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Standard Guide for Significance of Particle Size Measurements of Coating Powders[1]

This standard is issued under the fixed designation D 5861; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the significance of referencing the techniques used whenever specifying the particle size distribution of a coating powder.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1921 Test Methods for Particle Size (Sieve Analysis) of Plastic Materials[2]

D 3451 Guide for Testing Coating Powders and Powder Coatings[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *coating powders*—these are finely divided particles of organic polymer that generally contain pigments, fillers, and additives and that remain finely divided during storage under suitable conditions.

3.1.2 *powder coatings*—these are coatings that are protective, decorative, or both; and that are formed by the application of a coating powder to a substrate and fused into continuous films by the application of heat or radiant energy.

4. Significance and Use

4.1 This guide describes the need to specify the measuring technique used whenever quoting the particle size distribution of a coating powder.

4.2 This guide is for use by manufacturers of coating powders and by specifiers for process control and product acceptance.

5. Particle Size of Coating Powders

5.1 The size of the particles comprising a coating powder plays a critical role in the fluidization, application, and reclamation of the powder, and in the final appearance of the coated part. Coating powders are comprised of particles of widely differing sizes, from as low as about 1 µm to as high as about 150 µm. Collectively, the individual particles form a size distribution, defined by the percentages of particles present of a given size or within a given size range. There are generally few particles at the low and high ends of the distribution, the majority being in the 25 to 65-µm range. The distribution can be described by an actual plot of the particle size distribution, or by numerical attributes of the distribution, such as the calculated values of its mean, median or mode. The mean represents the average particle size (the sum of all the particle sizes divided by the number of particles). The median represents a size such that half the particles are larger than it and half the particles are smaller than it. The mode represents the most frequently occurring particle size. For all coating powders these three figures are numerically different.

5.2 The particle size distribution is generally chosen by the coating powder manufacturer from knowledge of the application technique, the required cured film thickness, surface appearance, and performance. Once the desired particle size distribution has been selected, it needs to be monitored to ensure consistency from batch to batch and, indeed, within each batch. Occasionally the coating powder applicator may specify the particle size from knowledge of the specific application equipment or customer requirements, or both.

5.3 It is important for all involved to understand that the numerical data comprising a particle size distribution are significantly dependent on the technique used to obtain them. It is, therefore, of little use to quote or specify a particle size distribution, and even less a single particle size, without also defining the technique used to obtain that measurement, or, if a single size, whether it is, for example, the mean, median or modal value.

6. Measurement of Particle Size

6.1 There are a wide variety of instruments currently available for measuring the particle size distributions of coating powders. Actual sieving, such as described in Test Methods D 1921, where the percentage weight of coating powder retained on sieves of known mesh size is measured, is relatively inexpensive and direct. It is, however, significantly slower than indirect measurement techniques, such as laser scattering and electrolytic conductivity, such as described in Guide D 3451. With indirect measurement techniques, a secondary effect, induced by the presence of the coating powder

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 06.02.

particles, is measured, such as changes in light scattering or in the conductivity of an electrolyte. These effects are analyzed using a specific theoretical algorithm, unique to the measurement technique, and the particle size distribution calculated that would cause the measured changes. Various other statistical data on the distributions, such as the mean, the median, and the mode are also often automatically calculated.

6.2 Secondary measurement techniques make assumptions such as the measured particles being spherical, and do not acknowledge the fractured, randomized shapes the particles actually possess. Others require the preparation of a suspension of the particles in a liquid, which could alter the physical state of particle agglomerates present in the dry state. Even the required processing for dry powder measurement techniques could mechanically break up larger particles or agglomerates into smaller ones, or both.

6.3 Thus not only can the theoretical algorithms for the measuring techniques be quite different, but each measurement technique can cause the particle size distribution to change during sample preparation or the measurement process itself, or both. This simply serves to emphasize that once a measurement technique has been selected, there is still need for consistency in all aspects of its operation.

7. Effect of Using Different Measurement Techniques

7.1 To illustrate the numerical differences in measured particle size that can be found when different measurement techniques are used, the same coating powder was provided to a number of participants, who measured the particle size of the sample, usually in triplicate, using their own preferred technique. Participants included coating powder manufacturers, raw material suppliers to the powder coating market, and manufacturers of particle size measuring equipment.

7.2 The data obtained can be found in Annex A1 and Annex A2. They have been transposed into two respective standard formats for ease of comparison. Where possible, additional numerical data were extracted from the original plots of particle size distribution. In these instances, such figures are enclosed in parentheses in Annex A1 (see Figs. A1.1-A1.14). Some of the original plots of particle size distribution were replotted for clarity, with a consistent ordinate and abscissa, of "percentage of particles in a given range" and "log (particle size in μm)" respectively. These standardized distributions constitute Figs. A1.1-A1.14.

7.3 It can be seen that there are distinct differences between the data acquired by different techniques, and by the same technique when the machine manufacturer or model is changed. There are even differences when instruments with the same model number are used in different laboratories.

7.4 It must be emphasized that these data are not presented in order to recommend one measurement technique over another, or one participating piece of equipment over another nonparticipating piece of equipment, but rather to clearly illustrate the *necessity of defining how a size measurement is obtained* when quoting any numerical value regarding particle size.

8. Measurement Techniques Used

8.1 Agitated Sieving, Dry Sampling[4]

8.2 Electrolyte Conductivity, Wet Sampling[5]

8.3 Laser Scattering, Dry Sampling[6]

8.4 Laser Scattering, Wet Sampling[7]

8.5 Sedimentation/X-Ray Absorption, Wet Sampling[8]

8.6 Mercury Porosimetry, Dry Sampling[9]

8.7 Note that some of the instruments were used independently of each other, and by more than one participant.

9. Keywords

9.1 coating powder; electroconductivity; laser scattering; mercury porosimetry; particle size analysis; powder coating; sedimentation; sieve analysis; X-ray

[4] Jet sieve available from Alpine American Corporation, 5, Michigan Drive, Natick, MA 01760, and Sonic Sifter Separator Model L3P available from ATM® Corporation, 645 S94th Place, Milwaukee, WI 53214 have been found suitable for this purpose.

[5] Model TA II available from Coulter® Scientific Instruments Corp., 1950 West 8th Ave., Hialeah, FL 33010, has been found suitable for this purpose.

[6] Model LS-130 available from Coulter® Scientific Instruments Corp.; Model LA-900 available from Horiba Instruments Inc., 17671 Armstrong Ave., Irvine, CA 92714; Model EPCS-F available from Insitec Measurement Systems, 2110 Omega Road, Suite D, San Ramon, CA 94583; Mastersizer X and Series 2600 available from Malvern Instruments Inc., 10 Southville Road, Southborough, MA 01772, and Microtrac II, Model 158704 and SRA 9200 available from Leeds and Northrup Company, 3000 Old Roosevelt Blvd., St. Petersburg, FL 33716, have been found suitable for this purpose.

[7] Microtrac FRA 7995 available from Leeds and Northrup Company, and Par-Tec® 100 available from Laser Sensor Technology, Inc., 14926 NE 31st Circle, Redmond, WA 98052, have been found suitable for this purpose.

[8] SediGraph 5100 available from Micromeritics® Instrument Corp., 1 Micromeritics Drive, Norcross, GA 30093, has been found suitable for this purpose.

[9] AutoPore 9220 available from Micromeritics® Instrument Corp., has been found suitable for this purpose.

ANNEXES

(Mandatory Information)

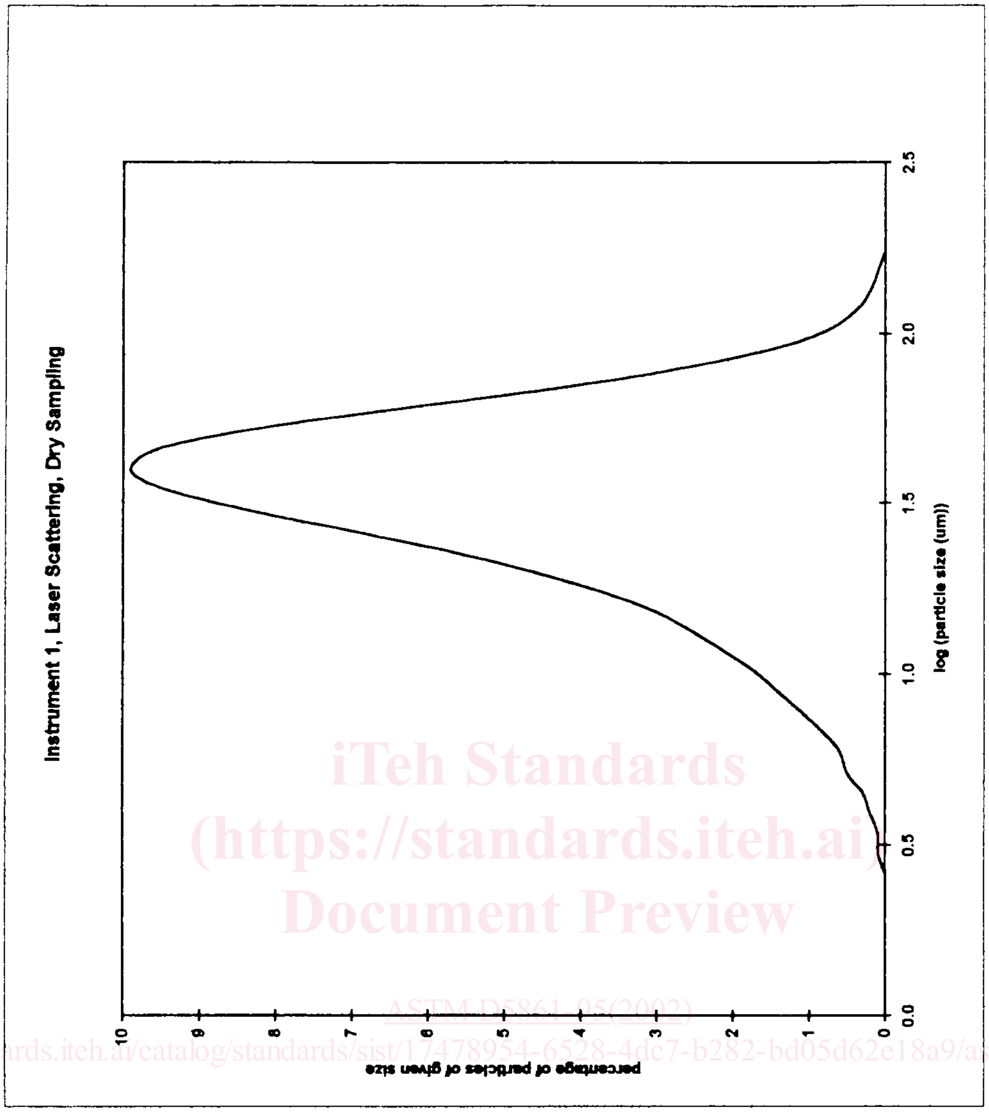
A1. DATA AS ILLUSTRATED IN Table A1.1

TABLE A1.1 Particle Size Data from Secondary Measurement Techniques[A]

Instrument Number	Method	Percent of Particles Less Than Micron Size in Body of Table													Mean, (µm)	Median, (µm)	Mode, (µm)
		5	10	20	25	30	40	50	60	70	75	80	90	95			
1	Laser		11.8			23.0		32.2		42.5			60.4			32.2	
	scattering		11.3			22.4		31.9		42.3			59.6			31.9	
	(dry)		11.6			22.7		32.0		42.3			59.3			32.0	
2	Laser		10.6					29.7					61.1		33.5	29.7	
	scattering		11.0					30.9					64.6		35.1	30.9	
	(wet)		10.8					30.2					62.2		34.2	30.2	
3	Laser		8.4					28.7					58.8		34.8	28.7	36.9
	scattering		8.5					28.6					58.8		35.2	28.6	36.8
	(dry)		8.4					28.6					58.9		35.0	28.6	37.0
4	Laser		8.3					26.1					51.9			26.1	
	scattering		8.4					26.2					52.4			26.2	
	(dry)		8.4					26.1					52.2			26.1	
5	Laser		12.6					33.3					63.2		36.1	33.3	
	scattering		12.8					33.2					63.3		36.1	33.2	
	(dry)		12.7					33.0					63.8		36.1	33.0	
6	Laser														46.1		(37.0)
	scattering														46.1		(37.0)
	(wet)														46.0		(37.0)
7	Laser		9.6					30.3					60.4		32.9	30.3	
	scattering		7.6					29.2					59.8		31.7	29.2	
	(dry)		9.8					30.5					60.0		32.9	30.5	
8	Laser		12.4					32.7					62.1		35.6	32.7	
	scattering		12.2					32.1					60.9		35.2	32.1	
	(dry)		12.4					32.5					61.2		35.6	32.5	
9	Laser	6.8	10.4	15.9		21.1	26.0	30.6	35.3	40.4		46.6	55.7	64.5		30.6	(36.2)
	scattering	6.4	9.8	14.8		19.4	24.0	28.4	32.9	38.1		44.4	54.5	64.5		28.4	(31.0)
	(dry)																
10	Electrolyte		14.5					35.7					74.0		34.0	35.7	38.9
	conductivity		11.0					23.6					43.3		22.7	23.6	26.3
	(wet)		11.2					26.2					58.6		25.7	26.2	27.4
11	Electrolyte		7.6					18.1					38.4		17.7	18.1	19.4
	conductivity		6.4					13.7					29.4		13.8	13.7	14.3
	(wet)		6.0					12.3					24.6		12.4	12.3	12.8
12	Laser		9.6		17.8			30.3			44.7		58.0		32.2	30.3	38.9
	scattering		9.8		18.4			31.3			45.8		60.3		33.6	31.3	38.9
	(dry)		9.9		18.5			31.4			45.8		59.7		33.3	31.4	38.9
13	Sedimentation		(11)					(25)					(42)			(25)	
	(X-ray		(10)					25.1					(46)			25.1	27.2
	absorption)		(10)					25.0					(46)			25.0	27.1
14	Mercury		(9.0)					24.8					(130)			24.8	24.4
	porosimetry[B]		(9.0)					22.4					(90)			22.4	20.3
			(9.0)					24.2					(100)			24.2	24.5
15	Laser	(4.0)	(6.2)	(10.4)	(12.5)	(14.6)	(19.0)	(23.5)	(28.9)	(34.3)	(37.8)	(41.7)	(53.0)	(63.0)		(23.5)	35.8
	scattering	(3.8)	(6.2)	(10.3)	(12.3)	(14.4)	(18.6)	(23.4)	(28.5)	(34.1)	(37.4)	(41.2)	(52.1)	(62.4)		(23.4)	35.7
	(dry)	(3.8)	(6.2)	(10.2)	(12.2)	(14.2)	(18.5)	(23.2)	(28.4)	(34.0)	(37.4)	(41.0)	(52.0)	(62.2)		(23.2)	35.6

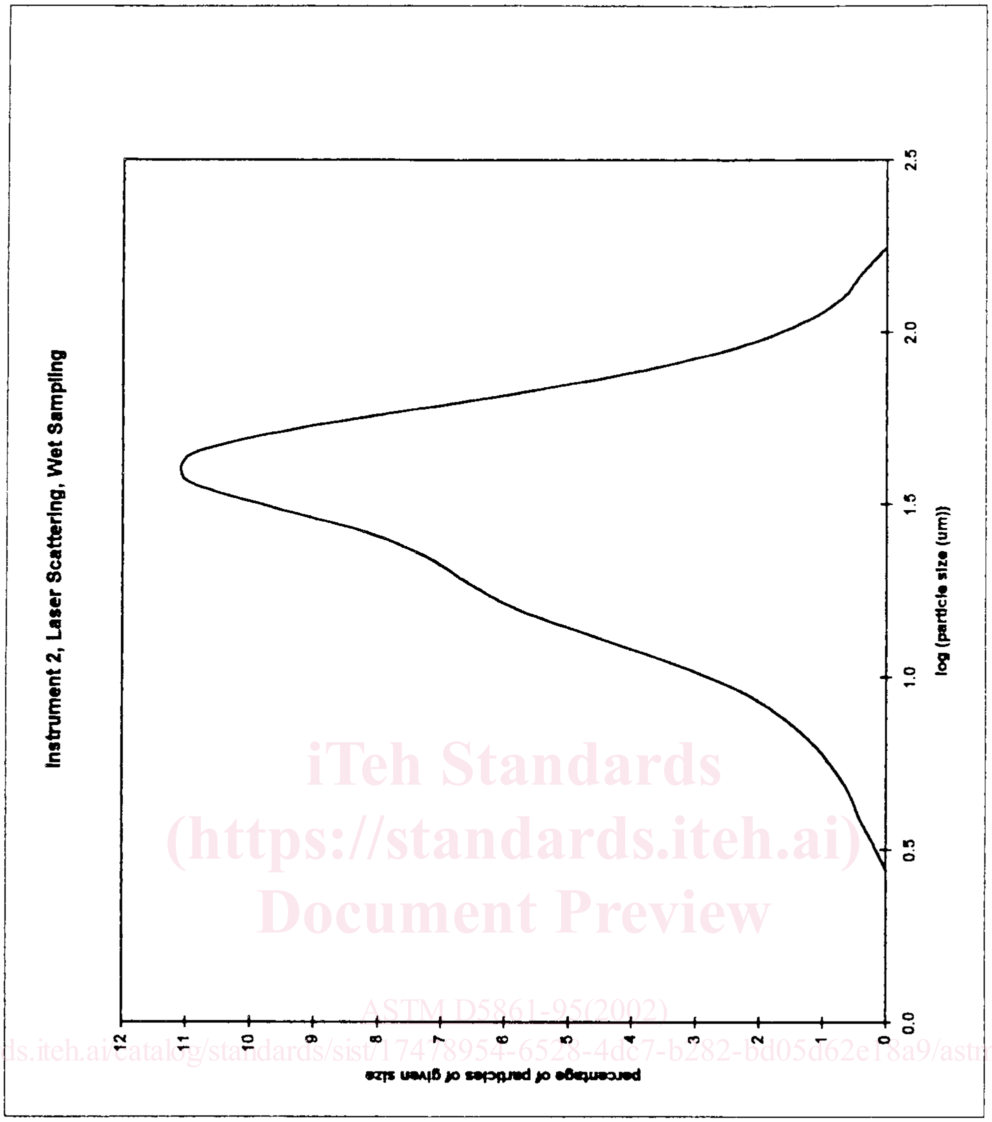
[A]All figures in the body of the table are in microns and are volume based except for instrument No. 13 data which are weight based. Figures in ( ) were not provided explicitly, and so have been estimated from the original data/graphs.

[B]Data processed after Mayer & Stowe.



Instrument 1:		
Particle Size (um)	Log (size)	Percentage of particles of given size
2.8	0.4150	0.0
3	0.4771	0.1
3.4	0.5315	0.1
3.9	0.5911	0.2
4.5	0.6532	0.3
5.1	0.7076	0.5
5.9	0.7709	0.6
6.7	0.8261	0.8
7.7	0.8865	1.1
8.8	0.9445	1.4
10.1	1.0043	1.7
11.6	1.0645	2.1
13.2	1.1206	2.5
15.2	1.1818	3.0
17.4	1.2405	3.7
19.9	1.2989	4.6
22.8	1.3579	5.7
26.1	1.4166	7.0
29.9	1.4757	8.3
34.3	1.5353	9.4
39.2	1.5933	9.9
44.9	1.6522	9.6
51.5	1.7118	8.4
59	1.7709	6.6
67.5	1.8293	4.6
77.3	1.8882	2.9
88.6	1.9474	1.6
101.4	2.0060	0.8
116.2	2.0652	0.4
133.1	2.1242	0.2
152.4	2.1830	0.1
174.6	2.2420	0.0

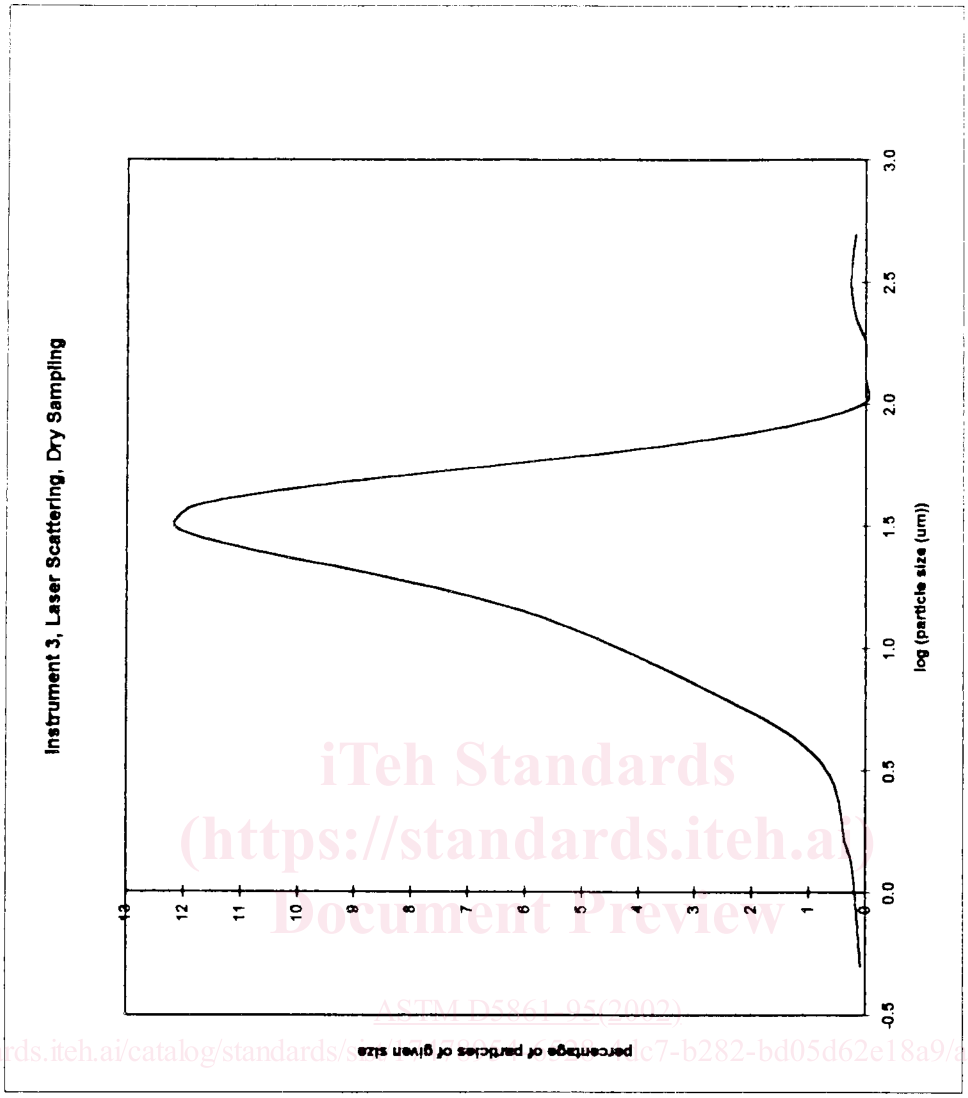
FIG. A1.1 Instrument 1, Laser Scattering, Dry Sampling



Instrument 2:

Particle Size (um)	Log (size)	Percentage of particles of given size
2.8	0.4393	0.0
3.3	0.5145	0.2
3.9	0.5899	0.4
4.6	0.6646	0.6
5.5	0.7404	0.9
6.5	0.8156	1.2
7.8	0.8910	1.7
9.3	0.9681	2.4
11.0	1.0414	3.4
13.1	1.1173	4.6
15.6	1.1931	5.8
18.5	1.2672	6.6
22.0	1.3424	7.2
26.2	1.4183	8.2
31.1	1.4928	9.7
37.0	1.5682	11.0
44.0	1.6435	10.9
52.3	1.7185	9.2
62.2	1.7938	6.7
74.0	1.8692	4.3
88.0	1.9445	2.5
104.7	2.0199	1.4
124.5	2.0952	0.7
148.0	2.1703	0.4
176.0	2.2455	0.0

FIG. A1.2 Instrument 2, Laser Scattering, Wet Sampling



Instrument 3:

Particle Size (um)	Log (size)	Percentage of particles of given size
0.5	-0.3010	0.1
1.3	0.1206	0.3
1.6	0.2041	0.4
2.0	0.2900	0.4
2.4	0.3766	0.5
2.9	0.4624	0.6
3.5	0.5478	0.8
4.3	0.6335	1.3
5.2	0.7193	1.9
6.4	0.8055	2.6
7.8	0.8910	3.3
9.5	0.9768	4.1
11.6	1.0626	5.0
14.1	1.1486	6.0
17.2	1.2343	7.4
20.9	1.3201	9.1
25.5	1.4059	11.0
31.0	1.4915	12.2
37.8	1.5774	11.8
46.0	1.6630	9.6
56.1	1.7489	6.3
68.3	1.8346	3.3
83.3	1.9204	1.1
101.4	2.0062	0.0
123.6	2.0920	0.0
150.6	2.1777	0.0
183.4	2.2635	0.0
223.5	2.3493	0.2
272.3	2.4351	0.2
331.8	2.5208	0.3
404.2	2.6066	0.2
492.5	2.6924	0.2

FIG. A1.3 Instrument 3, Laser Scattering, Dry Sampling