

July 9, 2010 E-29650

U. S. Nuclear Regulatory Commission Attn: Document Control Desk One White Flint North 11555 Rockville Pike Rockville, MD 20852

Subject: BORAL[®] Boron Carbide Particle Size (Docket No. 72-1030; TAC NO. L24153)

References: 1. EPRI Technical Report 1011819, Neutron Transmission Through BORAL[®]: Impact of Channeling on Criticality, Final Report, June 2005.

- 2. AAR Topical Report 1829, Credit for 90% of the ¹⁰B in BORAL[®], October 27, 2004.
- 3. Matco Services Inc. Report, Determination of B₄C Particle Sizes in BORAL[®] Neutron Absorber Material, May 13, 2010, Project 910-90193 (note typographical error corrected for report date, actually issued in 2010 vs. 2009).
- Matco Services Inc. Report, Determination of B₄C Particle Sizes in BORAL[®] Neutron Absorber Material – Report #2, New Measurements Using Laser Light Scattering, Revision 2, July 1, 2010, Project 910-90193.

As discussed in recent conversations with NRC Staff regarding the determination of average boron carbide (B₄C) particle size contained in BORAL[®] neutron absorber material, Transnuclear (TN) provides the following information regarding continued compliance to the NUHOMS[®] HD System Technical Specifications associated with NRC Certificate of Compliance (CoC) 1030.

Summary

TN recently learned that the manufacturer's description for particle size of the boron carbide in BORAL[®] does not match the average size of approximately 85 microns *before rolling* described in the Technical Specifications for CoC 1004 Amendment 10. With the particle size before rolling in question, the particle size *after rolling*, described in the Technical Specifications for CoC 1030 Amendment 0 also comes into question. Specifically, Section 9.1.7.3 of the NUHOMS[®] HD UFSAR (incorporated by reference in the CoC 1030 Technical Specification Section 4.3.1) provides a description of the BORAL[®] poison plate product utilized for criticality control in the 32PTH DSC indicating an average boron carbide particle size in the finished product after rolling of approximately 50 microns. Subsequent laboratory testing of excess BORAL[®] material used in 32PTH DSCs fabricated for Dominion's Surry and North Anna plants indicates that the average size of the boron carbide particles for those test coupons satisfies that description. Therefore, the Dominion 32PTH DSCs that use BORAL[®] fabricated for Surry and North Anna plants comply with the CoC 1030 Technical Specifications. Other than those fabricated for Dominion, no canisters using BORAL[®] have been fabricated under CoC 1030.

NMSSDI

Background

BORAL[®] has been provided to the commercial nuclear power industry for several decades and has been manufactured by three different companies, Brooks & Perkins, AAR Corporation and most recently Ceradyne Inc. A consistent particle size specification has been imposed by the two most recent manufacturers (at least since 1993) for the boron carbide in its initial powder form (i.e., prior to processing/manufacturing) and is based on the following particle size distribution (specific particle sizes and percentages are Ceradyne proprietary information).

- No particles shall be greater than X1 microns (separate out all particles retained on size X Sieve - equivalent to X1µm).
- "A" wt-% maximum of particles may be between Y1 and X1 microns (utilize "A" wt-% maximum particles which are retained on size Y Sieve equivalent to Y1µm).
- "B" wt-% maximum of particles may be less than Z1 microns (utilize "B" wt-% maximum particles which pass through size Z Sieve - equivalent to Z1µm).

This particle size distribution establishes a maximum particle size of X1 microns and requires that the bulk of the material lie in the range between Z1 and Y1 microns in the initial powder form. The material supplier certifies their boron carbide powder to this specification.

Technical Significance of Particle Size

From a design perspective, a fine powder is desired to minimize the potential for self-shielding effects and neutron channeling in the finished product. NRC guidance provided in ISG-15 cautions that neutron poison materials with large poison particles (defined as 80 microns) have been shown to absorb significantly fewer neutrons than homogeneous materials with the same poison loading. Neutron channeling occurs when a neutron takes a path between boron carbide particles that allows it to penetrate the absorber sheet without attenuation. However, technical literature (Reference 1) indicates neutron channeling is reduced by finer granulation of boron carbide particles and that it has a negligible effect on criticality calculations in the flooded condition for boron carbide particle sizes up to 150 microns.

BORAL[®] is typically manufactured in various thicknesses from 0.075" to up to 0.300". Due to the rolling process utilized in manufacturing, the thinner material will generally contain smaller average particles as the initial particles are crushed to a finer size in the thinner product. The ¹⁰B loading also affects particle size reduction due to rolling with the higher loaded ¹⁰B product containing smaller average particle size after rolling. Therefore, considering that the initial range of the majority of particles is between Z1 and Y1 microns, a relatively large average particle size range is possible for the entire family of BORAL[®] product thicknesses and ¹⁰B loading.

Basis for Particle Size Reported in NUHOMS® HD UFSAR

The description of the approximate 50 micron average particle size was initially provided in an AAR topical report (Reference 2) intended to justify a 90% credit for the ¹⁰B in BORAL[®] for criticality analyses. AAR reported an average particle size after rolling of "about 50 microns" relative to the concern stated in ISG-15 for neutron channeling. The AAR topical report states that the 50 micron size is smaller than the particle size that may cause significant channeling between particles. Note that the overall justification developed by AAR for reducing the poison loading penalty in criticality design from 25% to 10% was never accepted by the NRC and therefore TN continues to take a conservative poison loading credit of only 75% in the criticality analyses performed for CoC 1030.

This same descriptive language regarding average particle size was incorporated in the initial NUHOMS[®] HD FSAR. TN is not aware of prior direct size measurements performed by the manufacturer intended to quantify the entire range of particle size for the family of BORAL[®] products. Therefore, the average particle size provided in the material description represents a *reasonable approximation* by the manufacturer to support the arguments made in their topical report for minimizing neutron channeling effects.

Conclusion Regarding License Compliance

In order to quantify the particle size in BORAL[®] utilized in TN products, TN contracted with a materials laboratory to perform direct measurement of particle size for representative material. Coupons were cut from spare sheets of BORAL[®] material remaining from the prior fabrication of Dominion 32PTH DSCs. Those coupons were subjected to dissolution in acid separating the boron carbide particles for subsequent measurement. The lab results provided in Reference 3 (attached as Enclosure 1) utilizing image analysis from scanning electron microscope (SEM) micrographs indicate a lower average particle size than that provided in the BORAL[®] description. Additional lab results provided in Reference 4 (attached as Enclosure 2) utilizing a more sophisticated measurement technique (i.e., laser light scattering particle size distribution analysis) indicate an average particle size of 31.7 microns determined by particle size frequency method and 55.8 microns determined by mass fraction method, essentially equal to that provided in the BORAL[®] description. Although the average particle size of 31.7 microns determined by particle size frequency method and 55.8 microns determined by ass fraction method, essentially equal to that provided in the BORAL[®] description. Although the average particle size of 31.7 microns determined by particle size frequency method is less than that provided in the BORAL[®] description, the performance of BORAL[®] material is enhanced by a smaller particle size as discussed above. Therefore, TN has concluded that these results are consistent with the Technical Specification description.

Regarding the scope of the testing program, one random sheet was selected from spare inventory for each of three lots of BORAL[®] material remaining from production and analyzed accordingly. This testing is analogous to qualification testing rather than production acceptance testing such that additional test coupons are deemed unnecessary to support the license compliance conclusion.

Additional testing was performed on coupons provided by Ceradyne Inc. representative of more recent BORAL[®] manufacturing. Although these results reported in Reference 4 have no relevance to the Dominion 32PTH canisters which utilized only AAR manufactured BORAL[®], they are provided as an indication that the particle size after rolling remains consistent, at least for the specific BORAL[®] material parameters tested.

Effect on Previously Certified DSC Products under NRC CoC 1030

BORAL[®] has been utilized in a total of twenty four (24) 32PTH DSCs supplied to Dominion's Surry and North Anna plants. A total of three (3) lots of BORAL[®] were utilized in those canisters, with all lots manufactured by AAR in their former facility in Livonia Michigan, involving the same manufacturing processes and procedures with the same specified material thickness and ¹⁰B loading.

TN had previously evaluated three (3) recently loaded DSCs for Surry and determined that they remained compliant with the Technical Specifications based on the initial test results (Reference 3). Although those results indicated a smaller average particle size, this was deemed acceptable relative to the BORAL[®] product line based on the material thickness and poison loading. The spare sheet of BORAL[®] analyzed in this lab report was taken from the same lot of material utilized for the vast majority of the poison plate contained in the canisters loaded in Surry's recent campaign (100% from analyzed lot for S/N's DOM-32PTH-018-C and DOM-32PTH-023-C, and 99% for S/N DOM-

32PTH-024-C) such that the test results were representative for those specific canisters. Therefore, it was concluded that the canisters involved in the recent loading campaign at Surry were compliant with the CoC 1030 license and Technical Specifications.

Regarding the balance of Dominion 32PTH canisters for the Surry and North Anna plants; those canisters previously loaded (16 total) and those canisters which have been certified but not yet loaded (5 total), subsequent testing (Reference 4) included the other two lots of BORAL[®] material representative of those canisters by selecting one spare sheet from each of the two lots for testing. As expected, the test results for all 3 lots reported in Reference 4 are similar, since the manufacturing process and technical requirements for these lots were essentially identical. Therefore, the test results reported in References 3 and 4 support the conclusion that all 24 Dominion 32PTH canisters which utilize BORAL[®] poison plate are compliant with the CoC 1030 license and Technical Specifications.

Should the NRC staff require additional information regarding this matter, please do not hesitate to contact Donis Shaw at 410-910-6878 or me at 410-910-6881.

Sincerely,

your Som

ayant Bondre, PhD Vice President - Engineering

cc: B. Jennifer Davis (NRC SFST)

Enclosures:

- 1. Matco Services Inc. Report, Determination of B₄C Particle Sizes in BORAL[®] Neutron Absorber Material, May 13, 2010, Project 910-90193 (note typographical error corrected for report date, actually issued in 2010 vs. 2009).
- 2. Matco Services Inc. Report, Determination of B₄C Particle Sizes in BORAL[®] Neutron Absorber Material Report #2, New Measurements Using Laser Light Scattering, Revision 2, July 1, 2010, Project 910-90193.

Enclosure 1 to TN E-29650

Matco Services Inc. Report, Determination of B₄C Particle Sizes in BORAL[®] Neutron Absorber Material, May 13, 2010, Project 910-90193 (note typographical error corrected for report date, actually issued in 2010 vs. 2009)



Report of: Determination of B₄C Particle Sizes in Boral Neutron Absorber Material

Report To: Peter Quinlan TransNuclear 7135 Minstrel Way, Suite 300 Columbia, MD 21045

Phone: 410-910-6895 Fax: 410-910-6902

Summary:

Boral neutron absorber material consists of a layer of boron carbide (B₄C) particles in an aluminum matrix, sandwiched between two sheets of aluminum. The manufacturer's product description gives the average boron carbide particle size as 50μ m. Recently, concerns have emerged that the actual average particle size may be larger.

To date, particle sizes have been determined for one sample of Boral material. The average particle size appears to be significantly less than 50μ m. Data collected so far indicate that the peak of the B₄C particle size distribution lies between 2.5 and 5.0 μ m. Despite the presence of larger (~40 μ m) particles, the high population of small particles brings the average particle size down below 10 μ m.

Procedure:

In order to determine the particle size, the boron carbide particles were first isolated by dissolving away the aluminum matrix in a 50% HCl solution. The resulting powder was extracted to a 1.2μ m Millipore filter and imaged in Scanning Electron Microscopy (SEM). The length scale used in SEM image acquisition was calibrated to a Scanning Electron Microscope Magnification Standard produced by the National Standards Bureau (Specimen ID: JY-55-PE).

The SEM micrograph images were then analyzed in the ImagePro software package to determine particle sizes and statistics. Particle sizes were measured as Feret's statistical diameters. Feret's diameter measures the perpendicular distance between parallel lines, tangent to opposite sides of the outline of a particle. When Feret's diameter is determined for randomly oriented particles, it produces an average over all possible orientations. The Feret's diameters were taken horizontally in the image (ie. between two vertical lines) for all particles measured.

Conclusion:

SEM images consistently show jagged, angular particles. The vast majority of particles are under 50μ m in their longest dimension. A typical SEM image is shown in Figure 1. A total of 251 particles were measured in this image, using Feret's statistical diameters. The most frequent particle size was a Feret's diameter between 2.5 and 5.0 μ m. Only two particles with Feret's diameters greater than 30μ m are present in this image.



The average particle size is significantly smaller than 50μ m. The population distribution of Feret's diameters is illustrated by the histogram in Figure 2. Based on 251 Feret's diameter measurements, the statistics of the boron carbide particles are as follows:

Statistic	Value (in μ m)
Average	6.64
Minimum	0.7
Maximum	34.0
Standard Deviation	4.81

Figures:



Figure 1.

This 248x SEM image shows a typical field of boron carbide particles on the polycarbonate Millipore filter substrate. The 1.2μ m pores in the filter material appear as dark dots on the background. No particle in this image is larger than 50μ m in any dimension.

Figure 2.

This is a histogram of the particle sizes measured (as horizontal Feret's diameters) in the SEM micrograph in Figure 1. Each column represents the number of particles in a particular "bin" of diameters.

The first column represents the number of particles having Feret's diameters between zero and 2.5μ m, the second column represents particles between 2.5μ m and 5.0μ m, etc. Most bins are 2.5μ m "wide". Wider bins were used at the high end, all of which are empty. The largest Feret's diameter measured in the image in Figure 1 was 34.0μ m.

Prepared By:

Sam Scheinman Physicist NACE C.T.

"IMPORTANT NOTICE: It is the policy of MATCO Associates that samples submitted as part of contracted investigations are the responsibility of MA TCO for only one month after final reports on those samples have been issued. They may then be discarded or otherwise disposed of. If you would like samples returned or safeguarded for longer than one month, please make such arrangements with this office in writing (include shipping provider and account number). If the submitted samples are part of a claim or potential lawsuit it is the client's responsibility to make arrangements to have the samples returned. Any testing not performed in MATCO's facility has been performed by established laboratories used by MATCO Associates. MATCO's policy is to submit partial invoices during the progress of a project, especially if the project has a longer time line. These partial invoices will usually be submitted along with interim reports, or as major steps are taken toward completion of the project. These partial invoices are necessary to support the on-going expenses of major projects. Clients are welcome to solve the client's problem, as mutually understood by the client and MATCO. Enclosure 2 to TN E-29650

Matco Services Inc. Report, Determination of B₄C Particle Sizes in BORAL[®] Neutron Absorber Material – Report #2, New Measurements Using Laser Light Scattering, Revision 2, July 1, 2010, Project 910-90193

July 1, 2010

Report of: Determination of B₄C Particle Sizes in Boral Neutron Absorber Material – Report #2, New Measurements Using Laser Light Scattering: Rev. 2 – 7/1/2010: NIST traceable standard data, updated mass statistics

- Report to: Peter Quinlan Transnuclear 7135 Minstrel Way, Suite 300 Columbia, MD 21045
- Phone:
 410-910-6895
 Fax:
 410-910-6902

 E-mail:
 peter.quinlan.ext@transnuclear.com
 Fax:
 410-910-6902

Summary:

Boral neutron absorber material consists of a layer of boron carbide (B₄C) particles in an aluminum matrix, sandwiched between two sheets of aluminum. The manufacturer's product description gives the average boron carbide particle size as 50μ m in the finished product. Transnuclear has requested that Matco attempt to quantify the average size of the boron carbide particles in the finished Boral material.

A particle size population distribution was measured previously by image analysis from scanning electron microscope (SEM) micrographs of one sample of Boral material. The average particle size indicated in that survey was significantly less than 50μ m.

New data have been compiled for 11 samples (3 from each of three large Boral sheets, and one sample from each of two smaller coupons received from Ceradyne). The new measurements were made using a Horiba LA-920 Laser Scattering Particle Size Distribution Analyzer. Average particle size (by diameter) was 31.7298 μ m and the average particle size (by mass fraction) was 55.8320 μ m.

Procedure:

In order to determine the particle size, the boron carbide particles were first isolated by dissolving away the aluminum matrix in a 50% HCl solution. Once the B_4C powder settled out, the spent acid was decanted off and the B_4C powder was rinsed with deionized water. The powder sample was washed with deionized water, allowed to settle, and decanted repeatedly until the rinse water pH measured greater than 3.

The particles were suspended by ultrasonic agitation in a recirculating deionized water

system for analysis by laser light scattering in a Horiba LA-920 Laser Scattering Particle Size Distribution Analyzer.

Results and Discussion:

SEM images (see previous report, dated 5/13/2010) consistently showed jagged, angular particles. The vast majority of particles were under 50μ m in their longest dimension. However, it was determined that using SEM image analysis was problematic for obtaining good statistics regarding the population distribution of larger particles. Laser light scattering does not have this limitation.

In the following charts, the population size distributions are given, as measured, for each sample processed, along with a corresponding mass fraction plot. The mass fractions were calculated from the particle size distribution data. The calculations assume that each size-frequency bin contains spherical particles of identical diameter and that the particles are homogeneous and of equal density. Particle volumes were calculated from particle diameters as spherical volumes using $V = \frac{4}{\pi} \left(\frac{d}{d}\right)^3$. The

calculated from particle diameters as spherical volumes, using $V = \frac{4}{3}\pi \left(\frac{d}{2}\right)^3$. The density was set to 1 g/µm³ for simplicity. This assumption concerning the density did not affect mass fraction calculations. The resulting dataset gives mass percent, per bin.

All samples analyzed exhibited strongly bimodal particle size distributions, with peaks often around 10μ m and 90μ m diameters. Mass fraction distributions were plotted on semi-log (log₁₀ X-axis) charts to match the presentation in the particle size distribution charts. Even with the semi-log presentation, the peak at smaller particle sizes ($\approx 10\mu$ m) vanishes in the mass fraction plots. Larger particles account for a majority of the mass.

About the statistical measures:

Since these distributions are bimodal, the statistical bin with the highest population is referred to as the bin having "maximum concentration" of particles, as opposed to calling it the "mode".

The average, median, and maximum concentration particle diameters are reported here, exactly as they were reported by the Horiba analyzer.

The corresponding "from mass fraction" median and maximum concentration statistics were computed using the mass fractions calculated for each particle size bin, under the assumption of identical, homogeneous, spherical particles. The median "from mass fraction" was found by linear interpolation of the cumulative mass distribution. The value reported is the particle diameter for which the cumulative mass distribution is equal to 50%. This is the particle diameter for which half the sample mass is present as smaller particles and half the sample mass is comprised of larger particles.

Project No. 910-90193 Page 3 of 17

The "average diameter, from mass fraction" is the arithmetic average volume diameter, $D_{3,0}$ in moment-ratio notation (ISO 9276-2) This is the diameter for which, if all particles in the sample had this diameter, the total sample mass would be unchanged.

Prepared By:

Sam Scheinman Physicist NACE C.T.

Reviewed by:

Martin Latona Senior Materials Engineer

"IMPORTANT NOTICE: It is the policy of MATCO Services, Inc. that samples submitted as part of contracted investigations are the responsibility of MATCO for only one month after final reports on those samples have been issued. They may then be discarded or otherwise disposed of. If you would like samples returned or safeguarded for longer than one month, please make such arrangements with this office in writing (include shipping provider and account number). If the submitted samples are part of a claim or potential lawsuit it is the client's responsibility to make arrangements to have the samples returned. Any testing not performed in MATCO's facility has been performed by established laboratories used by MATCO Services. MATCO's policy is to submit partial invoices during the progress of a project, especially if the project has a longer time line. These partial invoices will usually be submitted along with interim reports, or as major steps are taken toward completion of the project. These partial invoices are necessary to support the on-going expenses of major projects. Clients are welcome to request interim reports during the progress of major and longer projects. MATCO's intention is to do all that is necessary to solve the client's problem, as mutually understood by the client and MATCO Services, Inc.

4640 Campbells Run Road · Pittsburgh, PA 15205 · Tel: (412) 788-1263 · Fax: (412) 788-1283

Table 1.	Average,	median,	and	maximum	concentration	statistics;	by	particle	size f	requenc	y and b	y mass	fractio	n.
	· · · · · · · · · · · · · · · · · · ·						~ ,		0.20		,	·		•

			Diar	neter from pa frequencies	irticle size (µm):	Diamete	r from mass f	raction (µm):
Sample ID	Location	Matco Specimen ID	Average	Median	Max. Concentration	Average	Median	Max. Concentration
	Left	OL	20.4093	13.8547	12.3513	34.1058	51.3347	58.9530
MF 710156-2	Center	OC	34.7903	19.2151	83.5045	62.4415	94.9741	101.4600
	Right	OR	45.8319	24.7561	136.6400	84.7317	131.9809	152.4530
	Left	SL	35.2775	20.7216	82.8503	61.2040	91.0343	101.4600
SI 510179-1	Center	SC	24.9971	17.2122	12.3698	42.2738	65.7971	77.3390
	Right	SR	43.8468	29.2708	93.4127	73.4682	107.2165	116.2100
	Left	HL	32.0951	17.2474	82.7051	59.9412	94.8097	101.4600
HD 410060-2	Center	HC	26.7247	16.7792	61.7998	44.8990	64.0493	67.5230
	Right	HR	31.5870	18.2126	72.2190	56.3755	85.5088	88.5830
Z1035-1030- 07-02-1		C1	34.5496	21.3064	72.0963	58.6536	83.9613	88.5830
Z1035-1030- 07-02-2		C2	18.9189	11.4866	12.3100	36.0581	60.9502	67.5230
Average for all Samples	All	All samples	31.7298	19.0966	65.6599	55.8320	84.6924	92.8679
NIST traceable Particle Standard Whitehouse Scientific Catalog: PS 213 Bottle No.: 02411-02420			42.8409	38.3011	36.5604	58.7244	80.3240	67.5230

The mass fraction median was found by linear interpolation of the cumulative mass distribution, to find the particle size at which the cumulative mass distribution was equal to 50%. For this value of the diameter, 50% of the sample mass is present as smaller particles and 50% as larger particles. Mass distribution data were calculated from the size distribution data.

Sample OL, taken from the left side of panel MF 710156-2:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	20.4093	34.1058
Median (µm)	13.8547	51.3347
Max. Concentration (µm)	12.3513	58.9530

Table 2. Average, median, and max. statistics, by diameter and mass, for Sample OL.

Figure 1. Sample OL particle size distribution. The red line is the cumulative size distribution (right hand axis).

<u>Figure 2</u>. Sample OL mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Sample OC, taken from the center of panel MF 710156-2:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	34.7903	62.4415
Median (µm)	19.2151	94.9741
Max. Concentration (µm)	83.5045	101.4600

Table 3. Average, median, and max. statistics, by diameter and mass, for Sample OC.

Figure 3. Sample OC particle size distribution. The red line is the cumulative size distribution (right hand axis).

<u>Figure 4</u>. Sample OC mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Sample OR, taken from the right side of panel MF 710156-2:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	45.8319	84.7317
Median (µm)	24.7561	131.9809
Max. Concentration (µm)	136.6400	152.4530

Table 4. Average, median, and max. statistics, by diameter and mass, for Sample OR.

<u>Figure 5</u>. Sample OR particle size distribution. The red line is the cumulative size distribution (right hand axis).

Sample SL, taken from the left side of panel SI 510179-1:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	35.2775	61.2040
Median (µm)	20.7216	91.0343
Max. Concentration (µm)	82.8503	101.4600

Table 5. Average, median, and max. statistics, by diameter and mass, for Sample SL.

Figure 7. Sample SL particle size distribution. The red line is the cumulative size distribution (right hand axis).

Figure 8. Sample SL mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Project No. 910-90193 Page 9 of 17

Sample SC, taken from the center of panel SI 510179-1:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	24.9971	42.2738
Median (µm)	17.2122	65.7971
Max. Concentration (µm)	12.3698	77.3390

<u>Table 6</u>. Average, median, and max. statistics, by diameter and mass, for Sample SC.

Figure 10. Sample SC mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Sample SR, taken from the right side of panel SI 510179-1:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	43.8468	73.4682
Median (µm)	29.2708	107.2165
Max. Concentration (µm)	93.4127	116.2100

Table 7. Average, median, and max. statistics, by diameter and mass, for Sample SR.

Figure 11. Sample SR particle size distribution. The red line is the cumulative size distribution (right hand axis).

Figure 12. Sample SR mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Sample HL, taken from the left side of panel HD 410060-2:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	32.0951	59.9412
Median (µm)	17.2474	94.8097
Max. Concentration (µm)	82.7051	101.4600

Table 8. Average, median, and max. statistics, by diameter and mass, for Sample HL.

<u>Figure 13</u>. Sample HL particle size distribution. The red line is the cumulative size distribution (right hand axis).

Figure 14. Sample HL mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Sample HC, taken from the center of panel HD 410060-2:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	26.7247	44.8990
Median (µm)	16.7792	64.0493
Max. Concentration (µm)	61.7998	67.5230

Table 9. Average, median, and max. statistics, by diameter and mass, for Sample HC.

2

Sample HR, taken from the right side of panel HD 410060-2:

Statistic	From particle size frequencies:	From mass fraction:
Average diameter (µm)	31.5870	56.3755
Median (µm)	18.2126	85.5088
Max. Concentration (µm)	72.2190	88.5830

Table 10. Average, median, and max. statistics, by diameter and mass, for Sample HR.

Figure 17. Sample HR particle size distribution. The red line is the cumulative size distribution (right hand axis).

4640 Campbells Run Road · Pittsburgh, PA 15205 · Tel: (412) 788-1263 · Fax: (412) 788-1283

Sample C1, taken from Ceradyne coupon Z1035-1030-07-02-1:

Statistic	From particle size frequencies:	From mass fraction: 58.6536	
Average diameter (µm)	34.5496		
Median (µm)	21.3064	83.9613	
Max. Concentration (µm)	72.0963	88.5830	

Table 11. Average, median, and max. statistics, by diameter and mass, for Sample C1.

Sample C1 mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Sample C2, taken from Ceradyne coupon Z1035-1030-07-02-2:

Statistic	From particle size frequencies:	From mass fraction: 36.0581	
Average diameter (µm)	18.9189		
Median (µm)	11.4866	60.9502	
Max. Concentration (µm)	12.3100	67.5230	

Table 12. Average, median, and max. statistics, by diameter and mass, for Sample C2.

Figure 21. Sample C2 particle size distribution. The red line is the cumulative size distribution (right hand axis).

Figure 22. Sample C2 mass distribution, as calculated from particle size distribution data. Red line is the cumulative mass distribution (right hand axis).

Sample QC; Quality Control test of NIST Traceable Polydisperse Particle Standard. Whitehouse Scientific Catalog No: PS 213. Bottle No: 02411-02420

Statistic	From particle size frequencies:	From mass fraction:	
Average diameter (µm)	42.8409	58.7244	
Median (µm)	38.3011	80.3240	
Max. Concentration (µm)	36.5604	67.5230	

Table 13. Average, median, and mode statistics, by diameter and mass, from QC test.

Figure 24. NIST traceable particle size standard mass distribution, as calculated from particle size distribution data. The red line is the cumulative mass distribution (right hand axis).

Analysis of Whitehouse Scientific 10-100µm Polydisperse Particle Standard: Percentage Undersize

Figure 25. "Graphical Review" from the Whitehouse Scientific Certificate of Analysis.

Cumulative % undersize	10%	25%	50%	75%	90%
Final average dia. (µm)	25.37	31.68	41.26	52.62	62.93
95% confidence (µm)	±2.08	±3.00	±3.26	±2.88	±3.08

Cumulative % undersize	10%	25%	50%	75%	90%
Diameter (μm)	24.475	30.215	38.351	50.332	66.249

<u>Table 15</u>. Particle diameter at five points on the cumulative population distribution, found by linear interpolation of the measured size distribution.