PARTICLE SIZE AND SIZE DISTRIBUTION OF SUPERABRASIVE POWDERS

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The purpose of this article is to present the techniques and standards that are currently employed in the superabrasive industry for the determination of particle size/particle size distribution of diamond and CBN powders.

CENTRIFUGAL SEDIMENTATION PARTICLE SIZE ANALYZER

PARTICLE SIZE CHARACTERIZATION

Knowledge of particle size and size distribution of a powder system is a prerequisite for most production and processing operations. Particle size and size distribution have a significant effect on the surface and bulk properties (mechanical, electrical, thermal), of the finished product. In most instances powder suppliers provide size and size distribution information, but that information needs to be checked for quality control purposes. An adequate control of size and size distribution of the superabrasive powders is imperative to avoid production losses due to high rejection rates of the finished product. Particle size and size distribution can be determined using numerous commercially available instruments. It is important to understand that as different instruments are based on different physical principles, there are bound to be differences in the results obtained from these instruments. Furthermore, even when using instruments based on the same physical principle, the use of proprietary components and variations in adaptations of the same basic physical principle, as well as, the use of proprietary mathematical algorithms, can result in significant variations of the size measurement results. In addition, powder size and size distribution is greatly impacted by particle shape. Thus, comparison of size and size distribution results from different instruments should be conducted with great care and should be based on adequate protocols. The development of such protocols would require instrument calibration coupled with extensive analysis of "standard", well characterized powders that are similar to the powders under investigation. The use of particle size and size distribution data as relative measurements is extremely important when using particle size distribution as a process control tool. The magnitude and/or direction of change are indicative of the changes in the manufacturing process, which can significantly impact on the quality of the product. In such cases, acceptable limits for these fluctuations have to be defined and particle size and/or size distribution has to be monitored to ensure that it lies within these limits. Absolute precision in particle size analysis can only be achieved when the two principle parameters of length and weight are directly traceable to International Standards. Techniques that fulfill these criteria are called "primary" or direct methods and include: microscopy and image analysis, sieve analysis, electrical sensing zone, gravity sedimentation and centrifugal sedimentation. On the other hand, "secondary" or indirect methods of particle size characterization depend on second order effects such as diffraction patterns, Brownian motion, turbidity, etc. In addition, these techniques involve computer modeling (mathematical algorithms coupled with a number of hypothesis) for the interpretation of experimental data. Laser light diffraction, dynamic light scattering and photon correlation spectroscopy are the most popular secondary or indirect particle size measurement techniques/methods. Absolute measurements can be conducted with some degree of reliability when using techniques such as microscopy-based analysis. With these techniques, the particles being measured are visually examined. However, the reliability of the absolute measurement is affected by the number of particles that are being measured, the representative nature of the particles included in the analysis, the particle shape, the state of dispersion and the specimen preparation technique followed. Furthermore, the particles to be analyzed must be spherical so a single dimension can describe their size. *Given the above considerations, it is obvious that powder size and size distribution measurements should be regarded as relative measurements, where results from one run can be compared with those from another run, obtained on the same or, different instrument under similar measurement conditions.* Therefore, the measurement precision is of far greater importance than the accuracy. Effective communication between powder manufacturers/suppliers and customers/end users is a critical factor in the development of robust size measurement procedures, protocols and specifications. Incorporating appropriate audits also enable tracking performance and conformity to the agreed upon protocols and specifications. A recommended general procedure for particle size and size distribution analysis is presented in Figure 1 below. Some of the definitions and calculations used for particle size and size distribution characterizations are presented in Appendix 1. The correspondence between Mesh grit size, equivalent micron size and approximate number of particles in one carat are presented in Appendix 2.

ELECTRICAL SENSING ZONE PARTICLE SIZE ANALYZER

GRIT OR SIEVE SIZES DIAMOND AND CBN POWDERS - SIZING AND STANDARDS SIZE DETERMINATION BY SIEVING TECHNIQUE

In the most general sense, sieving consists of shaking the free flowing dry powder sample through a stacked series of sieves with decreasing mesh size. The mesh with the largest aperture is at the top, and that with the smallest aperture is at the bottom of the stack. Size distribution is reported as the mass of the material retained on a mesh of a given size, but may also be reported as the cumulative mass retained on all sieves above a mesh size or as the cumulative mass fraction above a given mesh size. The following similar ASTM and ISO standards describe the requirements for sieve construction including details on aperture openings, means to measure the same, wire diameter, frame diameter and techniques for maintenance and cleaning of sieves: ASTM E11-95 – Defines specifications for metal wire sieves, ASTM E161-96 – Defines specifications for electroformed sieves. ISO 3310-1 – Technical requirements and testing / Part 1 – Test sieve of metal wire sieves and ISO 3310-3 – Technical requirements and testing / Part 3 – Test sieve of electroformed sheets

TYPICAL SIEVES

CALIBRATION OF SIEVES

Sieve calibration is most practically conducted by sieving a material with a known size distribution and determining the similarity of the test results to that of the test material. The National Institute has developed standard Reference Materials, (SRM), for sieve calibration for Standards and Technology (NIST). These materials are certified by electron and optical microscopy methods for dimension and are intended for sieve calibration. The appropriate NIST SRMs for sieve calibration are listed in Appendix 3 – Part 1. Other proposed techniques for sieve calibration include optical and photometric examination of sieve to determine distribution of aperture size in a particular sieve.

APPLICABLE STANDARDS

The purpose of the standards is to establish a common basis for checking the size of diamond and CBN powders, which are used to manufacture a wide range of industrial superabrasive products (i.e. saws, grinding wheels, etc.). It is intended to serve as common basis of understanding for the producers of superabrasive powders, and for the manufacturers, distributors and users of the superabrasive products. The following equivalent standards apply to grit or sieve size diamond and CBN powders: American Standard ANSI B74.16 / July 12, 2002 – Checking the Size of Diamond and Cubic Boron Nitride Abrasive Grains International Standard ISO 6106 / May 16, 2005 – Abrasive Products – Checking the Grit Size of Superabrasives

SPECIFICATIONS FOR PARTICLE SIZE DISTRIBUTION OF DIAMOND AND cBN GRIT OR SIEVE SIZES

The specification for particle size distribution from electroformed sieves applicable to grit or sieve sizes diamond and CBN powders according to American Standard ANSI B74.16-2002, is defined in Table 1.1.

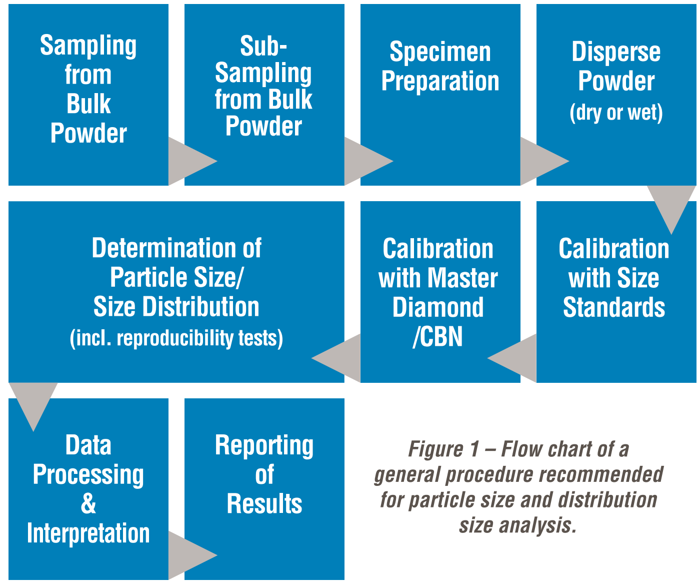


Figure 1 – Flow chart of a general procedure recommended for particle size and distribution size analysis.

Table 1.1 – SPECIFICATION FOR PARTICLE SIZE DISTRIBUTION FROM ELECTROFORMED SIEVE ACCORDING TO ANSI B74.16-2002

USA Mesh Size	ISO Size Design	Nominal ISO Sieve	Test Weight for (Sieve (g))		0.1% Max. On	Oversize Control		Undersize Control			0.5% Max. Through
		Aperature Range (µm)	8" (200mm)	3" (75mm)	Sieve (µm)	Sieve (µm)	Max. % On	Sieve (µm)	Min. % On	Max. % Thru	Sieve (µm)
NARROW RANGE GRADES											
16/18	1181	1180/1000			1830	1280		1010			710
18/20	1001	1000/850			1530	1080		840			600
20/25	851	850/710			1280	915		710			505
25/30	711	710/600			1080	770		600			425
30/35	601	600/500			915	645		505			360
35/40	501	500/425	80-120	9.6-14.5	770	541	5	425	93	5	302
40/45	426	425/355			645	455		360			255
45/50	356	355/300			541	384		302			213
50/60	301	300/250			455	322		255			181
60/70	251	250/212			384	271		213			151
70/80	213	212/180			322	227		181			127
80/100	181	180/150			271	197		151			107
100/120	151	150/125			227	165	7	127	90	7	90
120/140	126	125/106	40-60	4.8-7.2	197	139		107			75
140/170	107	106/90			165	116		90			65
170/200	91	90/75			139	97	8	75	88	8	57
200/230	76	75/63			116	85		65			49
230/270	64	63/53			97	75		57			41
270/325	54	53/45	20-30	2.4-3.6	85	65	12	49	80	12	37
325/400	46	45/38			75	57		41			32

USA Mesh Size	ISO Size Design	Nominal ISO Sieve	0.1% Max. On	Oversize Control		Midpoint Control			Undersize Control		0.5% Max. Through	
		Aperature Range (µm)	Sieve (µm)	Sieve (µm)	Max. % On	Sieve (µm)	Min. % On	Max. % Thru	Sieve (µm)	Min. % On	Max. % Thru	Sieve (µm)
WIDE RANGE GRADES												
16/20	1182	1180/850	1830	1280		1046			840			600
20/30	852	850/600	1280	915		741			600			425
30/40	602	600/425	915	645	5	525	45	55	425	93	5	302
35/40	502	500/355	770	541		440			360			255
40/50	427	425/300	645	455		372			302			213
60/80	252	250/180	384	271		221			181			127

The specification for particle size distribution from wire woven sieves applicable to grit or sieve sizes diamond and CBN powders according to American Standard ANSI B74.16-2002 standard is defined in Table 1.2.

Table 1.2 – SPECIFICATION FOR PARTICLE SIZE DISTRIBUTION FROM WOVEN WIRE SIEVES – ANSI B74.16-2002 STANDARD

USA Grit Size	Test Weight 8" (200mm) Sieves	Sieve through which 99.9% Must Pass	Max. of Oversize on Sieve		Sieve	Min. Retained	Max. Through	Max. of 2% Through Sieve
	GRAMS	MESH	%	MESH	MESH	%	%	MESH
8/10	80 to 120	6	8	8	10	90	8	14
10/12		7		10	12			16
12/14		8		12	14			18
14/16		10		14	16			20

The specification for particle size distribution from electroformed sieves applicable to grit or sieve sizes diamond and CBN powders according to International Standard ISO 6106-2005, is defined in Table 2.0.

Table 2.0

ISO Size Design	Equivalent Mesh Sizes	Test Weight for		99.9% Must Pass Through	Upper Control Sieve	Max. On Sieve	Lower Control Sieve	Min. On Sieve	Max. Through Sieve	0.5% Max. Through
		200 mm Sieves (g)	75 mm Sieves (g)	Sieve (µm)	Sieve (µm)	%	Sieve (µm)	%	%	Sieve (µm)
NARROW RANGE GRADES										
1181	16/18	80-120	9.6-14.5	1830	1280	5	1010	93	5	710
1001	18/20			1520	1080		850			600
851	20/25			1280	915		710			505
711	25/30			1080	770		600			425
601	30/35			915	645		505			360
501	35/40			770	541		425			302
426	40/45			645	455		360			255
356	45/50			541	384		302			213
301	50/60			455	322		255			181
251	60/70			384	271		213			151
213	70/80			322	227		181			127
181	80/100	40-60	4.8-7.2	271	197	7	151	90	7	107
151	100/120			227	165		127			90
126	120/140			197	139		107			75
107	140/170			165	116	8	90	88	8	65
91	170/200			139	97		75			57
76	200/230	20-30	2.4-3.6	116	85		65			49
64	230/270			97	75		57			41
54	270/325			85	65	12	49	83	12	37
46	325/400			75	57		41			32

ISO Size Design	Equivalent Mesh Sizes	Test Weight for		99.9% Must Pass Through	Upper Control Sieve	Max. On Sieve	Lower Control Sieve	Min. On Sieve	Max. Through Sieve	0.5% Max. Through
		200 mm Sieves (g)	75 mm Sieves (g)	Sieve (µm)	Sieve (µm)	%	Sieve (µm)	%	%	Sieve (µm)
NARROW RANGE GRADES										
1182	16/20	80-120	9.6-14.5	1830	1280	5	840	93	5	600
852	20/30			1280	915		600			425
602	30/40			915	645		425			302
502	35/40			770	541		360			255
427	40/50			600	455		302			213
252	60/80			384	271		181			127

The specification for particle size distribution from electroformed sieves applicable to grit or sieve sizes diamond and CBN powders according to Chinese Standard GB/T6406-1996 are defined in Table 3.0.

Table 3.0

Grit Size		99.9% thru Sieve	Upper Control Sieve		Lower Control Sieve			Max. 2% thru Sieve
Mesh	(µm)	(µm)	Sieve (µm)	Pass (%)	Sieve (µm)	Pass (%)	Not Pass (%)	(µm)
NARROW RANGE GRIT SIZES								
16/18	1180/1000	1700	1180		1000			710
18/20	1000/850	1400	1000		850			600
20/25	850/710	1180	850		710			500
25/30	710/600	1000	710		600			425
30/35	600/500	850	600		500			355
35/40	500/425	710	500		425			300
40/45	425/355	600	455	8	360	90	8	255
45/50	355/300	500	384		302			213
50/60	300/250	455	322		255			181
60/70	250/212	384	271		213			151
70/80	212/180	322	227		181			127
80/100	180/150	271	197		151			107
100/120	150/125	227	165	10	127	87	10	90
120/140	125/106	197	139		107			75
140/170	106.90	165	116		90			65
170/200	90/75	139	97	11	75	85	11	57
200/230	75/63	116	85		65			49
230/270	63/53	97	75		57			41
270/400	53/45	85	65	15	49	80	15	–
325/400	45/38	75	57		41			–
WIDE RANGE GRIT SIZES								
16/20	1180/850	1700	1180		850			600
20/30	850/600	1180	850		600			425
30/40	600/425	850	600	8	425	90	8	300
40/50	425/300	600	455		302			213
60/80	250/180	384	271		181			127

The specification for particle size distribution from electroformed sieves applicable to grit or sieve sizes diamond and CBN powders according to Russian Standard GOST 9206-1980 is defined in Table 4.0.

Table 4.0

Size Designation	99.9% thru Sieve	Oversize Control Max. % of Sieve					Main Fraction Min. Retain on Sieve			Max. 2% thru Sieve
	(µm)	8	10	12	13	15	90	80	75	(µm)
NARROW RANGE GRADES										
2500/2000	3000	2500					2000			1600
2000/1600	2500	2000					1600			1250
1600/1250	2000	1600					1250			1000
1250/1000	1600	1250					1000			800
1000/800	1250	1000					800			630
800/630	1000		800					630		500
630/500	800		630					500		400
500/400	630		500					400		315
400/315	500		400					315		250
315/250	400		315					250		200
250/200	315		250					200		160
200/160	250			200				160		125
160/125	200			160				120		100
125/100	160			125				100		80
100/80	125			100					80	63
80/63	100				80				63	50
63/50	80				63				50	40
50/40	63					50			40	

Table 4.0 … *Continued*										
Size Designation	**99.9% thru Sieve**	**Oversize Control Max. % of Sieve**					**Main Fraction Min. Retain on Sieve**			**Max. 2% thru Sieve**
	(µm)	**8**	**10**	**12**	**13**	**15**	**90**	**80**	**75**	**(µm)**
WIDE RANGE GRADES										
2500/2000	3000	2500					1600			1250
1600/1000	2000	1600					1000			600
1000/630	1250	1000					630			500
630/400	800		630					400		315
400/250	500		400					250		200
250/160	315		250					160		125
160/100	200			160					100	80
100/63	125			100					63	50
63/40	80				63				40	

Standard specifications for particle size distribution of grit size diamond and CBN powders as defined by American Standard ANSI B74.16-2002, International Standard ISO 6106-2005, Japanese Standard JIS 4130-1982, Chinese Standard GB/T6406-1996 and Russian Standard GOST 9206-1980 are presented in Table 5.1.

Table 5.1

Grit Designation #	**International Standard ISO 6106-2005**		**USA Standard ANSI B74.6-2002**		**Japanese Standard JIS 4130-1982**		**Chinese Standard GB/T 6406-1996**		**Russian Standard GOST 9206-80**	
	Mesh	**(µm)**	**Mesh**	**(µm)**	**Mesh**	**(µm)**	**Mesh**	**(µm)**	**Mesh**	**(µm)**
										2000/1600
										1600/1250
1182	16/20		16/20	1280/840			16/20	1180/840		
1181	16/18		16/18	1280/1010	16/18	1180/1000	16/18	1180/1000		1250/1000
1001	18/20		18/20	1080/840	18/20	1000/850	18/20	1000/850		1000/800
852	20/30		20/30	915/600	20/30	850/600	20/30	850/600		
851	20/25		20/25	915/710			20/25	850/710		
711	25/30		25/30	770/600			25/30	710/600		800/630
602	30/40		30/40	645/425	30/40	600/425	30/40	600/425		630/400
601	30/35		30/35	645/505			30/35	600/500		630/500
502	35/45		35/45	541/360						
501	35/40		35/40	541/425			35/40	500/425		500/425
427	40/50		40/50	455/302	40/50	425/300	40/50	425/300		
426	40/45		40/45	455/360			40/45	425/355		
356	45/50		45/50	384/302			45/50	355/300		400/315
301	50/60		50/60	322/255	50/60	300/250	50/60	300/250		315/250
252	60/80		60/80	271/181	60/80	250/180	60/80	250/180		250/160
251	60/70		60/70	271/213			60/70	250/212		250/200
213	70/80		70/80	227/181			70/80	212/180		
181	80/100		80/100	197/151	80/100	180/150	80/100	180/150		200/160
151	100/120		100/120	165/127	100/120	150/125	100/120	150/125		160/125
126	120/140		120/140	139/107	120/140	125/106	120/140	125/106		125/100
107	140/170		140/170	116/90	140/170	106/90	140/170	106/90		
91	170/200		170/200	97/75	170/200	90/75	170/200	90/75		100/80
76	200/230		200/230	85/65	200/230	75/63	200/230	75/63		80/63
64	230/270		230/270	75/57	230/270	65/53	230/270	63/53		63/50
54	270/325		270/325	53/45	270/325	53/45	270/325	53/45		50/40
46	325/400		325/400	45/38	325/400	45/38	325/400	45/38		
										< 40

A cross reference chart for grit size diamond and CBN powders, between American Standard ANSI B74.16-2002, International Standard ISO 6106-2005, Chinese Standard GB/T6406-1996 and Russian Standard GOST 9206-1980, based on percentage of main fraction of the particle size distribution of the powder that falls between the upper and lower control sieves (µm) is presented in Table 5.2.

Table 5.2

USA Mesh Size Design	ISO Design	ANSI B74.16-200			ISO 6106-2005			Chinese Standard			Russian Standard		
		Upper Control Sieve µm	Lower Control Sieve µm	Main Fraction %	Upper Control Sieve µm	Lower Control Sieve µm	Main Fraction %	Upper Control Sieve µm	Lower Control Sieve µm	Main Fraction %	Upper Control Sieve µm	Lower Control Sieve µm	Main Fraction %
											2000	1600	
											1600	1250	
16/20	1182	1280	840		1280	840		1180	850				
16/18	1181	1280	1010		1280	1010		1180	1000		1250	1000	90
18/20	1001	1080	840		1080	850		1000	850		1000	800	
20/30	852	915	600		915	600		850	600				
20/25	851	915	710		915	710		850	710				
25/30	711	770	600		770	600		710	600		800	630	
30/40	602	645	425		645	425		600	425		630	400	
30/35	601	645	505		645	505		600	500		630	500	
35/45	502	541	360		541	360							
35/40	501	541	425	93	541	425	93	500	425	90	500	400	
40/50	427	455	302		455	302		455	302				
40/45	426	455	360		455	360		455	360				
45/50	356	384	302		384	302		384	302		400	315	80
50/60	301	322	255		322	255		322	255		315	250	
60/80	252	271	181		271	181		271	181		250	160	
60/70	251	271	213		271	213		271	213		250	200	
70/80	213	227	181		227	181		227	181				
80/100	181	197	151		197	151		197	151		200	160	
100/120	151	165	127	90	165	127	90	165	127	87	160	125	
120/140	126	139	107		139	107		139	107		125	100	
140/170	107	116	90		116	90		116	90				
170/200	91	97	75	88	97	75	88	97	75	85	100	80	
200/230	76	85	65		85	65		85	65		80	63	
230/270	64	75	57		75	57		75	57				
270/325	54	65	49		65	49		65	49		63	50	75
325/400	46	57	41	80	57	41	83	57	41	80	50	40	
											40	/	

MICRON OR SUB-SIEVE SIZES DIAMOND AND CBN POWDERS SIZING AND STANDARDS TECHNIQUES AND EQUIPMENT FOR THE CHARACTERIZATION OF PARTICLE SIZE

Optical microscopy has been traditionally used to develop, define and qualify sub-sieve diamond and CBN powders. Based on optical microscopy, particle size is defined as the diameter of the minimum circumscribed circle that completely encloses the projected image of the particle; another common measure of particle size being the longest single dimension (LSD). With an appropriate calibration scale, an operator can characterize a distribution of particles by visually classifying and manually accumulating counts of particles in different size ranges. Automated optical counting systems (optical or SEM microscopes coupled with a computerized image analyzer) eliminate most of the fatiguing work of this type of analysis. Optical microscopy allows the operator to "really" see the particles and evaluate their range of shape and sizes. However, its major drawback is represented by the fact that it may be difficult to collect enough data to give reliable results. The number of particles measured is usually small compared to other particle sizing methods, so representative sampling becomes critical. In addition, optical microscopy is a tedious and tiresome technique that requires a long time to perform the analysis. Over the years, more sophisticated techniques have been developed to overcome drawbacks and limitations of optical microscopy. Electrical sensing zone, forward and right angle light scattering, dynamic light scattering, centrifugal sedimentation and automated image analysis (optical or SEM microscopy) are techniques used most widely today for determination of particle size distribution. A number of instruments/devices, known as particle size analyzers, are currently available for the determination of particle size distribution of sub-sieve diamond and CBN powders. These instruments are capable of measuring large numbers of micron size particles quickly and accurately. Most are relatively easy to operate, and in one form or another have replaced older methods of characterizing the particle size of diamond and CBN powders based on optical microscopy. As expected, not a single technique/device can be used to measure the whole size spectrum of micron powders with consistent accuracy. Each technique/device is capable of providing optimal performance over a certain size range. Therefore, it is almost impossible to choose a single technique/device to develop a standard for the particle size of micron powders. The main criteria in choosing the right device to measure the size distribution of a particular powder are: 1) highest accuracy over the whole size range under investigation, and 2) lowest interference between the size range, and lower and upper detection limits of the device.

CALIBRATION OF TEST EQUIPMENT

The use of primary and secondary standards for calibration can significantly improve the reliability of analysis. The measurement of particle size is ultimately based on primary standards of length. The use of light or electron microscopy based techniques that can be calibrated to standards certified for length serve as the primary size measurements methods to which other methods, such as electrical sensing zone, light scattering, centrifugal sedimentation, etc. are often calibrated. Following the calibration procedure recommended by the equipment manufacturer, the particle size analyzer employed for the characterization of particle size of diamond and CBN powders shall be calibrated against the following standards:

Primary Standards – National Institute of Standards and Technology (NIST) Standard Reference Materials (SRM)
These SRMs are used for evaluating and calibrating specific types of particle size measuring instruments, including light scattering, electrical sensing zone, flow-through counters, optical and scanning electron microscopes, sedimentation systems, and wire cloth sieving devices.

Secondary Standards – National Institute of Standards and Technology (NIST) Traceable Polymer Spheres Size Standards
Nano-sphere size standards are uniform polystyrene spheres in size range 20 nm to 900 nm, which are calibrated with NIST traceable methodology. The methods used to calibrate the diameters of the nano-spheres are photon correlation spectroscopy (PCS) and transmission electron microscopy (TEM). Nano-sphere size standards are ideal for the calibration of electron and atomic force microscopes. They are also used in laser light scattering studies. Nano-sphere size standards are packaged as aqueous suspensions in 15 milliliter (ml) dropper-tipped bottles; concentrations being optimized for ease of dispersion and colloidal stability. The spheres have a density of 1.05g/cm3 and an index of refraction of 1.59 @ 589 nanometers. Micro spheres size standards are uniform polystyrene spheres in size range 1.0 µm to 160 µm, which are NIST-traceable by calibration methods, such as optical or electron microscopy. Products from 1µm to 160µm in diameter are packaged as aqueous suspensions of polystyrene spheres in dropper-tipped bottles. Diameters of 200 µm and larger are packaged as dry spheres in screw-capped bottles. The spheres have a density of 1.05g/cm3 and an index of refraction of 1.59 @ 589 nanometers. Details on the recommended NIST Standard Reference Materials (SRM), as well as, NIST traceable polymer spheres size standards are presented in Appendix 3 – Part 2.

APPLICABLE STANDARDS

The purpose of the standards is to establish a common basis for checking the size of diamond and CBN powders, in sub-sieve sizes. It is intended to serve as common basis of understanding for the producers of micron superabrasive powders, and for the distributors and users of micron superabrasive powders. The following American Standard applies to micron or sub-sieve size diamond and CBN powders: ANSI Standard B74.20 / February 3, 2004 – Specification for Diamond and Cubic Boron Nitride Powders in Sub-Sieve Sizes

SPECIFICATIONS FOR PARTICLE SIZE DISTRIBUTION OF DIAMOND and cBN MICRON SIZES

The specification for particle size distributions applicable to micron size diamond and CBN powders according to American Standard ANSI B74.20-2004 is defined in Table 6.0.

Table 6.0

Size Range (µm)	D5 Minimum (µm)	D50 ± Tolerance (µm)	D95 Maximum (µm)	D99.9 Largest Particle (µm)
0-0.25	0.0	0.125 ± 0.025	0.25	0.75
0-0.5	0.0	0.25 ± 0.050	0.5	1.5
0-1	0.0	0.5 ± 0.1	1.0	3.0
0-2	0.0	1.0 ± 0.2	2.0	4.0
1-2	1.0	1.5 ± 0.22	2.0	6.0
2-4	2.0	3.0 ± 0.3	4.0	9.0
2-6	2.0	4.0 ± 0.4	6.0	12.0
4-8	4.0	6.0 ± 0.6	8.0	15.0
6-12	6.0	9.0 ± 0.9	12.0	20.0
8-16	8.0	12.0 ± 1.2	16.0	24.0
10-20	10.0	15.0 ± 1.5	20.0	26.0
15-25	15.0	20.0 ± 2.0	25.0	34.0
20-30	20.0	25.0 ± 2.5	30.0	40.0
25-35	25.0	30.0 ± 3.0	35.0	48.0
30-40	30.0	35.0 ± 3.5	40.0	52.0
40-50	40.0	45.0 ± 4.5	50.0	68.0
40-60	40.0	50.0 ± 5.0	60.0	78.0
50-70	50.0	60.0 ± 6.0	70.0	90.0

The specification for particle size distributions applicable to micron size diamond and CBN powders according to Chinese Standard JB/T7900 is defined in Table 7.0.

Table 7.0

Size	Nominal Diameter D µm)	Max. Particle Size D max (µm)	Min. Particle Size D min µm)	Particle Size Distribution
M0/0.5	0 ~ 0.5	0.7	-	1. No particles are larger than the maximum size (Dmax) 2. The content of large particles can not be more than 3% 3. The content of fine particles is <8% for M3/6 or smaller grades, <18% for M4/8 to M10/20 grades, and <28% for M12/22 to M36/54 grades 4. The content of the finest particles is <2% for all particle grades
M0/1	0 ~ 1	1.4	-	
M0.5/1	0.5 ~ 1	1.4	0	
M0.5/1.5	0.5 ~ 1.5	1.9	0	
M0/2	0 ~ 2	2.5	-	
M1/2	1 ~ 2	2.5	0.5	
M1.5/3	1.5 ~ 3	3.8	1	
M2/4	2 ~ 4	5.0	1	
M2.5/5	2.5 ~ 5	6.3	1.5	
M3/6	3 ~ 6	7.5	2	
M4/8	4 ~ 8	10.0	2.5	
M5/10	5 ~ 10	11.0	3	
M6/12	6 ~ 12	13.2	3.5	
M8/12	8 ~ 12	13.2	4	
M8/16	8 ~ 16	17.6	4	
M10/20	10 ~ 20	22.0	6	
M12/22	12 ~ 22	24.2	7	
M20/30	20 ~ 30	33.0	10	
M22/36	22 ~ 36	39.6	12	
M36/54	36 ~ 54	56.7	15	

The specification for particle size distribution applicable to micron size diamond and CBN powders according to Russian Standard GOST 9206-1980 is defined in Table 8.0

Table 8.0

Size Range		Grain Size (µm)	
	Oversize (µm) Max. 5%	Main Fraction (µm) Min. 70%	Undersize (µm) Max. 5%
NARROW RANGE GRADES			
1/0	Over 1 to 2	1 & smaller 95%	---
2/1	Over 2 to 3	From 1 to 2	Smaller than 1
3/2	Over 3 to 5	From 2 to 3	Smaller than 2
5/3	Over 5 to 7	From 3 to 5	From 1 to 2
7/5	Over 7 to 10	From 5 to 7	From 2 to 3
10/7	Over 10 to 14	From 7 to 10	From 3 to 5
14/10	Over 14 to 20	From 10 to 14	From 5 to 7
20/14	Over 20 to 28	From 14 to 20	From 7 to 10
28/20	Over 28 to 40	From 20 to 28	From 10 to 14
40/28	Over 40 to 60	From 28 to 40	From 14 to 20
60/40	Over 60 to 80	From 40 to 60	From 20 to 28
WIDE RANGE GRADES			
2/0	Over 2 to 3	2 and finer	---
3/0	Over 3 to 5	3 and finer	---
3/1	Over 3 to 5	From 1 to 3	---
5/2	Over 5 to 7	From 2 to 5	Smaller than 2
7/3	Over 7 to 10	From 3 to 7	From 1 to 2
10/5	Over 10 to 14	From 5 to 10	From 2 to 3
14/7	Over 14 to 20	From 7 to 14	From 3 to 5
20/10	Over 20 to 28	From 10 to 20	From 5 to 7
28/14	Over 28 to 40	From 14 to 28	From 7 to 10
40/20	Over 40 to 60	From 20 to 40	From 10 to 14
60/28	Over 60 to 80	From 28 to 60	From 14 to 20

Standard specifications for particle size distribution of micron size diamond and CBN powders as defined by American Standard ANSI B74.20-2004, Japanese Standard, JIS6002-63, Chinese Standard JB/T7900 and Russian Standard GOST 9206-1980 are presented in Table 9.0.

Table 9.0

USA Standard ANSI B74.20-2004	Japanese Standard JIS6002-63		Chinese Standard JB/T7900		Russian Standard GOCT 9206-80	
Size Range (µm)	Size Designation	Size Range (µm)	Size Designation	Size Range (µm)	Size Designation	Size Range (µm)
					0.1/0	< 0.1
0-0.25						
					0.3/0	< 0.3
0-0.5			M0/0.5	0-0.5	0.5/0	< 0.5
					0.5/0.1	0.5-0.1
					0.7/0.3	0.7-0.3
			M0.5/1	0.5-1	1/0.5	1-0.5
0-1	15000	1/0	M0/1.0	0-1	1/0	< 1
			M0.5/1.5	0.5-1.5		
0-2			M0/2	0-2	2/0	< 2
					3/0	< 3
1-2	8000	2/1	M1/2	1-2	2/1	2-1
					3/1	3-1
			M1.5/3	1.5-3		
	5000	3/2			3/2	3-2
2-4			M2/4	2-4		
					5/2	5-2
			M2.5/5	2.5-5		
	4000	4/3				
					5/3	5-3
2-6						
	3000	5/4				
			M3/6	3-6		
	2500	6/5				
					7/3	7-3
					7/5	7-5
4-8			M4/8	4-8		
	2000	8/6				
					10/7	10-7
	1500	10/8	M5/10	5-10	10/5	10-5
6-12			M6/12	6-12		
			M8/12	8-12		
	1200	13/10				
8-16			M8/16	8-16		
					14/7	14-7
					14/10	14-10
	1000	16/13				
10-20			M10/20	10-20	20/10	10-20
			M12/22	12-22		
					14/20	20-14
15-25					28/14	28-14
20-30			M20/30	20-30	28/20	28-20
25-35	700	24/20	M22/36	22-36		
	600	38/24				
500	34/28					
					40/20	40-20
30-40					40/28	40-28
	400	37/34	M36/54	36-54		
40-50						
					60/28	60-28
40-60					60/40	60-40
50-70						

The purpose of these standards is to establish a common basis for checking the size of diamond and CBN powders, which are used to manufacture a wide range of industrial superabrasive products (i.e. saws, grinding wheels, etc.) and also powders, in sub-sieve sizes used for polishing or fine texturing applications. It is intended to serve as common basis of understanding for the producers of superabrasive powders, and for the manufacturers, distributors and users of the superabrasive products. This article will be available through the Industrial Diamond Association of America for crossreference and guidelines on standards.

REFERENCES:

Particle Size Characterization – NIST Special Publication 960-1
ANSI Standard B74.16 / 2002 – Checking the Size of Diamond and Cubic Boron Nitride Abrasive Grains
ANSI Standard B74.20 /2004 – Specification for Diamond and Cubic Boron Nitride Powders in Sub-Sieve Sizes
ISO Standard 6106 / 2005 – Abrasive products – Checking the grit Size of Superabrasives
Chinese Standard GB/T6406-1996
Chinese Standard JB/T7900
Russian Standard GOST 9206-1980
Japanese Standard, JIS6002-63
Japanese Standard JIS 4130-1982

APPENDIX 1

Units for Particle Size:

Micro Meter (µm) = 10^{-6} meter (m)
Nano Meter (nm) = 10^{-3} µm = 10^{-9} m
Angstrom (Å) = 10^{-1} nm = 10^{-4} µm = 10^{-10} m

Mesh: Number of (sieve) openings per linear inch, starting with a ruler zeroed on the center of any wire

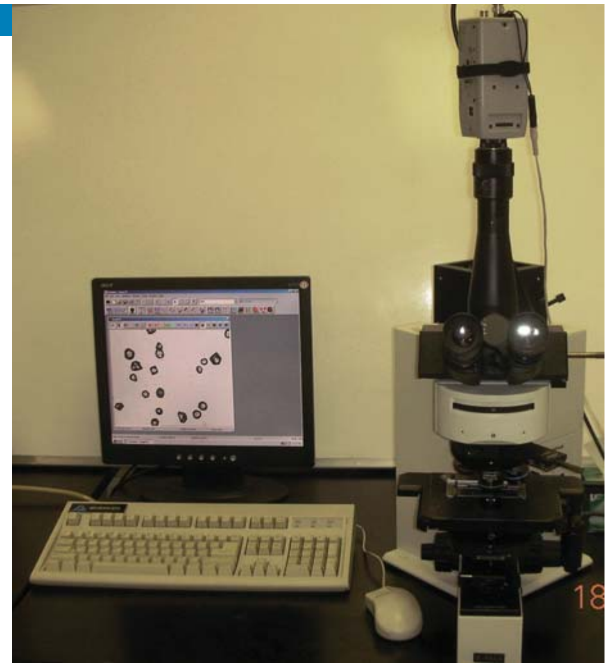
Accuracy: Measure of how close a measured value is to the true value

Precision: Measure of the variation in repeated measurements (same instrument & operator)

Resolution: Measure of the minimum detectable differences between features in a size distribution or the capability of the equipment to resolve or separate adjacent narrow peaks that differ in size.

Reproducibility: Measure of the variation between different instruments, operators and sample preparation

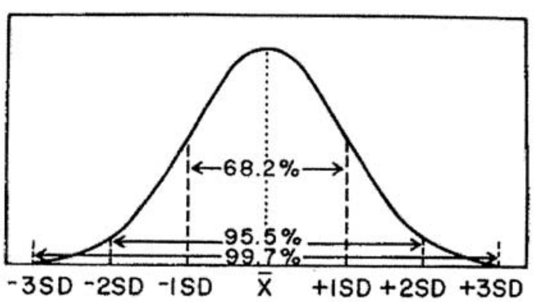
Mean: The mean is determined by adding a group of measured values, then dividing the total by the number of measurements in the group: X = $\sum Xi/n$. The mean value is related to accuracy or systematic error. The mean value for a control material provides an estimate of the central tendency of the distribution that is expected if method performance remains stable. Any change in accuracy, such as schematic shift or drift, would be reflected in change in mean value of the control, which would be shown by a shift or drift of the distribution of control results.



OPTICAL MICROSCOPY IMAGE ANALYZER

Standard Deviation: Standard deviation is determined by first calculating the mean, then taking the difference of each control result from the mean, squaring that difference, dividing by n-1, then taking the square root: _ = $\sqrt{} [\sum (Xi - X)^2 / (n-1)]$. The standard deviation is a measure of the distribution and is related to imprecision or random error. The bigger the standard deviation, the wider the distribution, the greater the random error, and the poorer the precision of the method; the smaller the standard deviation, the narrower and sharper the distribution, the smaller the random error, and the better the precision of the method. For a measurement procedure, it is generally expected that the distribution of control results will be normal or Gaussian. For a Gaussian distribution, the percentage of results that are expected with certain limits can be predicted. For example, for control results that fit a Gaussian distribution, it would be expected that 68.2% of observed results will be within $\pm 1\sigma$ of the mean; 95.5% within $\pm 2\sigma$ of the mean and 99.7% within $\pm 3\sigma$ of the mean.

Control Limits: Given the mean and standard deviation for a control material, control limits are calculated as the mean plus a certain multiple (n) of the standard deviation, such as 2σ or 3σ. Upper control limit (UCL) = Mean + $n\sigma$. Lower control limit (LCL) = Mean - $n\sigma$



Sub-sieve size diamond and CBN powders - Conformance to ANSI B74.20: Nominal standard particle size distributions and acceptability limits for those distributions are summarized in Table 6.0 – Specification for particle size distribution of sub-sieve diamond and CBN powders - ANSI B74.20-2004. Standard definitions for each of the columns in the table are listed below:

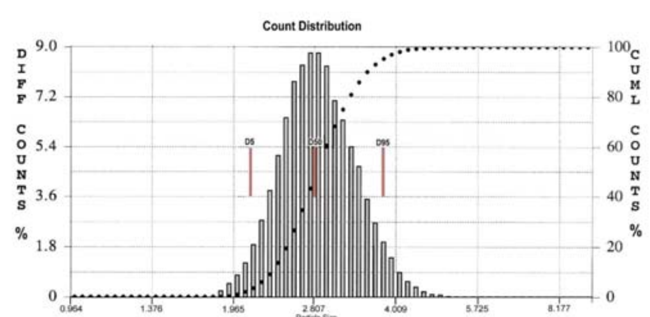
Size Range: The range of the distribution is a more practical way of designating the standard particle size distributions. While the range designation is used as standard practice when specifying sub-sieve distributions, it does not represent the actual particle size range of the distribution. Rather, it has become an arbitrary method for designating certain size distributions, which are now commonly practiced by those in the industry.

D5 (Minimum): The D5 (Minimum) value represents the particle size for which 95 percent of the particles are coarser. By specifying a minimum value, the standard specifies a "lower limit" for 95 percent of the particles in the count distribution.

D50 & Tolerance: The D50 value represents the particle size for which 50 percent of the particles are coarser, and 50 percent of the particles are finer. The tolerance on the D50 value represents the permissible deviation from the number that will be allowed and while still conforming to this standard.

D95 (Maximum): The D95 (Maximum) value represents the particle size, for which 95 percent of the particles are finer. By specifying a maximum value, the standard specifies an "upper limit" for 95 percent of the particles in the count distribution.

D99.9 (Largest Particle): The D99.9 (Largest Particle) value represents the particle size, for which no particles coarser are present. By specifying a largest IMAGE particle value, the standard specifies an "upper threshold" for the entire population of particles in the distribution. The D99.9 figure is read as the last channel of measurable particles. Confirmation of oversize particles should be made by microscopy.



Example of 2-4 Microns Diamond Particle Size Distribution

APPENDIX 2

USA Grit Size (Mesh)	Approximate Particle Size (μm)	Number of Particles in One Carat
GRIT or SIEVE SIZES		
8/10	2380-2000	4.2-7
10/12	2000-1680	7-12
12/24	1680-1410	12-20
14/16	1410-1190	20-33
16/18	1190-1000	35-57
18/20	1000-840	57-97
20/25	840-710	97-160
25/30	750-590	160-282
30/35	590-500	282-460
35/40	500-420	460-770
40/45	420-350	770-1334
45/50	350-297	1,334-2,080
50/60	297-250	2,080-3,240
60/80	250-177	3,240-10,400
80/100	177-149	10,400-17,140
100/120	149-125	17,140-20,920
120/140	125-105	20,920-49,400
140/170	105-88	49,400-82,400
170/200	88-74	82,400-140,000
200/230	74-62	140,000-252,000
230/270	62-33	252,000-384,000
270/325	33-44	384,000-660,000
325/400	44-37	660,000-1,120,000
MICRON OR SUB-SIEVE SIZES		
400-500	60-40	1,120,000-1,580,000
500/600	50-25	1,580,000-2,046,000
600/700	37-22	~ 2,046,000
700/800	36-40	~ 9,503,000
1200	18-12	~ $1.696*10^{7}$
1800	12-8	~ $1.696*10^{7}$ – $2.62*10^{8}$
2200	10-6	~ $7.86*10^{7}$
3000	8-4	~ $2.62*10^{8}$
6000	6-2	~ $8.82*10^{8}$
8000	4-2	~ $2.05*10^{9}$
12000	3-1	~ $2.05*10^{9}$ – $6.2*10^{10}$
15000	2-0	~ $6.2*10^{10}$
25000	1-0	$6.2*10^{10}$

APPENDIX 3 – Part 1

STANDARDS FOR CALIBRATION OF TEST EQUIPMENT
PART 1, STANDARD REFERENCE MATERIALS FOR SIEVE CALIBRATION

PRIMARY STANDARDS – Standard Reference Materials (SRM) National Institute for Standards and Technology (NIST) has developed Standard Reference Materials (SRM) for sieve calibration that are certified for dimension by electron and optical microscopy methods. SRM 8010 is a three bottle set of different sands (A, C and D), intended for use in sieving only, and covers the sieve size range from 30 mesh to 325 mesh, as follows:

SRM	Description	Particle Diameter Distribution	Unit Size
8010A	Sand	30 to 100 mesh	130g
8010C	Sand	70 to 200 mesh	130g
8010C	Sand	110 to 325 mesh	130g

SRMs 1003c, 1004b, 1017b, 1018b and 1019b each consist of soda-lime glass beads covering a particular size distribution (PSD) range.

SRM	Description	Particle Diameter Distribution	Unit Size
1003c	Glass Beads	10 to 60 μm / 600 to 325 mesh	25g
1004b	Glass Beads	40 to 150 μm / 270 to 120 mesh	43g
1017b	Glass Beads	100 to 400 μm / 140 to 45 mesh	70g
1018b	Glass Beads	220 to 750 μm / 60 to 25 mesh	87g
1019b	Glass Beads	750 to 2450 μm / 20 to 10 mesh	200g

APPENDIX 3 – Part 2

STANDARDS FOR CALIBRATION OF TEST EQUIPMENT PART 2

STANDARDS FOR CALIBRATION OF PARTICLE SIZE DISTRIBUTION ANALYZERS PRIMARY STANDARDS – STANDARD REFERENCE MATERIALS (SRM) NIST has developed various standards, available as SRMs, for calibration and performance evaluation of particle size distribution analyzers. Following SRMs are recommended for the calibration of the laser diffraction instruments:
– SRMs 1690, 1691, 1692, 1963 and 1964 are commercially manufactured mono-disperse latex particles in a water suspension.
– SRMs 1960 and 1961 are mono-disperse latex particles in a water suspension produced by the National Aeronautics and Space Administration (NASA).
– SRM 1965 consists of two different groupings of the SRM 1960 particles mounted on a microscope slide.

Following SRMs are recommended for the calibration of instruments based on gravitational sedimentation:
– SRM 659 consists of equiaxed silicon nitride particles measured using sedimentation.
– SRM 1978 consists of granular, irregular shaped zirconium oxide particles measured using sedimentation.
– SRM 1982 consists of spherical particles measured using scanning electron microscopy, laser scattering, and sieving.

SRM	Description	Particle Diameter Distribution	Unit Size
659	Silicon Nitride	0.2 to 10 µm	2.5g
1978	Zirconium Oxide	0.33 to 2.19 µm	5g
1982	Zirconium Oxide	10 to 150 µm	10g
1984	Tungsten Carbide/Cobalt	9 to 30 µm	14g
1985	Tungsten Carbide/Cobalt	18 to 55 µm	14g

SECONDARY STANDARDS – NIST TRACEABLE POLYMER SPHERES SIZE STANDARDS In addition to the NIST Standard Reference Materials, numerous secondary standards are available from NIST, as well as, from instrument manufacturers and vendors of scientific supplies. These secondary standards are all calibrated against primary standards developed by international standards accreditation agencies.

SRM	Description	Particle Diameter Distribution	Unit Size
1690	Polystyrene Spheres (1 µm)	0.895 µm	5 ml vial
1691	Polystyrene Spheres (0.3 µm)	0.269 µm	5 ml vial
1692	Polystyrene Spheres (3.0 µm)	2.982 µm	5 ml vial
1961	Polystyrene Spheres (30 µm)	29.64 µm	5 ml vial
1963a	Polystyrene Spheres (0.1 µm)	0.1018 µm	5 ml vial
1964	Polystyrene Spheres (0.06 µm)	0.0639 µm	5 ml vial
1965	Polystyrene Spheres (10 µm) (on slide)	9.94 µm (hexagonal array) 9.89 (unordered clusters)	1 slide

DEFINITIONS

Reference Material Certificate – Document accompanying a certified reference material stating one or more property values and their uncertainties, and confirming that the necessary procedures have been carried out to ensure their validity and traceability. (ISO Guide 30: 1992)

NIST Standard Reference Material® (SRM) - A CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material (NIST SP 260-136). Note: An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs. The terms "Standard Reference Material" and the diamond-shaped logo that contains the term "SRM," are registered with the United States Patent and Trademark Office.

NIST Reference Material - Material issued by NIST with a report of investigation instead of a certificate to: (1) further scientific or technical research; (2) determine the efficacy of a prototype reference material; (3) provide a homogeneous and stable material so that investigators in different laboratories can be ensured that they are investigating the same material; and (4) ensure availability when a material produced and certified by an organization other than NIST is defined to be in the public interest or when an alternate means of national distribution does not exist. A NIST RM meets the ISO definition for a RM and may meet the ISO definition for a CRM (depending on the organization that produced it).

NIST Traceable Reference Material TM (NTRMTM) – A commercially produced reference material with a well-defined traceability linkage to existing NIST standards for chemical measurements. This traceability linkage is established via criteria and protocols defined by NIST to meet the needs of the metrological community to be served (NIST SP 260-136). Reference materials producers adhering to these requirements are allowed use of the NTRM trademark. A NIST NTRM may be recognized by a regulatory authority as being equivalent to a CRM.

NIST Certified Value - A value reported on an SRM certificate or certificate of analysis for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST. (NIST SP 260-136)

NIST Reference Value - A best estimate of the true value provided on a NIST certificate, certificate of analysis, or report of investigation where all known or suspected sources of bias have not been fully investigated by NIST. (NIST SP 260-136)

NIST SRM Certificate or Certificate of Analysis - In accordance with ISO Guide 31: 2000, a NIST SRM certificate is a document containing the name, description, and intended purpose of the material, the logo of the U.S. Department of Commerce, the name of NIST as a certifying body, instructions for proper use and storage of the material, certified property value(s) with associated uncertainty (ies), method(s) used to obtain property values, the period of validity, if appropriate, and any other technical information deemed necessary for its proper use. A Certificate is issued for an SRM certified for one or more specific physical or engineering performance properties and may contain NIST reference, information, or both values in addition to certified values. A Certificate of Analysis is issued for an SRM certified for one or more specific chemical properties. Note: ISO Guide 31 is updated periodically; check with ISO for the latest version.

NIST Certificate of Traceability – Document stating the purpose, protocols, and measurement pathways that support claims by an NTRM to specific NIST standards or stated references. No NIST certified values are provided, but rather the document references a specific NIST report of analysis, bears the logo of the U.S. Department of Commerce, the name of NIST as a certifying body, and the name and title of the NIST officer authorized to accept responsibility for its contents.

Standards for calibration should also be developed from the powders being measured on a regular basis. In addition, it is critical to compare the material used as "standard" against a traceable primary standard. It is also recommended to develop protocols for sample preparation, analysis and data interpretation, particular to the material investigated and the instrument used.