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May 8, 2006

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Author's Name: JONATHAN DUFFEY

Location: 773-A Phone (803)725-3859

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Author's User ID	Primary Author/Contact (Last Name, First)	Location	Phone No.	Org Code	Organization
a3903	DUFFEY JONATHAN	773-A	(803)725-3859	L3420	SRNL

Additional Author's User ID	Additional Author's (Last Name, First)
o8459	RONALD, LIVINGSTON

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JONATHAN DUFFEY	4/26/2006
Author's Name (Print)	Date

Manager's User ID A3761 **Manager's Full Name (Last, First)** NORATO, MICHAEL ANTHONY

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WSRC-TR-2003-00388-R0

Characterization of Neptunium Oxide Generated Using the HB-Line Phase II Flowsheet

by
JONATHAN DUFFEY

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

Additional Authors:
RONALD LIVINGSTON

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Characterization of Neptunium Oxide Generated Using the HB-Line Phase II Flowsheet

J. M. Duffey and R. R. Livingston

August 2003

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

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Characterization of Neptunium Oxide Generated Using the HB-Line Phase II Flowsheet

J. M. Duffey and R. R. Livingston

August 2003

Approvals

Jonathan M. Duffey, Author Date

Ronald R. Livingston, Author Date

George C. Rodrigues, Technical Reviewer Date

James W. McClard, Technical Reviewer Date

Robert H. Smith Jr., Technical Reviewer Date

Alice M. Murray, L4 Manager, Actinide Laboratory Programs Date

Alan F. Riechman, L3 Manager, Actinide Technology Section Date

Jeffrey B. Schaade, Manager, H-Area Technical Services Date

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Characterization of Neptunium Oxide Generated Using the HB-Line Phase II Flowsheet

J. M. Duffey and R. R. Livingston

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

Summary

Approximately 98 grams of neptunium(IV) oxide (NpO_2) were produced at the Savannah River Technology Center (SRTC) for use in gas generation tests to support the neptunium stabilization program at the Savannah River Site (SRS).[1] The NpO_2 was produced according to the anticipated HB-Line flowsheet consisting of anion exchange, oxalate precipitation, filtration, and calcination.[2, 3] Characterization of the NpO_2 product to be used in gas generation tests included bulk and tap density measurements, X-ray diffraction, particle size distribution, specific surface area measurements, and moisture analysis.

Background

The SRS plans to convert neptunium solutions currently stored in H-Canyon to NpO_2 for transfer to Y-12 for storage and eventual use by the Oak Ridge National Laboratory (ORNL). The NpO_2 will be transported in the 9975 shipping package. Due to the potential for radiolytic production of hydrogen during transport, the SRTC is performing gas generation tests and moisture measurements to support the necessary revisions to the 9975 Safety Analysis Report for Packaging (SARP). About 98 grams of NpO_2 were produced at SRTC for use in these tests.

The NpO_2 was produced according to the anticipated HB-Line flowsheet consisting of anion exchange, oxalate precipitation, filtration, and calcination. Production of the NpO_2 from samples of stored neptunium solution is described in a separate report.[3] Following anion exchange to concentrate neptunium solution from H-Canyon Tank 8.5, neptunium(IV) oxalate ($Np(C_2O_4)_2$) was precipitated, filtered, and calcined to NpO_2 powder in four separate batches. Two batches of $Np(C_2O_4)_2$ were calcined to the oxide at 600 °C (600C-1 and 600C-2) and the other two batches were calcined to the oxide at 650 °C (650C-1 and 650C-2). Calcination temperatures of 600 °C and 650 °C were chosen based on the expected HB-Line Phase II furnace operating temperature of 625 °C ± 25 °C.[i]

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[i] At the time of lab-scale production of NpO_2 at SRTC the expected HB-Line Phase II furnace operating temperature was 625 °C ± 25 °C. Currently, however, it is expected that the furnace will be operated at 650 °C (range uncertain).

This report describes the characterization of the NpO_2 powder produced for use in gas generation tests. Characterization methods included bulk and tap density measurements, X-ray diffraction analysis, particle size distribution analysis, specific surface area measurements, and moisture analysis before and after exposure to 75% relative humidity (RH) for several days.

Experimental Results and Discussion

Density Measurements

The density of unsettled NpO_2 powder (bulk density) was measured for the first batch of NpO_2 calcined at 600 °C (600C-1) by weighing the NpO_2 into a 25-mL graduated cylinder and recording the volume of unsettled powder. The uncertainty in the mass reading was estimated to be ± 0.005 g, and the uncertainty in the net mass of NpO_2 was ± 0.007 g. Because the top of the powder column was not perfectly level in the graduated cylinder, the recorded volume was taken as the average of the high and low volume readings for the powder level. The uncertainty of each individual volume reading was estimated to be ± 0.2 cm^3 (minimum cylinder graduation) and the propagated uncertainty of the recorded volume was also ± 0.2 cm^3 (rounding up) because the volume was averaged. The density of settled powder (tap density) was then measured by tapping the graduated cylinder firmly on a solid surface for 1.0 min and recording the new volume as described above. The NpO_2 was emptied from the graduated cylinder and the measurement process repeated. Results of these tests are given in Table 1. Average results for the two measurements were 1.90 ± 0.03 g/cm^3 for bulk density and 2.50 ± 0.04 g/cm^3 for tap density. These values are similar to densities measured previously by Rudisill (average of eight measurements ± 1 standard deviation: 1.83 ± 0.08 g/cm^3 and 2.48 ± 0.07 g/cm^3 for bulk and tap densities, respectively) for NpO_2 calcined at 650 °C. Rudisill used a similar experimental technique but smaller sample size.[4] Because the densities measured for NpO_2 batch 600C-1 and Rudisill's NpO_2 calcined at 650 °C were so similar, no additional density measurements were made.

Table 1: Bulk and Tap Density Measurements of NpO_2 Powder 600C-1

Trial #	Cylinder (g)[a]	Cylinder + NpO_2 (g)[a]	NpO_2 (g)[b]	Unsettled NpO_2 (cm^3)[c]	Settled NpO_2 (cm^3)[c]	Bulk Density (g/cm^3)[d]	Tap Density (g/cm^3)[d]
1	59.607	84.877	25.270	13.4	10.1	1.89 ± 0.03	2.50 ± 0.05
2	59.637	84.813	25.176	13.1	10.1	1.92 ± 0.03	2.49 ± 0.05
Average	NA	NA	25.223	13.3	10.1	1.90 ± 0.03	2.50 ± 0.04

[a] Gross mass uncertainties are ± 0.005 g.

[b] Net mass uncertainties are ± 0.007 g.

[c] Volume uncertainties are ± 0.2 cm^3.

[d] Error bars are propagated uncertainties based on the estimated mass and volume uncertainties.

X-Ray Diffraction

Samples of NpO_2 powder calcined at 600 °C (600C-1) and at 650 °C (650C-1) were analyzed by X-ray diffraction using a Bruker D8 Advance X-ray diffractometer contained in a radioactive glovebox. The data were collected by step-scanning over the 2-theta (2θ) range of 10° to 160° with a step size of 0.02° and a dwell time of 10 s. The instrument parameters are given in Table 2.

The powder X-ray diffraction patterns for the 600C-1 and 650C-1 NpO_2 are shown in Figures 1 and 2, respectively. The unit cell dimensions measured were 5.4365 Å for 600C-1 and 5.4352 Å for 650C-1. Both values are in good agreement with literature values for NpO_2 of 5.433 Å[5] and 5.4328 Å.[6] By comparison, the unit cell dimension reported for plutonium(IV) oxide (PuO_2) is 5.3960 Å.[6] A third sample of NpO_2 (650C-2) was analyzed with lanthanum hexaboride (LaB_6)[ii] added as an internal d-spacing and instrument profile standard to more accurately measure the unit cell dimension and crystallite size (Figure 3). Lattice parameter refinement and crystallite size determinations (integral breadth) were made with Jade Plus software. No residual $Np(C_2O_4)_2$ was detected in these materials and the NpO_2 unit cell was stabilized.

Table 2: X-ray Diffractometer Instrument Parameters

Radiation source	CuKα X-ray
Source power	45 kV, 40 mA
Wavelength	1.5405982 Å
Goniometer	Bruker D8 Advance; radius 217 mm
Divergence soller slit	None
Divergence	1°
Specimen rotation	No
Diffracted beam antiscatter	2 mm
Diffracted beam soller slit	2°
Secondary monochromator	Curved pyrolytic graphite
Receiving slit	0.2 mm
Detector slit	2 mm
Detector	NaI scintillation
2θ range	5° to 150°
Step interval	0.02° (2θ)
Fixed counting time	10 s/step

[ii] The LaB_6 standard was a National Institute of Standards and Technology (NIST) standard reference material (SRM 660a).

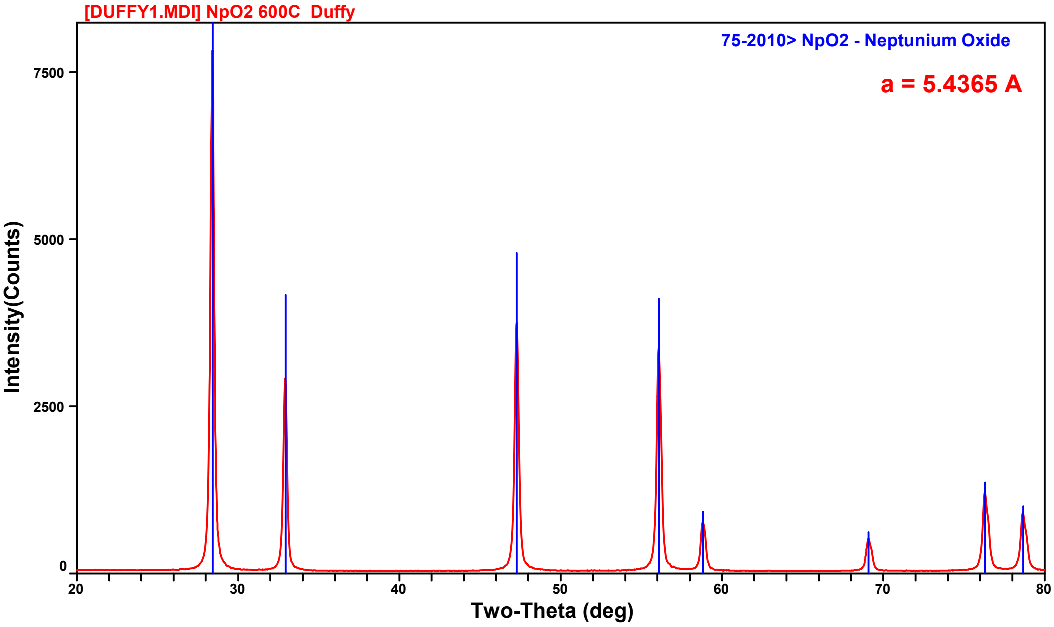


Figure 1. Powder X-ray diffraction pattern for NpO_2 calcined at 600 °C (600C-1).

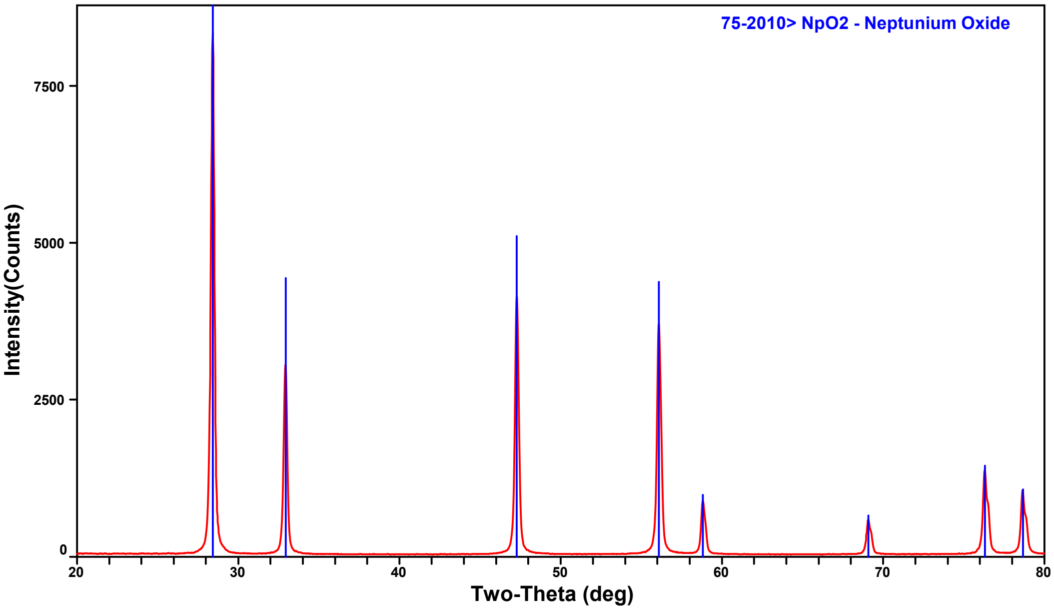


Figure 2. Powder X-ray diffraction pattern for NpO_2 calcined at 650 °C (650C-1).

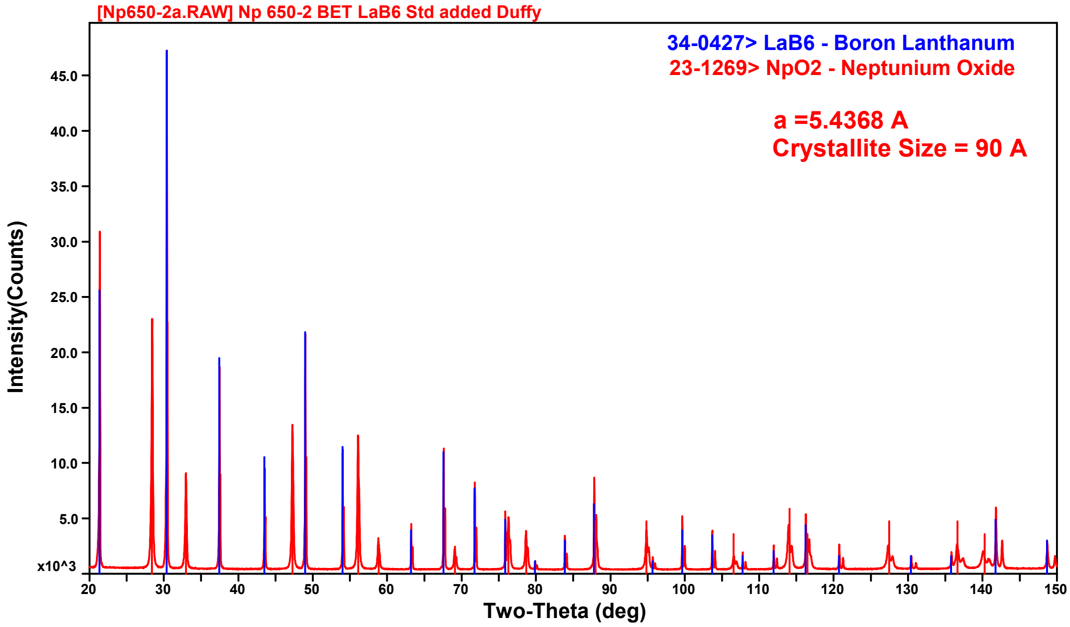


Figure 3. Powder X-ray diffraction pattern for NpO_2 calcined at 650 °C (650C-2) with lanthanum hexaboride (NIST SRM 660a) added as an internal d-spacing and instrument profile standard.

Particle Size Distribution

Scanning Electron Microscopy

The particle size distributions of the first two NpO_2 batches (600C-1 and 650C-1) were evaluated qualitatively using a Cambridge Model 250 contained scanning electron microscope (CSEM) located in a radioactive glovebox. Sample mounts were prepared in a radioactive glovebox by "dusting" a few milligrams of powder from each of the first two NpO_2 batches onto the adhesive surfaces of two sample plates. The sample plates were then removed from the preparation glovebox and transferred to the radioactive glovebox containing the CSEM for analysis.

Three separate areas were examined on each sample plate—one near the left side of the plate, one near the center, and one near the right side of the plate. For each area, four scans were made at increasing magnification levels of 50X, 250X, 500X, and 1000X. The resulting micrographs are shown in Figures 4 through 6 (600C-1) and Figures 7 through 9 (650C-1). An approximate sizing bar is shown at the lower left of each micrograph to aid the viewer in particle size estimation.

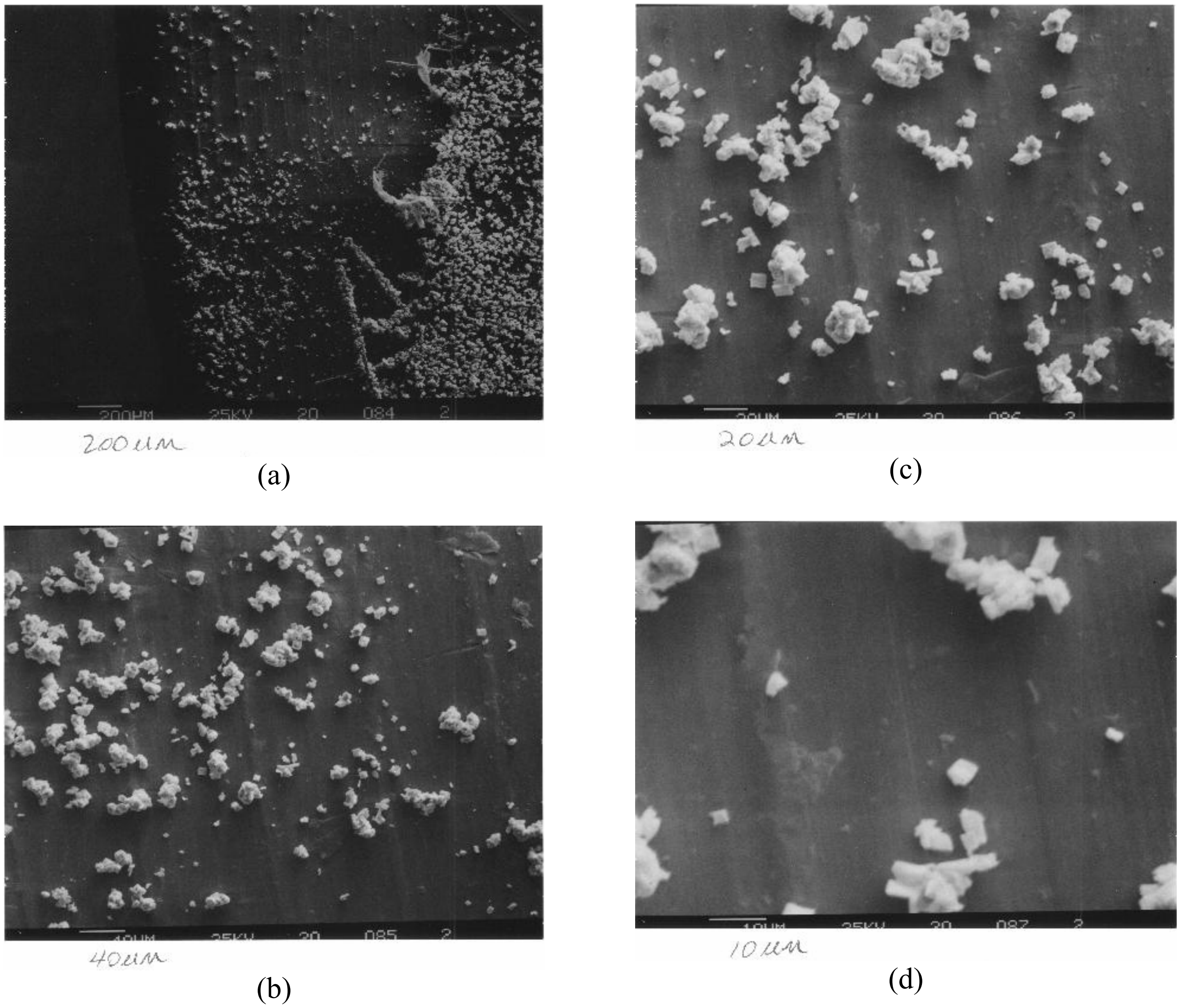
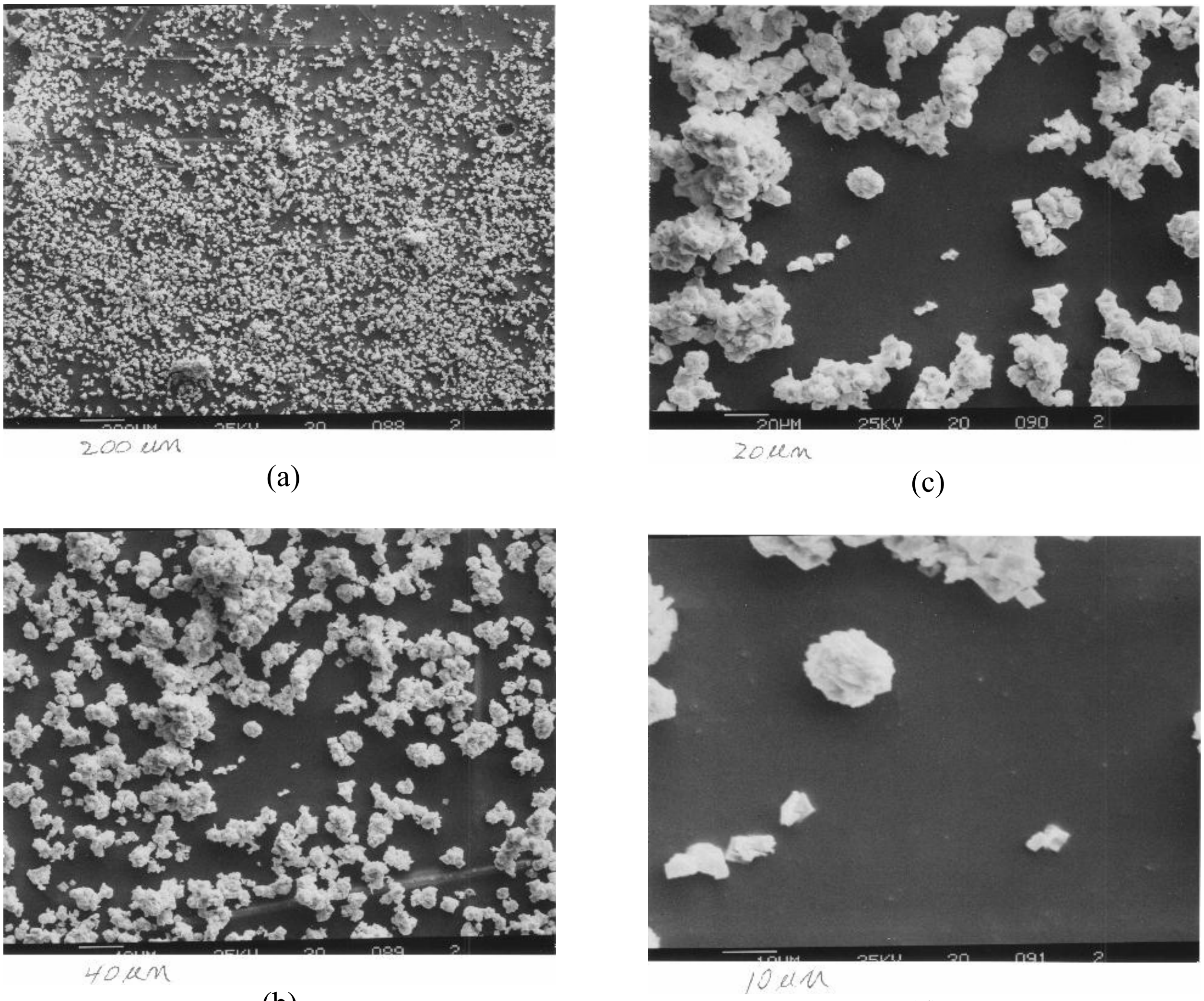


Figure 4. SEM micrographs of 600 °C NpO_2 (600C-1) taken near left side of sample mounting plate. Magnifications are (a) 50X, (b) 250X, (c) 500X, and (d) 1000X. Sizing bars are shown at the lower left of each micrograph with sizes of (a) 200 μm, (b) 40 μm, (c) 20 μm, and (d) 10 μm.



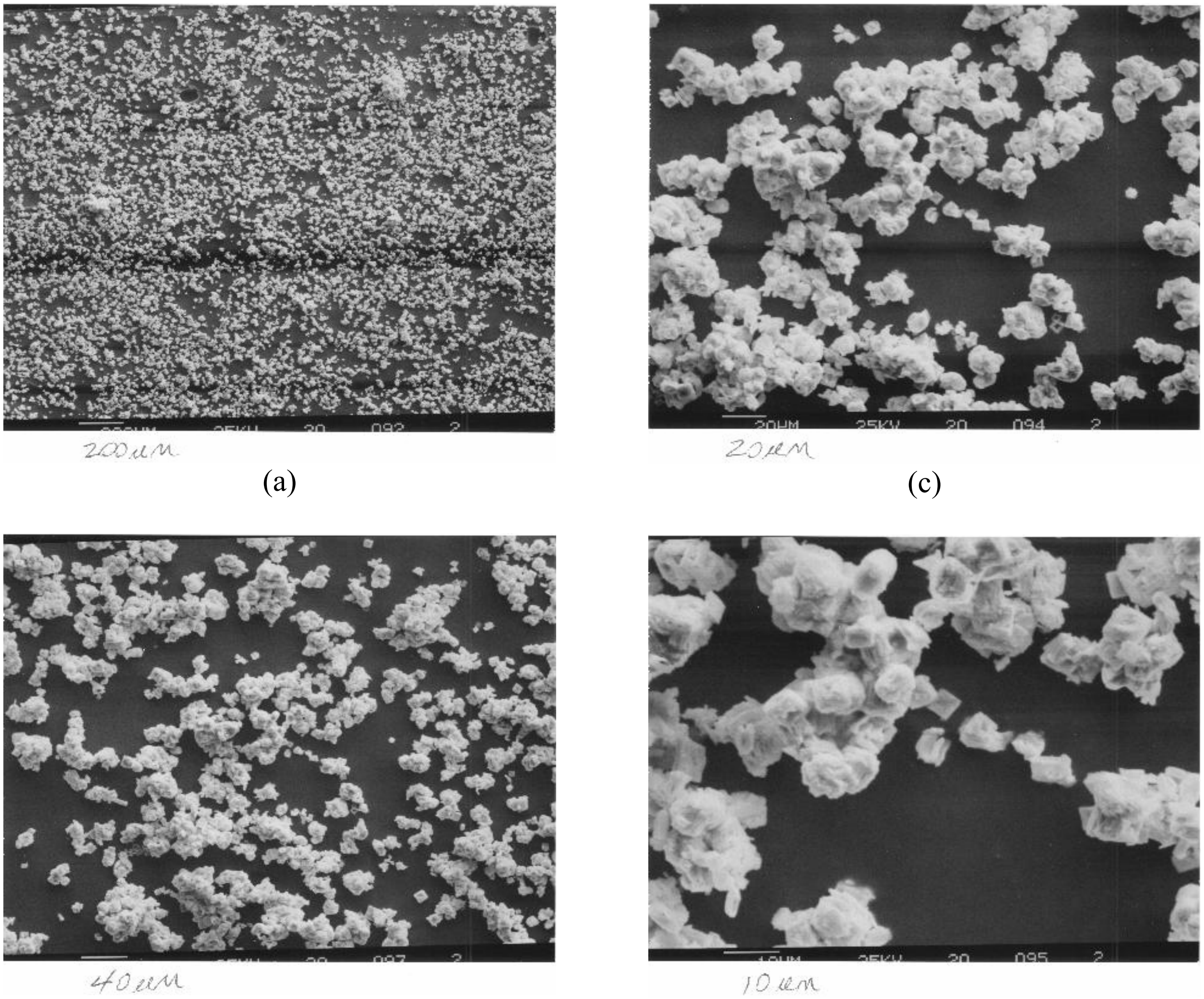
(a)

(c)

(b)

(d)

Figure 5. SEM micrographs of 600 °C NpO_2 (600C-1) taken near center of sample mounting plate. Magnifications are (a) 50X, (b) 250X, (c) 500X, and (d) 1000X. Sizing bars are shown at the lower left of each micrograph with sizes of (a) 200 μm, (b) 40 μm, (c) 20 μm, and (d) 10 μm.



(a) (c)

(b) (d)

Figure 6. SEM micrographs of 600 °C NpO_2 (600C-1) taken near right side of sample mounting plate. Magnifications are (a) 50X, (b) 250X, (c) 500X, and (d) 1000X. Sizing bars are shown at the lower left of each micrograph with sizes of (a) 200 µm, (b) 40 µm, (c) 20 µm, and (d) 10 µm.

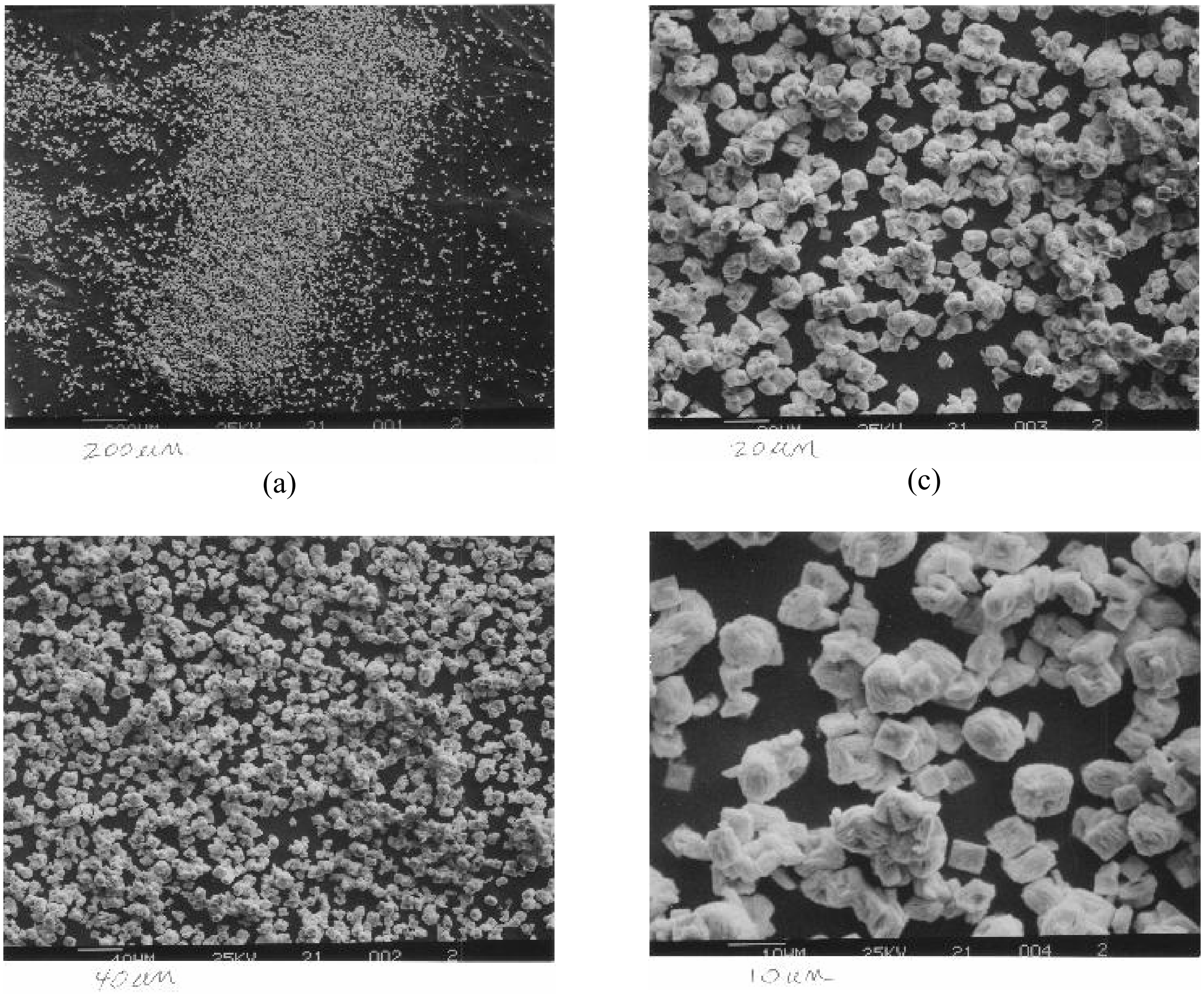
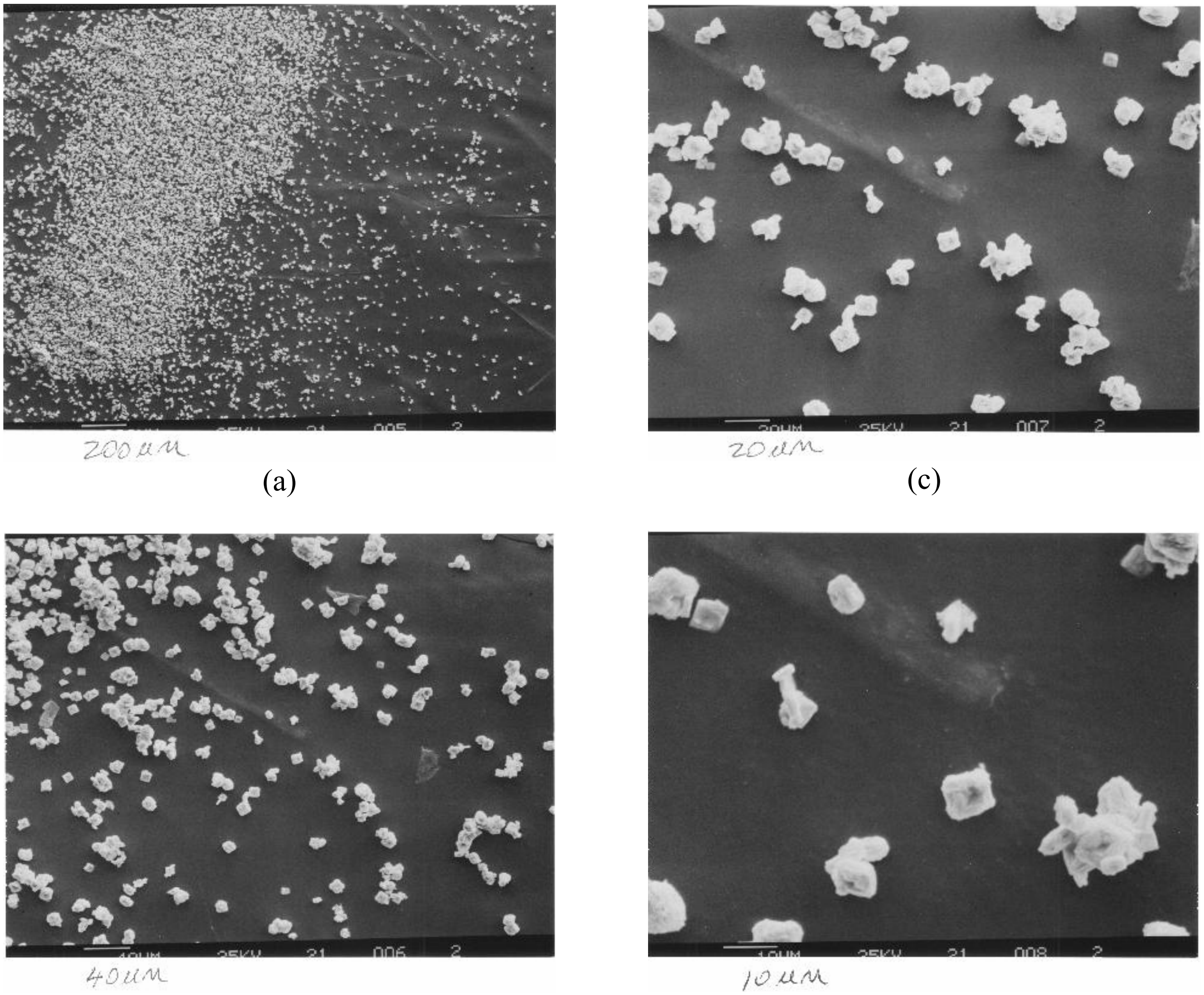


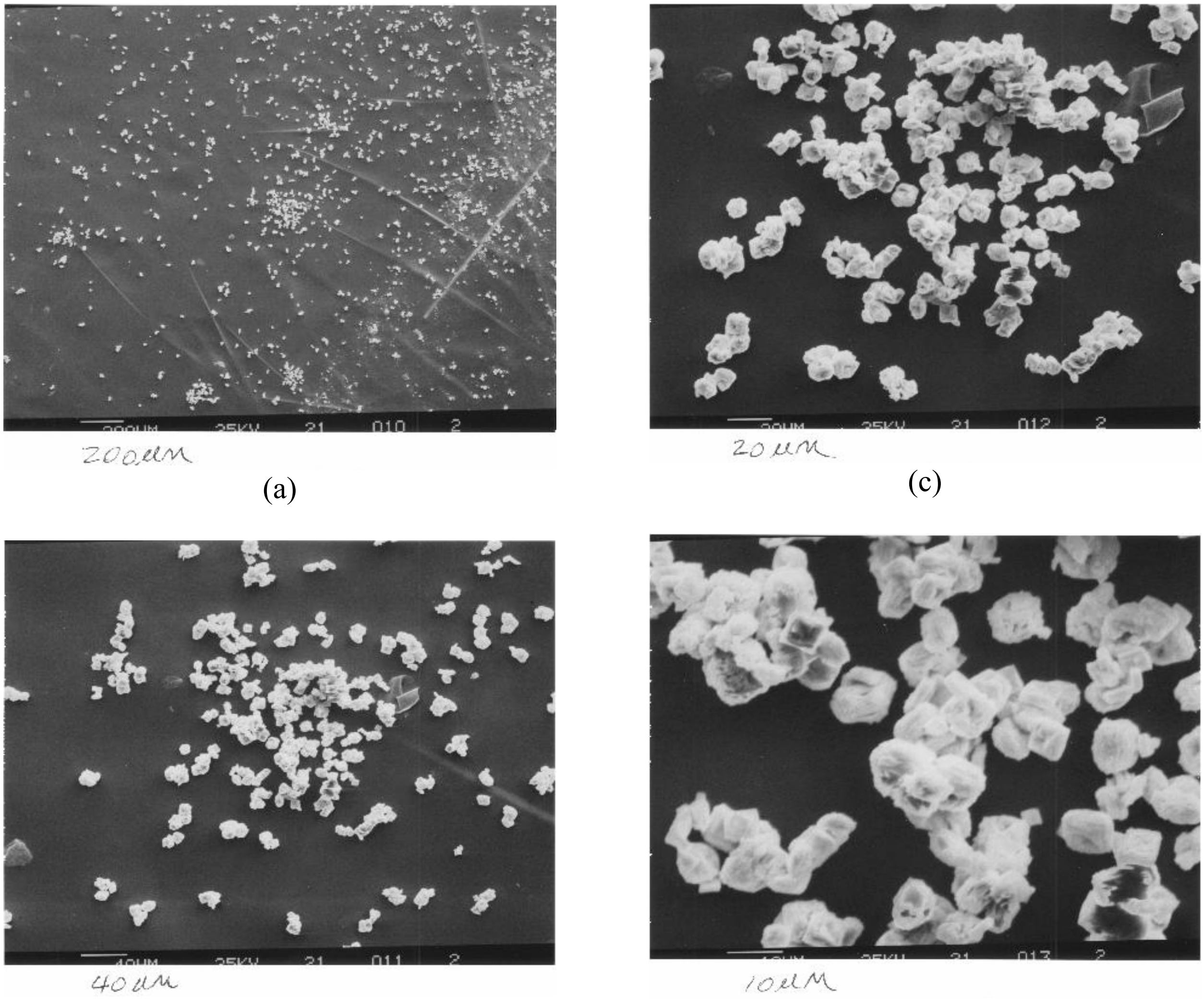
Figure 7. SEM micrographs of 650 °C NpO_2 (650C-1) taken near left side of sample mounting plate. Magnifications are (a) 50X, (b) 250X, (c) 500X, and (d) 1000X. Sizing bars are shown at the lower left of each micrograph with sizes of (a) 200 μm, (b) 40 μm, (c) 20 μm, and (d) 10 μm.



(a) (c)

(b) (d)

Figure 8. SEM micrographs of 650 °C NpO_2 (650C-1) taken near center of sample mounting plate. Magnifications are (a) 50X, (b) 250X, (c) 500X, and (d) 1000X. Sizing bars are shown at the lower left of each micrograph with sizes of (a) 200 μm, (b) 40 μm, (c) 20 μm, and (d) 10 μm.



(a) (c)

(b) (d)

Figure 9. SEM micrographs of 650 °C NpO$_2$ (650C-1) taken near right side of sample mounting plate. Magnifications are (a) 50X, (b) 250X, (c) 500X, and (d) 1000X. Sizing bars are shown at the lower left of each micrograph with sizes of (a) 200 µm, (b) 40 µm, (c) 20 µm, and (d) 10µm.

The NpO_2 particle morphologies seen in Figures 4 through 9 are similar to particle morphologies observed for PuO_2.[7] Based on these micrographs, one can say qualitatively that the majority of particles for both 600C-1 and 650C-1 oxides appear to be less than about 20 μm in diameter but greater than about 5 μm. Also, the 650C-1 oxide particles appear to be somewhat more uniform in size and shape than do the 600C-1 oxide particles. However, due to the relatively limited number of particles viewed in the micrographs, the degree of confidence in estimation of particle size was not very high and an alternative method for determining particle size distribution is desirable.

Laser Diffraction Analysis

The particle size distribution of each batch of the NpO_2 powder was evaluated quantitatively by a laser diffraction technique using a Microtrac® particle size analyzer (Leeds and Northrup Microtrac II, model number 158704). In this technique, about 250 mg of NpO_2 powder was introduced into the particle size analyzer as an aqueous slurry. The light diffraction pattern produced as the slurry passed through the laser beam was mathematically analyzed by the instrument software to determine the particle size distribution of the sample. Particle size distributions are reported as volume size distributions by this technique. Volume size distributions are equivalent to mass size distributions if the material is of constant density. The instrument is calibrated at least monthly using two different NIST standard reference materials (SRM 1984 and SRM 1003b). Certificates of analysis for the NIST reference materials and calibration data obtained before and after analysis of the NpO_2 samples are provided in Attachment 1. Based on comparison of the calibration data reports with the certificates of analysis for the standard reference materials, the method precision is less than ± 5% and the bias is less than ± 15%.

The data generated for each sample by the Microtrac® particle size analyzer are given in Tables 3 through 7. Column 1 of each table lists the channel numbers for the particle size analyzer. Each channel spans a particle size range (in microns), and the size of the channel decreases exponentially with increasing channel number. Column 2 lists the maximum size of each channel in microns. Column 3 lists the volume percent of the sample that is below the maximum channel size and column 4 lists the volume percent of the sample that falls into a given channel size range. Column 5 gives the volume percents of sample (percentile points) that are smaller than the particle diameters (in microns) listed in column 6. In Table 3, for example, 75.62% of the total sample volume consists of particles smaller than 22.00 μm in diameter (channel 11), and 25.87% of the sample volume consists of particles between 15.56 μm and 22.00 μm in diameter.

Table 3: Microtrac Particle Size Data for 600 °C NpO_2 (600C-1)

				Percentile Points[a]	
Channel #	Channel Top (μm)	Volume % Below Channel	Volume % in Channel	Volume % below Diameter	Diameter (μm)
	ABOVE	100.00	0.00	10%	5.54
1	704.00	100.00	0.00	20%	8.76
2	497.80	100.00	0.00	50%	15.62
3	352.00	100.00	0.00	80%	24.38
4	248.90	100.00	0.00	90%	29.83
5	176.00	100.00	0.00	—	—
6	124.45	100.00	0.00	—	—
7	88.00	100.00	0.00	—	—
8	62.23	100.00	0.16	—	—
9	44.00	99.84	7.48	—	—
10	31.11	92.36	16.74	—	—
11	22.00	75.62	25.87	—	—
12	15.56	49.75	22.08	—	—
13	11.00	27.67	11.03	—	—
14	7.78	16.64	6.77	—	—
15	5.50	9.87	4.89	—	—
16	3.89	4.98	2.88	—	—
17	2.75	2.10	1.89	—	—
18	1.94	0.21	0.21	—	—
19	1.38	0.00	0.00	—	—
20	0.97	0.00	0.00	—	—
	BELOW	0.00	0.00	—	—

[a] Percentile points give the volume (or mass) percent of sample with particle size below the stated diameter.

Table 4: Microtrac Particle Size Data for 600 °C NpO_2 (600C-1 Rerun)

				Percentile Points[a]	
Channel #	Channel Top (μm)	Volume % Below Channel	Volume % in Channel	Volume % below Diameter	Diameter (μm)
	ABOVE	100.00	0.00	10%	—
1	704.00	100.00	0.00	20%	8.40
2	497.80	100.00	0.00	50%	15.12
3	352.00	100.00	0.00	80%	23.46
4	248.90	100.00	0.00	90%	—
5	176.00	100.00	0.00	—	—
6	124.45	100.00	0.00	—	—
7	88.00	100.00	0.00	—	—
8	62.23	100.00	1.51	—	—
9	44.00	98.49	6.19	—	—
10	31.11	92.30	14.65	—	—
11	22.00	77.65	25.47	—	—
12	15.56	52.18	22.81	—	—
13	11.00	29.37	11.63	—	—
14	7.78	17.75	6.57	—	—
15	5.50	11.17	5.66	—	—
16	3.89	5.52	3.38	—	—
17	2.75	2.14	1.75	—	—
18	1.94	0.39	0.39	—	—
19	1.38	0.00	0.00	—	—
20	0.97	0.00	0.00	—	—
	BELOW	0.00	0.00	—	—

[a] Percentile points give the volume (or mass) percent of sample with particle size below the stated diameter.

Table 5: Microtrac Particle Size Data for 600 °C NpO_2 (600C-2)[a]

Channel #	Channel Top (μm)	Volume % Below Channel	Volume % in Channel	Percentile Points[b] Volume % below Diameter	Percentile Points[b] Diameter (μm)
	ABOVE	100.00	0.00	10%	9.87
1	704.00	100.00	0.00	20%	13.20
2	497.80	100.00	0.00	50%	20.14
3	352.00	100.00	0.00	80%	29.31
4	248.90	100.00	0.00	90%	35.50
5	176.00	100.00	0.00	—	—
6	124.45	100.00	0.00	—	—
7	88.00	100.00	0.00	—	—
8	62.23	100.00	0.90	—	—
9	44.00	99.10	13.79	—	—
10	31.11	85.30	26.79	—	—
11	22.00	58.52	29.55	—	—
12	15.56	28.96	17.29	—	—
13	11.00	11.67	4.75	—	—
14	7.78	6.92	2.30	—	—
15	5.50	4.62	1.47	—	—
16	3.89	3.15	1.73	—	—
17	2.75	1.42	1.42	—	—
18	1.94	0.00	0.00	—	—
19	1.38	0.00	0.00	—	—
20	0.97	0.00	0.00	—	—
	BELOW	0.00	0.00	—	—

[a] NpO_2 batch 600C-2 was not used in any moisture uptake or gas generation tests due to a process upset during production (see p. 16).

[b] Percentile points give the volume (or mass) percent of sample with particle size below the stated diameter.

Table 6: Microtrac Particle Size Data for 650 °C NpO_2 (650C-1)

Channel #	Channel Top (μm)	Volume % Below Channel	Volume % in Channel	Percentile Points[a] Volume % below Diameter	Percentile Points[a] Diameter (μm)
	ABOVE	100.00	0.00	10%	5.98
1	704.00	100.00	0.00	20%	7.72
2	497.80	100.00	0.00	50%	11.40
3	352.00	100.00	0.00	80%	16.29
4	248.90	100.00	0.00	90%	19.94
5	176.00	100.00	0.00	—	—
6	124.45	100.00	0.00	—	—
7	88.00	100.00	0.00	—	—
8	62.23	100.00	0.00	—	—
9	44.00	100.00	0.00	—	—
10	31.11	100.00	4.36	—	—
11	22.00	95.64	17.64	—	—
12	15.56	78.00	30.71	—	—
13	11.00	47.28	26.93	—	—
14	7.78	20.35	13.13	—	—
15	5.50	7.23	4.29	—	—
16	3.89	2.93	1.54	—	—
17	2.75	1.40	1.19	—	—
18	1.94	0.21	0.21	—	—
19	1.38	0.00	0.00	—	—
20	0.97	0.00	0.00	—	—
	BELOW	0.00	0.00	—	—

[a] Percentile points give the volume (or mass) percent of sample with particle size below the stated diameter.

Table 7: Microtrac Particle Size Data for 650 °C NpO_2 (650C-2)

Channel #	Channel Top (μm)	Volume % Below Channel	Volume % in Channel	Percentile Points[a] Volume % below Diameter	Diameter (μm)
	ABOVE	100.00	0.00	10%	5.03
1	704.00	100.00	0.00	20%	8.16
2	497.80	100.00	0.00	50%	13.15
3	352.00	100.00	0.00	80%	19.55
4	248.90	100.00	0.00	90%	23.26
5	176.00	100.00	0.00	—	—
6	124.45	100.00	0.00	—	—
7	88.00	100.00	0.00	—	—
8	62.23	100.00	0.00	—	—
9	44.00	100.00	2.01	—	—
10	31.11	97.99	9.28	—	—
11	22.00	88.71	22.90	—	—
12	15.56	65.82	29.93	—	—
13	11.00	35.88	18.03	—	—
14	7.78	17.86	6.68	—	—
15	5.50	11.18	4.05	—	—
16	3.89	7.13	3.31	—	—
17	2.75	3.82	2.69	—	—
18	1.94	1.13	0.85	—	—
19	1.38	0.28	0.28	—	—
20	0.97	0.00	0.00	—	—
	BELOW	0.00	0.00	—	—

[a] Percentile points give the volume (or mass) percent of sample with particle size below the stated diameter.

Figure 10 is a plot of the volume percent of sample in each channel versus the channel center size in microns for all four batches of NpO_2, and Table 7 compares the percentile points, average particle sizes, and distribution widths for all four batches of NpO_2. The analysis was run twice on the same sample from batch 600C-1 (dark blue and light blue curves) to demonstrate reproducibility of the instrument. The particle size distributions for batches 600C-1 and 600C-2 are slightly broader than batches 650C-1 and 650C-2, and the average particle sizes are larger. There is a larger difference in particle size distributions for batches 600C-1 and 600C-2 as compared to 650C-1 and 650C-2. This difference is probably due to the fact that a Teflon stir bar was inadvertently calcined along with batch 600C-2 during oxide production.[3] Therefore, because of the uncertainty associated with the effect of stir bar decomposition on the material, batch 600C-2 was not used in any of the subsequent moisture uptake or gas generation tests. Nevertheless, additional characterization data including SSA and moisture content of the as-calcined 600C-2 NpO_2 are presented in this report along with the data for the other three NpO_2 batches.

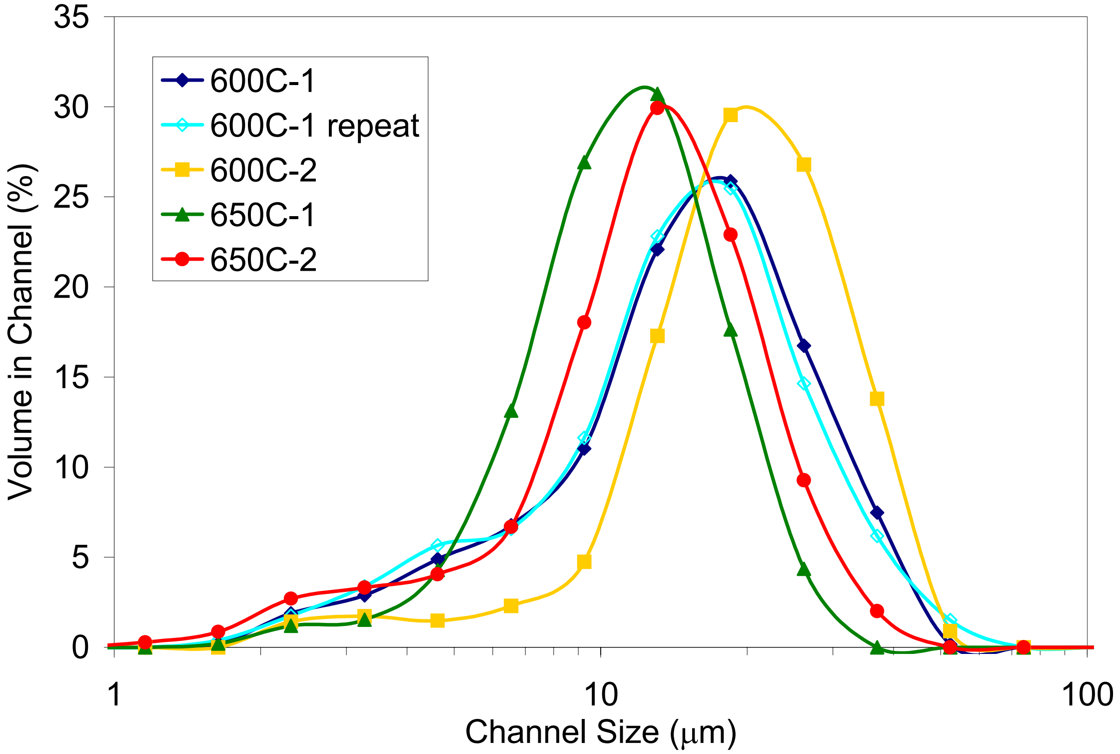


Figure 10. Particle size volume distributions for NpO_2 batches 600C-1, 600C-2, 650C-1 and 650C-2. (Batch 600C-2 was not used in moisture uptake or gas generation tests due to a process upset during production (see p. 16).)

Table 8. Summary of Particle Size Distribution Data for all NpO_2 Batches

Batch #	Percentile Points[a] (μm) 10%	20%	50%	80%	90%	Mean Diameter[b] (μm)	Particle Size Distribution Width[c] (μm)
600C-1	5.54	8.76	15.62	24.38	29.83	16.74	9.50
600C-1 (rerun)	—	8.40	15.12	23.46	—	16.53	9.39
600C-2[d]	9.87	13.20	20.14	29.31	35.50	21.06	9.26
650C-1	5.98	7.72	11.40	16.29	19.94	12.05	5.36
650C-2	5.03	8.16	13.15	19.55	23.26	13.80	6.76

[a] Percentile points show the given percent of the sample volume that is smaller than the indicated particle size (microns). The 50% point is the median particle size and is one indication of average particle size.

[b] The mean diameter is a weighted value of the average particle size, or the center of gravity of the distribution.

[c] The width of the particle size distribution (in microns) is calculated by the Microtrac software as (the particle size below which 84% of the sample volume falls minus the particle size below which 16% of the sample volume falls) divided by two.

[d] NpO_2 batch 600C-2 was not used in any moisture uptake or gas generation tests due to a process upset during production (see p. 16).

Specific Surface Area Measurements

Gas generation testing of PuO_2 has shown that specific surface area (SSA) and moisture content are two important factors in hydrogen (and oxygen) gas generation of PuO_2.[8] For PuO_2 produced by calcination of the oxalate, SSA and moisture uptake of PuO_2 decrease with increasing calcination temperature.[7, 8] Therefore, knowledge of the SSA and moisture content of the NpO_2 product as they relate to gas generation measurements is important for establishing processing controls to ensure the HB-Line NpO_2 product is adequately described by SRTC gas generation tests.

The SSA of each batch of NpO_2 produced for gas generation testing was determined by the BET (Brunauer, Emmet, and Teller) isotherm method using a Micromeretics Gemini 2360 surface area analyzer and FlowPrep 060 de-gassing unit contained in a radioactive glovebox. Table 9 lists the instrument parameters for both of these units. The BET analysis of NpO_2 is described in a separate report.[9]

Table 10 summarizes the results of the BET SSA analyses for NpO_2 and for a glass standard of similar SSA obtained from Micromeretics (Norcross, GA, USA). Based on mulitiple measurements of the glass standard, the method precision is ± 0.9% and the bias is ± 2.8%. As expected, NpO_2 calcined at 650 °C had a lower SSA than NpO_2 calcined at 600 °C. However, the specific surface areas (SSAs) were less than would be expected for PuO_2 produced in a similar manner. For example, SSAs of 39.9 ± 1.0 m^2/g, 10.2 ± 1.0 m^2/g, and 1.74 ± 0.17 m^2/g were measured for PuO_2 calcined at 450 °C, 700 °C, and 950 °C, respectively.[9] Therefore, NpO_2 calcined at 650 °C has a SSA only about twice that of PuO_2 calcined at 950 °C. This behavior is fortunate because a lower SSA should correspond to lower moisture adsorption and lower gas generation rates.

Table 9: BET Specific Surface Area Results

Batch ID	Calcination Temperature (°C)	Average SSA (m^2/g)	Precision (1σ) (%)	Bias[a] (%)
Glass Standard[b]	NA	5.14	0.9	2.8
600C-1[c]	600	5.34	0.6	NA
600C-2[d, e]	600	6.37	2.4	NA
650C-1[c]	650	3.68	1.0	NA
650C-2[c]	650	3.67	1.9	NA

[a] Assumes that the center value for the standard (Micromeritics glass standard; 5.0 ± 0.3 m^2/g) is the true surface area.
[b] Based on the average of five measurements.
[c] Based on the average of two measurements.
[d] Based on the average of seven measurements.
[e] NpO_2 batch 600C-2 was not used in any moisture uptake or gas generation tests due to a process upset during production (see p. 16).

Moisture Analysis and Uptake Studies

As discussed in the previous section, SSA and moisture content are two very important factors in the generation of hydrogen (and oxygen) by PuO_2. In gas generation tests with PuO_2, we have observed increasing gas generation rates with increasing moisture content at constant SSA.[8] Moisture content and SSA are also expected to be important factors in gas generation tests with NpO_2. Therefore, to evaluate the impact of moisture content on NpO_2 gas generation, gas generation tests were conducted with as-calcined NpO_2 (i.e., no additional moisture added) and with NpO_2 exposed to 75% RH for several days. The gas generation tests are described in a separate report.[10]

Exposure to 75% RH

Prior to the first series of gas generation tests, approximately one-half of the NpO_2 was exposed to an atmosphere of 75% RH for about four days to increase the moisture content. Seventy-five percent (75%) RH was considered a realistic upper bound for RH in the HB-Line Phase II facility. Due to the uncertainty associated with the effect of the calcined stir bar on NpO_2 batch 600C-2, this material was not included in the moisture uptake or gas generation tests. Therefore, NpO_2 batch 600C-1 was divided into two roughly equal samples (600C-1A and 600C-1B). Samples 600C-1A and 650C-2 were maintained in vials sealed in glovebox air while samples 600C-1B and 650C-1 were exposed to 75% RH.

Exposure to 75% RH for test series one was accomplished by storing the samples over a saturated sodium chloride (NaCl) solution maintained in a small desiccator. The samples were contained in quartz dishes and an empty quartz dish was included as a control. The saturated NaCl solution was prepared according to an American Society for Testing and Materials (ASTM) standard, "Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions".[11] The sample and control dishes were weighed before, during and after exposure to 75% RH to monitor moisture uptake by weight gain. The maximum net weight gains observed during the four-day exposure were about 0.15 wt % and 0.08 wt % for samples 600C-1B and 650C-1, respectively. However, due to the relatively small amount of moisture adsorbed by the samples and the rate at which moisture was desorbed when the samples were removed from the humid atmosphere, tracking moisture uptake by weight gain was not considered a reliable indication of moisture content. After four days exposure to 75% RH, sample aliquots of NpO_2 were removed from each container and stored in sealed vials for about eight weeks until they could be analyzed for moisture content. The first gas generation test was then initiated with four samples: 600C-1A and 650C-2 (as-calcined); 600C-1B and 650C-1 (exposed to 75% RH).[10]

Following the first series of gas generation tests, the four NpO_2 samples were removed from the test containers and all four samples were exposed to 75% RH. To minimize the chance that the sample containers came into contact with the saturated sodium chloride solution and to ensure equilibration of the samples with the humid atmosphere, a different method of exposure to 75% RH was devised. For this exposure method, the saturated NaCl solution was placed in a 1-L plastic bottle, and the NpO_2 samples were stored in stainless steel inserts in a separate glass container. The stainless steel inserts were used to contain the NpO_2 samples during the gas generation tests. The sample container and the NaCl container were connected with two pieces of Tygon plastic tubing to allow the headspace gas to be circulated continuously between the containers. The samples were exposed in this manner for a total of about 20 days. Again, the sample containers were weighed before, during and after exposure to 75% RH, and again the weight changes were relatively small, and the samples lost weight rapidly when removed from the humid atmosphere. The maximum observed net weight gains were as follows: 0.18 wt % (600C-1A), 0.20 wt % (600C-1B), 0.10 wt % (650C-1), and 0.12 wt % (650C-2). Prior to initiating the second gas generation test series, aliquots of each NpO_2 sample were removed and analyzed immediately for moisture content.

Moisture Analysis

The moisture contents of NpO_2 samples used in gas generation tests were measured by thermogravimetric analysis coupled with mass spectrometry (TGA-MS). This measurement technique allows determination of both total mass losses at 1000 °C and identification and, in some instances, quantification of the constituents volatilized during heating. The precision (1σ) of the TGA mass loss measurement was found to be ± 0.6% based on six measurements of a gypsum standard over a two-month period. The accuracy of these measurements was ± 0.5%. The estimated precision (1σ) for duplicate NpO_2 samples was about ± 2%. Details of the TGA-MS analysis are described in a separate report.[9]

Figure 11 shows the TG mass loss curves for NpO_2 samples from the first gas generation test series. Total mass losses of approximately 0.2 wt % and 0.3 wt % were observed for NpO_2 calcined at 650 °C and 600 °C, respectively. In both instances samples exposed to 75% RH had slightly smaller mass losses than the as-calcined samples, and the reason for this is not yet known.

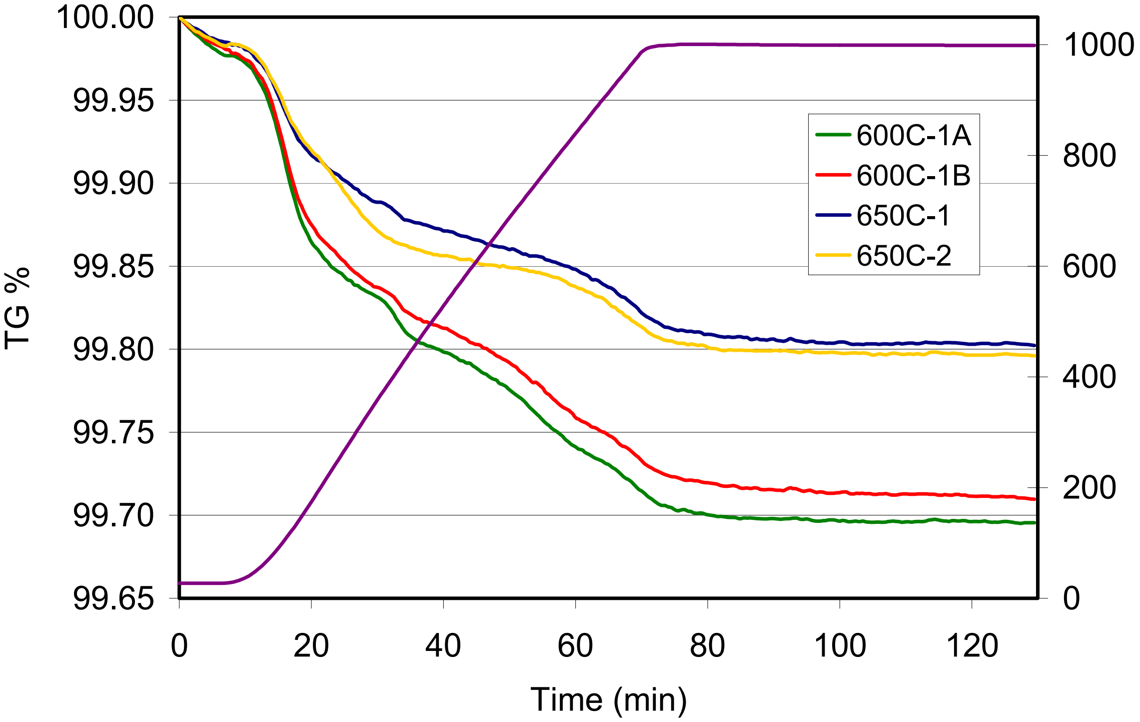


Figure 11. TG mass loss plot for NpO_2 samples from the first gas generation test series. Samples were conditioned as follows: 600C-1A (as-calcined); 600C-1B (75% RH); 650C-1 (75% RH); 650C-2 (as-calcined).

Figure 12 is a combined plot of the TGA mass loss curve and the predominant masses detected by the MS for sample 600C-1A. The rate of heating was 20 °C per minute. In addition to water (mass 18), other species detected in significant quantities included oxygen (O, mass 16); nitric oxide (NO, mass 30), sulfur dioxide (SO_2, mass 64), and carbon (C, mass 12). For these NpO_2 samples, the most likely sources of NO, SO_2, and C are residual nitrate (NO_3^-), sulfamate ($SO_3NH_2^-$) or sulfate (SO_4^{2-}), and oxalate ($C_2O_4^{2-}$) or carbonate (CO_3^{2-}) ions, respectively. Based on the relative intensities of the various masses, one can see that only about one-third of the total mass loss is due to water.

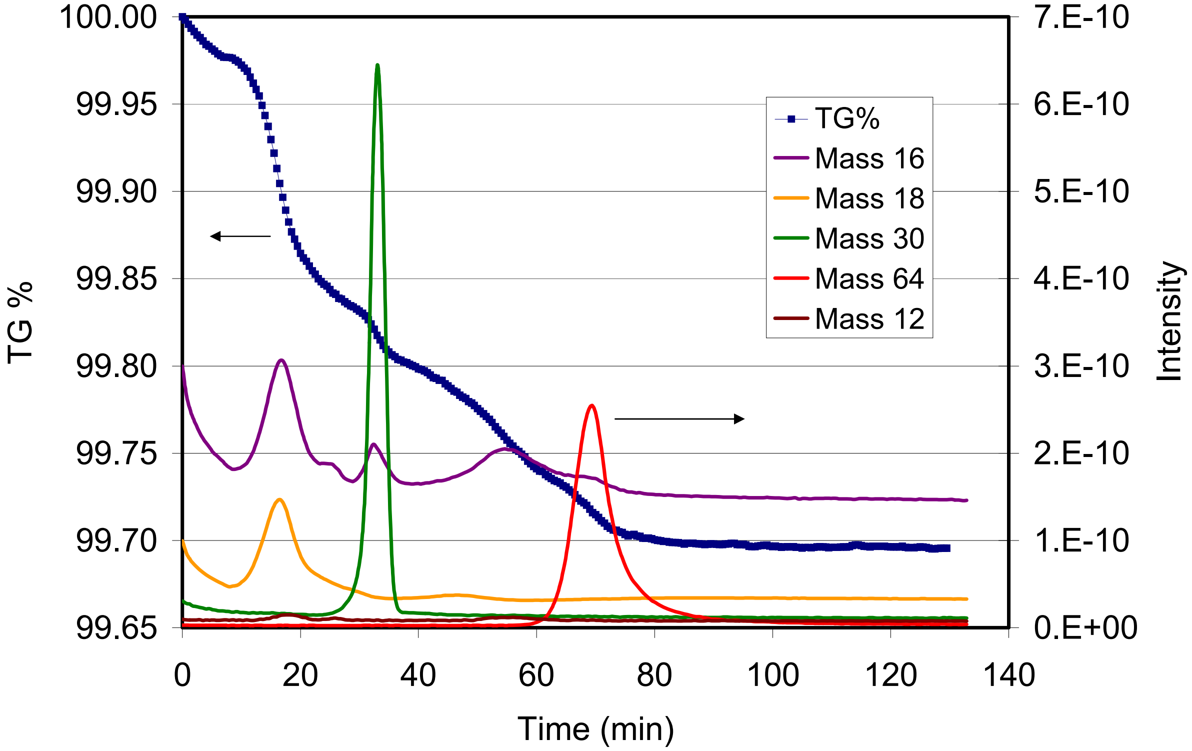


Figure 12. TGA-MS results for NpO_2 sample 600C-1A. The rate of heating was 20 °C per minute.

Table 10 summarizes the TGA-MS results for NpO_2 from both the first and second gas generation test series. The SSA measurement results are also included for ease of evaluating the effect of SSA on total mass loss and moisture content. As might be expected, samples having higher SSA were found to have higher mass losses when fired to 1000 °C. The percentage of total mass loss due to loss of water ranged from about 30 to 50%. Water losses measured by TGA-MS for samples not exposed to 75% RH ranged from 0.076 to 0.092 wt %. During exposure of the samples to high humidity, maximum weight gains from 0.08 to 0.20 wt % were observed. However, water losses measured by TGA-MS for samples exposed to 75% RH were only about 0.02 to 0.04 wt % greater than for samples that were not exposed to high humidity. Thus, the difference in water loss measured by TGA-MS before and after exposure to 75% RH was only 20 to 25% as great as expected based on the observed weight gains. We do not know at this time if this lower than expected water content is due primarily to a negative bias in the MS water measurement or to co-adsorption of some species other than water vapor during exposure to 75% RH.

Table 10: NpO_2 TGA-MS Results

Test Series	Sample ID	Exposed to 75% RH (yes/no)	Total Mass Loss (Wt %)	H_2O Loss (Wt %)	SSA (m^2/g)
1	600C-1A	No	0.304	0.092	5.34
1	600C-1B	Yes	0.290	0.13	5.34
NA[a, b]	600C-2	No	0.339	0.17	6.38
NA	650C-1	No	0.210	0.076	3.68
1	650C-1	Yes	0.198	0.091	3.68
1	650C-2	No	0.204	0.077	3.67
2	600C-1A	Yes	0.323	0.13	5.34
2	600C-1B	Yes	0.350	0.14	5.34
2	650C-1	Yes	0.207	0.11	3.68
2	650C-2	Yes	0.213	0.071	3.67

[a] NA indicates materials were analyzed by TGA-MS but were not used in gas generation tests.

[b] NpO_2 batch 600C-2 was not used in any moisture uptake or gas generation tests due to a process upset during production (see p. 16).

Conclusions

Characterization of NpO_2 produced for use in gas generation tests included (1) bulk and tap density measurements, (2) X-ray diffraction, (3) particle size distribution, (4) SSA measurements, and (5) total mass loss and moisture analysis before and after exposure to 75% RH. The bulk and tap densities for NpO_2 calcined at 600 °C were determined to be 1.90 ± 0.03 g/cm^3 and 2.50 ± 0.04 g/cm^3, respectively. The unit cell dimension of NpO_2 calcined at 650 °C was determined by X-ray diffraction analysis using an internal reference standard and found to be 5.4368 Å with a crystallite size of 90 Å. No gross impurities were detected. The average particle size ranges were 16 ± 10 μm and 13 ± 7 μm for NpO_2 calcined at 600 °C and 650 °C, respectively. The average SSA of the 600 °C NpO_2 (600C-1) was 5.34 ± 0.03 m^2/g, and the average SSA of the 650 °C NpO_2 (650C-1 and 650C-2) was 3.68 ± 0.08 m^2/g. These SSAs are intermediate between the SSA measured for PuO_2 calcined at 700 °C (10.83 m^2/g) and 950 °C (1.89 m^2/g). Total mass losses on heating to 1000 °C were about 0.3 wt % for 600 °C NpO_2 and about 0.2 wt % for 650 °C NpO_2. Maximum moisture contents determined by TGA-MS following exposure to 75% RH were about 0.14 wt % and 0.11 wt % for NpO_2 calcined at 600 °C and 650 °C, respectively.

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Attachment

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1984

Thermal Spray Powder – Particle Size Distribution
Tungsten Carbide/Cobalt (Acicular)

This Standard Reference Material (SRM) is primarily intended for use in the calibration of equipment used to measure particle size distributions (PSD) in the 9 um to 30 μm range. SRM 1984 consists of a single bottle containing approximately 14 g of tungsten carbide/cobalt powder.

The PSD values at five mass percentiles were measured by scanning electron microscopy (SEM) and laser light scattering (LLS). The certified PSD values by SEM are listed in Table 1. These certified values were determined by the measurement of over 10 000 individual particles from three bottles. The reference PSD values by LLS are listed in Table 2. A comparison of the two methods is shown in Figure 1.

Table 1. Certified PSD Values by SEM

Cumulative Mass Fraction (%)	Certified Diameter (μm)	Uncertainty* (μm)
10	10.3	0.9
25	13.2	0.9
50	17.1	2.2
75	21.3	1.6
90	26.3	0.9

* The uncertainty at each percentile, computed according to the ISO and NIST Guides [1], is an expanded uncertainty at the 95 % level of confidence.

Expiration of Certification: This certification is valid until **August 2010** provided the sample dispersion procedures are rigorously followed. The powder contains no organic binder and is, therefore, considered chemically stable.

The concept for the development of this SRM was provided by S.J. Dapkunas of the NIST Ceramics Division.

The SRM measurement technique, development, and certification were performed by J.F. Kelly and P. Pei of the NIST Ceramics Division.

Statistical analyses were performed by H-k. Liu of the NIST Statistical Engineering Division.

The support aspects of the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by R.J. Gettings and B.S. MacDonald.

Stephen W. Freiman, Chief
Ceramics Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 December 2000

Nancy M. Trahey, Chief
Standard Reference Materials Program

Measurement of SRM: The starting material was a 10 kg batch from a single lot (W2203A) of Type SD 251 tungsten carbide/cobalt powder obtained from OSRAM Sylvania Products, Inc.[1], Towanda, PA. This powder was chosen for its size distribution, irregularly shaped particle morphology (Figure 2), resistance to fracture on handling, and low level of aggregation.

The powder was split into bottle units containing approximately 14 g each by using spinning rifflers. A randomized set of 100 bottles from 672 bottles was selected for homogeneity testing, round robin study using LLS instruments, and certification analyses by SEM. Homogeneity testing of 14 randomly selected bottles, measured in duplicate, was performed with a LLS instrument at NIST. The data showed no evidence of size heterogeneity.

Scanning Electron Microscopy Analysis: SEM based image analysis was carried out on three bottles. Sample preparation for microscopy entailed both a reduction in the mass of powder and a separation into size fractions. The size fractionation was accomplished by sieving using sieves with nominal openings of 10 µm, 20 µm, and 30 µm. Subsamples from each of the sieve splits were then produced by successive division using a spinning riffler.

SEM images were acquired for each of the sieve fractions. The backscatter electron images of the particles were acquired as greyscale image files into a computer via a digital interface. The 2048 × 2048 pixel images were analyzed to obtain the projected areas of the tungsten carbide/cobalt particles. These areas were fitted with ellipses and the major and minor axes converted to particle volumes (prolate ellipsoids) and particle diameters using the average of the three ellipsoid axes. The pixel-to-length conversion was calibrated using a micrometer slide calibrated at NIST using laser interferometry. Several hundred particles of each sieve fraction were measured for a total of approximately 2400 particles measured from each bottle. Particle size distributions describing the percent of powder mass represented by particles with diameters less than a given length were calculated using the weighting factors obtained from the sieving results. The diameter values corresponding to the specific mass fractions of 10 %, 25 %, 50 %, 75 %, and 90 % are listed in Table 1. Current practice in the thermal spray industry is to specify these values to define the particle size distribution. A graphical comparison of the distribution measured by SEM with the mean distribution obtained by LLS is shown in Figure 1.

Laser Light Scattering Method (Round Robin Study): Ten laboratories participated in this round robin study. Each round robin participant received two bottles for analysis using their light scattering instruments. The reference distribution values given in Table 2 are based on 20 measurements by the 10 participating laboratories. The LLS data from all 10 laboratories were obtained using such instruments as the Horiba LA910, Microtrac X100, Sympatec HELOS, and Beckman Coulter LS 230 following the sample preparation procedure specified by NIST. Therefore, in the use of this SRM, the sample dispersion procedure is mandatory; otherwise, the data may not be comparable. Although the light scattering instruments produce a continuous plot of weight percent finer than a given diameter, five cumulative percentiles were selected as a representative data set for certification since it is consistent with the industrial practice.

The following individuals and companies participated in the development of this SRM:

T. Weigel and P. Plantz, Honeywell, Microtrac Solution Center, Clearwater, FL
J. Wolfgang, SYMPATEC, Inc., Princeton, NJ
L. Vu, P. Fernando, and M. Pohl, Horiba Instruments, Inc., Irving, CA
L. Updegraff and R. Neiser, Sandia National Laborabories, Albuquerque, NM
J. Bohan, API, Hadley, MA
J. Vasiliou, Duke Scientific Corporation, Palo Alto, CA
K. Cowan and R. Iacocca, P/M Laboratory, Pennsylvania State University, State College, PA
H.D. Garrelts, Stellite Coatings, Goshen, IN
H. Hildebrand, Beckman Coulter, Miami, FL
R. Simmons, Dirats Laboratories, Westfield, MA
F. Venskytis, OSRAM Sylvania Products, Inc., Towanda, PA

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[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 2. PSD Reference Values by LLS

Cumulative Mass Fraction (%)	Reference Value (μm)	Uncertainty* (μm)
10	9.5	0.8
25	12.5	0.8
50	16.5	0.9
75	21.3	1.2
90	26.5	1.3

* The uncertainty at each percentile, computed according to the ISO and NIST Guides [1], is an expanded uncertainty at the 95 % level of confidence.

SAMPLE DISPERSION AND MEASUREMENT PROCEDURES FOR LIGHT SCATTERING METHOD

Sample Dispersion Procedure: Application of the reference values produced by LLS requires following this sample dispersion procedure; otherwise, results may not be comparable to the reference values listed in Table 2. **NOTE:** Each sample bottle contains approximately 14 g tungsten carbide/cobalt powder that is sufficient for many analyses by LLS instruments.

1. Use a microriffler for splitting the sample into subsamples of the appropriate mass as specified by the manufacturer's instructions.

2. Add 4 % aqueous sodium pyrophosphate solution to each subsample at the ratio of 1.00 mL/g of powder and make a paste by mixing gently with a spatula. **DO NOT use a magnetic stirrer since the powder will attach to the magnet and may be crushed during stirring.**

3. Transfer the paste quantitatively (totally) into the measuring cell containing distilled water whose pH has been pre-adjusted to 9.5 ± 0.1 with 1 mol/L sodium hydroxide solution. Flush the container with pH-adjusted distilled water to complete the transfer.

4. Follow the instrument manufacturers' instructions for instrument calibration and operation.

REFERENCE

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); available at http://physics.nist.gov/Pubs/.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

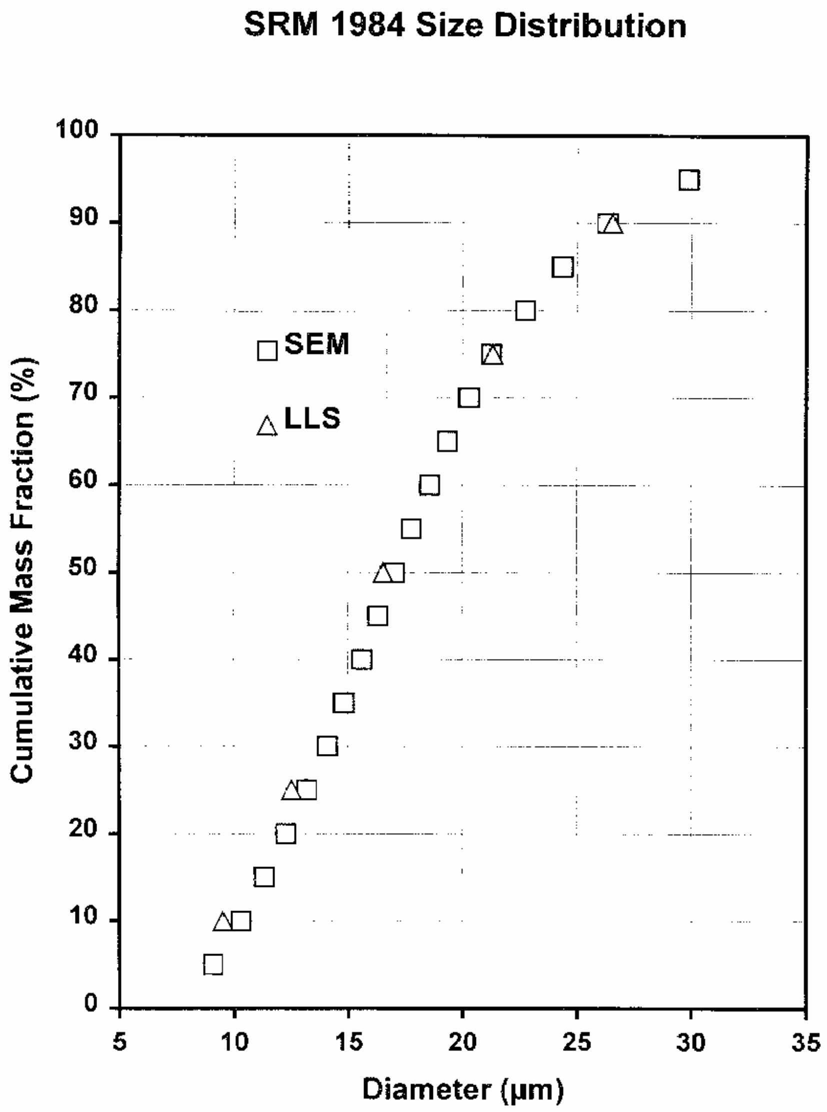


Figure 1. SRM 1984 Size Determination by SEM and LLS

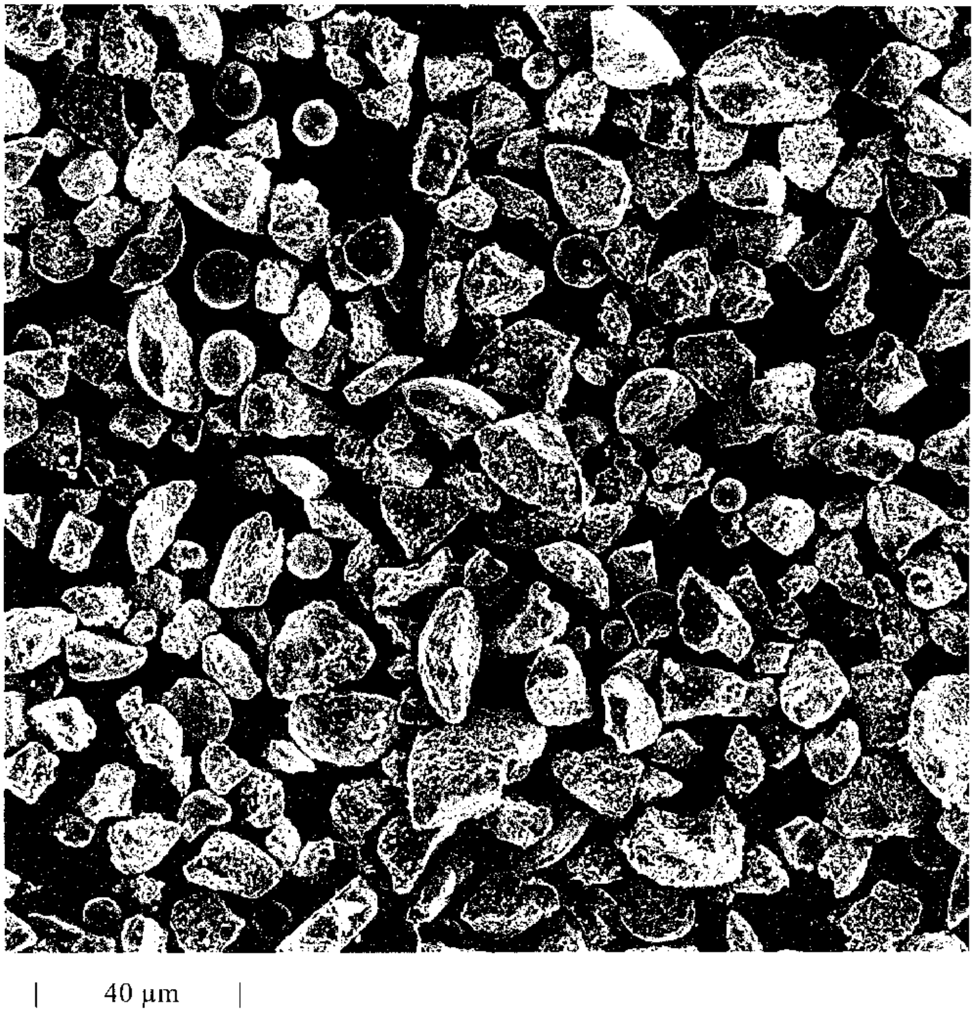
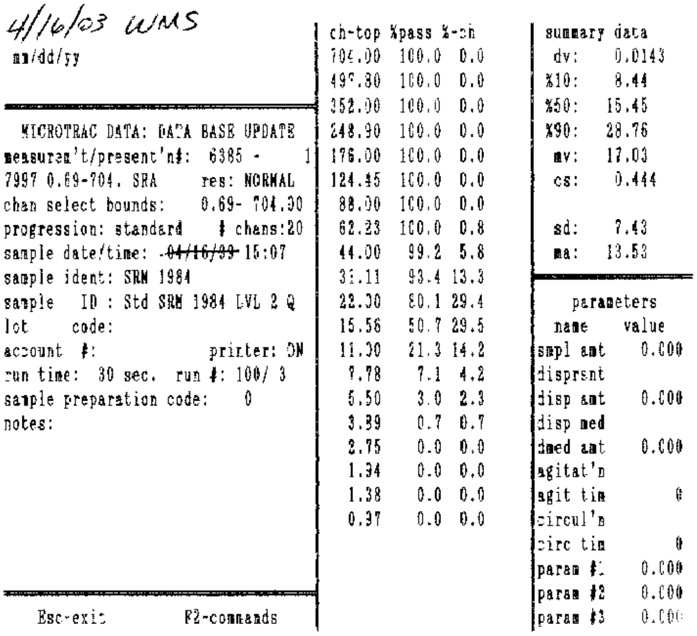


Figure 2. SEM Micrograph of SRM 1984 Powder



4/16/03 WMS

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mm/dd/yy

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7997 0.69-704. SRA      res: NORMAL
chan select bounds:     0.69- 704.00
progression: standard   # chans:20
sample date/time: ~~04/16/99~~ 15:07
sample ident: SRM 1984
sample   ID : Std SRM 1984 LVL 2 Q
lot     code:
account  #:                printer: ON
run time:  30 sec.  run #: 100/ 3
sample preparation code:     0
notes:

  Esc-exit           F2-commands
```

```
ch-top %pass %-ch
704.00  100.0  0.0
497.30  100.0  0.0
352.00  100.0  0.0
248.90  100.0  0.0
176.00  100.0  0.0
124.45  100.0  0.0
 88.00  100.0  0.0
 62.23  100.0  0.8
 44.00   99.2  5.8
 31.11   93.4 13.3
 22.00   80.1 29.4
 15.56   50.7 29.5
 11.00   21.3 14.2
  7.78    7.1  4.2
  5.50    3.0  2.3
  3.89    0.7  0.7
  2.75    0.0  0.0
  1.94    0.0  0.0
  1.38    0.0  0.0
  0.97    0.0  0.0
```

```
summary data
 dv:     0.0143
%10:     8.44
%50:    15.45
%90:    28.76
 mv:    17.03
 cs:     0.444

 sd:     7.43
 ma:    13.53

   parameters
 name     value
smpl amt    0.000
disprsnt
disp amt    0.000
disp med
dmed amt    0.000
agitat'n
agit tim        0
circul'n
circ tim        0
param #1    0.000
param #2    0.000
param #3    0.000
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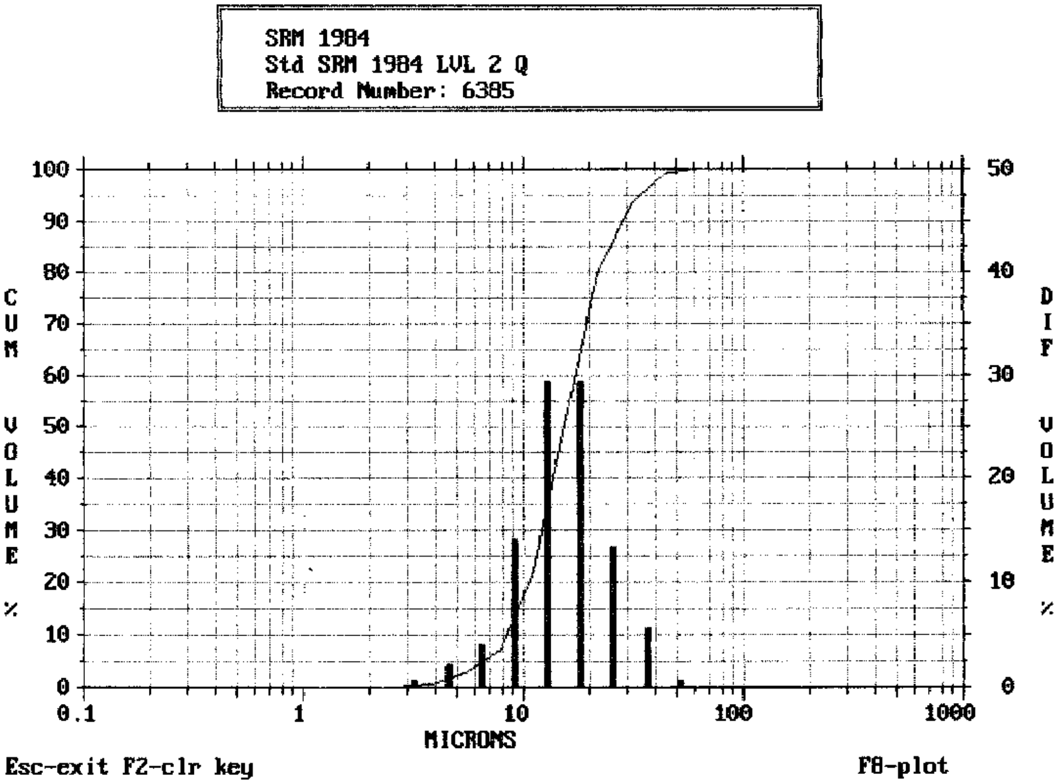
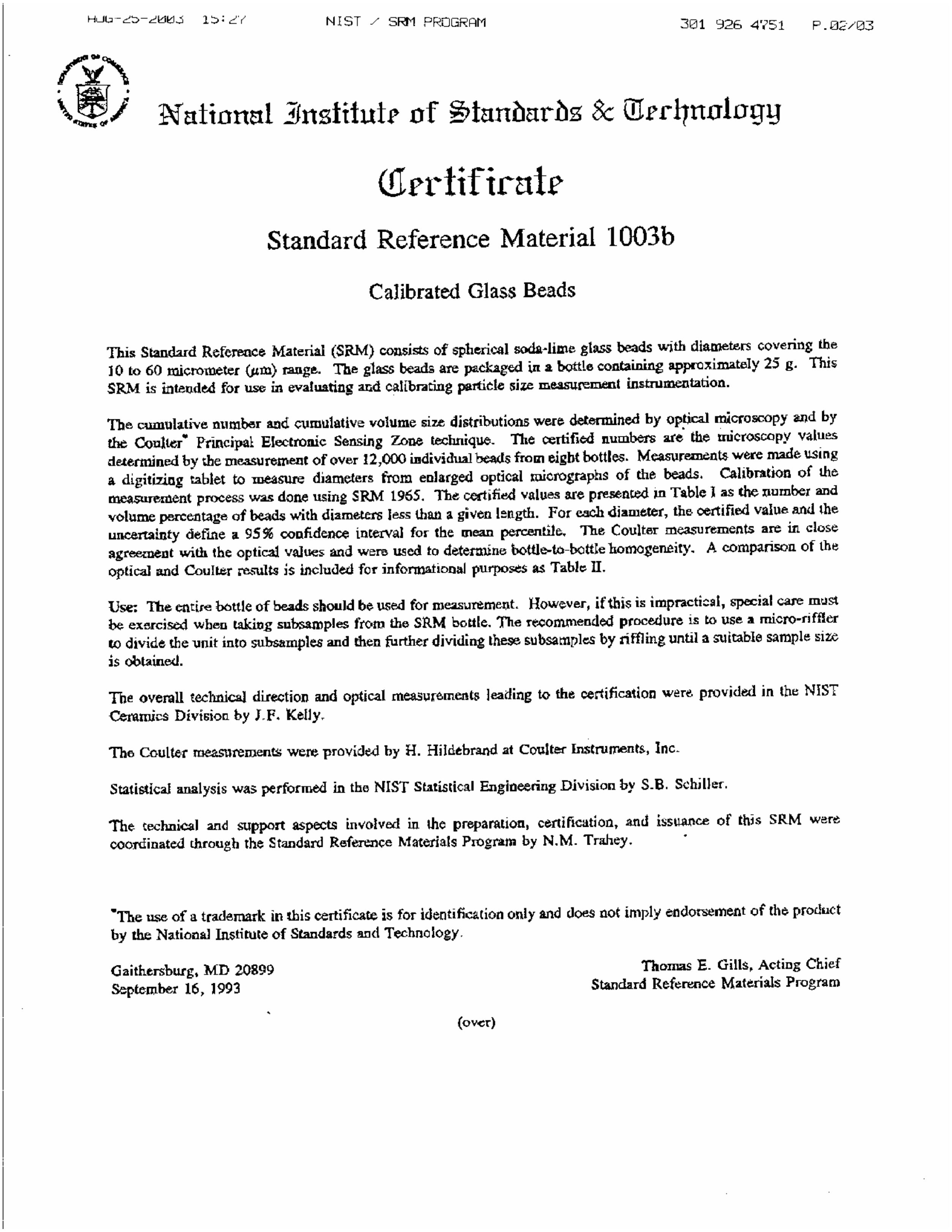


SRM 1984
Std SRM 1984 LVL 2 Q
Record Number: 6385



AUG-25-2003 15:27 NIST / SRM PROGRAM 301 926 4751 P.02/03

National Institute of Standards & Technology

Certificate

Standard Reference Material 1003b

Calibrated Glass Beads

This Standard Reference Material (SRM) consists of spherical soda-lime glass beads with diameters covering the 10 to 60 micrometer (µm) range. The glass beads are packaged in a bottle containing approximately 25 g. This SRM is intended for use in evaluating and calibrating particle size measurement instrumentation.

The cumulative number and cumulative volume size distributions were determined by optical microscopy and by the Coulter* Principal Electronic Sensing Zone technique. The certified numbers are the microscopy values determined by the measurement of over 12,000 individual beads from eight bottles. Measurements were made using a digitizing tablet to measure diameters from enlarged optical micrographs of the beads. Calibration of the measurement process was done using SRM 1965. The certified values are presented in Table I as the number and volume percentage of beads with diameters less than a given length. For each diameter, the certified value and the uncertainty define a 95% confidence interval for the mean percentile. The Coulter measurements are in close agreement with the optical values and were used to determine bottle-to-bottle homogeneity. A comparison of the optical and Coulter results is included for informational purposes as Table II.

Use: The entire bottle of beads should be used for measurement. However, if this is impractical, special care must be exercised when taking subsamples from the SRM bottle. The recommended procedure is to use a micro-riffler to divide the unit into subsamples and then further dividing these subsamples by riffling until a suitable sample size is obtained.

The overall technical direction and optical measurements leading to the certification were provided in the NIST Ceramics Division by J.F. Kelly.

The Coulter measurements were provided by H. Hildebrand at Coulter Instruments, Inc.

Statistical analysis was performed in the NIST Statistical Engineering Division by S.B. Schiller.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

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*The use of a trademark in this certificate is for identification only and does not imply endorsement of the product by the National Institute of Standards and Technology.

Gaithersburg, MD 20899
September 16, 1993

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

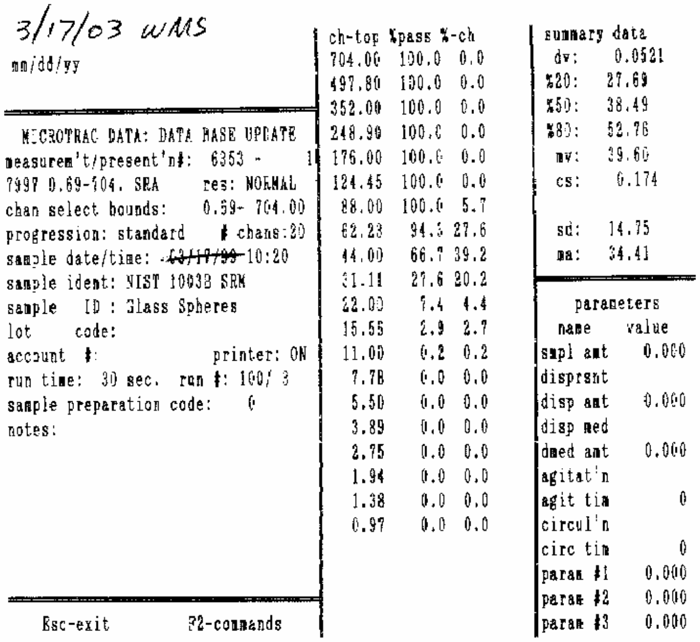
Table I. Certified Cumulative Size Distribution
Percent of Spheres with Diameters Less Than the Size Indicated

Diameter (μm)	Number (%)	Uncertainty (%)	Volume (%)	Uncertainty (%)
6	0.08	0.07	0.00	0.00
8	0.54	0.13	0.009	0.003
10	1.61	0.35	0.03	0.01
12	3.70	0.62	0.13	0.03
14	6.98	1.13	0.39	0.08
16	11.69	1.80	0.93	0.19
18	17.44	2.54	1.92	0.38
20	24.76	3.17	3.62	0.65
22	32.85	3.60	6.18	0.94
24	40.87	3.62	9.47	1.18
26	48.90	3.66	13.72	1.53
28	55.86	3.44	18.33	1.70
30	62.00	3.25	23.38	2.03
32	67.89	2.99	29.29	2.13
34	73.19	2.49	35.67	2.12
36	77.83	2.05	42.38	1.93
38	82.48	1.65	50.23	1.93
40	86.39	1.35	57.99	2.30
42	89.75	1.22	65.74	2.92
44	92.70	0.84	73.60	2.05
46	95.08	0.54	80.92	1.50
48	97.15	0.20	88.02	0.85
50	98.91	0.23	95.01	1.06
52	99.74	0.09	98.69	0.42
54	99.96	0.05	99.79	0.27
56	99.98	0.04	99.94	0.14
58	99.99	0.02	100.00	0.00
60	100.00	0.00	100.00	0.00

Table II. Volume % Less Than Diameter Values for Optical and Coulter Techniques
(Results are given for information only)

	Diameter (μm)	
Volume %	Optical	Coulter
10	24.2	25.9
20	28.8	30.0
30	32.2	33.3
40	35.3	36.2
50	37.9	38.9
60	40.5	41.6
70	43.2	44.1
80	45.8	46.7
90	48.6	49.7

The density of the glass is 2.445 g/cm^3 as determined by helium gas pycnometry. This value is not certified, but is provided for those wishing to apply Stokes' Law calculations.



3/17/03 wMS

```
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                                         704.00  100.0  0.0      dv:    0.0521
                                         497.80  100.0  0.0     %20:   27.69
                                         352.00  100.0  0.0     %50:   38.49
 MICROTRAC DATA: DATA BASE UPDATE        248.90  100.0  0.0     %80:   52.76
measurem't/present'n#:  6353 -      1    176.00  100.0  0.0       mv:  39.60
7997 0.69-704. SRA      res: NORMAL      124.45  100.0  0.0       cs:   0.174
chan select bounds:    0.69- 704.00       88.00  100.0  5.7
progression: standard    # chans:20       62.23   94.5 27.6       sd:  14.75
sample date/time: ~~03/17/99~~ 10:20          44.00   66.7 39.2       ma:  34.41
sample ident: NIST 1003B SRM              31.11   27.6 20.2
sample   ID : Glass Spheres               22.00    7.4  4.4       parameters
lot     code:                             15.55    2.9  2.7      name     value
account  #:              printer: ON      11.00    0.2  0.2    smpl amt   0.000
run time:  30 sec.  run #: 100/ 3          7.78    0.0  0.0    disprsnt
sample preparation code:    0              5.50    0.0  0.0    disp amt   0.000
notes:                                     3.89    0.0  0.0    disp med
                                           2.75    0.0  0.0    dmed amt   0.000
                                           1.94    0.0  0.0    agitat'n
                                           1.38    0.0  0.0    agit tim       0
                                           0.97    0.0  0.0    circul'n
                                                               circ tim       0
                                                               param #1   0.000
                                                               param #2   0.000
    Esc-exit         F2-commands                               param #3   0.000
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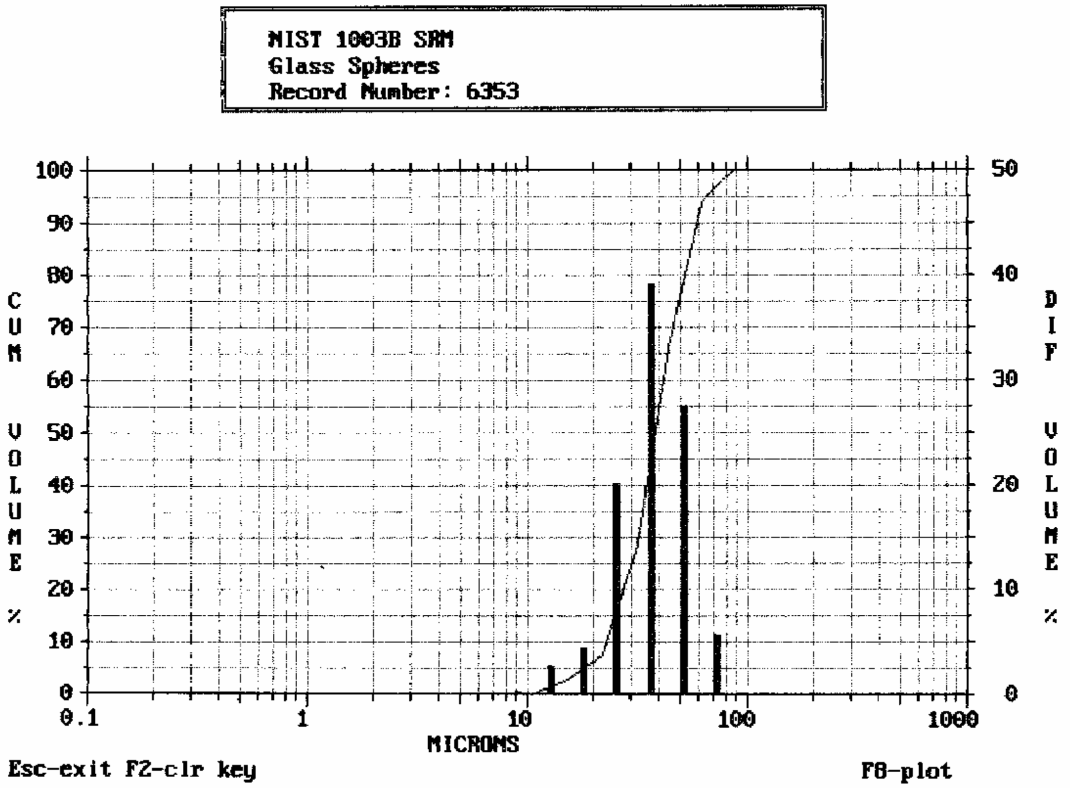
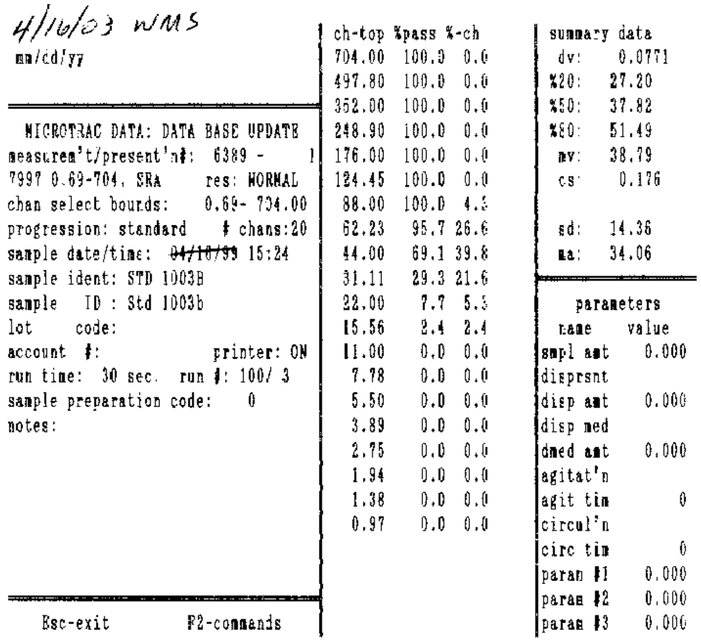


NIST 1003B SRM
Glass Spheres
Record Number: 6353



4/16/03 WMS

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                                           497.80  100.0  0.0     %20:   27.20
                                           352.00  100.0  0.0     %50:   37.82
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measurem't/present'n#:  6389 -      1      176.00  100.0  0.0      mv:   38.79
7997 0.69-704, SRA      res: NORMAL        124.45  100.0  0.0      cs     0.176
chan select bounds:     0.69- 704.00        88.00  100.0  4.3
progression: standard     # chans:20        62.23   95.7 26.6      sd:   14.36
sample date/time:  ~~04/10/99~~ 15:24       44.00   69.1 39.8      ma:   34.06
sample ident: STD 1003B                     31.11   29.3 21.6
sample   ID : Std 1003b                     22.00    7.7  5.3       parameters
lot     code:                               15.56    2.4  2.4      name    value
account  #:                printer: ON      11.00    0.0  0.0     smpl amt    0.000
run time:  30 sec.  run #: 100/ 3            7.78    0.0  0.0     disprsnt
sample preparation code:    0                5.50    0.0  0.0     disp amt    0.000
notes:                                       3.89    0.0  0.0     disp med
                                             2.75    0.0  0.0     dmed amt    0.000
                                             1.94    0.0  0.0     agitat'n
                                             1.38    0.0  0.0     agit tim        0
                                             0.97    0.0  0.0     circul'n
                                                                  circ tim        0
                                                                  param #1    0.000
                                                                  param #2    0.000
     Esc-exit         F2-commands                                 param #3    0.000
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STD 1003B
Std 1003b
Record Number: 6389