
**Pacific Northwest
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**Surrogate Development for
Fernald Silos 1 & 2
Accelerated Waste Retrieval Program**

D. Mullen

September 2002



Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RL01830

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Richland Washington 99352

Summary

The Department of Energy contracted with PNNL to prepare specifications for surrogate waste materials to be used in testing equipment for the retrieval and stabilization of wastes from Fernald Silos 1 & 2. This document outlines the rationale for the specification of those surrogates.

The surrogates were designed to replicate, as nearly as possible, the known properties of the silo waste most significant to the processes of hydraulic mining and pipeline transport as slurry. The significant properties for which characterization data were available were the density and particle size distribution.

To support project requirements and budget constraints, two surrogate formulations were proposed; one for the full-scale sluicing tests requiring about 400 cubic meters of material, and one for the component test loop, requiring about 300 kg.

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1. Introduction

This document describes the rationale and methodology for the preparation of surrogate waste materials in support of the engineering design and testing of apparatus for retrieval and processing of wastes stored in the Department of Energy Fernald Site Silos 1 & 2. The design and testing results for that equipment will be reported elsewhere.

2. Background

2.1. Waste Characterization

The K-65 material was sampled and characterized by International Technology Corporation in 1990 and again in 1991-2. A total of 27 samples were taken from three silos, 11 from Silo 1, 11 from Silo 2, and 5 from Silo 3. The results of these efforts were presented by Tank and Rhyne as Certificates of Analysis, dated March 2 1990 and June 29, 1992 (References 2, 3). These Certificates were partially reproduced in Reference 1. Complete copies of the documents were obtained and used for this work.

Extensive characterization work was also performed by Florida International University (FIU) and reported in Reference 4.

2.1.1. Particle Size Distribution

Particle size distributions (PSD) were reported^{1,2,3} for 1989 and 1991 samples in terms of “weight% finer than” for a range of particle diameters. The salient observations to be made about the summarization of the data averaged for each silo are:

- Both silos exhibit a bimodal distribution of particle size with significant fraction of mass concentrated in the 1 to 7 μ and 100 to 250 μ ranges.
- Silo 2 has a relatively large fraction of 20 – 75 μ particles and +425 μ particles at the expense of the smaller ranges.
- The mass median particle diameter for Silo 1 is 10 μ where the mass median particle diameter for Silo 2 is 100 μ .

Particle size distributions determined by Florida International University (FIU)⁴ were expressed in terms of “population % of particles larger than” the range of particle diameters, making correlation to Tank and Rhyne data (“mass % finer than”) indirect. Generalizations in this document are based on the assumption that the material furnished to FIU was a mixture of K-65 samples having properties representative of an average of in-situ properties. The provenance of the K-65 material used by FIU was not reported. Particle size distributions reported by FIU were for samples taken at different levels in partially-settled specimens, so proved to be of little use for characterization of bulk K-65 material.

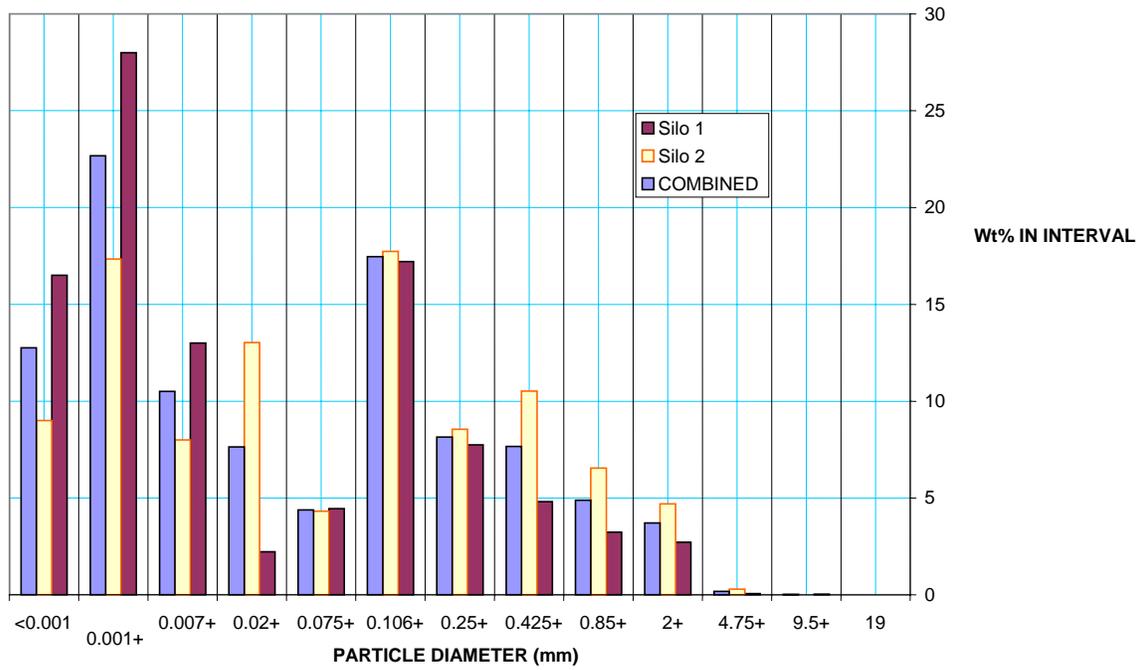


Figure 1. Particle size distribution for K-65 sampled 1989 & 1991.

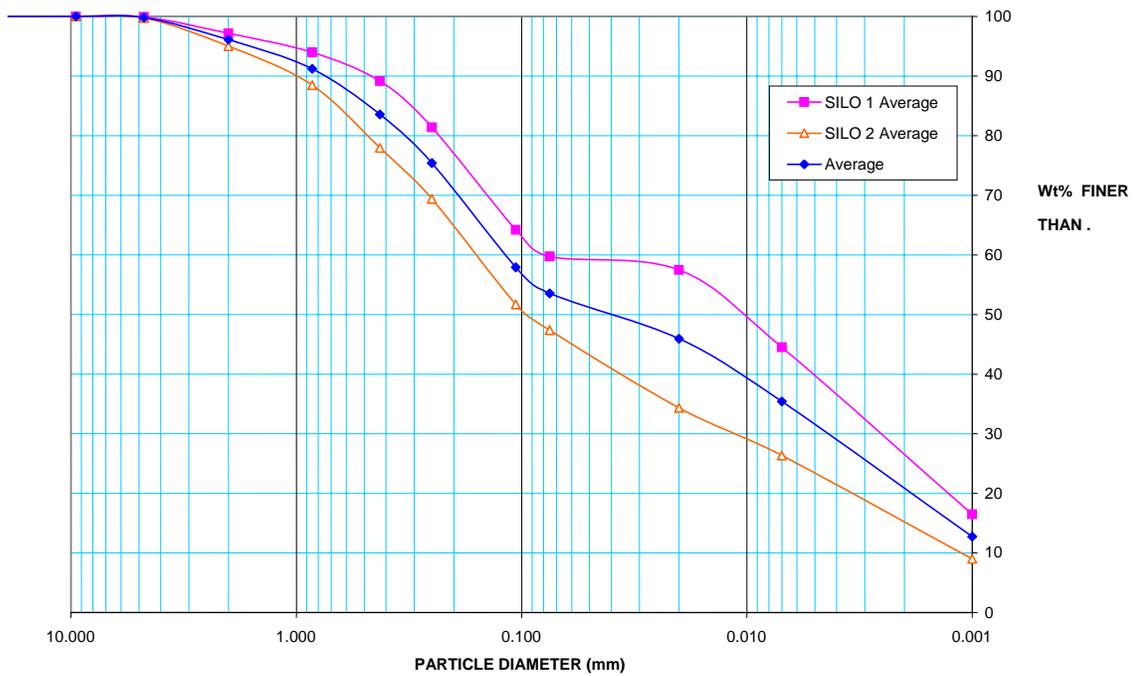


Figure 2. Particle size distribution as weight % finer than particle diameter.

2.1.2. Water Content

The water content of the K-65 samples, where reported, varied within the 20 to 82% range.

2.1.3. Plasticity

Atterberg Limits

Where soil plasticity properties were reported^{2,3}, the liquid limit varied from 34 to 70, with most values in the 50s, corresponding to poorly to moderately graded soils.

2.1.4. Settling

The settling tests reported by FIU⁴ were performed on sampled material and sampled material mixed with Bentonite at varying concentrations. As little as 5 wt% Bentonite had a significant effect on settling rates, and 10 wt% bentonite completely inhibited settling.

2.1.5. Specific Gravity/Density

Reported specific gravity of the Silo 1 material samples averaged 2.95 with a standard deviation of 0.23. For Silo 2, the SG average was 3.00 with standard deviation of 0.02.

2.1.6. Compaction/Consolidation

One-dimensional consolidation and compaction tests were performed by IT Analytical Services^{2,3} on a limited number of different samples from each silo. Optimum moisture content for compaction ranged from 24 to 46%, and dry density from 1120 to 1760 kg/m³ (70 to 100 lb/ft³). Moisture content in the samples used for consolidation testing was generally much higher than the optimum content, ranging from 49 to 82%. Void ratios for undisturbed samples were from 1.82 to 2.76, and were typically reduced by a factor of 0.25 to 0.3 under 1530 kPa (16 tons/ft²) of pressure during lab consolidation testing. Assuming a maximum 8.23 m (27 ft) depth of material, having density of 1600 kg/m³ (100 lb/ft³), the pressure at the silo floor is 129 kPa (2700 lb/ft²). At this pressure, consolidation test data indicates that consolidation was typically about 5% of the starting void ratio, e.g. a starting void ratio of 2.00 might be reduced to 1.90 if the material was near optimum moisture. In many cases the material in the samples characterized by consolidation or proctor tests was well above optimum moisture and most of it is under less overburden *in situ*, so the material *in situ* could be expected to be under-consolidated.

2.1.7. Elemental Composition

Silicon (51%), lead (9%), carbon (5%), iron (4%) and barium (4%) are the most prevalent elements present, expressed as wt% oxide basis. The specific minerals or compounds present are reported in the Technical Requirements Document¹.

2.1.8. Radiological

Radiological properties are not of concern to the development of surrogates.

2.1.9. Organic Carbon

Oil and grease were reported in concentrations of 0.36 to 2.70% in Silo 1 and 0.03 to 0.05% in Silo 2. The overall concentration is unknown for either silo. Total organic carbon was reported in concentrations of 1.92 to 2.62% in Silo 1 and 0.60 to 2.44% in Silo 2. Process knowledge accounts for the presence of kerosene in Silo 2. The source of the oil and grease is not accounted for.

No attempt is to be made to duplicate the organic carbon or oil/grease component in the surrogate. The composition of the reported organics is unknown and adding plausible analogues to the surrogate would complicate disposal of the material to an unwarranted extent.

2.1.10. Slurry Viscosity

FIU has developed correlations⁴ for viscosity of the K-65 with and without BentogROUT (trade name for bentonite grout marketed by CETCO – Colloid Environmental Technologies Company) on the basis of a small number of data points. In many cases the slurry settled too rapidly for the viscosimeter to be useful. It seems likely that settling affected the readings for most of the samples tested, except those with significant fractions of bentonite.

The pipeline pumping resistance data from FIU⁴ is probably a better basis for comparison for surrogates, as long as it is not scaled. Using the correlations for a friction factor to Reynolds number, $f(Re)$, is tenuous at best, given the considerable uncertainty of the viscosity data.

2.2. Test Objectives

Two test programs⁵ are to be conducted by The Providence Group at the test facility in Oak Ridge, Tennessee.

2.2.1. Integrated Test Loop (ITL)

The Integrated Test Loop was designed to evaluate the effectiveness of the sluicing equipment and the submersible pump for bulk waste retrieval. The test cell included a test tank of ~305 m³ (400 cubic yards) capacity, two sluicing monitors (nozzle with an aiming system) and mounting frames, and the slurry pump and its mounting/positioning system. Ancillary equipment included sluicing supply pumps, a raw water supply pump for a high-pressure spray nozzle set at the slurry pump inlet, and settling tanks in which to separate the working fluids from the surrogate slurry recovered from the test tank.

Testing in the ITL was designed to evaluate the retrieval system under various combinations of sluicing jets pressure/flow, using various sluicing procedures. It provided a basis for estimating the characteristics of the slurry output stream and for development of efficient mining strategies for the retrieval project.

2.2.2. Component Test Loop (CTL)

The Component Test Loop was designed to evaluate the performance and wear characteristics of the slurry pump, proposed instrumentation, and other transfer system components. The CTL consisted of a small slurry reservoir in which a slurry pump was supported. The discharge from the pump was routed through a short pipe loop that included the components under test and then back to the reservoir.

3. Requirements for Surrogate

3.1. Quantity

The ITL required about 305 m³ (400 cubic yards) of surrogate to fill to the test depth. The CTL required only about 320 kg (700 lb) of solids to charge the tank and pump loop with 15 wt% solids.

3.2. Cost

The budget for surrogates was initially set at \$30,000, including procurement, mixing, placement and disposal. This proved to be a serious constraint, especially for the large quantity of ITL material.

3.3. Availability

Because the budget was inadequate to cover shipping, the materials to be used for the ITL surrogate had to be procured locally to the Oak Ridge test location.

3.4. Handling

Surrogate materials were required to present no special hazards of toxicity beyond what could be mitigated with simple protective measures. They were to be handled with standard construction equipment (loaders, dump trucks, hand tools). Nuisance dust and minor respiratory irritants were allowable since the operators mixing and placing the material could be adequately protected by masks, gloves, and dust mitigation.

3.5. Disposition

The surrogate materials were required to be suitable for disposal as unregulated fill material.

3.6. Properties

The properties of interest for the surrogate materials were primarily those related to the hydraulic processes to which the waste would be subjected (sluicing, pumping, pipe transport and settling) and secondarily the associated factors of abrasion and seal effectiveness.

4. Methodology

Following is a discussion of the methodology for development of the specification for the surrogate.

4.1. Selection of Properties

Only a few properties could be practically specified, otherwise the economical procurement of the requisite quantities would have been impossible. The properties believed to be most significant to sluicing and slurry transport were the particle size distribution, material density, particle shape and hardness.

We knew that the K-65 material was processed from raw uranium ore by milling (likely in a crusher, then in a roller or ball mill), so the particle shape would be generally angular rather than spherical, and probably could be approximated well by similarly milled mineral material. We also had good information on the particle size distribution and density. The hardness of the K-65 is unknown, but it does contain a significant fraction of siliceous materials, which typically have a hardness of up to 7 on the Mohs scale.

4.2. Simplification of Characterization Data

Some of the K-65 particle size distribution (PSD) data was available in graphical form and some in tabular. There were typically 20 size ranges, of which only 16 or 17 were populated. Prospective vendors generally did not have data for the fine end of the PSD for their materials, so to simplify the specification, the values from several of the smaller-size range “bins” were lumped together. The particle size ranges used for initial specification were:

Screen Size	Particle Size (mm)
No. 4	4.75
No. 8	2.36
No. 16	1.18
No. 30	0.60
No. 50	0.30
No. 100	0.15
No. 200	0.075
Finer	<0.075

Later the specification was refined to use the following screens and sizes:

Screen Size	Particle Size (mm)
0.375in	9.5
#4	4.75
#10	2
#20	0.85
#40	0.425
#60	0.25
#140	0.106
#200	0.075
*	0.02
*	0.007
*	0.001
*	<0.001

* Sizes are not measured with physical screens, but with laser particle counters.

The PSD data from each silo was averaged and the average of both silos combined was also calculated. Because the test program and budget did not allow for multiple surrogates to represent each silo, the decision was taken to use the averaged silo waste properties as the basis for the surrogate.

4.3. Density Compensation

The average specific gravity for the K-65 materials was 2.97, which is a little higher than the 2.65 to 2.8 typical of the common minerals such as limestone and silica. Because slurry transport properties are closely related to settling properties, we decided to adjust the particle size in the surrogate such that the velocity response time

$$\tau = \rho D^2 / 18\mu$$

for a given surrogate particle size would match the average velocity response time for the corresponding K-65 particle size. To do this, the particle diameters were multiplied by

$$\left(\frac{\rho_1}{\rho_0}\right)^{.5}$$

so

$$\tau = \rho_0 D_0^2 / 18\mu = \rho_0 \left(D_0 \left(\frac{\rho_1}{\rho_0} \right)^{.5} \right)^2 / 18\mu = \rho_1 D_1^2 / 18\mu$$

Obviously, it is not practical to simply multiply the screen mesh sizes by a factor and arrive at a usable specification. The particle size distribution was instead adjusted to effectively “shift” the initial distribution in the appropriate direction. This was done with the following algorithm using the factor *pdf*, where

$$pdf = (SG_0 / SG_1)^{.5} - 1 \text{ or } pdf = (\rho_0 / \rho_1)^{.5} - 1$$

where n = the number of “bins”

$$i = 0, 1, \dots, n+1$$

$$A_i = \text{wt\% in bin } i$$

$$A_0 = 0 \text{ and } A_{n+1} = 0$$

$$\text{for } i = 1, \dots, n+1;$$

$$A'_i = A_{i-1}(pdf) + A_i(1-pdf)$$

$$A''_n = A'_n + A'_{n+1} \text{ (for } pdf > 0).$$

4.4. Blending

The available stock materials were “Block Mat” and “Limestone”, both of which are milled from limestone rock and were available from the local vendor. Neither of the available stock materials matched the specified particle size distribution, but mixtures of the stocks could provide a better approximation. The PSD for a blend of materials was calculated by multiplying the bin weight percent (wt%) values for each material by the proposed weight fraction for that material and summing the results by bin.

4.5. Blend Quality Criteria

Six numerical criteria were considered to evaluate the conformity of a proposed surrogate blend PSD to the specified surrogate PSD. They derived from two general categories, absolute % error (AE) and relative % error (RE). Absolute error is the difference between the proposed and specified bin wt% values. Relative error is the absolute error as a fraction of the specified value. The root mean square of the relative error (*RMS_{re}*)

$$RMS_{re} = \sqrt{\sum (RE^2)}$$

was used as the primary criterion for blend quality.

As will be observed later, the “nominal” surrogate blend specified for the ITL did not match the K-65 PSD very well at all, so the RMS_{re} criterion was of limited value. For the task of tailoring the CTL surrogate, where good variety of graded particles was available and affordable, it was much more valuable.

5. Implementation

This section discusses the practical implementation of the surrogate specification.

5.1. Material Sourcing

Only one vendor in the test facility area was able to provide material with marginally suitable and known particle size distributions in the quantity required and within budget allowed for the ITL material.

The smaller amount of CTL surrogate materials could be economically procured and shipped from vendors outside the test location. This enabled specification of a blend having much higher fidelity to the K-65 material particle size distribution and greater hardness. AGSCO Products supplied graded silica sands and TiO_2 as stock inventory items, delivered promptly.

5.2. Material Sampling

The recommended procedure for sampling the delivered truckload quantities of material was as follows.

- The truckload deliveries of ITL surrogate stock materials were dumped out onto a gravel pad covered with steel plates.
- A 250 ml sample was taken from each of four random locations from within the pile (grabbed at intervals during the placement of the material on the pad) creating a 1-liter composite sample for each mixed truckload.
- The sample number was recorded corresponding to the load tag number in the sample log.

5.3. ITL Surrogate

The ratio of three parts limestone to one part block mat was selected as being simple to specify and implement. This formulation was expected to have a RMS_{RE} value of about 1.85, however this was based on PSD data for #200 mesh and coarser, with all finer particles lumped in one “bin”, per Figure 3.

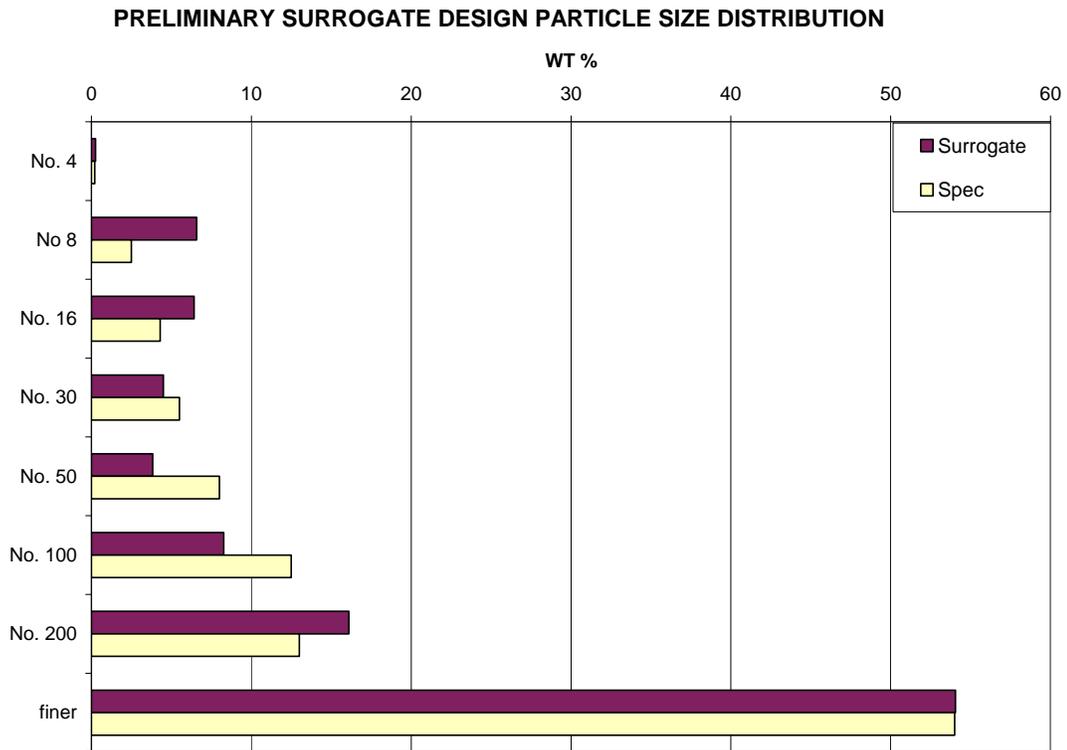


Figure 3: ITL Surrogate PSD based on initial components PSD.

5.3.1. Mixing

Following are the instructions suggested for mixing and placement of the materials.

- Spread the load across the working end of the pad (while dumping if the driver can do it) so it uses the whole width (the short dimension) of the pad. Working from the side of the pile closest to the storage end of the pad, drag or push about a yard at a time from the pile and spread it back on the pad toward the storage end. Do this repeatedly across the width of the pile until you have about a 15cm (6”) deep bed as wide as you can make it in the space available. Now, working from the side of the bed (90 degrees from the direction it was dragged) take scoops of the material with shallow running cuts across the whole width. Try to set the cut depth to about 1/3 of the bed depth so it will take 3 passes to move it all. Pile these scoops at the storage end of the pad, placing them randomly.
- Continue this operation until an entire truckload is mixed. Pile up the material from a truckload in a discrete pile at the end of the pad.
- Perform this operation on each truckload, creating a discrete pile of mixed material on the pad from each truckload.
- Place a tag on each mixed truckload denoting the load number, and date it was mixed.

5.3.2. Placement

- Upon receipt of the sample analysis, consult the lead test engineer for additional mixing or material addition instructions to any specific truckloads.
- Make load adjustments as the loads are taken off the storage area for placement. Drag about half the load out onto the working area, spread the adjustment material onto it fairly evenly, drag the rest of the load out over that, then pile the load on the working end, scooping from the long side of the pad and taking scoops from the pad up. Drag the material off this pile as for mixing (above) and place it per instructions (either back in the storage pile pending sampling and analysis, or directly to the test area).
- If adjustments are made, note the adjustments on the load tag and resample the load in accordance with the instructions listed above.
- Identify the load numbers to be placed in a batch and denote the batch number and date on each load tag for that batch. A batch is defined as the truckloads of material that are placed in the ITL surrogate test tank during any given week.
- Move the piles from the pad and place them in the ITL surrogate tank, placing all materials from a batch in a single horizontal lift covering the full length of the tank if possible.
- Make an entry in the surrogate batch log (Attachment #2) of the date(s) the batch was placed, its approximate depth and location in the tank, and the load numbers the batch is comprised off. Attach the sample analysis results to the batch log sheet.

Continue to fill the ITL surrogate test tank until it reaches the level stated in the “Test Plan for the Fernald Accelerated Waste Retrieval (AWR) Project”

5.3.3. Surrogate Characterization

The PSDs for the ITL and CTL surrogates are presented graphically. The data points shown represent the weight fraction held on the corresponding “screen” (sizes less than 75μ were counted with a Coulter counter, not a screen, and equivalent volume and mass calculated). The smallest size (0.0001mm) represents all material finer than 1μ ; the non-zero value is used to avoid difficulties with generating a log-scale plot.

Raw Materials

More detailed PSD data taken as the materials were delivered revealed that the distribution of the fines was not ideal (Figure 4, Figure 5), and there was considerable variation in the PSD of the raw materials from load to load (Figure 6). The corrected RMS_{RE} value of the material as placed was 4.36 (7).

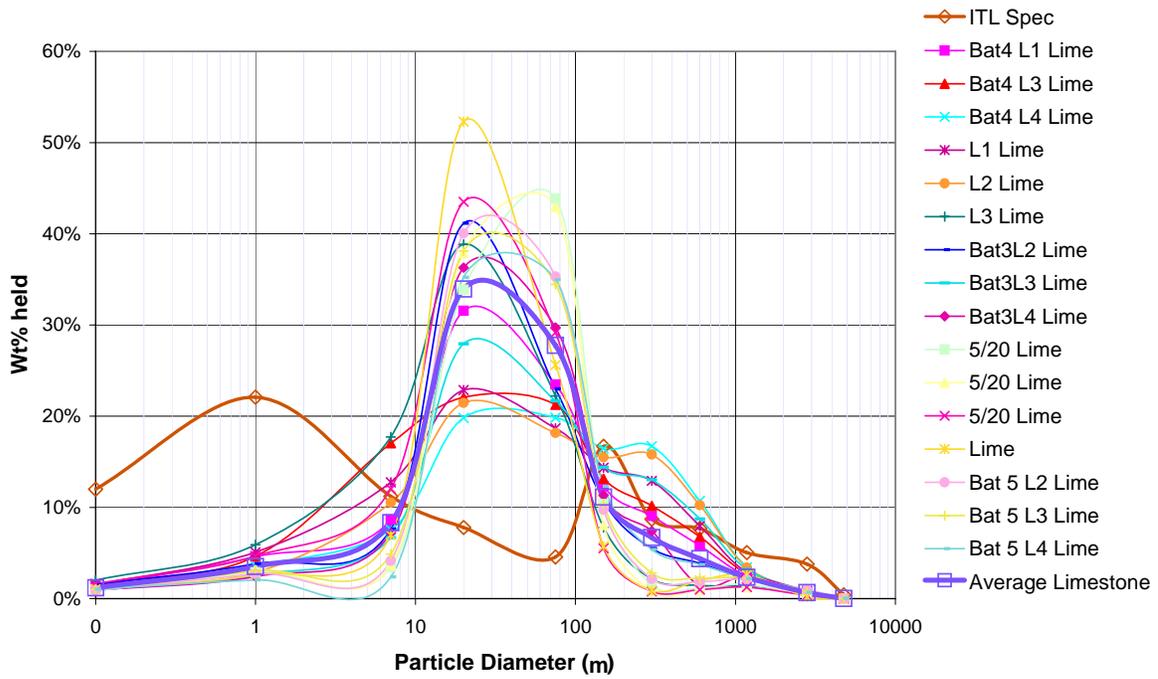


Figure 4: Crushed limestone surrogate material PSD. “Average Limestone” is not weighted.

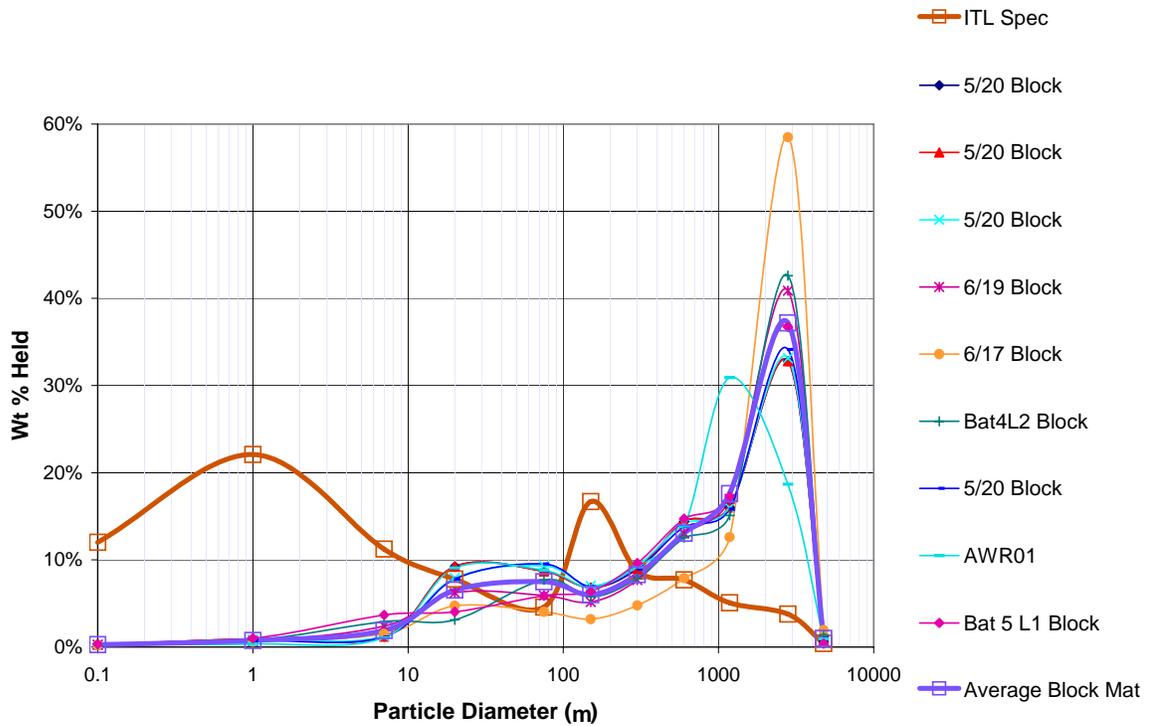


Figure 5: Block Mat PSD “Average Block Mat” is not weighted.

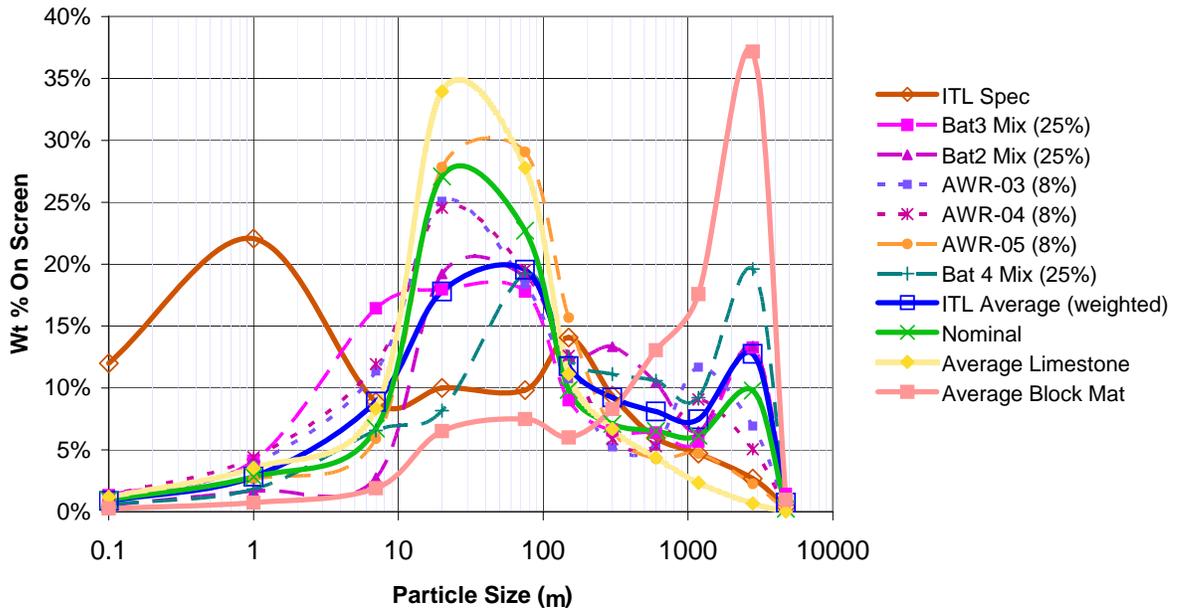


Figure 6: ITL Surrogate PSD by batch and weighted average.

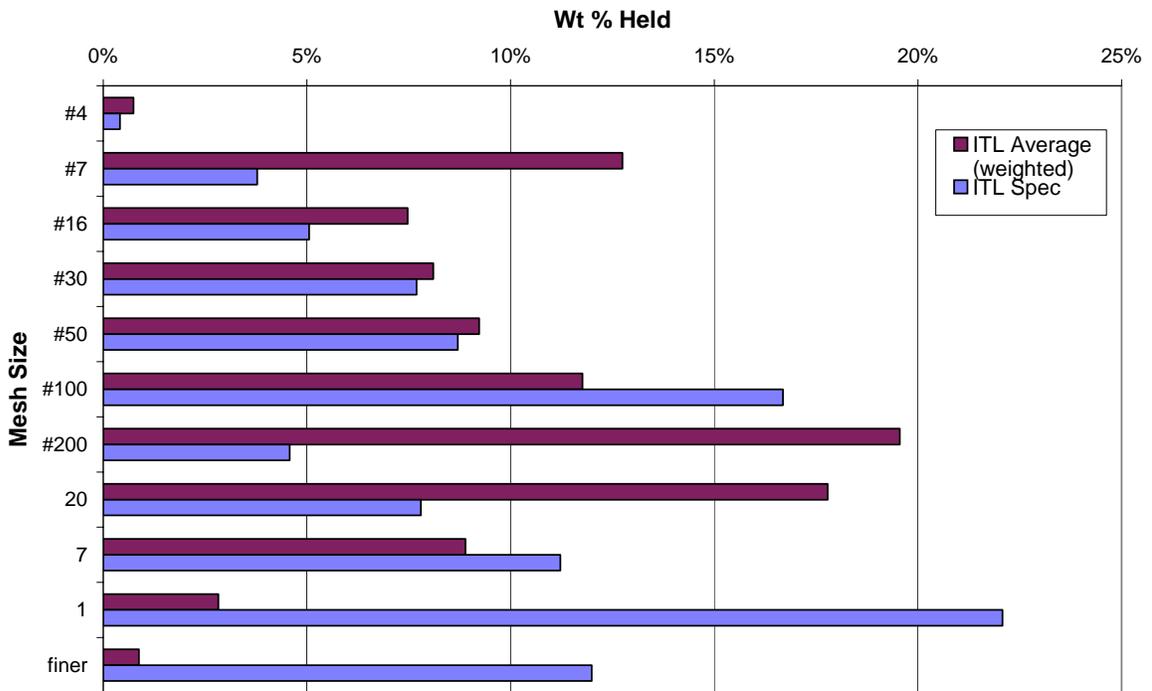


Figure 7: ITL Surrogate PSD from sampling and detailed analysis.

Figure 4 and Figure 5 show the PSD's for the two raw materials, "Block Mat" and "Limestone", compared to the "Specification" for a PSD corresponding to K-65, adjusted for density. 7 and Figure 6 show "Nominal" surrogate PSD (calculated from the raw material PSD's and the mix proportions, and the "Average Batch" data – averaged sample data for ITL simulant. It is quite obvious that neither the "Nominal" surrogate (RMS% error value of 4.76) nor the "Average Batch" (RMS% error value of 3.26) is a particularly good match for K-65. They both are, however, somewhat conservative formulations for testing the pumping system and instrumentation, being generally weighted toward the coarser particles at the expense of the sub-10 μ fraction. The finer particles are generally easier to transport and hinder settling of the larger particles, thereby lowering the saltation velocity for a fluid in which they are present.

"Nominal" is the calculated PSD for the specified blend of "Average Limestone" and "Average Block Mat". Those component PSD's are raw averages of all the sample data, not weighted by the masses of the lots the samples represent, so "Nominal" should not be expected to match the "ITL Average" PSD

5.4. CTL Surrogate

Late in the program, practically on the eve of starting CTL tests, a vendor was located who offered graded silica sand, novaculite (99.5% SiO₂ mineral with rounded platy grains) and TiO₂ (in sub-micron particles) in useful quantities at reasonable pricing and had considerable PSD data in their catalog. A blend of the stock limestone materials and the new materials was formulated which was a substantially closer approximation to the K-65 PSD, and materials were ordered. The new formula called for 15% block mat, 15% limestone and 70% of the new materials blend. The formula theoretically would have a RMS% error of 1.76.

The CTL had already been loaded with ITL surrogate in the 25/75% ratio, so a procedure was devised to iteratively pump off slurry and add water to reduce the concentration, then to add solids to reach a 15% loading. This strategy relied on the assumption, which turned out to be inaccurate, that the slurry in the CTL was being uniformly mixed by the pump/agitator. After the adjustments had been made and the loop run, it was observed that the slurry solids loading was in fact much lower than the target 15%, because the pump tank was too large for the pump to sustain thorough mixing. The larger particles (>75 μ) were evidently settling out in the tank, and had been in that state during the surrogate modifications. Alterations were made to the tank to reduce the volume and additional mixing was induced, probably entraining more of the larger particles.

Further uncertainty as to the composition of the surrogate actually circulating in the CTL loop is apparent in the limited grab sample data available at this time (see Figure 8).

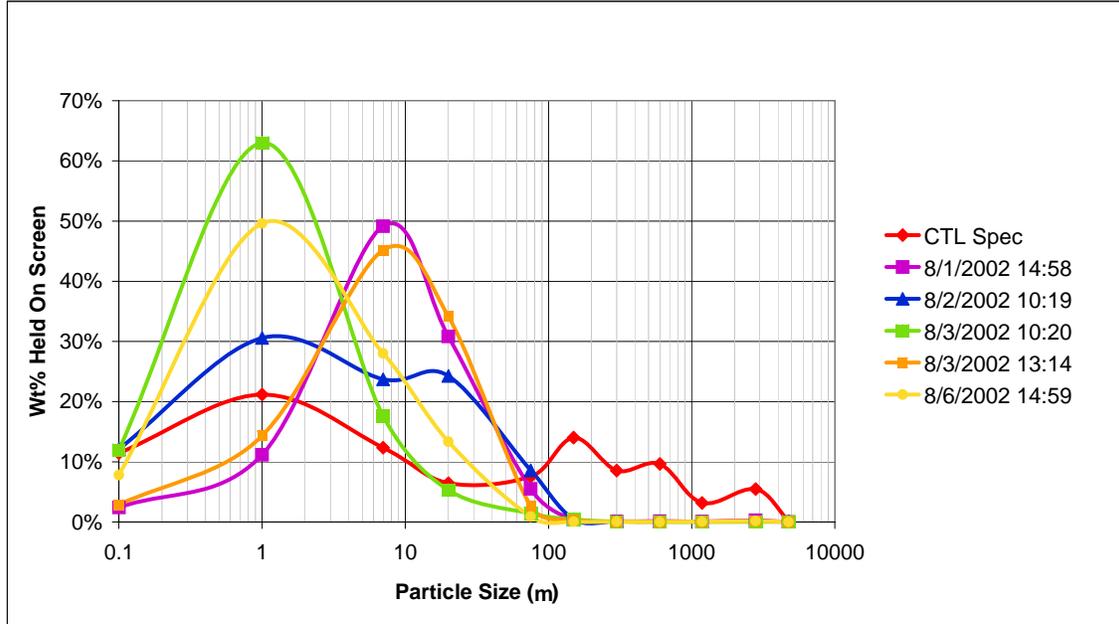


Figure 8: CTL PSD data from sampling, in chronological order.

Note that the data presented in Figure 8 is taken from the pipe loop return, and is not necessarily representative of all the solids in the CTL apparatus. There has been some speculation that the surrogate solids were being ground into finer particles by the pump and self-erosion, but evidence for that is inconclusive. It is also arguable that the larger particles settled out into parts of the reservoir, and would not be present in a grab sample from the circulating medium. It will be necessary to sample the solids in the tank after the test to get a clearer indication, however, the uncertainty regarding the initial charge and the effect of the revisions makes any determination of further change during the test tenuous at best.

Some of the CTL tests were to be repeated using a formulation of graded silica sands matching the specification with a RMS% error value closer to 1.

5.5. Pumping Test Loop Surrogate

As part of the associated PNNL task to consult on the design of the Silos 1 & 2 treatment facility, a K-65 surrogate was designed using graded silica sands for the base, with 3 wt% Al_2O_3 added to enhance abrasiveness. Addition of novaculite and TiO_2 was allowed as required to make up deficiencies in the PSD of the silica feedstocks. This surrogate as purchased pre-mixed and split by the vendor into ~100lb bags to avoid settling segregation in a bulk package. The RMS% error criterion value for this formula is 0.5.

The abrasive-enhanced surrogate was used for endurance testing of plant equipment, particularly the slurry pumps.

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