains. Sieving is completely impractical below about 5-10µm. Consequently, sieving gives no information on the size distribution below ~10 µm, which includes the important submicrometer and nanoparticle size ranges. Finally, sieving creates a limited number of size bins and may therefore miss fine structure of the distribution which would be revealed by other methods that produce many smaller size bins.

Because of the known complexity of lunar surface soil-forming processes, particles in the finer size range may be created and modified by processes totally different from the processes that create coarser particles, and such differences may leave a record in the grain size distribution. The only data in this finer size range is from Coulter counter or optical/electron microscope techniques [4, 12]. Knowledge of the grain size distribution of material finer than about 10µm is necessary to help evaluate possible exposure health hazards to humans at a lunar location. Nanometer size particles may cause health issues primarily because of their small size, regardless of their composition. These

effects may be particularly pronounced for fresh lunar soil because the grain surfaces may have been activated by UV, solar flare radiation, shock effects, solar and galactic particle etching and structural effects, and reactive vapor coatings from sputtering and impacts.

**Alternative methods:** A number of alternative methods for determining grain size distribution have existed for many years (NBS Special Paper 260-85, 1983). Major techniques include those based on optical and electron microscope imaging, volume displacement and electrical sensing flow through detectors, light scattering including laser light, laser Doppler techniques based on Brownian motion detection, sieving, impactor, and sedimentation methods. Our new data is based upon laser light scattering.

**Instrument:** For grain sizes down to about 1 micrometer, we use an advanced laser light scattering instrument (Microtrac™). This instrument uses a proprietary modified Mie scattering algorithm to account for the irregular shapes of the particles and their non-transparent nature. This instrument was chosen because of the demonstrated reproducibility and accuracy of the method, the relative ease of use, and the ability to analyze a large number of samples in a relatively short time. It is now feasible to analyze multiple splits from the same sample to allow for an evaluation of the homogeneity of the initial material and the variability caused by any size fractionation during sampling and splitting. Such analysis is not practical when using sieving because of the time and labor required for each analysis.

**Method:** The method consists of adding a small amount of soil or dust to a working fluid, usually either water or isopropyl alcohol, and introducing this fluid into a circulating system containing the analysis cell illuminated by lasers and surrounded by detectors at known geometries. Care is taken to disperse the sample and eliminate clumping. The data are accumulated in a few minutes and are then analyzed, reduced, and displayed in a variety of formats. Between runs, the system can be flushed and evaluated for a particle-free initial condition.

For sizes below ~1 µm, and well down into the nanometer scale we are using another laser-based instrument (Nanotracer™) that detects Brownian motion of the finest grains. These results are not reported here.

Percent Bin	NIST Reported	Microtrac	$\Delta$
10	0.33 $\mu\text{m}$	0.368 $\mu\text{m}$	$\sim+10\%$
25	0.57 $\mu\text{m}$	0.530 $\mu\text{m}$	$\sim-08\%$
50	0.98 $\mu\text{m}$	0.959 $\mu\text{m}$	$\sim-02\%$
75	1.52 $\mu\text{m}$	1.683 $\mu\text{m}$	$\sim+10\%$
90	2.19 $\mu\text{m}$	2.808 $\mu\text{m}$	$\sim+22\%$

**Table 1. Comparison of our result against NIST Standard Reference Material SRM1978.**

Percent Bin	Certified Glass Standard	Microtrac	$\Delta$
10	47 - 54 $\mu\text{m}$	52.86 $\mu\text{m}$	Within range
50	55.5 - 59.5 $\mu\text{m}$	56.26 $\mu\text{m}$	Within range
90	57 - 70 $\mu\text{m}$	57.06 $\mu\text{m}$	Within range
Width	Less than 25	7.86	Within range

**Table 2. Comparison of our result against a certified (traceable to NIST) glass standard [13].**

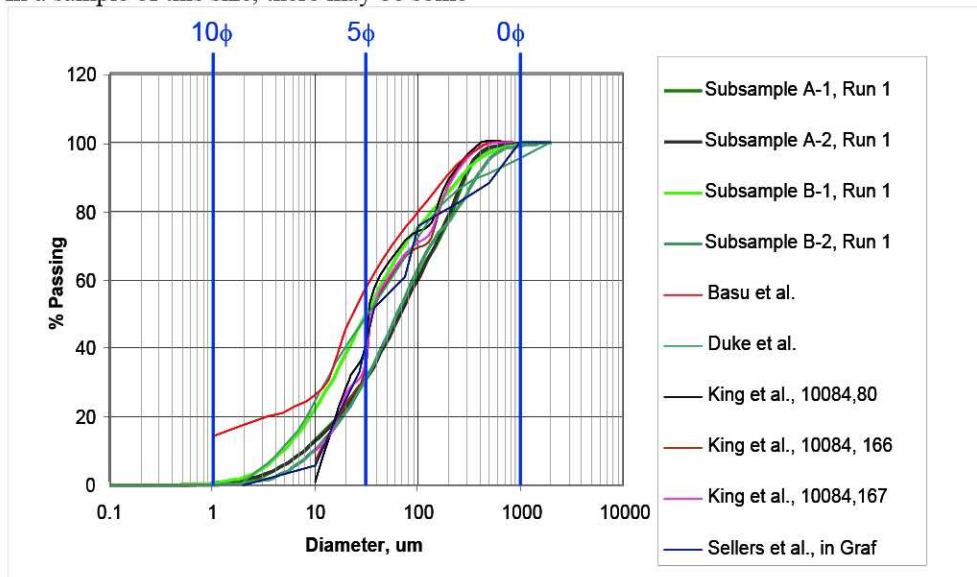
**Discussion:** This measurement was done using distilled water as the working (carrier) fluid, which allows us to directly compare our results with those of [1, 2, 11], who also used distilled water for their wet sieving processes on subsamples. The overall shape of the distribution is in agreement with that of Basu et al. for particles smaller than  $3.47 \phi$  ( $90\mu\text{m}$ ). For the coarser sizes, our data suggest that there are more particles in the  $2 \phi$  to  $2.74 \phi$  ( $150\mu\text{m}$  to  $250\mu\text{m}$ ) than revealed in the Basu et al. analysis.

The difference between our result and those of Basu may be due to variation between subsamples. The subsample that we used was a few tens of milligrams. In a sample of this size, there may be some

variation in the largest size fraction because there may not be enough particles to achieve a statistically valid measurement.

**Conclusion:** Direct comparison of sieve data to light-scattering data is difficult and the results must be carefully checked with standard known particle sizes and distribution. Our analyses of NIST or NIST-traceable grain size material shows that the Microtrac results are within acceptable ranges for particle sizes between  $0.33\mu\text{m}$  and  $\sim 60\mu\text{m}$ . The correspondence of the sieve data from 10084 to the light-scattering data is good and provides confidence that the two methods produce comparable results. These results open the door to future automated, rapid, and reproducible grain size analysis of planetary soils and dust.

References: [1] Duke, M.B., et al. (1970) Proc. Apollo 11 Lunar Sci. Conf., Geochim. et Cosmochim. Acta Suppl., 1: p. 347; [2] King Jr, E.A., et al. (1971) Proc. 2<sup>nd</sup> Lunar Sci. Conf., 2: p. 737-746; [3] Carrier, W.D. (1973) The Moon, 6(3-4): p. 250-263; [4] McKay, D.S., et al. (1974) Proc. 5<sup>th</sup> Lunar Sci. Conf., Suppl. 5, Geochimica et Cosmochimica Acta, 5: p. 887-906; [5] McKay, D.S., et al. (1976) Proc. 7<sup>th</sup> Lunar & Planet. Sci. Conf., 7: p. 295-313; [6] McKay, D.S., et al. (1977) Proc. 8<sup>th</sup> Lunar Sci. Conf.: p. 2929-2952; [7] McKay, D.S., et al., *Grain size and the evolution of Luna 24 soils*, in *Mare Crisium: The View from Luna 24. Suppl. 9, Geochimica et Cosmochimica Acta*. 1978, Pergamon Press. p. 125-136; [8] McKay, D.S., et al. (1978) Proc. 9<sup>th</sup> Lunar & Planet. Sci. Conf.: p. 1913-1932; [9] McKay, D.S., et al. (1980) Proc. 11<sup>th</sup> Lunar & Planet. Sci. Conf.: p. 1531-1550; [10] McKay, D.S., et al., *The Lunar Regolith*, in *Lunar Sourcebook*, G. Heiken, D. Vaniman, and B.M. French, Editors. 1991, Cambridge University Press: Cambridge. p. 285-356; [11] Basu, A., et al. (2001) Meteoritics and Planetary Science, 36: p. 177; [12] Greene, G.M., et al. (1975) Proc. 6<sup>th</sup> Lunar Sci. Conf., 1: p. 517-527; [13] Plantz, P.E., *Traceability and Standards Applied to Microtrac Instruments*. 2007, Microtrac Inc. (download at microtrac.com).



**Figure 1. Comparison of particle size distribution results from previous workers and our current research.** Our data were measured using water, which was also used for the Basu data. This allows straightforward comparison; however, because of the effect of water on lunar soil, we will re-measure this sample in isopropyl alcohol which we have found to minimize clumping artifacts.

