Oxide Milling and Blending

Using a

Resodyn® LabRAM Acoustic Mixer

Final Report

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Executive Summary

The purpose of this study is to investigate acoustic mixing as an alternative technology for blending, mixing and size reduction for the Pit Disassembly Conversion (PDC) Project. This study also investigated the potential application of acoustic mixing in the HB-Line process, where it would be used for large (3 kg) payload blending. Based on these potential applications, tests were done to assess the ability of the acoustic mixer to mix small and large payloads and to size reduce plutonium oxide surrogate materials both autogenously and with milling media. Brown and white aluminum oxides were the surrogates used for the blending studies and cerium oxide also known as cerium dioxide was the surrogate used for the size reduction studies.

There were four components to the small payload blending studies:

- Blending: The LabRAM acoustic mixer was able to produce mix indexes in excess of 0.95 in 5 seconds when blending 83 grams of <200 micron white and brown aluminum oxide. Container geometry (cylindrical versus annular) had no significant effect on blending.
- Autogenous Particle Size Reduction: Autogenous size reduction studies using 200-500 micron cerium oxide indicated that only a small degree of particle size reduction can be achieved autogenously, and that the majority of the reduction occurs in the first 10 minutes of blending. Studies using double roll mill processed cerium oxide revealed no significant particle size reduction, but an analysis of USB and EM images showed a significant reduction in the small amount of "pancake-shaped" particles.
- Milling Media Induced Particle Size Reduction: Studies using 200-500 micron cerium oxide demonstrated that the acoustic mixer is capable of rapid particle size reduction. Under the right conditions (50 grams of the cerium oxide at a Feed/Media ratio of 64/36 milled for 2 minutes at 84 g's acceleration), a 50 gram sample was milled to 99.3% <212 microns in only 2 minutes. Size reduction studies using milling media also showed that a sample of 200-500 micron cerium oxide milled under optimum conditions to the target value of 99% <212 microns, generated 32-34% material < 38 microns and an estimated 7-8% material < 5 microns. These optimum results could be achieved under a variety of test conditions, but milling with cylindrical media at 60 g's produced consistently favorable results with respect to minimizing the production of fines.
- Milling Media Density Studies: Increasing milling media density increases the degree or rate of particle size reduction. Decreasing the number of milling media particles decreases the degree or rate of particle size reduction.

Large payload blending studies using the LabRAM acoustic mixer demonstrated that the mixer is capable of blending payloads in excess of 3kg. For a 3kg payload of 90-grit white and brown aluminum oxide, the LabRAM was able to achieve a mix index of 0.99 after 10 minutes of blending.

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1.0 Introduction

The Pit Disassembly and Conversion (PDC) process includes milling, mixing, and blending of plutonium oxide, and mixing of uranium oxide[1]. These processes occur in three glovebox systems: Plutonium (Pu) Oxide Product Handling, S&P Stabilization, and HEU Oxide Product Handling. The current design for the Pu Oxide Handling System consists of the following main components: bottle inverter, oxide mill feeder, double roll mill, conical blender, discharge valve, sampler, and a loss-in-weigh feeder. The purpose of the double roll mill is to reduce the size of plutonium oxide to less than 200 microns and the purpose of the conical blender is to blend the oxide.

The purpose of this study was to investigate acoustic mixing as an alternative technology for blending to be potentially applied to three elements of the PDC Project involving mixing and blending, and possibly a technology that will also provide size reduction. Blending and successful size reduction of the plutonium oxide could be potentially applied to the Pu Oxide Product handling element of the PDC Project. This study also investigated acoustic mixing for potential use in the HEU Oxide Handling Systems as opposed to other equipment such as a Turbula Mixer. Acoustic mixing technology is mature and widely applied in numerous industries.

Recent double roll mill studies using cerium oxide as a surrogate for plutonium oxide showed that a small percentage of larger pancake-shaped particles were being produced by the roll mill which were larger than the desired 200 micron upper size limit. There were some indications that these pancakes were influenced by humidity, and that they may not be produced under the low humidity processing conditions present in the glovebox that contains the oxide handling system. A separate study was performed to investigate this.

This study also investigated the possibility of using acoustic mixing technology for combined blending and particle size reduction in one geometrically favorable designed vessel, replacing two stages of the process such as the conical blender and double roll mill. The effect of acoustic mixing on particle size reduction was studied with and without milling media.

Mixing and blending studies were performed using white and brown aluminum oxide (Al_2O_3) as a surrogate, whereas particle size reduction studies were performed using cerium oxide (CeO_2). Both materials were used in previous PDC Project conical blender and double roll mill tests at CETL. To match with these prior studies, all acoustic milling and blending studies were performed under ambient room conditions. Relative humidity and temperature were recorded for all studies. The basis for using brown and white aluminum oxide for the blending/mixing tests was to utilize existing techniques and procedures to determine the blending index of similar sized particles. It is unknown quantitatively how well cerium oxide represents plutonium oxide for grinding. Future tests, separate from this acoustic scoping test series, will be performed to determine this. However, since cerium oxide was used with the double roll mill tests it was used for this scope of work. These scoping tests were sequenced in a manner

[1] Refer to the definitions for mixing and blending in sections 4.2 and 4.7.

that allowed relative comparison of results with similar tests and surrogates run to date, for the PDC Project, on the CETL conical blender and double roll mill mockup, as well as for comparison between the small scale and large scale acoustic results achieved in this series. In this series CETL conducted the initial series of small scale scoping acoustic runs using a LabRAM Resodyn® unit, which will be followed by a series of larger scale runs performed at Resodyn®, using their RAM5 Resodyn® system, having a nominal 80 lb capacity. All tests were run with clean (non-radioactive) surrogate material in laboratory scale tests and ambient environmental conditions. Overall results of these scoping tests were compared amongst each other, in a summary report produced by CETL, together with comparisons with corresponding results achieved in prior CETL conical blender and roll mill tests run for the PDC Project. Included in the summary report is a description of relative effectiveness, anomalies, and recommendations for potential testing in subsequent acoustic evaluations based on findings and considerations for optimization. The report was issued upon completion of the first series of small scale tests described herein. Large scale testing was eliminated due to a technical change of direction.

2.0 Objectives

The objectives of this test series were to:

- Demonstrate blending of white and brown aluminum oxide blending surrogate in a cylindrical and an annular cylindrical vessel (to simulate a geometrically favorable design).
- Determine if pancake-shaped cerium oxide surrogate particles are broken up by the acoustic mixing process.
- Determine if size reduction of cerium oxide surrogate is possible using acoustic mixing, with and without milling media.
- Two items were added to the original test scope and are listed below. The first was added in order to explore an additional variable (milling media density) with respect to size reduction studies. The second item below was added to investigate another potential application of the LabRAM.
    - Determine the effect of milling media density on the particle size reduction of 200-500 micron cerium oxide.
    - Determine the capability of the LabRAM to mix payloads up to 3 kg in mass[2].

[2] Max loading studies are reported in a separate report. See reference 3.8.

3.0 References

3.1 Studies are planned using the LabRAM and the RAM5 mixers. See: (http://www.resodynmixers.com/products/labram/) and (http://www.resodynmixers.com/products/ram5/)

3.2 Washington Group Report. MOX Feed Specification Impact Study Report #1. Sept 13, 2000.

3.3 Washington Group Report. MOX Feed Specification Impact Study Report #2. Jan 14, 2001.

3.4 Interim Double Roll Mill Results – First Run of 2.5 kg Feed Through the Roll Mill, Clemson Engineering Technologies Laboratory, Rev.2, June 9, 2008.

3.5 Steve Hoeffner and Luke Beaver (CETL). Final Report: Evaluation of Two DBY 50 Laboratory Nautamixer Designs for the Plutonium Oxide Handling System. October 1, 2009.

3.6 Steve Hoeffner, Caleb Pittman, and Colton Cauthen (CETL). Summary of Sieving Studies. In process.

3.7 Steve Hoeffner (CETL) Greg Wunderlich (URS) Phil Rodwell (SRNS). Oxide Acoustic Milling and Mixing Scoping Test Plan. December 7, 2010.

3.8 Steve Hoeffner and Colton Cauthen (CETL). Acoustic Mixing Max Loading Final Test Report. March 22, 2012.

3.9 Steve Hoeffner , Colton Cauthen, and Taylor Yarborough(CETL). Acoustic Milling and Mixing: Pre-Operational Test Report. In Process.

3.10 Steve Hoeffner, Caleb Pittman. Comparing DRM-Processed Cerium Oxides Report.

4.0 Terms and Definitions

4.1 Acoustic Mixing – The application of a low-frequency high intensity acoustic energy to a charge creating a uniform shear field with micro-mixing zones throughout the system, resulting in efficient mixing.

4.2 Blending – Blending as related to the PDC project refers to the blending of heterogeneous material for the purpose of generating a mixing index of 0.5 or greater.

4.3 Charge – The Material (surrogate and milling media) placed in the acoustic mixer vessel.

4.4 F/M Ratio – The Feed-to-Media ratio (F/M ratio) refers to the weight-based ratio of surrogate material to milling media.

4.5 Feed – The surrogate material portion of the charge[3].

4.6 Milling Media – Also known as grinding or size reduction media. The milling media consists of spherical beads of aluminum oxide, or other hard and wear resistant material. The milling media will interact with the feed via the energy that is imparted to the charge thus size reducing the surrogate material.

4.7 Mixing – Mixing as related to the PDC project refers to the mixing of a single batch of material so that a sample can be taken from the batch that is representative of the entire population based on a qualitative level of confidence.

4.8 Mixing Index – The mixing index is a measure of degree of mixing. It ranges from 0 (no mixing) to 1 (completely blended) and is calculated by:

$$M = ({s_0}^2 - s^2)/({s_0}^2 - {s_r}^2)$$

where, M is the mixing index and s^2 is the variance. The subscripts o and r refer to the initial and random values of s^2, whereas s^2 refers to the variance of replicate samples. See Appendix A for additional details.

4.9 Particle Size Distribution – "In sieve analysis, the percentages, by mass or number, of all fractions into which various sizes of particles are classified" [ASTM E1638-94]. This shall be recorded in grams retained on each aperture size and shall be plotted as the cumulative percent retained on each aperture size. See Reference 3.6 for additional details.

4.10 Sieve Efficiency – Sieve efficiency refers to the accuracy of sieving with respect to the separation of material based on particle diameter. A sieve machine/process with a high sieve efficiency is one which retains only particles with a diameter greater than the size of the openings in a given sieve screen.

[3] See Appendix H for ordering information on feed material.

4.11 Surrogate – A surrogate material is ideally one which has physical and mechanical properties (e.g., grinding, crushing, and density) that are similar to oxide obtained from the DMO process at LANL. However, because no quantitative data was available, surrogates were selected by MOX/URS based on qualitative knowledge, which was available at the time. The surrogate used in this experiment is cerium oxide, which is sometimes referred to as cerium dioxide (CeO_2).

5.0 Test Scope

Prior to any testing with the LabRAM a detailed experimental test plan was written, which outlined all of the tests to be performed for the acoustic mixing studies (see Reference 3.7). The scope of tests performed deviate from this report in some cases due to insight from pre-operational testing and small-scale testing as well as changes in the technical direction of the project and application of the acoustic mixing technology. For details on pre-operational testing and results, see Reference 3.9. Table 1 shows the updated test scope. Figure 1 shows a schematic of the bench-scale acoustic mixer, the LabRAM, which was used for all acoustic mixing studies. The containers that were designed for blending and milling on the LabRAM are often referred to using an alphanumeric "Container Number." The letter is used to denote whether the container is annular or cylindrical (A=annular, C=cylindrical), whereas the number refers to the actual container number, as defined historically. See Appendix B for container details.

Table 1: Acoustic Mixing Test Scope

Set	Study to Perform	Variables					Estimated # Samples	Estimated # Tests	Container Type	Date of Completion
		Time, mins	Vessel	Aluminum Oxide Milling Media		Mixing Intensity				
				Loading	Size					
Bench-Scale Testing at Clemson Engineering Technologies Laboratory Using LabRAM Acoustic Mixer										
I	Pre-testing Studies Using <200 Micron White and Brown Aluminum Oxide									
P1	G's v Weight[1]	N/A	Cylinder	---	---	100%	---	---		July, 2011
P2		N/A	Cylinder	20, 50%	1/8", 1/4"	100%	---	---		July, 2011
P3	G's v H/D [2]	N/A	Cylinder	---	---	100%	---	---		July, 2011
P4	G's v Headspace[3]	N/A	Cylinder	---	---		---	---		July, 2011
II	Mixing/Blending Studies Using <200 Micron White and Brown Aluminum Oxide									
1	Section 6.2.1	0.083-0.5	Cylinder	---	---	100%	3-7	3-7	1	1/2/2012
2			Annular	---	---	100%	3-7	3-7	2	1/2/2012
III	Size Reduction Studies Using Roll Mill Processed Cerium Oxide									
4	Section 6.2.2	10	Cylinder	---	---	100%	1	1	Split 3	1/4/2012
5			Annular	---	---	100%	1	1	4	1/4/2012
IV	Size Reduction Studies Using 200-500 Micron Cerium Oxide									
6	Section 6.2.3.1	10,30	Cylinder	---	---	100%	1	2	Split 3	11/28/2011
7			Annular	---	---	100%	1	2	4	11/28/2011
8	Section 6.2.3.2	3.5-25	Cylinder	---	1/4" - Spherical	Target 90, 60, 30 G's	9	9	5	1/11/2012
9		4-20	Cylinder		1/4" - Satellite	Target 90, 60, 30 G's	3	3	5	1/11/2012
10		4-20	Cylinder		1/4" - Cylindrical	Target 90, 60, 30 G's	6	6	5	1/11/2012
11	Section 6.2.3.3	5-30	Cylinder	---	1/4" - Spherical	Target 90, 60, 30 G's	6	6	6	1/16/2012
10		5-30	Cylinder		1/4" - Satellite	Target 90, 60, 30 G's	3	3	6	1/16/2012
11		5-30	Cylinder		1/4" - Cylindrical	Target 90, 60, 30 G's	5	5	6	1/16/2012
8	Section 6.2.3.4	3.5-10	Annular	---	1/4" - Spherical	Target 90, 60 G's	5	5	7	2/1/2012
9		3.67-12.25	Annular		1/4" - Satellite	Target 90, 60 G's	5	5	7	2/1/2012
10		4-16	Annular		1/4" - Cylindrical	Target 90, 60 G's	5	5	7	2/1/2012
11	Section 6.2.3.5	1-9	Annular	---	1/4" - Spherical	Target 90, 60 G's	9	9	8	2/1/2012
10		2-12.5	Annular		1/4" - Satellite	Target 90, 60 G's	4	4	8	2/1/2012
11		2-15	Annular		1/4" - Cylindrical	Target 90, 60 G's	11	11	8	2/1/2012
12	Section 6.2.3.6	2-62	Cylinder	---	1/4" - Spherical	Target 90, 60 G's	12	12	7	2/20/2012
13		3-62		---	1/4" - Cylindrical	Target 90, 60 G's	9	9		2/20/2012

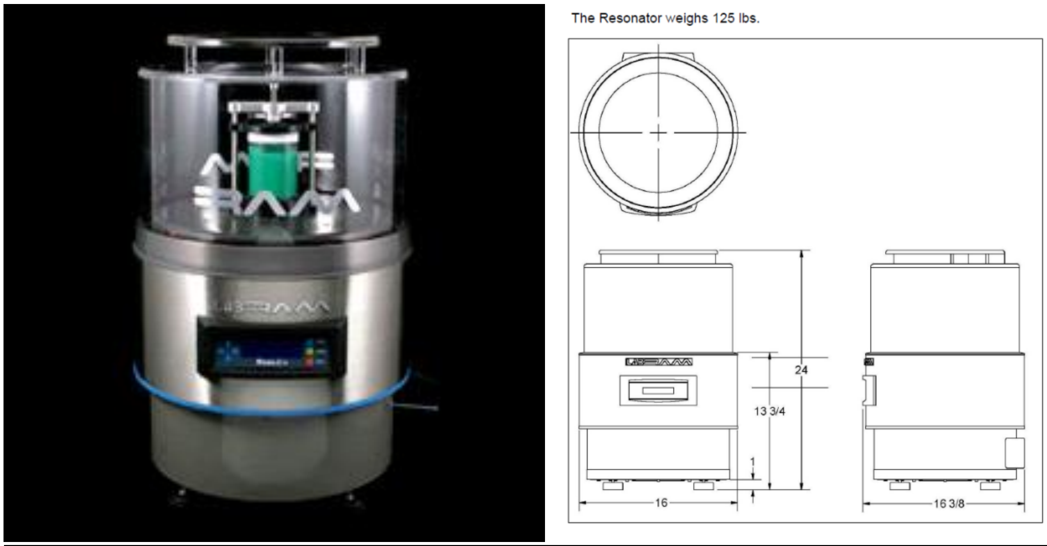


Figure 1: LabRAM Image and Dimensions

The LabRAM also comes with software that tracks intensity, frequency, and acceleration when the LabRAM is operating. This data can then be exported into an excel file and plotted. Figure **2** shows an example of what these three parameters look like when plotted in excel. For most tests performed using the LabRAM this data was tracked and saved and is available from CETL upon request.

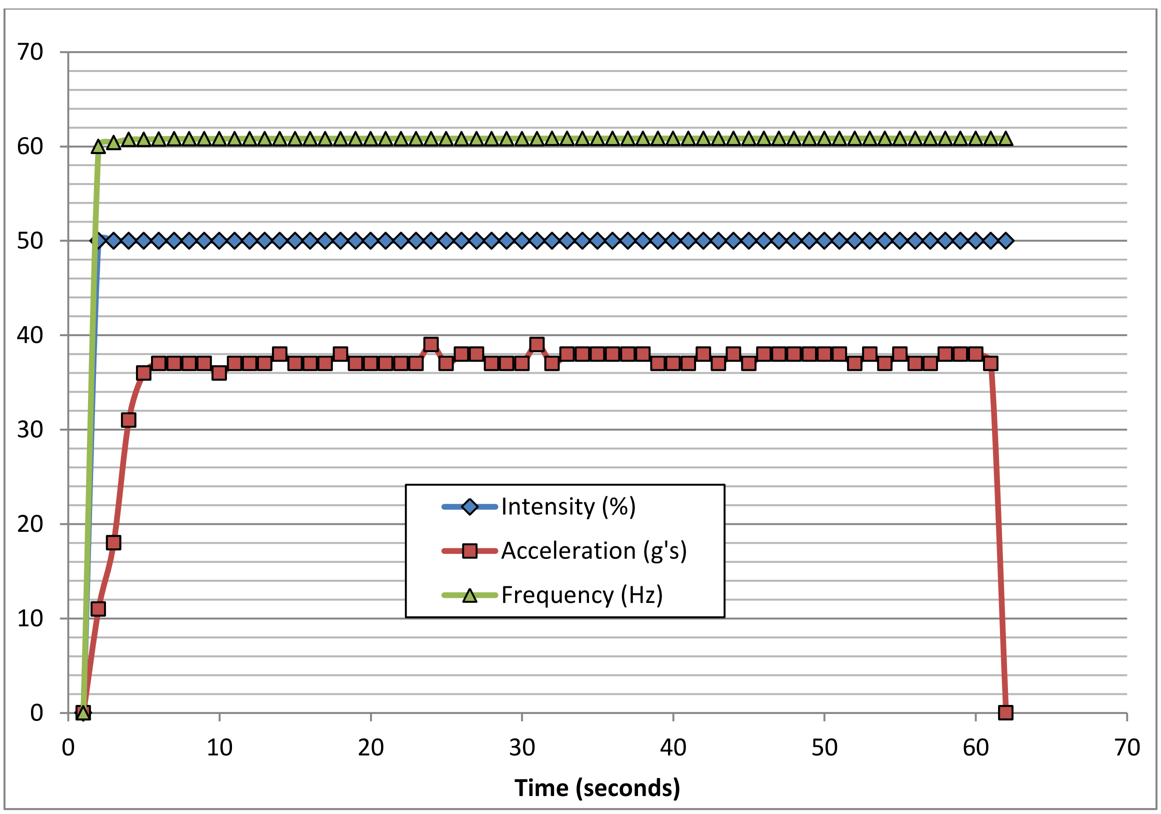


Figure 2: Example of LabRAM Data Plotted in Excel

5.1 Bench-Scale Acoustic Mixer Blending Studies Using <200 Micron White and Brown Aluminum Oxide

In order to determine the blending capabilities of the LabRAM, a set of blending studies were performed. These studies tested the LabRAM's ability to mix white and brown 90-grit aluminum oxide in two types of containers and at several different blending durations. The geometries of the containers used were cylindrical and annular cylindrical. A sample charge of 83.1 grams, composed of 50% white and 50% brown aluminum oxide, was mixed in each container type at 50% intensity for 15, 30, 60, and 120 seconds and at 100% intensity for 5, 15, and 30 seconds. A mix index was then calculated for each of the 14 samples. In order to achieve results comparable to those of the conical blender, a target mix index of 0.9 was established. However, the criterion for the PDC project is a mix index of 0.5.

Blending the Samples
Samples of as-received 90-grit brown and white aluminum oxide, each of mass 41.55 grams, were prepared. The sample of brown aluminum oxide was then poured into the container and the container was gently tapped to level the material in the container. The white aluminum oxide was then poured into the container and the container was tapped gently to level the material in the container. No blending of white and brown aluminum oxide was induced by this tapping, as seen in Figure 3 and Figure 4, which show the material in the containers after being gently tapped. The container was then placed on the LabRAM and mixed for the appropriate duration and intensity.

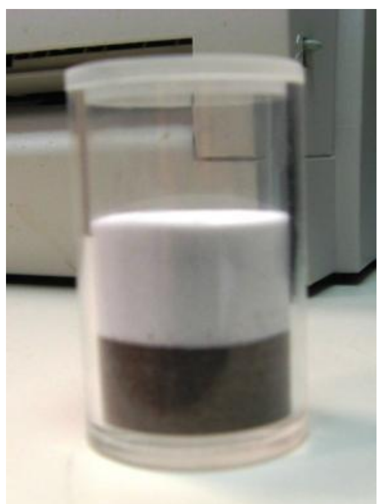


Figure 3: Container C1

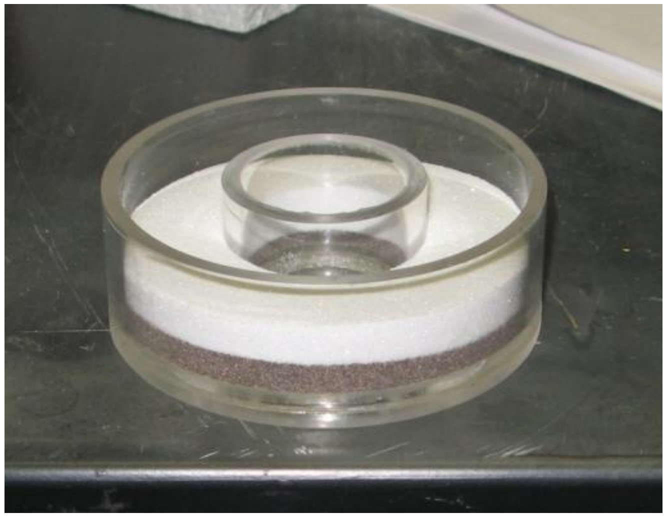


Figure 4: Container A2

Calculating the Mix Index

The mix index was calculated for each sample per the method used in calculating the mix index for the conical blender studies with the following exceptions: (1) the images were not modified using ArcSoft software prior to being processed with Matrox Inspector 8.0. (2) The low pass thresholds were selected manually on a picture-by-picture basis. The implementation of these minor changes were necessary due to the use of a new USB microscope that was purchased for the acoustic mixing study and also due to some poorly mixed samples from the max loading test (see Section 5.4) that had to be analyzed. The mix indexes for this (Section 5.1) study were originally calculated using pre-determined thresholds (eliminating the second difference listed above). However, during the max loading study it became apparent that the thresholds would need to be manually adjusted to accurately analyze both well and poorly mixed samples that were created during that study. Mix indexes for the max loading study were, therefore, calculated by manually adjusting the thresholds and the mix indexes for this Section 5.1 study were then re-calculated (in order to be consistent) by manually adjusting the thresholds. It should be noted that there was very little difference in the mix indexes calculated by the two different methods for the samples from this Section 5.1 study. For additional information on the calculation of the mix index for the conical blender study see Reference 3.5. For a detailed procedure on determining the mix index using Matrox Inspector 8.0, see Appendix A.

Measuring the Surrogate Temperature

In order to determine the change in temperature as a result of blending, the temperature of the aluminum oxide was measured immediately before and after blending. The temperature was measured by inserting a digital thermometer into the aluminum oxide and recording the thermometer's reading once a static (or maximum) reading was observed. The thermometer used was a Fisher Scientific digital traceable thermometer with a stainless steel stem and an accuracy of 0.1 °C from -50 to 150 °C[4].

[4] Similar to Fisher Scientific model 14-648-12

Some additional photos were taken during testing that are not included in this report because they were not considered necessary in understanding the testing, results, or conclusions of this study. However, these photos remain in CETL's possession and are available upon request.

5.2 Bench-Scale Acoustic Mixer Autogenous Size Reduction Studies Using Roll Mill Processed Cerium Oxide

In order to determine the autogenous size reduction abilities of the LabRAM, a set of tests were done using cerium oxide as a surrogate. The test series outlined below was performed using double roll mill processed cerium oxide, which was size reduced using the double roll mill technology. The double roll mill processed cerium oxide contained a small amount of oversized material which contained some "pancake-shaped" particles. The purpose of the tests outlined below was to determine if the LabRAM would break up these "pancake-shaped" particles such that the double roll mill processed material would meet the particle size criteria after being autogenously mixed in the LabRAM. Per the test plan, the cerium oxide was to be mixed on the LabRAM at 90 g's. However, for the cylindrical container (Container C3a) the LabRAM was not able to achieve 90 g's, or even 50 g's. The amount of feed to be used was then reduced from 198 grams to 145 grams and testing was performed under this condition for the cylindrical and annular cylindrical container. The LabRAM was able to achieve an average output (g-forces) of 70 g's for the cylindrical container. When using the annular cylindrical container, the intensity of the LabRAM was adjusted so that the average output was around 70 g's. USB and EM images were also taken of the mixed samples in order to determine if the "pancake-shaped" particles produced by the double roll mill would be broken up during blending.

For the tests using the cylindrical container with rings, a 145 gram sample of double roll mill processed cerium oxide was poured into the container and mixed in the LabRAM for 10 minutes at 100% intensity. The cylindrical container used for this study was designed to have three rings that would allow for the separation of material by layer after blending. This was done to test for stratification of material based on particle size (i.e. to determine the homogeneity of the LabRAM mixed samples as a function of depth). After blending, the material present in each of the layers was separated and placed in labeled containers. A particle size analysis was then performed on each of the layers.

For the test using the annular cylindrical container, a 145 gram sample of double roll mill processed cerium oxide was poured into the container and mixed in the LabRAM for 10 minutes at intensities ranging from 79-88% such that the acceleration (g's) was held fairly constant around 70 g's.

Some additional photos were taken during testing that are not included in this report because they were not considered necessary in understanding the testing, results, or conclusions of this study. However, these photos remain in CETL's possession and are available upon request.

5.3 Bench-Scale Acoustic Mixer Size Reduction Studies Using 200-500 Micron Cerium Oxide

In order to determine if acoustic mixing is a technology capable of producing results similar or superior to the feeder/spreader and double roll mill, a set of size reduction studies were performed using 200-500 micron cerium oxide. The first of these studies tested the ability of the LabRAM to size reduce as-received 200-500 micron Bayville cerium oxide autogenously (without the addition of any milling media.) The second set of studies tested the ability of the LabRAM to size reduce 200-500 micron cerium oxide using a variety of milling media.

5.3.1 Autogenous Size Reduction

Autogenous Size Reduction Using a Cylindrical Container (Container C3b)
The container used for this study was designed such that the inner shell is split into several rings. In order to prepare the container for use in the study, the rings were taped together using electrical tape. With the previously prepared container standing upright, the outer shell was then flexed and lowered over the inner container such that the gap in the shell encompassed the beginning and ending edge of the tape strips. A nominal 199 gram sample of as received 200-500 micron Bayville cerium oxide was then poured into the container and the lid was placed on the container. Two hose clamps were fastened around the container ensure the outer shell was secured tightly around the inner container. Figure 5 shows the final container setup, but without the presence of feed. The container was then secured in the LabRAM and mixed for the appropriate duration. Per the Acoustic Mixing Test Plan, the LabRAM was to be run at an intensity that yielded 90 g's. However, for the given container and amount of feed, the LabRAM was not able to yield 90 g's even at 100% intensity. The LabRAM was therefore run at 100% intensity, which resulted in g values ranging from 71-77 g's. Once mixed, the container was removed from the LabRAM and the hose clamps were removed. The container was placed in a weigh boat and the outer shell was removed by flexing it and sliding it upward. The top strip of tape was then carefully removed (without disturbing the container). Once the top strip of tape was removed, the top ring was lifted upward, allowing the material to spill out into the weigh boat. Using a straight edge, the surplus material (material extending above the top surface of the existing ring) was scraped off into the weigh boat until the material in the container was level with the top surface of the container. The container was then carefully transferred into a second weigh boat and the contents of the first weigh boat were emptied into a labeled container. The procedure for removing the tape, lifting the ring, and scraping and saving the excess material was then repeated for the remaining layers. The material from each layer was then sieved individually on the Ro-Tap for 40 minutes and a particle size analysis was performed (See Reference 3.6 for sieve analysis procedure).

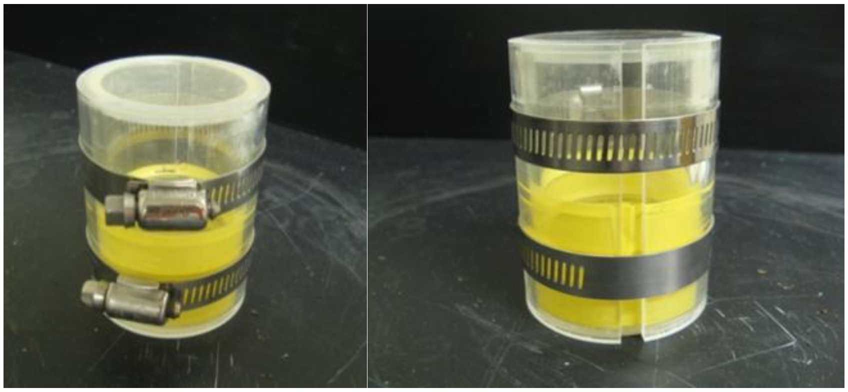


Figure 5: Container C3b Set-up

Autogenous Size Reduction Using an Annular Cylindrical Container (Container A4)

The study using the annular cylindrical container (container number A4) required no container preparation. A nominal 198 gram sample of as received 200-500 micron Bayville cerium oxide was poured into the container and the lid was placed on the container. The container was then secured in the LabRAM and mixed for the appropriate duration at 100% intensity, which yielded an average output of 77 g's. Once mixed, the sample was split into two 97 gram samples. One of the 97 gram samples was then sieved on the Ro-Tap for 40 minutes and a particle size analysis was performed.

5.3.2 Size Reduction Using Milling Media

A series of tests were performed to determine the optimum test conditions for milling 200-500 micron cerium oxide such that 99%[5] of the resulting material is less than 212 microns while producing as little material with a particle size less than 38 microns as possible. The variables explored in these tests were milling media geometry, LabRAM g's, F/M ratio, container height, container geometry, headspace, and milling duration. Note: new feed and milling media were used for every test. Figure 6 shows the following containers used throughout the experiments.

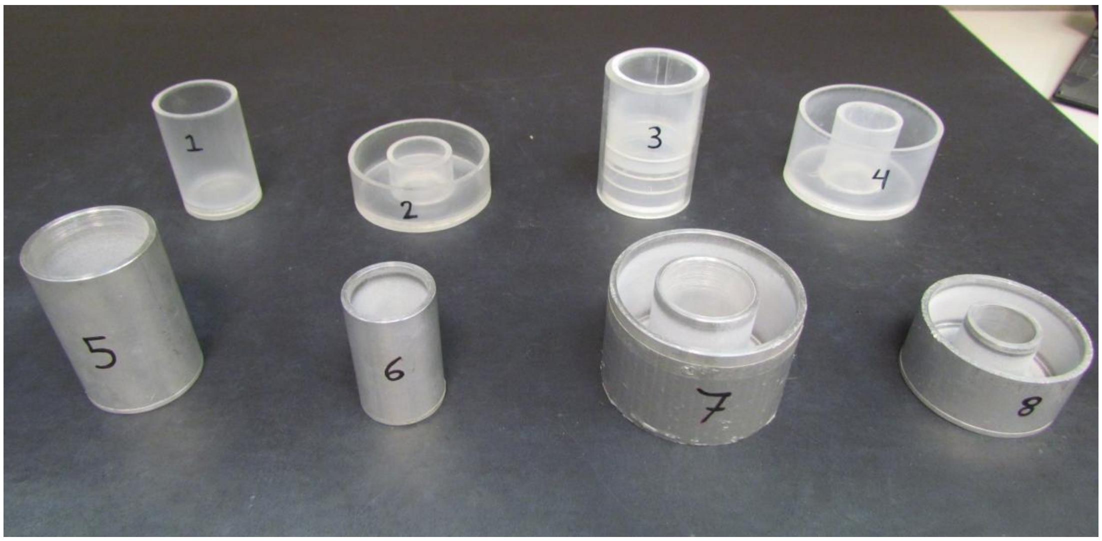


Figure 6: Container types (C1, A2, C3, A4, C5, C6, A7, and A8)

The objective of each test was to determine how much material below 38 microns would be produced when the sample was milled to 99% below 212 microns—leaving 1% greater than 212 microns. This was done using a trial-and-error method in which historical data was used in an attempt to predict the milling duration at which 1% greater than 212 microns would occur. In most cases several data points near 1%>212 microns were obtained and interpolation or extrapolation was used to create a data point at exactly 1%>212 microns. A total of 94 unique tests were performed in order to obtain data points at 1% greater than 212 microns for each of the desired testing conditions. Figure 7, Figure 8, and Figure 9 show a test chart for all of the desired testing conditions. Originally, each milling media type was to be used at 30, 60, and 90 g's; each acceleration value (g's) was to be run using a F/M ratio of 36/64 and 64/36; and each F/M ratio was to be used in a cylindrical and annular cylindrical container. However, as testing proved some conditions to be highly unfavorable, those testing conditions were removed from the test plan. Test conditions that were removed include all testing at 30 g's, and a variety of tests using satellite milling media. The satellite milling media tests were removed due to the fact that satellite and

[5] The actual performance criteria is 100% <200µ. However, the closest sieve that is commonly available is 212 µ, and a lenience of 1.0% was allowed for these studies. (e.g., if a sample had at least 99% of the material passing the 212µ sieve (less than 1% > 212µ), it was considered acceptable).

spherical milling media were producing similar results, making it unnecessary to test both spherical and satellite milling media under every condition. It should be noted that the target headspace for these tests was between 40 and 60 percent. However, as seen in Figure 7 and Figure 8, some tests were performed using a headspace of 85 percent. The purpose of these tests was to determine the effect[6] of varying headspace/container height for a given F/M ratio. The results obtained also allowed for the determination of the effect of varying the F/M ratio for a given container. This test series utilized container A7, (previously used for the F/M=36/64 study) at a F/M ratio of 64/36, and the same quantities of feed and media used in a previous study which utilized container A8. See Appendix B for container drawings.

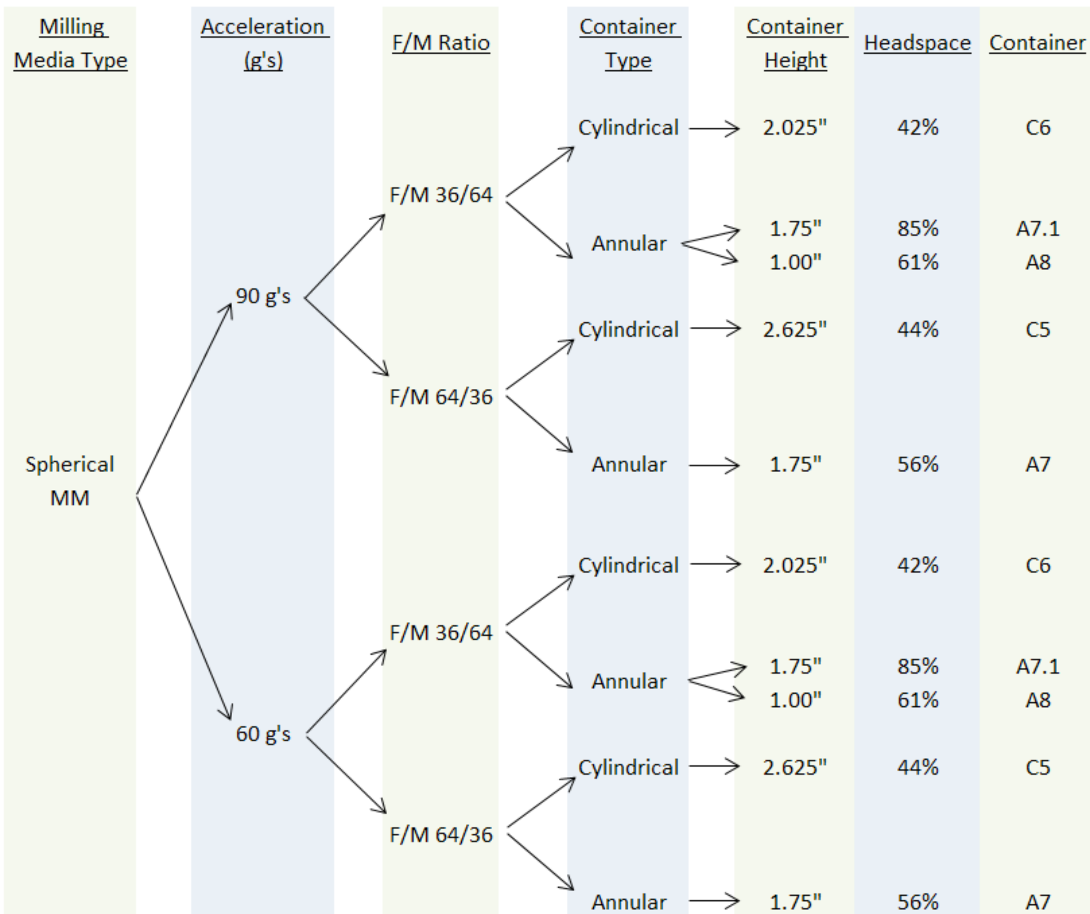


Figure 7: Spherical Milling Media Test Condition Chart

[6] Effect on the particle size reduction of cerium oxide such that 100%[6] of the milled material is milled to less than 212 microns and as few fines (material <38 microns) are produced as possible.

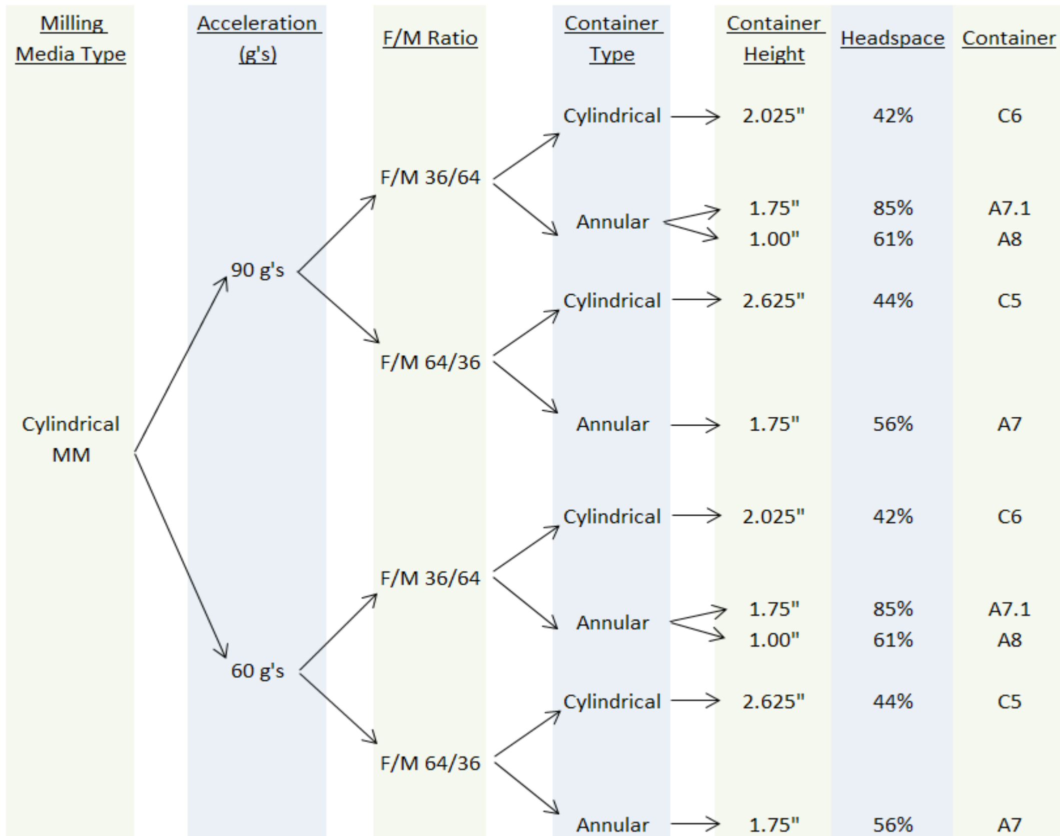


Figure 8: Cylindrical Milling Media Test Condition Chart

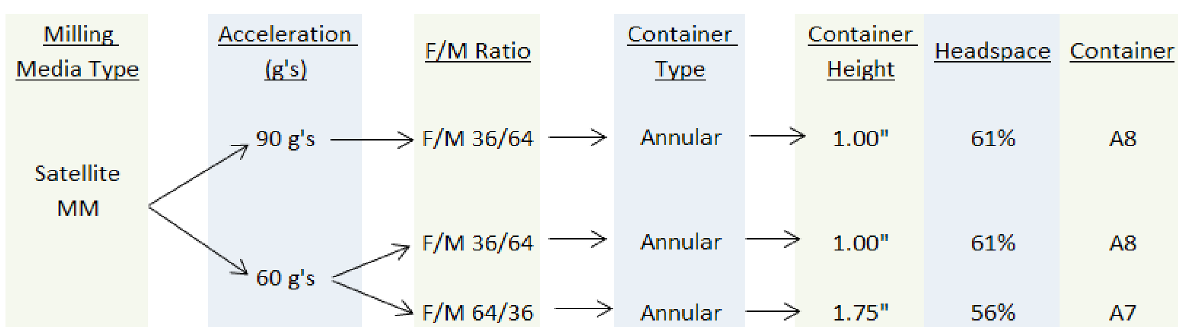


Figure 9: Satellite Milling Media Test Condition Chart

For each test, the designated amounts of cerium oxide and milling media were placed in the appropriate container and milled on the LabRAM for the pre-determined milling duration. The LabRAM software was used to record the LabRAM intensity, frequency, and acceleration for each test. Once milling was complete, the container was removed from the LabRAM and the milling media and cerium oxide were separated by pouring the mixture into a 4 inch, 2000 micron sieve then shaking the sieve rigorously (by hand) over a weigh boat until all of the cerium oxide had sifted through the sieve screen. The cerium oxide was then sieved on the Ro-Tap for 40 minutes and the fractions retained on each sieve were weighed. The pan material was kept separate for laser particle size analysis.

Some additional photos were taken during testing that are not included in this report because they were not considered necessary in understanding the testing, results, or conclusions of this study. However, these photos remain in CETL's possession and are available upon request.

5.3.3 Effect of Milling Media Density on Particle Size Reduction

The following test scope outlines a study that was conceived after the publication of the Oxide Acoustic Milling and Mixing Scoping Test Plan, and therefore was not included in the test plan. This study researched the effect of milling media density on particle size reduction of cerium oxide and was comprised of two mains sets of tests. The first set of tests was entirely weight based using set feed/milling media ratios (by weight) and keeping the feed and milling media weight constant. For the second set of tests, the feed weight was kept constant, but instead of keeping the milling media weight constant, the number of milling media particles was kept constant. Table 2 shows each of the tests that were run, what type of milling media was used for each, how much feed and media were used, and other important information about the test conditions. While there are 8 tests outlined in this report and listed in Table 2, only 6 of them are unique. Therefore, only 6 tests were run, as the results from 2 of the tests were each used twice.

For each test, the LabRAM was run at 90 g's for 10 minutes. At the end of each test, the container was removed from the LabRAM and the feed and milling media were manually separated. The container, lid, and media were then tapped to remove any loose feed hold-up, and all of the feed was placed in a weigh boat. A sieve analysis was then performed to determine the degree of particle size reduction that occurred during each test.

Table 2: Milling Media Density Study Test Plan

Table 1A - Constant Mass Testing								
Test No.	Feed/Media Ratio (Weight)	Total Weight (g)	Feed Weight (g)	Media Weight (g)	Media Type	Head-space	Media Volume (cm^3)	No. of Media Particles
1	63/37	150	95	55	Al_2O_3	86%	16.2	143
2	63/37	150	95	55	YSZ	87%	9.2	64
3	37/63	150	55	95	Al_2O_3	87%	27.8	246
4	37/63	150	55	95	YSZ	89%	15.8	110

Table 1B - Constant Volume Testing								
Test No.	Feed/Media Ratio (Weight)	Total Weight (g)	Feed Weight (g)	Media Weight (g)	Media Type	Head-space	Media Volume (cm^3)	No. of Media Particles
1	63/37	150	95	55	Al_2O_3	86%	16.2	143
5	44/56	217	95	122	YSZ	86%	20.6	143
3	37/63	150	55	95	Al_2O_3	87%	27.8	246
6	21/79	265	55	210	YSZ	87%	35.4	246

Some additional photos were taken during testing that are not included in this report because they were not considered necessary in understanding the testing, results, or conclusions of this study. However, these photos remain in CETL's possession and are available upon request.

5.4 Acoustic Mixing Max Loading Study

The LabRAM has a variable mix capacity of 1 pound (454 grams) and a nominal volumetric capacity of 1 pint. The HB-Line[7] process of the H Canyon facility requires up to 3 kg of material to be mixed. In order to determine the ability of the LabRAM to operate outside of its rated capacity such that it could be used for blending in the HB-Line process, a set of tests were performed with the LabRAM acoustic mixer. For additional details on the scope of this study see Reference 3.8.

[7] The HB Line is located on top of H Canyon and is the only chemical processing facility of its kind in the DOE complex.

6.0 Test Results and Discussion

The results of tests performed at CETL are presented below. All data and results considered necessary to understand each study and the results thereof have been included. However, CETL does possess "raw data" generated from testing that is not included in this report, but are available upon request.

6.1 Bench-Scale Acoustic Mixer Blending Studies Using <200 Micron White and Brown Aluminum Oxide

In order to assess the LabRAM's blending capabilities, the mix index was calculated for the blending of brown and white aluminum oxide in both cylindrical and annular cylindrical containers. The payload for these studies was 83.1 grams, which was composed of 50% white and 50% brown aluminum oxide by weight. The blending intensity and duration were also varied to determine their effect on the mixing efficiency (mix index). It is expected that a mix intensity of 100% would be used in production, but testing at 50% intensity was also performed and is included in this report. Table 3 shows a summary of the mix index results for each 50% intensity test as well as the change in temperature of the feed immediately before and after blending. The ambient temperature was typically around 20° C. The temperature data in Table 3 shows a general increase in temperature with respect to blending time, as expected. While none of the temperature increases seen in this study were dramatic, the results do suggest that blending in an annular container (opposed to a cylindrical container) may reduce the degree of temperature increase during blending. Figure 10 is a graph of the mix index data from Table 3 (tests performed at 50% intensity). For all of the scenarios tested, the mix index was above the targeted value of 0.9. As shown in Figure 10, container geometry (cylindrical versus annular cylindrical) appears to have no effect on blending. While the results do vary between the tests using the different container geometries, this variance is likely within the scatter of the data and does not provide reasonable ground for inference. Figure 11 shows the results of the tests that were run at 100% intensity. As seen in Figure 11, even a 5-second blending duration is sufficient, with a mix index in excess of 0.98. Figure 10 and Figure 11 show little difference between a sample mixed for equal durations at 50% versus 100% intensity (comparing 15 and 30 second mix durations). However, because the material has been thoroughly mixed long before 30 seconds at both intensities, this comparison is not indicative of the effect of mix intensity on the resulting mix index of the material.

Table 3: Mix Indexes and Temperature Changes for 50% Intensity Tests

	Cylindrical Container		Annular Container	
	ΔT	**MI**	**ΔT**	**MI**
15 Second Mix	1.8°C	0.982	1.7°C	0.989
30 Second Mix	2.2°C	0.994	1.0°C	0.990
60 Second Mix	5.4°C	0.999	2.3°C	0.993
120 Second Mix	9.4°C	1.000	4.8°C	0.996

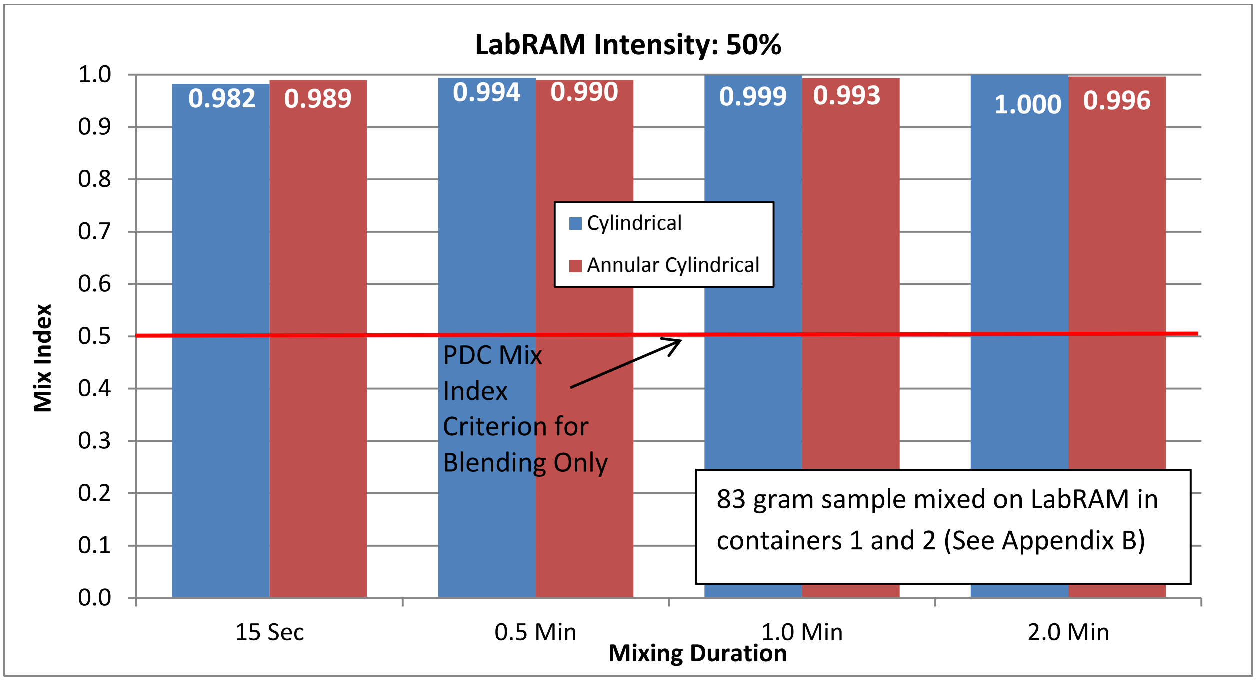


Figure 10: Mix Index for all 50% Intensity Tests

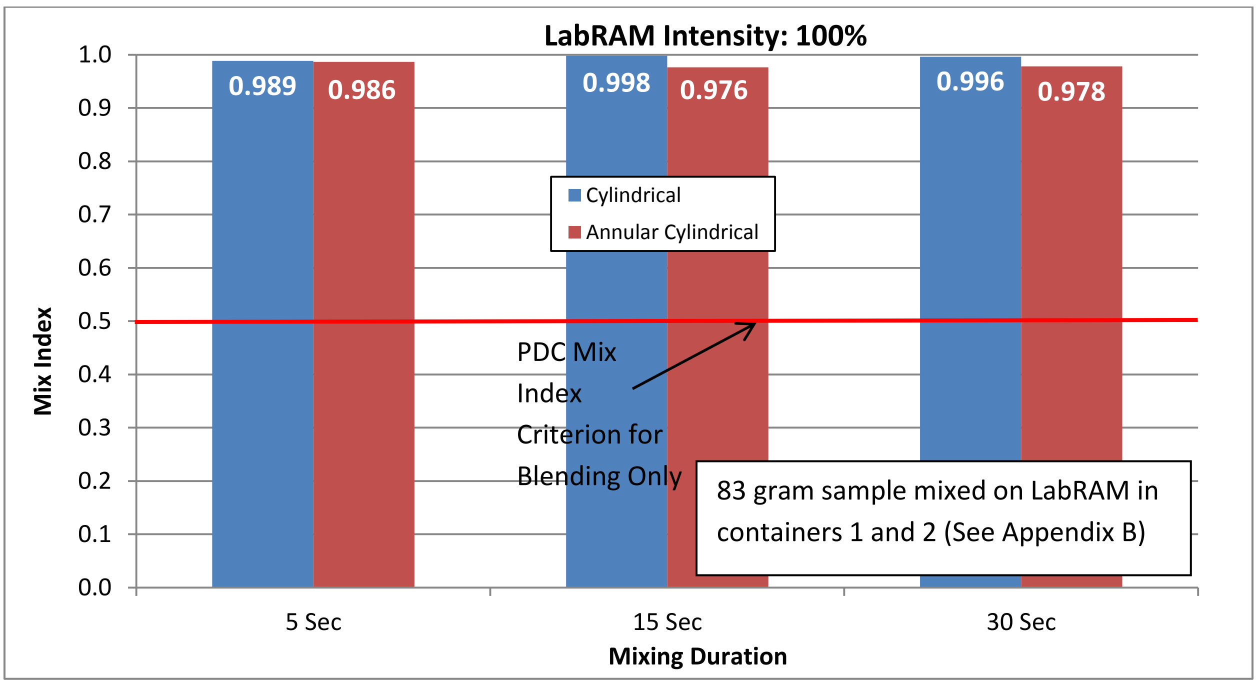


Figure 11: Mix Index for all 100% Intensity Tests

Table 4 shows the LabRAM output data for each of the tests. The values in the table represent the average acceleration (g's) exerted throughout the course of the test. For the tests that were video recorded, a hyperlink has also been inserted so that the video for the given test can be viewed by holding the ctrl key and clicking the appropriate cell in the table.

Table 4: LabRAM Output Data (Average Acceleration) for Each Test with Links to Videos

	50 % Intensity		**100% Intensity**	
	Cylindrical	**Annular**	**Cylindrical**	**Annular**
5 Second Mix	N/A	N/A	Unknown[8]	Unknown
15 Second Mix	36 g's	34 g's	73 g's	64 g's
30 Second Mix	37 g's	35 g's	75 g's	67 g's
60 Second Mix	37 g's	35 g's	N/A	N/A
120 Second Mix	38 g's	35 g's	N/A	N/A

6.2 Bench-Scale Acoustic Mixer Autogenous Size Reduction Studies Using Double Roll Mill Processed Cerium Oxide

A 145 gram sample of double roll mill processed cerium oxide was mixed on the LabRAM in a cylindrical container for 10 minutes at 100% intensity, which resulted in an average acceleration of around 70 g's. The container was designed such that the material could be separated into 3 layers after blending. Once separated, each of the layers was sieved to develop particle size distribution curves. Figure 12 shows the results of the particle size analyses of the three layers compared to the original sample. There appears to be slight particle size reduction, uniformly distributed across the three layers. A second test was done using an annular cylindrical container. The sample size and acceleration were kept constant between the two tests. Figure 13 shows a comparison between samples mixed in the cylindrical and annular cylindrical vessels. The results suggest that container geometry (annular versus cylindrical) has no effect on autogenous particle size reduction of double roll mill processed cerium oxide.

[8] Since test was short, values were not recorded. But the values should be in the range of 73-75 G's for the cylindrical vessel and 64-67 G's for the annular vessel.

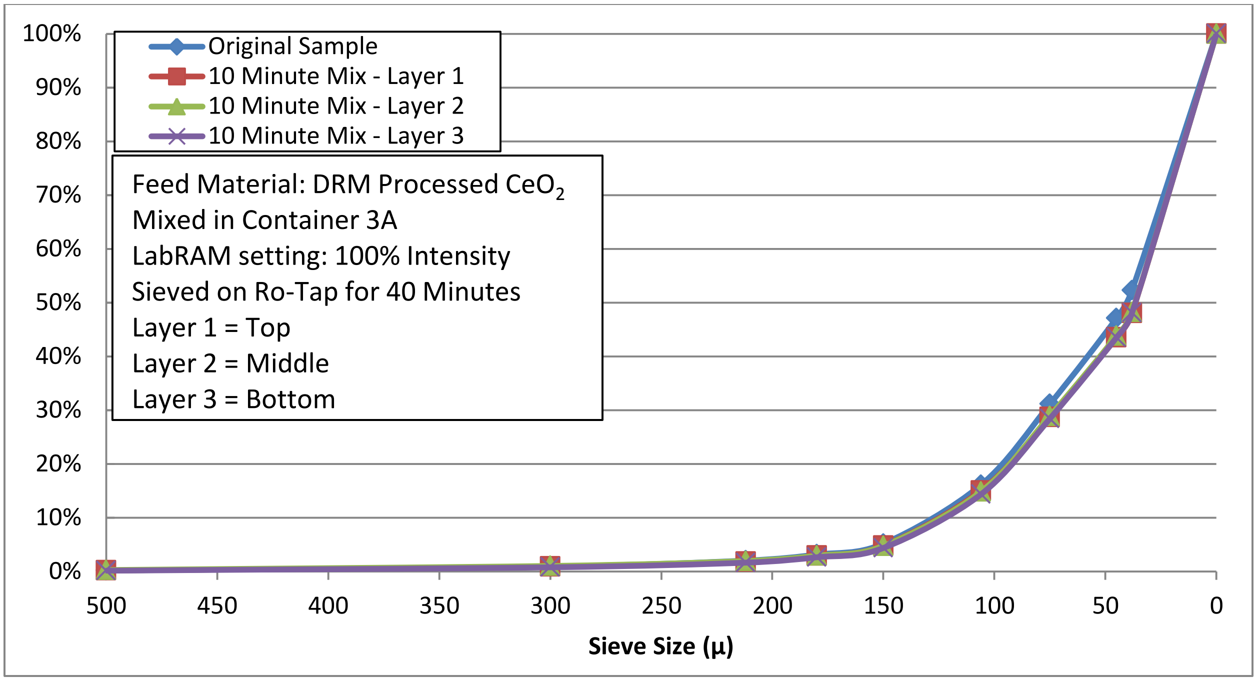


Figure 12: PSA of Sample Mixed for 10 Minutes - Per Layer

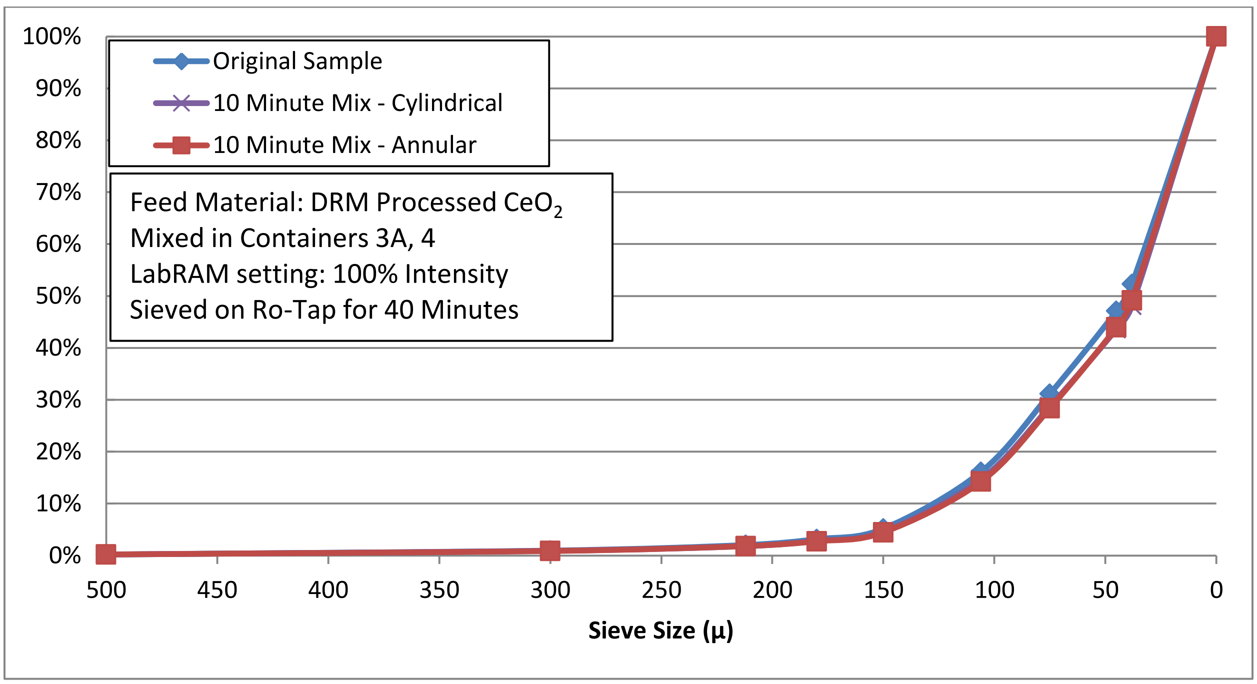
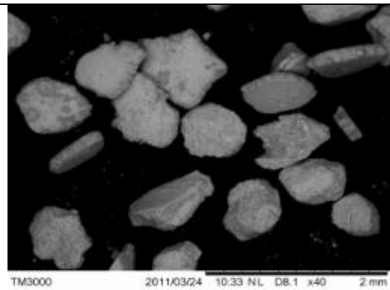
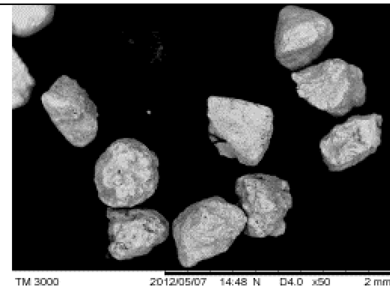
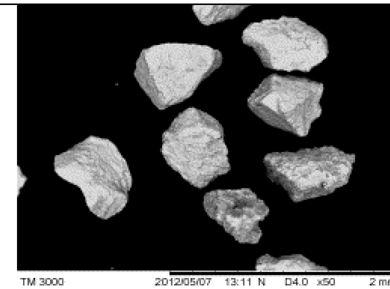
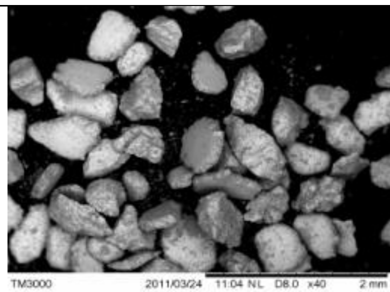
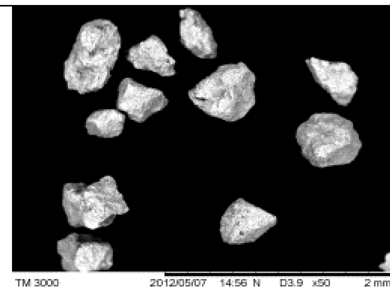
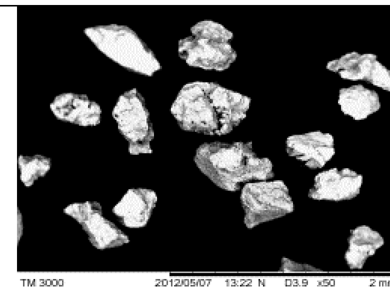
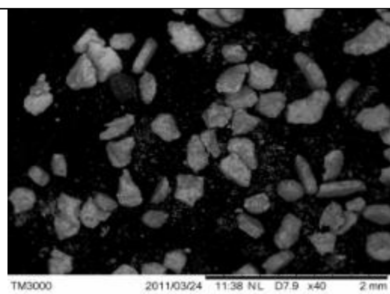
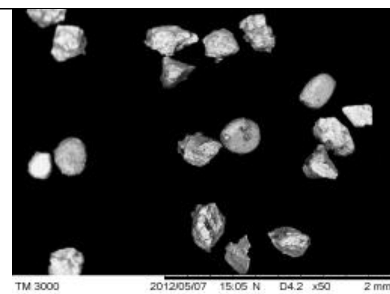
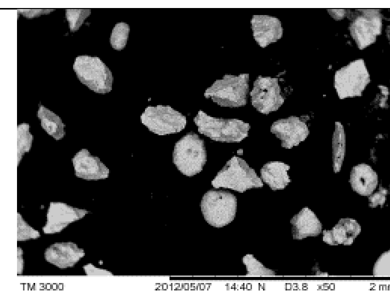


Figure 13: PSA of Samples Mixed for 10 Minutes – Annular versus Cylindrical

A comparison of USB and EM images taken before and after blending the double roll mill processed cerium oxide in the LabRAM revealed a reduction of "pancake-shaped" particles. Two samples were created during testing, one from blending in a cylindrical container and one from an annular cylindrical container. Three subsamples were created from the sample mixed in the cylindrical container (one from each of the layers described above). Several before and after EM images are shown in Table 5.

Additional 'before and after' USB and EM images can be found in Appendix C. It should be noted that the pancake-shaped particles represent a very small fraction of the sample, which explains why no significant particle size reduction is observed even after the pancake-shaped agglomerates are broken up. Additional EM and USB images were taken during testing that are not included in Appendix C because they were not considered necessary in understanding the testing, results, or conclusions of this study. However, these photos remain in CETL's possession and are available upon request.

Table 5: EM Images of DRM CeO_2 Before and After Blending

40x	Before Blending	After Blending (10 min) [Annular Container]	After Blending (10 min) [Cylindrical Container]
>500 micron			
>300 micron			
>212 micron			

6.3 Bench-Scale Acoustic Mixer Size Reduction Studies Using 200-500 Micron Cerium Oxide

6.3.1 Autogenous Size Reduction

A 199 gram sample of as received 200-500 micron Bayville cerium oxide was poured into container C3b and mixed in the LabRAM for 10 minutes at 100% intensity. Per the methods outlined in the *Test Scope* section of this report, the four layers of cerium oxide were individually removed from the container and stored for particle size analysis. Some material (<0.5 grams) was also retained on the lid (see Figure 14). This material was placed in a separate container. A particle size analysis was then performed on each of the layers. Figure 15 shows the results of the particle size analyses of the four layers compared to a sample of the as received 200-500 micron Bayville cerium oxide. Figure 15 and Table 6 show the results of the particle size analyses of the 4 layers and suggests that the particle size distribution was uniform across layers 1-3 (the top 3 layers). Based on the particle size distribution curves in Figure 15, it appears that the bottom layer (layer 4) was composed of slightly finer material than the layers above (layers 1-3).

Figure 14: CeO_2 Retained on Container C3b Lid after Blending

Comparing the percent retained on each sieve in Table 6 shows that, compared to the top 3 layers, the bottom layer (layer 4) had 3% less material retained on the 300 sieve and about 1% less material retained on the 212 sieve. This would indicate that the bottom layer had less coarse (>212µ) material. Comparing the percent retained in the pan shows that the bottom layer had 2% more fine (<38µ) material than the top 3 layers (about 1.5 grams versus a nominal 0.5 grams present in each of the <38 micron fractions in the top 3 layers)[9]. About 1% of this difference could be due to the 0.5 grams of so that adheres to the top and bottom lids.

[9] Each layer contained approximately 53 grams of material with the exception of the top layer, which only contained 38 grams.

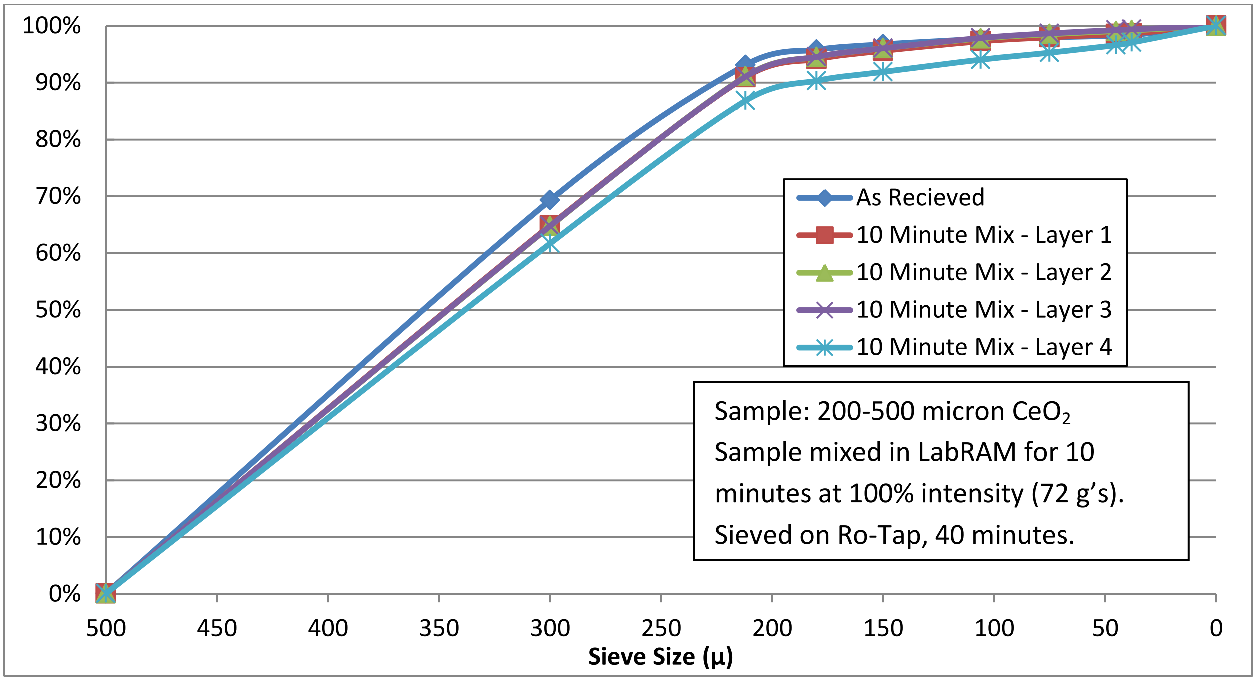


Figure 15: PSA of Sample Mixed for 10 Minutes – Per Layer

Table 6: Bayville CeO_2 After 10 Minutes Blending – PSA for Each Layer

Sieve	Layer 1	Layer 2	Layer 3	Layer 4
500	0.1%	0.1%	0.1%	0.1%
300	64.8%	64.7%	64.7%	61.6%
212	26.1%	26.4%	26.3%	25.1%
180	3.2%	3.4%	3.5%	3.5%
150	1.5%	1.5%	1.5%	1.6%
106	1.7%	1.7%	1.8%	2.2%
75	0.7%	0.7%	0.8%	1.2%
45	0.5%	0.5%	0.6%	1.3%
38	0.1%	0.1%	0.1%	0.5%
Pan	1.3%	0.8%	0.6%	2.9%
TOTAL	100%	100%	100%	100%

The 4 layers and the material retained on the lid were then combined and poured into container C3b for a second set of tests. The purpose of the second set of tests was to determine the effect of mix duration on autogenous particle size reduction and on the homogeneity of the mixed material. The material was mixed in the LabRAM for an additional 20 minutes at 100% intensity. Figure 16 shows the results, which show slightly more scatter between the top 3 layers (compared to Figure 15). Figure 16 also suggests a small degree of stratification between the upper 3 layers (layers 1-3) and the bottom layer (layer 4).

Comparing the percent retained on each sieve in Table 7 shows that, compared to the top 3 layers, the bottom layer (layer 4) had an average about 2% less material retained on the 300 sieve and about 1% less material retained on the 212 sieve. This would indicate that the bottom layer had less coarse

(>212µ) material. Comparing the percent retained in the pan shows that the bottom layer had approximately 1.3% more fine (<38µ) material than the top 3 layers (about 1.2 grams versus a nominal 0.3 grams present in each of the <38 micron fractions in the top 3 layers)[10]. About 1% of this difference could be due to the 0.5 grams or so that adheres to the top and bottom. Other than the slight difference in the amount retained on the 300 micron sieve, the results in Table 7 are very similar to those in Table 6—the top 3 layers have nearly identical distributions while the bottom layer contains less coarse (>212µ) material and more fine (<38µ) material.

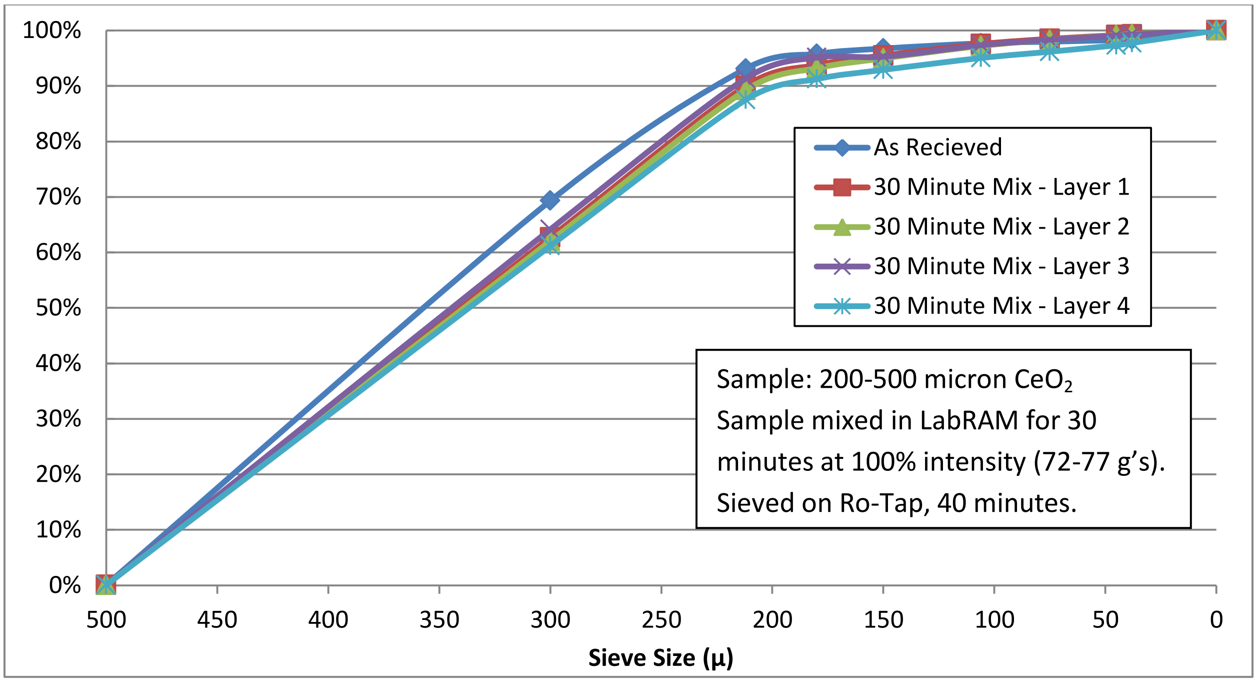


Figure 16: PSA of Sample Mixed for 30 Minutes – Per Layer

[10] Each layer contained approximately 53 grams of material with the exception of the top layer, which only contained 38 grams.

Table 7: Bayville CeO_2 After 30 Minutes Blending – PSA for Each Layer

Sieve	Layer 1	Layer 2	Layer 3	Layer 4
500	0.1%	0.1%	0.1%	0.1%
300	62.7%	62.0%	64.1%	61.1%
212	27.3%	27.2%	27.2%	26.3%
180	3.8%	3.9%	3.8%	3.7%
150	1.7%	1.8%	0.2%	1.7%
106	2.1%	2.2%	2.1%	2.1%
75	0.9%	1.1%	1.0%	1.1%
45	0.7%	0.8%	0.8%	1.2%
38	0.1%	0.2%	0.2%	0.4%
0	0.7%	0.7%	0.7%	2.3%
TOTAL	100%	100%	100%	100%

Figure 17 shows the particle size distribution for the sample mixed in the annular cylindrical container (container A4) for 10 and 30 minutes. The particle size distribution for a sample mixed in the cylindrical container (container C3b) is also included in Figure 17 for comparative purposes. The particle size reduction appears to follow the same trend for both container geometries, with very little size reduction occurring during the range of 10-30 minutes of blending time, indicating little or no particle size reduction occurs after the first 10 minutes. However, the sample mixed in the annular cylindrical container did experience slightly more particle size reduction than the sample mixed in the cylindrical container. This may be due to the slightly higher acceleration (g-forces) exerted during the annular container tests. Table 8 shows a summary of the g-forces exerted during each test.

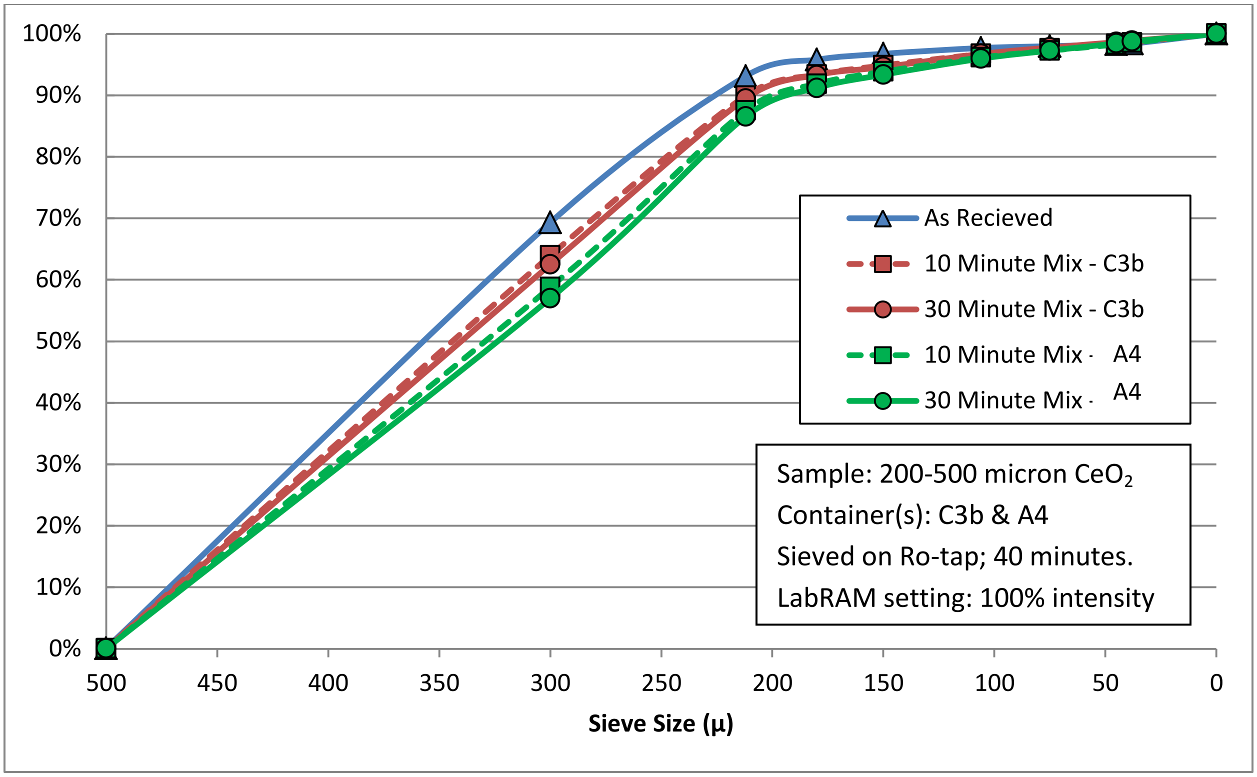


Figure 17: PSA per Container Geometry and Blending Duration

Table 8: LabRAM Output Data (Average Acceleration)

Cumulative Mix Duration	10 Minutes	30 Minutes
Cylindrical Container (C3b)	70 g's	73 g's
Annular Cylindrical Container (A4)	77 g's	79 g's

During the testing discussed above, temperature data was collected for the cerium oxide at different durations of blending. Table 9 and Table 10 summarize this data. The results indicate that cerium oxide is increasing in temperature very rapidly during blending. Figure 18 displays is a plot of the data from Table 9 that shows the post-blending temperature of the cerium oxide with respect to time during blending. Plotting a third order polynomial trendline yields an equation for the temperature of cerium oxide as a function of blending time (See Figure 18). Note that this equation is only valid for a range of 0 to 20 minutes and may vary based on container dimensions and LabRAM output.

Table 9: Change in Surrogate Temperature during Blending in Cylindrical Container

Cumulative Mix Duration	5 Minutes	10 Minutes	20 Minutes
Pre-Blending Temperature	20.8 °C	21.0 °C	21.0 °C
Post-Blending Temperature	80.1 °C	114.4 °C	143.5 °C
Change in Temperature (ΔT)	59.3 °C	93.4 °C	122.5 °C

Table 10: Change in Surrogate Temperature during Blending in Annular Cylindrical Container

Cumulative Mix Duration	**5 Minutes**	**10 Minutes**	**20 Minutes**
Pre-Blending Temperature	22.0 °C	No Data	26.2 °C
Post-Blending Temperature	61.0 °C	No Data	98.0 °C
Change in Temperature (ΔT)	39.0 °C	No Data	71.8 °C

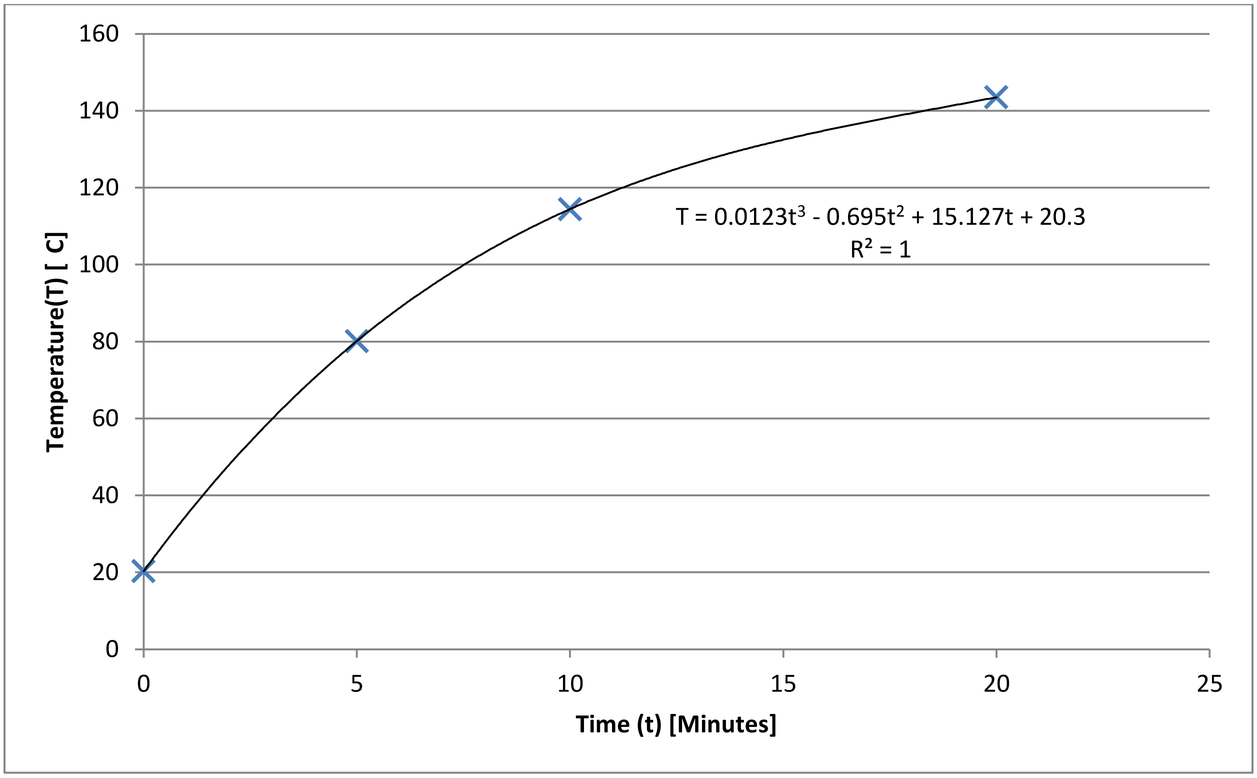


Figure 18: Temperature of Cerium Oxide vs. Blending Time for Cylindrical Container

6.3.2 Size Reduction Using Milling Media

There were four containers used for this test series (See Appendix B). Each container was designed for a specific loading, F/M ratio, and resulting headspace. The first subset of tests was done using two cylindrical containers (container C5 and C6) designed for F/M ratios of 36/64 and 64/36, respectively. The cerium oxide was milled in each of these containers at 60 and 90 g's[11] and using each of the three milling media types. The second subset of tests was done using two annular cylindrical containers (container A7 and A8) designed for F/M ratios of 36/64 and 64/36, respectively. The cerium oxide was milled in each of these containers at 60 and 90 g's and using each of the three milling media types. Appendix D contains a table with all of the unique test conditions that were explored in this study. Figure 19 – Figure 26 show the resulting particle size distributions of samples milled under the conditions indicated. Each figure can be used to determine the effect of milling media type on the rate of particle size reduction for the given set of test conditions. Figure 24 and Figure 26 do not contain a

[11] Some tests were done at 30 g's, but due to the lengthy milling duration required and the large amount of material <38 microns that was produced during milling, the 30 g's tests were removed from the scope of testing.

curve for cylindrical milling media because cylindrical milling media was not used at the same milling duration as the satellite and spherical milling media for these two tests. For each of the test conditions shown, satellite milling media appears to induce particle size reduction the fastest. Cylindrical milling media typically appears to have the slowest rate of particle size reduction, with the exception of Figure 22.

A possible explanation for the difference in rate of particle size reduction induced by each milling media type is the number of milling media particles that were present for each test. Because each of the milling media types have a unique mass-per-particle, a different number of particles had to be used for each milling media type in order to keep the total milling media weight constant. Table 11 shows the approximate number of milling media particles that were used for each test. In light of Figure 19 – Figure 26, Table 11 suggests that the more milling media particles that are present for a given F/M ratio and total payload, the higher the rate of particle size reduction.

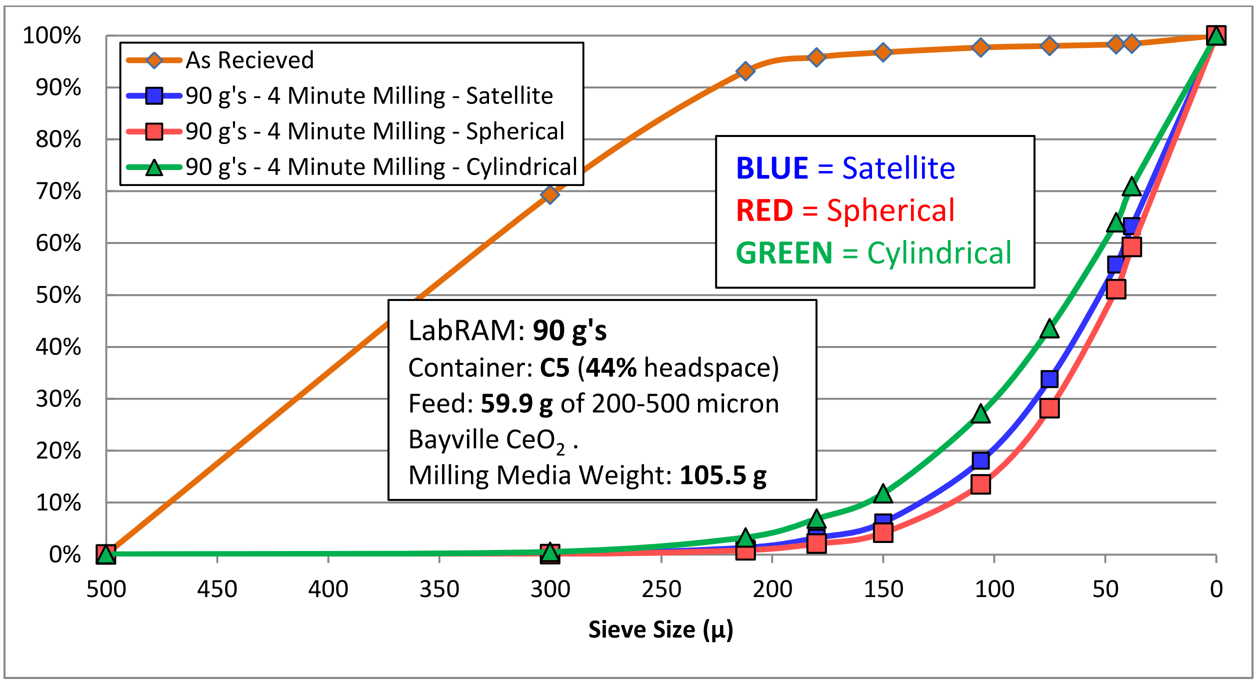


Figure 19: PSA Results for F/M Ratio of 36/64 (Container C5)

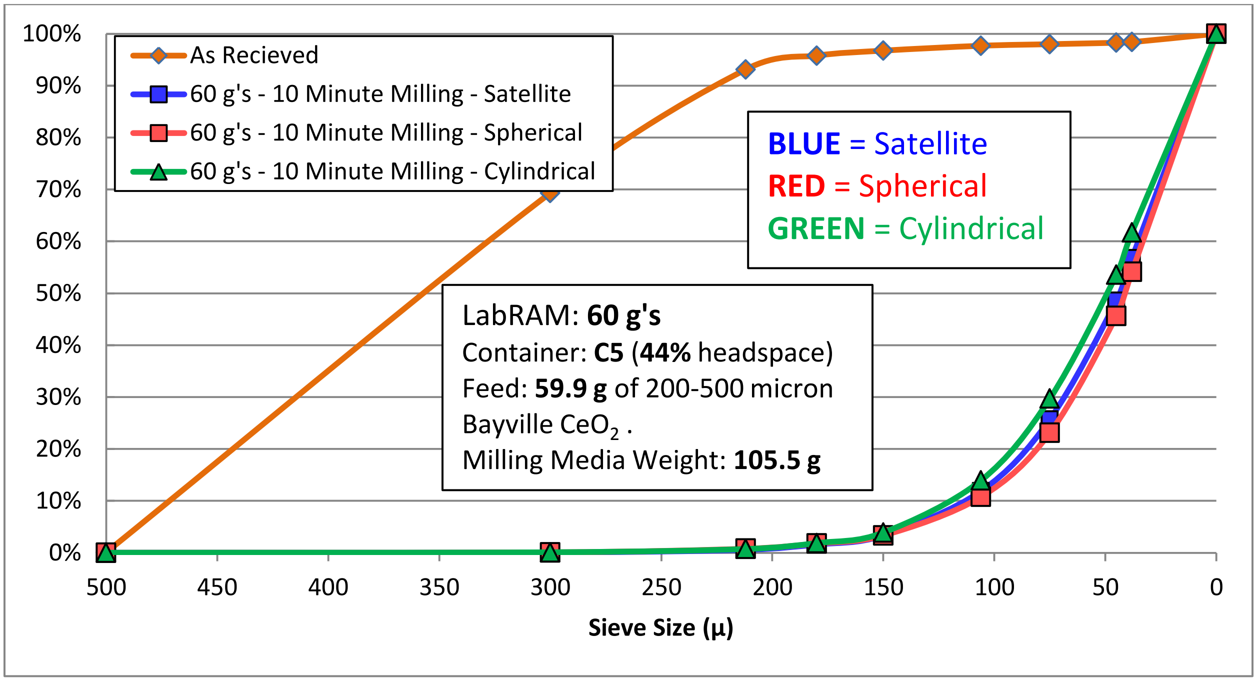


Figure 20: PSA Results for F/M Ratio of 36/64 (Container C5)

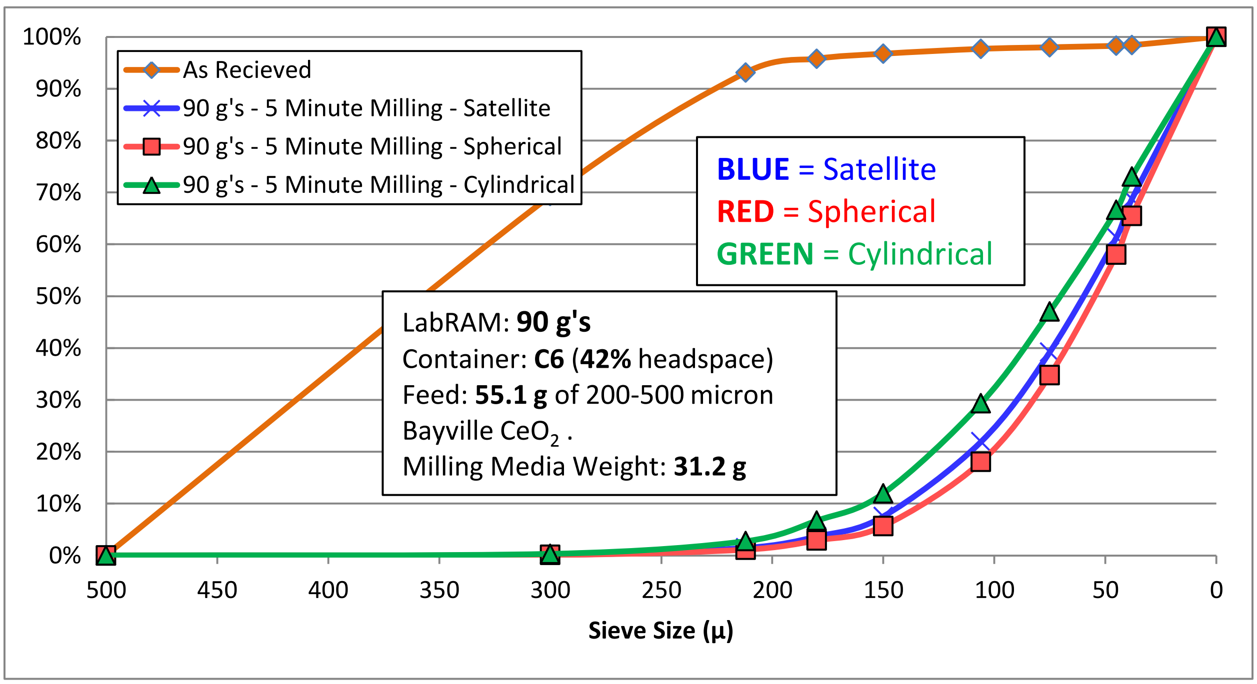


Figure 21: PSA Results for F/M Ratio of 64/36 (Container C6)

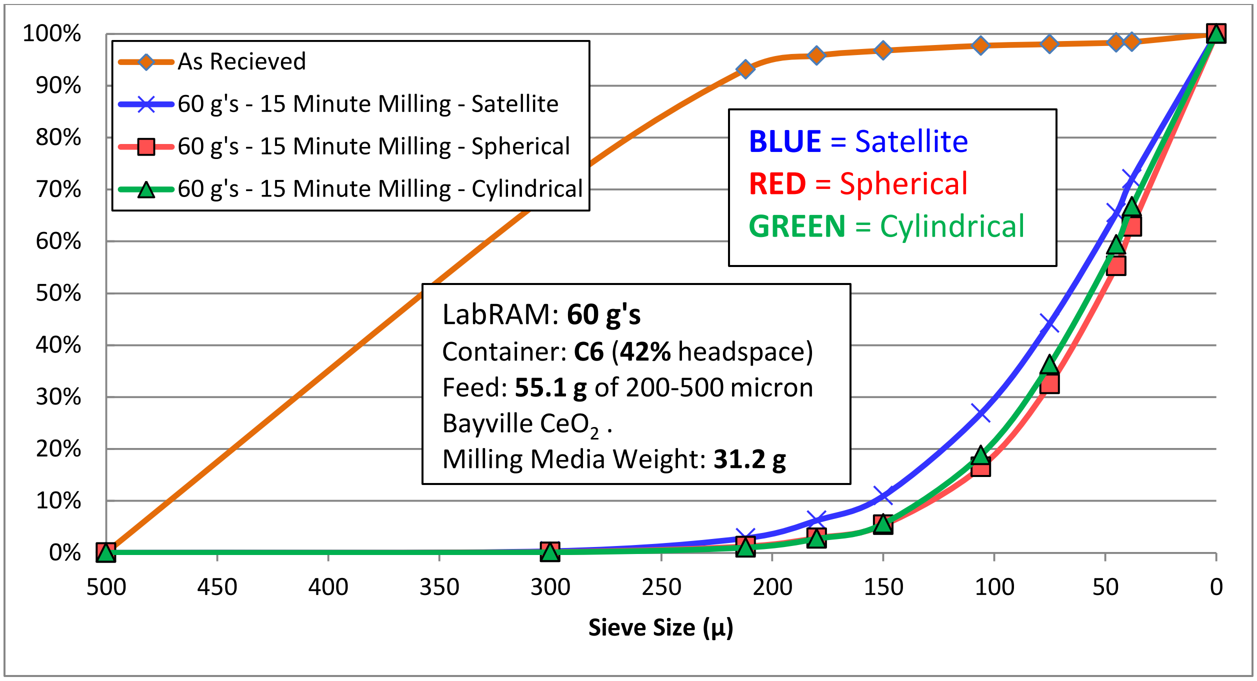


Figure 22: PSA Results for F/M Ratio of 64/36 (Container C6)

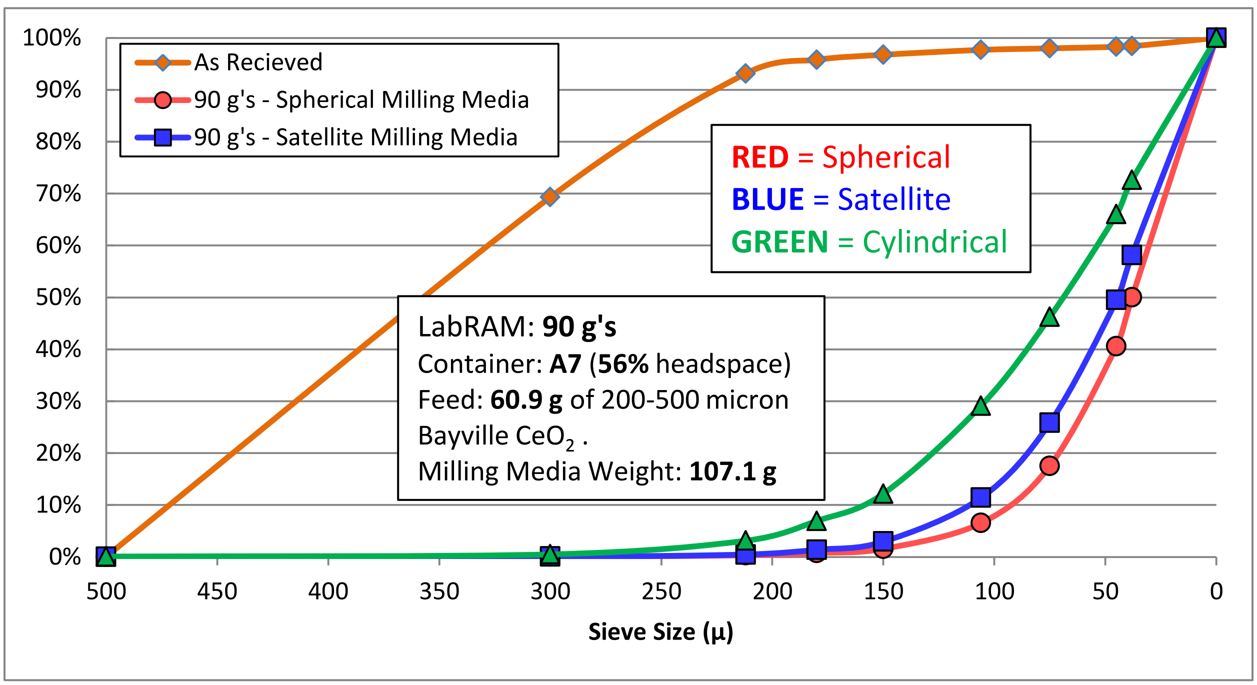


Figure 23: PSA Results for F/M Ratio of 36/64 (Container A7)

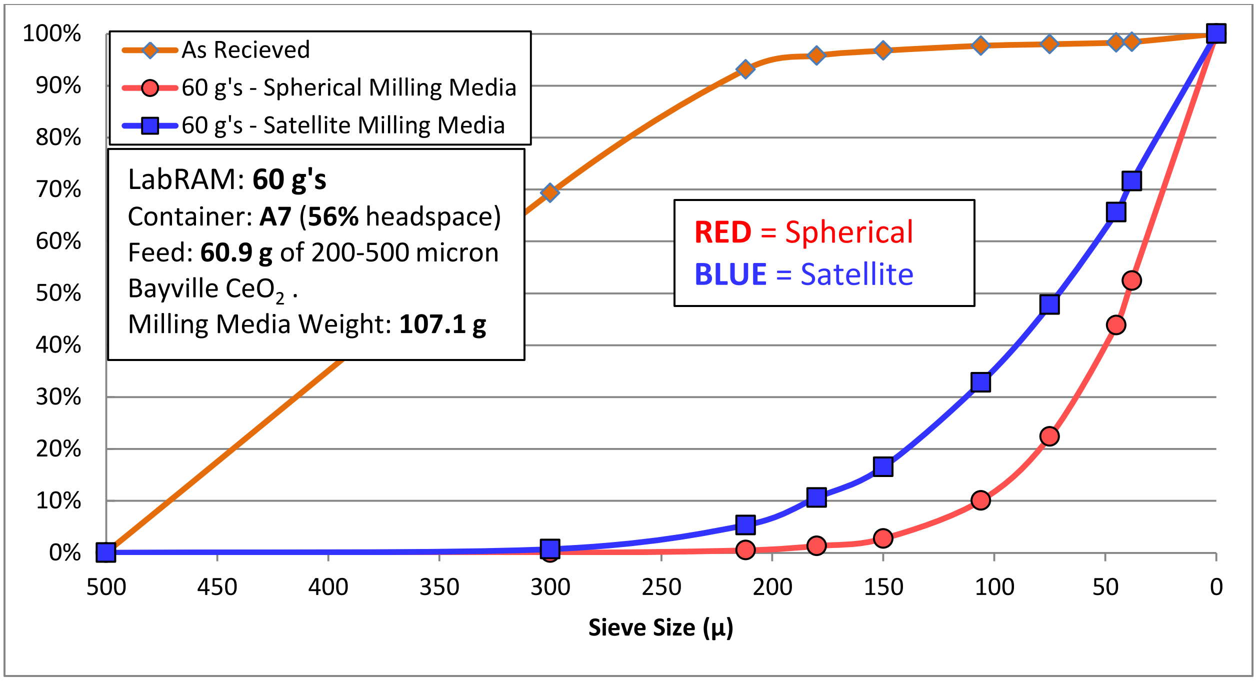


Figure 24: PSA Results for F/M Ratio of 36/64 (Container A7)

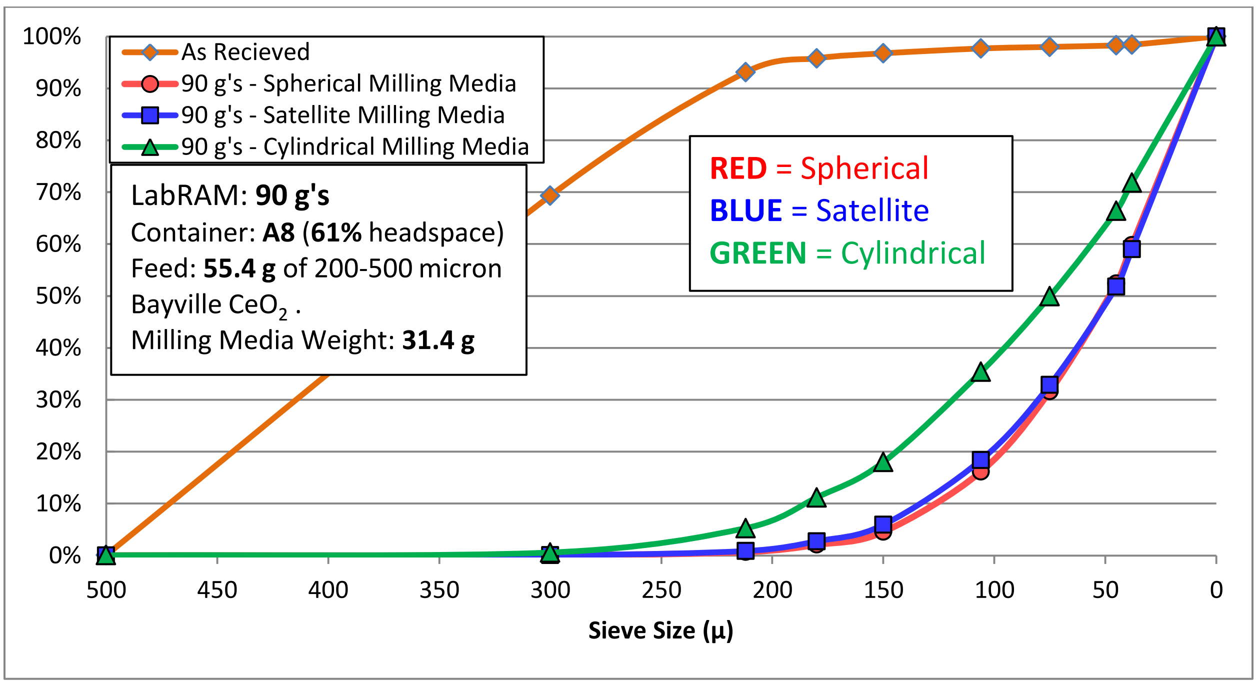


Figure 25: PSA Results for F/M Ratio of 64/36 (Container A8)

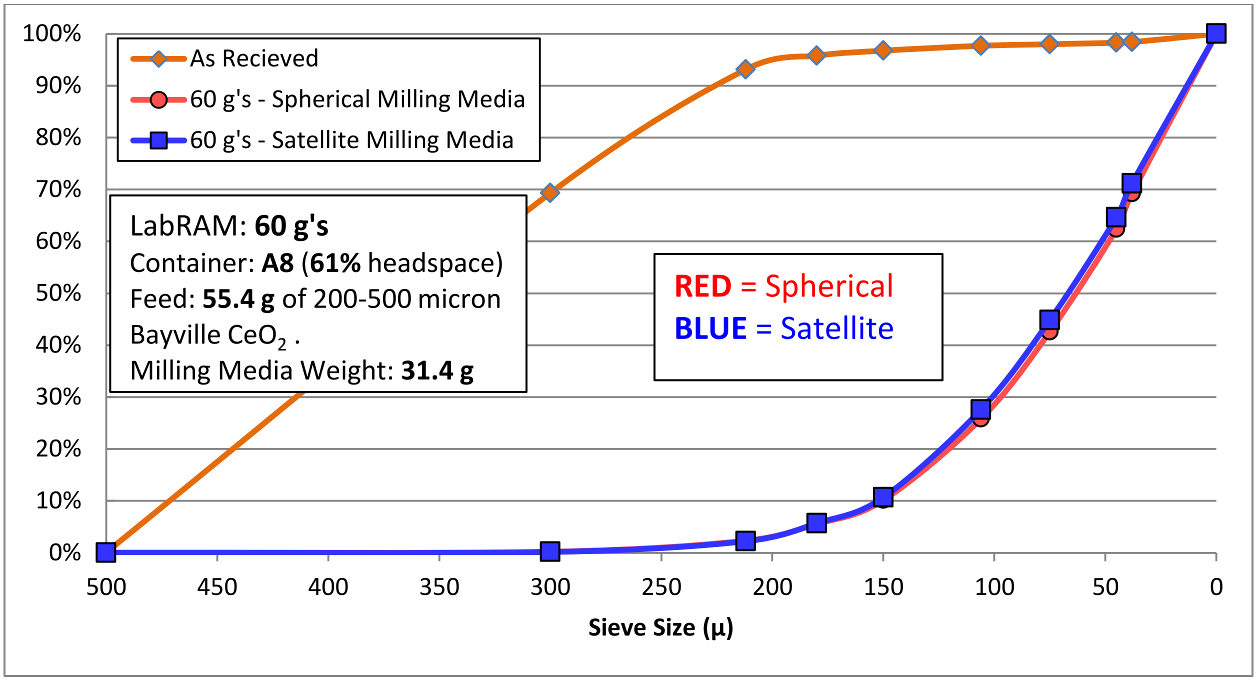


Figure 26: PSA Results for F/M Ratio of 64/36 (Container A8)

Table 11: Approximate Number of Milling Media Particles Used for Each Test

Milling Media Type	F/M Ratio: 64/36	F/M Ratio 36/64
Spherical	80	274
Satellite	58	198
Cylindrical	42	144

As mentioned in section 5.3.2 of this report, the main objective of this test series was to determine the amount of material <38 microns that is produced when milling a sample of 200-500 micron cerium oxide to 99% <212 microns. However, it would take hundreds of tests to mill samples to exactly 99% <212 microns for every test condition being explored in this test series. Instead, tests were performed until enough data was obtained to confidently interpolate or extrapolate to 99% <212 microns. Interpolation was preferred over extrapolation for accuracy, but in a few cases where interpolation was not feasible, extrapolation was used. Figure 27 shows an example of the type of curves that were produced for interpolating and extrapolating data. To obtain the desired data from Figure 27, a horizontal line was drawn from 1.0% on the left axis (% >212) over to the curves defined by squares which represent the % >212. From the intersection of the horizontal line and the curves defined by the squares, vertical lines were projected up or down to the corresponding curves defined by triangles. From the intersection of the vertical lines and the curves defined by triangles, horizontal lines were projected over to the right-hand axis. The value where the horizontal line intersects the right-hand axis represents the % <38 microns that would theoretically be produced if the sample was milled to exactly 99% <212 microns.

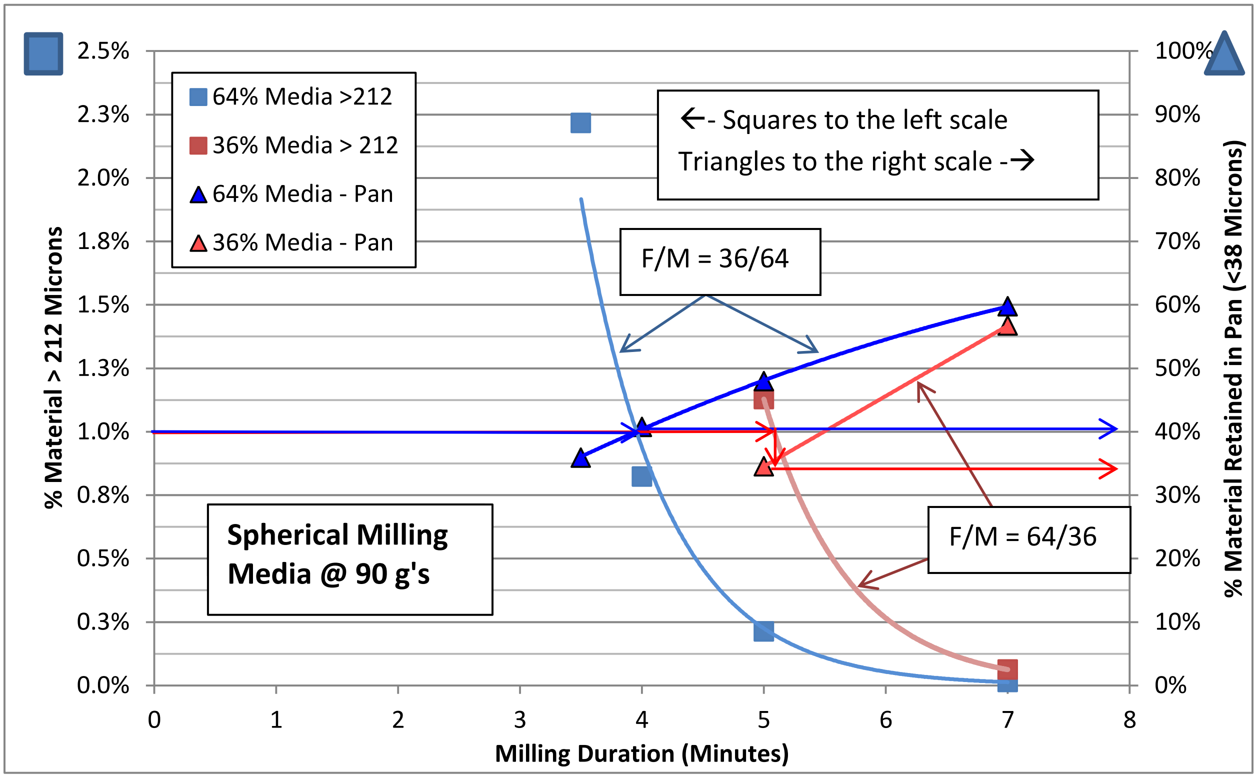


Figure 27: Effect of F/M Ratio on Particle Size Reduction

Using the method outlined in the previous paragraph, the percent of material <38 microns that would result from milling the 200-500 micron cerium oxide to 99% <212 microns was determined for each test condition. Table 12 shows a summary of this data for each of the test conditions of interest. Appendix E contains all of the figures (similar to Figure 27) that were used to extract the data presented in Table 12.

Table 12: Percent of Material < 38 Microns @ 1% > 212 microns with Test Conditions

Milling Media	G's	F/M Ratio	Milling Time (Min)	Container Number	Container Geometry	Container Height	Headspace	%<38μ
Cylindrical	60	64/36	57.2	A7.1	Annular	1.75"	85%	33%
Cylindrical	60	64/36	15.0	C6	Cylindrical	2.025"	42%	33%
Cylindrical	60	36/64	16.5	A7	Annular	1.75"	56%	34%
Cylindrical	60	64/36	11.8	A8	Annular	1.00"	61%	34%
Cylindrical	60	36/64	9.8	C5	Cylindrical	2.625"	44%	34%
Cylindrical	83	64/36	20.8	A7.1	Annular	1.75"	85%	32%
Cylindrical	83	64/36	2.7	A8	Annular	1.00"	61%	38%
Cylindrical	89	36/64	5.0	A7	Annular	1.75"	56%	33%
Cylindrical	90	64/36	6.4	C6	Cylindrical	2.025"	42%	32%
Cylindrical	86	36/64	4.8	C5	Cylindrical	2.625"	44%	35%
Spherical	60	64/36	46.0	A7.1	Annular	1.75"	85%	34%
Spherical	60	64/36	8.3	A8	Annular	1.00"	61%	35%
Spherical	60	64/36	15.3	C6	Cylindrical	2.025"	42%	38%
Spherical	60	36/64	9.6	C5	Cylindrical	2.625"	44%	44%
Spherical	60	36/64	7.5	A7	Annular	1.75"	56%	45%
Spherical	83	64/36	17.8	A7.1	Annular	1.75"	85%	33%
Spherical	83	64/36	1.9	A8	Annular	1.00"	61%	39%
Spherical	89	36/64	3.4	A7	Annular	1.75"	56%	43%
Spherical	89	64/36	5.1	C6	Cylindrical	2.025"	42%	35%
Spherical	89	36/64	4.0	C5	Cylindrical	2.625"	44%	40%

The 5th column in Table 12 displays an alphanumeric "Container Number." The letter is used to denote whether the container is annular or cylindrical (A=annular, C=cylindrical). The number refers to the actual container number, as defined historically. See Appendix B for container details. Container number A7.1 refers to container 7, but contains the ".1" to differentiate between the tests where container 7 was loaded according to its design (F/M of 36/64; 56% headspace) and the tests where container 7 was loaded using a F/M ratio of 64/36 and a headspace of 85%. The latter was used for a group of tests that were performed in order to determine the effect[12] of varying headspace/container height for a given F/M ratio. The results obtained also allowed for the determination of the effect[5] of varying the F/M ratio for a given container. It should be noted that Table 12 does not include any data for satellite milling media. This is because testing was focused primarily on spherical and cylindrical milling media, since these were determined early on to be the extremities with respect to the production of material < 38 microns. Table 13 compares the results from tests using satellite milling media where values could be

[12] Effect on the particle size reduction of cerium oxide such that 100%[12] of the milled material is milled to less than 212 microns and as few fines (material <38 microns) are produced as possible.

interpolated for 1% > 212 microns to the corresponding spherical and cylindrical milling media results. As seen in Table 13, at a F/M ratio of 36/64, satellite milling media produces less material <38 microns than spherical media, but produces more than cylindrical media—this applies for both 60 and 90 (nominal) g's. At a F/M ratio of 64/36 and 60 g's, all 3 milling media types produce similar results (with respect to material < 38 microns). There is no data for satellite milling media at a F/M ratio of 64/36 and 90 g's.

Table 13: Percent of Material < 38 Microns at 1% > 212 Microns

Container	F/M Ratio	G-Forces	Satellite %<38µ	Cylindrical %<38µ	Spherical %<38µ
A7	36/64	60 g's	38%	34%	45%
A7	36/64	89 g's	38%	33%	43%
A8	64/36	60 g's	33%	34%	34%

All of the data in Table 14 and Table 15 is already shown in Table 12, but Table 14 and Table 15 present the data so that it is easier to make the necessary comparisons. The purpose of Table 14 is to show the effect of varying the F/M ratio[13] in a given container, milling media type, and g-force. Comparing the milling durations required to mill the material to 99% less than 212 microns shows an extraordinary difference between the 64/36 and 36/64 F/M ratio tests (a 247-513% increase in the time required), which was most likely a result of the difference in headspace as shown in Figure 28 (Containers 7.1 and 7, respectively). Table 14 also shows that for *cylindrical milling media*, varying the F/M ratio and headspace has little or no effect on the amount of material produced below 38 microns (despite the tremendous difference in milling time). However, for *spherical milling media* there is an 11% increase in the amount of material below 38 microns that is produced when milling at a F/M ratio of 64/36 as opposed to 36/64, suggesting that for spherical milling media, a F/M ratio of 64/36 and headspace of 85% are preferred over a F/M ratio of 36/64 and headspace of 56%, respectively.

[13] Varying the F/M ratio while maintaining about the same amount of feed material, resulted in a significant change in headspace (56% to 85%). Because of this, any variance induced by changing F/M ratio may also have been caused by the change in headspace.

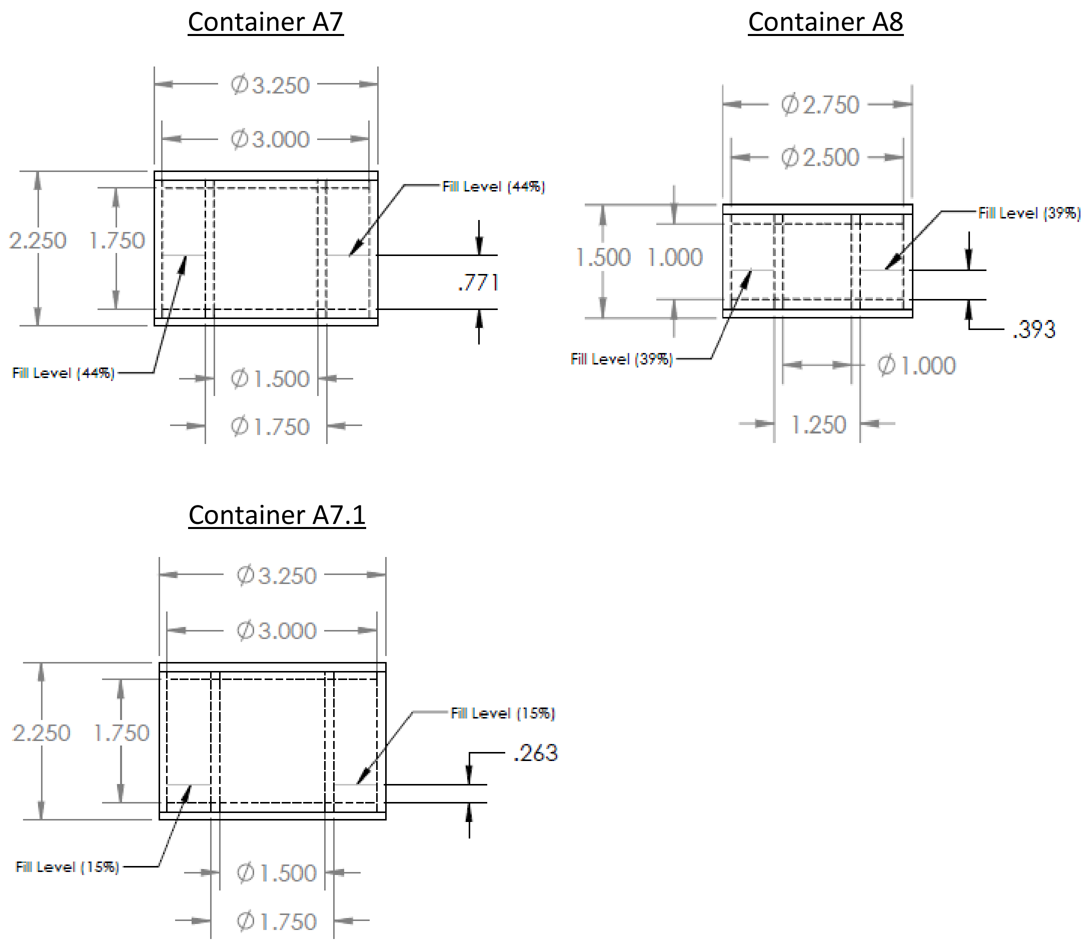


Figure 28: Container A7, A7.1, and A8 Drawings, Dimensions, and Fill Levels

Table 14: Effect of F/M Ratio and Headspace on the Size Reduction of Cerium Oxide (to 1%>212µ)

% > 38µ[14]	F/M Ratio	Headspace	G's	Milling Media Type	Container	Milling Time (Min)	Container Height (in)[15]
33%	36/64	56%	89	Cylindrical	A7	5.0	1.75
32%	64/36	85%	83	Cylindrical	A7.1	20.8	1.75
43%	36/64	56%	89	Spherical	A7	3.4	1.75
33%	64/36	85%	83	Spherical	A7.1	17.8	1.75
34%	36/64	56%	60	Cylindrical	A7	16.5	1.75
33%	64/36	85%	60	Cylindrical	A7.1	57.2	1.75
45%	36/64	56%	60	Spherical	A7	7.5	1.75
34%	64/36	85%	60	Spherical	A7.1	46.0	1.75

[14] Percent of material produced below 38 microns when milling the sample such that 99% of the material is less than 212 microns.
[15] Inside Height

The purpose of Table 15 is to show the effect of varying headspace while keeping the F/M ratio constant. Based on the results shown in Table 15, it seems that varying the headspace between 61-85% affects the amount of material produced below 38 microns for the higher g-forces (83 g's), but does not have any effect when milling at a lower g-force of 60 g's. As shown in Table 14, varying the headspace does significantly impact the amount of milling time required to size reduce the sample to 99% less than 212 microns (a 316-837% increase in the time required).

Table 15: Effect of F/M Ratio on the Size Reduction of Cerium Oxide (to 1%>212μ)

% > 38μ	F/M Ratio	Headspace	G's	Milling Media Type	Container	Milling Time (Min)	Container Height (in)[8]
38%	64/36	61%	83	Cylindrical	A8	5.0	1.00
32%	64/36	85%	83	Cylindrical	A7	20.8	1.75
39%	64/36	61%	83	Spherical	A8	1.9	1.00
33%	64/36	85%	83	Spherical	A7	17.8	1.75
34%	64/36	61%	60	Cylindrical	A8	11.8	1.00
33%	64/36	85%	60	Cylindrical	A7	57.2	1.75
34%	64/36	61%	60	Spherical	A8	8.8	1.00
34%	64/36	85%	60	Spherical	A7	46.0	1.75

Figure 29 contains four charts which show all of the directly comparable results obtained for the acoustic mixer milling studies (i.e. interpolated results for which there was 1%>212 microns). The red horizontal lines represent the range of optimum results (31%-34%). Each of the four charts in Figure 29 represents samples milled at the g-force and milling media type specified in the lower right corner of each chart. Each chart contains results from tests run at both F/M ratios (64/36 and 36/64) and in all of the containers for which the relevant data is available. Each point in the charts represents a unique container (which implies a unique height and/or headspace as indicated in the legend). The legends at the top of each chart show an alphanumeric description for each container where the letter (C or A) represents whether the container used was annular or cylindrical, and the number represents the container number, which is consistent with the numbers assigned to each container in previous reports as well as in container drawings and images. Figure 29 can be used to quickly compare the results of changing several variables. Figure 29 also indicates that, with the data that is currently available, chart A (cylindrical milling media at 60 g's) produced the most consistently favorable results in comparison with the other three charts (i.e. it has the most data points in the optimum range). This suggests that cylindrical milling media at 60 g's is less sensitive to other variables (such as headspace, container height, and F/M ratio) and therefore represents the test conditions for which the optimum range (31%-34% < 38 microns) can be most easily achieved and would be less influenced by other variables. It should be noted that Figure 29 does not include any data for satellite milling media. This is because testing was focused primarily on spherical and cylindrical milling media, since these were determined early on to be the extremities with respect to the production of material < 38 microns. Figure 29 also omits 30 g's data since this test condition was found to be unfavorable early on due to a large production of fines and slow rate of particle size reduction.

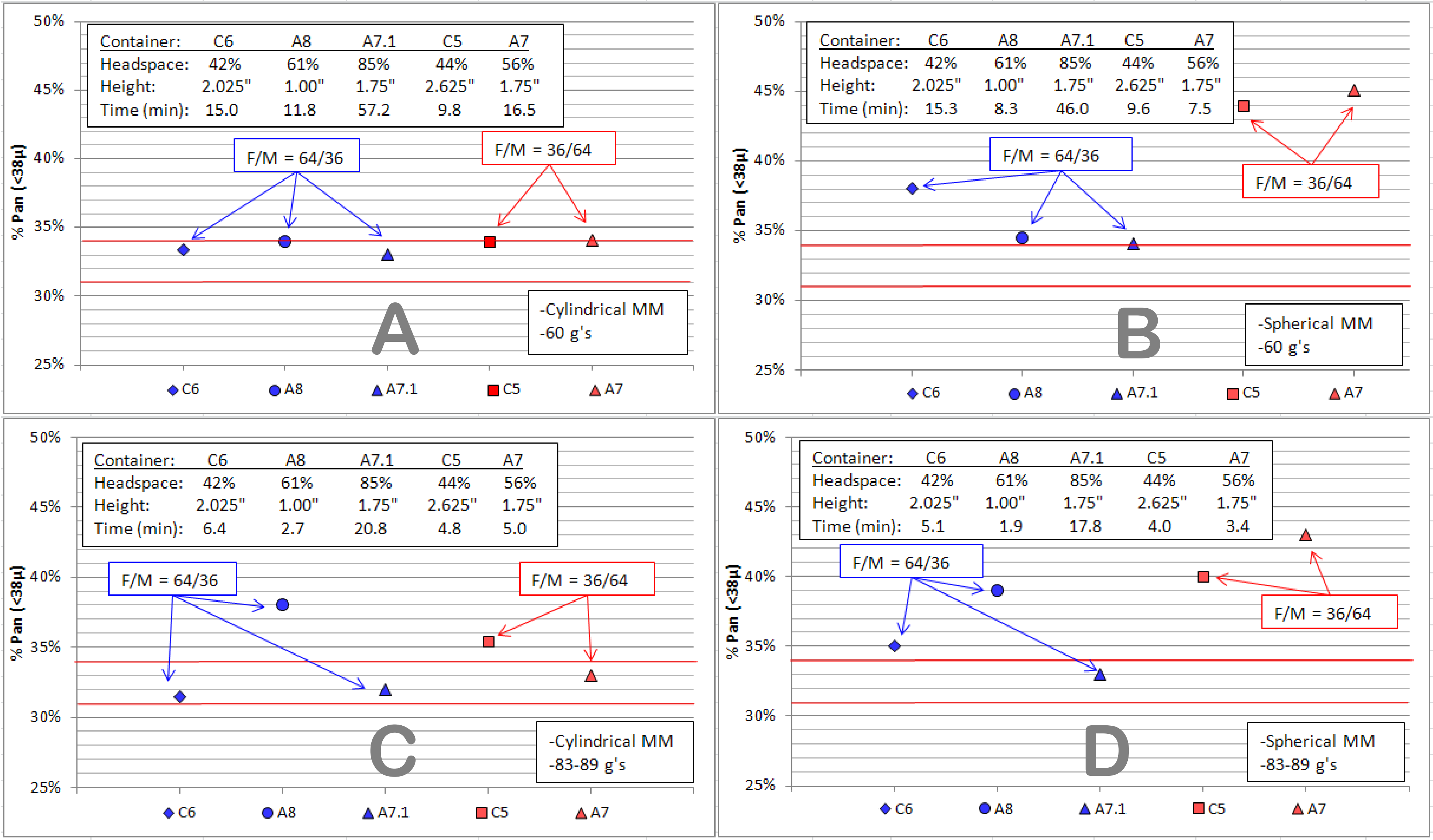


Figure 29: Percent of Material < 38 Microns @ 1% > 212 Microns

Some limited analysis of material less than 38 microns was also performed using a laser particle size analyzer. The results are presented in Appendix G and indicate that at the optimized result of <1% greater than 212 microns and 30-34% less than 38 microns there is approximately 7-8% of material less than 5 microns (all on weight basis).

Although cylindrical media produces fewer fines than spherical media during milling, a potential disadvantage to using cylindrical milling media was observed during testing. Figure 30 shows new and once used cylindrical milling media. As demonstrated in the images, the cylindrical milling media particles are damaged during milling. As a result, chips from the milling media were observed in the 180, 212, 300, and 500 micron sieves when performing the sieve analyses on cerium oxide that had been milled with cerium oxide. Figure 30 is a picture taken of the material retained on the 212 micron sieve from a sample milled with cylindrical media at 90 g's. While this is a concern when using cylindrical aluminum oxide milling media particles, using a more ductile or abrasive milling media may reduce or eliminate the chipping/fracturing observed in Figure 30. It is also possible that milling at a lower acceleration would reduce or eliminate this phenomenon. This damage did not occur to the spherical or satellite milling media.

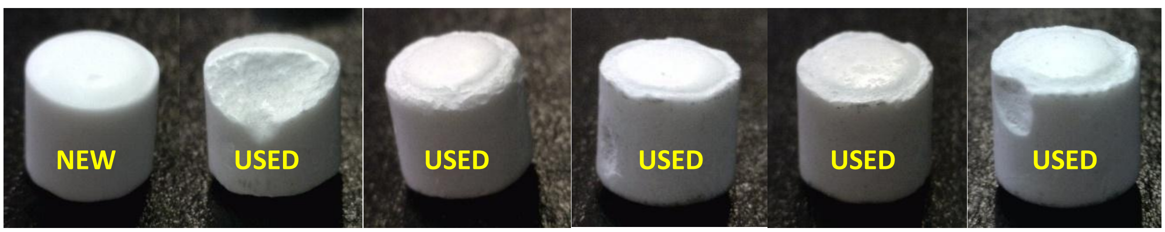


Figure 30: New and Used Cylindrical Milling Media (Once Used)

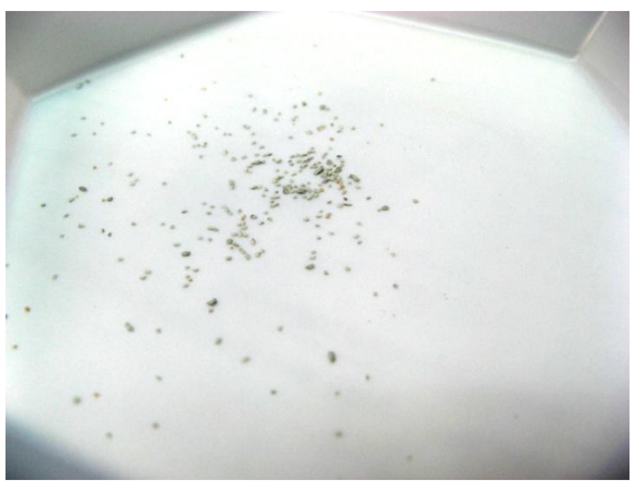


Figure 31: Photo of Cylindrical Media Chips

6.3.3 Effect of Milling Media Density on Particle Size Reduction

The results supporting the major conclusions from this study are shown in Figure 32. A more detailed report is included in Appendix F. Looking at the results from tests 2 and 5 in Figure 32, it is clear that using more YSZ particles increased the particle size reduction. Comparing tests 1 and 5 shows that increased milling media density does increase particle size reduction- both tests used an equivalent number of particles for reduction, but the test with the YSZ media yielded significantly more size reduction. Comparing tests 3 and 5 shows that less dense milling media (Al_2O_3) can outperform denser milling media (YSZ) if there is a sufficient difference in the number of particles used, such that more media particles are used for milling with the less dense media than for milling with the denser media.

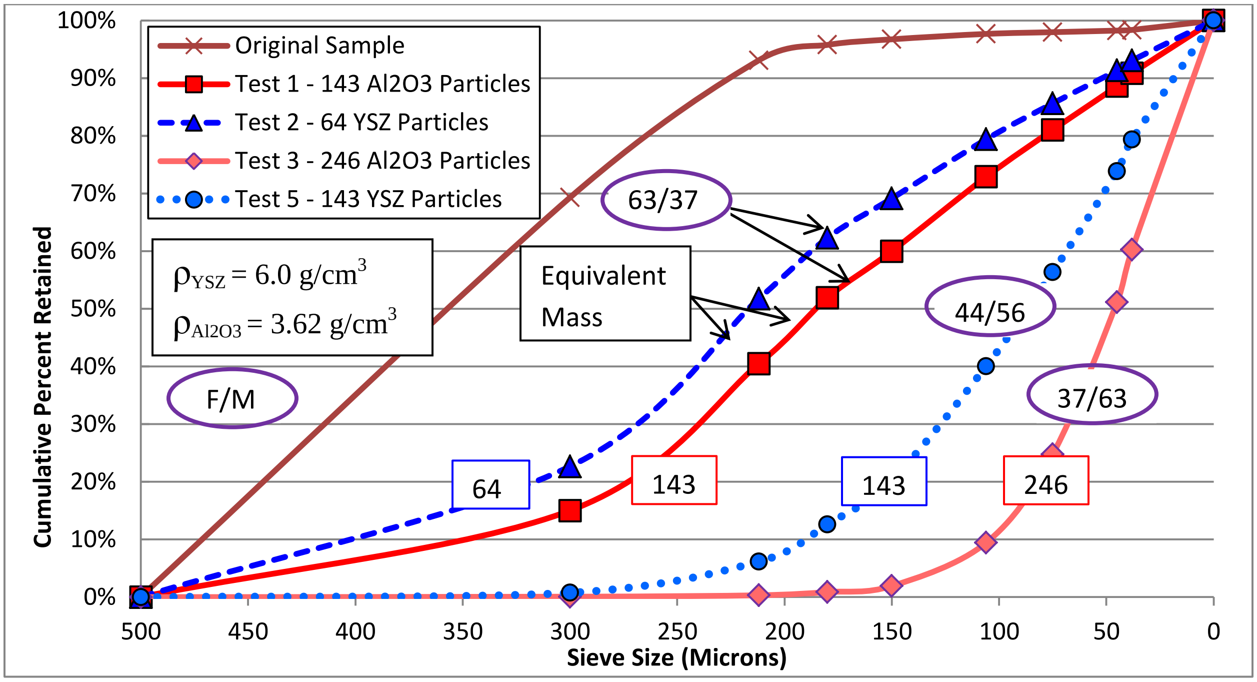


Figure 32: Effect of Number of Media Particles and Density on Particle Size Reduction

6.4 Acoustic Mixing Max Loading Study

A blending study was performed using 1.5 kg and 3.0 kg of 90-grit brown and white aluminum oxide. For blending durations and other testing information, see Table 16 (Test #2.1-2.7). For each test a mix index was calculated. See Figure 33 for mix index results. The operational limitations and behavior of the LabRAM were studied using 3 different surrogates at 8 different loadings ranging from 1 kg to 4.5 kg. The LabRAM was able to operate under all of the conditions tested, and some degree of blending was observed during all tests. Results from these studies are presented in Table 16. Table 16 also includes hyperlinks to videos of the blending that can be accessed by clicking on the video number in the far right column. There was a noticeable difference in the blending behavior of each surrogate. For a more thorough presentation and explanation of the results of this study, see Reference 3.8.

Table 16: Summary of Max Loading Test Conditions and Results

Test #	Loading (kg)	Duration (sec)	Max g's (g's)	Avg g's (g's)	Video Number
Brown & White 90-grit Aluminum Oxide - Blending Study					
2.1	1.5 kg	30	7	6	400
2.2	1.5 kg	150	7	6	403
2.3	1.5 kg	349	7	6	402
2.4	3.0 kg	30	7	6	395
2.5	3.0 kg	150	7	6	399
2.6	3.0 kg	349	7	6	398
2.7	3.0 kg	600	7	6	404
Brown 90-grit Aluminum Oxide – LabRAM Operating Limit Study					
Test #	Loading (kg)	Duration (sec)	Max g's (g's)	Avg g's (g's)	Video Number
1.1	1.0 kg	156	11	6	N/A
1.2	1.5 kg	12	6	6	N/A
1.3	2.0 kg	42	6	6	N/A
1.4	2.5 kg	42	8	8	N/A
1.5	3.0 kg	60	7	6	N/A
1.6	3.5 kg	54	5	5	N/A
1.7	4.5 kg	48	4	4	N/A
200-500µ As-received Bayville Cerium Oxide - LabRAM Operating Limit Study					
Test #	Loading (kg)	Duration (sec)	Max g's (g's)	Avg g's (g's)	Video Number
3.1	1.0 kg	78	28	16	405
3.2	1.5 kg	144	16	15	406
3.3	2.0 kg	78	11	11	409
3.4	2.5 kg	60	8	8	N/A
3.5	3.0 kg	66	6	6	N/A
3.6	3.5 kg	78	5	5	N/A
3.7	4.0 kg	36	4	4	N/A
3.8	4.5 kg	90	4	4	412
Double Roll Mill-Processed Cerium Oxide - LabRAM Operating Limit Study					
Test #	Loading (kg)	Duration (sec)	Max g's (g's)	Avg g's (g's)	Video Number
4.1	1.0 kg	102	14	10	413
4.2	1.5 kg	72	5	4	414
4.3	2.0 kg	72	3	2	415
4.4	2.5 kg	84	4	4	416
4.5	3.0 kg	36	7	5	N/A
4.6	3.5 kg	96	21	17	417
4.7	4.0 kg	54	19	17	N/A
4.8	4.5 kg	126	18	15	N/A

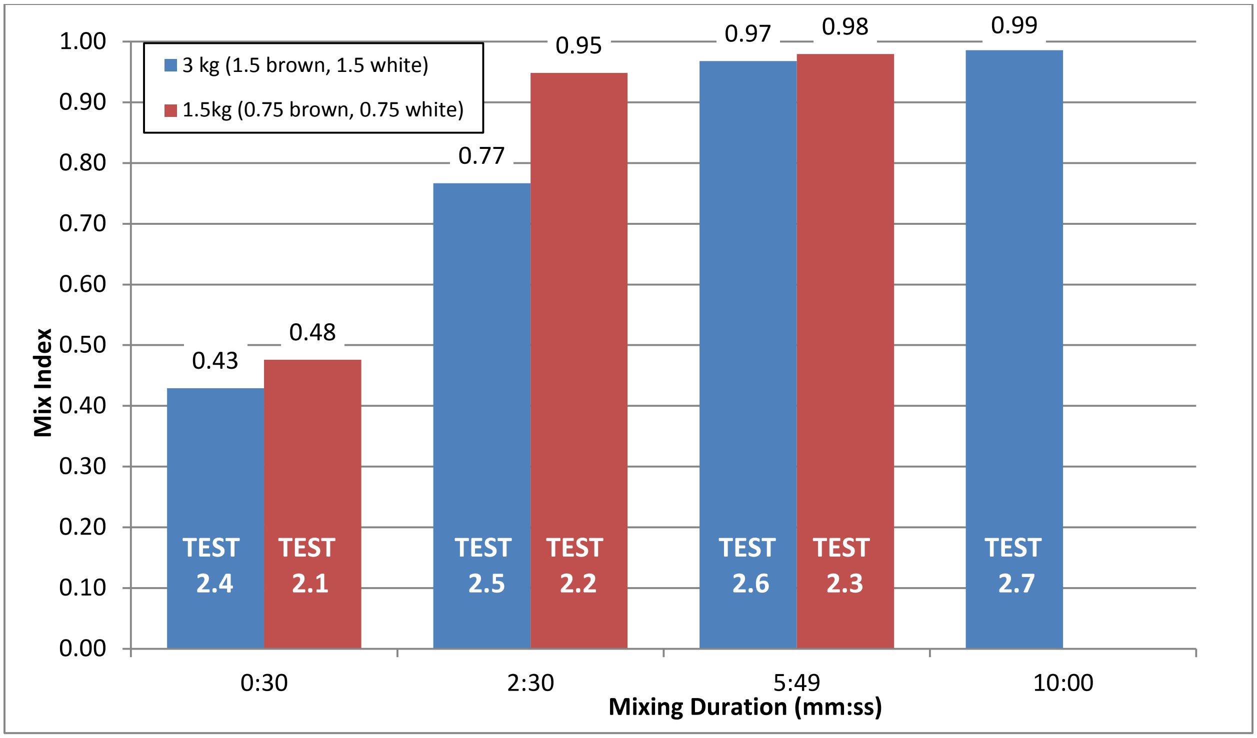


Figure 33: Mix Index for 1.5 kg and 3.0 kg Payloads as a Function of Blending Duration

6.5 Container Design Guidance

The containers for blending and milling on the LabRAM were designed using results from the pre-operational study. Once calculations were performed based on these results, a 25% reduction factor was applied to the payload to be used in each container. This reduction factor was applied to account for the possibility of additional damping that would occur when performing tests using test conditions that had not been previously studied. However, even with the 25% reduction factor, the LabRAM was still unable to achieve the desired acceleration (g's) for some of the tests performed. As a result, it is recommended that for future testing, a 50% reduction factor be applied in order to ensure the capability of the LabRAM to achieve the desired acceleration under the prescribed test conditions.

7.0 Conclusions

7.1 Bench-Scale Acoustic Mixer Blending Studies Using <200 Micron White and Brown Aluminum Oxide

- The LabRAM was capable of producing results comparable to those of the conical blender (mix index greater than 0.90) and met the PDC project criterion (mix index greater than 0.5 for blending). It should be noted that less material was used in the LabRAM study (< 1 kg) than in the conical blender study (Approximately 10 kg).
- Under the conditions tested, container geometry (cylindrical versus annular cylindrical) did not affect blending results.
- Under the conditions tested, the LabRAM was able to reach a mix index greater than 0.95 in 5 seconds.

7.2 Bench-Scale Acoustic Mixer Autogenous Size Reduction Studies Using Roll Mill Processed Cerium Oxide

- Testing showed no indication of significant particle size reduction.
- For double roll mill processed cerium oxide, homogeneity was observed throughout the cylindrical container after blending.
- There was no significant difference between results obtained using a cylindrical container versus an annular cylindrical container.
- Mixing double roll mill processed cerium oxide on the LabRAM appeared to break up most of the "pancake-shaped" particles.

7.3 Bench-Scale Acoustic Mixer Size Reduction Studies Using 200-500 Micron Cerium Oxide

7.3.1 Autogenous Size Reduction

- The LabRAM was not capable of autogenously size reducing 200-500 micron cerium oxide to a target particle size range between 5 and 200 microns under the conditions tested.
- When blending cerium oxide in the LabRAM, a small degree of autogenous size reduction occurred. Most of the size reduction occurred within the first 10 minutes of blending, suggesting that the size reduction may have been the result of agglomerated material being broken up.
- The temperature of cerium oxide increased dramatically with respect to blending time, but began to level off at around 150° C after about 20 minutes of blending (under the testing conditions explored in this study).
- The particle size distribution of the material in the container after blending did not exhibit complete vertical uniformity. The bottom layer contained slightly more fines than the other layers. The sample used (300-500 micron cerium oxide) contained only a small amount of fines. Using a finer sample of cerium oxide or the same

sample after further particle size reduction, may produce results with less uniformity than the tests outlined in this report.

- The sample mixed in the annular cylindrical container showed a slightly higher degree of particle size reduction than the sample mixed in the cylindrical container. However, this may have been a result of higher g-forces rather than the difference in container geometry.

7.3.2 Size Reduction Using Milling Media

- All test series have shown that the effect each variable has on the characteristics of particle size reduction (%>212μ and %< 38μ) varies with respect to the other variables. For example, it was observed that varying the headspace in a container has a significant effect at 83 g's, but not at 60 g's. This is just one example of a common occurrence that has been observed throughout testing.
- Overall, milling a sample at 60 g's using cylindrical milling media minimized the effects of other variables, while still producing optimum results (31%-34% < 38 microns at 1% > 212 microns).
- The following list presents the milling media types in order from least to most favorable with respect to the production of material < 38 microns during milling: spherical, satellite, cylindrical. For the conditions tested the degree of improvement when switching from spherical to satellite was similar to that achieved by switching from satellite to cylindrical.
- It was observed that cylindrical milling media was damaged during milling, resulting in flakes from the milling media particles being mixed in with the feed material being milled (cerium oxide). Reducing the acceleration (g's) at which the sample is milled may reduce or eliminate this phenomenon.
- Milling at 30 g's was determined to be undesirable based on large production of fines (compared to milling at 60 or 90 g's) and long milling duration, both of which are characteristic of this milling condition.
- The effect of F/M ratio on the production of fines (material < 38 microns) during milling was dependent upon variables such as acceleration (g's) and milling media type. For spherical milling media, a F/M ratio of 64/36 typically produced less fines than a F/M ratio of 36/64. For cylindrical milling media at 60 g's, the F/M ratio had no effect, whereas at 90 g's the effect varied and was therefore indeterminate. It is also important to note that neither F/M ratio consistently produces optimum results, except when milling is done using cylindrical media at 60 g's.
- Size reduction studies using milling media also showed that a sample of 200-500 micron cerium oxide milled under optimum conditions to the target value of 99% <212 microns, generated 32-34% material < 38 microns and an estimated 7-8% material < 5 microns.

7.3.3 Effect of Milling Media Density on Particle Size Reduction

- When an equivalent number of particles were used (constant volume), the denser milling media induced a higher degree of particle size reduction (samples were milled for the same duration).
- When an equivalent weight of feed and milling media were used (constant mass) the less dense milling media (Al_2O_3) induced a higher degree of particle size reduction due to the presence of additional milling media particles.

7.4 Acoustic Mixing Max Loading Study

- The LabRAM was able to achieve a mix index of 0.97 after 5 minutes and 49 seconds for a 3kg payload.
- The LabRAM was able to achieve a mix index of 0.95 after 2.5 minutes for a 1.5kg payload.
- The LabRAM was able to operate and induce some degree of blending under all of the conditions tested.

7.5 Container Design Guidance[16]

- Accelerations as high as 90 g's were desired for this test series.
- The LabRAM has limited power and is rated at 0.5 kg maximum payload.
- Data in this report indicates there are numerous variables that can influence the maximum G's achievable for a given payload and that often a payload of less than 0.5 kg is required if 90 G's of acceleration is desired.
- Based on preliminary studies the maximum load that would allow 90 G's of acceleration to be obtained were estimated. Then loads were then decreased by 25%.
- This reduction was insufficient to achieve the desired 90 g's for some tests.
- Based on these results, in the future, it would be more efficient to estimate container design and loading based on historical data, then apply a 50% reduction factor rather than attempting to precisely predict the response of the LabRAM to unique test conditions.

[16] Only applies to the LabRAM mixer as it has limited capacity

8.0 Appendices

8.1 Appendix A – Matrox Analysis Method

8.2 Appendix B – Container Design and Drawings

8.3 Appendix C – USB and EM Images

8.4 Appendix D – Table of Test Conditions

8.5 Appendix E – Charts used for the Extraction of % < 38µ Data

8.6 Appendix F – Milling Media Density Study Report

8.7 Appendix G – Laser Particle Size Distribution Results

8.8 Appendix H – Ordering Information for Cerium Oxide