



A Honeywell Company

Particle Size Distribution of Powders by Laser Light Scattering

UOP Method 856-07

Scope

This method is for determining the particle size distribution of powders and slurries using laser light scattering. Most applications are for particle sizes in the 1 to 100 μm range, but the instrument is capable of measuring particles from 0.02 to 2800 μm . Samples are analyzed as received or prepared using sonication to disperse agglomerated particles.

Reference

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

Forward light scattering is used as the basis for measurement of particle size distribution. The instrument incorporates three lasers to measure the particles over the entire size range. A beam of laser light is projected through a circulating stream of particles and the intensity of the resultant diffraction patterns is measured by a photodetector. The intensity of the detected light at various angles is proportional to the number and size of the particles. The diffraction patterns are processed using optical models selected by the operator. The particle size distribution is presented in histogram format. The data are also summarized to include mean and median diameter and other particle characteristics.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Beaker, polypropylene, 30-ml, VWR, Cat. No. 13915-067

Bottle, wash, polyethylene, 1000-mL, VWR, Cat. No. 16651-664

Ultrasonic Probe, Branson, Model S-250D, VWR, Cat. No. 33995-594

Particle size analyzer, Microtrac, Model S3500

Spatula, double blade, flat end, VWR, Cat. No. 57952-005

Stir plate, VWR, Cat. No. 58940-158

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Nonconfidential UOP Methods are available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, USA. The UOP Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

Stir bar, VWR, Cat. No. 58948-988

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Surfactants and Dispersing Aids, Assorted Cationic, Anionic, Non-Ionic: Isopropanol, Photoflo®, TSPP (Tetra Sodium Pyro Phosphate), and TritonX-100®, VWR, Cat. Nos. (consecutively, BDH1133-1LP, GRKK146-4510, JT3850-1, and 82022-980). Typical concentrations are less than 0.1 weight percent.

Water, deionized, or as recommended by the manufacturer

Water, for sampling system, flow rate, 35 L (5 gal) per minute; pressure 30-100 psig (210-690 kPa)

- The Microtrac analyzer has its own filter and pressure regulator. If the water supply contains excessive particulate matter, installation of a pre-filter is advisable. Water is the typical dispersing medium for the majority of sample types. Water with surfactants may also be used if the suspension is unstable. With the use of additional accessories from the manufacturer, non-aqueous liquids or air may also be used as the dispersing medium.

Procedure

The analyst is expected to be familiar with general laboratory practices and the equipment being used. This Procedure section gives specific instructions for the Microtrac analyzer listed in *Apparatus*. If a different analyzer is used, use these instructions as a guide, along with the instructions from the instrument manufacturer, to perform the analysis.

1. Install the analyzer according to the manufacturer's instructions. Complete the initial operating check and startup procedure as described in the Instrument Operating Manual to verify instrument operation. Once this is completed, it need not be done again unless service is required. The Microtrac S3500 software is entirely automated.
2. Standard analysis methods can be developed for each material type that will reduce the set up and analysis time. Enter the parameters listed in Table 1 for the typical "UOP Standard Analysis." Enter other parameters in different analytical method files as needed for the sample types to be analyzed. See the Instrument Operating Manual for instructions on how to set up and store multiple analysis methods.
 - Automated background correction can also be initiated using the Set Zero function prior to each sample that is analyzed.

Sample Preparation

Obtain a representative sample of the powder or slurry to be tested. Samples may be analyzed without pretreatment if the assumption can be made that the sample contains no particles larger than the upper limit of the selected range.

1. Fill a 30-mL beaker containing a stir bar with deionized water to the 27 ml mark. Using a spatula, place approximately one gram of sample into the beaker. If sonication is not required, proceed to Sample Analysis.
2. Transfer the beaker to the stir plate with the ultrasonic probe and turn on the stir plate. Align the probe tip with the side of the beaker at the 10 ml mark. Sonicate using the standard conditions of 90 watts for four minutes, setting the timer on the ultrasonic probe.

Sample Analysis

1. Select the instrument to be used from the tool bar. Select Measure → Select Instrument → S3500/3000.
2. Recall the appropriate analysis method from the drop down list by selecting Set Up and recall the method from saved methods (see Procedure Number 2).
3. Run Background Correction (Set Zero) and initiate the sample analysis by selecting “Auto” from the tool bar.
4. Add the prepared sample to the analysis chamber when prompted by the software. Follow the on screen instructions to determine the amount of sample required. The instrument will automatically start the analysis when the correct sample concentration is measured.
5. The sample chamber will automatically rinse, disposing of the sample down the drain, following the completion of the analysis sequence. Since the sample prep is dilute with low solids and neutral pH, it is acceptable to discard the small amount used for analysis down the drain. The remainder of the sample prep is allowed to settle, is decanted, and the solids are dried and disposed of in Industrial Waste.
 - Check local regulations to confirm proper disposal procedures.

Table 1

Instrument Parameters: UOP Standard Analysis			
Timing		Particle Information	
Set Zero Time (s):	30	Transparency:	Absorbing
Run Time (s):	30	Refractive Index:	N/A
Number of Runs:	3	Shape:	Irregular
Perspective		Fluid Information	
Progression:	Standard	Water	
Distribution:	Volume	Refractive Index:	1.333
Minimum:	0.0215		
Maximum:	1408		
Residual:	Disabled		
Percentile:	10, 25, 50, 75, 90		
SDC/ASVR (Options)		SDC/ASVR (Auto Sequence)	
SDC/ASVR Information:	Default	Fill/Rinse:	Disable
New Parameters:	Default	Perform Set Zero:	Enabled
Control		Continue Valid:	Enabled
Number of Rinses:	4	Auto Sequence:	1
De-aeration Cycles:	4	Sample Loading	
Flow Rate (%):	35	UltraSound:	None
Ultrasound (%):	30	Pre-Run Circ (s):	60
Ultrasound Time (s):	120		

Calculations

All calculations are carried out by the software and printed automatically using Adobe. Typical data reports are shown in Figures 1 and 2. ASCII and other file types may also be generated for electronic data transfer. Results are reported to three significant figures.

UOP - Riverside Analytical

13XI M-34-196 QA Reference

- LIMS Task ID =

05/09/2007 06:34

S3000/S3500

S3792

- LIMS ID =220188702

- Sample Prep = 4Min@45%200W



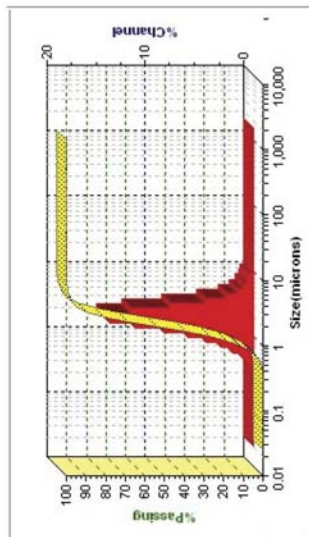
Data Item	Value
MV(um):	2.058
MN(um):	0.964
MA(um):	1.597
CS:	3.76
SD:	0.895

Size(um)	%Tilt
10.00	0.926
25.00	1.326
50.00	1.861
75.00	2.507
90.00	3.32

%Tilt	Size(um)
10.00	0.926
25.00	1.326
50.00	1.861
75.00	2.507
90.00	3.32

Dia	Vol%	Width
1.861	100.0	1.79

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.89	4.78	94.30
1184	0.00	100.00	3.27	8.31	89.52
995.6	0.00	100.00	2.750	12.42	81.21
837.2	0.00	100.00	2.312	14.96	68.79
704.0	0.00	100.00	1.945	14.62	53.83
592.0	0.00	100.00	1.635	12.05	39.21
497.8	0.00	100.00	1.375	9.06	27.16
418.6	0.00	100.00	1.156	6.59	18.10
352.0	0.00	100.00	0.972	4.73	11.51
296.0	0.00	100.00	0.818	3.28	6.78
248.9	0.00	100.00	0.688	2.06	3.50
209.3	0.00	100.00	0.578	1.05	1.44
176.0	0.00	100.00	0.486	0.39	0.39
148.0	0.00	100.00	0.409	0.00	0.00
124.5	0.00	100.00	0.344	0.00	0.00
104.7	0.00	100.00	0.2890	0.00	0.00
88.00	0.00	100.00	0.2430	0.00	0.00
74.00	0.00	100.00	0.2040	0.00	0.00
62.23	0.00	100.00	0.1720	0.00	0.00
52.33	0.00	100.00	0.1450	0.00	0.00
44.00	0.00	100.00	0.1220	0.00	0.00
37.00	0.00	100.00	0.1020	0.00	0.00
31.11	0.00	100.00	0.0860	0.00	0.00
26.16	0.00	100.00	0.0720	0.00	0.00
22.00	0.00	100.00	0.0610	0.00	0.00
18.50	0.00	100.00	0.0510	0.00	0.00
15.56	0.00	100.00	0.0430	0.00	0.00
13.08	0.00	100.00	0.0360	0.00	0.00
11.00	0.00	100.00	0.0300	0.00	0.00
9.25	0.34	99.66	0.02550	0.00	0.00
7.78	0.52	99.66			
6.54	0.83	99.14			
5.50	1.42	98.31			
4.62	2.59	96.89			



Distribution:	Volume Standard	Run Time:	30 Sec	Fluid:	WATER	Fluid Ref. Index:	Loading Factor:
Upper Edge:	1408	Particle:	0.00	Above Residual:	0	Transmission:	0.020
Lower Edge:	0.0215	Transparency:	0.00	Below Residual:	0	RMS Residual:	1.58E-02
Residuals:	Disabled	Absorbing:	N/A	Cell ID:	0636	Flow:	35 %
Num. Channels:	64	Part. Shape:	Irregular	Unsonic Power:	N/A	Unsonic Time:	N/A
Analysis Mode:	S31000	DB Record:	757	Recalc. Status:		Serial Num:	S3792
Filter:	Enabled	Database:	C:\Program Files\Microtrac\FLEX 10.3.1\Default\ASAP\ASAP2007.MDB				
Analysis Gain:	Default						

Warnings: NONE

Figure 1
Typical Report, Small Particles

UOP - Riverside Analytical



FCC FCat

- LIMS Task ID =

07/26/2007 15:15

S3000/S3500
S3792

- LIMS ID = 220194265

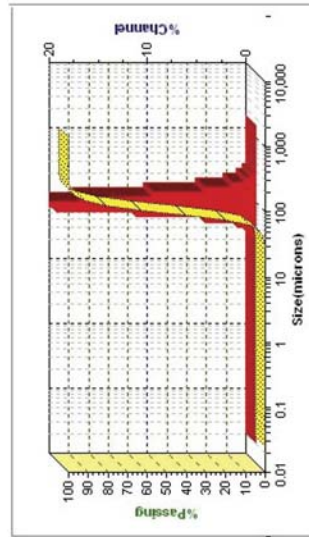
- Sample Prep = No Ultrasound

Data Item	Value
MV(um):	100.7
MN(um):	73.23
MA(um):	90.56
CS:	6.60E-02
SD:	29.85

Size(um)	%Tile
10.00	64.36
25.00	77.41
50.00	94.51
75.00	116.8
90.00	144.3

Dia	Vol%	Width
94.51	100.0	59.69

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.89	0.00	0.00
1184	0.00	100.00	3.27	0.00	0.00
995.6	0.00	100.00	2.750	0.00	0.00
837.2	0.00	100.00	2.312	0.00	0.00
704.0	0.00	100.00	1.945	0.00	0.00
592.0	0.00	100.00	1.635	0.00	0.00
497.8	0.00	100.00	1.375	0.00	0.00
418.6	0.00	100.00	1.156	0.00	0.00
352.0	0.00	100.00	0.972	0.00	0.00
296.0	0.41	100.00	0.818	0.00	0.00
248.9	0.96	99.59	0.688	0.00	0.00
209.3	2.28	98.63	0.578	0.00	0.00
176.0	5.16	96.35	0.486	0.00	0.00
148.0	10.46	91.19	0.409	0.00	0.00
124.5	17.62	80.73	0.344	0.00	0.00
104.7	22.52	63.11	0.2890	0.00	0.00
88.00	20.23	40.59	0.2430	0.00	0.00
74.00	12.14	20.36	0.2040	0.00	0.00
62.23	5.20	8.22	0.1720	0.00	0.00
52.33	1.90	3.02	0.1450	0.00	0.00
44.00	0.75	1.12	0.1220	0.00	0.00
37.00	0.37	0.37	0.1020	0.00	0.00
31.11	0.00	0.00	0.0860	0.00	0.00
26.16	0.00	0.00	0.0720	0.00	0.00
22.00	0.00	0.00	0.0610	0.00	0.00
18.50	0.00	0.00	0.0510	0.00	0.00
15.56	0.00	0.00	0.0430	0.00	0.00
13.08	0.00	0.00	0.0360	0.00	0.00
11.00	0.00	0.00	0.0300	0.00	0.00
9.25	0.00	0.00	0.02550	0.00	0.00
7.78	0.00	0.00			
6.54	0.00	0.00			
5.50	0.00	0.00			
4.62	0.00	0.00			



Distribution	Volume	Run Time	30 Sec	Fluid	WATER	Loading Factor	0.100
Preparation:	Standard	Run Num:	Avg of 3	Fluid Ref. Index	1.333	Transmission	0.97
Upper Edge:	1408	Particle	UOP	Above Residual	0	RMS Residual	7.02E-03
Lower Edge:	0.0215	Transparency	STANDARD	Below Residual	0	Floor	35 %
Residual:	Disabled	Part. Ref. Index	ANALYSIS			Ultrasonic Power	N/A
Num. Channels:	64	Part. Shape	Absorbing	Cell ID:	0036	Ultrasonic Time	N/A
Analysis Mode:	S1000	DB Record	Irregular	Recalc Status:		Serial Num:	S3792
Filter:	Enabled						
Analysis Date:	Default	Database	C:\Program Files\Microtrac\FLEX 10.3.1\DATABASES\REV0307.MDB				

Warnings: NONE

Figure 2
Typical Report, Large Particles

Precision

Precision statements were determined using UOP Method 999.

Repeatability and Intermediate Precision

A nested design was carried out for determining particle size of two different samples with two analysts in one laboratory. Each analyst carried out tests on two separate days, performing two tests each day per sample. The total number of tests for each of the two samples was eight. The precision data are summarized in Table 2. Two tests performed by the same analyst on the same day should not differ by more than the repeatability allowable difference with 95% confidence. Two tests performed in one laboratory by different analysts on different days should not differ by more than the intermediate precision allowable difference with 95% confidence.

The data in Table 2 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Table 2
Repeatability and Intermediate Precision, μm

<u>Sample</u>	<u>Mean Size</u>	<u>Repeatability</u>		<u>Intermediate Precision</u>	
		<u>Within-Day esd</u>	<u>Allowable Difference</u>	<u>Within-Lab esd</u>	<u>Allowable Difference</u>
Zeolite	1.86	0.005	0.02	0.008	0.03
Catalyst	94.07	0.58	2.29	0.46	1.66

Reproducibility

There is insufficient data to calculate reproducibility of the test at this time.

Time for Analysis

The elapsed time for one analysis is 0.25 hour. The labor requirement is 0.25 hour.

Suggested Suppliers

Microtrac Inc., 148 Keystone Drive, Montgomeryville, PA 18936, www.microtrac.com

VWR Scientific, 1310 Goshen Parkway, West Chester, PA 19380, www.vwr.com