Low-Level Cementitious Waste Sample Retrieval and Analysis at Savannah River Site - 11149

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ABSTRACT

In order to better define the physical properties of cementitious low-level wastes processed at Saltstone, a team of engineers and scientists from Savannah River Remediation (SRR) and the Savannah River National Lab (SRNL) developed and implemented several tools designed to allow for remote sample retrieval of grout cores from an active disposal vault. The effort was successful at remotely retrieving three sample replicates from each of three different locations inside the waste disposal vault. These cores were stored at SRNL and a variety of analyses have been performed to determine the accuracy with which lab-prepared samples compare with actual waste forms produced by the operating facility. Lessons learned from the initial core sampling effort have resulted in further technology development to ensure that future waste form sampling can be achieved with minimal direct radiological exposure of the facility worker.

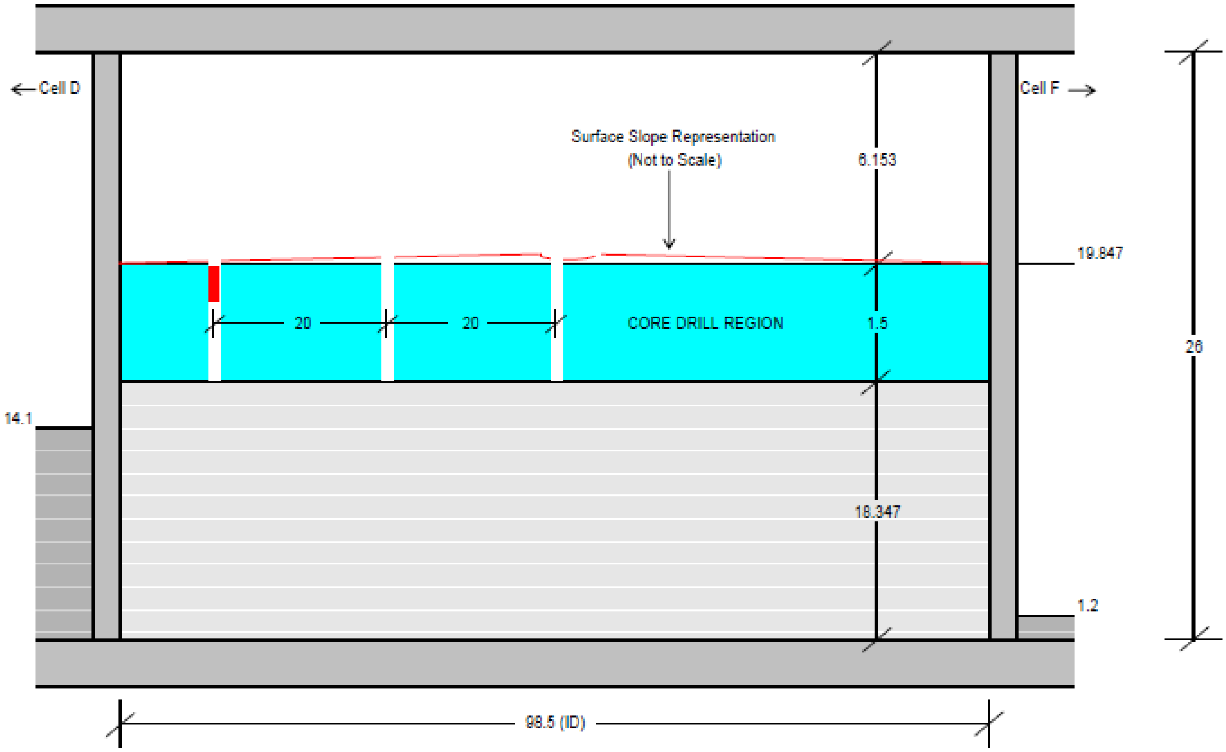
INTRODUCTION

The Saltstone Production and Disposal Facilities located at the Savannah River Site (SRS) in Aiken, SC were originally commissioned in 1990 for the purpose of immobilizing decontaminated low-level salt solutions in a cementitious grout form for long term storage in a near-surface landfill. A radiological performance assessment of the disposal facility was completed in 1992, using waste form property data taken from laboratory-prepared grouts intended to represent the emplaced product. Since 1992, an increased amount of regulatory oversight and the desire to add more technical rigor to the baseline for waste form performance created a need to retrieve samples of grout being stored in an active disposal vault. These samples would be valuable in validating laboratory data as well as defining the process facility's ability to correctly achieve targeted grout mix designs.

CORE SAMPLING EFFORT

Core drilling was the simplest technology available for retrieving samples of already-emplaced saltstone. The tools and techniques for core drilling are widely used in the construction industry for the testing and evaluation of structural concretes in a wide variety of applications. However, sampling inside of a Saltstone disposal vault presented specific challenges created by the limited access to the waste form along with significant administrative controls due to the radiological nature of the samples themselves.

The basic geometry of the disposal vault is shown below in Figure 1. Personnel were able to occupy the area on top of the vault roof, but access to the waste form below was possible only through a series of 7.6 cm (3 in.) penetrations spaced at approximately two meter (6 ft.) intervals over the top of the vault. Detailed observation of the sample equipment was possible only through the use of portable borescope-style cameras.



Note: All dimensions referenced on the diagram are in feet. One inch cell height = 6,048 gallons saltstone.

Fig. 1: Elevation Plan of Saltstone Disposal Vault

Tool Development

The initial core sampling was accomplished through the modification of commercially available core sampling equipment. An electric drill motor was purchased along with several 5 cm (2 in.) coring bits of various kerf thicknesses. Because all the tools would need to be operated inside of a glovebag for contamination control purposes, the components were set up in such a way to provide for ease of operation (e.g., drill motor was separated to operate outside of glovebag, drill pipe sections were short to ensure they could be easily inserted/removed from glove bag). Because the actual strength of the grout was not known, the core drill was designed to have cooling water available from a portable, pressurized tank if lubrication and cooling of the drill bit were required. Upon completion of the basic sampling rig, a series of simulant tests were performed to ensure performance of the equipment prior to field implementation.

Mockup Testing

Testing of the Saltstone core sampler was performed in several stages. SRNL personnel core drilled several pre-existing concrete coupons of various compressive strengths to verify the basic operation of all major components. Because it was recognized that these concretes did not likely represent the actual waste form, SRNL also prepared several simulant grouts in advance and placed them into 200 L (55 gal) drums for coring. As development continued on the facility work control documentation that would govern the sampling evolution, SRNL ultimately performed a full-scale mockup of the equipment with Operations personnel from the facility to ensure that the radiological and work controls identified in work planning would be adequate for success. The top of the vault was simulated using scaffolding and plank flooring, and a drum containing simulant saltstone was placed underneath the scaffold to mimic the geometry of the actual sample vault.

Field Work and Sample Retrieval

The core sampling evolution was performed over the course of two days, September 16th and 17th, 2008 [1]. Due to the extensive preliminary testing and thorough pre-job briefing, the physical work did not encounter significant obstacles other than those presented by the waste form itself. Because saltstone is a low-strength material, SRNL personnel did have difficulty with fouling of the drill bit from fines/tailings and the samples were generally difficult to extract from the drill pipe. As a result, there was significant variation in the condition of the cores from location to location. A typical core sample pipe is shown below in Figure 2.

Fig. 2: A six-inch grout sample shows cracking when removed from the vault.

Transfer and Storage

Discussions with laboratory researchers were held long before sample retrieval was attempted to identify what analytical methods and techniques were available to characterize the grout once it was removed from the disposal vault. Since this was a first-of-a-kind evolution for the Saltstone facility, SRNL recognized that several months of method development and testing would be required before any actual analysis of the grout samples could be performed. This presented a problem for ensuring the integrity of the samples. The sample in Fig. 2 can clearly be identified as greenish or teal in color. This is consistent with simulant samples taken during mockup testing. However, core samples obtained during preliminary testing were observed to oxidize quickly once they were removed from the surrounding monolith. SRNL fabricated sample transporters which were evacuated after the grout was placed into them for shipment to the laboratory receipt area. Upon receipt at the lab, the samples were then transferred into storage cylinders that were nitrogen-blanketed until the samples were ready for analysis. Visual observation of the samples several months after receipt did not indicate any significant change during the time that they were stored [2].

ANALYTICAL RESULTS

The method development, analysis and reporting of results has been a major effort that began two years ago and is still in the final stages of completion. While final results of all property data have yet to be reported, much of the characterization has been finished. A full discussion of the analytical techniques and property data is beyond the scope of this report, but some of the significant conclusions are summarized below. Referenced reports will provide additional detail [2, 6, 7].

Density and Porosity

A large amount of density and porosity data is available for simulant grouts that have been mixed in the laboratory [3]. While the operating facility uses an on-line density monitor for process control during production, this measurement cannot account for changes that occur after placement (e.g., bleed, evaporation, etc.) As a result, there was no data available for comparison to simulant grouts prior to this effort. The density of the cored grout samples varied between 1.86 and 1.92 g/cm^3, while porosity varied between 58 and 62%. This data correlates very well to the simulant grouts previously tested.

X-Ray Diffraction

A portion of one of the cores was sub-sections and submitted for XRD analysis. The results are described in Reference 2:

> *Phases common to each of the samples are mullite, quartz, and gypsum. Mullite and quartz are associated with the crystalline portion of fly ash. Although less common, gypsum can also be attributed to the fly ash. The occurrence of gypsum in fly ash is indicative of wet treatment or storage. In addition to these phases being common to all of the samples, they are also the most predominant crystalline phases. This corresponds well with the abundance of fly ash in the mix and the limited reactivity of the fly ash. Hydrotalcite, a hydration phase associated with slag, is only identified in the 3-2 XRD pattern. Hydrotalcite may be present in all of the samples; however, the peaks associated with this phase can be obscured by the peaks associated with gypsum. Sodium nitrate was identified in the XRD pattern for sample 3-1. The sodium nitrate is a component of the salt solution and could be expected in all of the samples. However, since the*

> *samples were prepared and analyzed with minimal handling, there may be sufficient pore water to preclude the sodium nitrate from crystallizing to an extent sufficient enough to be positively identified by XRD. The amorphous hump near 30° 2θ in the spectra is indicative of the presence of calcium silicate hydrate (CSH) gel.*

Thus the samples showed phases typical of hydrated cementitious materials consistent with what one would expect for the dry feed composition used in the production of Saltstone. No atypical crystalline structures were identified.

Optical Microscopy

A sample fragment was also mounted and polished for microscopic analysis. Figure 3 shows this sample at 100x magnification.

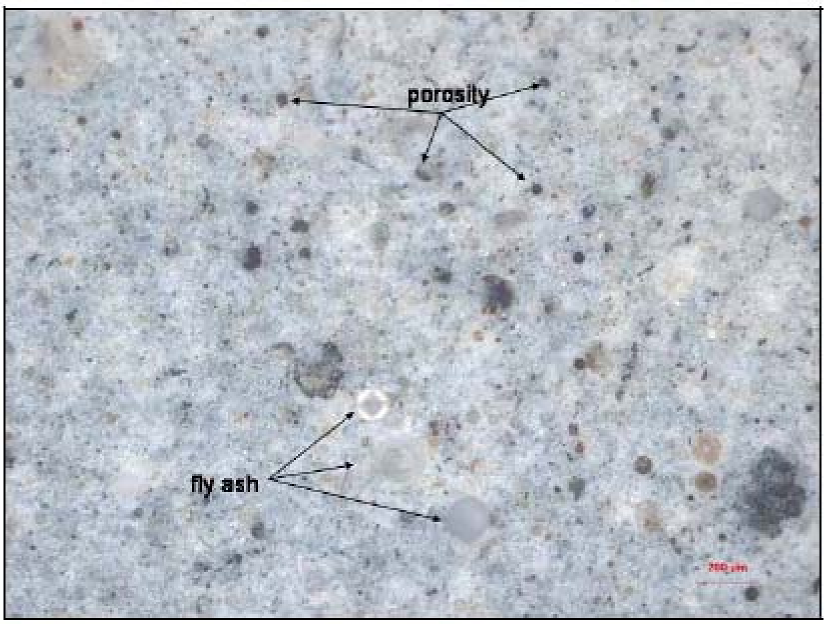


Fig. 3: Vault 4 core sample noting porosity and fly ash sites

This image is also consistent with microscopic observation done on simulant grout. One of the unknown items that this sample effort was intended to address was to determine the success with which the full-scale operating facility produced a waste form that was consistent with the product of lab-scale mixes that were used for the overwhelming majority of property measurements during flowsheet testing and performance assessment over the years. The initial characterization has provided added confidence that the simulant grouts generally resemble the actual emplaced and cured waste form.

FUTURE SAMPLING CONCEPTS

The sample evolution as previously discussed was successful in retrieving several grout cores for analysis at SRNL. However, the actual performance of the work was quite difficult and could be completed only through the intense effort and co-operation of several different work groups. The sampling effort ultimately involved representatives from Engineering, Operations, Maintenance, Industrial Hygiene, SRNL, and Radiological Control (Health Physics). The physical work was taxing, conducted in a radiation field, under significant heat stress. Thus it was immediately recognized that alternative methods for sample retrieval should be considered if there is a continuing need to monitor waste form properties.

Formed Core Samplers

One of the challenges that the core sampling effort did not address was the difficulty of obtaining samples at several different depths to quantify the amount of sample variability over different days of processing. The design of the core sampler lent itself to near-surface sampling and a much more laborious effort would have been required to take samples below the first few feet of the existing grout level.

Formed core sampling is based on the concept of a pipe-within-a-pipe design. Individual sample carriers would be preloaded into sample pipes and then placed in a disposal cell prior to the introduction of radioactive grout. The sample carrier and sample pipe would have matched inlet and vent holes and would fill as the grout level increased in the disposal cell. This concept offers significant advantages in providing for variable depth sampling as well as greatly reducing the physical work required for sample retrieval.

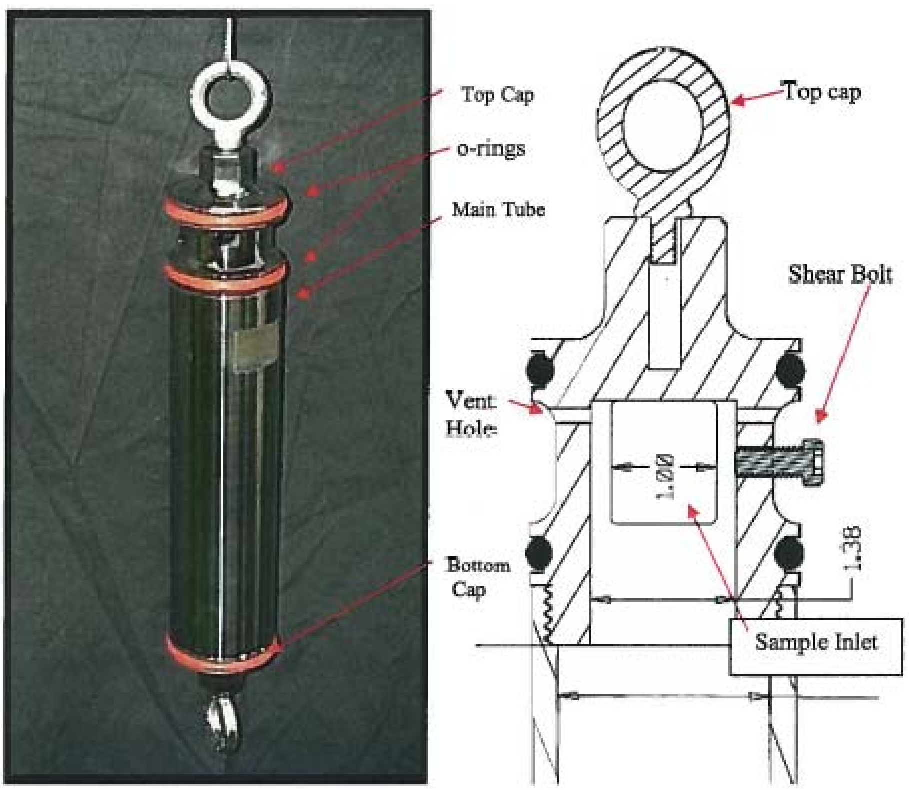


Fig 4: An early conceptual design of a formed core sampler prior to testing.

A scaled mockup test was performed with simulant grout that filled formed core samplers at two different fill rates expected to bound the conditions seen in the disposal vault [4]. Both samplers were filled with minimal void space, and the sample carrier extraction forces were well within ranges that can be applied at the actual disposal location. The test indicated some additional development work was necessary to ensure good sample extraction from the carrier, but in general it is expected that a design of this type is fundamentally sounds for long term use in core sample retrieval.

In Situ Monitoring

One of the long term objectives of analytical method development for Saltstone has been to correlate readily-quantified mechanical properties (e.g., elastic modulus) to more-difficult-to-measure properties that are important to waste performance (e.g., porosity, permeability). To this end, part of an on-going variability study has collected a significant amount of data in an attempt to define an empirical relationship between these properties [5]. The success of this effort offers two potential benefits: data collection is cheaper and faster for mechanical properties, and the potential exists for in-situ monitoring of the emplaced waste form.

All work in this area is at a conceptual level, but it may be possible to install an array of sensing devices at the time of disposal cell construction which would then be covered by grout as waste processing is performed. This is similar to the implementation of the formed core samplers, but an in-situ monitor would eliminate exposure to operations personnel and would create the potential for data collection at a resolution that is essentially un-obtainable with core sampling. Core sampling is a destructive technique with economic constraints due to the radiological exposure and operational difficulty of retrieving samples; thus at best the data obtained can give only snapshots of grout properties over a period of time. If in-situ monitoring turns out to be practicable, the opportunity will exist to understand with much greater detail both how the properties of the Saltstone change throughout the progress of early hydration and how the product degrades over time.

CONCLUSIONS

Core samples of radioactive grout have been obtained from an active Saltstone disposal cell. The difficulty of retrieving the core samples has led to several technology development efforts which will reduce the physical work needed to obtain samples, or which could potentially eliminate the need for destructive sample removal in the future.

Initial characterization has shown that the emplaced saltstone is consistent with grouts prepared using simulants in the laboratory. Physical characterization and microscopic examination of the waste form has shown the density and porosity to be within expected ranges. Additionally the structure and crystalline phases present demonstrate that cementitious hydration products have formed in a manner similar to simulant grouts. Although the initial characterization is promising, additional data from permeability and leach testing is still pending. This information will be needed before a full judgment can be made comparing the laboratory and field emplaced grout.

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