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Simulants for Testing the Blend Can Loading System

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April 2021

SRNL-STI-2021-00205, Revision 0

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EXECUTIVE SUMMARY

Eleven simulants have been provided to the Savannah River National Laboratory (SRNL) Mechanical Systems & Custom Equipment Development (MSCED) organization to further test the capabilities of the Blend Can Loading System (BCLS). The basis for the materials used in these simulants are based on SRNL-STI-2020-00503, "Simulant Recommendation for the Blend Can Loading System" (Ref. i). The 11 different simulants provided to MSCED are summarized in Table ES- 1. This table provides a basis of why they were selected and what properties were specifically targeted, given the composition of the procured materials.

Table ES- 1 Summary of Simulants Provided to MSCED

Simulant	Basis
1A	Highest density material, tungsten carbide. PSD of the powder matching that of the nominal ARIES distribution. Oversized PSD matching up with the unprocessed ARIES distribution. Nominal wt. % distribution between the under and oversized ARIES distribution.
1B	Highest density material, tungsten carbide. PSD of the powder matching that of the greatest fraction of Modes 1 and 2 of the ARIES distribution. Oversized PSD matching up with the unprocessed upset ARIES/furnace distribution. Nominal wt.% distribution between the under and oversized ARIES/furnace distribution was targeted.
1C	Most representative density material, molybdenum for undersized ARIES distribution, targeting the volumetric fraction of Modes 1 and 2. Oversized material was stainless steel targeting the ARIES/furnace distribution. Maintained the same volumetric distribution if density were the same, hence the mass of molybdenum is greater than that of the stainless steel.
2A	Highest density material, tungsten carbide. PSD of the powder matching that of the nominal ARIES distribution.
2B	Highest density material, tungsten carbide. PSD of the powder matching that of the greatest fraction of Modes 1 and 2 of the ARIES distribution.
2C	Most representative density material, molybdenum for undersized ARIES distribution, targeting the volumetric fraction of Modes 1 and 2.
3A	Made with batches of stainless steel powders blended with various combinations and concentrations of salts having varying melting points. Granular and patty-cake forms were made. Typical calcine temperature of 900 °C with one batch at 600 °C. Fifteen different batches were used to make up this simulant.
3B	Made with a single blend of stainless steel powders and salts in granular and patty-cake forms. Low melting salt was selected to provide strong bonding of the stainless steel and salts when calcined at 900 °C.
4A	Most representative density material, molybdenum. Not chemically similar. Targeting %RF and mode 2 particle size respectively. Blend was targeted to provide the %RF.
4B	Chemically similar processed material, cerium oxide. Targeting mode 2 particle size and %RF respectively. No blending with other materials.
5A	Chemically similar processed material, cerium oxide. %RF range is the target, respectively, PSD as is. No blending with other materials.

The simulant profile at 16% RH is provided in Table ES- 2. The only properties that could differ at different relative humidity conditions are the bulk and tap densities, the other physical properties are constant. The 16% RH data is provided since this is how the simulants were delivered to MSCED and once exposed to a

different RH environment, the properties could differ. The bulk/tap densities were obtained using a 250 mL graduated cylinder.

Table ES- 2 Simulant Profile at 16% RH

Process category	Simulant Number and Subcategory	Particle Density (g/cm^3)	Mode 1 (µm)	Mode 2 (µm)	Mode 3 (µm)	% RF	Bulk Density (g/cm^3)	Tap Density (g/cm^3)	Carr's Index (%)	SSA (m^2/g)
DMO	1A Unprocessed ARIES	13.60	3	20.2	62.2	13.9	7.56	8.01	5.6	N/M
DMO	1B Unprocessed ARIES	14.45	3	20.2	62.2	17.4	8.07	9.06	10.9	N/M
DMO	1C Unprocessed ARIES	10.08	7.1		28.5	14.2	2.58	2.96	13.0	N/M
DMO	2A Processed ARIES	15.60	3	20.2	62.2	13.9	7.12	7.72	7.7	0.16
DMO	2B Processed ARIES	15.53	3	20.2	62.2	17.4	6.50	7.08	8.30	0.23
DMO	2C Processed ARIES	10.43	3.57	14.3	37	20.4	1.88	2.28	17.4	0.39
Pyro-chemical	3A Pyro-chemical worst case clumps	N/A					1.47	N/A		
Pyro-chemical	3B Pyro-chemical worst case clumps	N/A					1.17	N/A		
Aqueous	#4A Aq. Worst case flowability	9.94	-	10.1	-	9.0	1.71	2.00	14.3	0.50
Aqueous	#4B Aq. Worst case flowability	5.94	-	8.7	-	21.0	0.71	1.00	29.4	N/M
Aqueous	#5A Aq. Worst case RF	7.15	-	-	-	85.1	1.12	1.35	17.0	3.90

N/M = Not Measured

The physical characteristics and makeup of the 11 simulants are summarized in the data sheets shown in Table ES- 3 through Table ES- 13 and are for simulants 1, 2, 3, 4 and 5. The bulk/tap density of the batched material is for 16% RH using a 250 mL graduated cylinder. The reported bulk/tap densities of the powders used to make the simulant are obtained at room conditions (e.g. uncontrolled environment).

Details of how simulant 3 was made are provided in section 3.3 of this document and are one time use simulants, given they can be size reduced in the BCLS process and if so, their characteristics will be different. Simulant 2 can be modified with oversized materials to provide additional simulant 1 compositions, if needed.

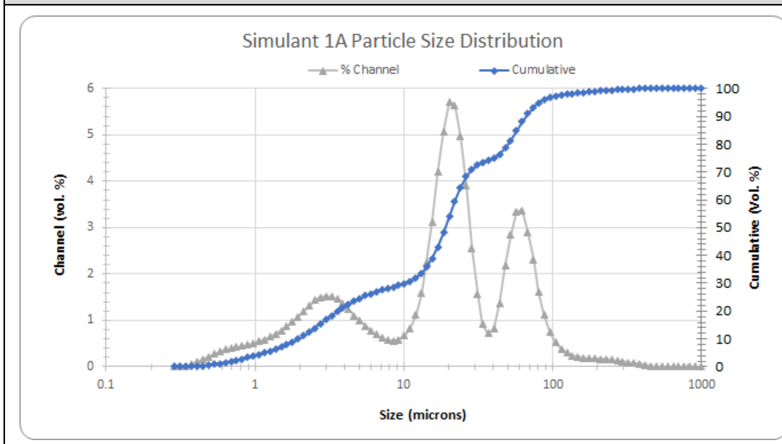
The bulk density and tap density were obtained at two different RH, approximately 16% and 62% and the temperature was maintained around 70 ^{o}F. The %CI was calculated from these densities. Additionally, 100 mL and 250 mL graduated cylinders were used to obtain the bulk/tap densities, primarily due to Simulant 2C did not have sufficient material to obtain data using the 250 mL cylinder. The averages and standard deviation are provided in Table 3-5 and Table 3-6 for the 250 and 100 mL graduated cylinders respectively. The general trend was as the %RH went from 16% to 62%, the %CI increased, indicating the material might be less flowable as the %RH increases. Furthermore, the 100 mL data %CI are typically larger than that of the 250 mL data, such cannot be explained. Simulant 4B is cerium oxide and its true density is lower than other cerium oxides that have been characterized. This could be due to trapped gas when the agglomerates were formed.

The recommendation for simulant 6 is to use the existing brown fused aluminum oxide MSCED has on hand for size reduction efforts. Additional quantities of simulants similar to simulant 3 A/B characteristics can be made to support the size reduction effort.

Table ES- 3 Simulant 1A: Unprocessed ARIES – Average Distribution Tungsten Carbide

Temperature (°F)	72	Percent Relative Humidity			16
Mass (g)	Volume (mL)	Density (g/cc)			%CI
		Particle	Bulk	Tap	
5000	710	13.60	7.56	8.01	5.6
Photograph					

Distribution of Solids	Mesh	Wt%
Buffalo Tungsten 900-0820	10 (2000 µm)	2.1
Buffalo Tungsten 900-0820	20 (850 µm)	29.6
Buffalo Tungsten 903-3060	40 (425 µm)	26.2
Buffalo Tungsten 903-3060	50 (300 µm)	13.4
Buffalo Tungsten 903-3060	60 (250 µm)	1.3
Buffalo Tungsten 903-3060	70 (212 µm)	3.6
Undersized Material – See Below		23.8

Particle size distribution – Unsized Material		Mode		
		1	2	3
	Peak (mm)	3.0	20.2	62.2
	Range (mm)	0.4 – 4.0	4.0 - 30	30 – 497
	Vol. %	22.5	50.0	27.5
	Respirable Fraction (%)			
	13.9			

Vendor	Material	Mass fraction	Density (g/cc)			%CI
			Particle	Bulk	Tap	
Buffalo Tungsten	WCI-1107	0.166	15.59	3.31	4.17	20.6
Buffalo Tungsten	WCIV-648	0.500	15.60	7.06	8.03	13.8
Buffalo Tungsten	WCVI-1256	0.334	15.57	7.40	8.08	8.4

Table ES- 4 Simulant 1B:Unprocessed Upset/Muffle Furnace ARIES – Max Mode 1&2 Tungsten Carbide

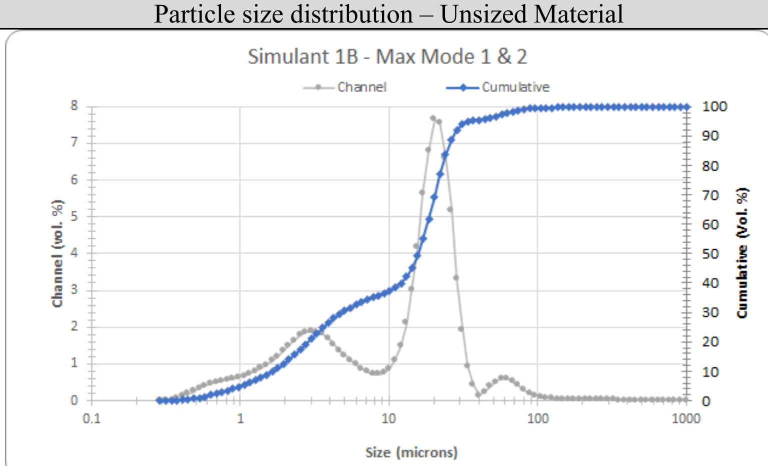
Temperature (°F)	72	Percent Relative Humidity			16
Mass (g)	Volume (mL)	Density (g/cc)			%CI
		Particle	Bulk	Tap	
5000	617	14.45	8.07	9.06	10.9

Photograph

Distribution of Solids

Buffalo Tungsten	WCZ917-2040	WCZ926-4080	Mesh	Wt%
	X		30 (600 µm)	7.74
	X		35 (500 µm)	8.11
	X	X	40 (425 µm)	5.90
	X	X	50 (300 µm)	10.25
		X	60 (250 µm)	3.31
		X	70 (212 µm)	5.96
		X	80 (180 µm)	6.01
		X	<80 (180 µm)	1.01
Undersized Material – See Below				51.71

Particle size distribution – Unsized Material



	Mode 1	Mode 2	Mode 3
Peak (mm)	3.0	20.2	62.2
Range (mm)	0.4 – 4.0	4.0 - 30	30 – 497
Vol. %	28	66	6
Respirable Fraction (%)	17.4		

Vendor	Material	Mass fraction	Density (g/cc) Particle	Density (g/cc) Bulk	Density (g/cc) Tap	%CI
Buffalo Tungsten	WCI-1107	0.379	15.59	3.31	4.17	20.6
Buffalo Tungsten	WCIV-648	0.573	15.60	7.06	8.03	13.8
Buffalo Tungsten	WCVI-1256	0.048	15.57	7.40	8.08	8.4

Table ES- 5 Simulant 1C: Unprocessed Upset/Muffle Furnace ARIES– Max Mode 1&2 Moly & S/S

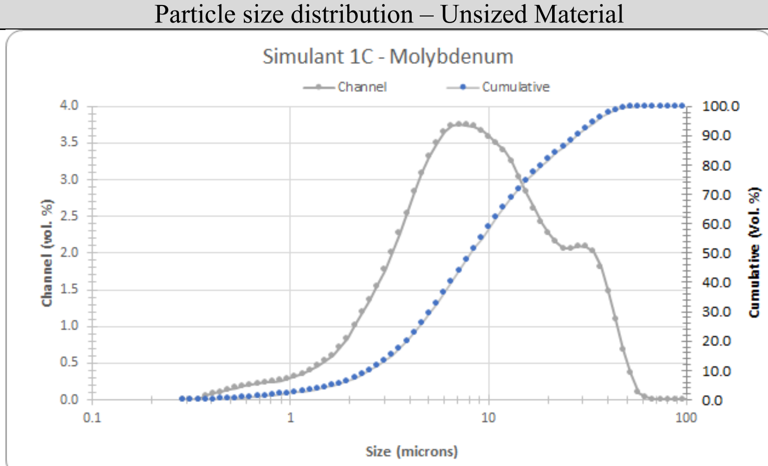
Temperature (°F)	72	Percent Relative Humidity			16
Mass (g)	Volume (mL)	Density (g/cc)			%CI
		Particle	Bulk	Tap	
4384	1700	10.08	2.58	2.96	13.0

Photograph

Distribution of Solids

VULKAN STAINLESS STEEL GRIT	GRT-40	GRT-30	30 (600 µm)	6.24
	X		35 (500 µm)	4.94
	X		40 (425 µm)	4.41
		X	50 (300 µm)	15.15
		X	60 (250 µm)	5.00
		X	70 (212 µm)	2.17
		X	80 (212 µm) (pan)	2.97
Undersized Material – See Below				59.12

Particle size distribution – Unsized Material

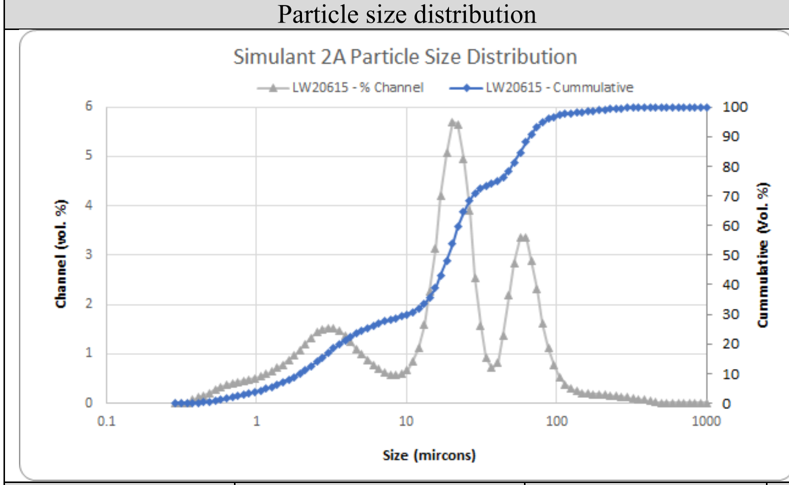


	Mode 1	Mode 2	Mode 3
Peak (mm)	7.1		28.5
Range (mm)	0.4 – 30		30 – 352
Vol. %	93.1		6.9
Respirable Fraction (%)	14.2		

Vendor	Material	Mass fraction	Density (g/cc)			%CI
			Particle	Bulk	Tap	
Atlantic Equipment Engineers	Molybdenum – 325 Mesh (2004514)	0.349	10.11	2.87	3.23	11.21
EdgeTech	Molybdenum - 200 Mesh (977-210128)	0.651	10.06	1.69	2.40	29.47

Table ES- 6 Simulant 2A: Processed ARIES – Average Distribution Tungsten Carbide

Temperature (°F)	72	Percent Relative Humidity			16	
Mass (g)	Volume (mL)	Density (g/cc)			%CI	SSA (m^2/g)
		Particle	Bulk	Tap		
5000	710	15.60	7.12	7.72	7.7	0.16
Photograph						

Particle size distribution		Mode		
		1	2	3
	Peak (µm)	3.0	20.2	62.2
	Range (µm)	0.4 – 4.0	4.0 - 30	30 – 497
	Vol. %	22.5	50.0	27.5
	Respirable Fraction (%)			
	13.9			

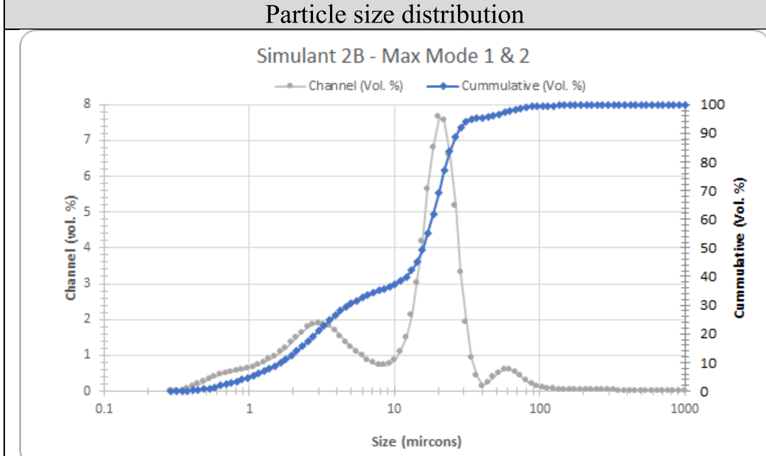
Vendor	Material	Mass fraction	Density (g/cc)			%CI
			Particle	Bulk	Tap	
Buffalo Tungsten	WCI-1107	0.166	15.59	3.31	4.17	20.6
Buffalo Tungsten	WCIV-648	0.500	15.60	7.06	8.03	13.8
Buffalo Tungsten	WCVI-1256	0.334	15.57	7.40	8.08	8.4

Table ES- 7 Simulant 2B: Processed ARIES – Max Mode 1&2 Tungsten Carbide

Temperature (°F)	72	Percent Relative Humidity			16	
Mass (g)	Volume (mL)	Density (g/cc)			%CI	SSA (m^2/g)
		Particle	Bulk	Tap		
5000	782	15.53	6.50	7.08	8.3	0.23

Photograph

Particle size distribution



	Mode		
	1	2	3
Peak (μm)	3.0	20.2	62.2
Range (μm)	0.4 – 4.0	4.0 - 30	30 – 497
Vol. %	28	66	6

Respirable Fraction (%)
17.4

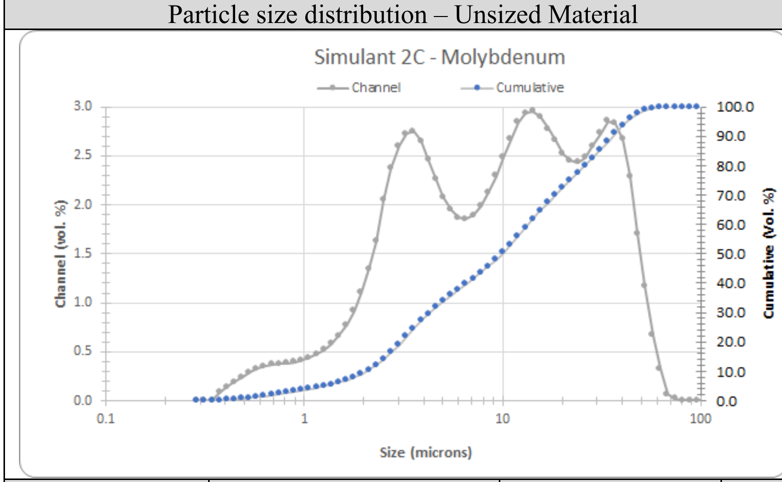
Vendor	Material	Mass fraction	Density (g/cc)			%CI
			Particle	Bulk	Tap	
Buffalo Tungsten	WCI-1107	0.379	15.59	3.31	4.17	20.6
Buffalo Tungsten	WCIV-648	0.573	15.60	7.06	8.03	13.8
Buffalo Tungsten	WCVI-1256	0.048	15.57	7.40	8.08	8.4

Table ES- 8 Simulant 2C: Processed ARIES – Max Mode 1&2 Molybdenum

Temperature (ºF)	72	Percent Relative Humidity			16	
Mass (g)	Volume (mL)	Density (g/cc)			%CI	SSA (m^2/g)
		Particle	Bulk	Tap		
3625	1700	10.43	1.88	2.28	17.4	0.39

Photograph

Particle size distribution – Unsized Material



	Mode		
	1	2	3
Peak (µm)	3.57	14.3	37
Range (µm)	0.4 – 4.0	4.0 - 30	30 – 352
Vol. %	28	58.3	13.7
Respirable Fraction (%)			
20.4			

Vendor	Material	Mass fraction	Density (g/cc)			%CI
			Particle	Bulk	Tap	
Atlantic Equipment Engineers	Molybdenum – 325 Mesh (2012516)	0.349	10.17	2.87	3.23	11.21
EdgeTech	Molybdenum - 200 Mesh (977-210128)	0.651	10.06	1.69	2.40	29.47

Table ES- 9 Simulant 3A: Pyrochemical – Variable Salt Batches Compositions

Temperature (ºF)	72	Percent Relative Humidity			16
Mass (g)	Volume (mL)	Density (g/cc)			%CI
		Particle	Bulk	Tap	
2491	1700$	N/M	1.47	N/M	N/M
Photograph					

Vendor	Material	Mass fraction	Particle Density (g/cc)
Sandvik Osprey Powders	316L stainless steel	0.489	7.97
Atomising Systems Limited	316B stainless steel	0.319	7.79
Blue Line	Cerium Oxide	0.046	7.15
Alfa Aesar	Calcium Fluoride	0.040	3.18*
Fisher Scientific	Calcium Chloride	0.038	2.15*
Alfa Aesar	Titanium Oxide	0.031	4.23*
Sigma-Aldrich, Fisher Scientific	Magnesium Chloride	0.023	2.32*
Fisher Scientific	Sodium Chloride	0.014	2.16*
Respirable Fraction (%)			Not calculated, no particle size data, though somewhat dusty

$ Filled to the 1.7 liter mark, measured mass and calculated density
* Literature values

Table ES- 10Simulant 3B: Pyrochemical – Single Salt Batch Composition

Temperature (°F)	72	Percent Relative Humidity			16
Mass (g)	Volume (mL)	Density (g/cc)			%CI
		Particle	Bulk	Tap	
1949	1700$	N/M	1.15	N/M	N/M
Photograph					

Vendor	Material	Mass fraction	Particle Density (g/cc)
Sandvik Osprey Powders	316L stainless steel	0.33	7.97
Atomising Systems Limited	316B stainless steel	0.28	7.79
Fisher Scientific	Calcium Chloride	0.10	2.15*
Alfa Aesar	Titanium Oxide	0.03	4.23*
Sigma-Aldrich, Fisher Scientific	Magnesium Chloride	0.07	2.32*
Fisher Scientific	Sodium Chloride	0.19	2.16*
Respirable Fraction (%)			Not calculated, no particle size data, not too dusty.

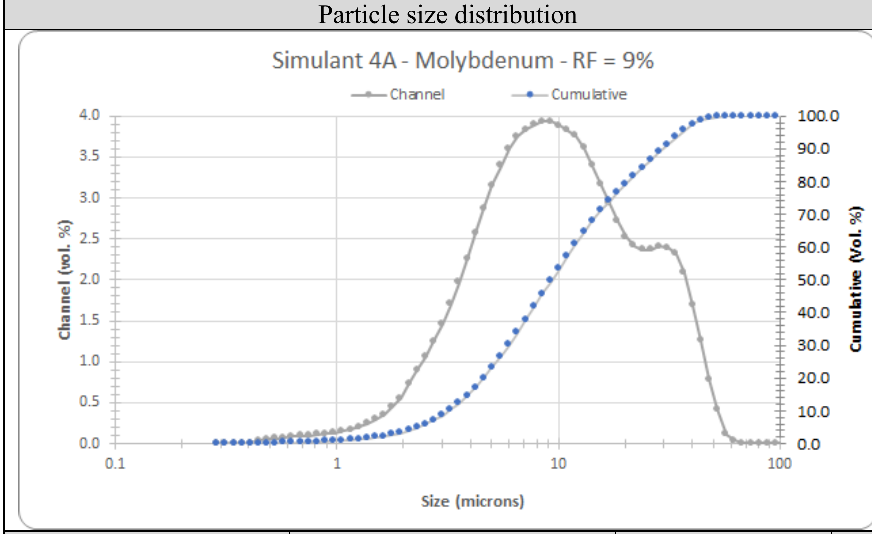
$ Filled to the 1.7 liter mark, measured mass and calculated density
* Literature values

Table ES- 11 Simulant 4A: Aqueous Processing High Density RF 9% – Molybdenum

Temperature (ºF)	72	Percent Relative Humidity			16	
Mass (g)	Volume (mL)	Density (g/cc)			%CI	SSA (m^2/g)
		Particle	Bulk	Tap		
2912	1700	9.94	1.68	2.05	18.1	0.50

Photograph

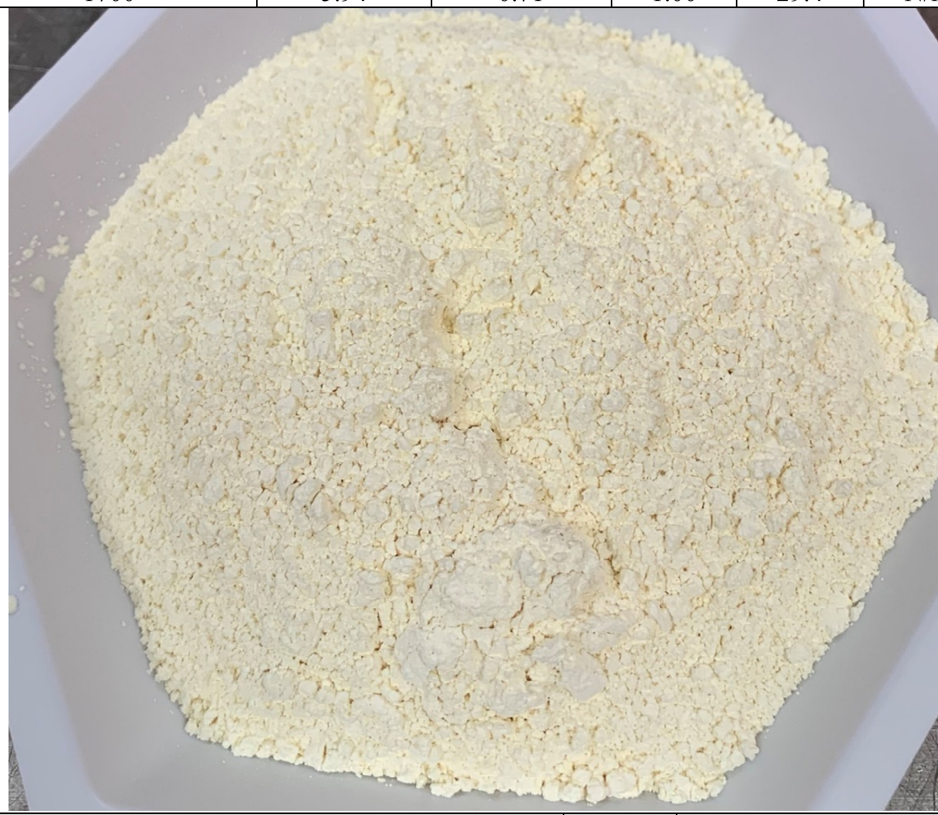
Particle size distribution

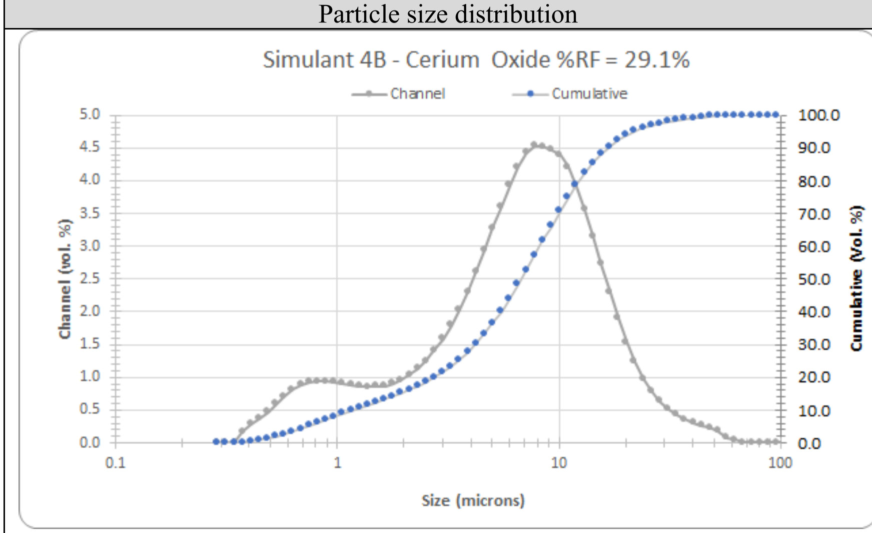


	Mode 2 (microns)
D50 (from PSD curve)	10.1
Respirable Fraction (%)	
9.0	

Vendor	Material Name Lot Number	Mass fraction	Density (g/cc)			%CI
			Particle	Bulk	Tap	
Edgetech	2-3 µm 977-210129	0.816	10.11	1.54	2.07	25.5
Atlantic Equipment Engineers	1-5 mircons 2012501	0.184	10.19	2.81	3.32	15.5

Table ES- 12 Simulant 4B: Aqueous Processing – Similar Processing Low %RF Cerium Oxide

Temperature (ºF)	72	Percent Relative Humidity			16	
Mass (g)	Volume (mL)	Density (g/cc)			%CI	SSA (m^2/g)
		Particle	Bulk	Tap		
1241	1700	5.94	0.71	1.00	29.4	N/M
Photograph						

Particle size distribution		Mode 2 (microns)
	D50 (from PSD curve)	Mean = 8.4 50% = 6.7
	Respirable Fraction (%)	
	29.1	

From Vendor – SRNL analysis to follow

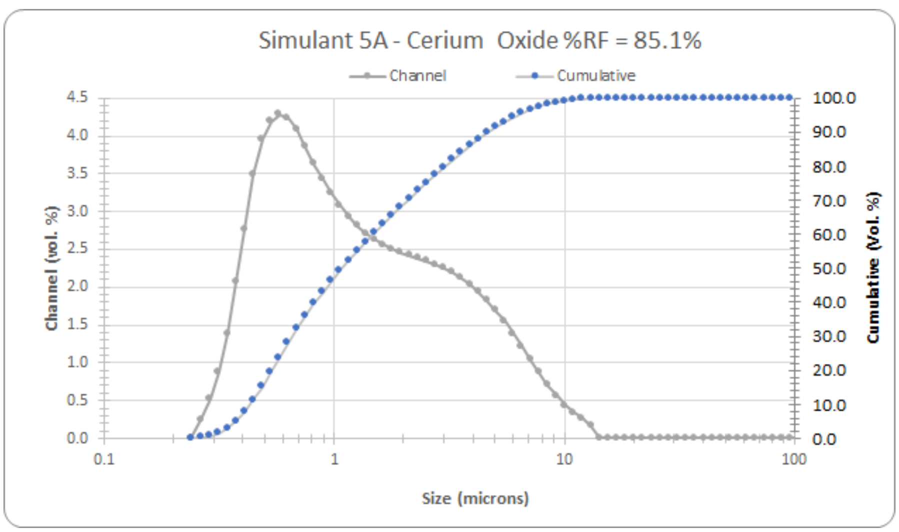
Vendor	Material Name Lot Number	Mass fraction	Density (g/cc)			%CI
			Particle	Bulk	Tap	
Blue Line	Cerium Oxide 8.2 µm D30593	1.0	TBD	0.73	0.97	24.8

Table ES- 13 Simulant 5A: Aqueous Processing – Similar Processing High %RF Cerium Oxide

Temperature (°F)	72	Percent Relative Humidity			16	
Mass (g)	Volume (mL)	Density (g/cc)			%CI	SSA (m^2/g)
		Particle	Bulk	Tap		
2163.0	1700*	7.15	1.12	1.35	17.0	3.90

Photograph

Particle size distribution – Unsized Material	Respirable Fraction (%)
(chart below)	85.1



Vendor	Material	Density (g/cc)			%CI
		Particle	Bulk	Tap	
Atlantic Equipment Engineers	CE-602 (obtained from MSCED)	7.15	1.13	1.55	27.1

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LIST OF ABBREVIATIONS

ASAP	Accelerated Surface Area and Pore Analyzer
ARIES	Advanced Recovery and Integrated Extraction System
BET	Brunauer-Emmett-Teller
BCLS	Blend Can Loading System
CI	Carr's Index
DI	Deionized
DMO	Direct Metal Oxidation
MSCED	Mechanical Systems & Custom Equipment Development
NIST	National Institute of Standards and Technology
N/M	Not Measured
PSD	Particle Size Distribution
RH	Relative Humidity
RF	Respirable Fraction
SEM	Scanning Electron Microscope
SRNL	Savannah River National Laboratory
SSA	Specific Surface Area
Wt. %	Weight Percent
Vol. %	Volume Percent

1.0 Introduction

This report is to provide the physical characterization [true density, bulk density, tap density, percent Carr's index (%CI)], and particle size analysis and photos of the material that were obtained to provide the simulants recommended in SRNL-STI-2020-00503 [Ref. i] to support testing of the BCLS. The list of materials recommended for the different simulants provides are provided in Table 1-1 Details of the recommended materials are provided in Reference i.

Table 1-1. Material Recommended to Support

Simulant Profile	Recommended Materials
1	Stainless steel powder Molybdenum powder Tungsten carbide powder Stainless steel grit Tungsten carbide grit
2	Stainless steel powder Molybdenum powder Tungsten carbide powder Copper tungsten carbide powder
3	Stainless steel powder Salts
4	Cerium oxide
5	Cerium oxide
6	Ruby Sapphire Brown fused aluminum oxide

The batched simulants from these materials to support the various processes in Reference i are described in this report and their physical properties are either calculated or measured. The simulant profiles of the Pu-oxide processes are provided in Table 1-2. The objective of the batched simulants are to satisfy as close as possible the requirements in as shown in Table 1-2 and as discussed in Reference 1 given the availability of materials that can be procured and characterized to support the simulant profiles.

Table 1-2. Simulant Profiles for Pu-Oxide from Various Processes (Ref. ii)

Process category	Simulant Number and Subcategory	Particle Density (g/cm^3)	Mode 1 (µm)	Mode 2 (µm)	Mode 3 (µm)	%<RF of <180 (µm)	Wt% <180 (µm)	Wt% 180-1000 (µm)	Wt% 1000-2000 (µm)	Bulk Density (g/cm^3)	Tap Density (g/cm^3)	Carr's Index (%)	SSA (m^2/g)	Flow-ability
DMO	#1 unprocessed ARIES	11.56	1.2-2.8	16.2-18.5	48-63	11-22	36-73	23-64	0-15	4.2-5.0	4.9-6.3	9.5-22.3	0.24-0.52	Excellent to poor
DMO	#2 Processed ARIES	11.56	0.9-3.2	10.9-18.5	37-63	12-32	100	0	0	3.6-4.9	4.8-6.3	21-25.8	0.24-0.52	Fair-poor
Pyro-chemical	#3 Pyro-chemical worst case clumps	7.1	1.9	12.2	66	22.5	0-100	0-100	0-100	2.72	3.49	22.1	2-5	Poor
Aqueous	#4 Aq. Worst case flowability	3.0 - 12	n/a	9.7	n/a	9.0	100	0	0	<1.5	*	28-38	10-15	Cohesive very poor
Aqueous	#5 Aq. Worst case RF	4.6	1.8	9.0	n/a	78-95	100	0	0	<1.0	*	<23.1	<2.9	Poor

2.0 Experimental Procedure

2.1 Primary Simulant Characteristics

The primary characteristics targeted for the various simulants are provided in Table 2-1. This is based on the recommendations of materials for use given the various simulants in Reference i for the given process categories.

Table 2-1. Primary Characteristics of Simulants

Simulant	Process Category	Subcategory	Primary Characteristics
1	DMO	Unprocessed ARIES*	Modes of the powders Mass fraction distribution of solids Average composition Variation in particle density
2	DMO	Processed ARIES	Modes Variation in particle density
3	Pyro-Chemical	Worse Case Clumps	Granular Patty Cakes
4	Aqueous	Worst Flowable Case	Mean particle size % RF Variation in particle density Bulk Density Carr Index
5	Aqueous	Worst RF Case	Modes %RF

* ARIES = Advanced Recovery and Integrated Extraction System

2.2 Materials

The materials obtained for this task were based on reviewing suppliers who provided powders or grits (oversized materials). Suppliers were determined by reviewing the list on www.thomasnet.com for the material of interest. Suppliers who stated they provided powders or grits on their webpages were then accessed to determine if these powders/grit could potentially be used and readily available. If the vendor webpage did not provide any specifics on the material, they were not considered.

2.3 Characterization

The measurements and/or calculations that will be performed are:

- Bulk and tap densities,
- Carr's Index
- True (particle) density,
- Particle size distribution,
- Scanning electron microscope (SEM),
- Specific surface area (SSA).

The materials that were received were measured in laboratory conditions for bulk and tap densities. For the batched simulants, the bulk and tap densities were obtained in a glovebox where the relative humidity (RH) is controlled at two different RH levels.

2.3.1 Bulk and Tap Density and % CI

ASTM D7481 (Ref. iii) was used to determine the bulk and tap densities of the powders. Graduated cylinders (250 mL) were used in this activity. Additional 100 mL graduated cylinders were used to further assess the impact of using a smaller cylinder. The volume markings in the region of measurements were verified using deionized (DI) water at room temperature and if corrections are required, such was done after the measurements were completed. The cylinders were cleaned and dried with instrument air prior to use. In between measurements, dry paper towels were used to remove residue. If a different sample was to be used, the cylinders were cleaned with instrument air. The graduated cylinders were weighed and the results were recorded. The powder/grit sample were placed into a sealable plastic bag and a corner was cut to allow for the powder to flow out of the bag and into a funnel on top of a graduated cylinder. For the samples in the glovebox, 500 mL sample bottles were used rather than plastic bags. The funnel was moved side to side to load the powder as evenly as possible during the filling process and to fill the cylinders in a repeatable manner. The graduated cylinders were loaded between 150 to 250 mL, and the powder volume and total mass recorded. In the case where 100 mL graduated cylinders were used, they were filled between 70 to 100 mL. The recorded volume is to the smallest marking on the cylinder, which is 2 mL for the 250 mL graduated cylinder and 1 mL for the 100 mL graduated cylinder. The mass of the added sample is calculated using equation (1). The scale used was M&TE and had a reading to 0.1 grams. The bulk density was determined using equation (2). The graduated cylinder was secured to the tapping platform on the Varian tapper. The Varian tapper taps at 250 taps/minute with a fixed drop of 3 mm. The tapper was set to 500 taps, started, and upon completion the volume recorded. The tapper was then set to 750 taps and started and upon completion the volume recorded. If the volume change between the 500 taps and 750 taps is less than 2 volume percent (vol. %) of the 500 taps volume, then the measurement is complete. If not, the tapper was then set at 1250 taps and the volume recorded upon completion of the taps. If the volume between the 750 taps and 1250 taps is less than 2 vol. % of the 750 taps, then the measurement is complete. If not, the procedure would continue with 1250 taps until the 2 vol.% difference is satisfied. The tap density is calculated using equation (3) with the final measured volume. The %CI is calculated using equation (4). The powder flowability (Ref. iv) is based on %CI and can be estimated using Table 2-2 and is provided to the reader for reference.

$$m_{sample} = m_{sample+cylinder} - m_{cylinder} \tag{1}$$

$$\rho_{bulk} = \frac{m_{sample}}{V_{inital}} \tag{2}$$

$$\rho_{tap} = \frac{m_{sample}}{V_{final}} \tag{3}$$

$$CI = \left(1 - \frac{\rho_{bulk}}{\rho_{tap}}\right) \cdot 100\% \tag{4}$$

Where: ρ_{bulk} = bulk density (g/mL)
ρ_{tap} = tap density (g/mL)
CI = Carr’s Index (%)
$m_{cylinder}$ = mass of 250 mL cylinder (g)
$m_{sample+cylinder}$ = mass of powder added and 250 mL cylinder (g)
m_{sample} = mass of powder added (g)
V_{inital} = initial volume of powder added (mL)
V_{final} = final tap volume of powder (mL)

Table 2-2. Powder Flowability in Terms of Carr's Index

Carr's Index %	Description of flow
5-15	Free Flowing – excellent flow granular
12 - 16	Free Flowing – good flow powders
18 – 21	Fair to passable powdered granule flow
23 – 28	Easy fluidizable powders – poor flow
28 – 35	Cohesive powders – poor flow
35 – 38	Cohesive powders – very poor flow
> 40	Cohesive powders – very very poor flow

2.3.2 True (Particle) Density

The particle density was determined using a Micromeritics AccuPyc II 1340 gas pycnometer. The gas used was helium. The gas pycnometer was functional checked (calibrated) using a sphere of known mass and volume. The powder/grit was then placed into the sample holder and the mass of the powder was logged into the AccuPyc and recorded. The AccuPyc calculates both the volume of the powder and particle density.

2.3.3 Particle Size Distribution

For powders that are not visually granular in nature, particle size distribution (PSD) was measured using a Microtrac S3500 laser particle size analyzer. The S3500 was calibrated using National Institute of Standards and Technology (NIST) traceable spheres of known diameters. A small sample of powder (approximately 0.3 to 0.4 grams) is mixed with DI water containing a surfactant (4% sodium hexametaphosphate). The S3500 will not perform the measurement unless there is sufficient material for the instrument to detect. The flow was set to 60% of the maximum flow and the measurement began 30 seconds after sufficient material for the measurement was detected. Each particle size measurement consists of four 30-second measurements and the vol. % is recorded for each micron size bin for volumetric and number distributions. The 30-second measurements are averaged, equation (5), included the % tile values (e.g., 10%, 16%, 25%, etc.) for each of the particle size bins. Percent tile is the particles size for which a vol. % of the material has a smaller diameter. For example, a 10% tile for a powder is 5 µm, which means that 10 vol% of material is below 5 µm in diameter. The volumetric data is the same as that of the mass distribution for a given powder. This might not be true for blended material. The averaged mean, D25, D50, and D75 particles sizes and the PSD are provided where D25, D50, and D75 correspond to the 25th, 50th, and 75th percentile respectively.

$$vol.\%_{j,avg,k} = \sum_{i=1}^{4} vol.\%_{j,i,k} \tag{5}$$

Where: $vol.\%_{j,avg,k}$ = the average vol. % of the 30 second measurements
$vol.\%_{j,i,k}$ = vol. % in particle size bin j, measurement i and sample k
i = the 30 second measurement
j = bin with a specific micron size
k = a single powder sample

For oversized materials, either the vendors supplied characterization or the material was sieved to obtain the distribution. Sieves used are not NIST traceable due to the time frame required to obtain NIST traceable sieves through the Savannah River Nuclear Solutions procurement process. Level 2 procurement is required and can take up to six months in obtaining the sieves and such would not satisfy the timeline required to complete this task. The sieves used in this task are mesh sizes 10, 18, 20, 30, 40, 50, 60, 70, 80, and 170. SRNL could not obtain a 45 mesh sieve.

2.3.4 Morphology

The morphology of the powders and blends was obtained using either SEM or optical images.

2.3.5 Specific Surface Area

The specific surface area (SSA) was obtained using an Micromeritics Corporation Accelerated Surface Area and Pore Analyzer (ASAP). The SSA was determined using nitrogen adsorption and the Brunauer, Emmett and Teller (BET) theory that is integrated into the ASAP software. The data reported using BET is m^2/g. Only batched materials consisting of powders were analyzed. This was due to the small sample used for analysis and the potential for large errors that could result when trying to sub-sample a heterogeneous blend.

2.4 Batching

The powders/grits were blended to obtain targeted volumetric and/or respirable fractions, which were calculated based on the individual powder PSDs and particle densities. The methods used are described below. After the batches were made, they were placed into an oven at 110 °C for at least one day. They were then moved to the plastic glovebox and exposed to the internal environment for at least one day.

The method for making simulant 3 is described in section 3.3. Simulant 3 was not characterized for densities due to the large variability in the makeup of the components.

The resulting blends were not analyzed for PSD, but were characterized for bulk/tap/true densities and for the powder only blend, SSA.

The simulants provided to the customer were based on either reaching a maximum of 5000 grams or 1700 mL of material for each simulant.

2.4.1 Volumetric Targets of Powders

Batching of materials requires the use of mass, not volume. Given the particle size distributions of the powders were provided as a volumetric distribution, the mass fraction of each material was determined using equation (6). The volume of material from this mass fraction and the total volume of the blend is determined using equation (7). The volume fraction for a given material is given by equation (8). The volumetric contribution for a given bin (particle size) for a batch is given by equation (9). The blends were targeted to provide volumetric distributions or mode, such as that shown in Table 2-3. To obtain the vol. % in a mode, the vol. % from the bins in the range of a mode are summed as shown in equation (10). For example, for Mode 1 in Table 2-3, the vol. % of bins from 0.4 to 4 µm is summed and compared to the targeted vol. % for that mode. If the PSD has vol. % of bins below 0.4 µm, these will be added to the range. The same can be said if the PSD has vol. % greater than the 210 µm in Mode 3. These calculations were performed using EXCEL where the mass or volume fractions were adjusted to provide a batch having targeted values.

$$x_{mass,i} = \frac{m_i}{m_T} \text{ where } \sum_i x_{mass,i} = 1 \tag{6}$$

$$v_{vol,i} = \frac{x_{mass,i}}{\rho_i}, \qquad v_{vol,total} = \sum_i v_{vol,i} = \sum_i \frac{x_{mass,i}}{\rho_i} \tag{7}$$

$$f_{vol,i} = \frac{v_{vol,i}}{v_{vol,total}} = \frac{\frac{x_{mass,i}}{\rho_i}}{\sum_i \frac{x_{mass,i}}{\rho_i}} \tag{8}$$

$$vol\%_{i,blend} = \sum_j f_{vol,j} \cdot vol\%_{i,avg,j} \tag{9}$$

$$vol\%_{mode\ j,blend} = \sum_i vol\%_{i,blend} \text{ and } \sum_i vol\%_{mode\ j,blend} = 1 \tag{10}$$

Where: $x_{mass,i}$ = mass fraction of material in batch (g/g-total)
m_i = mass of material in batch (g)
m_T = total mass in batch (g)
$v_{vol,i}$ = volume fraction for a given mass fraction of a material
$v_{vol,total}$ = total volume fraction of the batch
ρ_i = true density of material (g/cm^3)
$f_{vol,i}$ = volume fraction of material in the batch
$vol\%_{i,blend}$ = volume percent for a given bin for the batch
$vol\%_{mode\ j,blend}$ = volume percent given the range of particle size for a mode

Table 2-3. ARIES Tri-Modal Particle Size Distribution (Table 4 from Ref. i)

Mode	Range (µm)	Peak (µm)	Volume Percent
1	0.4 – 4	1 – 2.5	17 – 28
2	4 – 30	11 – 19	34 – 66
3	30 – 210	40 – 60	17 – 38

Table 2-4 provides targeted blends for various combinations of the Modes.

Table 2-4. Potential Blended Targets for ARIES Powders (Table 5 from Ref. i)

Mode	Blends (vol. %)			
	Average	Max Mode 1	Max Mode 3	Max Mode 1 & 2
1	22.5	28	17	28
2	50.0	55	45	66
3	27.5	17	38	6

2.4.2 Blending of Powders and Oversized Materials

The batching of powders and oversized materials targeted the average values stated in Table 7 of Ref. i for the ARIES unprocessed DMO and Table 8 of Ref. i for the ARIES upset DMO and Muffle furnace. Given these are mass fractions, the powders were batched per section 2.4.1 and added to the oversized material. If the densities of the undersize and oversized material are different, the volume contribution from each distribution was targeted. The added oversized material was either sieved to obtain the targeted oversized distribution (or as close as possible) or the as-received distribution was used. If there was insufficient material for a given sieve size, the difference was added to the next lower sieve size. For instance, SRNL did not have a 45 mesh sieve, hence the mass fraction from this sieve was added to the 50 mesh sieve.

2.4.3 Respirable Fraction

The respirable fraction (RF) can be targeted in the batch. First the RF diameter (microns) is calculated using equation (11) for the given material in a blend. For a given material, the vol. % RF is calculated by linearly interpolating the cumulative volume between two particle size bins where the RF diameter is located. The %RF for a batched material is shown in equation (12) where the volume fraction of each material and %RF of the material were multiplied and summed. EXCEL was used to perform this calculation where the batch was determined by satisfying the %RF. The %RF can be determined for any batched material consisting of powder but is specifically targeted for simulants 4 and 5.

$$RF_{dia,i} = \frac{10}{\sqrt{\rho_i}} \tag{11}$$

$$\%RF = \sum \%RF_i \cdot f_{vol,i} \tag{12}$$

Where: $RF_{dia,i}$ = upper diameter for respirable fraction (microns)
$\%RF_i$ = vol. % of respirable particles in a material
$\%RF$ = vol. % of respirable particles in a blend

2.4.4 Simulant 3

Simulant 3 was made from stainless steel powders, cerium oxide powder, titanium oxide powder, and various salts. The list of salts used and their melting temperature are provided in Table 2-5. The salts were blended with the dry powders. Two different types of blends were made, granular and patty-cakes. The granular solids were made by adding the water slowly such that granular material forms while the blend was being mixed. The patty-cakes were made by adding the water to the dry material and pouring them into steel trays. This wetted simulant was then placed into an oven at 110 °C for a minimum of an overnight bakeout to remove free water. The dried materials were then placed into a alumina crucibles, placed into a furnace, and calcined at either 600 or 900 °C for two hours.

Table 2-5. Salts Used in Simulant 3

Salt Name	Compound	Melting Temperature (°C)
Calcium Fluoride	CaF_2	1418
Calcium Chloride	$CaCl_2$	775
Magnesium Chloride	$MgCl_2$	714
Sodium Chloride	NaCl	801

2.4.5 Relative Humidity

A plastic glove bag was used to control the RH within the chamber and is shown in Figure 2-1. Salts were used to control the RH inside the glovebox. The lower RH of 16% was controlled using lithium chloride, and for the high RH of 62%, sodium bromide was used. When using lithium chloride, extra dry salts were added to the bath due to potential moisture ingress into the glove bag. For the sodium bromide, water was added to the bath due to evaporative losses and potential out leakage. The RH was measured using two OMEGA RH650 handheld temperature/relative humidity instruments. Additional details about the plastic glove bog and its use for RH control are documented in SRNL-L3100-2021-00005 (Ref. v).

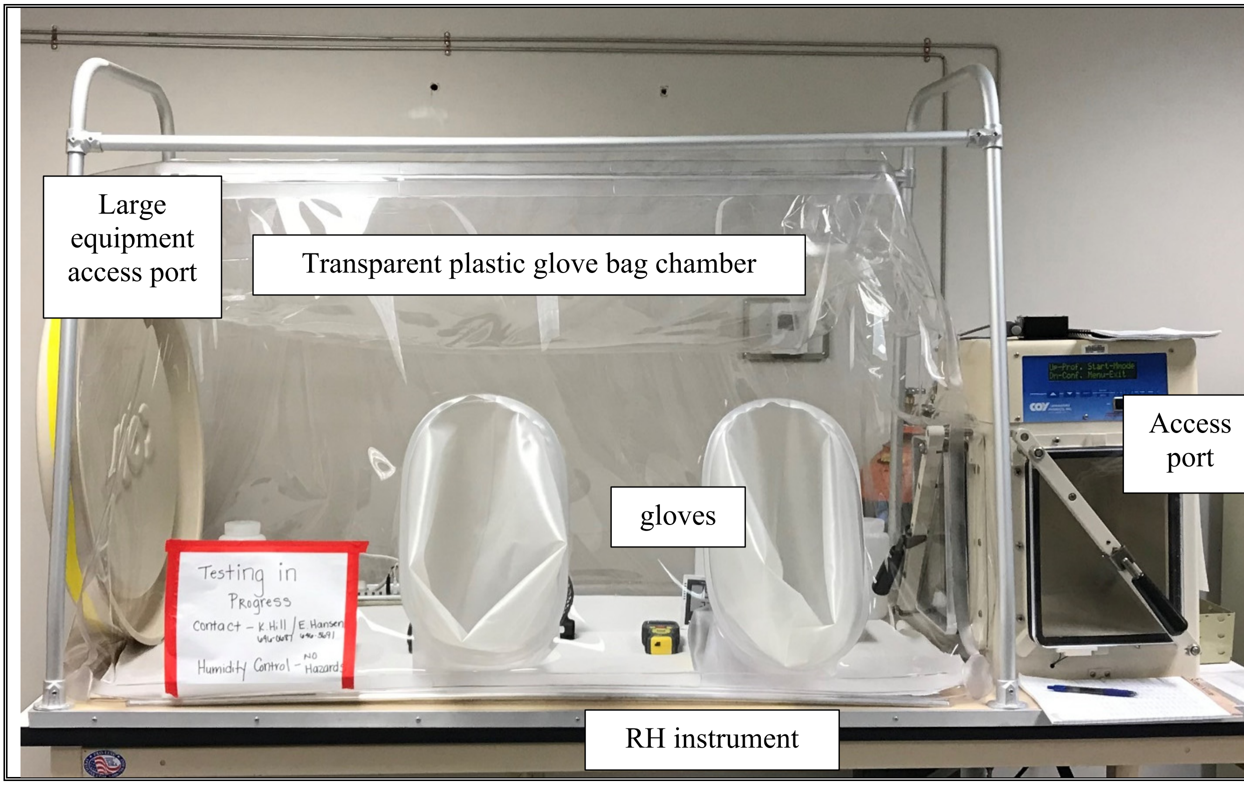


Figure 2-1. Plastic Glove Bag Chamber for Relative Humidity Control

2.5 <u>Quality Assurance</u>

The data obtained is for general use. Analytical equipment used in this task were calibrated and verified prior to use.

3.0 Results and Discussion

3.1 <u>Procured Materials</u>

The vendors and materials that were obtained are listed in Table 3-1. This table also lists the product name and the size of the material. The material/size information was used to determine if it could be of use for this application and procured for analysis. The only material from Table 1-1 for simulants 1 through 5 that was not procured was copper tungsten carbide.

Table 3-1. Materials Obtained from Vendors

Vendor	Material	Name	Size stated by Vendor
Buffalo Tungsten	Tungsten Carbide	WCI	1.0 – 2.0 µm FSSS[1]
		WCIV	9.0 – 14.0 µm FSSS[1]
		WCVI	25.0 – 50.0 µm FSSS[1]
		WCX	-270 Mesh
		WCZ	-8 + 20 Mesh
		WCZ	-30 + 60 Mesh
		WCZ	-40 + 80 Mesh
		WCZ	-20 + 40 Mesh
		WCY	-80 +200 Mesh
		WCY	-80 +300 Mesh
Atlantic Equipment Engineers	Cerium Oxide	CE-602	-325 Mesh
	Stainless Steel	SS-103	-325 Mesh
	Molybdenum	MO-103	-325 Mesh
		MO-102	1 – 5 µm
		MO-101	2 – 8 µm
	Tungsten Carbide	WP-301	1 – 5 µm
		WP-302	-325 Mesh
		WP-305	-100 + 270 Mesh
Blue Line	Cerium Oxide	Mean 10 µm	10 µm
		Mean 25 µm	25 µm
		Mean 8.4 µm	8.4 µm
Good Fellow	Molybdenum	MO006015	105 µm
		MO006011	350 µm
Pellets	Stainless Steel	Unscreened	As stated on package
		PWDR + 60	+250 µm
		PWDR – 100	-150 + 45 µm
		PWDR – 200	-75 + 45 µm
		PWDR – 325	- 44 µm
		PWDR – 350	As stated on package
Vulkan	Stainless Steel	G30	140 – 500 µm
		G40	400 – 800 µm
		G60	700 – 1250 µm
		G150	1250 – 1700 µm
		G200	1400 – 2000 µm
		G300	1700 – 3000 µm
EdgeTech	Molybdenum	-200 mesh	As stated on package
		2 – 3 µm	As stated on package
		2.55 µm	As stated on package
ACROS	Cerium Oxide	AC199120025	Powder
Alfa Aesar	Cerium Oxide	12925	Powder
Aldrich	Cerium Oxide	211575	- 5 µm
Sandvik	Stainless Steel	- 32 µm	- 32 µm

1 – FSSS = Fisher Particle Size Analysis

3.2 Characterization of Procured Materials

The characterization of the as-received materials for PSD (mean, D25, D50 and D75 tiles), densities and %CI are provided in Table 3-3. Table 3-3 also includes the lot number, which was how the vendor distinguished materials of the same type (name) that were produced at different times. There is variability lot to lot. The PSD of each lot was analyzed. The same lot numbers were repeated to determine if the distribution was consistent or measured again if another batch was received from the vendor with the same lot number. The initial PSD measurement was used in all subsequent calculations if the material was used in a batch. The particle size distribution curves, including both channel and cumulative for each of the procured powders are provided in Appendix A. The tungsten carbide, molybdenum, and stainless steel powders had PSDs that could be added to produce ARIES type tri-modal PSD distribution. The density of the molybdenum most represents the particle density of the ARIES and aqueous processes.

Vulkan provided test certificates of their oversized materials, but the reported screens openings were not consistent to the oversized mesh distribution stated in Reference i. Two materials from Buffalo Tungsten were sieved to obtain oversized particle size distributions and their distributions are listed in Table 3-2. These materials were used without sieving to provide oversized material for the upset ARIES/furnace oversized distribution.

Table 3-2. Oversized Distribution of Buffalo Tungsten Material

WCZ926-4080		WCZ917-2040	
Mesh	Mass Fraction	Mesh	Mass Fraction
35	0.0001	20	0.0000
40	0.0235	30	0.3081
50	0.2736	35	0.3228
60	0.1428	40	0.2131
70	0.2570	50	0.1558
80	0.2594	Pan	0.0002
Pan	0.0436		

Buffalo Tungsten WCZ 900-0820 and WCZ 903-3060 were sieved, and these sieved materials were used to provide the oversized particles for unprocessed ARIES. Their distributions were not used.

Vulkan G30 and G40 were sieved, and these sieved materials were used to provide the oversized particles for unprocessed ARIES/furnace. Their distributions were not used.

Appendix B contains the micrographs for any given name of material. In general, none of the material were truly spherical in nature and hard agglomerates seemed to be present for many of the materials. The stainless steel from Pellets LLC had the characteristics of shaved material and can have large aspect ratios as compared to the other materials.

SRNL did not procure any cobalt tungsten carbide powder. Oerlikon-Metco provides thermal spray powders, but the particles were spherical and porous. Furthermore, multiple PSDs were not readily available, and the quantity required was large.

Table 3-3. Characterization of As-Received Material, PSD, Densities, and %CI

Vendor	Material	Name	Lot - Number	Particle Size Distribution (microns)				Densities (g/cm^3)			%CI
				Mean	D25	D50	D75	Bulk	Tap	Particle	
Buffalo Tungsten	Tungsten Carbide	WCI	1103	4.0	1.7	2.9	4.7	3.28	3.95	15.55	16.95
			1107	3.9	1.6	2.7	4.4	3.31	4.17	15.59	20.64
			1111	3.7	1.8	3.4	4.9	3.06	3.51		12.98
		WCIV	648	19.6	16.0	19.4	23.0	7.33	8.06	15.60	9.04
		WCVI	1256	72.6	50.0	59.8	74.1	7.41	8.09	15.57	8.38
			1212	58.3	41.2	54.4	70.5	7.18	7.69	15.58	6.86
		WCX	1183	44.9	34.6	42.7	52.3	7.50	8.24	15.59	9.04
		WCZ	900-0820	OVERSIZED				7.28	7.89	14.05	7.67
			903-3060	OVERSIZED				5.13	5.78	12.35	11.57
			926-4080	OVERSIZED				5.52	6.08	13.49	9.20
			927-2040	OVERSIZED				5.19	5.65	13.06	8.10
		WCY	2011-80200-4	80.3	56.1	70.2	92.8	8.27	9.57	16.47	13.59
			2101-80300-1	140.0	103.3	132.2	169.4	7.94	8.58		7.44
Atlantic Equipment Engineers	Cerium Oxide	CE-602	Not Specified	1.9	0.6	1.1	2.5	1.13[a]	1.55[a]	7.15[a]	27.05[a]
	Stainless Steel	SS-103	1711521	35.7	24.8	33.7	43.8	2.84	3.07	7.91	7.56
				34.7	24.5	33.0	42.5				
	Molybdenum	MO-103	2012516	3.4	2.3	3.2	4.2	2.71	3.20	10.17	15.31
				3.2	1.9	2.9	4.0				
			2004514	4.1	1.8	3.5	5.6	2.87	3.23	10.09	11.21
				4.5	2.4	4.0	5.9				
		MO-102	2012501	4.0	2.3	3.6	5.1	2.81	3.32	10.19	15.49
				3.8	1.7	3.4	5.1				
				3.8	2.0	3.5	5.1				
		MO-101	2004514	4.7	2.8	4.3	6.1	2.80	3.33	10.11	15.97
				4.1	1.8	3.4	5.5				
			2007508	3.7	2.0	3.5	4.9	2.67	2.99	10.08	10.58
	Tungsten Carbide	WP-305	1306512	106.9	85.0	102.8	124.2	7.84	8.69	15.50	9.78
			2101507-3	74.2	58.6	69.8	84.4				
		WP-302	1901510	38.9	11.7	22.6	64.9				
			2101507-2	78.9	4.9	82.7	121.6	6.80	7.72	15.31	11.83
		WP-301	1908505	5.9	3.0	4.7	7.4	3.73	4.69	15.37	20.42
Blue Line	Cerium Oxide	Cerium Oxide 10um	D12207	42.7	8.9	25.4	61.7	1.76	2.26	7.21	21.96
		Cerium Oxide 25um	D12206	39.0	29.0	36.7	46.2	1.74	2.10	7.30	17.13
		Cerium Oxide 8.4um	D30593	8.4	3.51	6.7	11.0	0.73	0.97	5.94	24.77
Good Fellow	Molybdenum	MO0060105	Not Specified	62.5	26.2	49.2	82.4	1.57	1.90	10.12	17.12
		MO0060350	Not Specified	159.7	121.8	157.7	194.2	2.64	2.91	10.02	9.44

Table 3-3. Characterization of As-Received Material, PSD, Densities, and %CI

Vendor	Material	Name	Lot - Number	Particle Size Distribution (microns)				Densities (g/cm^3)			%CI
				Mean	D25	D50	D75	Bulk	Tap	Particle	
Pellets LLC	Stainless	unscreened	Not Specified	116.3	51.2	89.6	158.4	2.85	3.38	7.69	15.76
		60	Not Specified	87.6	47.0	77.4	121.2	2.70	3.21	7.68	15.79
		-100	Not Specified	123.7	89.8	119.4	152.3	2.81	3.17	7.77	11.53
		-200	Not Specified	71.9	41.9	63.1	89.9	2.33	2.88	7.61	19.11
		-350	Not Specified	94.3	50.2	84.1	131.9	2.68	3.36	7.58	20.28
		-350 + 170	Sieved	239.9	159.2	222.8	297.1	material sieved from -350			
		-350 - 170	Sieved	51.2	27.6	42.9	63.3	material sieved from -350			
		-325		77.3	36.3	66.8	107.3	2.31	2.93	7.56	21.17
Vulkan	Stainless Grit	G200	23422	OVERSIZED				3.61	3.97	7.51	9.03
		G300	14598	OVERSIZED				3.33	3.51	7.56	5.20
		G40	24006	OVERSIZED				3.47	3.99	7.53	13.02
		G60	24149	OVERSIZED				3.43	3.87	7.55	11.34
		G30	24427	OVERSIZED				3.58	4.04	7.53	11.38
		G150	08311	OVERSIZED				3.84	4.06	7.45	5.32
EdgeTech	Molybdenum	-200mesh	977-210128	20.6	10.2	16.9	29.5	1.69	2.40	10.06	29.47
			977-210301	18.3	9.6	15.4	24.3	1.74	2.16		19.25
				18.0	8.9	14.5	22.7				
				19.7	9.9	16.0	25.9				
			1010-210308	16.2	10.1	15.3	21.3	1.61	2.04	10.18	20.84
		2-3micron	977-210129	15.8	7.6	12.3	21.7	1.54	2.07	10.11	25.54
				21.3	8.1	13.8	27.7	1.54	2.07		25.54
				22.1	8.2	15.0	31.3				
		2.55 micron	977-210129	17.5	7.3	12.6	23.6	1.45	1.82	10.16	20.44
ACROS	Cerium Oxide	AC199120025	A0413188	1.0[b]	0.5[b]	0.7[b]	1.2[b]	1.33	1.81	7.58	26.66
Alfa Aesar	Cerium Oxide	12925	N15H053	6.2	0.8	4.0	9.5	1.58	2.09	7.13	24.41
Sandvik	Stainless steel	-32 microns	20D1401	15.4	7.8	14.2	21.0	3.81	4.38	7.96	12.92
Aldrich	Cerium Oxide	211575	MKCL9679	1.4	0.6	1.2	1.9	0.96	1.23	7.22	21.90

[a] from Reference v

[b] obtained from last PSD measurement due to breakup of large agglomerate.

3.3 Simulants

The descriptions of the batching of simulants 1, 2, 4, and 5 are provided in Table 3-4. This table provides a basis of why they were selected and what properties were specifically targeted, given the composition of the available material.

Table 3-4. Batching of Simulants 1, 2, 4, and 5

Simulant	Basis
1A	Highest density material, tungsten carbide. PSD of the powder matching that of the nominal ARIES distribution. Oversized PSD matching up with the unprocessed ARIES distribution. Nominal wt. % distribution between the under and oversized ARIES distribution.
1B	Highest density material, tungsten carbide. PSD of the powder matching that of the greatest fraction of Modes 1 and 2 of the ARIES distribution. Oversized PSD matching up with the unprocessed upset ARIES/furnace distribution. Nominal wt.% distribution between the under and oversized ARIES/furnace distribution was targeted.
1C	Most representative density material, molybdenum for undersized ARIES distribution, targeting the volumetric fraction of Modes 1 and 2. Oversized material was stainless steel targeting the ARIES/furnace distribution. Maintained the same volumetric distribution if density were the same, hence the mass of molybdenum is greater than that of the stainless steel.
2A	Highest density material, tungsten carbide. PSD of the powder matching that of the nominal ARIES distribution.
2B	Highest density material, tungsten carbide. PSD of the powder matching that of the greatest fraction of Modes 1 and 2 of the ARIES distribution.
2C	Most representative density material, molybdenum for undersized ARIES distribution, targeting the volumetric fraction of Modes 1 and 2.
4A	Most representative density material, molybdenum. Not chemically similar. Targeting %RF and mode 2 particle size respectively. Blend was targeted to provide the %RF.
4B	Chemically similar processed material, cerium oxide. Targeting mode 2 particle size and %RF respectively. No blending with other materials.
5A	Chemically similar processed material, cerium oxide. %RF range is the target, respectively, PSD as is. No blending with other materials.

Details of the batching for each simulant are provided in the simulant data sheets located in the Executive Summary section. These data sheets summarize the temperature and RH of the bulk/tap density at 16% RH, the batch size (either mass or volume limited), %CI, SSA (if applicable), composition of the components that made up the batch by mass and a PSD of the materials that were considered powder. The 16% data is reported for the batched material, given this is the condition in which the simulants were provided to MSCED. The PSDs for the powdered materials for each simulant are provided in Appendix C to allow the reader a better view of the distribution. The bulk density, tap density and %CI for the individual powders are room condition measurements of the as-received powders.

The bulk density and tap density were obtained at two different RH, approximately 16% and 62% and the temperature varied between 70 to 72 °F for these measurements. The %CI were calculated from these densities. Two different size graduated cylinders, 100 mL and 200 mL, were used to obtain the bulk/tap densities. The 100 mL graduated cylinders were used because the volume of one of the simulants was not sufficient in obtaining the targeted volumes for the 250 mL cylinder. The averages and standard deviation are provided in Table 3-5 and Table 3-6 for the 250 and 100 mL graduated cylinders respectively. The general trend was as the %RH went from 16% to 62%, the %CI increased, indicating the material might be less flowable as the %RH increases. Furthermore, the 100 mL data %CI are typically larger than that of the 250 mL data, such cannot be explained. Simulant 4A is cerium oxide and its true density is lower than other cerium oxides that have been characterized. This could be due to trapped gas when the agglomerates were formed.

Table 3-5. Densities, %CI, and SSA Using 250 mL Cylinder for Simulants 1, 2, 4, and 5 @~70ºF

Simulant	% RH	Densities (g/cm^3)				%CI		True Density (g/cm^3)	SSA (m^2/g)
		Bulk		Tap					
		Avg	Stdev	Avg	Stdev	Avg	Stdev		
1A	16	7.56	0.06	8.01	0.03	5.6	1.1	13.60	N/M
	62	7.54	0.10	8.21	0.10	8.2	1.8		
1B	16	8.07	0.12	9.06	0.13	10.9	2.1	14.45	
	62	8.00	0.24	8.89	0.16	10.0	1.2		
1C	16	2.58	0.04	2.96	0.04	13.0	2.0	10.08	
	62	2.40	0.08	2.76	0.02	12.8	2.3		
2A	16	7.12	0.04	7.72	0.06	7.7	1.2	15.60	0.16
	62	6.76	0.06	7.38	0.12	8.5	2.2		
2B	16	6.50	0.10	7.08	0.02	8.3	1.5	15.53	0.23
	62	6.02	0.11	6.73	0.08	10.6	1.1		
2C[a]	16	1.88	0.00	2.28	0.05	17.4	1.8	10.43	0.39
	62	Not measured due to insufficient sample volume							
4A	16	1.71	0.08	2.00	0.08	14.3	0.9	9.94	0.50
	62	1.51	0.01	1.78	0.02	15.0	1.0		
4B	16	0.71	0.03	1.00	0.05	29.4	0.6	5.94	N/M
	62	0.62	0.03	0.93	0.01	33.9	3.9		
5A	16	1.12	0.02	1.35	0.01	17.0	2.4	7.15[b]	3.90
	62	1.11	0.03	1.35	0.03	17.9	3.7		

[a] Bulk starting volume was less than 100 mL, decision not to measure for RH of 62%
[b] from Reference v

Table 3-6. Densities, %CI, and SSA Using 100 mL Cylinder for Simulants 1, 2, 4, and 5 @~70ºF

Simulant	% RH	Densities (g/cm^3)				%CI		True Density (g/cm^3)	SSA (m^2/g)
		Bulk		Tap					
		Avg	Stdev	Avg	Stdev	Avg	Stdev		
1A	16	7.75	0.12	8.72	0.11	11.2	2.3	13.60	N/M
	62	7.82	0.09	8.50	0.08	7.9	1.6		
1B	16	8.51	0.10	9.69	0.15	12.2	0.4	14.45	
	62	8.08	0.10	9.86	0.48	18.0	4.8		
1C	16	2.56	0.08	2.92	0.03	12.3	2.3	10.08	
	62	2.43	0.02	2.88	0.02	15.8	2.2		
2A	16	6.99	0.05	7.68	0.07	9.0	0.4	15.60	0.16
	62	6.68	0.07	7.67	0.04	12.9	2.9		
2B	16	6.29	0.04	7.00	0.11	10.1	0.8	15.53	0.23
	62	6.08	0.11	6.89	0.14	11.8	1.4		
2C	16	1.92	0.02	2.15	0.03	10.6	2.0	9.52	0.39
	62	1.80	0.06	2.11	0.02	14.5	2.5		
4A	16	1.64	0.01	1.82	0.03	10.2	1.6	9.94	0.50
	62	1.56	0.06	1.78	0.10	12.0	2.4		
4B	16	0.60	0.02	0.88	0.02	31.6	3.5	5.89	N/M
	62	0.60	0.05	0.93	0.01	35.4	4.5		
5A	16	1.13	0.02	1.31	0.01	14.0	1.9	7.15[a]	3.90
	62	1.14	0.01	1.38	0.02	17.1	2.7		

[a] from Reference v

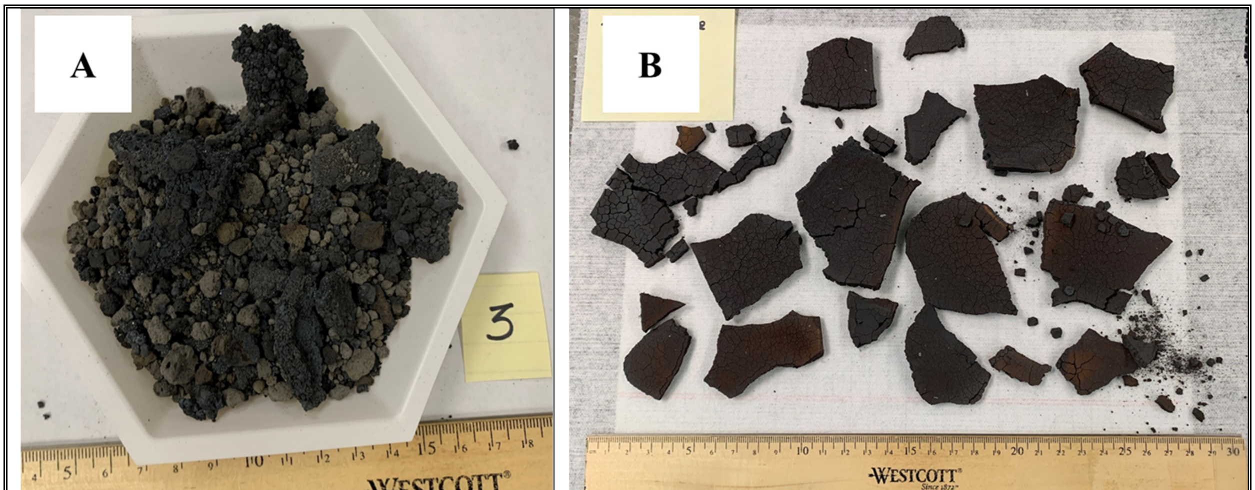
Simulant 3 represents the pyrochemical processed materials with high levels of salts. The salt composition and calcination temperature were based on Section 3.3 of M-ESR-K-00119 (Ref. ii). With two exceptions, all samples were calcined at 900 °C for 2 hours. Batch 7 was dried at 110 C overnight and not calcined. Batch 18b was calcined at 600 °C for 2 hours. While no specific compositional information was included in Reference ii, the following assumptions guided material selection. The total chlorine concentration ranged from 0 - 36.4 wt. %, and of that, $MgCl_2$ consisted of 1 - 7 wt. %, $CaCl_2$ consisted of 10 wt. %. Sodium chloride accounted for a significant portion. As such, these species were selected; however, $CaCl_2•2H_2O$ was utilized instead of the anhydrous form as it was readily available. Considering samples were calcined well above the dehydration temperature, this was not expected to considerably alter the results. In simulant 3B, the maximum NaCl content was utilized, as its melting temperature (Table 2-5) was below the calcination temperature and would promote liquid phase sintering, thereby producing strong, non-friable agglomerates. Several scoping batches also utilized CaF_2. These batches were incorporated into simulant 3B, but CaF_2 was not selected for use in simulant 3B as it was likely detrimental to agglomerate strength and decreased hygroscopicity.

Simulant 3A is a consolidation of Batches 1-4, 7, 9-16, 18a, and 18b. Table 3-7 is the nominal compositional makeup of the consolidated samples. The individual batch compositions and images of individual batches are included in Appendix D.

Table 3-7. Composition of Simulant 3A

Component	Wt. %
Sandvik 316L Stainless Steel < 32 microns	48.93
Atomising Systems 316LB Stainless Steel	31.90
CeO_2	4.58
TiO_2	3.07
$CaCl_2 \cdot 2H_2O$	3.79
$MgCl_2$	2.32
NaCl	1.37
CaF_2	4.05

All individual batches were dry mixed in a plastic bag prior to the addition of water. Water was introduced by 1 of 2 processing techniques: hand mixing with a spatula or direct addition to the batch bags. For the hand mixing technique, water was added a few drops at a time from a pipette while a spatula was used to continuously mix the powder. This technique generally resulted in clumps as seen in Figure 3-1-A. For direct addition to the batch bag, water was added to the same bag that dry powders were mixed in then kneaded into the powder to create a slurry. This slurry was poured/placed into a steel drying pan to form a patty-cake as seen in Figure 3-1-B.

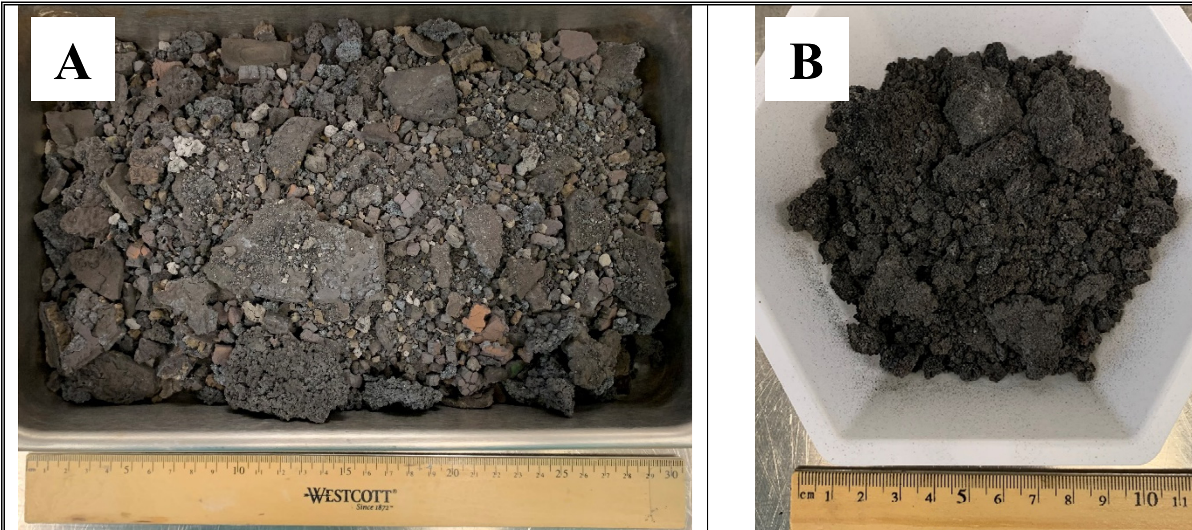


Batch 3 prepared by hand mixing after calcination.	Batch 16 prepared by bag mixing after calcination

Figure 3-1. Hand and Bag Preparation of Simulant 3

Batches were dried in an oven at 110 °C for a minimum of 12 hours before calcination. Samples were calcined at 900 °C for 2 hours (except for batch 18b which was calcined at 600 °C for 2 hours) in an alumina crucible with a heating and cooling rate of 10 °C/min. Samples were stored in a drying oven at 110 °C and ultimately consolidated (Figure 3-2-A).

The bulk density of simulant 3A was 1.47 g/cm^3. Due to the larger variability of size and shape of the resulting calcined material, the bulk density was calculated by measuring the mass of the 1.7 L of the simulant 3A placed into a 2 L bottle and dividing the mass by the volume.



Simulant 3A after consolidation	Simulant 3B

Figure 3-2. Simulants 3 After Calcination

Simulant 3B was a uniform composition listed in Table 3-8. Water was added by hand mixing and the drying and calcination profiles were identical to simulant 3A. Figure 3-2-B shows simulant 3B post calcination. The density of simulant 3B was measured in the same way as simulant 3A and was 1.15 g/cm^3.

Table 3-8. Composition of Simulant 3B

Component	Wt. %
Sandvik 316L Stainless Steel < 32 microns	38.42
Atomising Systems 316LB Stainless Steel	21.94
NaCl	19.35
$CaCl_2 \cdot 2H_2O$	10.12
$MgCl_2$	7.12
TiO_2	3.05

4.0 References

[i] Hansen, E. K., "Simulant Recommendation for the Blend Can Loading System", SRNL-STI-2020-00503, Rev. 1, December 2020
[ii] Hodges, J., "Surplus Plutonium Disposition Project Feed Characterization and Simulants for Blend Can Loading System Development," M-ESR-K-00119, October 2020
[iii] ASTM D7481 – 18, "Standard Test Method for Determining Loose and Tapped Bulk Densities of Powders using a Graduated Cylinder".
[iv] Caccavo, D., Cascone, S., Apicella, P., Lamberti, G., and Barba, A.A., "HPMC-Based Granules for Prolonged Released of Phytostrengtheners in Agriculture," Chemical Engineering Communications, 2017.
[v] Hansen, E. K., Hill, K. A., and Stanfield, A., "Selection of Relative Humidity Chamber for Conditioning Simulant for Testing the Blend Can Loading System (BCLS)", SRNL-L3100-2021-00005, Rev. 0, February 2021

Appendix A. PSD of Procured Powders

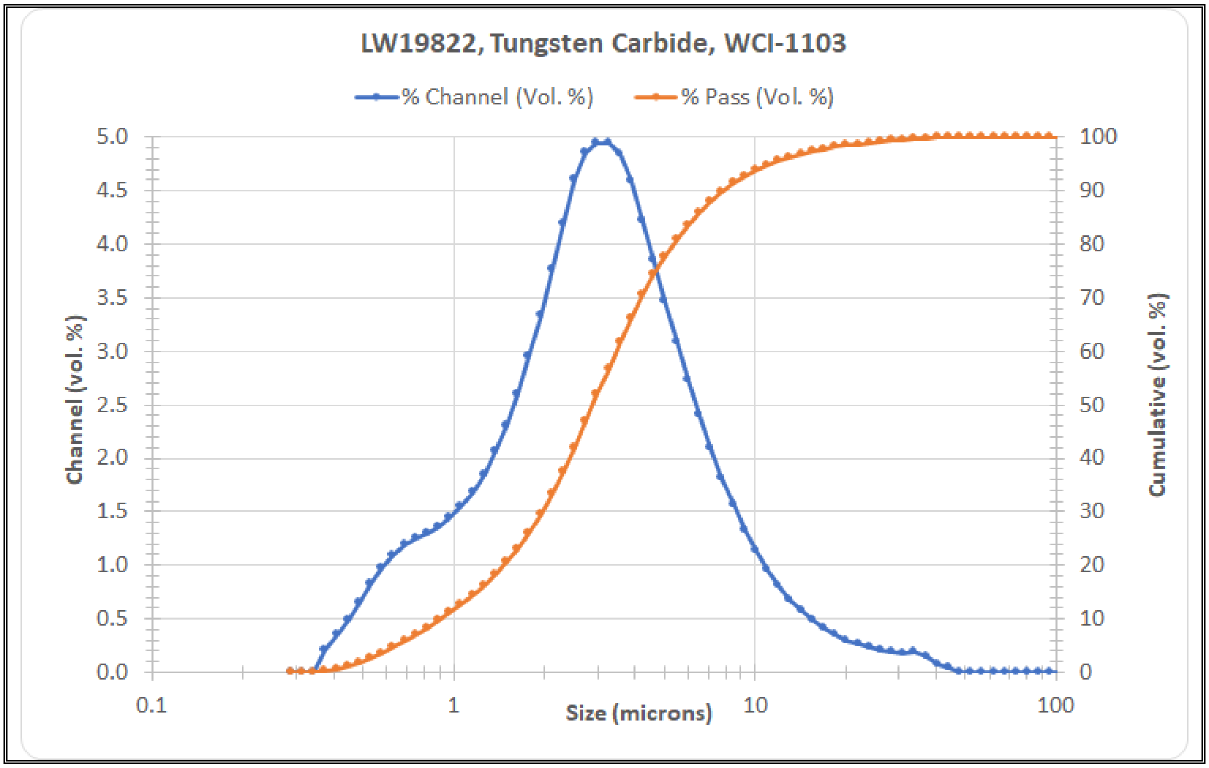


Figure A- 1 PSD Buffalo Tungsten WCI-1103

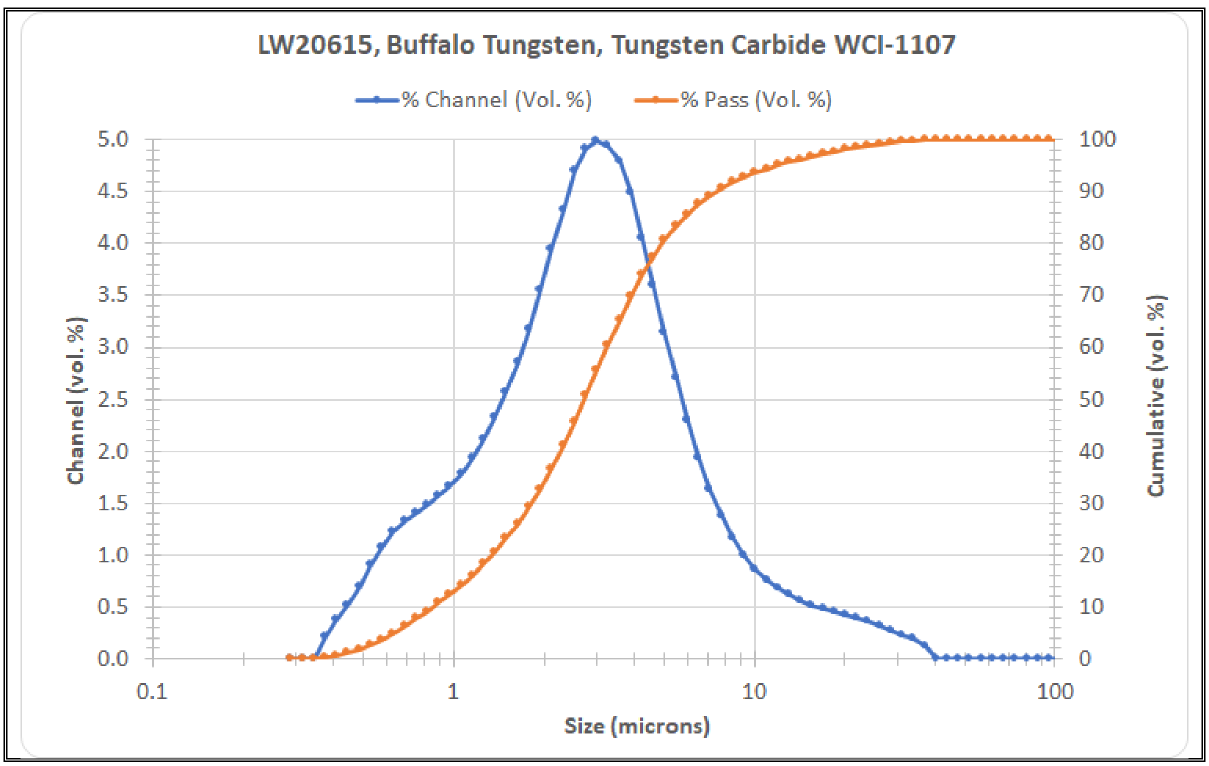


Figure A- 2 PSD Buffalo Tungsten WCI-1107

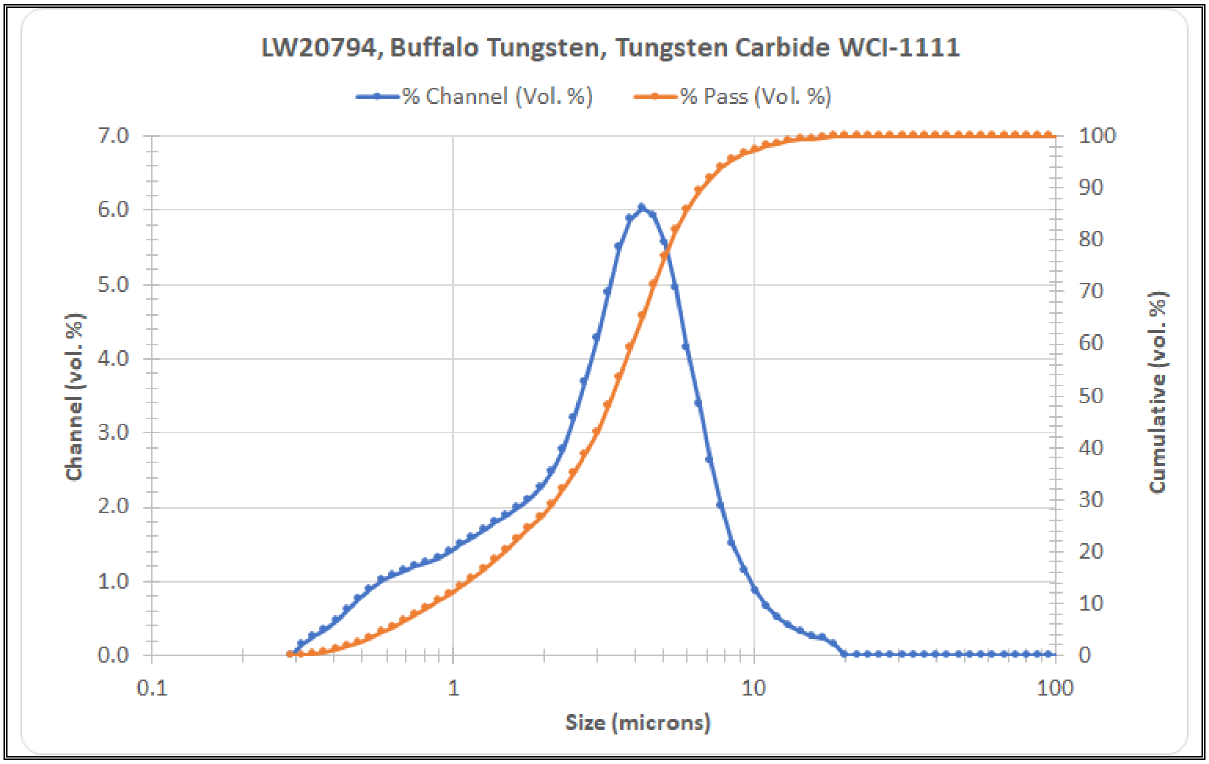


Figure A- 3 PSD Buffalo Tungsten WCI-1111

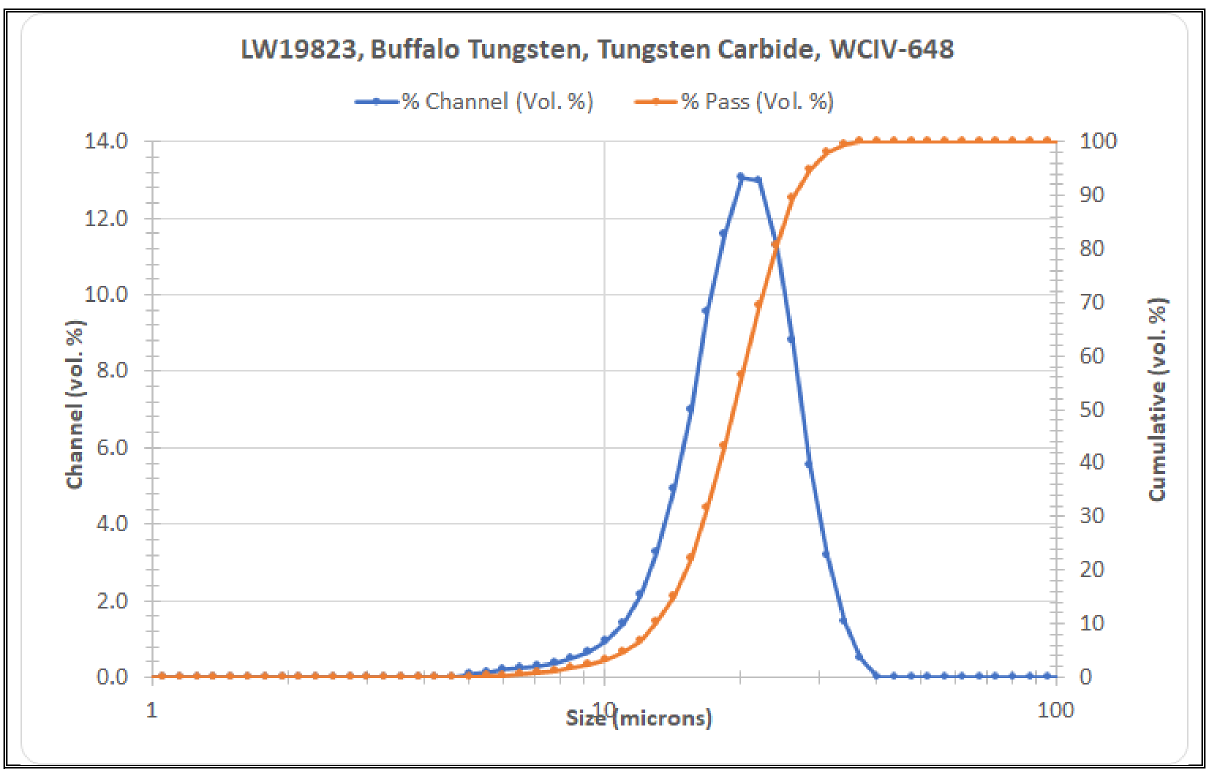


Figure A- 4 PSD Buffalo Tungsten WCIV-648

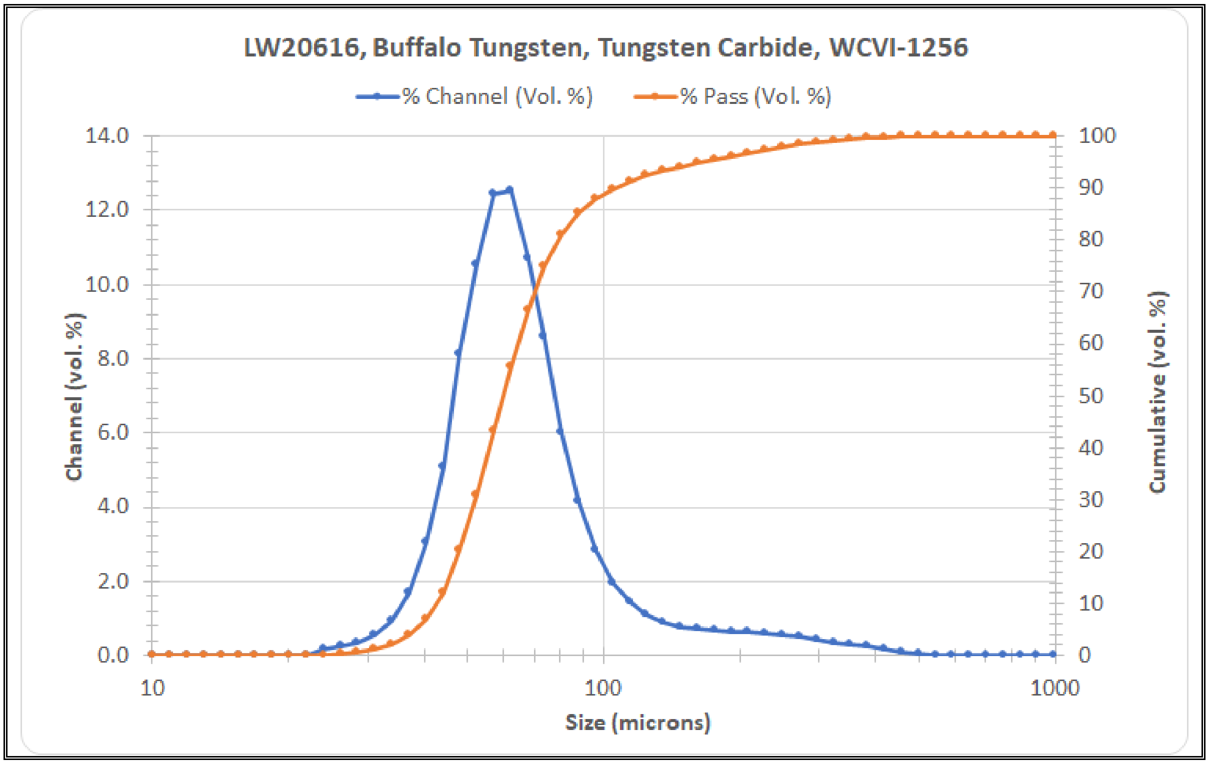


Figure A- 5 PSD Buffalo Tungsten WCVI-1256

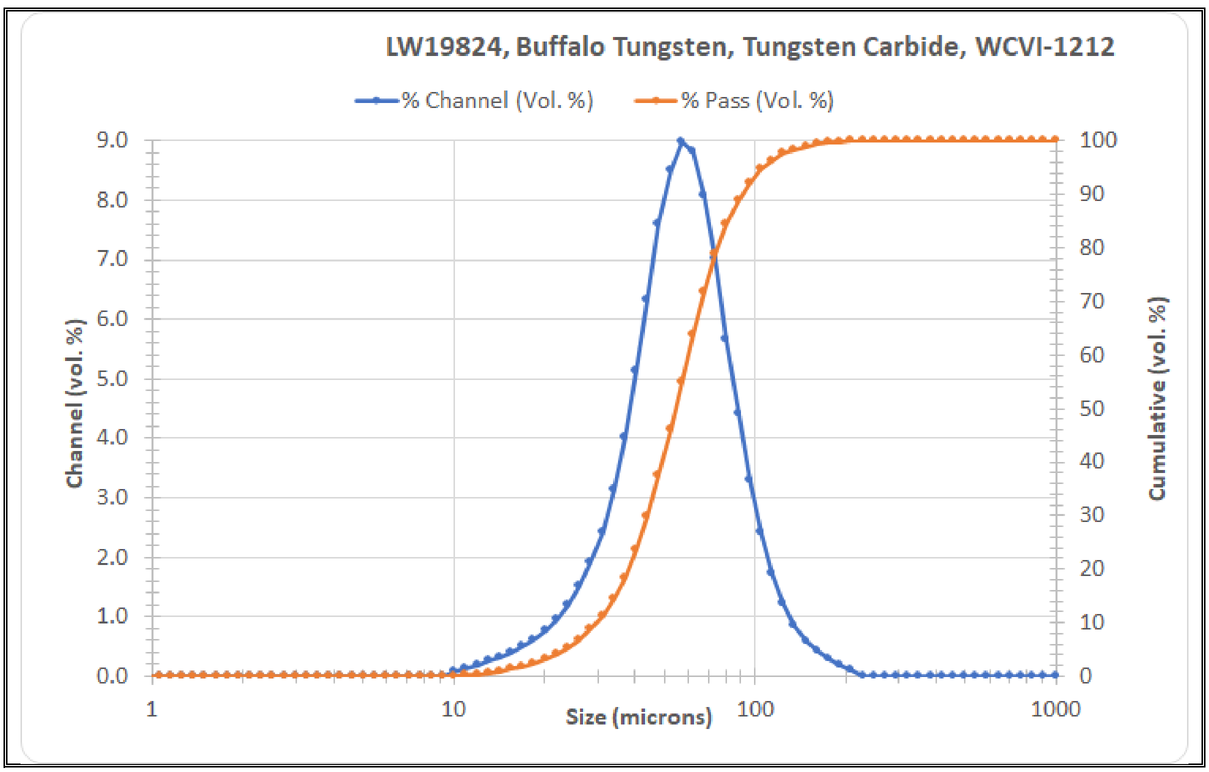


Figure A- 6 PSD Buffalo Tungsten WCVI-1212

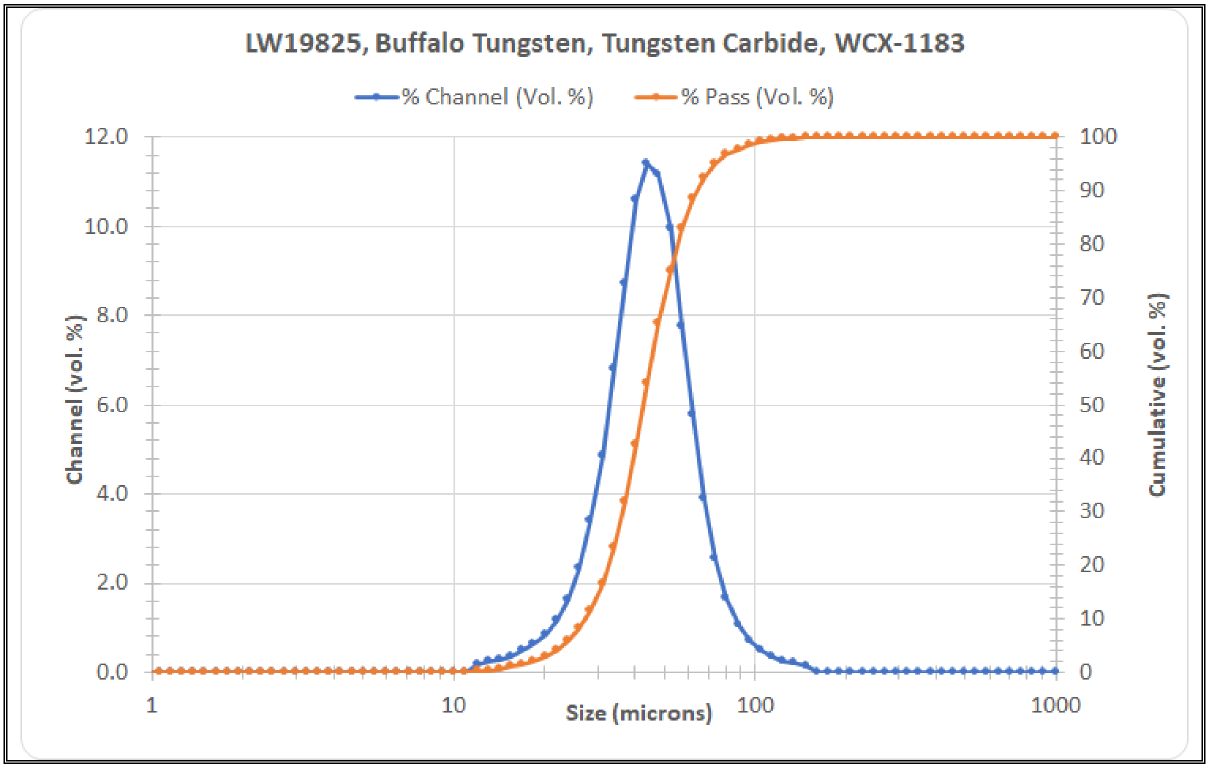


Figure A- 7 PSD Buffalo Tungsten WCX-1183

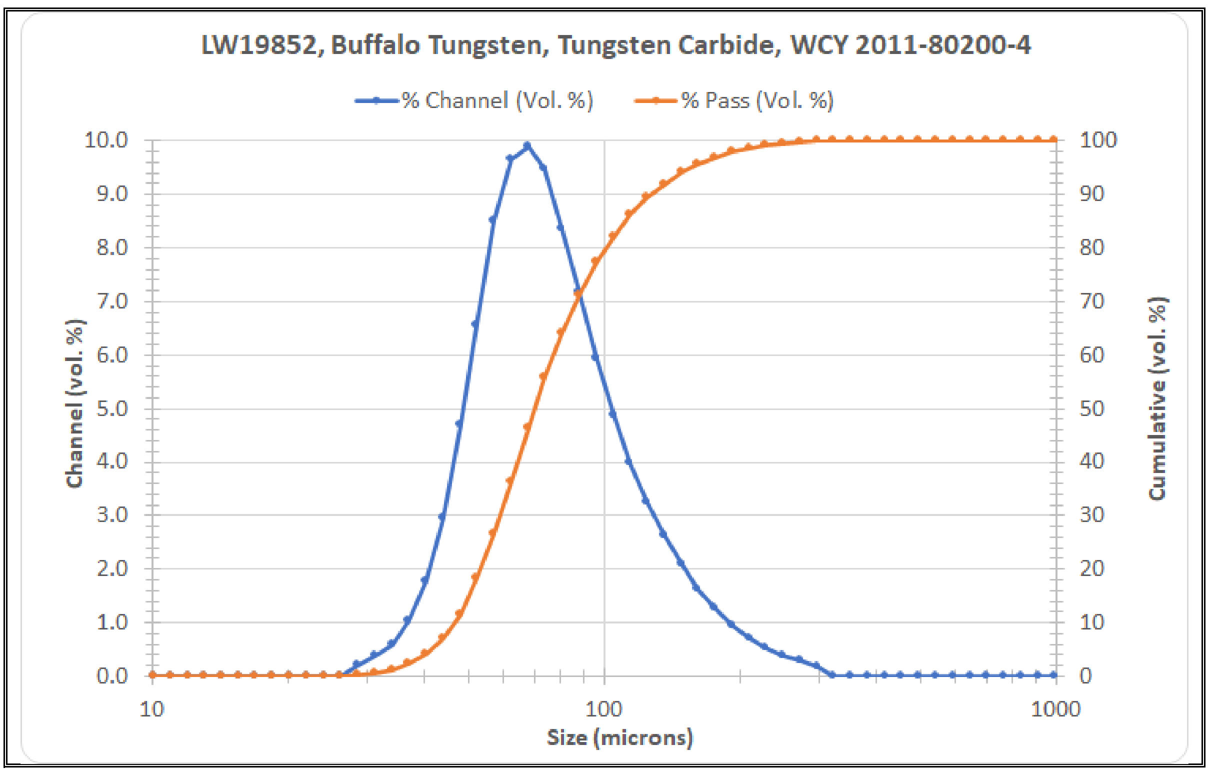


Figure A- 8 PSD Buffalo Tungsten WCY 2011-80200-4

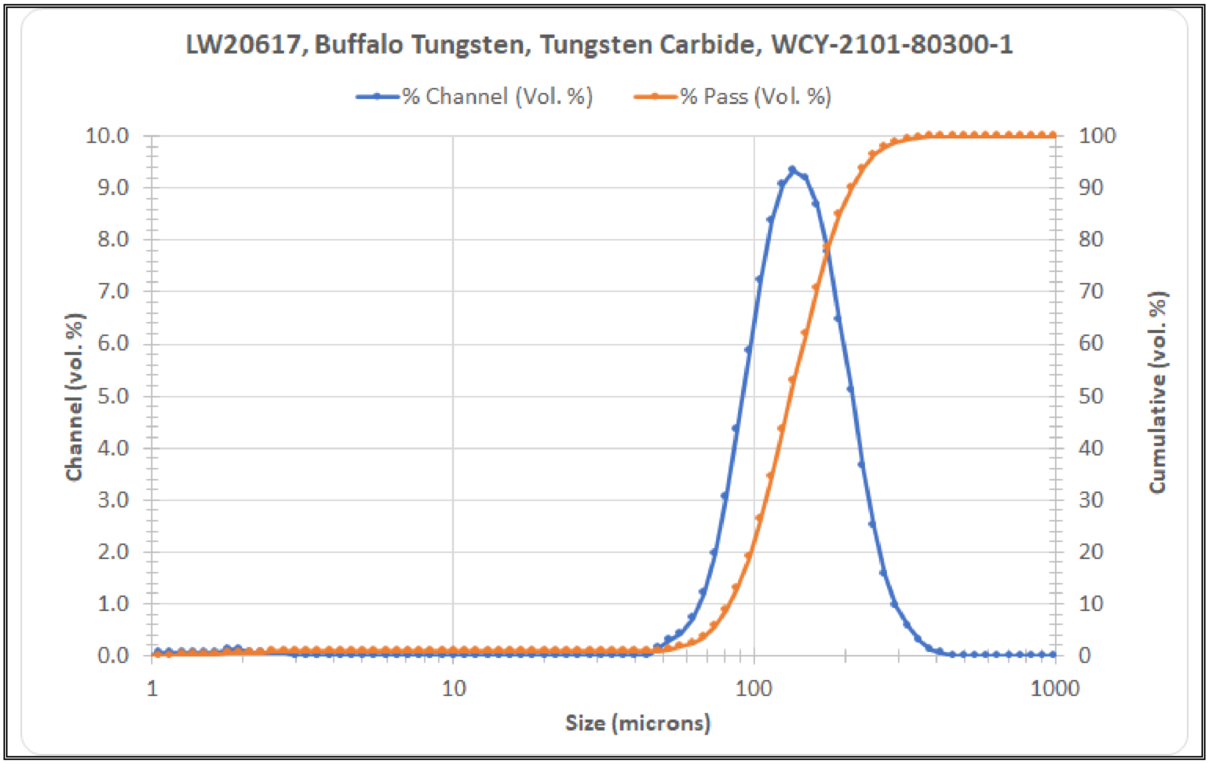


Figure A- 9 PSD Buffalo Tungsten WCY 2011-80300-1

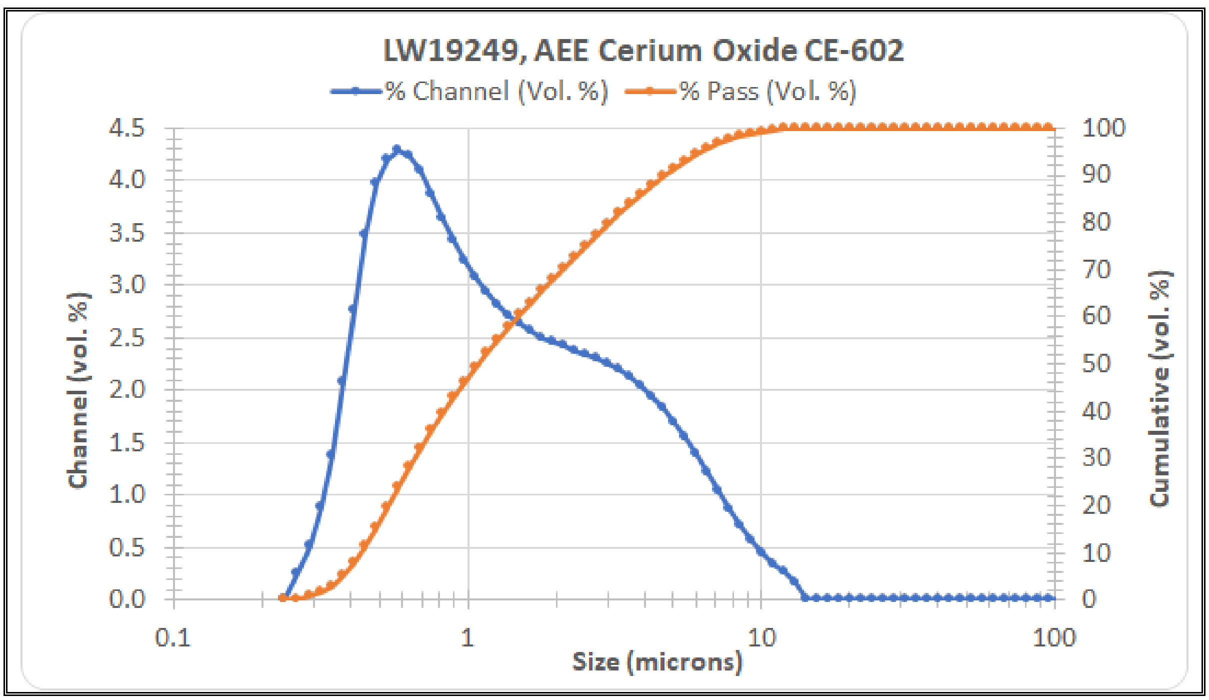


Figure A- 10 PSD Atlantic Engineering Equipment Cerium Oxide CE-602

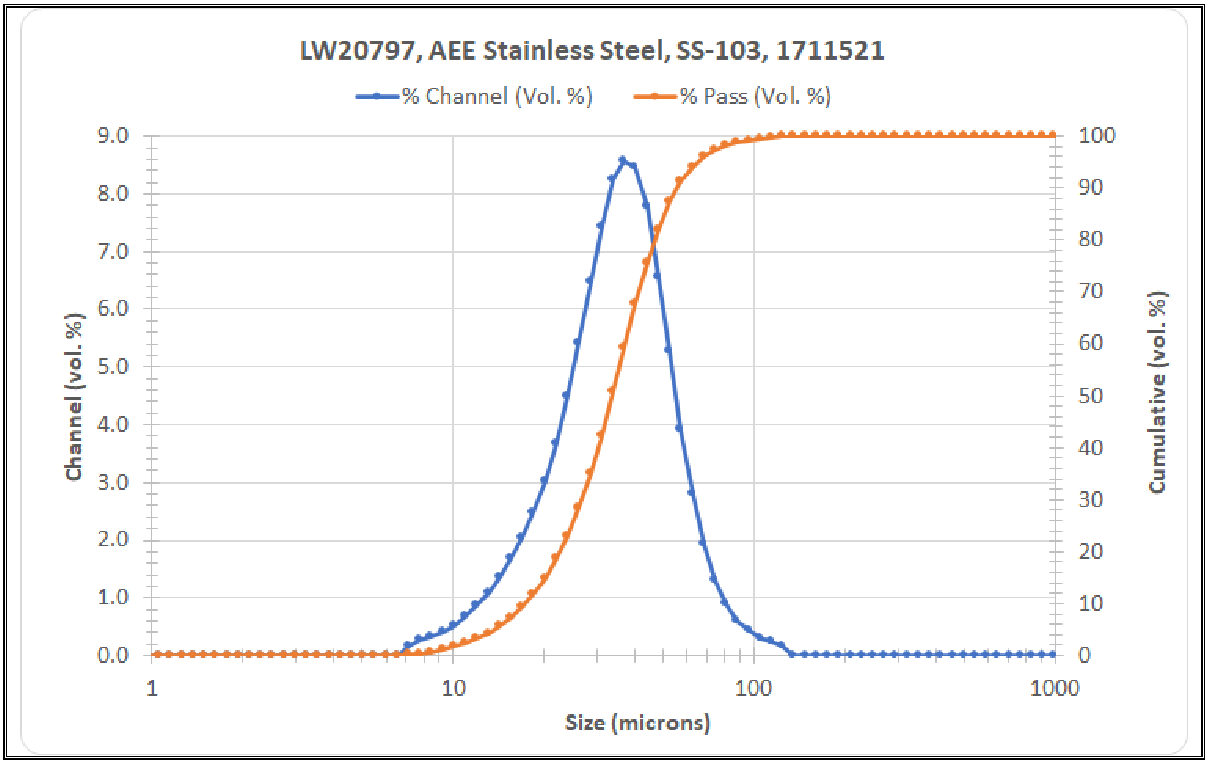


Figure A- 11 Atlantic Engineering Equipment Stainless Steel SS-103, LW20797

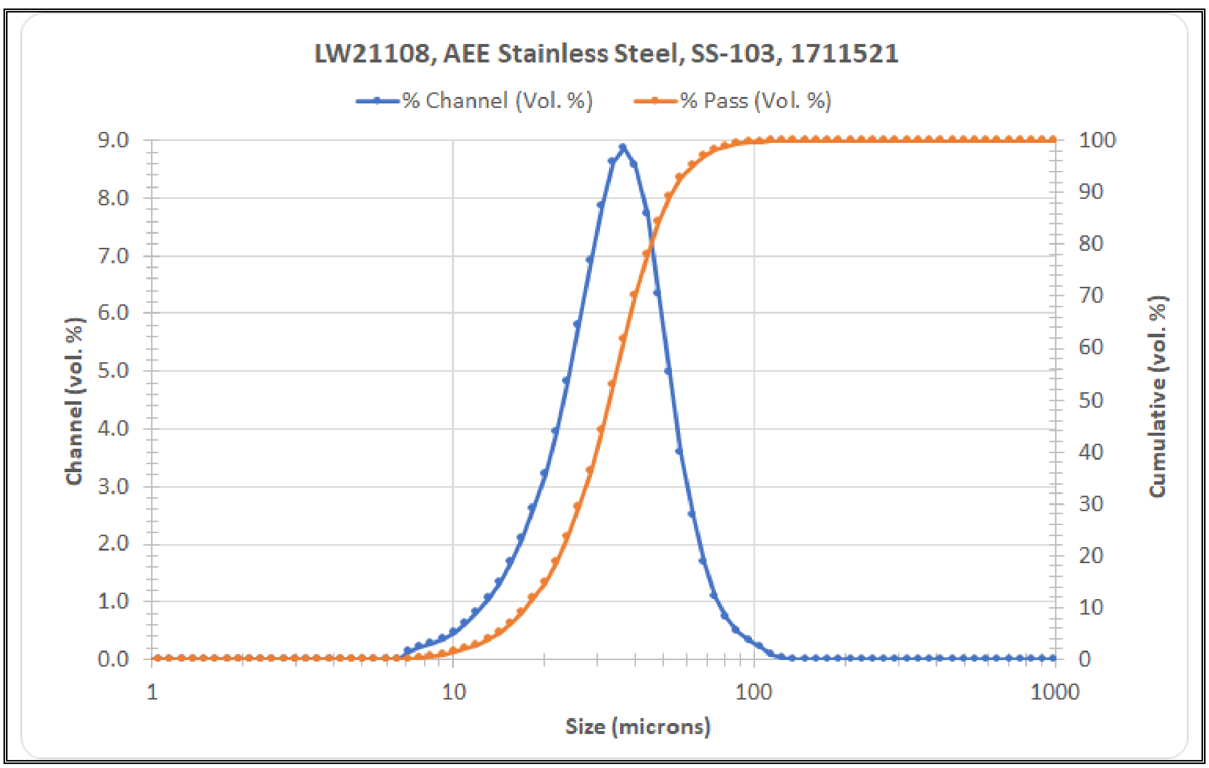


Figure A- 12 Atlantic Engineering Equipment Stainless Steel SS-103, LW21108

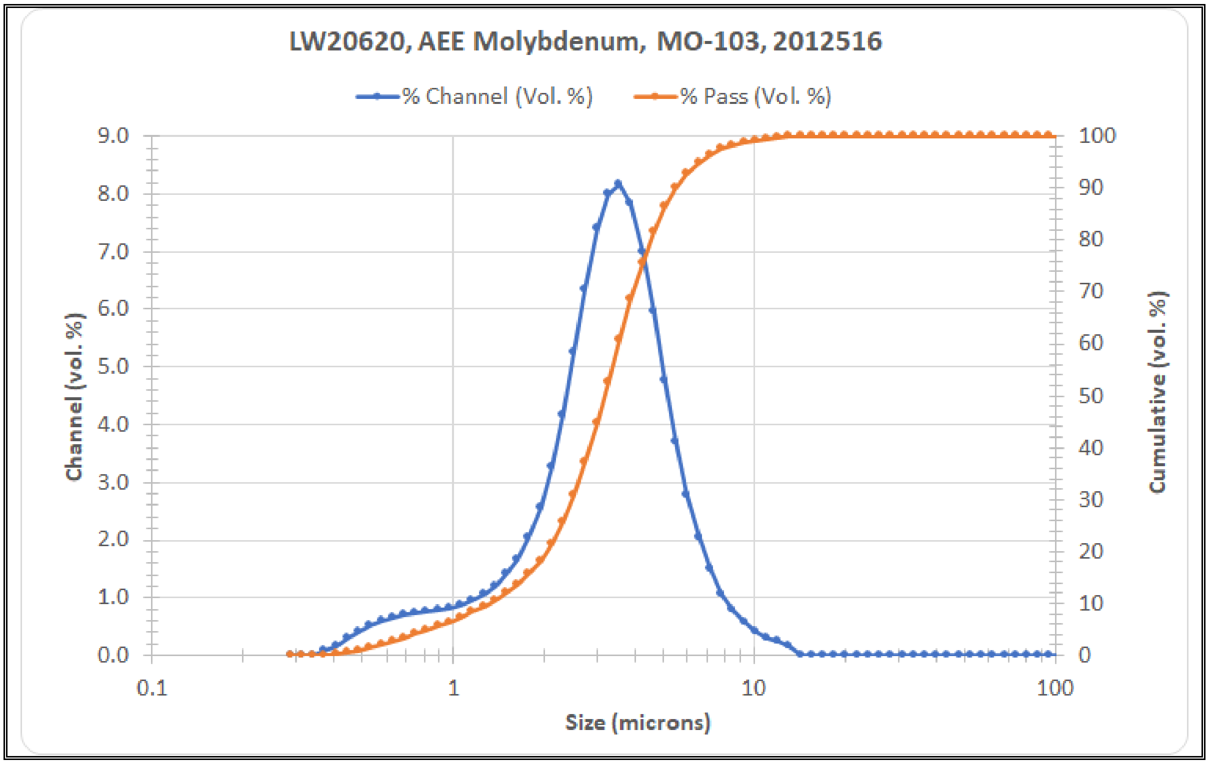


Figure A- 13 PSD Atlantic Engineering Equipment Molybdenum MO-103, 2012516

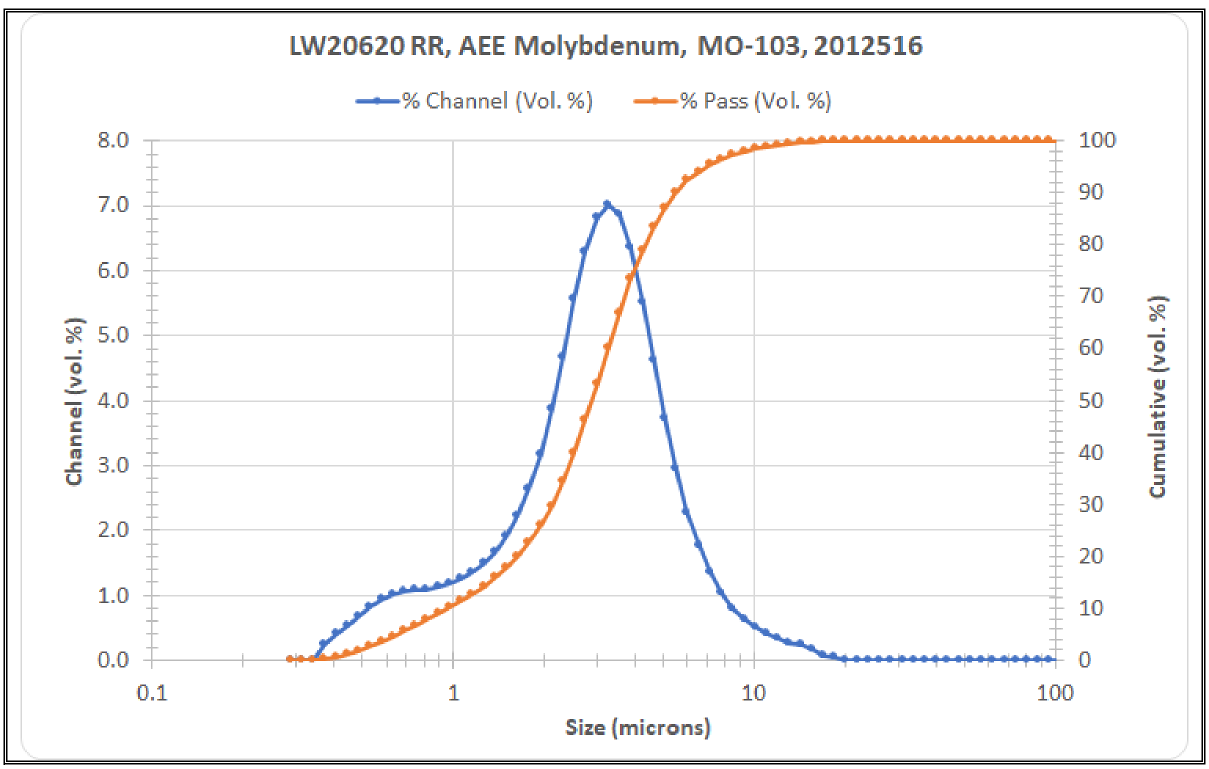


Figure A- 14 Atlantic Engineering Equipment Molybdenum MO-103, 2012516 RR

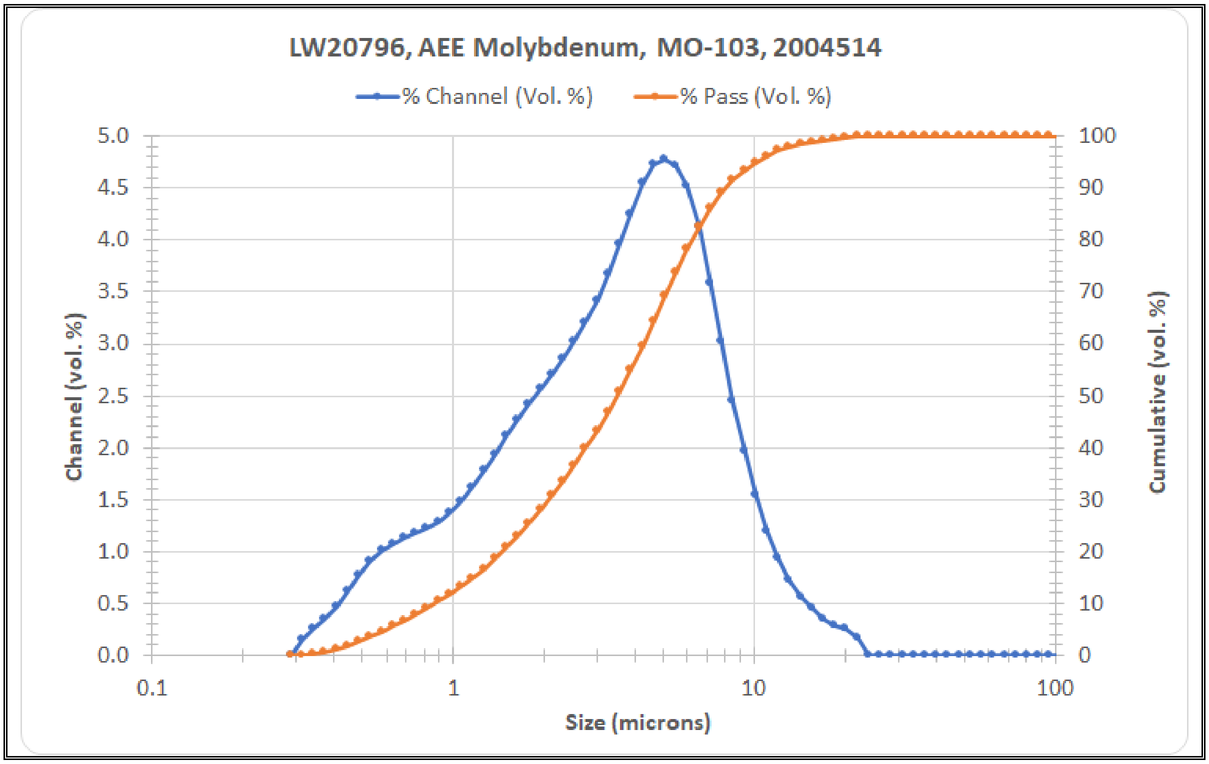


Figure A- 15 PSD Atlantic Engineering Equipment Molybdenum MO-103, 2004514

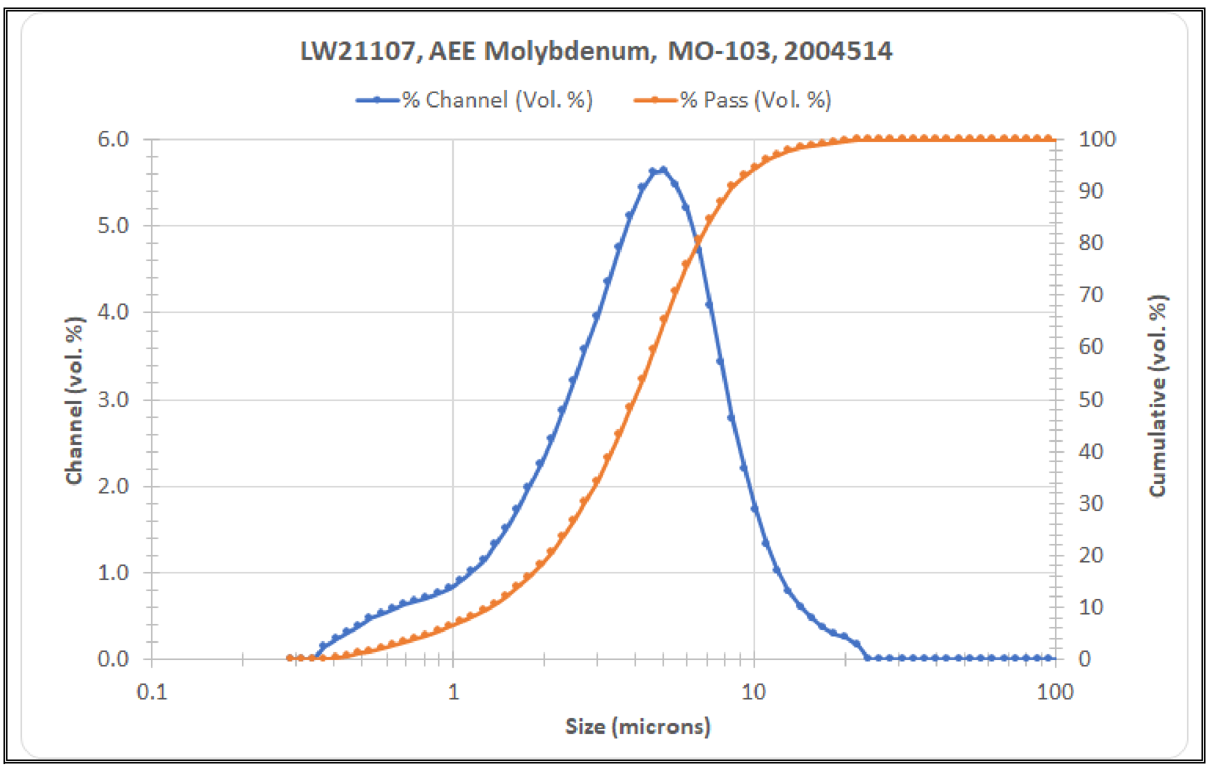


Figure A- 16 Atlantic Engineering Equipment Molybdenum MO-103, 2004514 RR

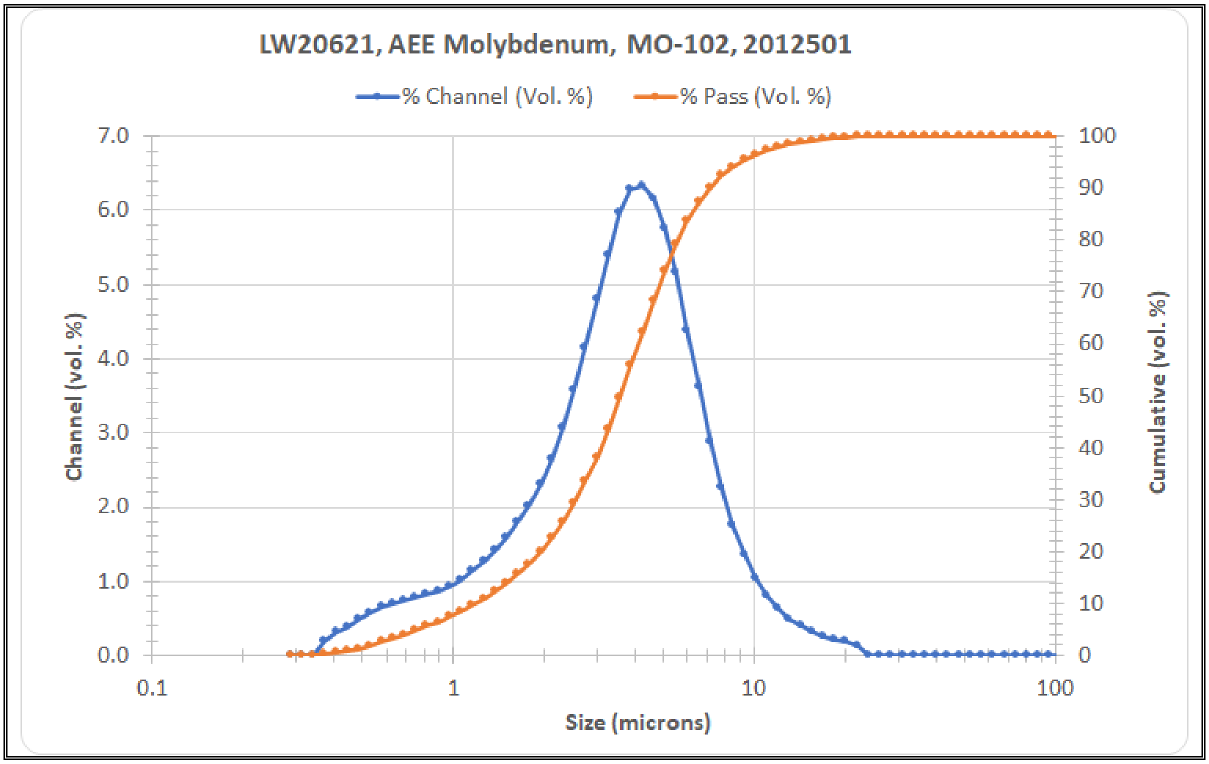


Figure A- 17 PSD Atlantic Engineering Equipment Molybdenum MO-102, 2012501

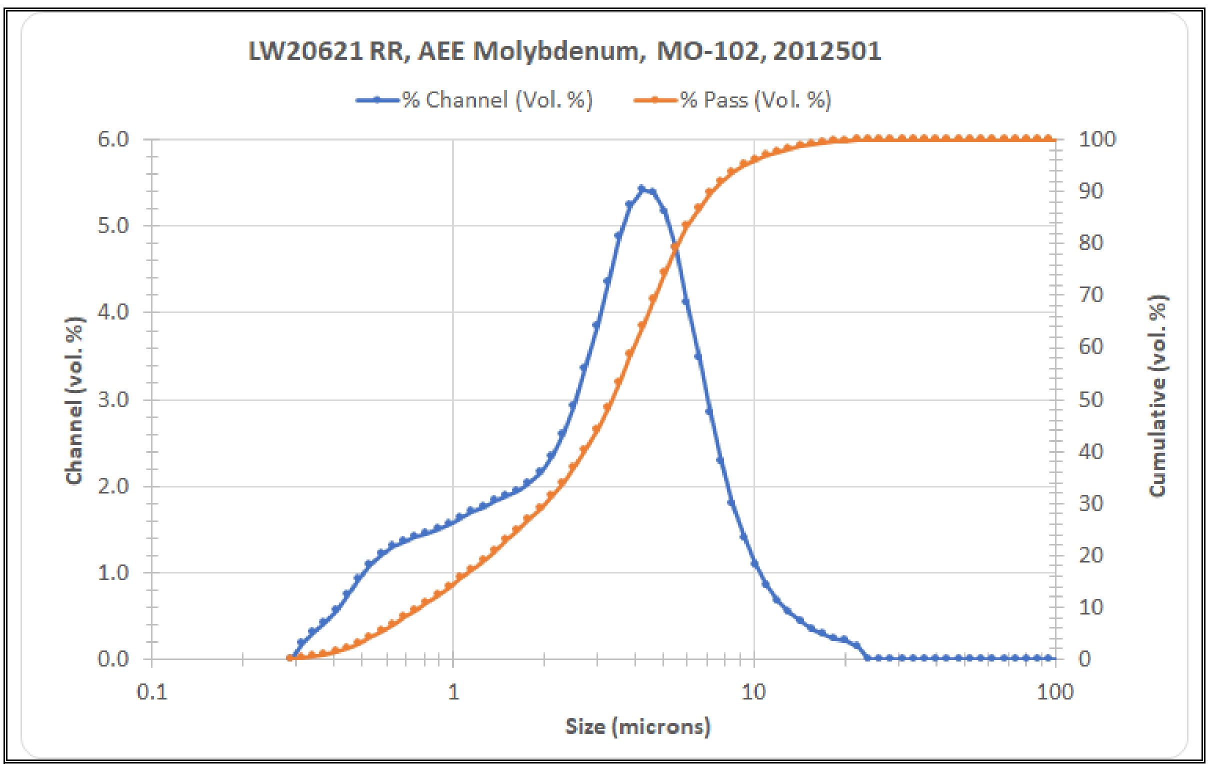


Figure A- 18 Atlantic Engineering Equipment Molybdenum MO-102, 2012501 RR

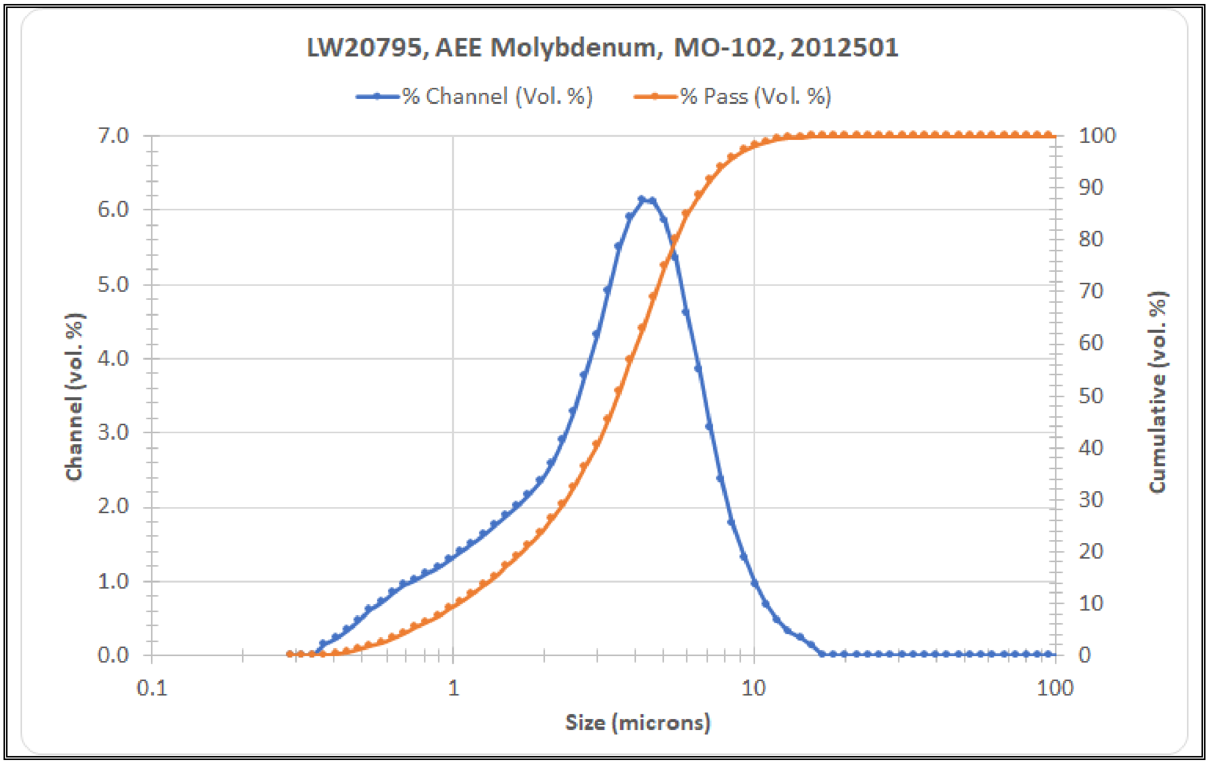


Figure A- 19 PSD Atlantic Engineering Equipment Molybdenum MO-102, LW20795

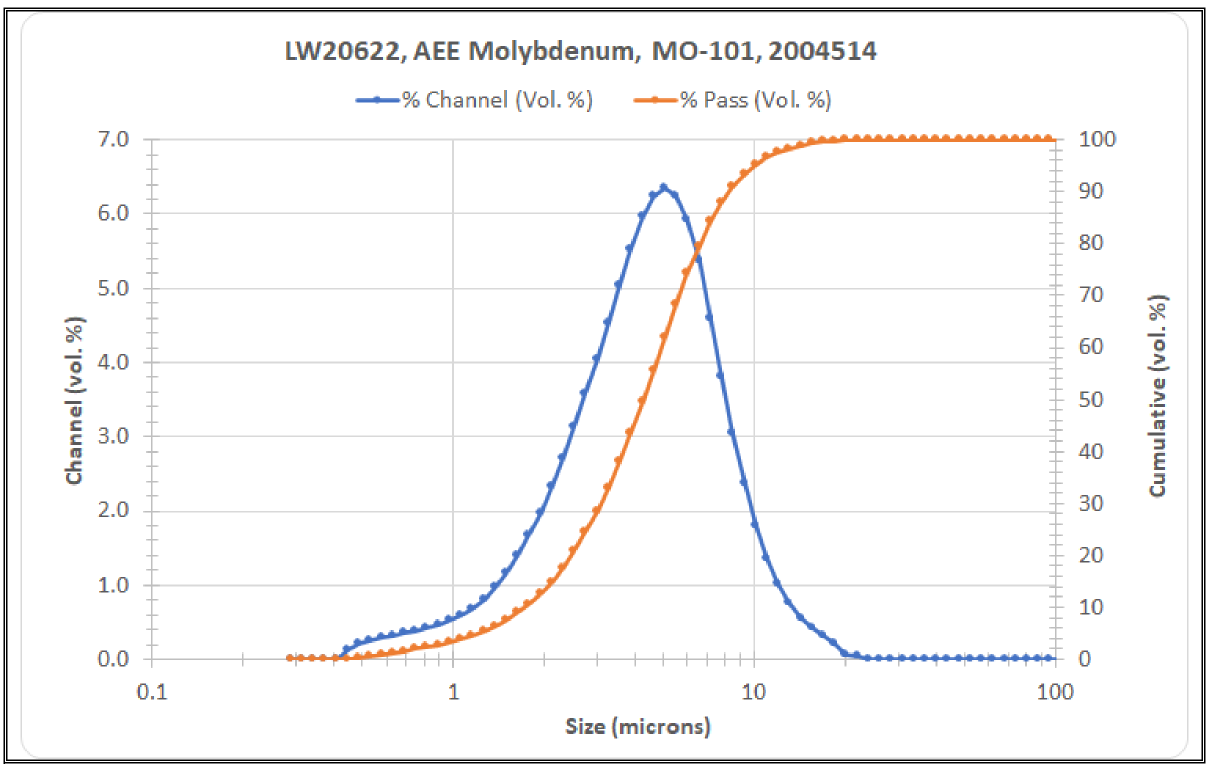


Figure A- 20 Atlantic Engineering Equipment Molybdenum MO-101, 2004514

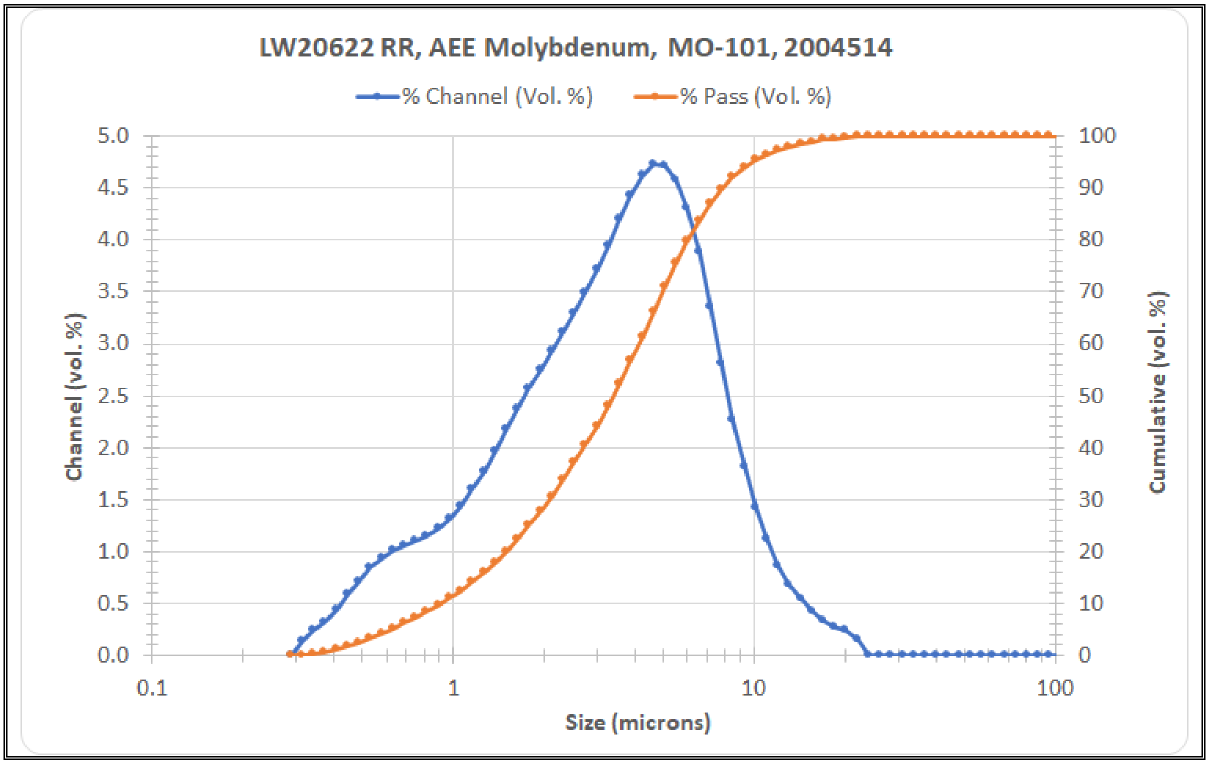


Figure A- 21 PSD Atlantic Engineering Equipment Molybdenum MO-101, 2004514RR

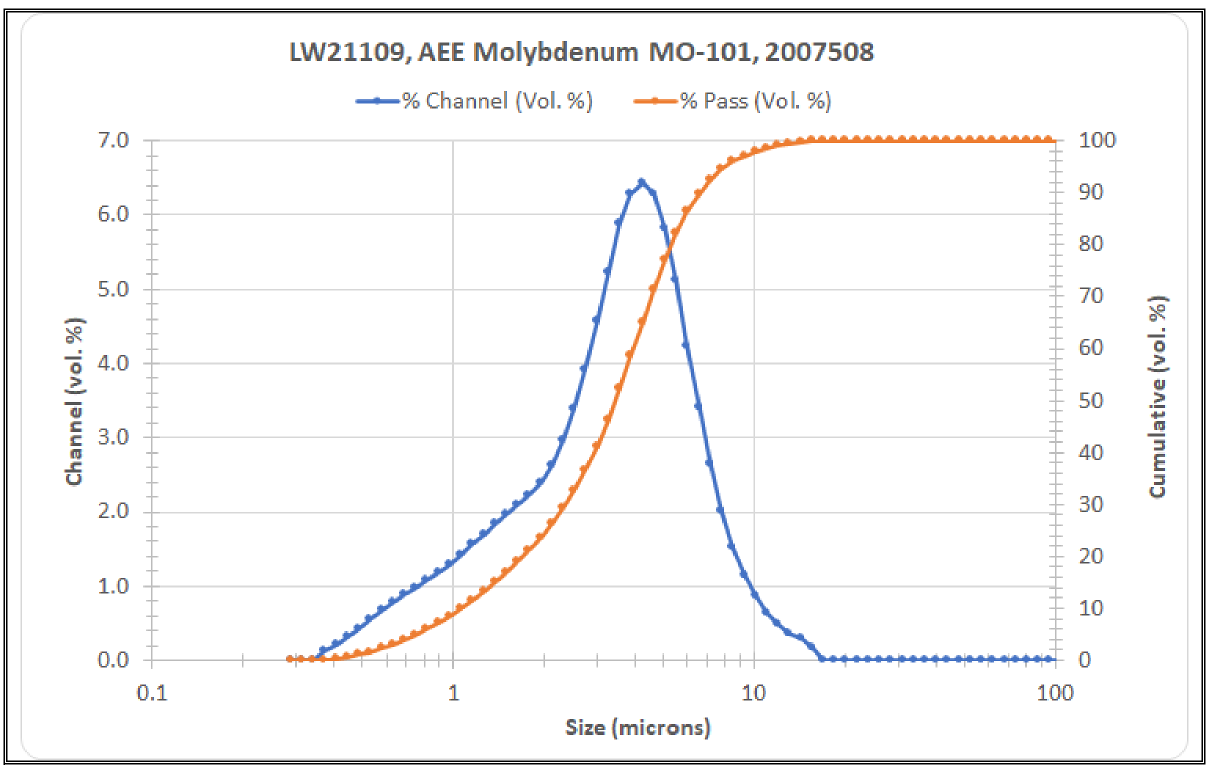


Figure A- 22 Atlantic Engineering Equipment Molybdenum MO-101, 2007508

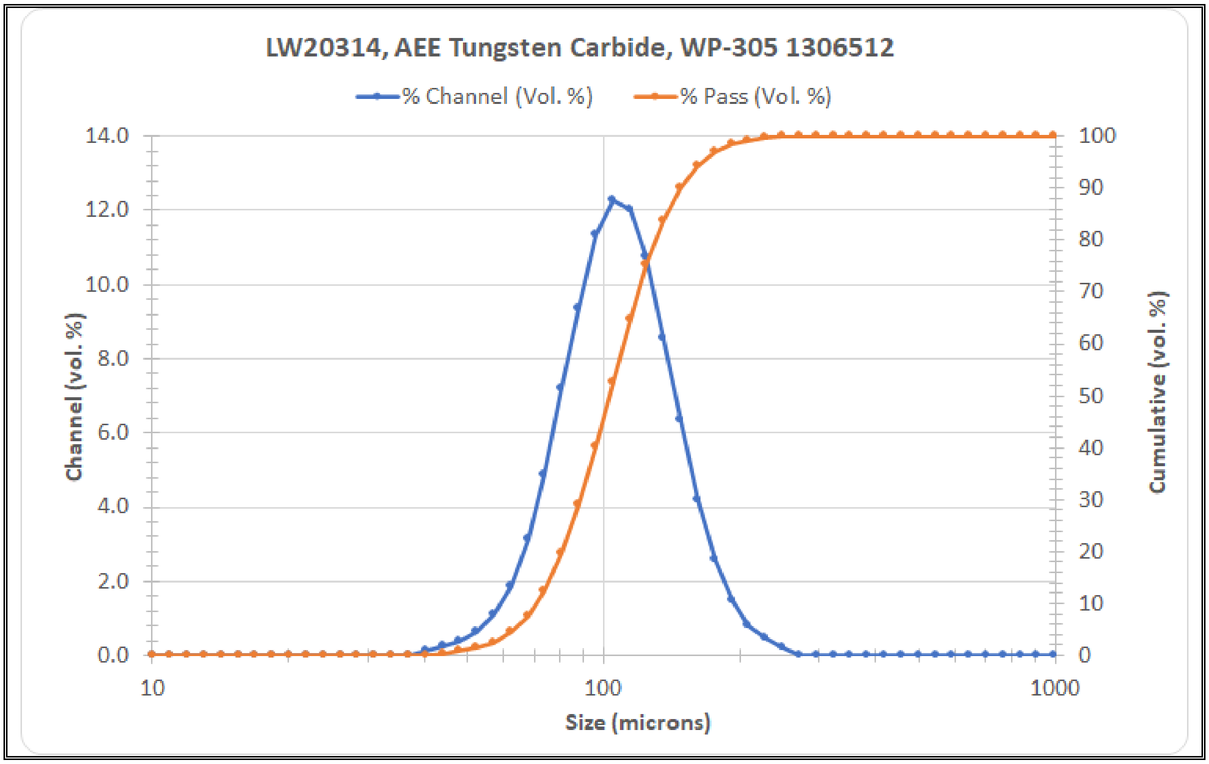


Figure A- 23 PSD Atlantic Engineering Equipment Tungsten Carbide WP-305, 1306512

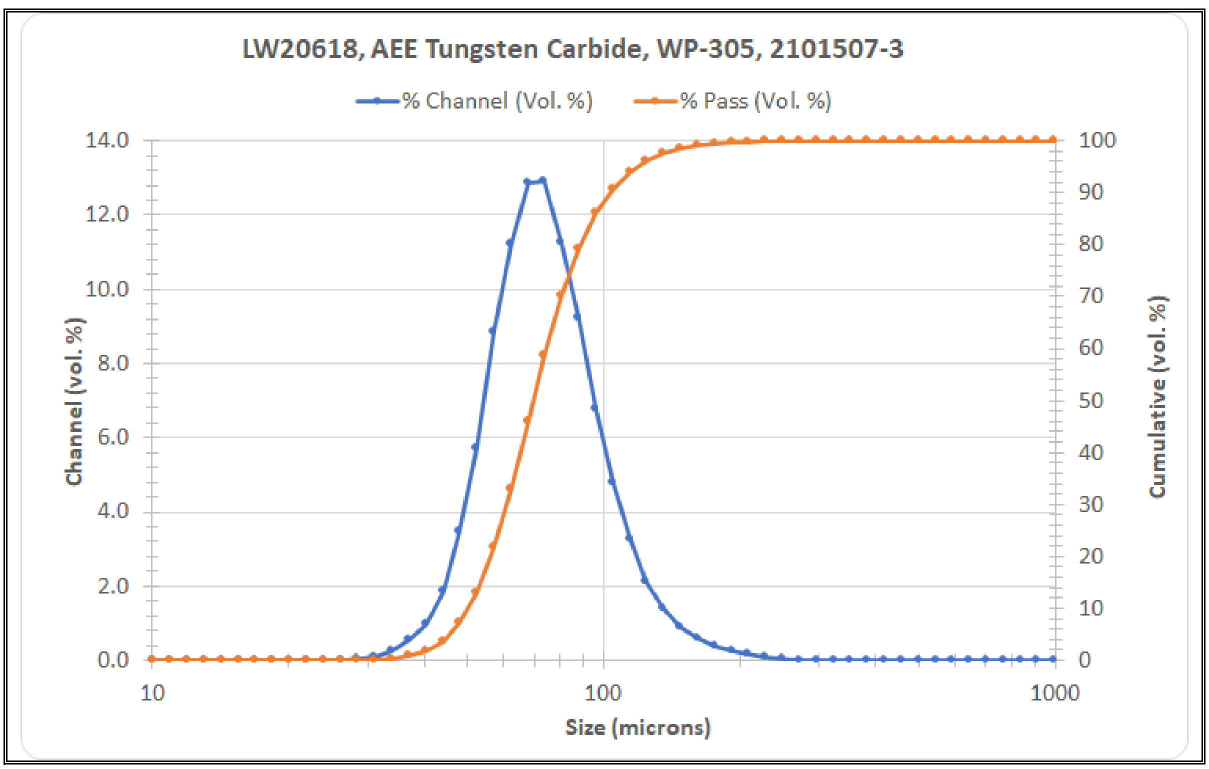


Figure A- 24 PSD Atlantic Engineering Equipment Tungsten Carbide WP-305, 2101507-3

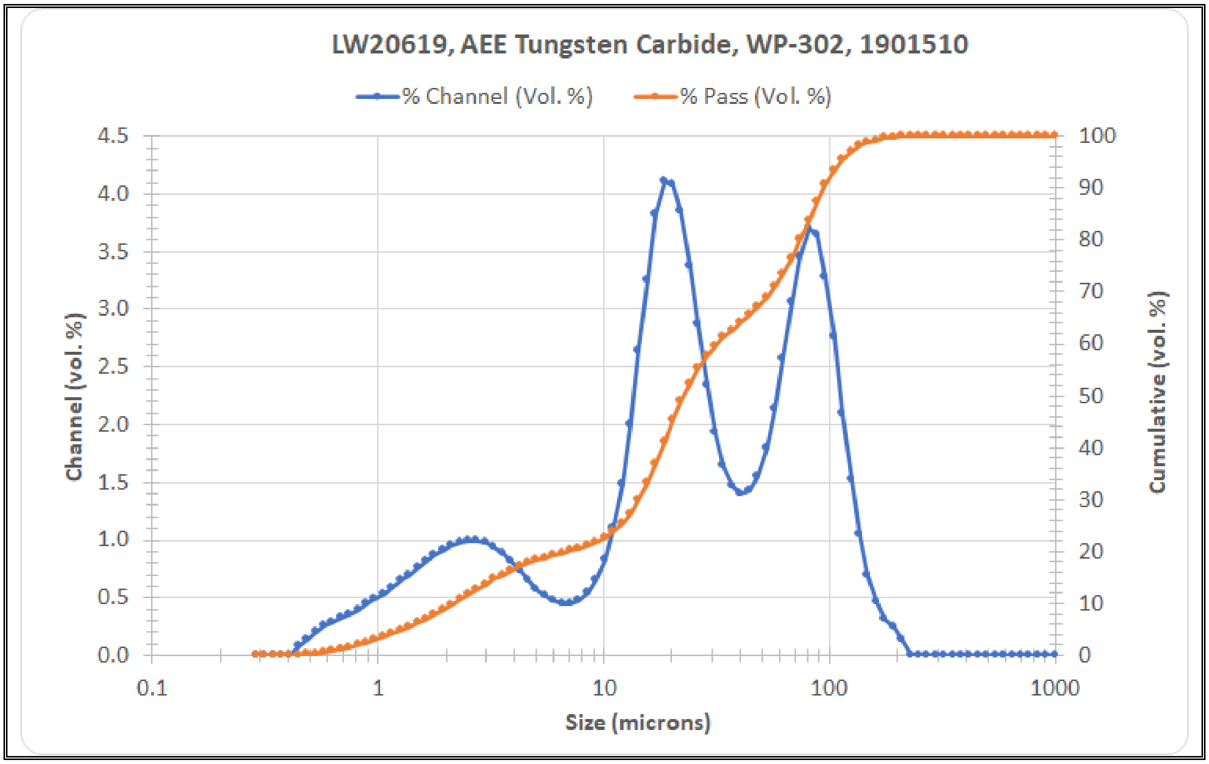


Figure A- 25 PSD Atlantic Engineering Equipment Tungsten Carbide WP-302, 1901510

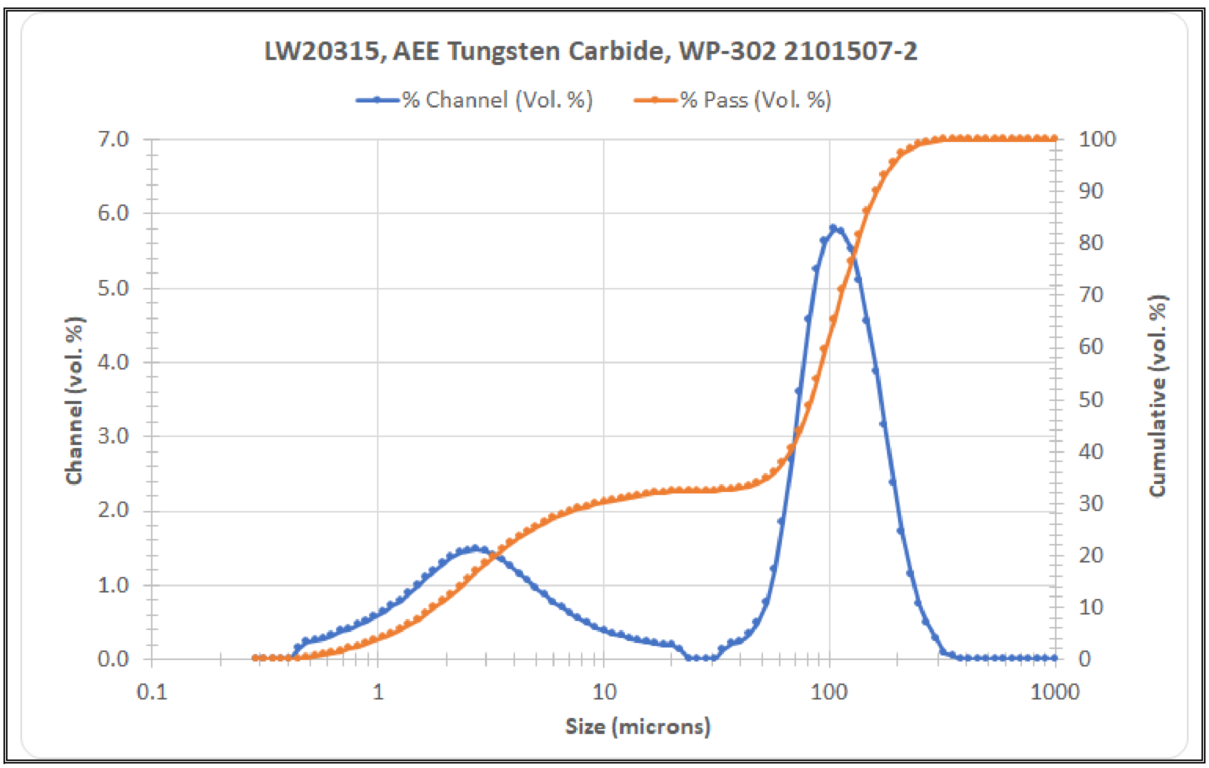


Figure A- 26 PSD Atlantic Engineering Equipment Tungsten Carbide WP-302, 2101507-2

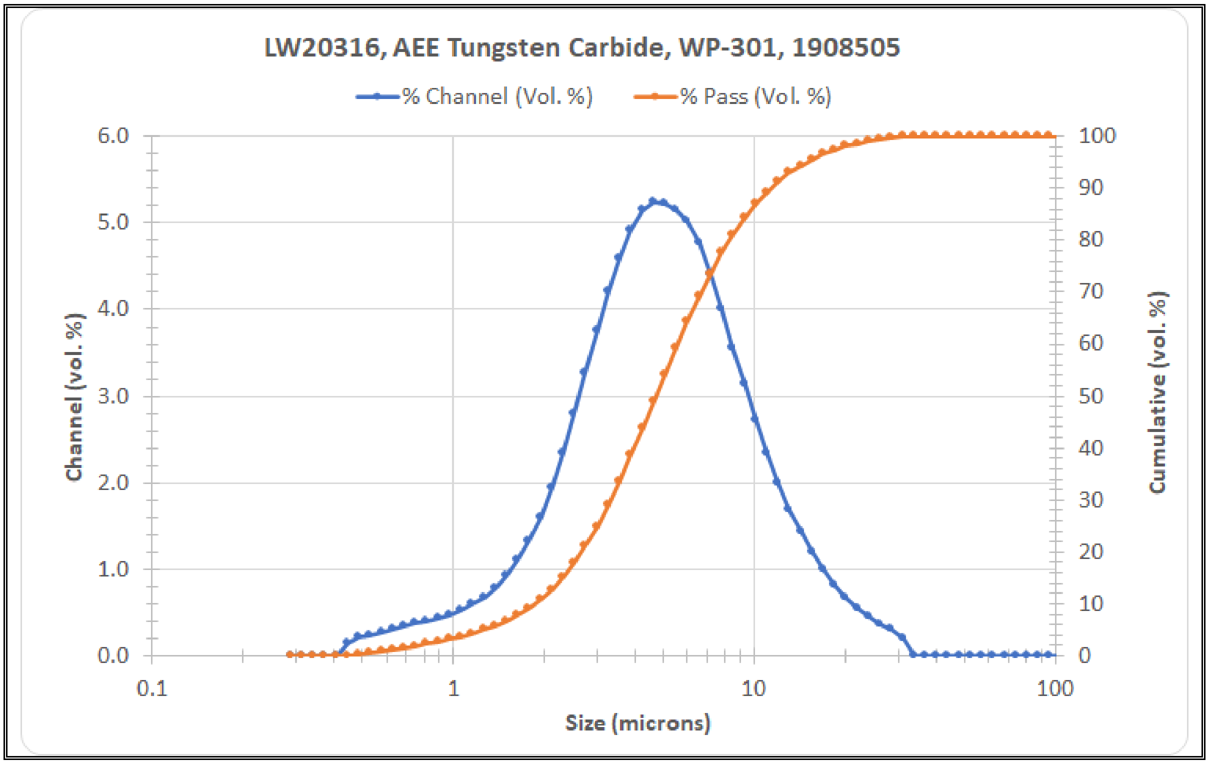


Figure A- 27 PSD Atlantic Engineering Equipment Tungsten Carbide WP-301, 1908505

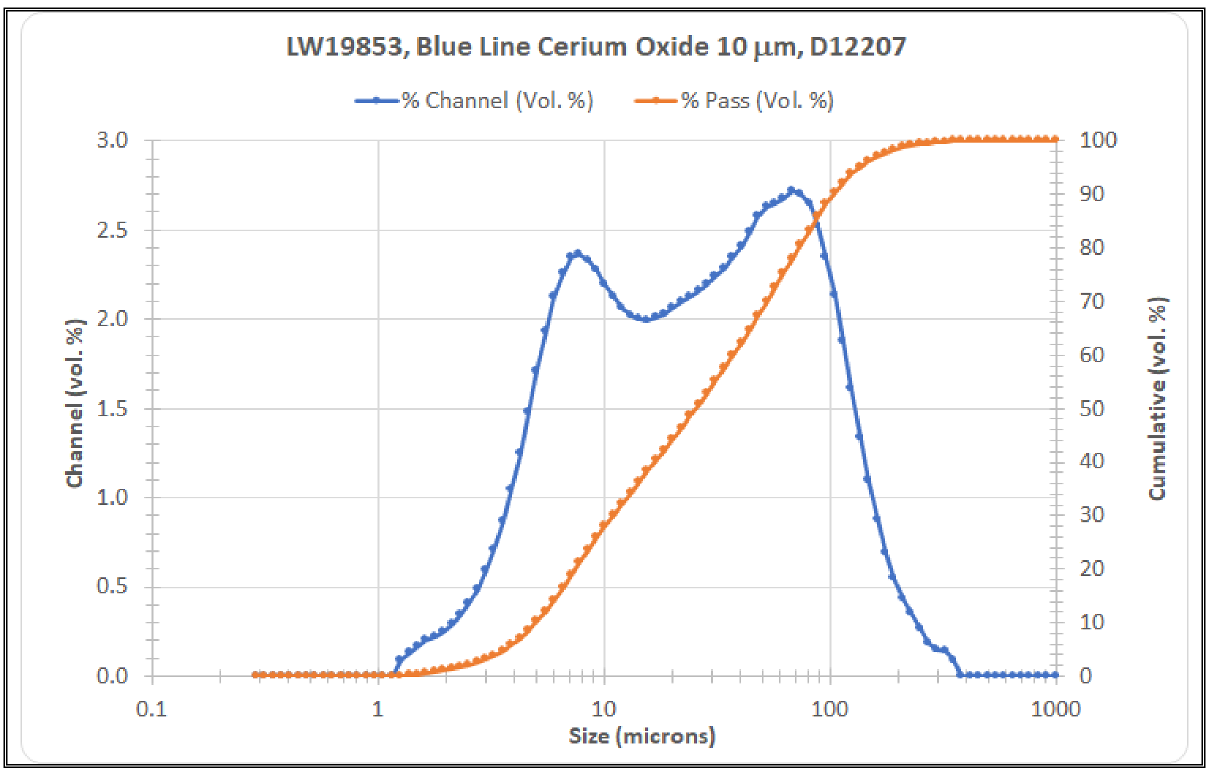


Figure A- 28 PSD Blue Line Cerium Oxide 10 Microns, D12207

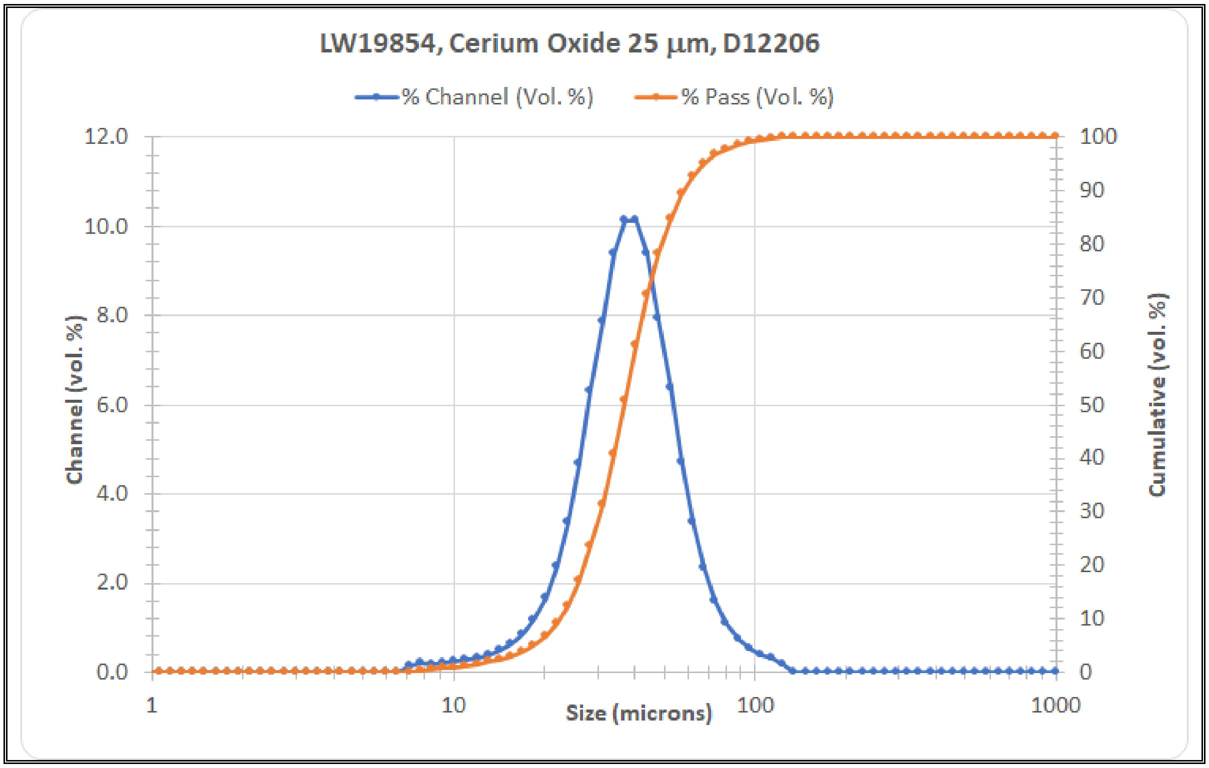


Figure A- 29 Blue Line Cerium Oxide 25 Microns, D12207

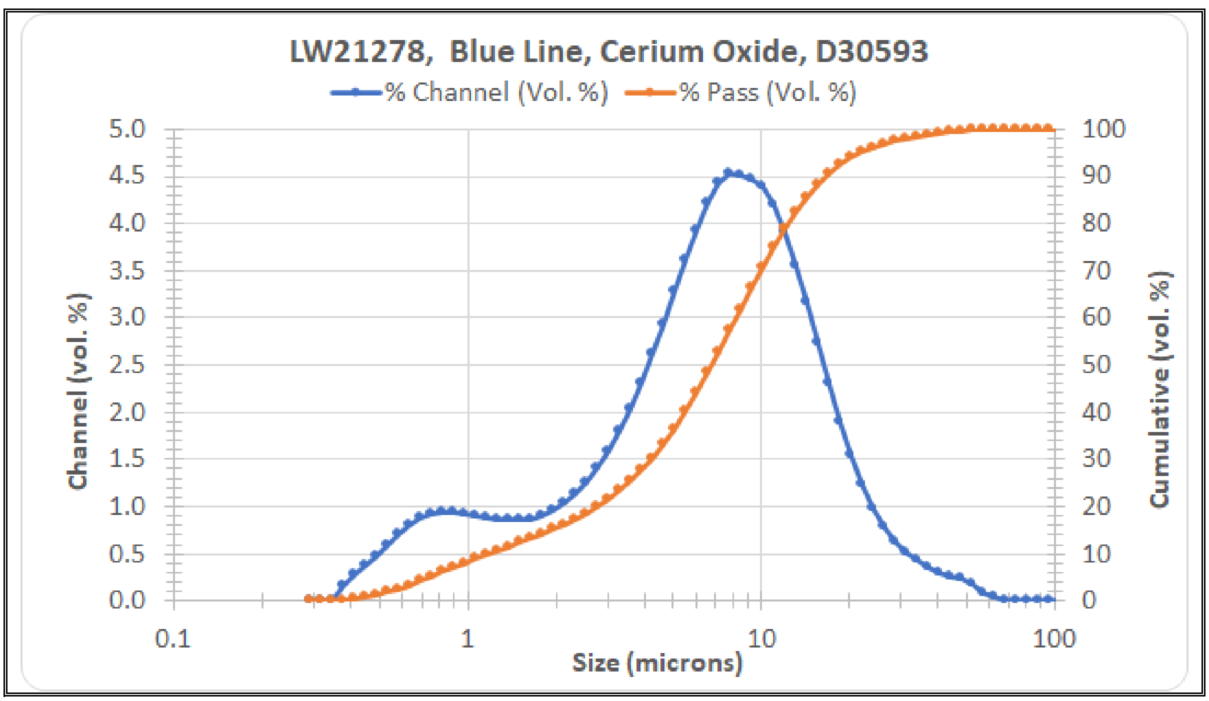


Figure A- 30 PSD Blue Line Cerium Oxide 8.4 Microns, D30593 (PSD From Vendor)

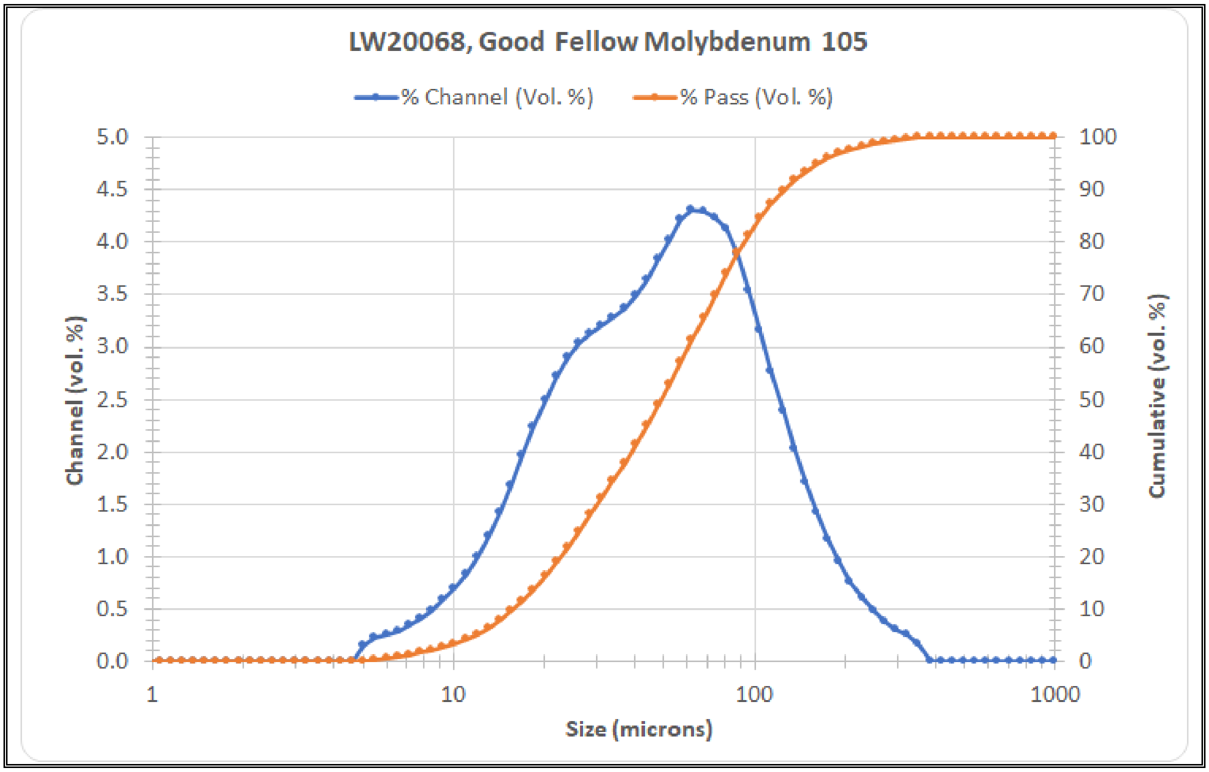


Figure A- 31 Good Fellow Molybdenum 105

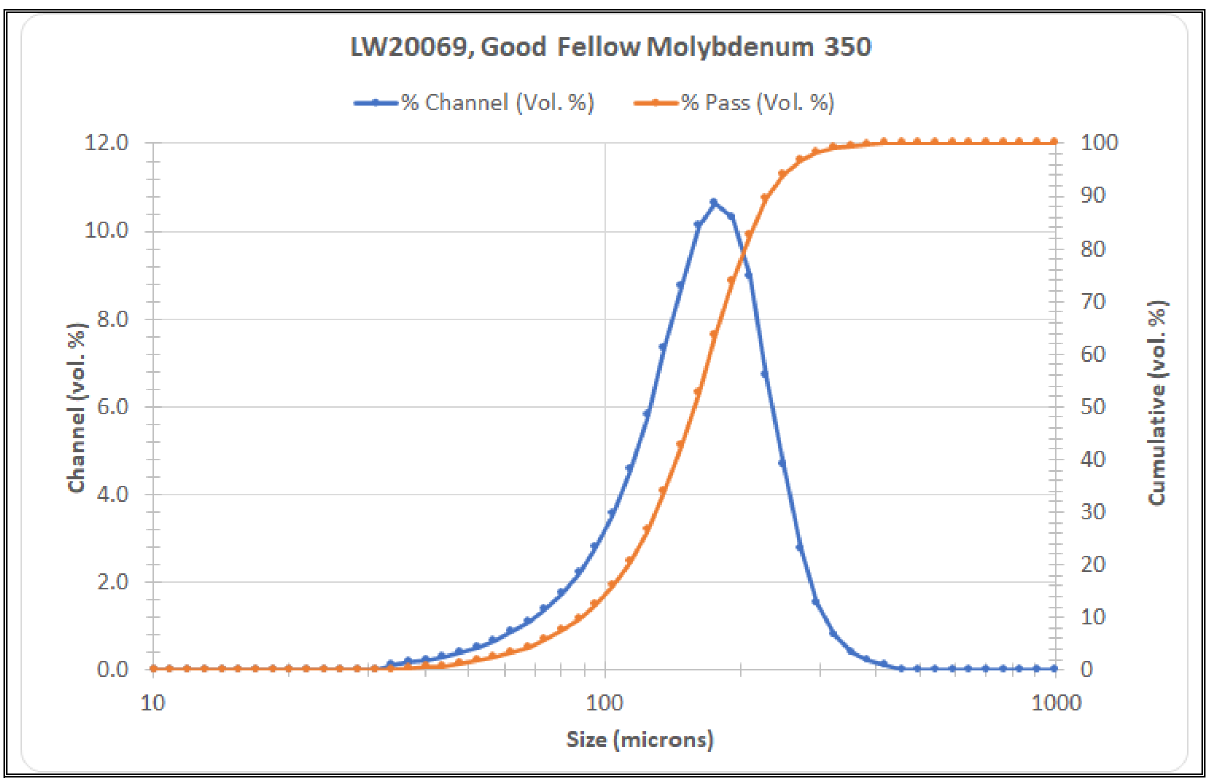


Figure A- 32 Good Fellow Molybdenum 350

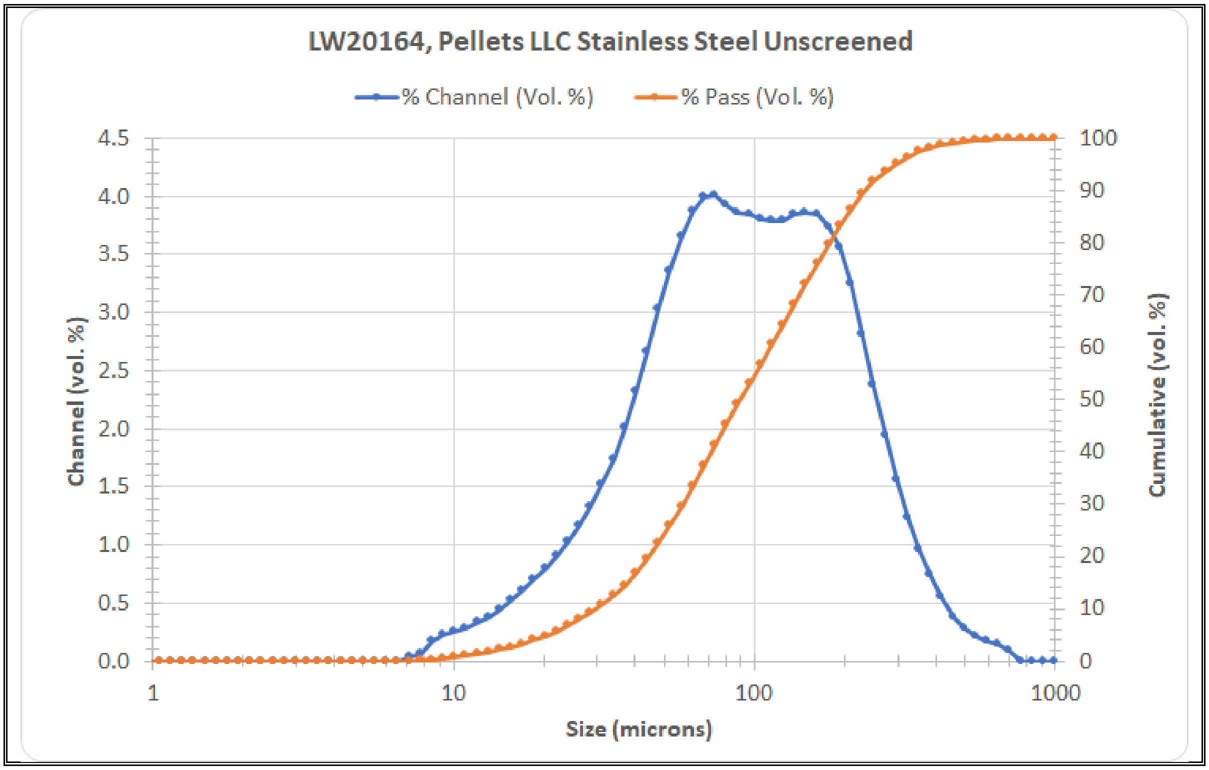


Figure A- 33 Pellets Stainless Steel Unscreened

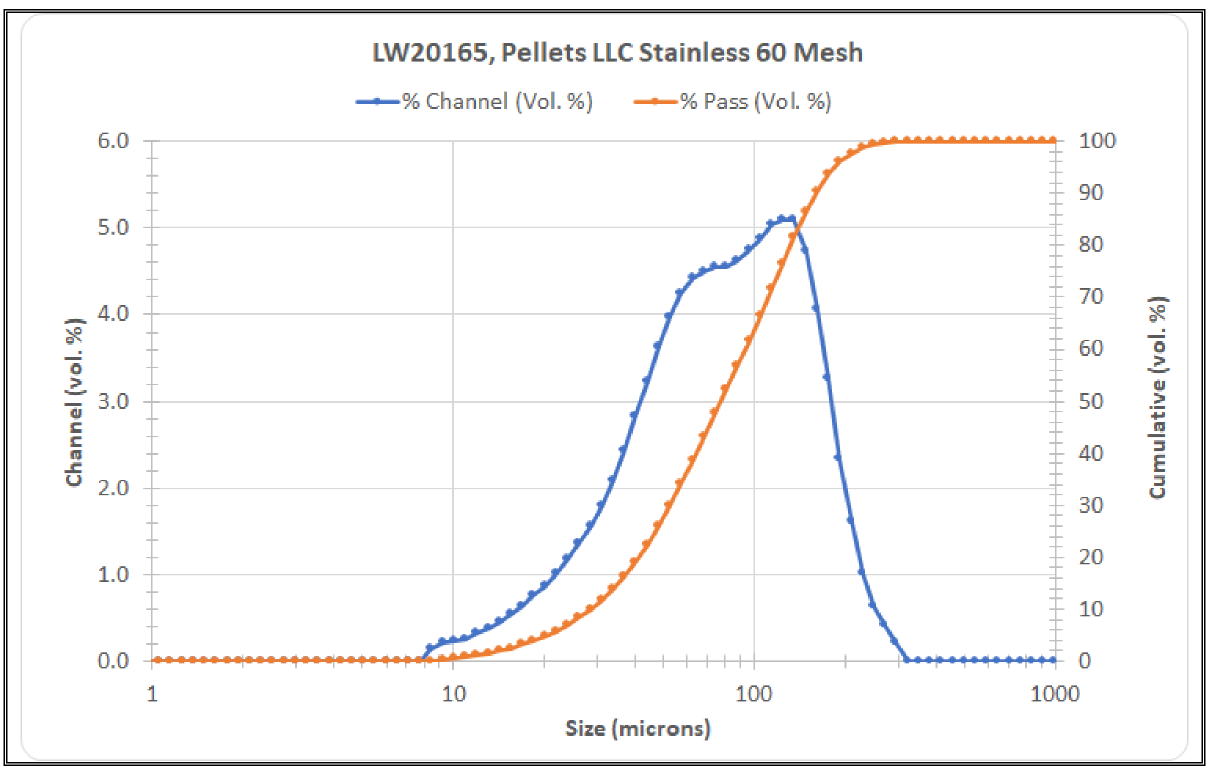


Figure A- 34 Pellets Stainless Steel 60 Mesh

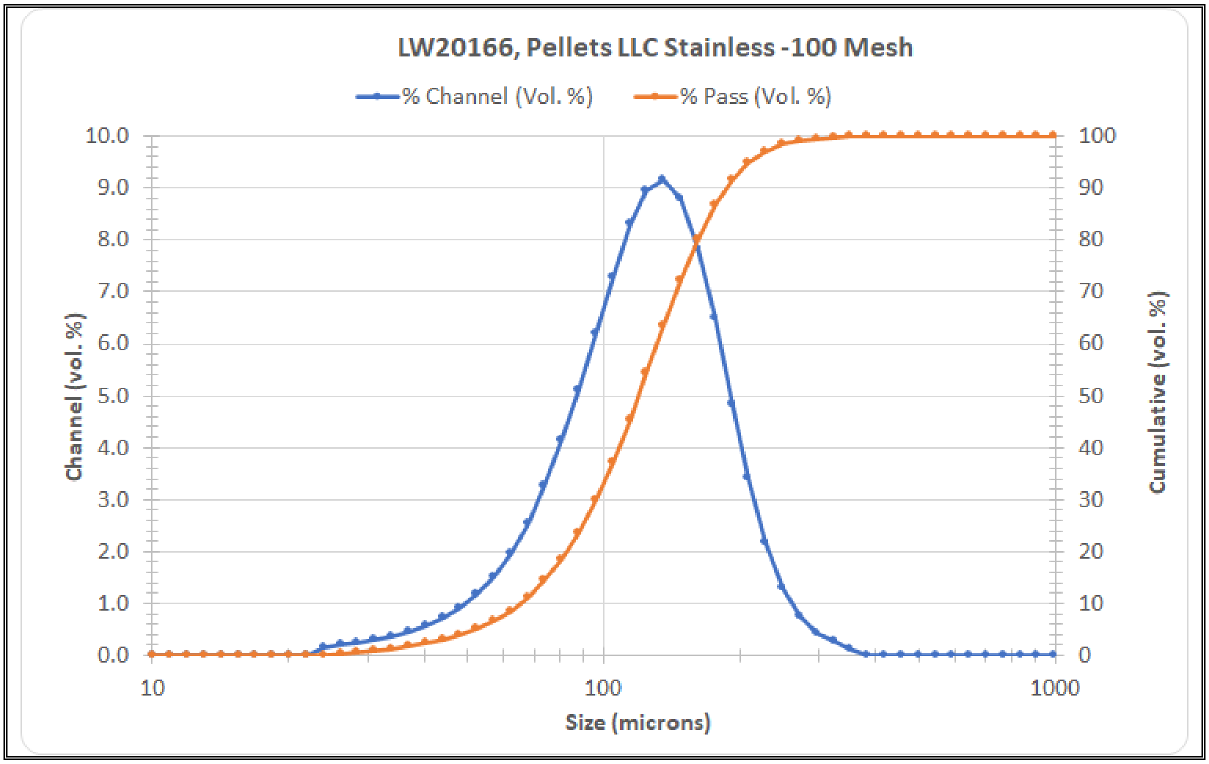


Figure A- 35 Pellets Stainless Steel -100 Mesh

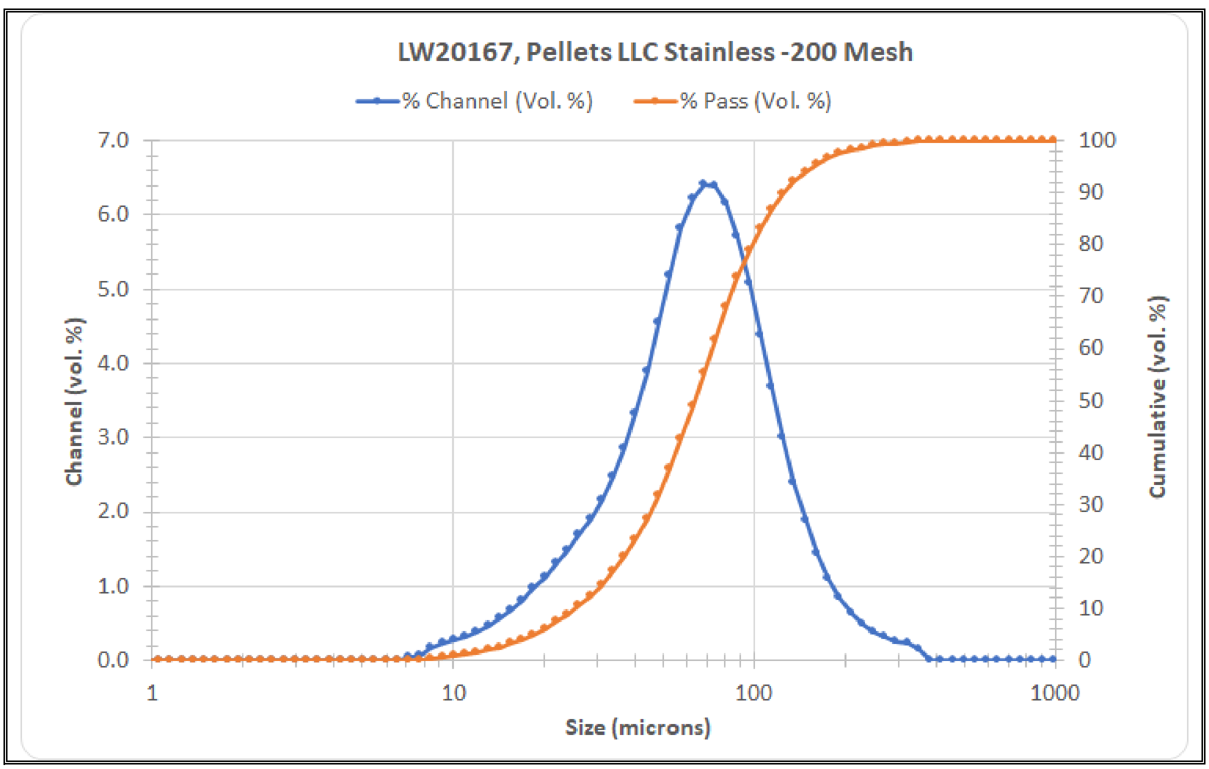


Figure A- 36 Pellets Stainless Steel -200 Mesh

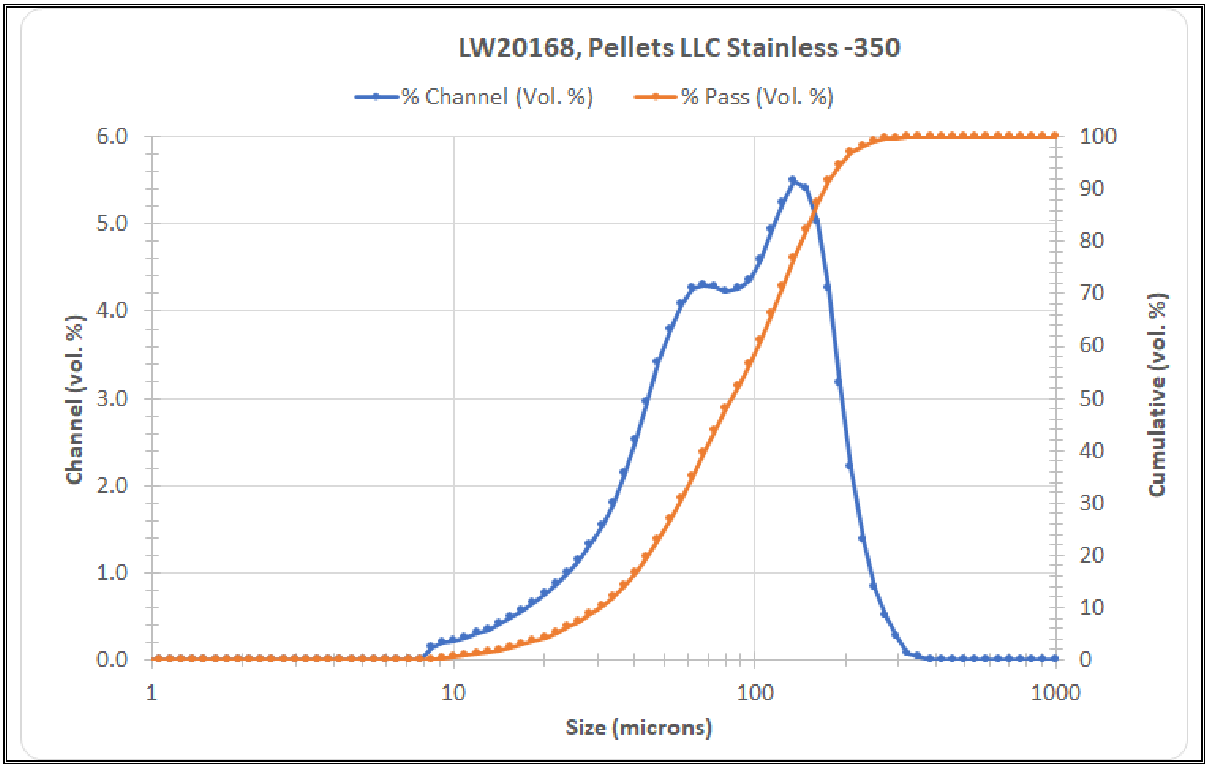


Figure A- 37 Pellets Stainless Steel -350

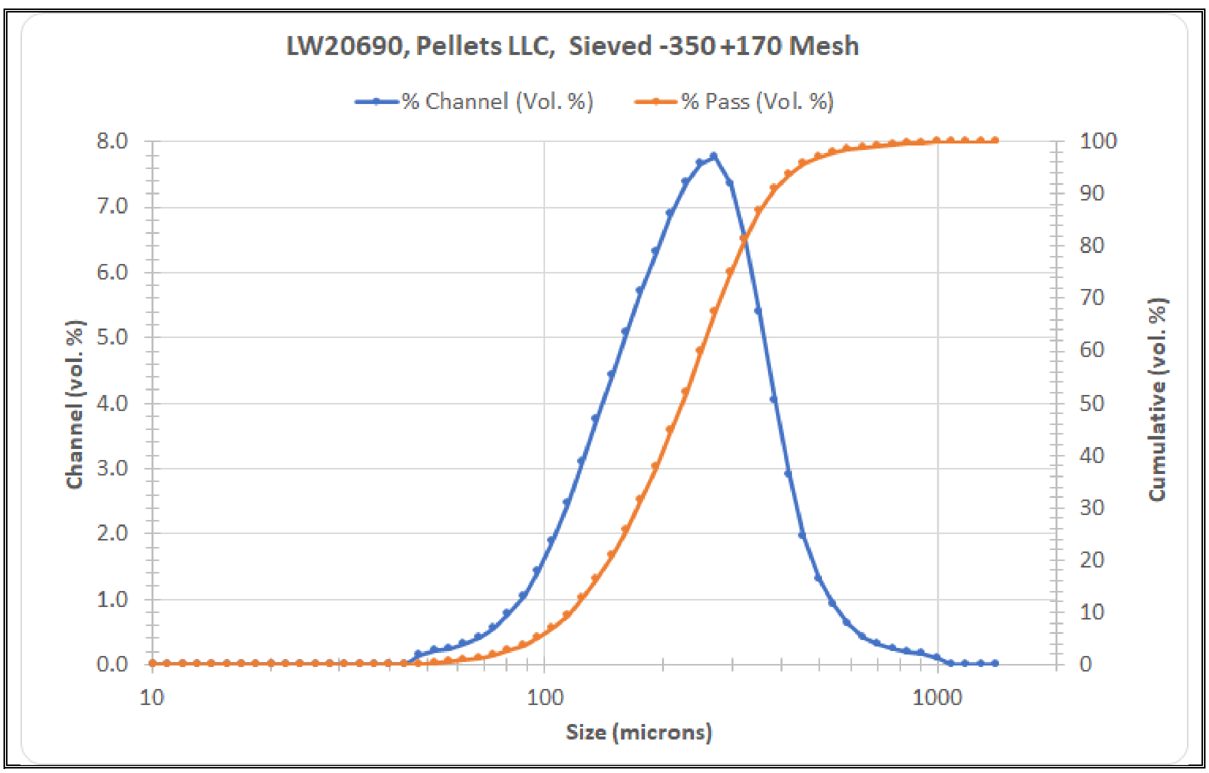


Figure A- 38 Pellets Stainless Steel -350, +170 Mesh (sieved)

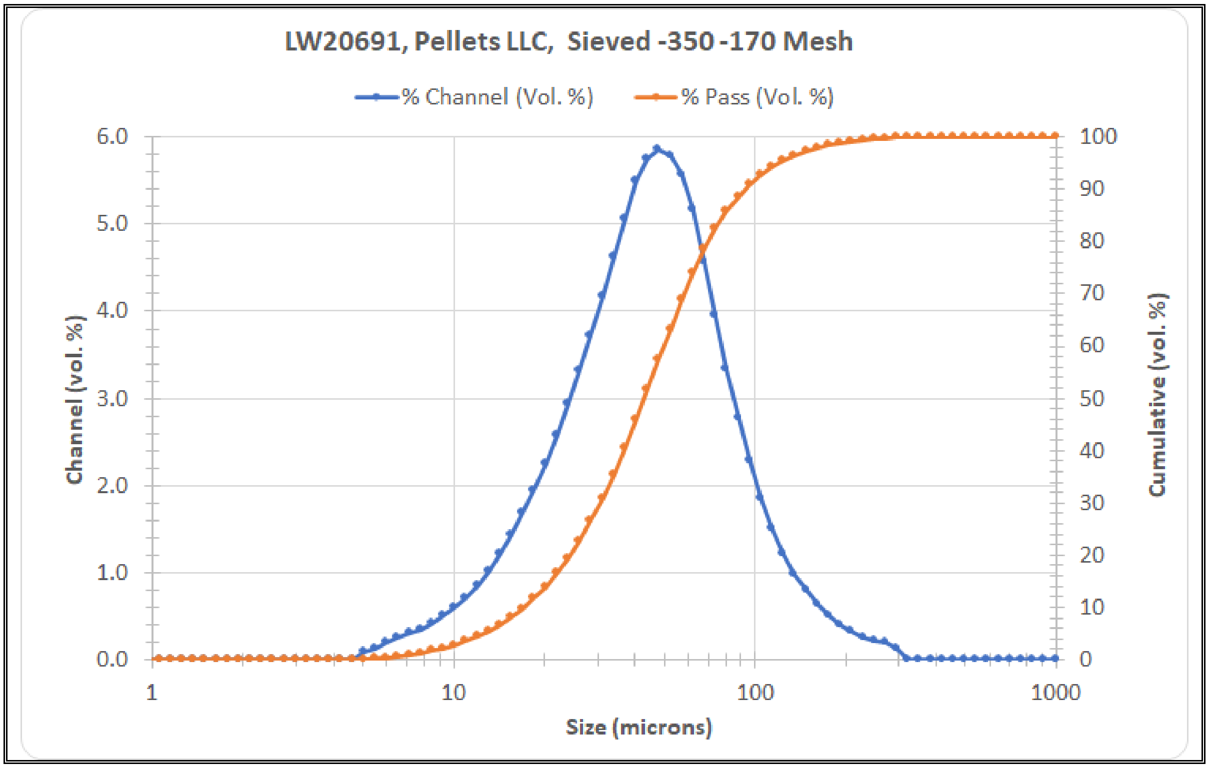


Figure A- 39 Pellets Stainless Steel -350, -170 Mesh (sieved)

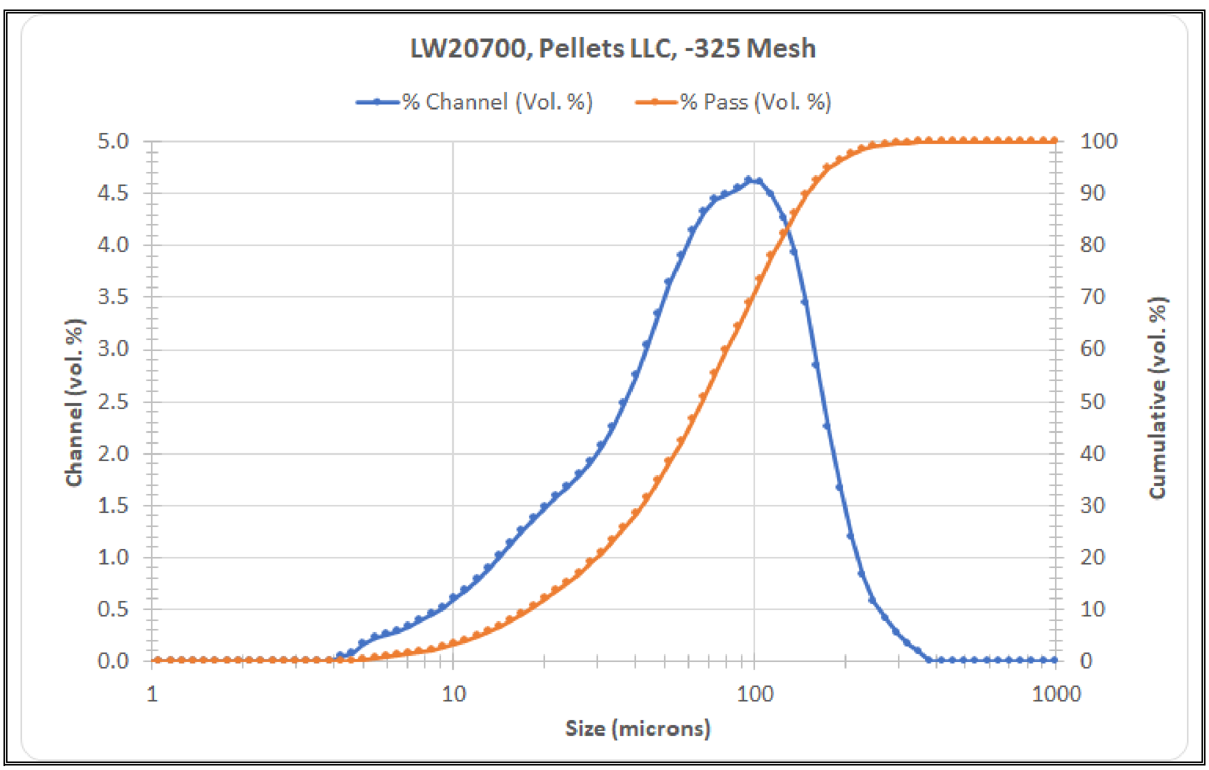


Figure A- 40 Pellets Stainless Steel -325 Mesh

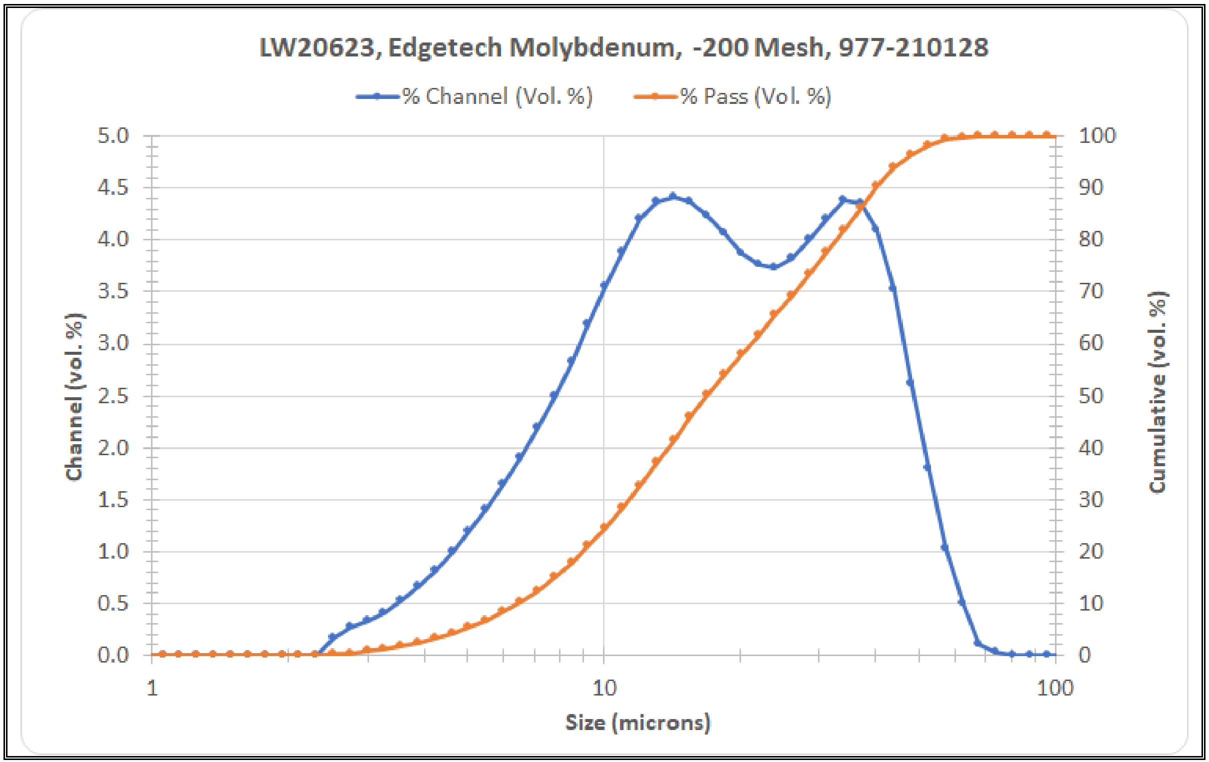


Figure A- 41 Edgetech Molybdenum, -200 Mesh, 977-210128

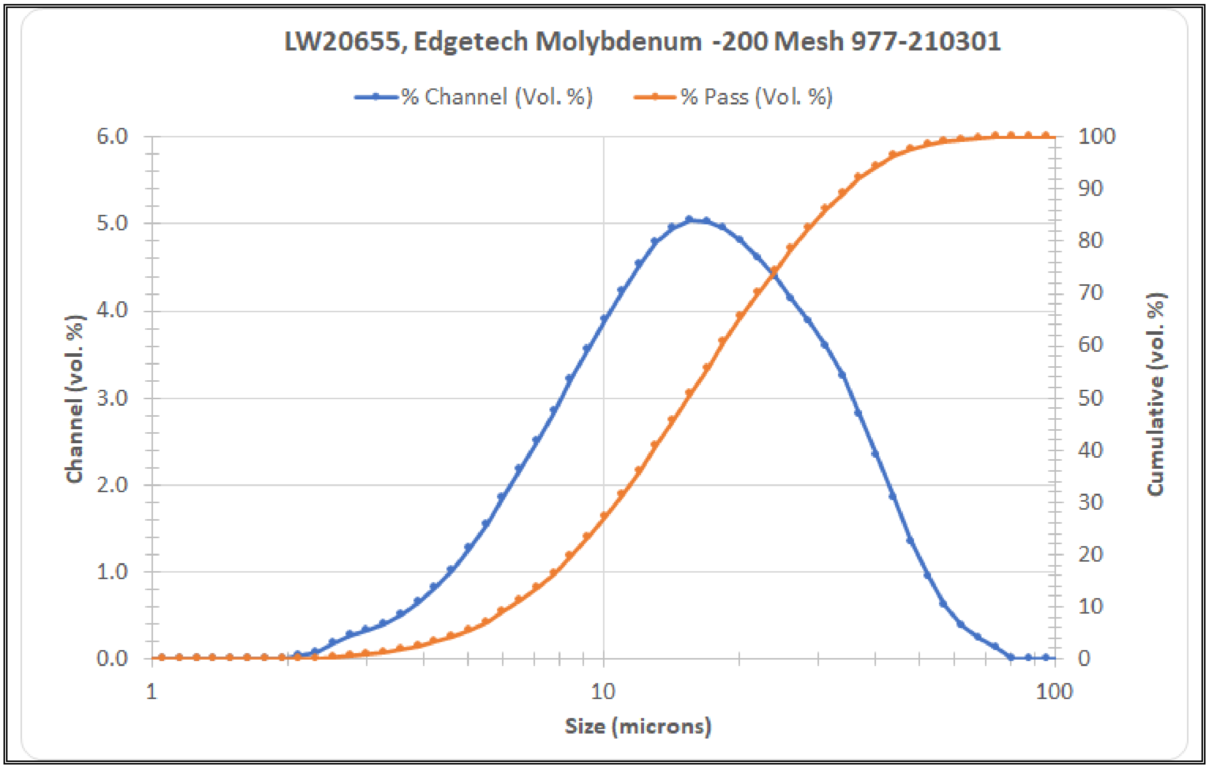


Figure A- 42 Edgetech Molybdenum, -200 Mesh, 977-210301, LW20655

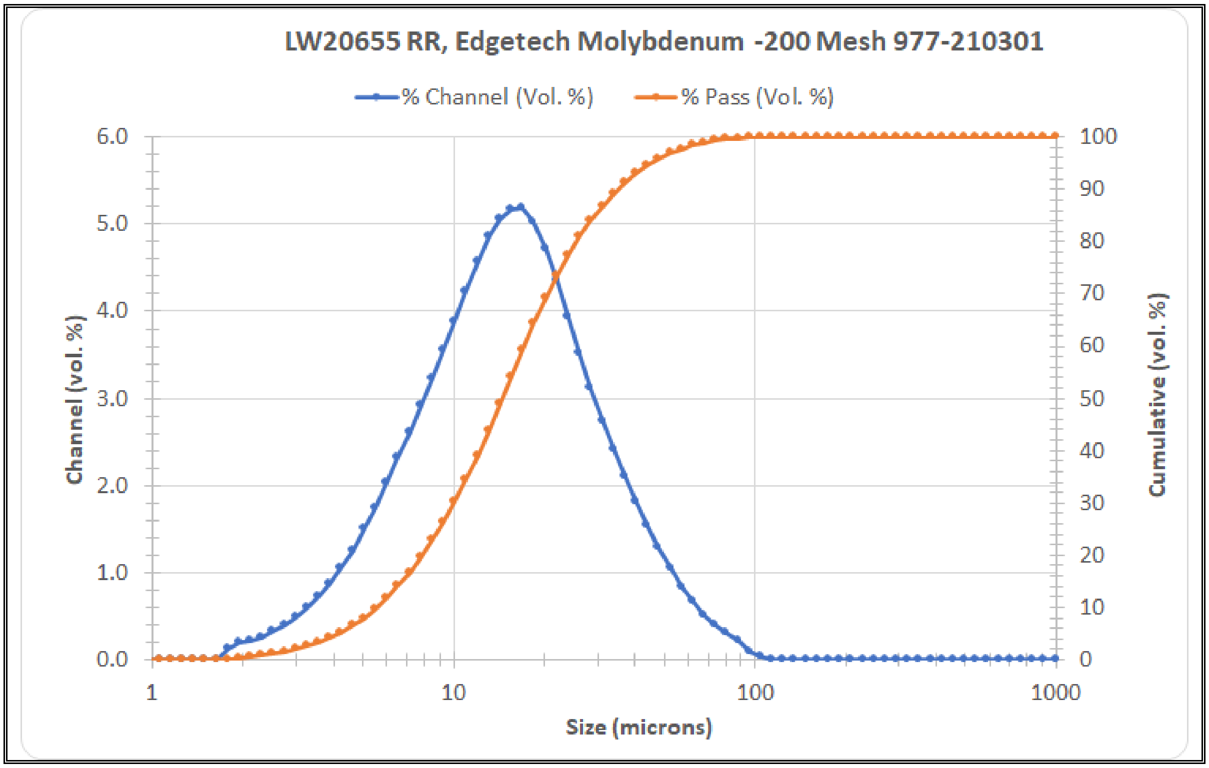


Figure A- 43 Edgetech Molybdenum, -200 Mesh, 977-210301, LW20655RR

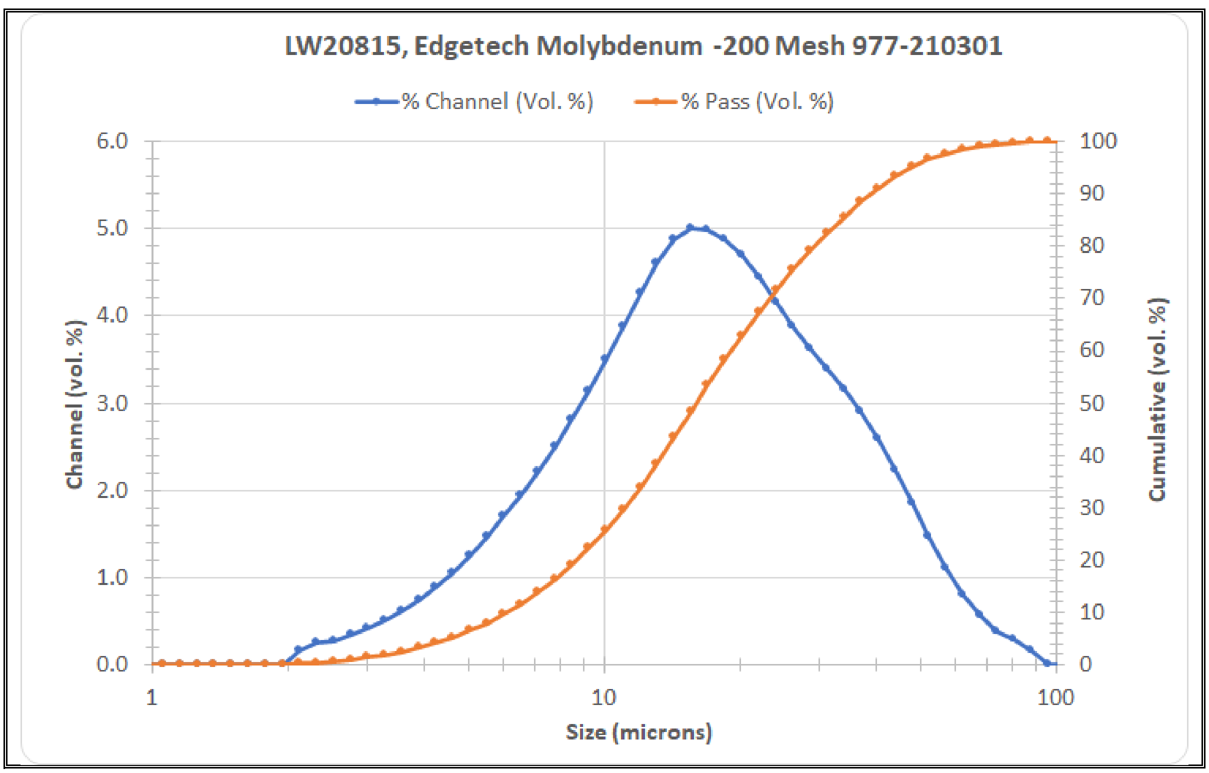


Figure A- 44 Edgetech Molybdenum, -200 Mesh, 977-210301, LW20815

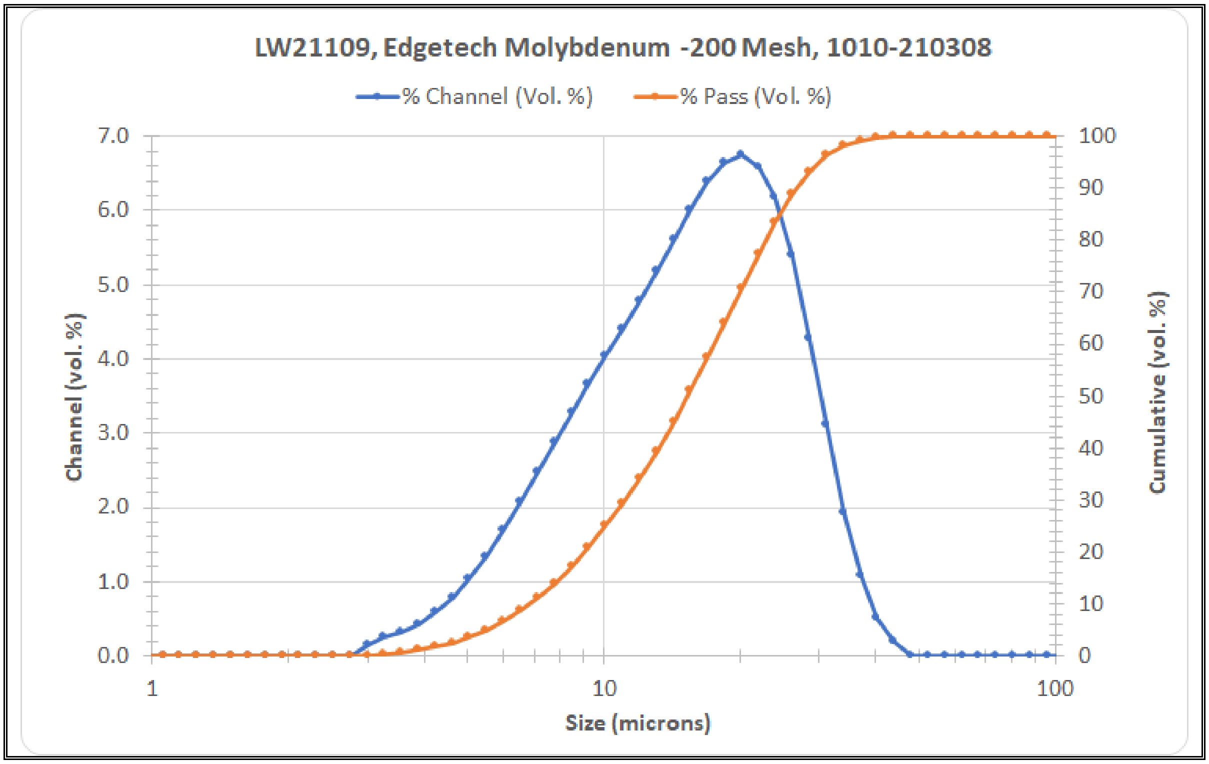


Figure A- 45 Edgetech Molybdenum, -200 Mesh, 1010-210308, LW21109

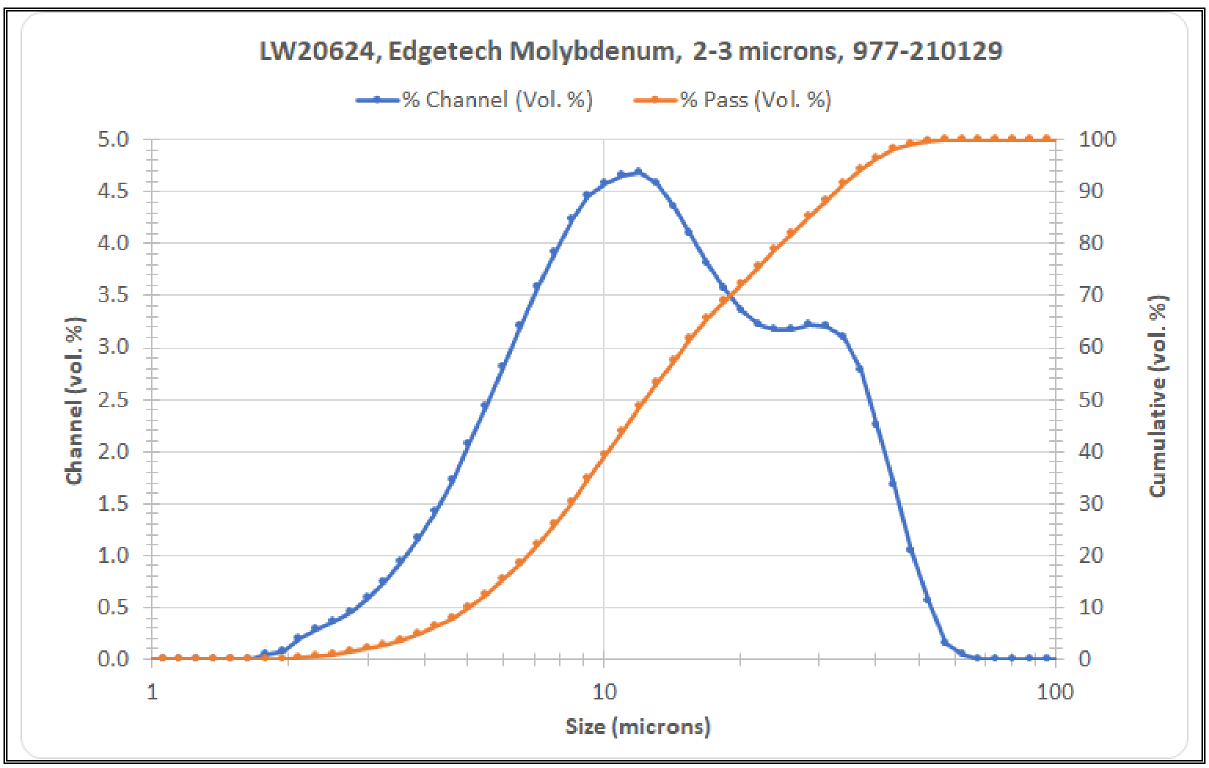


Figure A- 46 Edgetech Molybdenum, 2-3 microns, 977-210129, LW20624

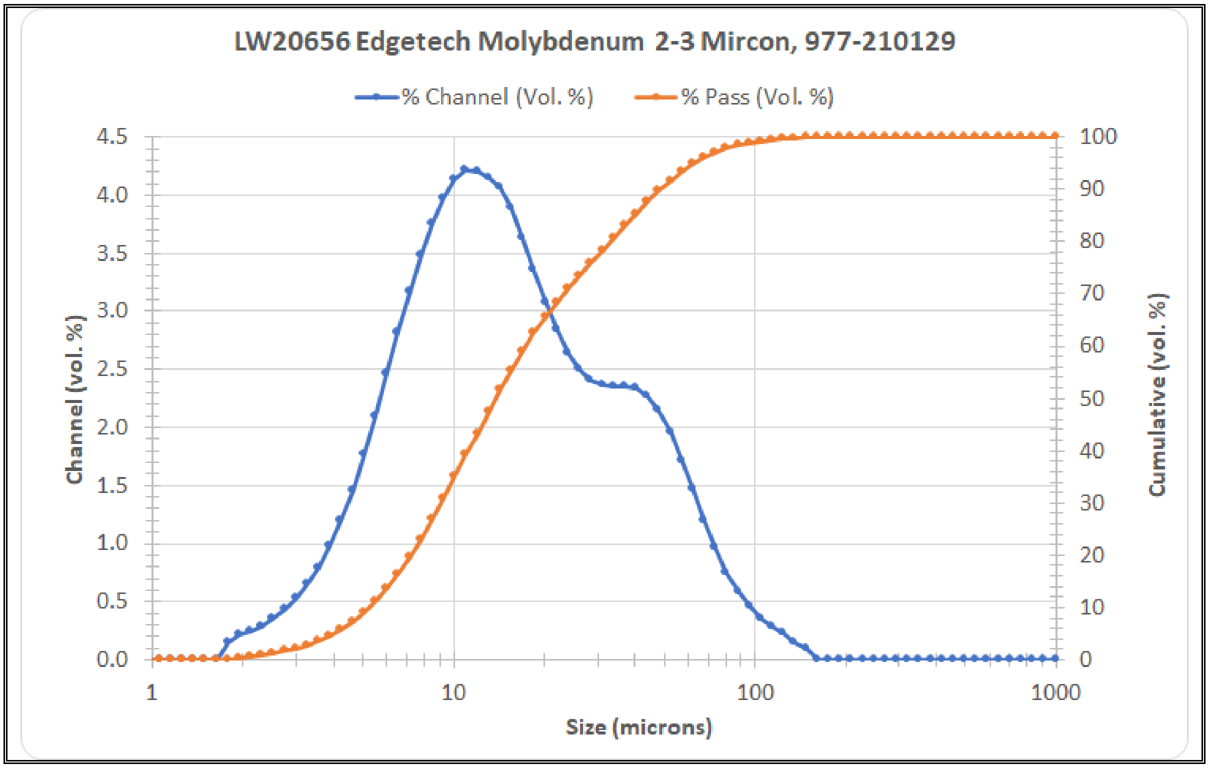


Figure A- 47 Edgetech Molybdenum, 2-3 microns, 977-210129, LW20656

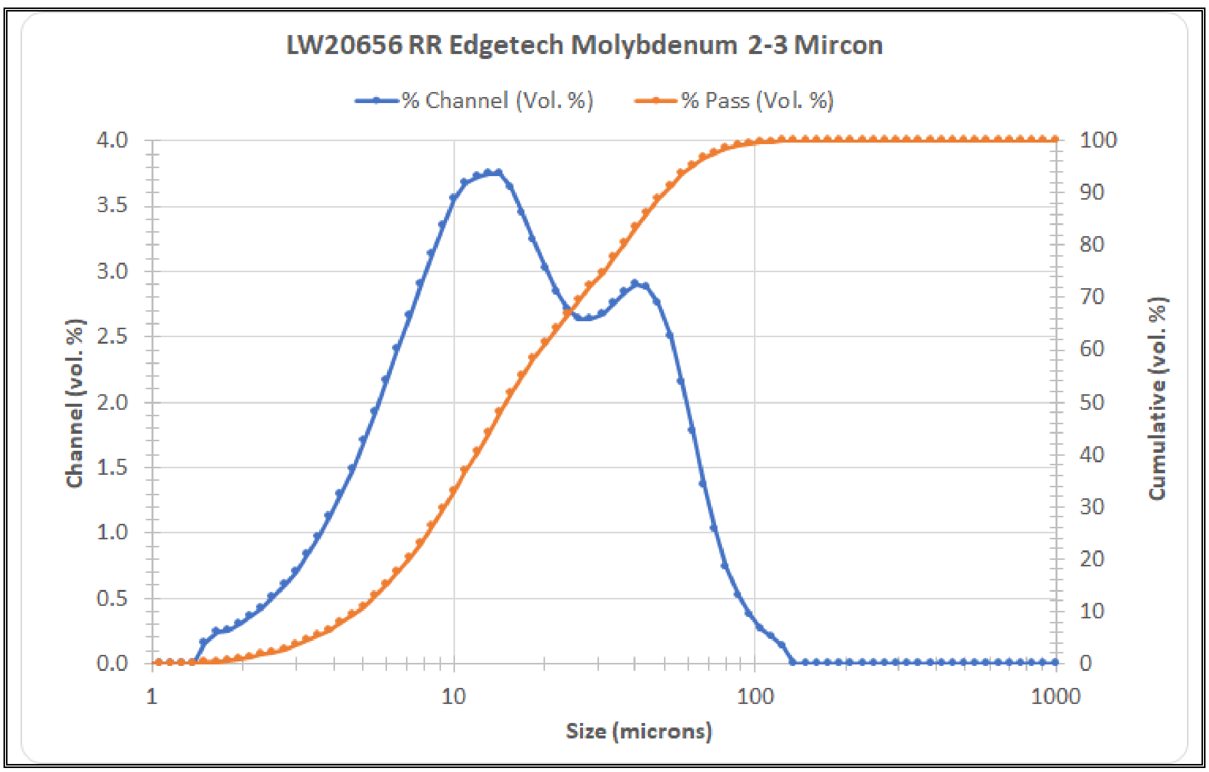


Figure A- 48 Edgetech Molybdenum, 2-3 microns, 977-210129, LW20656 RR

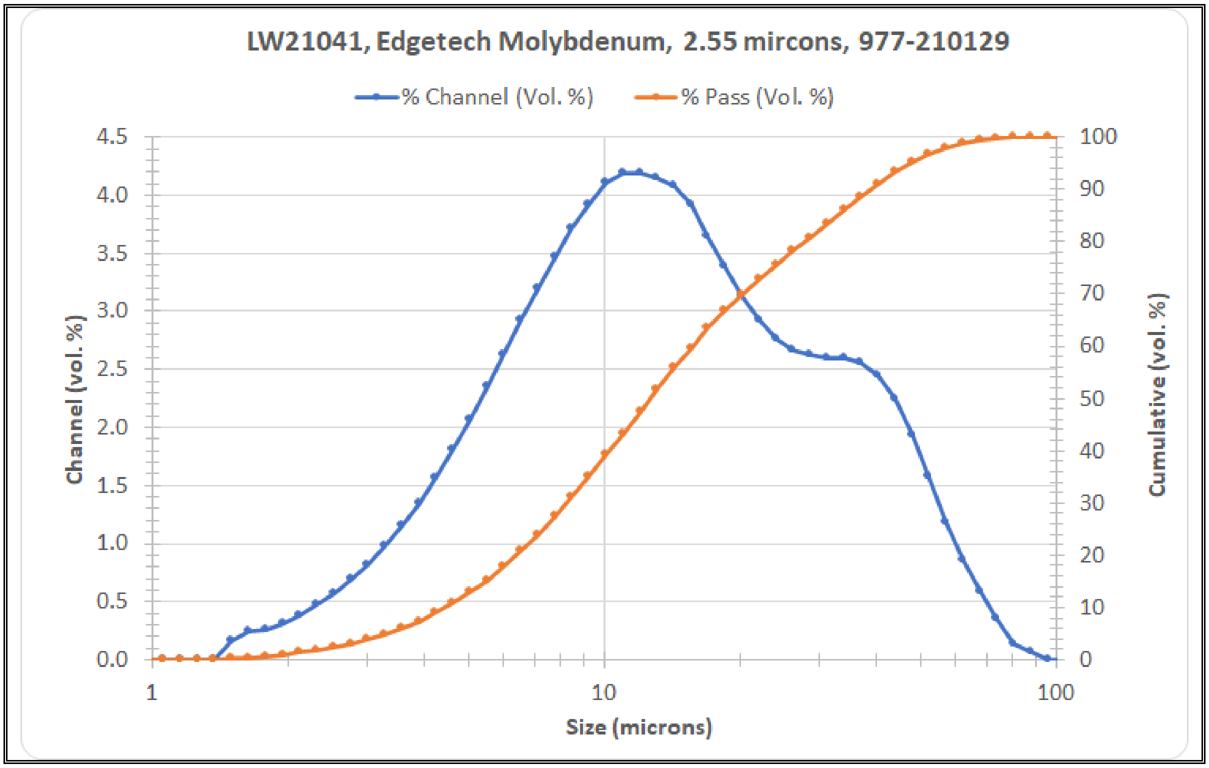


Figure A- 49 Edgetech Molybdenum, 2.55 microns, 977-210129, LW21041

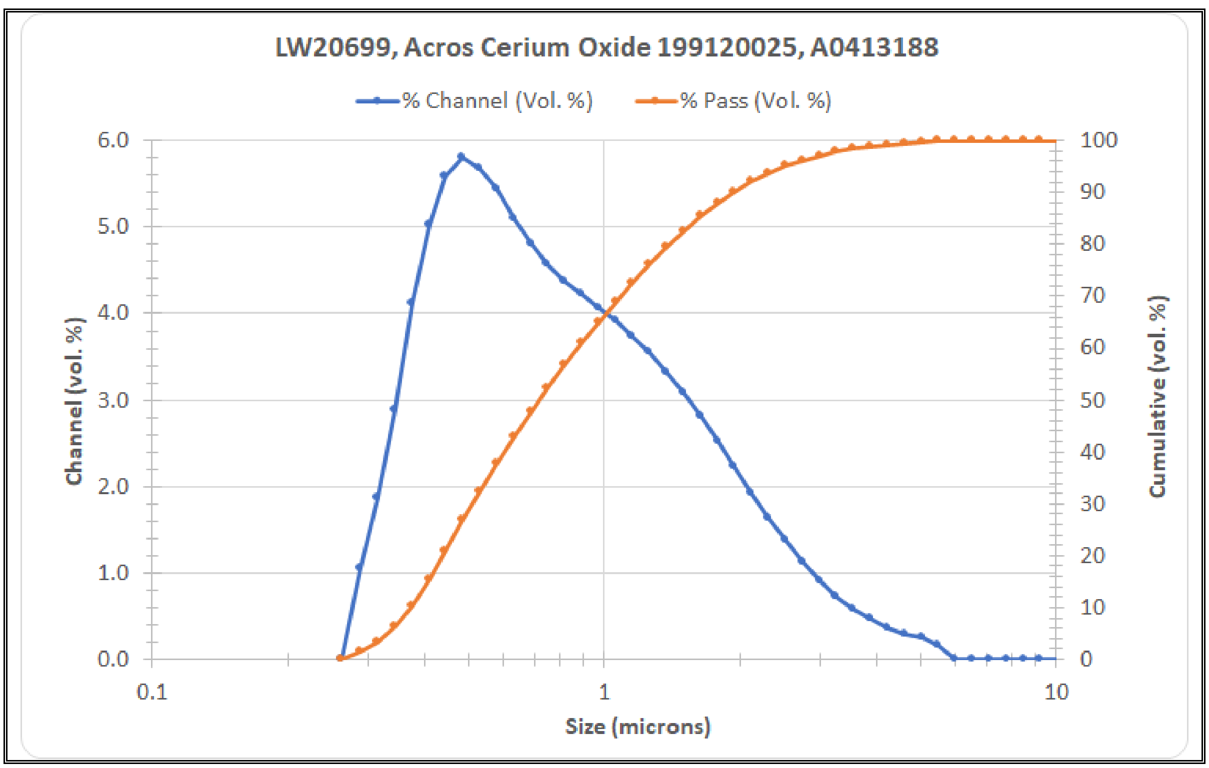


Figure A- 50 ACROS Cerium Oxide 199120025

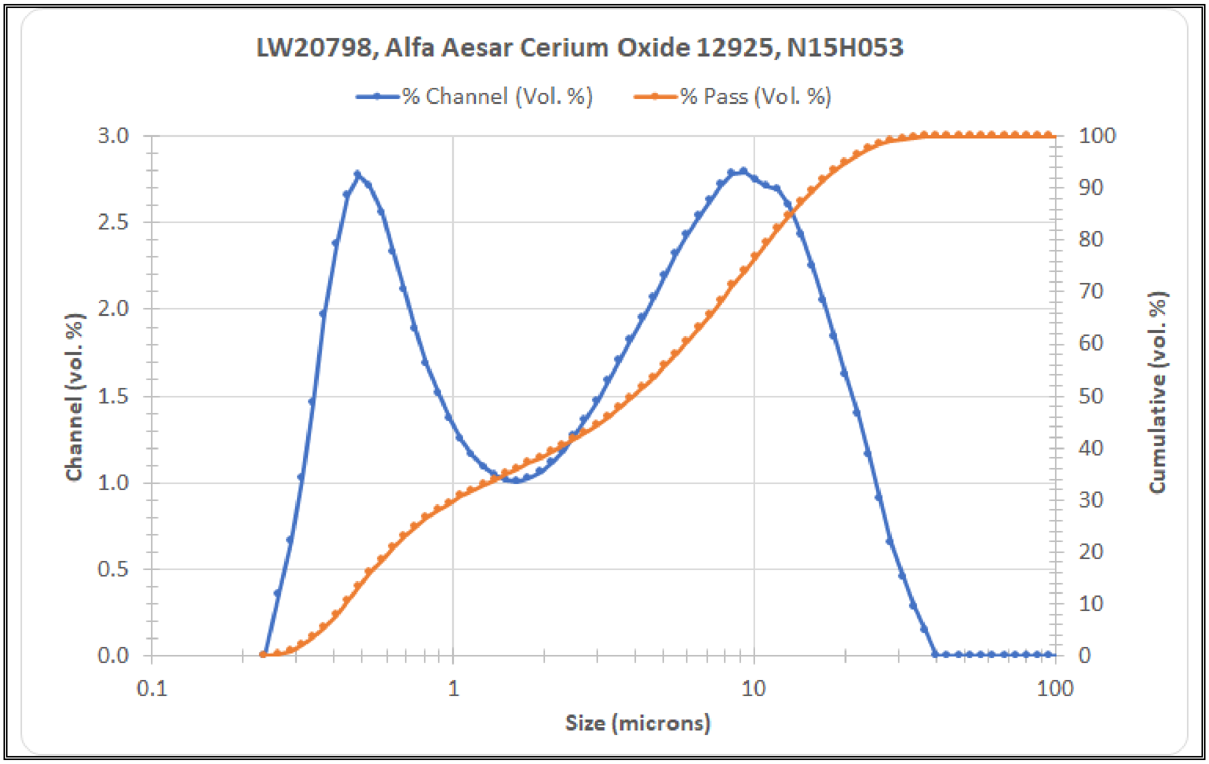


Figure A- 51 Alfa Aesar Cerium Oxide 12925

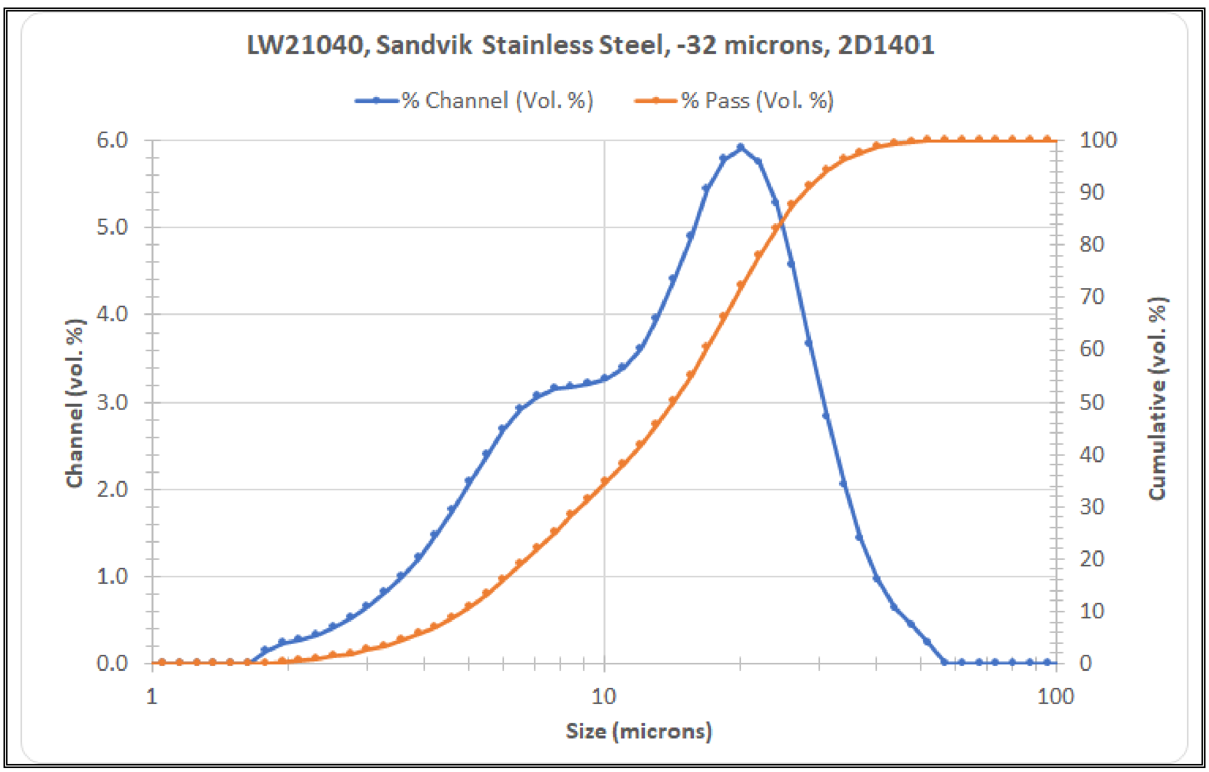


Figure A- 52 Sandvik Stainless Steel, -32 microns

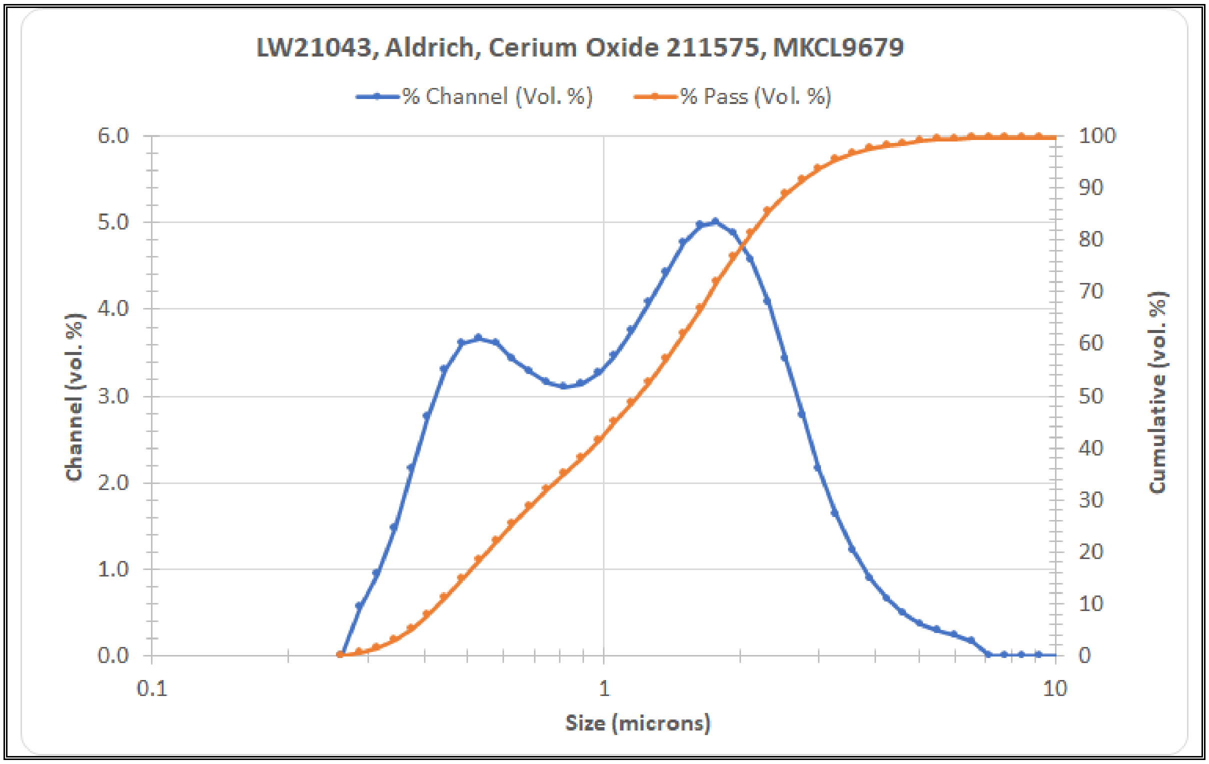
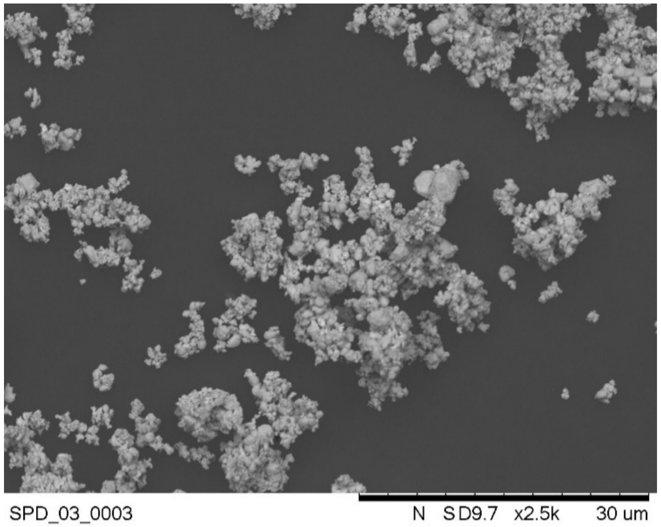
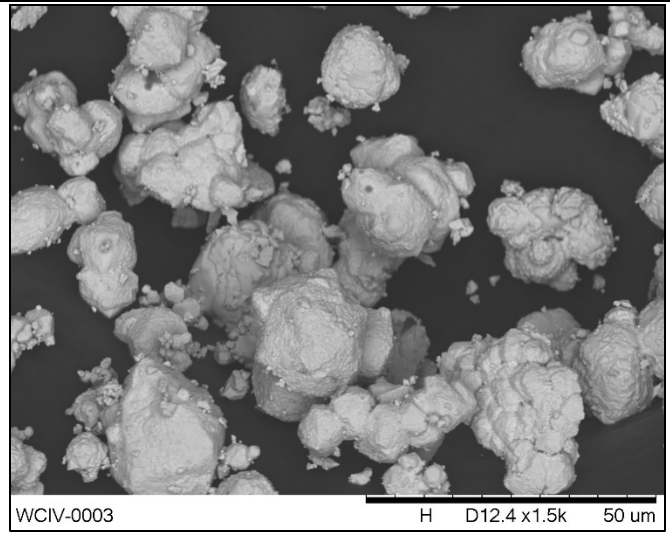


Figure A- 53 Aldrich Cerium Oxide -5 microns, 211575

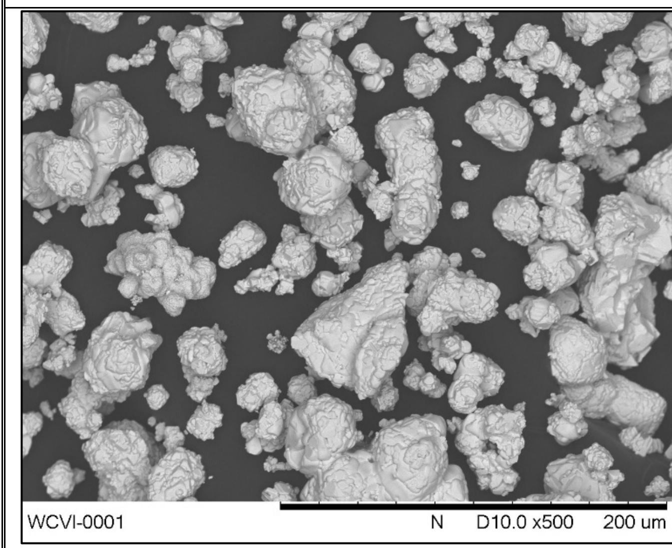
Appendix B. Scanning Electron Microscopy of Procured Powders



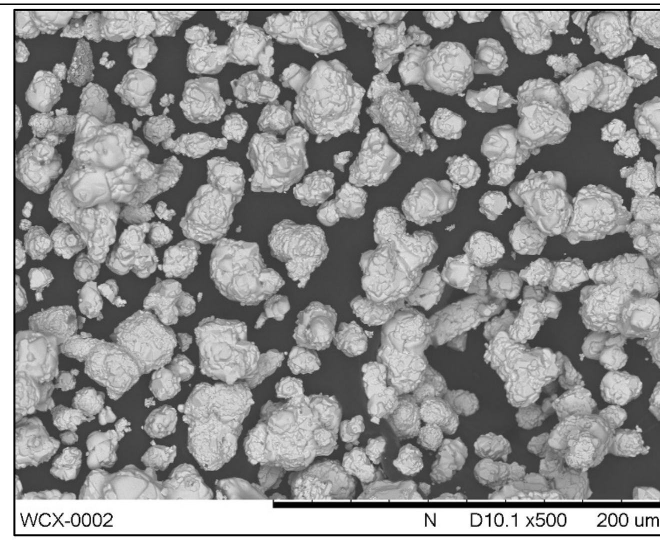
Buffalo Tungsten WCI



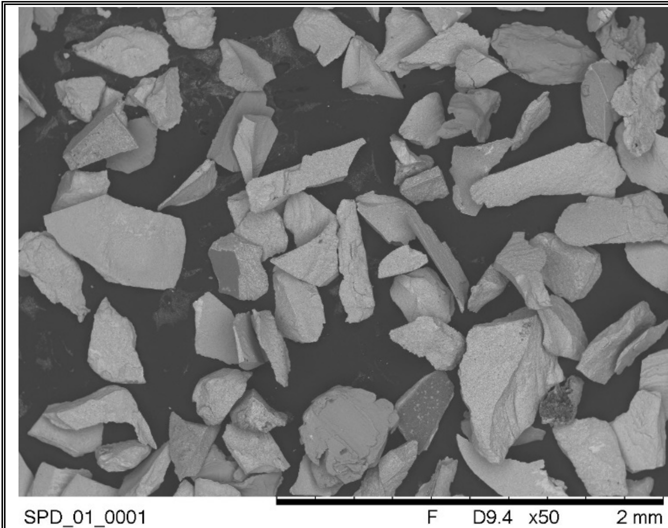
Buffalo Tungsten WCIV



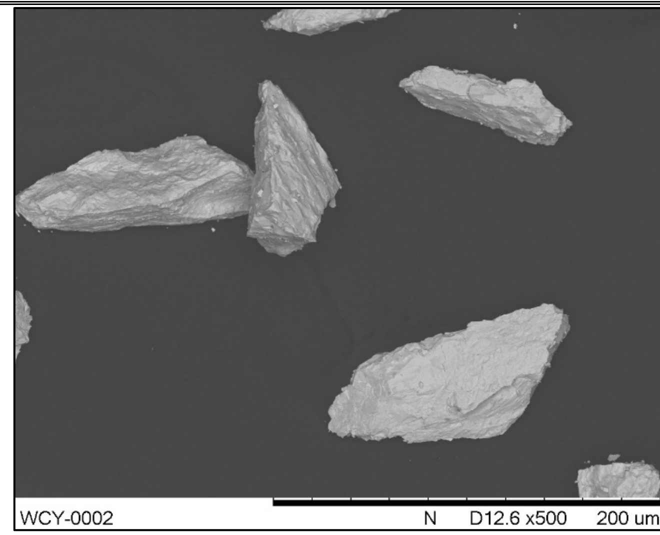
Buffalo Tungsten WCVI



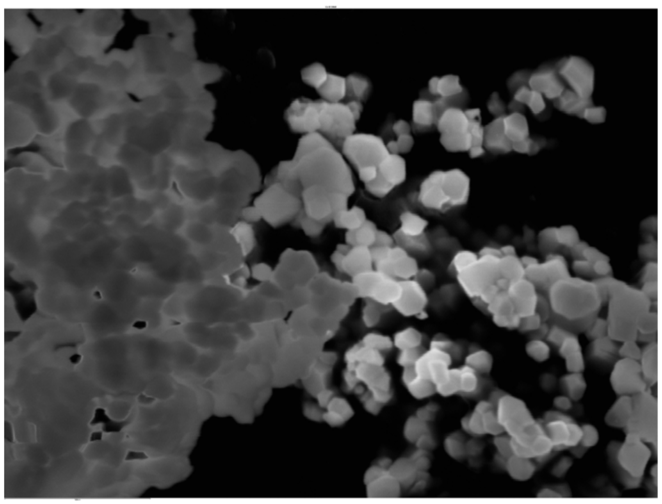
Buffalo Tungsten WCX



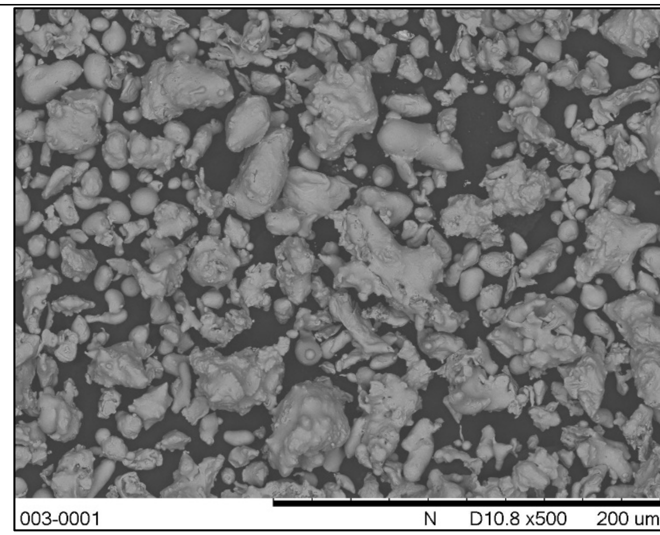
Buffalo Tungsten WCZ



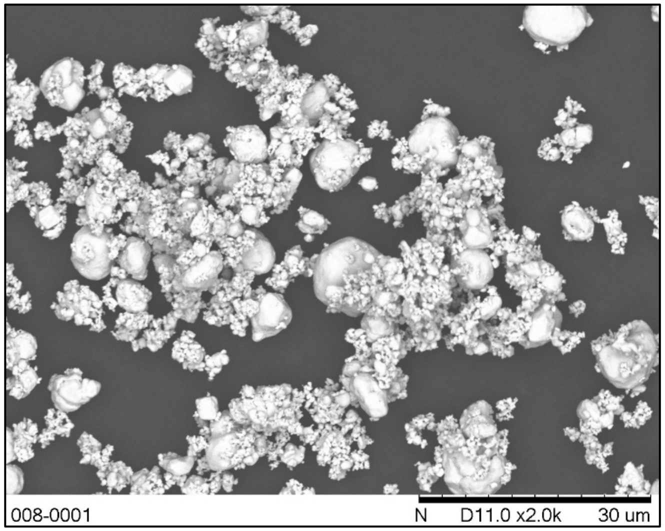
Buffalo Tungsten WCY



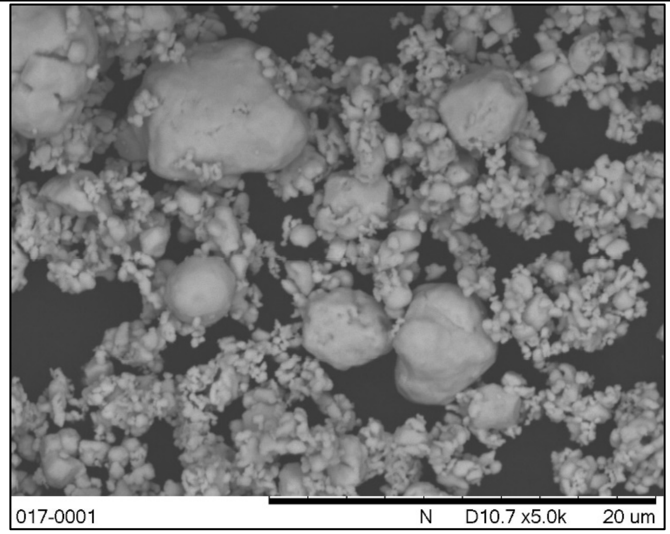
AEE Cerium Oxide CE-602 – 50kX



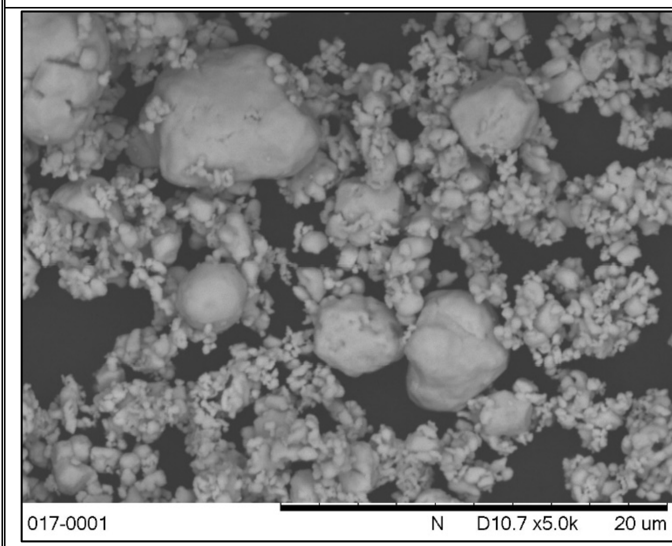
AEE Stainless Steel SS-103



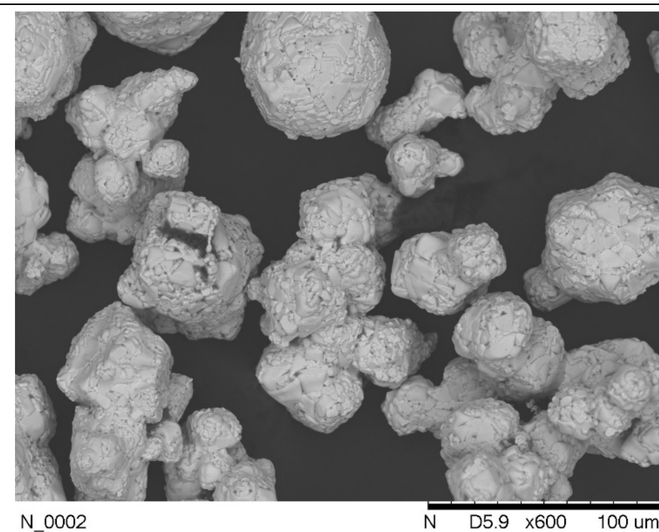
AEE Molybdenum MO-103



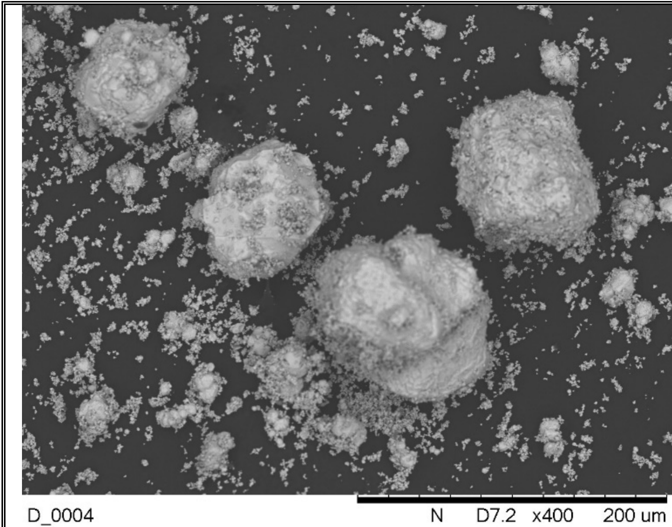
AEE Molybdenum MO-102



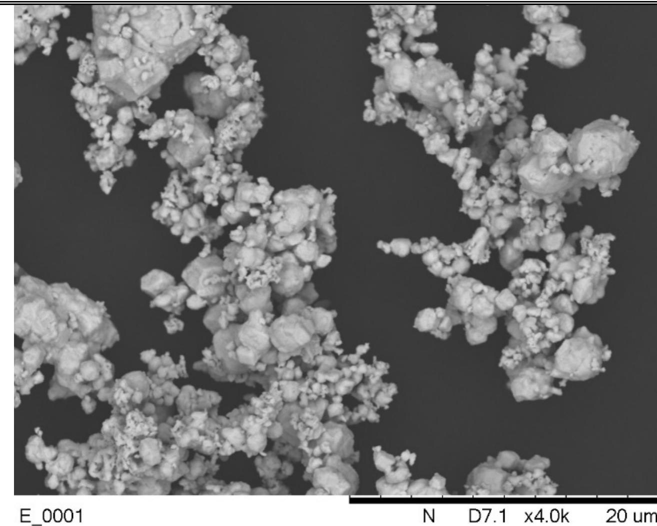
AEE Molybdenum MO-101



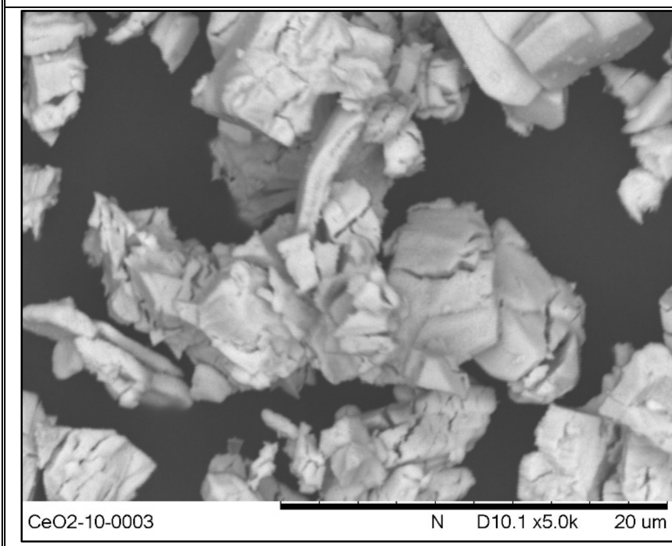
AEE Tungsten Carbide WP-305



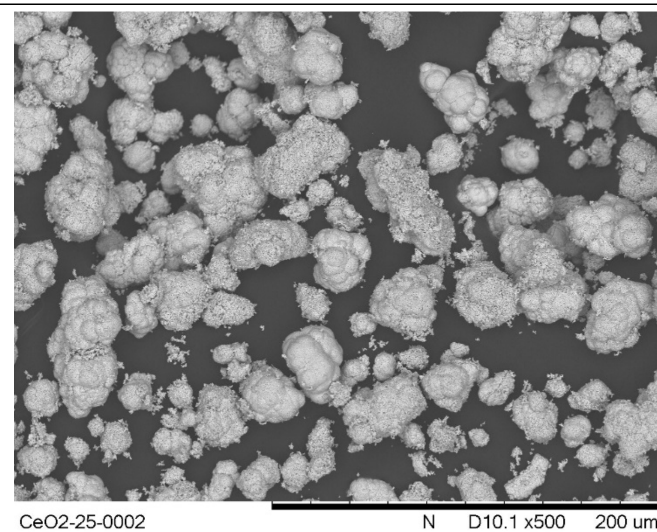
AEE Tungsten Carbide WP-302



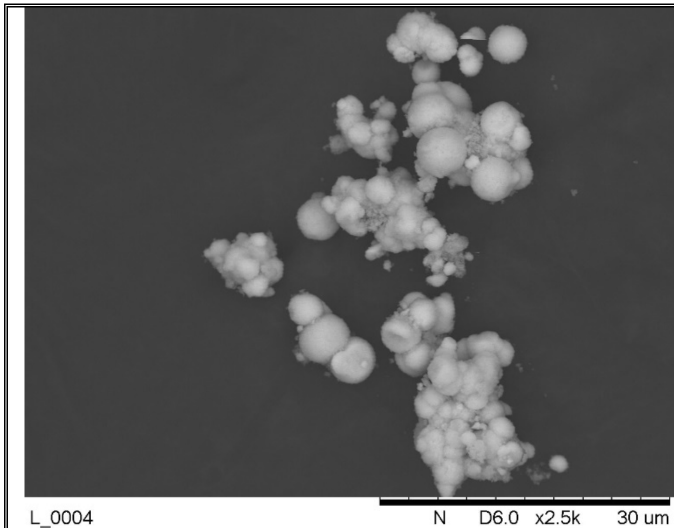
AEE Tungsten Carbide WP-301



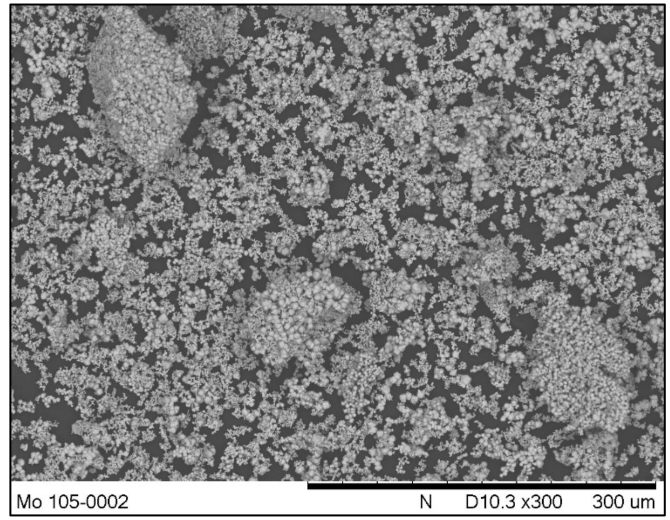
Blue Line Cerium Oxide 10 μm



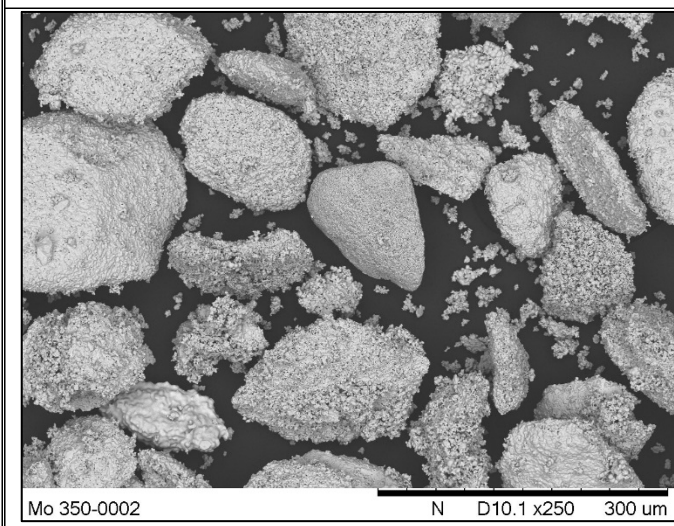
Blue Line Cerium Oxide 20 μm



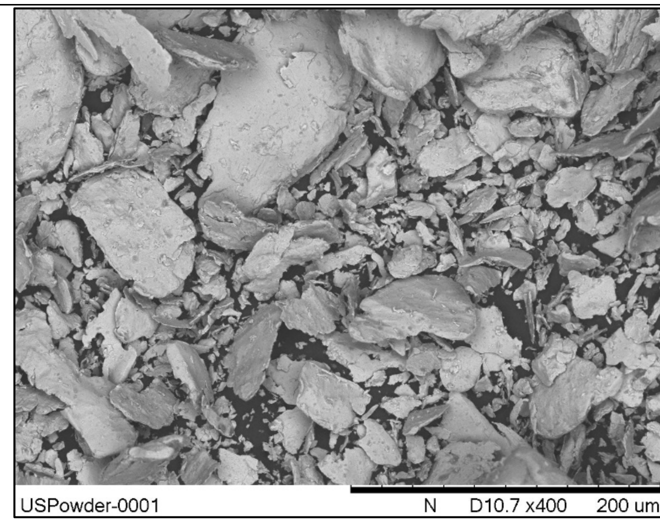
Blue Line Cerium Oxide 8.3 μm



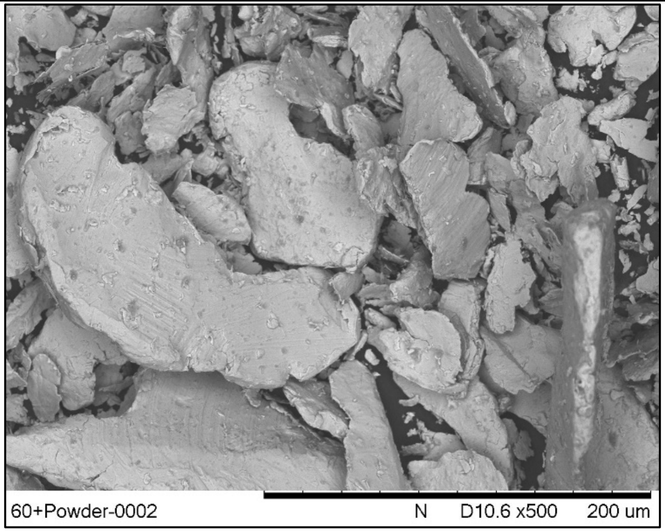
Good Fellow Molybdenum 105



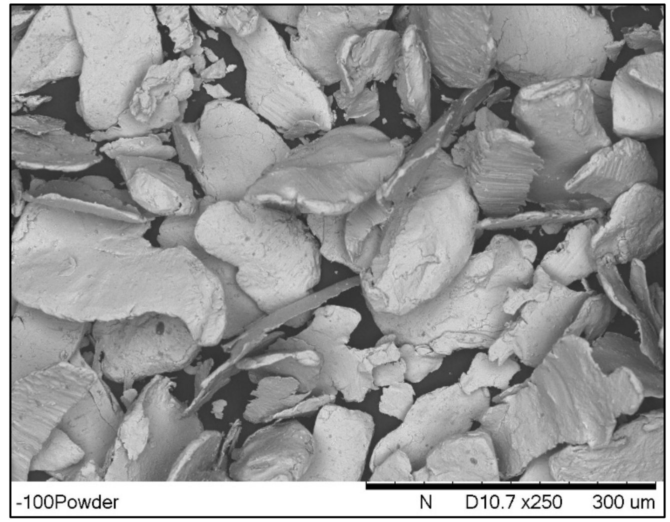
Good Fellow Molybdenum 350



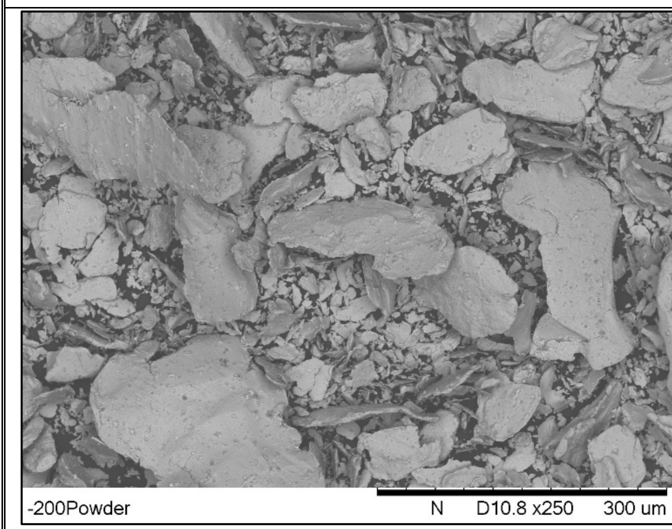
Pellets LLC Stainless Unscreened



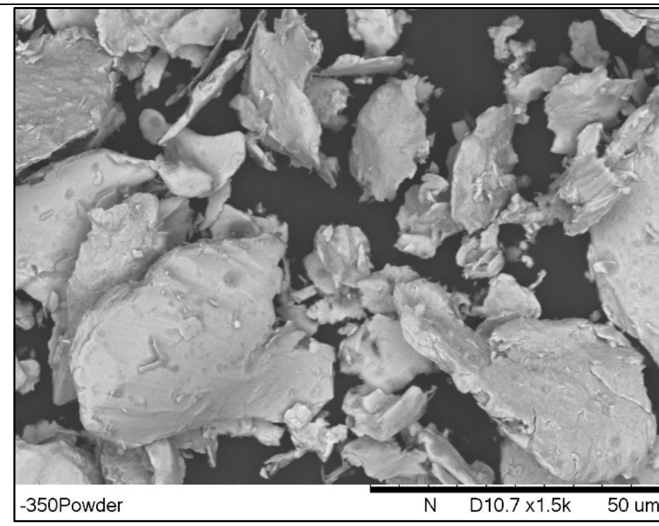
Pellets LLC Stainless 60



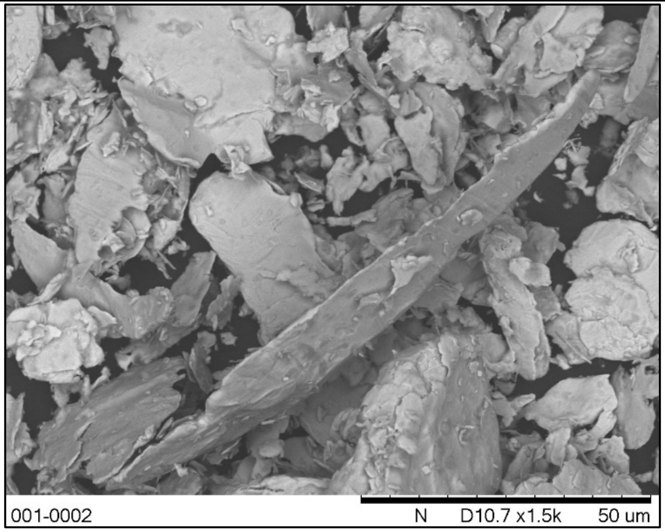
Pellets LLC Stainless -100



Pellets LLC Stainless -200

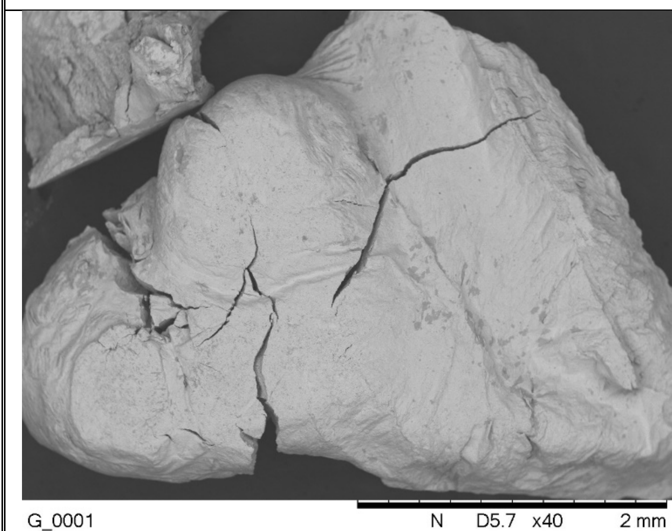


Pellets LLC Stainless -350

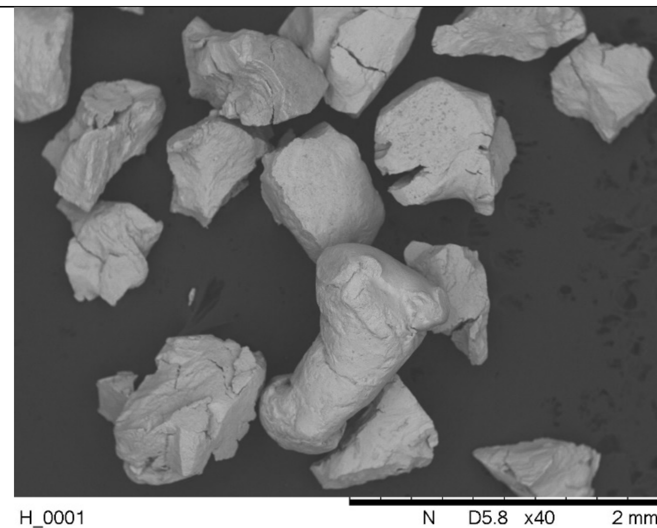


Pellets LLC Stainless -325

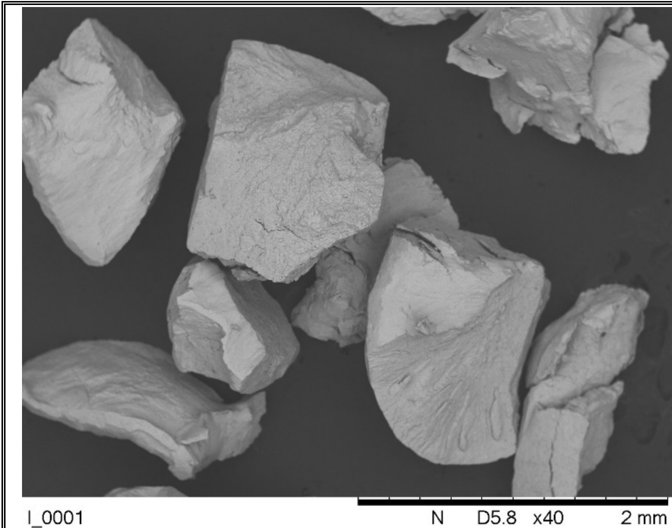
Vulkan Stainless Steel Grit G200



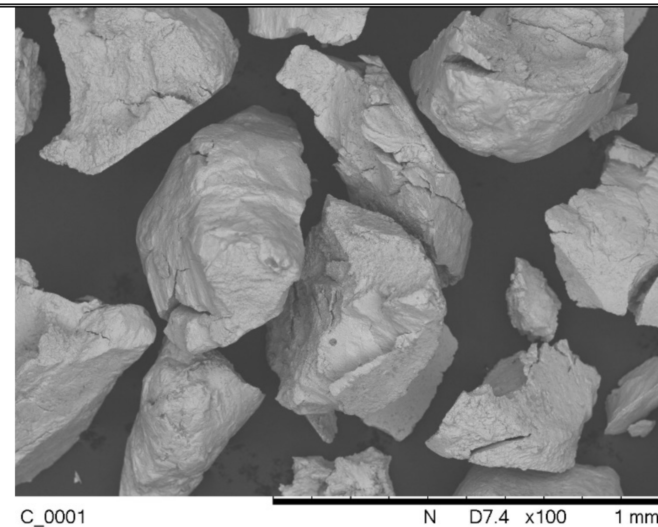
Vulkan Stainless Steel Grit G300



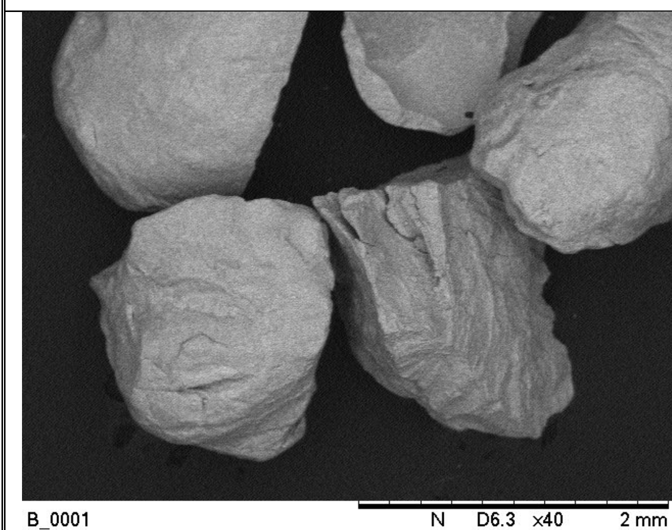
Vulkan Stainless Steel Grit G40



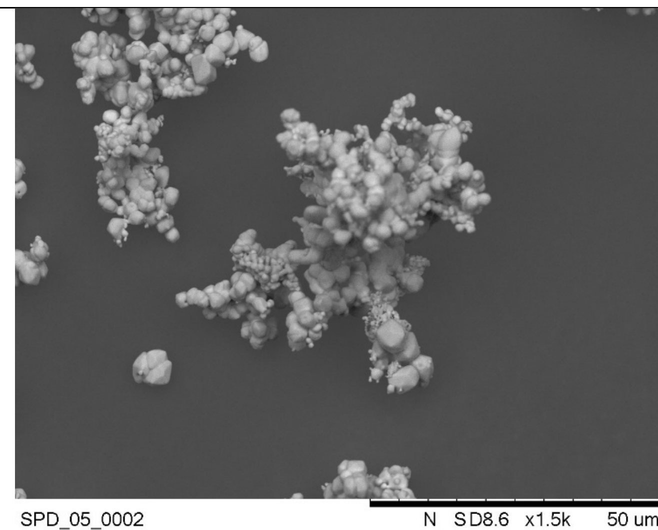
Vulkan Stainless Steel Grit G60



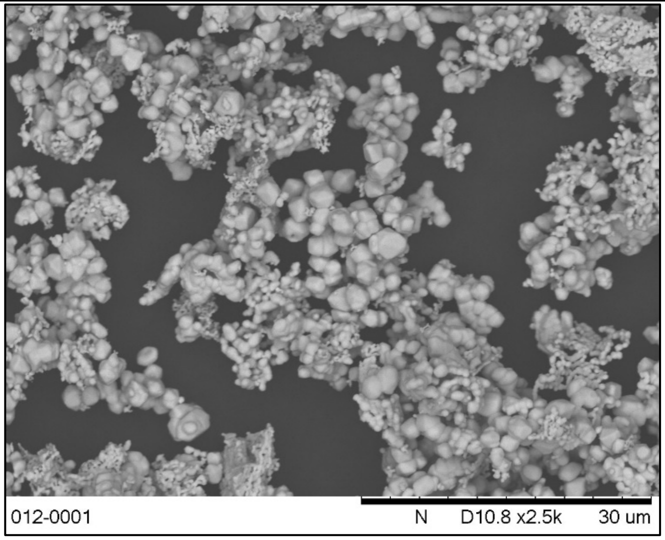
Vulkan Stainless Steel Grit G30



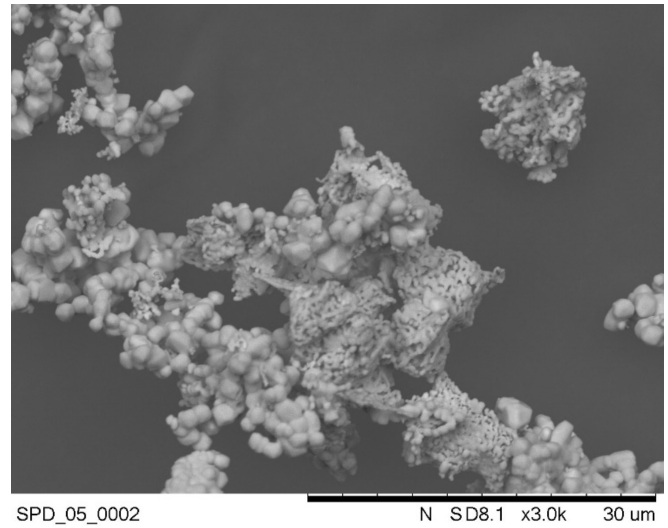
Vulkan Stainless Steel Grit G150



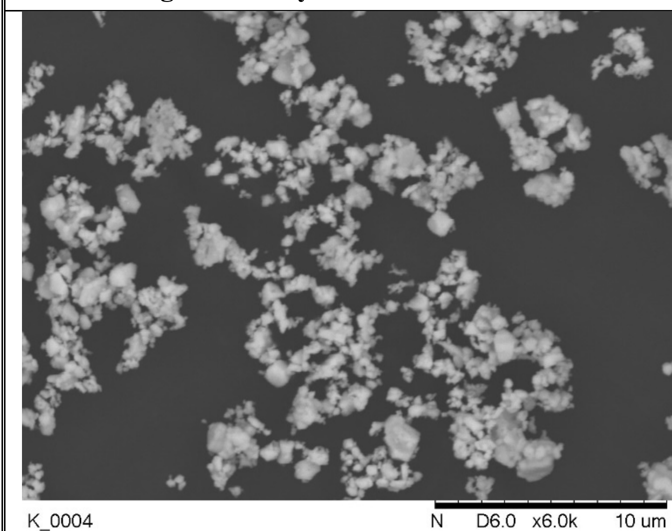
Edgetech Molybdenum -200 Mesh



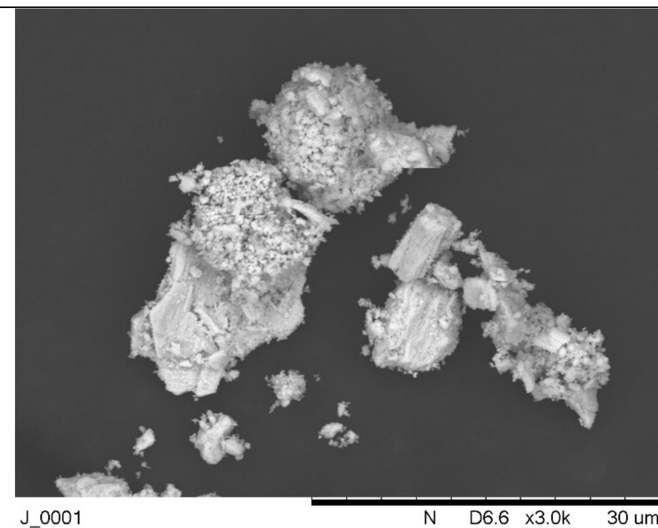
Edgetech Molybdenum 2-3 Microns



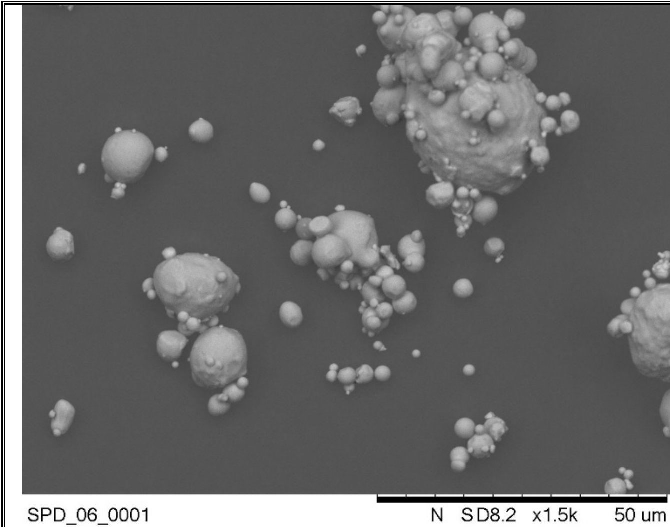
Edgetech Molybdenum -2.55 Microns



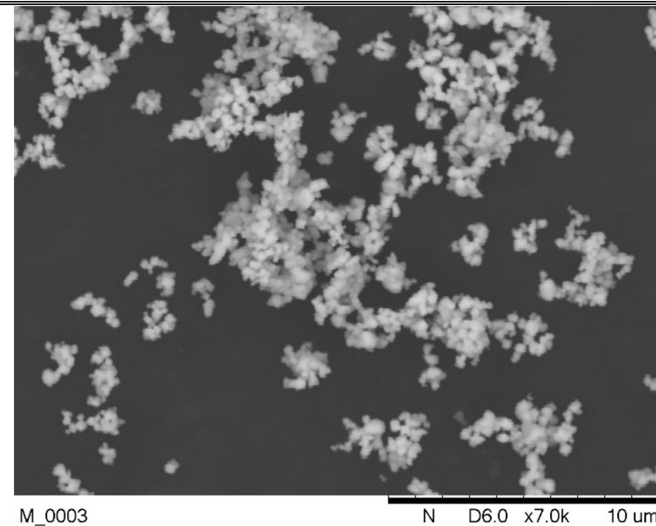
ACROS Cerium Oxide AC199120025



Alfa Aesar Cerium Oxide 12925

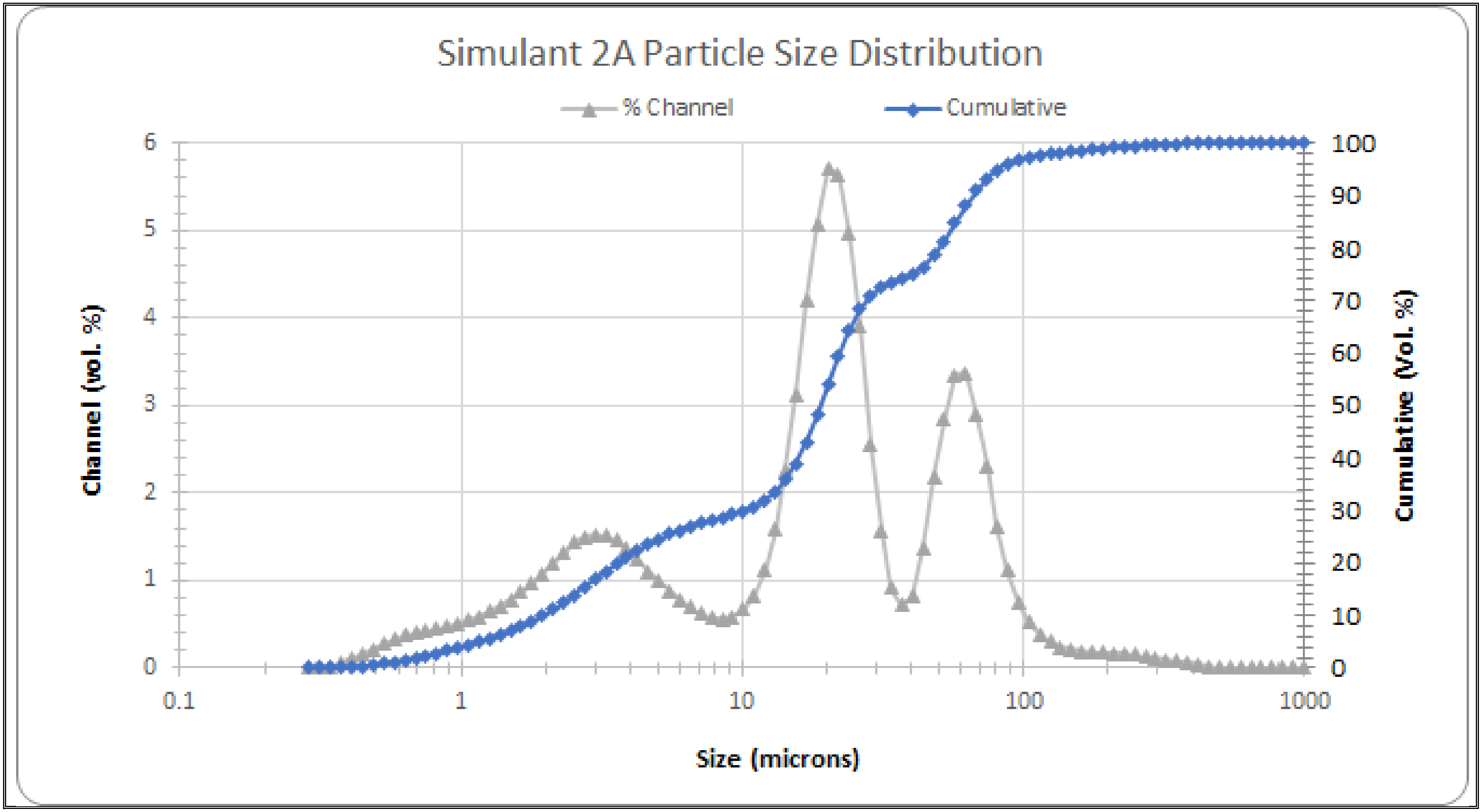


Sandvik Stainless Steel -32 microns

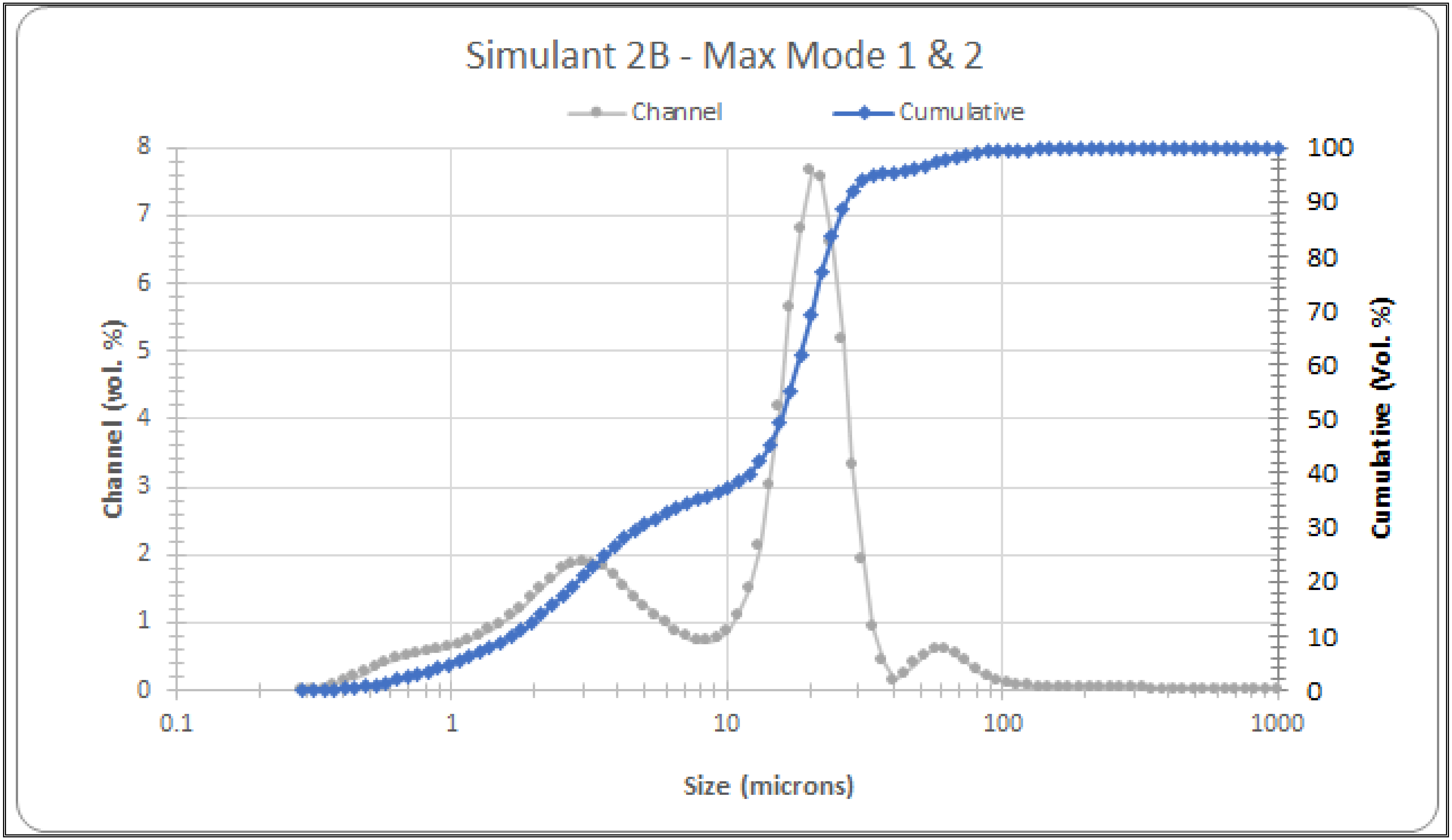


Aldrich Cerium Oxide 211575

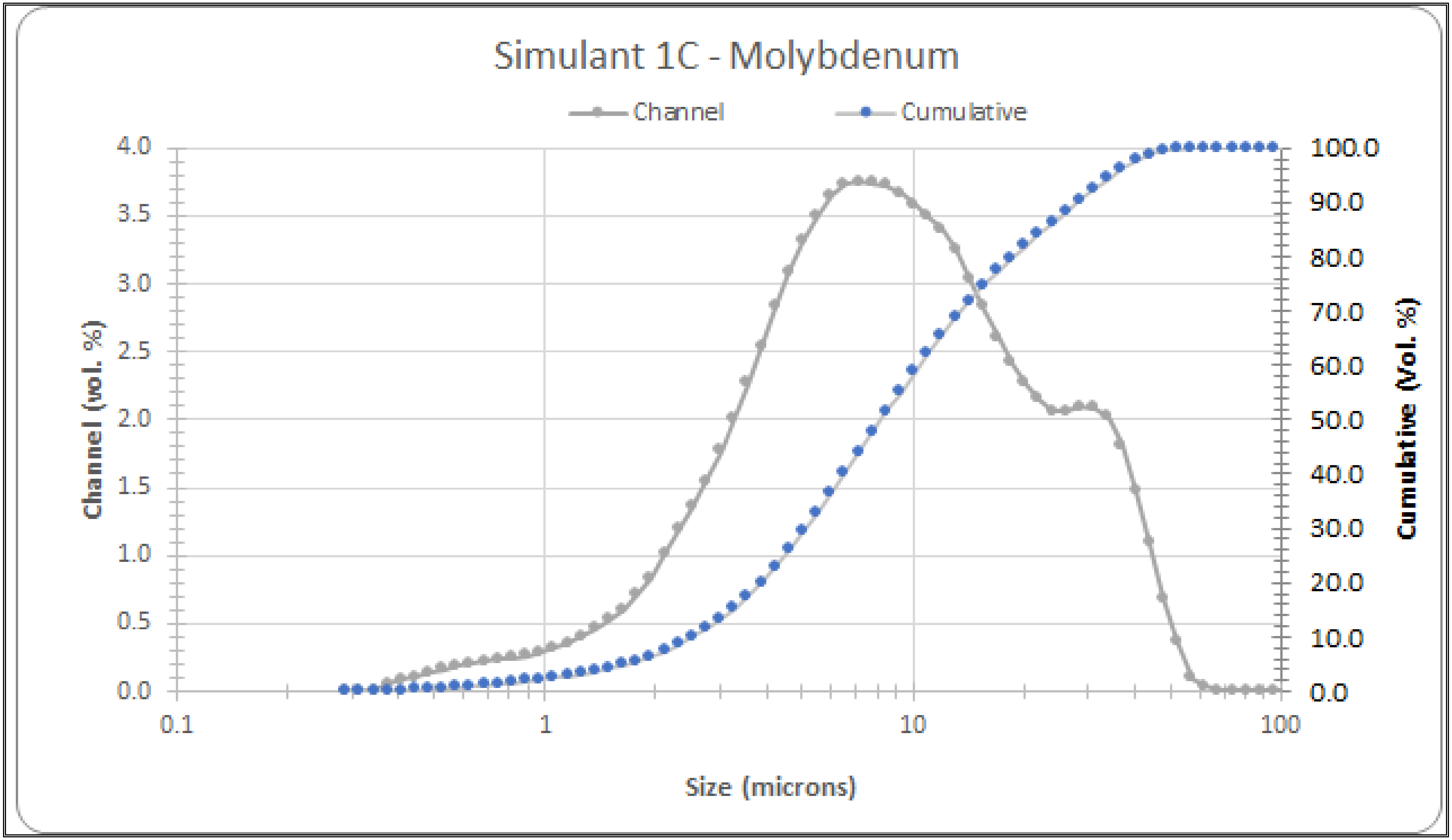
Appendix C. PSD of Undersized Batched Material



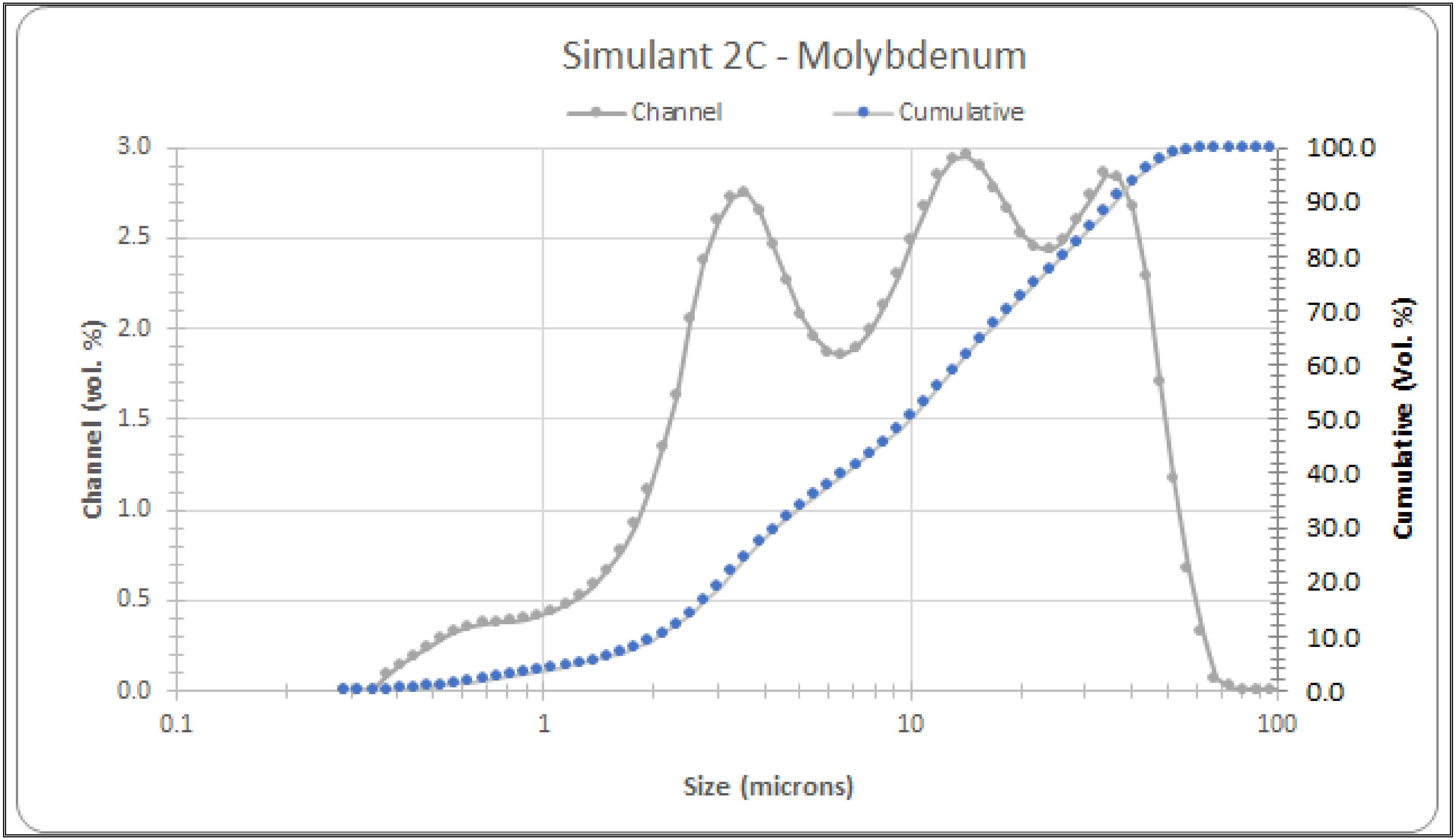
Simulant 1A and 2A Tungsten Carbide Particle Size Distribution



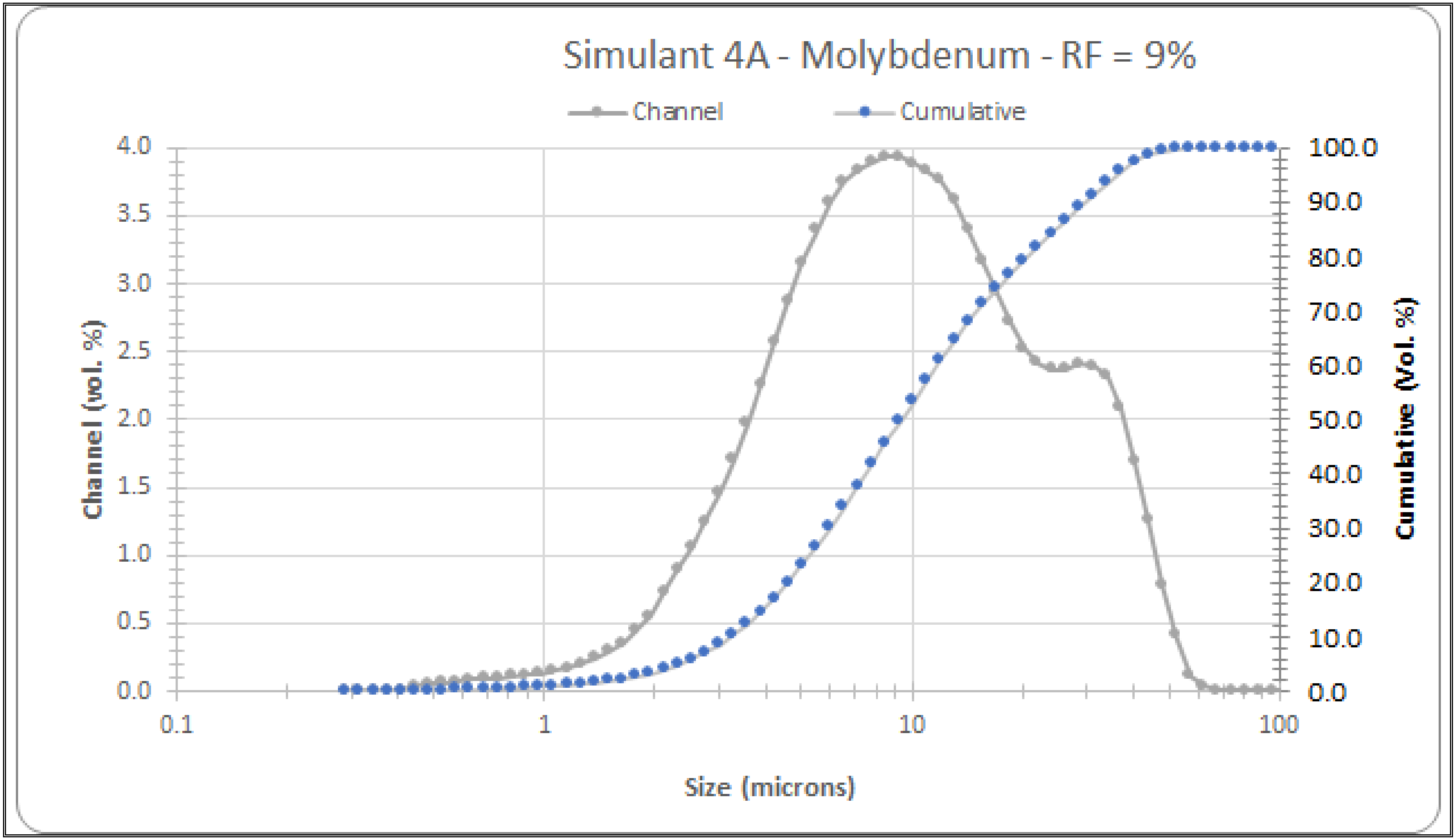
Simulant 1B and 2B Tungsten Carbide Particle Size Distribution



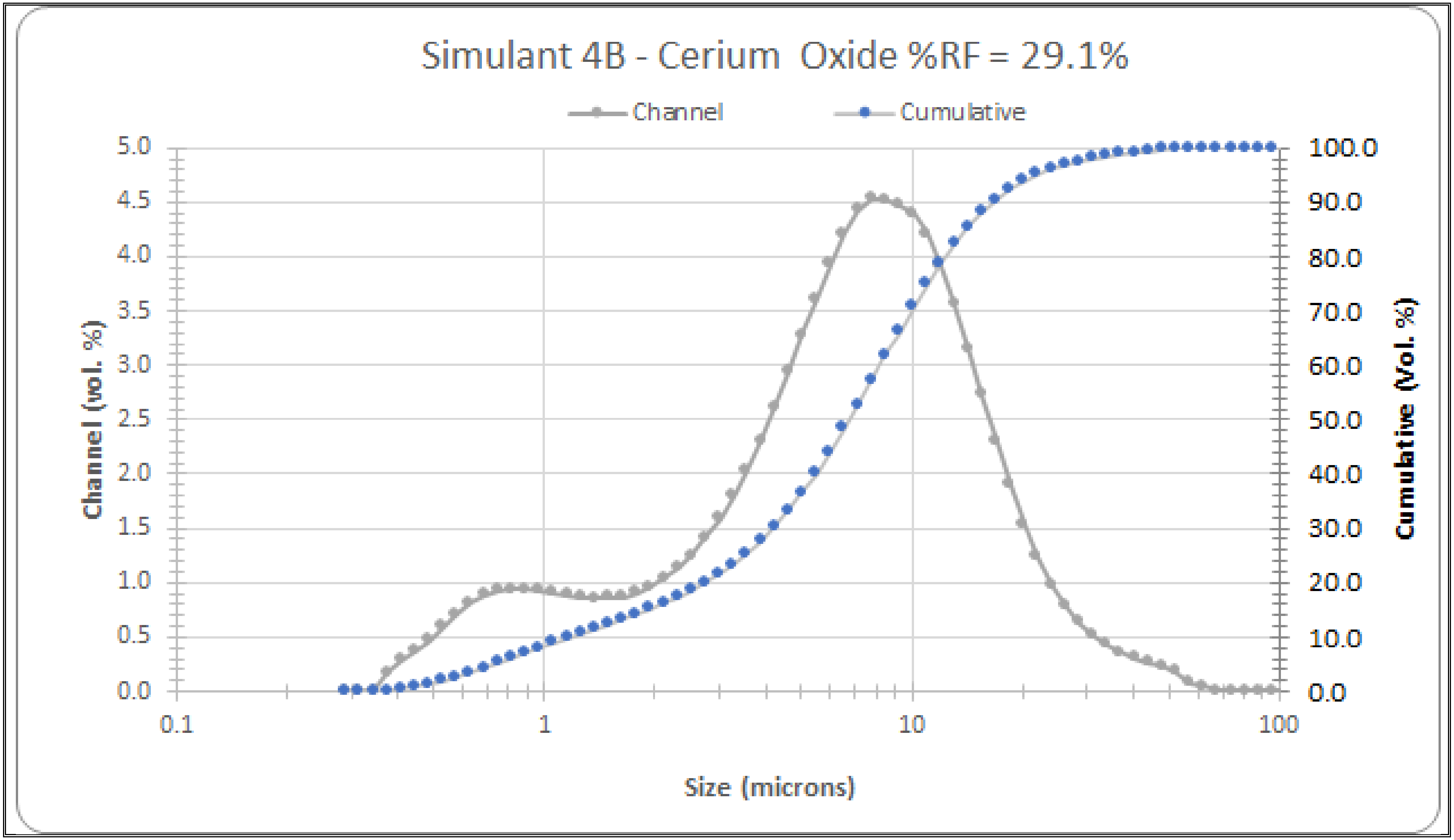
Simulant 1C Molybdenum Particle Size Distribution



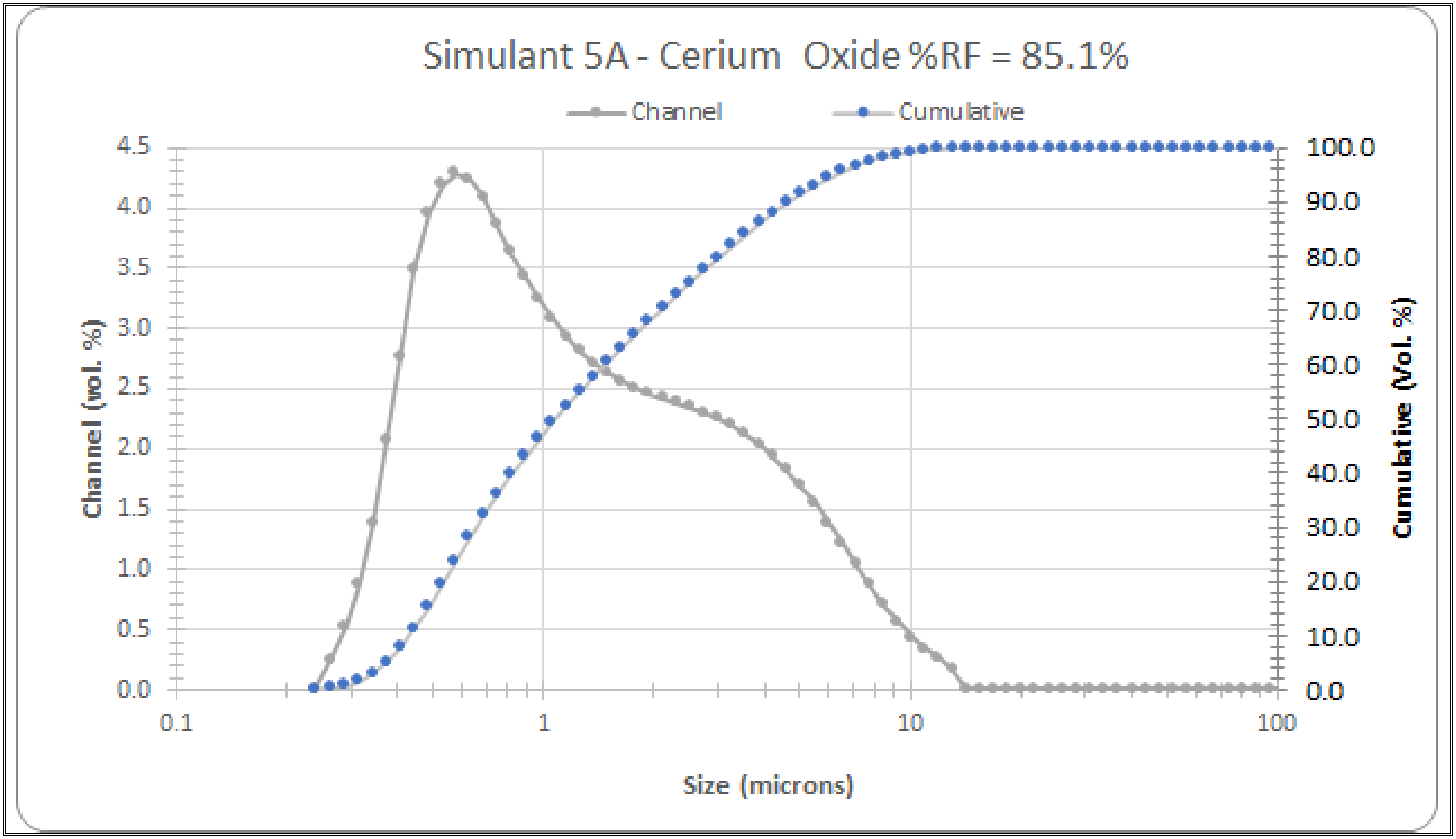
Simulant 2 Molybdenum Particle Size Distribution



Simulant 4A Molybdenum Particle Size Distribution



Simulant 4B Cerium Oxide Particle Size Distribution (data from Vendor)


Simulant 5A - Cerium Oxide %RF = 85.1%
Channel
Cumulative
Channel (vol. %)
Cumulative (Vol. %)
Size (microns)
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
0.1
1
10
100

Simulant 5A Cerium Oxide Particle Size Distribution

Appendix D. Simulant 3A Compositions

Simulant 3A Individual Batch Compositions

Batch #	Size (g)	Component (wt. %)						
		316L	316B	CeO_2	$CaCl_2 \cdot 2H_2O$	$MgCl_2$	CaF_2	TiO_2
1	200	85	0	0	4	4	5	3
2	100	0	85	0	4	4	5	3
3	100	85	0	0	7	0	5	3
4	100	0	85	0	7	0	5	3
7	70	85	0	0	7	0	5	3
9	100	85	0	0	7	0	5	3
10	100	0	85	0	7	0	5	3
11	100	85	0	0	12	0	0	3
12	100	85	0	0	12	0	0	3
13	100	85	0	0	12	0	0	3
14	100	0	85	0	12	0	0	3
15	300	85	0	0	4	4	5	3
16	300	43	0	43	4	4	5	3
18a	150	46	39	0	4	4	5	3
18b	150	46	39	0	4	4	5	3

Batch 1 – Calcined at 900 ºC

Batch 2 – Calcined at 900 ºC

Batch 3 – Calcined at 900 ºC

Batch 4 – Calcined at 900 ºC

Batch 7 – Dried at 110 ºC

Batch 10 – Calcined at 900 ºC

Batch 11 – Calcined at 900 ºC

Batch 17– Calcined at 900 ºC

Batch 18b - Calcined at 600 °C