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Solidification of Simulated Liquid Effluents Originating from Sodium-Bearing Waste at the Idaho Nuclear Technology and Engineering Center, FY-03 Report

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ABSTRACT

In this report, the mechanism and methods of fixation of acidic waste effluents in grout form are explored. From the variations in the pH as a function of total solids addition to acidic waste effluent solutions, the stages of gellation, liquefaction, slurry formation and grout development are quantitatively revealed. Experimental results indicate the completion of these reaction steps to be significant for elimination of bleed liquid and for setting of the grout to a dimensionally stable and hardened solid within a reasonable period of about twenty eight days that is often observed in the cement and concrete industry. The reactions also suggest increases in the waste loading in the direction of decreasing acid molarity. Consequently, 1.0 molar SBW-180 waste is contained in higher quantity than the 2.8 molar SBW-189, given the same grout formulation for both effluents. The variations in the formulations involving components of slag, cement, waste and neutralizing agent are represented in the form of a ternary formulation map. The map in turn graphically reveals the relations among the various formulations and grout properties, and is useful in predicting the potential directions of waste loading in grouts with suitable properties such as slurry viscosity, Vicat hardness, and mechanical strength. A uniform formulation for the fixation of both SBW-180 and SBW-189 has emerged from the development of the formulation map. The boundaries for the processing regime on this map are 100 wt% cement to 50 wt% cement / 50 wt% slag, with waste loadings ranging from 55 wt% to 68 wt%. Within these compositional bounds all the three waste streams SBW-180, SBW-189 and Scrub solution are amenable to solidification. A large cost advantage is envisaged to stem from savings in labor, processing time, and processing methodology by adopting a uniform formulation concept for fixation of compositionally diverse waste streams. The experimental efforts contained in this report constitute the first attempt at developing a uniform methodology.

EXECUTIVE SUMMARY

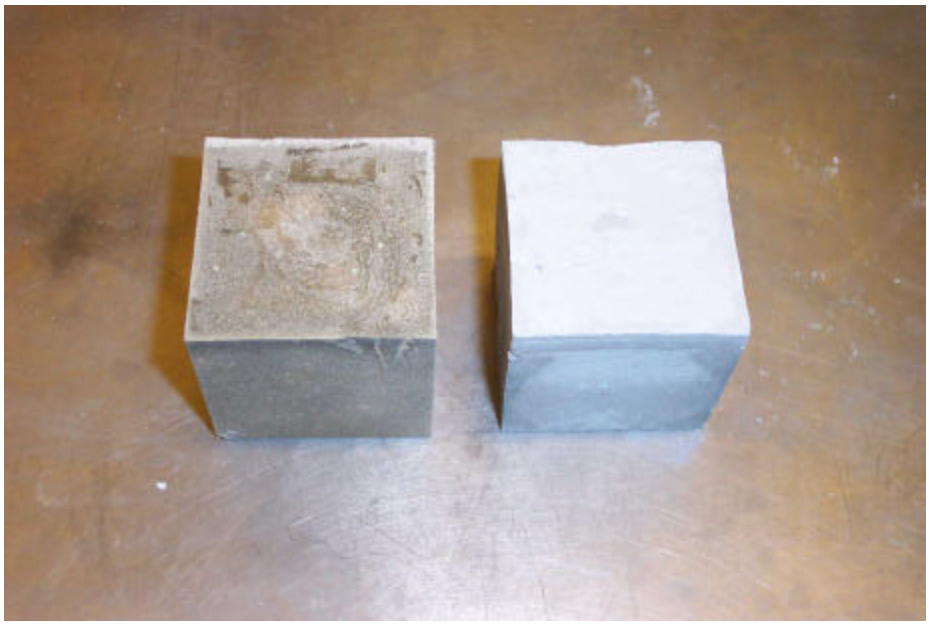
The sodium bearing waste (SBW) is an acidic liquid solution of several nitrate components. In view of its acidic nature, one of the aims of the Idaho Completion Project is to have it removed from the storage tanks at Idaho Nuclear Technology and Engineering Center (INTEC), and have it transformed into a disposable solid. Two types of disposable solids are under consideration: grout and silica gel. Commercially available Portland cement, slag and silica gel have been used in the present project for solidifying the three types of waste streams SBW-180, SBW-189 and NWCF Scrub solutions. A large part of this report concentrates on the investigation of the waste fixation in a grout matrix, while preliminary results of waste adsorption onto silica gel are also presented. Neutralizing the waste solutions to a weaker acid is considered important from the point of inhibiting the corrosive effects on cesium ion exchange silico-titanate resins, and for enhancing the retention of waste components in the grout and silica gel matrix. The silico-titanate resins are currently under consideration for absorption of the fission product cesium, so that the effluents contain no more than trace amounts (parts per million) of this element. The resulting waste streams are classified as low level wastes with low radioactivity, and their fixation in the grout must meet two properties 1. the grout must be devoid of any bleed liquid and 2. the grout must cure to a mechanically stable solid within a reasonable period of twenty eight days. While the first property is essential for qualifying the grout for disposal in the Waste Isolation Pilot Plant (WIPP), the second property is expected to facilitate handling and transportation.

Minimizing the quantity of grout for transportation to WIPP is also an important aspect under consideration for cost effective disposal. This relates to the quantity of waste loading in grout. All these criteria are related to the proportions of the neutralizing agent, the waste stream, Portland cement and slag and the methods by which these components are mixed to form the final grout. The experimental methodology of forming grout is further compounded by the chemical complexity of the waste streams and particularly the presence of RCRA metals. Thus, safeguarding the INTEC laboratory environment from hazardous metals contamination and minimizing the secondary waste production is also an important aim, and hence the experimental methodology is here evolved to progressively shift from an empirical to a more basic science approach. The experimental results are gathered to successively lead from (1) ad-hoc mixing, to (2) empirical matrix testing and to (3) systematic mapping of the grout components. In the third experimental approach, waste neutralization, and component proportioning bear a predictable relation to grout properties, and upon complete development, the method will potentially eliminate the uncertainties involved in relating grout types to properties.

A quaternary system is developed to sketch the relative proportions of the four components the neutralizing agent, cement, slag and waste. They are related simultaneously to the properties of grout formation, involving slurry pH, slurry viscosity, waste loading, the grout curing hardness (Vicat value) and mechanical strength of the cured monolith. The experiments thus far conducted have led to the development of formulation diagrams, which delineate the tolerable compositional ranges for forming grouts with marginal variations in waste loading. These formulation diagrams are in turn useful in predicting the desired compositional directions for optimizing the waste loading in association with desirable grout properties, namely the flow of grout into a container and its setting into a hardened monolith within a reasonable curing period not exceeding twenty eight days. The formulation diagram indicates the potential for developing a unified singular process for treating a variety of waste streams ranging in acidic strength. It is a ternary surface, which considers slag, cement and waste as the three end members at constant neutralizing concentration levels. In this methodology, the same compositional region is made to apply to all the waste streams and the waste loading is allowed to vary according to the changes in waste stream acidity. For example, it is noted

that given the same relative ratios of neutralizer-calcium hydroxide, slag and cement, the waste loading of the more acidic SBW-189 is about 3 to 5 weight% lower than the SBW-180. For these wastes the diagram also predicts the feasible waste loadings to occur within the range of 60 to 70 weight%, without any degrading effects, like anomalous shrinkage, separation of bleed water, or large scale surface crystallization.

A quantitative relation has been developed showing the dependence of pH on total solids addition to the waste stream. From this sketch the total solids required for completion of gellation, liquefaction, slurry formation and grout formation become apparent, leading to quantitative stabilization and prediction of waste in the grout matrix. With the completion of the above reaction steps, the subsequent release of bleed water from the grout during the curing stage is eliminated, for absence of liquid condensate is an important criterion in the waste form disposal. It is envisaged that a unified process will lead to cost effective optimization of the processing time, processing labor, equipment operation, waste loading and the quantity of the actual 55 gallon containers, while maintaining compliance of the waste form property with the waste acceptance criteria for disposal in WIPP (waste isolation pilot plant, New Mexico).



(A) (B)

Figures A and B. Examples of SBW-189 grout (A) and SBW-180 grout (B). Waste loadings are 68 wt% SBW-189, and 60 wt% SBW-180. Mechanical strengths are 1190 psi and 1500 psi respectively. The brown color in (A) is due to a higher slag content in the formulation. Both grouts have cured and register zero on the Vicat scale of penetrability.

The experimental objectives may be summarized as:

1. The development of a formulation map, indicating dependence of properties on grout and waste compositions, which in turn can be used to predict waste loading as a function of composition and properties.
2.The translation of composition parameters and properties obtained from small scale bench top experiments to an actual mixture in five and fifty five gallon drums.

3. The evaluation of the methodology to treat different SBW waste stream types by a unified process, leading to potential cost savings in process design, waste form qualification and equipment hardware.
4. The extension of the methodology developed from utilizing simplified waste simulants (Tables 2 and 3) to the full simulants that contain RCRA hazardous elements of mercury, nickel, cadmium, chromium and lead (Table 1).

The table below summarizes the formulations recommended for further evaluation. The formulations show that a range of options are available and will need to be "tailored" for the process, mixer, and equipment selected.

Waste Steam	Mixing Method	Loading wt%	50% NaOH wt%	$Ca(OH)_2$ Wt%	Slag wt%	Portland Cement wt%	Silica Gel wt%	Viscosity cP	Density g/cm^3
SBW-180	1	72	0	5	0	23		38200	1.42
SBW-180	2	75	0	8.5	0	16.5		20500	1.43
SBW-180	3	72	3	7	6	12			1.47
SBW-180	4	65	6.6				28.4		1.10
SBW-189	1	68	0	5	0	27		27500	1.61
SBW-189	2	68	0	12.4	0	19.6		28900	1.51
SBW-189	3	66	9	7	6	12			1.53
SBW-189	4	57.1	12.9				30.0		1.28
Scrub	2	35	18	1	41.3	4.7		12000	1.70
Scrub	3	54.4	15.6				30.0		1.21

Mixing Methods: 1 – combine calcium hydroxide and cement and mix in one step
2 – neutralize with calcium hydroxide and then mix in cement and slag
3 – pre-neutralize with sodium hydroxide and mix remaining powders
4 – neutralize with sodium hydroxide and mix with silica gel

Summary of Conclusions and Recommendations:

- SBW-180 can be grouted at 72 wt% to 75 wt%
- SBW-180 can be absorbed on silica gel at 65 wt% following partial neutralization
- SBW-189 can be grouted at 66 wt% to 68 wt%
- SBW-189 can be absorbed on silica gel at 57 wt% following partial neutralization
- Calcine Scrub can be grouted at 35 wt%
- Calcine Scrub can be absorbed on silica gel at 54 wt% after partial neutralization
- The grout formulations were satisfactory in the pilot scale continuous mixer
- Partial neutralization prior to ion exchange followed by grouting is possible
- Vibration and thermal cycling can cause free liquid in near saturated silica gel samples; thus, waste loading must be 5% to 10% lower and allow excess silica gel.
- Need to verify formulations "work" with full simulant including RCRA metals
- Need to investigate drum corrosion for the selected formulations

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A. Grout Test Data

B. Silica Gel Test Data

C. Absorption of NWCF Scrubber Solution on Calcine

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SOLIDIFICATION OF SIMULATED LIQUID EFFLUENTS ORIGINATING FROM SODIUM BEARING WASTE AT THE IDAHO NUCLEAR TECHNOLOGY AND ENGINEERING CENTER FY-03 REPORT

1. INTRODUCTION

1.1 Purpose and Scope

The sodium bearing waste (SBW) at the Idaho Technology and Engineering Center (INTEC) has originated from the second and third cycles of spent nuclear fuel reprocessing, waste calcination and decontamination activities [1,2]. It is an acidic liquid that is currently stored in the tanks at INTEC. Transformation of this waste into a disposable solid is among the important tasks of the Idaho Completion Project (ICP). The proposed plan is to evolve treatment options to process SBW liquid and transform the resulting secondary effluents into disposable low level waste solids. The SBW liquid treatment alternatives under consideration are processes of 1. cesium ion exchange (CsIX), 2. calcination, 3. direct evaporation, and 4. steam reforming. The liquid effluents originating from these treatment alternatives are expected to differ from one another and hence three solidification options of evaporation, grouting and absorption are being evaluated. At present data on liquid waste streams arising from the first three processes are shown in Tables 1 to 3 for SBW-180 and SBW-189 effluents from CsIX, scrubber solution from calcination, and concentrated acid from direct evaporation. The waste streams resulting from steam reforming tests would be added to the aforementioned list as they become available.

Two solidification options involving fixation in a grout matrix and/or absorption on silica gel were evaluated through a series of chronological experiments in accordance with the proposed test plans [3] for compositionally diverse waste simulants shown in Table 1. As the table indicates, the waste simulants are essentially nitrate solutions of several cations. They differ significantly in their acid molarity, which is on the order of 1 molar for SBW-180, 2.86 molar for SBW-189, 1.97 molar for calcination scrubber solution (NWCF Scrub), and 12 molar for evaporator bottom acid (LET&D Acid). In view of these large variations in acid molarity, it became imperative to conduct a series of small-scale experiments to evaluate fixation of the waste streams as a function of acid neutralization. The waste loading is expected to differ in the two matrices of silica gel and grout, as a function of the extent of neutralization from weakly acidic to strongly alkaline. The mechanism of fixation in the silica gel and grout matrices are different. In the former matrix physico-chemical adsorption is predominated by dehydration caused by exchange of OH^- in silica gel for positive ions in the waste, whereas the waste fixation in grout will largely occur by hydration reactions. The effects of cation valency and size on fixation mechanisms is expected to in turn influence the efficiency of waste retention in these matrices as a function of neutralization. In this regard effective retention of RCRA metals, cadmium, chromium, lead, nickel and mercury, and radioactive cesium is important, so that the waste forms comply with the waste acceptance criteria for disposal in WIPP [4,5]. Although, these elements are present in minor to trace abundance in the wastes of Table 1, the potential for hazard emerges from their rejection by the matrix due to higher valence and large ionic size, causing them to concentrate at the waste form surfaces as residual liquid (bleed water) or fibrous crystallites. Their concentration in the residual phases may be more imminent under higher waste loading, which is one of the factors for cost effective disposal. Thus the overall intent of the small-scale experimental methods undertaken in this project, has been to contribute towards optimization of the processing time, processing labor, equipment operation, waste loading and the quantity of the actual 55 gallon containers, while maintaining compliance of the waste form property with

Table 1. Projected Waste Stream Concentrations

Species	SBW-180 Simulant (*M*) (Ref. 11)	SBW-189 Simulant (*M*) (Ref. 12)	NWCF Scrub Simulant (*M*) (Ref. 8)	LET&D Acid Simulant (*M*) (Ref. 13)
Acid (H^+)	1.01E+0	2.86E+0	2.33E+0	1.20E+1
Aluminum	6.63E-1	7.11E-1	1.56E+0	5.60E-2
Arsenic [1,2]	4.99E-4	0	9.03E-5	
Barium [1,2]	5.58E-5	5.62E-5	2.42E-5	
Boron	1.23E-2	2.12E-2	5.42E-3	
Cadmium [2]	7.54E-4	3.91E-3	9.68E-4	
Calcium	4.72E-2	7.30E-2	5.04E-2	1.00E-3
Cesium [1]	7.73E-6	2.68E-5	8.38E-6	
Chlorine	3.00E-2	2.06E-2	3.81E-2	5.00E-2
Chromium [2]	3.35E-3	5.64E-3	1.73E-3	2.00E-3
Copper	6.97E-4	9.54E-4	1.56E-4	
Fluorine	4.74E-2	1.38E-2	8.88E-2	7.00E-3
Gadolinium [1]	1.77E-4	1.35E-4	3.42E-5	
Iron	2.17E-2	2.68E-2	1.22E-2	3.00E-4
Lead [2]	1.31E-3	1.16E-3	3.48E-4	
Lithium [1]	3.39E-4	3.84E-4	8.11E-5	
Magnesium	1.20E-2	2.21E-2	4.32E-3	
Manganese	1.41E-2	1.95E-2	4.28E-3	
Mercury [2]	2.02E-3	6.50E-3	2.10E-1	3.500E-3
Molybdenum [1]	1.93E-4	2.80E-4	4.15E-4	
Nickel [2]	1.47E-3	2.32E-3	7.28E-4	2.00E-3
Nitrate	5.01E+0	6.52E+0	8.24E+0	1.21E+1
Palladium [1]	2.35E-5	0	3.58E-6	
Phosphate	1.37E-2	2.07E-3	3.10E-2	
Potassium	1.96E-1	2.25E-1	7.90E-2	
Ruthenium [1]	1.25E-4	1.72E-4	3.26E-5	
Selenium [1,2]	1.46E-4	0	2.16E-5	
Silicon	3.02E-7	3.08E-4	3.68E-2	
Silver [1,2]	5.29E-6	0	4.12E-5	
Sodium	2.06E+0	2.04E+0	6.03E-1	2.40E-2
Strontium [1]	1.19E-4	1.41E-4	2.65E-5	
Sulfate	5.40E-2	1.07E-1	2.41E-2	1.00E-3
Vanadium [1,2]	9.23E-4	2.51E-5	7.78E-6	
Zinc [2]	1.05E-3	1.07E-3	2.47E-4	
Zirconium	6.33E-5	3.57E-4	1.86E-2	

[1] Species left out due to small amount.

[2] Species left out to render simulant RCRA non-hazardous.

Table 2. Simple Non-Hazardous Simulants

Species	SBW-180 (M)	SBW-189 (M)	NWCF Scrub (M)	LET&D Acid (M)
Al	6.63E-1	7.11E-1	1.56E+0	5.60E-2
B	1.23E-2	2.12E-2	5.42E-3	
Ca	4.72E-2	7.30E-2	5.04E-2	1.00E-3
Cl	3.00-E2	2.06E-2	3.81E-2	5.00E-2
Cu	6.97E-4	9.54E-4	1.56E-4	
F	4.74E-2	1.38E-2	8.88E-2	7.00E-3
Fe	2.17E-2	2.68E-2	1.22E-2	3.00E-4
H	1.01E+0	2.86E+0	2.33E+0	1.20E+1
K	1.96E-1	2.25E-1	7.90E-2	
Mg		2.21E-2	4.32E-3	
Mn		1.95E-2	4.28E-3	
Na	2.06E+0	2.04E+0	6.03E-1	2.40E-2
NO_3	5.01E+0	6.52E+0	8.24E+0	1.21E+1
PO_4	1.37E-2	2.07E-3	3.10E-2	
Si		3.08E-4	3.68E-2	
SO_4	5.40E-2	1.07E-1	2.41E-2	1.00E-3
Zn	1.05E-3	1.07E-3	2.47E-4	
Zr	6.33E-5	3.57E-4	1.86E-2	

the waste acceptance criterion of no bleed water, for disposal in WIPP. The task specific methods in this report evolve from the results of the previous tests, with theintent to mitigate empiricism in experimental work instructions. The scope of these new experiments is to place the empirical methodology on a firmer scientific platform. This approach has the potential to safe guard the large scale engineering process of waste immobilization from costly errors. The experiments focus on the immobilization of waste effluents originating from CsIX exchange, calcination, and evaporation processes. The experimental work was conducted in accordance with "Liquid Low Level Waste Stabilization /Solidification Experimental Testing, "Independent Hazard Review package IHR # INTEC-00-17.

2. HISTORY OF PREVIOUS STUDIES

2.1 CsIX and Grouting

The cesium removal via ion exchange (CsIX) process was proposed in 1998 [1] for stripping SBW of intense radioactivity, so that the resulting liquid could be classified as a transuranic waste for solidification into grout or for absorption into silica gel [3,4]. In the course of these experimental tests, two general formulations have emerged. The first formulation provided 40 weight% (wt%) loading of SBW to the total mass of the grout waste form. The test involved neutralization of the acidic waste by liquid sodium hydroxide and its subsequent cementation in the Portland cement plus blast furnace slag mixture. In the second formulation it was possible to increase the waste loading to 70 wt% by neutralizing the acidic waste with calcium hydroxide powder, which was followed by solidification in the Portland cement plus blast furnace slag matrix. The second formulation was recommended for disposal at WIPP facility for the CsIX SBW. For WIPP the waste form must be a solid with less than 1% free liquid and the radiation level must be less than 200 millirem (mR) for contact handled waste [5]. Further the recent RCRA regulations do not allow occurrence of free liquids in hydrofluoric acid containing wastes that are RCRA coded as U134 [6].

Table 3. Non-Hazardous Sodium Bearing Waste Simulant Make-Up (1 L Batch)

Species	Stock Chemical	Molecular Weight	SBW-180	SBW-189	NWCF Scrub	LET&D Acid
Al	2.2M $Al(NO_3)_3$		301.50 ml	323.18 ml	709.09 ml	25.45 ml
B	H_3BO_3	61.83	0.76 g	1.31 g	0.34 g	
Ca	$Ca(NO_3)_2.4H_2O$	236.15	11.14 g	17.24 g	11.90 g	0.24 g
Cl	12.0 M HCl		2.50 ml	1.72 ml	3.18 ml	2.17 ml
Cu	$Cu(NO_3)_22.5H_2O$	232.59	0.16 g	0.22 g	0.04 g	
F	27.6 M HF		1.70 ml	0.40 ml		0.25 ml
Fe	$Fe(NO_3)_39H_2O$	404.00	8.78 g	10.83 g	4.93 g	0.12 g
H	All acids					
K	KNO_3	101.10	19.84 g	22.75 g	7.99 g	
Mg	$Mg(NO_3)_26H_2O$	256.41		5.67 g	1.11 g	
Mn	$Mn(NO_3)_2$ [50% soln]	178.95		6.98 g	1.53 g	
Na	$NaNO_3$	85.00	174.92 g	173.39 g	51.25 g	1.40 g
NO_3	15.8 M HNO_3		46.83 ml	160.87 ml	128.66 ml	755.76 ml
PO_4	14.8 M H_3PO_4		0.93 ml	0.14 ml	2.09 ml	
Si	10 g/L Si0 in 5% (0.8M) HNO_3	28.08		0.86 g	2.21 g	
SO_4	18.0 M H_2SO_4		3.00 ml	5.94 ml	1.34 ml	0.06 ml
Zn	$Zn(NO_3)_26H_2O$	297.47	0.31 g	0.32 g	0.07 g	
Zr	1.53M Zr in 5.4 ZrDP	91.22	0.04 ml	0.23 ml	12.16 ml	
Actual NO_3			5.15 M	7.26 M	7.60 M	12.20 M
Actual Acid			1.01 M	2.86 M	2.33 M	12.08 M
Density g/ml			1.26	1.34	1.32	1.28

2.2 Silica Gel Absorption

In another method of immobilization, SBW was absorbed onto silica gel, yielding about 70 to 75 wt% waste loading without additional treatment. With subsequent heating/drying however, the effective waste loading onto silica gel could be increased to as high as 89 wt%. Nevertheless, for process simplicity, direct absorption without heating was explored in these studies, and 70 wt% loaded silica gel waste forms (70 wt% SBW + 30 wt% silica gel) met the WIPP requirement of no free liquid. In the initial study a rotary mixing/drying kiln process was adopted to feed SBW onto silica gel. In the absence of mixing, SBW addition to silica gel appeared to form a surface crust, preventing further percolation of waste liquid into the powdery silica gel mass. Additional tests led to simplification of absorption by elimination of the rotary mixing, and the silica gel was sprinkled onto the surface of the static SBW liquid. It was allowed to percolate to the bottom of the vessel, which resulted in complete absorption of the liquid with continued sprinkling. The silica gel waste forms thus made appeared to meet the TRU WIPP waste criteria of no free liquid. However, doubts are cast in disposal of silica gel waste forms in other low-level waste disposal sites such as Hanford, Nevada, and Envirocare of Utah. Unlike the WIPP, the land disposal restrictions in these low-level disposal sites require that the waste forms pass the toxic metal leach resistance tests. Because of the physically absorptive nature of SBW onto silica gel, it may seem that the

resistance to toxic metals leaching will be negligible and hence silica gel was not considered as a suitable mixed low-level waste form for immobilizing waste streams containing toxic metals.

2.3 Calcination and Scrubber Solution

In case of calcination option, SBW [7,8] would be treated in the existing New Waste Calcination Facility (NWCF). Here the SBW liquid will be solidified in a heated and fluidized bed. The process requires an off gas scrubber system. In the past, the scrubber solution was recycled to the tank farm and the calciner, however, with the closure of the tank farm, the scrubber solution needs to be disposed of directly as remote handled TRU WIPP waste. A separate study will evaluate absorption of the scrubber solution on the calcine or silica gel and grouting.

2.4 Steam Reforming

Steam reforming is another type of calcination process that is currently under consideration for treating the sodium bearing waste streams. In this process, steam is used as the fluidizing agent rather than air. The steam and added reductants create reducing conditions for the calcination reactions. It is anticipated that a scrubber system similar to NWCF will be used; however, the flowsheet is not available at this time. Presumably, the scrub solution would be caustic and contain unburned carbon. A grout and silica gel formulation are needed for this new solution.

2.5 Direct Evaporation of SBW

In this option, the SBW is evaporated and concentrated about 3 to 4 times and the solution is allowed to solidify with cooling. An off-gas cleanup system is proposed to condense all the acid and water that evolves from the SBW. This condensate would be concentrated in an evaporator and the liquid effluent treatment and disposal facility (LET&D). The final effluent is a highly acidic solution to be recycled or grouted and disposed of as contact handled mixed low-level waste.

2.6 Formulation Matrix Test Development from Multi-Dimensional Statistical Design

On the basis of the results obtained from preliminary tests, a formulation matrix was generated adopting a multi-dimensional statistical approach [9-13] to develop grout and silica gel waste forms with desirable properties and optimum waste loading. The three properties of importance for grout waste forms are 1. absence of free liquid, 2. fluid viscosity under 30,000 cp, and 3. hardening and setting of the grout to Vicat value of zero within 28 days. For silica-gel waste form the first property of no free liquid and an un-agglomerated flow of the gelatinous powder following waste loading was considered essential. The approach predicted 35 grout and 12 silica gel formulation tests for each waste stream to arrive at a range of feasible waste forms as a function of waste loading, neutralization, and cement/slag proportions. These tests have produced results with large variations in the bleed liquid concentration, Vicat values, setting times and waste loading. One possible cause for the large changes in the properties from sample to sample is the non-interactive and independent proportions of the components SBW, neutralizer, cement and slag in each formulation. Consequently, large uncertainties are introduced in determining the tolerance limits for these component variables in processing waste forms with desirable properties. The tolerance range for each component must be known, since potential uncertainties could arise in engineering grout waste forms in the actual scale-up process.

3. TEST OBJECTIVES

The objective of laboratory bench scale solidification tests is to provide experimental data to assist in the overall project design and selection efforts. Specific grout data are needed such as wet grout viscosity, cure time, waste form density and volume, and the recommended formulation for optimum waste loading that meets the waste acceptance criteria. For silica gel, data needs are similar with waste density, volume, loading, gas generation, and heat of absorption. These data must be provided for both SBW-180 and SBW-189 compositions, NWCF scrubber solution, LET&D acid, and steam reform scrubber solution [7-13]. It is imperative that the properties measured in small 5 cubic centimeter cubes made on a bench scale, confidently support and benefit the actual scale-up process involving production of five and fifty five gallon sized drums from a mixer. Since, there is a large volume difference, in transferring the technology from a laboratory scale to a plant production scale, the objective of the test plan is to develop small cubes leading to systematic gradations in properties. The data base thus generated will in turn provide the valuable tolerance limits for the components waste, neutralizer, cement, slag and silica gel. The tolerance range in the relative proportions of these components must be known so that potential uncertainties can be accommodated in the actual scale-up process. In case of grout formation particularly, the properties are determined by the relative interactions among the four components waste, neutralizer, cement and slag. These interactions are not apparent in the multi-dimensional design [12]. Consequently, the results obtained by this method have not been useful in determining the tolerance limits for component variations and in selecting candidate waste formulations with predictable confidence levels. As an outgrowth of this experimental experience, the objective of this test plan is to approach waste form development by evaluating the influence of interdependence among the components on properties of waste loading, viscosity, Vicat [14] hardening, density, mechanical strength and thermal cycle stability.

The experimental efforts may be summarized as:

1. The development of a formulation map, indicating dependence of properties on grout and waste compositions, which in turn can be used to predict waste loading as a function of composition and properties.

2. The translation of composition parameters and properties obtained from small-scale bench top experiments to an actual mixture in five and fifty five gallon drums.

3. The extension of the formulation concept developed for grout waste forms to immobilize waste streams in silica gels.

4. The evaluation of the methodology to treat different SBW waste stream types by a unified process, leading to potential cost savings in process design, waste form qualification and equipment hardware.

5. The extension of the methodology developed from utilizing simplified waste simulants (Tables 2 and 3) to the full simulants that contain RCRA hazardous elements of mercury, nickel, cadmium, chromium and lead (Table 1).

4. DISCUSSION OF INVESTIGATIVE TEST RESULTS FOR GROUT FORMATION

Ad-hoc tests were developed to resolve problems that were encountered in experiments planned under the previous test plan in sections 2.1 to 2.6. The problems seemed to arise from variations in waste

composition, waste neutralization, grout compositions and waste adsorption by silica gel particles. Accordingly new tests were conducted on an as needed basis. The results of these ad-hoc tests provide useful steps for the present experimental plan and are here discussed to support the objectives 3.1 to 3.5.

4.1 Stages of Grout Formation

The process of developing a grout waste form can be envisaged from previous experimental results as being made of five essential steps: 1. neutralization, 2. gellation, 3. liquefaction, 4. hydration and 5.solidification. These steps are interrelated and cumulatively influence the relevant properties of waste loading, fluid viscosity, curing time and mechanical strength. The SBW effluents are highly acidic and record zero or negative values on the pH meter. They need to be neutralized using appropriate reagents for cementitious grout formation occurs by hydration under basic (caustic) conditions with pH in the neighborhood of 10. Neutralization to a partial value of pH equaling 1 or 2 is also necessitated to avoid silico-titanate resin degradation, particularly in cesium ion exchange experiments.

4.2 Usage and Influence of Neutralizing Agents

Measured quantities of neutralizing reagents are added to increase the pH of the waste solution. The relative proportions of reagent to waste solution are predetermined to obtain the desired pH value. So far three reagents have been used to assess their relative influence on grouting properties. For the model experiment, the results of which are shown in Table 4, the total mass was 100 grams for each waste batch. The influence of reagents 50%NaOH (pH=12.5), NH_4OH (pH=13), and $Ca(OH)_2$ powder on the three types of waste streams SBW-180, SBW-189 and NWCF scrubber solution (NWCF Scrub) are shown in this table. For comparison the table also shows the changes imparted to the waste solution with cement addition. The mass proportions, pH values and temperatures (Table 4) were recorded at the gellation point (see also Table 5 for a volumetric basis). There is a short residence time needed for gellation and this is on the order of 7 minutes with continued stirring and addition of reagents. At the end of 7 minutes, gellation is rapid and a thick solid is formed with a sharp increase in the resistance to stirring. However, with continued stirring effort the solid mass liquefies. The resulting slurry now has a viscosity of about 3000 centipoise (cp) and also becomes more basic without further addition of neutralizing agents. The pH for the slurry is on the order of 10, and several tests have indicated a suitable pH range of 9.5 to 10.5 for curing of the cementitious mass into a hardened monolith within 28 days. It has also been noted that less basic cubes with pH on the order of 8.5 that are formed only by addition of ordinary Portland cement (OPC) do not readily cure to a hardened mass, clearly indicating the need for a neutralizing agent. Following liquefaction, the viscosity of the neutralized slurry is increased to about 25000 cp by further addition of cement and the pH remains buffered. The thick slurry is readily poured into plastic molds for curing, which is allowed to occur for a period of 28days in ambient atmosphere.

Table 4. Mass Proportions, pH and Temperature (^{0}C) for Neutralization and Gellation of Waste Streams SBW-180, SBW-189 and NWCF Scrub

	SBW-180	SBW-189	NWCF Scrub
50%NaOH	18g/100g waste pH=8.57, T=46.5C Gellation Time = 5 to 10 minutes	27g/100g waste pH=9.60, T=49C Gellation Time = 5 to 10 minutes	28g/100g waste pH=8.30 T=53C Gellation Time = 5 to 10 minutes
NH_4OH	14g/100g waste pH= 7, T=30C Gellation Time = 5 to 10 minutes	22g/100g waste pH=8.46, T=32C Gellation Time = 5 to 10 minutes	27g/100g waste pH=6.20 T=40C Gellation Time = 5 to 10 minutes
$Ca(OH)_2$	15g/100g waste pH=7, T=34C Gellation Time = 5 to 10 minutes	17g/100g waste pH=8.36, T=40C Gellation Time = 5 to 10 minutes	19g/100g waste pH=7.10 T=43.8C Gellation Time = 5 to 10 minutes
Cement	28g/100g waste, pH=4.89, T=38.4C Gellation Time = 5 to 10 minutes	28g/100g waste, pH=3.51, T=42C Gellation Time = 5 to 10 minutes	28g/100g waste pH= 3.3 T=46C Gellation Time = 5 to 10 minutes

Table 5. Titration Results for 100 mL of Simulant

50% Sodium Hydroxide mL	SBW-180 Simulant pH	SBW-189 Simulant pH	Calciner Scrub Simulant pH	Comments
0	0.08	0.14	-0.86	
5	0.57	0.16	-0.56	SBW-180 develops precipitates
10	2.82	0.17	-0.40	Scrub develops precipitates
15	3.80	0.41	1.37	SBW-180 gelled SBW-189 develops precipitates
20	11.75	2.27	2.05	SBW-180 thins to slush
25	13.03	2.82	2.42	
30		4.99	2.72	SBW-189 gelled
35		11.68	3.12	SBW-189 thins to slush Scrub gelled
40		12.99	5.50	
45			11.02	Scrub thins to slush
50			12.02	

4.3 Relations Among Grout Composition, Viscosity, and Curing

4.3.1 Neutralization by Calcium Hydroxide and Formulation Mapping

The preliminary empirical test results, as shown in Table 6, reveal a complex dependence of viscosity and curing on grout composition. In order to arrive at a clearer consensus among these variables, the data are here recast in the form of a composition map, Figures 1 and 2. In Figure 1 the four variables of waste loading (SBW-189), neutralizer ($Ca(OH)_2$), cement and slag are represented on a ternary diagram by maintaining the waste loading constant. The three independent variables are normalized to 100 wt%. The composition points experimentally tested, thus far on this diagram, point to two potential regions outlined as X and Y for processing SBW-189 waste. Outside of these boundaries in the region Z, there is less synergism noted among composition and the properties. Consequently, deviations in the results of this region are large, that inhibit selection of a central composition point to accommodate marginal errors. For example, the composition point A9 is bound by a very highly viscous grout A3 (50,000cp, Table 3) and an unsetting grout A1 (Vicat = 50, Table 6). A synergistic property of the two ends is observed in grout A9 for viscosity only, but it is not the case for Vicat. The latter value in A9 is the same as in A1, while the viscosity for A9 is on the order of 12800 cp. It is synergized between 50000 cp for A3 versus 8160 cp for A1.

Table 6. Formulations, Viscosity, and Vicat Numbers for SBW-189, SBW-180 and Scrub

Sample#	SBW-189 Wt%	SBW-180 Wt%	NWCF Scrub Wt%	NH_4OH Wt%	$Ca(OH)_2$	H_2O	Cement	Slag	Viscosity Centi-Poise	Vicat 10 day
A6	68				3		29	0	12880	0
A14	68				3.2		19.2	9.6	7100	10
A1	68				3		14.4	14.4	8160	50
A15	68				6.4		3.2	22.4	65760	0
A9	68				6.4		12.8	12.8	12800	50
A22	68				6.4		11.37	14.2	27000	10
A4	68				12.8		19		35360	0
A3	68				12.8		9.6	9.6	50000	0
A18	68				12.8			19.2	27000	7
C6		68			3		29.0		18000	5
C4		57.1			3.5	12.4	27.0		2240	0
C1		52.1			3.5	13.1	31.3		6620	0
C3		62			3	5	30		4320	0
C5		64			3	3	30		8800	50
C2		62			5.1	6.7	26.2		31000	0
C10		49.3			7.6	20.1	23		5760	0
C11		56			8	10	26		40000	0
NB15			40	14.4			45.6		26000	1
NB20			40	14.4				45.6	19600	5
NB21			40	14.4			13.68	31.92	8960	50
NB22			40	14.4			31.92	13.68	12000	1
NA7	70			22.4			7.6		6000	50
NB7			70	28			2		3000	50
NC7		70		16.1			13.9		7000	50
NA5	30			9.6			60.4		40000	0
NB5			30	12			58		40000	0
NC5		30		6.9			63.1		40000	0

The map also enables an experimentalist to formulate future combinations of components slag/cement/$Ca(OH)_2$ in relation to the existing ones, with predictable variations in the properties. The map so developed will eventually decrease the number of compositional points to be tested, leading to minimization of waste generation in the laboratory and elimination of uncertain sample production. Moreover, with sufficient data it would be possible to represent on the composition map the property of viscosity and curing as contours or lines of constant values. These contours in turn will bear a definite relation to composition, and thus enable prediction of grout behavior. The first set of samples A6 to A18 in Table 1 were developed by varying $Ca(OH)_2$/(Cement + Slag) ratio instead of fixing the $Ca(OH)_2$ content on the basis of amount required to neutralize the waste as shown in Table 4. By adopting the latter method, neutralizer ($Ca(OH)_2$) can be treated as a variable dependent on waste loading, which would then enable inclusion of waste loading and its influence on properties as one of the three variables on the formulation map.

4.3.2 Neutralization by Ammonium Hydroxide and Formulation Mapping

A second investigative test was conducted using NH_4OH as a neutralizer, because of the slow solution kinetics of $Ca(OH)_2$ in the waste solution. The step to use ammonia was undertaken mainly to evaluate the basicity needed to neutralize the waste, when a standard basic reagent is used. The reagent grade ammonia with a pH of 13 here forms the base case, relative to which the effects of other reagents are evaluated (Table 4). The results with ammonia as a neutralizer are shown in Figure 2 and Table 5. The proportion of ammonia to waste was determined from Table 4 to achieve the end point of gellation. Thus in this case, ammonia concentration is a waste dependent variable. The data of Table 5 have been shown in sets of NA, NB and NC that are followed by sample numbers. In this notation N stands for ammonia, A for SBW-189, B for Scrub and C for SBW-180. For brevity, all the sample points of Figure 2 are not shown in Table 6. The composition points may be viewed as progressing in waste loading towards the waste apex, paralleling the slag-cement join of the diagram. For example the waste loadings along the waste-cement join (where join is a line connecting or joining components or end members on the map) are on the order of 30 wt% for the NC5, NA5 and NB5 set, 40 wt% for NC6, NA6 and NB6 set, and 70 wt% for NC7, NA7 and NB7 set. Similarly other sets are shown along the joins Waste - 70cement/30slag or waste - 30cement/70 slag or waste - 100%slag. A general trend has been noted for all the waste grouts, such that below the median join of NB20-NB15 of 40 wt% waste loading, the viscosity increases and the curing is relatively rapid. Proceeding towards the waste apex above the 40 wt% join, there is a remarkable decrease in viscosity as would be expected from higher waste loading and also there are large variations in the kinetic impediment for curing as a function of waste type, pointing to the need of improving the formulation to accomplish desirable properties. Thus the 40 wt% parallel represents an optimum compositional region about which both viscosity and grout setting fall in the desired range and point to an universal grout preparation methodology that is applicable to all the three types of wastes. An universal methodology has the potential of cost savings in labor, production time and equipment hardware, and may open a venue to offset the relative production cost increase arising from decrease in waste loading to 40 wt%.

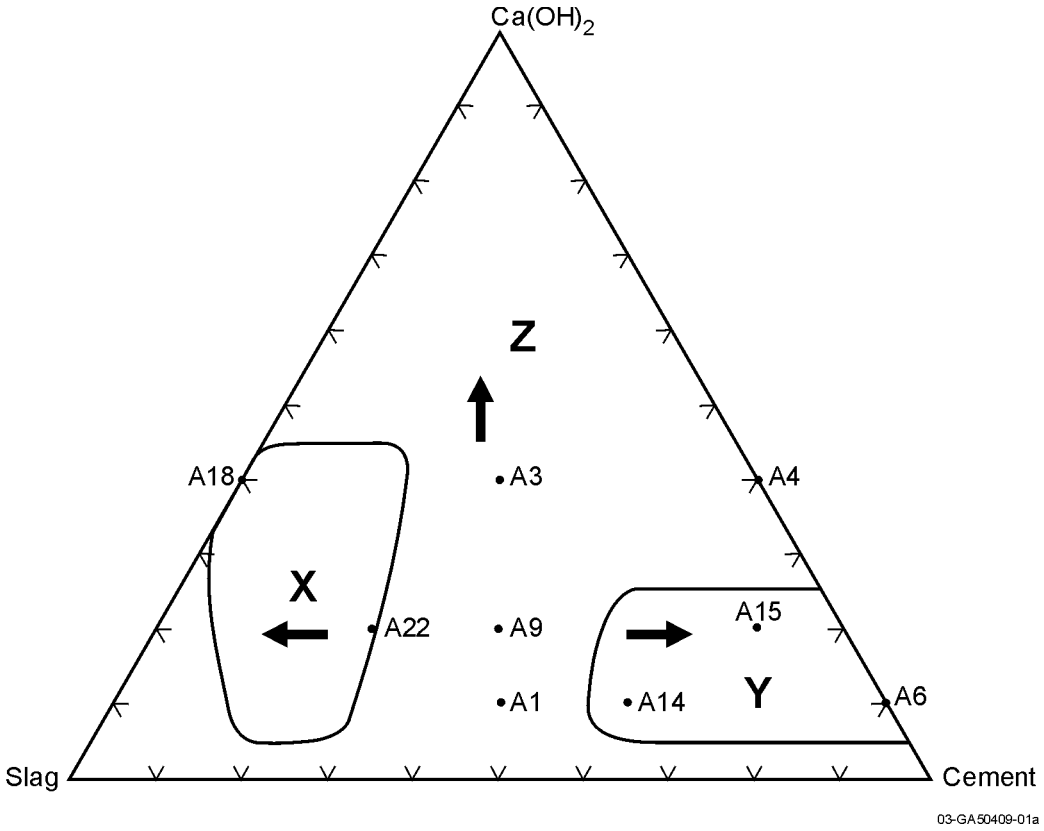

Figure 1. Grout Formulation Diagram at a constant loading of 68 wt% for sodium bearing waste SBW-189. X = compositional region for forming slag enriched grout, Y =compositional region for forming cement enriched grout. Composition points in region Z form grouts that are only partially set within 28days of curing time. Component proportions are in normalized weight% (cement+$Ca(OH)_2$+slag =100 wt%). Arrows point to compositional directions of increasing viscosity.

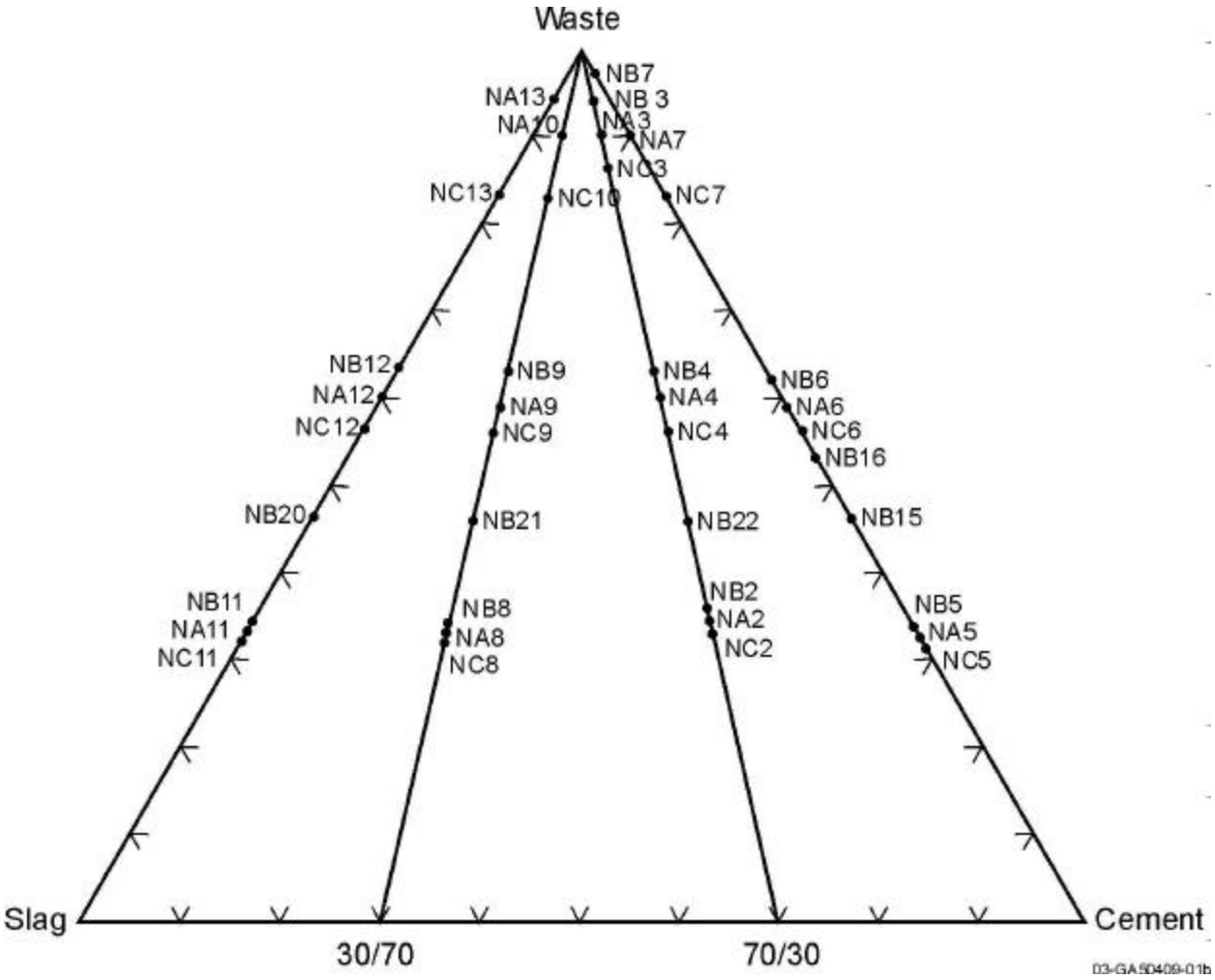

Figure 2. Grout formulation diagram with waste, slag and cement as independent variables. In this formulation ammonium hydroxide is the neutralizing agent whose concentration is dependent on waste concentration and waste type, causing the composition points to occur separated from one another for the same waste loading. Waste loadings vary from 30% for NC5 to 70 wt% for NC7 along cement-waste join. Identical waste loading variations compose the composition points along other joins - 70wt%cement/30wt%slag-waste; 30wt%cement/70wt%slag-waste; and 100wt%slag-waste. The waste types are: NC=SBW-180, NB=SrubRev.1, and NA=SBW-189, with corresponding sample numbers.

4.3.3 Neutralization by Alkaline Earth Oxides CaO and MgO

In previous experiments, attempts to neutralize the waste by calcium oxide addition have resulted in the degradation of CaO into $CaCO_3$ or $Ca(OH)_2$. For this reason an alternative alkaline earth oxide MgO was proposed (Kirkham, communication) and shall be tried in the near future to explore potential advantages, stemming from its relatively higher cationic potential and stability.

4.3.4 Neutralization by Sodium Hydroxide

A similar experimental exercise in the third investigative test with 50%NaOH in lieu of NH_4OH as a neutralizing agent (Table 4) did not yield results of matching quality. The grout waste forms made from NaOH neutralized waste streams showed swelling and surface crystallization that impeded curing to a hardened cube within 28 days. Further, the relatively high ionicity that is characteristic of NaOH, raised the temperature of the waste bath above 50^oC, where as this temperature remained in the 30 to 40C range with ammonia neutralization (Table 4). However, it is possible that the usage of ammonia would be restricted in an actual scale up process, due to its high vapor pressure and odor. Therefore, additional tests must be conducted to arrive at optimum concentration of NaOH needed to achieve differences in the neutralization levels. At present the plan is to pre-neutralize SBW to 0.5 Molar acid, to avoid degradation of sorbent. This partial neutralization of SBW will supposedly be carried out prior to solids filtering and cesium ion exchange. The resulting streams are expected to be different from the waste effluents 180, 189 or Scrub, requiring revision of grout formulations to match the projected changes in the chemical flow sheets.

4.3.5 Homogenization by Water Addition

In the fourth investigative test, water was used to decrease gellation thickening by $Ca(OH)_2$, and the corresponding bath temperature. Water is a coolant and also a weak neutralizer. Its addition raises the pH of the acidic waste from zero to 0.5 while the temperature remains ambient. The waste under study is SBW-180 denoted by symbol C in Table 6. While the waste loading is decreased from 68 wt% in C6 to 57 wt% in C4, at the same neutralizing concentration of 3 wt%, the changes in Vicat and viscosity are noted with variations in water content. C1 for example, has a low viscosity of 2240 cp and also has hardened to zero on Vicat within ten days, whereas C6 with higher waste loading and no water, recorded a higher viscosity of 18000 cp and also held a value of 5 in Vicat test. These two results may suggest an approach for improving the properties by decreasing the waste loading and concurrently balancing the relative mass proportions with addition of water. Formulations C1 and C3 with water contents of 13 and 5 wt% record a low viscosity of 6620 and 4320 cp and cure to a hardened mass of zero Vicat value within ten days. The failure of formulation C5 in Vicat value (50) is at present not clear. Formulations C2 and C11 record a higher viscosity of 31000 and 40000 cp because of higher $Ca(OH)_2$ concentration and would potentially inhibit the flow of grout into the container. In formulation C10 the higher neutralizing content is offset by higher water content of 20 wt%, causing the viscosity to drop to 5760 cp. The four formulations C1, C3, C4 and C10 (Table 6) cure to Vicat value of zero within ten days. The results suggest possible advantages stemming from water addition due to its cooling and homogenizing effects with nominal decrease in waste loading. The tests leading to optimization between waste loading and property would eventually be judged from the waste acceptance criteria for WIPP [5]. Thus in this preliminary stage water is considered an additional homogenizing and neutralizing agent, in the event the aforementioned neutralization methods also necessitate decreases in waste loading to meet the waste acceptance criteria for WIPP.

4.4 The Choice of Components and Mixing Sequence

Since grout formation is to occur on the scale of fifty-five gallon drums in an actual plant production setting, it is necessary to maintain a simple mixing process with fewer mixing steps, so that the cumulative errors arising from mixing are minimized. On the basis of investigative results the following mixing schemes are suggested:

4.4.1. Among the neutralizers investigated, calcium hydroxide is more effective. It is odorless, it limits the rapid rise in the temperature of the acidic waste to a lower value (40C), its neutralization effect is comparable to the standard ammonium hydroxide, and produces a noticeable gellation at the neutralization point (pH=7). As it is introduced to the waste in a powder form, there is a short residence time required during which continued stirring and gradual addition of the powder to the acid waste leads to gellation. In some of the previous studies the sequence of mixing involved addition of calcium hydroxide which was then followed by addition of cement. Some of the grouts thus formed were noted to bleed water during curing, possibly due to sluggish solubility of calcium hydroxide and inhomogeneous mixing with cement addition. The results were improved with no bleed water forming, by blending calcium hydroxide and cement powders prior to their introduction into the acid solution. This step is followed by gradual feeding of the blended powder into the waste solution, allowing residence time for mixing by continued stirring. The proportion of calcium hydroxide and cement in the blended powder and the quantity to be fed to achieve end point of gellation are determined from Table 4. Following gellation, the mass is liquefied by intense stirring. The syrup like mass now has a viscosity of 3000 cp. The viscosity of the syrup is now increased with further addition of the blended powder until the value reaches about 20000 cp. In several of the above experimental results the quantity of blend needed is on the order of 20 wt% for gellation, and an additional 5 wt% for completion of SBW-189 grout formation. These weight proportions are expected to vary with changes in acid molarity in different waste solutions.

4.4.2. In the initial experiments, water was introduced to decrease the viscosity and to increase the addition of cement following gellation, as greater cement quantities usually would lead to rapid setting of the grout with higher mechanical strength. But many experiments have shown this later addition of water leads to its absorption into grout in large quantities for a viscosity of about 20000 cp to be reached. Most of the grouts so formed have tended to not cure within 28 days and have also shown large shrinkage. An improvement in this direction has involved addition of water directly to the waste solution prior to addition of the blended powder. In this improved step, any further addition of water is avoided, once the blended powder has been introduced into the waste solution, and the viscosity of 20000 cp is achieved by controlled addition of the blended powder following gellation as was described above. The quantity of water needed is at present determined by the rise in pH from zero to a value of about 0.5 for the SBW-180 waste, and this value is expected to vary with differences in the acid molarity for the waste solutions.

5. EXPERIMENTAL TEST PLAN FOR GROUT FORMATION

The proposed test plan provides the framework for 1. conducting a set of experiments on a small bench top laboratory scale in a chronological step, 2. ensuring the safety of the working environment, 3. minimizing laboratory waste production resulting from experimental development, 4. generating experimental results to support customer confidence in the scale up process, and 5. developing systematic deviations from planned experiments to mitigate stumbling problems.

5.1 Experimental Procedure for Forming 5 cm Grout Cubes

1. Waste Loading -Assume a starting waste loading in weight% and measure corresponding quantity of waste solution in a suitable mixing container. Select three possible waste loadings at 70 wt%, 50 wt% and 30 wt% using Figures 1 and 2 as the basis.
2. Neutralizing - Weigh appropriate proportion of neutralizing agent $Ca(OH)_2$, referring to Table 4.
3. Cement + Slag Mixing - Compensate the difference in the total weight of 100 grams by equivalent proportion of cement + slag mixture. Select four possible mixtures as 100 wt% Cement, 70wt%cement+30 wt% slag, 30 wt% cement + 70 wt% slag and 100 wt% slag, using Figures 1 and 2 as the basis.
4. Blending - Thoroughly blend 5.1.2 and 5.1.3 powders in a suitable container prior to adding to the waste solution.
5. Gellation Gradually add 5.1.4 mixture into 5.1.1 waste solution with continued stirring, and stop addition of 5.1.4 at the gellation point. Record pH of Gellation, it will be nearly 7 if gellation is complete.
6. Liquefaction Following gellation, stir the mass to liquefaction and record viscosity, it should be on the order of 3000 cp.
7. Grouting Now resume addition of blended powder until the liquefied mass reaches of viscosity of about 25000 cp. Measure viscosity at 2.5, 5, and 10 RPM, to bracket a constant strain of 10%, and record the RPM and viscosity at this strain. Measure the total amount of blended powder added and calculate actual waste loading.
8. Pouring – Pour the grouted mass into a plastic cube to full capacity
9. Curing – Allow the cube to cure in ambient atmosphere and conduct Vicat tests to measure hardening at intervals of 1,3,5,7,14, 21 and 28 days.

5.2 Measure of Success

The extent of success is measured by visual examination of the cube and the Vicat values. A cube containing bleed water is considered a total failure. Similarly, cube with no bleed water but unset within 28 days is regarded as failed. These failed experimental products will form the cold waste. The experiment is considered most successful for the cubes that cure within three days to a hardness of 1 on the Vicat scale, with negligible shrinkage. Fast curing cubes with dimensional stability will have the potential to save time for curing and thus the cost of processing.

5.3 Remedy to Failure

On the basis of the observations and results from 5.1.1 to 5.1.9, ad-hoc experiments and new test plans must be evolved to remedy failures.

5.4 Scientific Output

Formulation maps as shown in Figures 1 and 2 are an important scientific output of the experimental steps 5.1.1 to 5.1.9. The map forms the matrix, which upon full development for each waste stream and each neutralizing agent, will become the basis for selecting a range of grouts with desirable properties. The map will also form the basis for mitigating failure by predicting the compositional course in relation to properties for subsequent processing. Judicious repetition of experimental steps 5.1.1 to 5.1.9 as a function of variations in Vicat values, viscosity, grout strength and waste loading is important to complete development of formulation maps.

5.5 Ensuring Safety

Safety is enforced by use of personal protective equipment (lab coat, safety glasses), recording all the usage of chemicals, recording all the observable results, modifying the experimental steps on the basis observed results, conducting optimum number of experiments on the scale of cubic molds and minimizing cold waste production.

5.6 Support for Scale Up Process

The scale up to 55 gallon drums is a robust process that should be tolerant to variations in component mixing, grout flow and grout composition, and yet produces grout that sets rapidly. Formulation map has the potential to mitigate the expensive failure at this stage for it would outline the tolerant variations in property and composition of grouts.

5.7 Significance to Objectives

All the objectives from 3.1 to 3.5 are covered by all the items 5.1 to 5.6 of this test plan. The experimental steps 5.1.1 to 5.1.9 will be separately applied for each RCRA element listed in the objective 3.5. On an as needed basis the effects of objective 3.5 on 3.1 will be evaluated using steps 5.1.1 to 5.1.9 and formulation maps similar to Figures 1 and 2 will be produced.

5.8 Number of Cubic Molds and Number of Tests

As indicated in Figure 2, twelve cubic molds for each waste stream at waste loadings shown in 5.1 form the basic need for developing a preliminary diagram. In the twelve mold set the neutralizing agent bears a specific relation to the waste concentration as shown in Table 4. For each change in this relation, a separate set of 12 molds would be needed to generate a formulation diagram. On the basis of previous experimental results described in sections 2 and 4 of this test plan, it is tentatively speculated that twelve plus additional ten molds should yield a complete database for one waste stream. Each mold would be subject to 7 Vicat tests and three viscosity tests as indicated in experimental procedures 5.1.7 and 5.1.9. Each mold will be subjected to one strength test, assuming every mold passes the Vicat test of 28 day hardening. Thus, total number of tests for 25 molds per waste stream are 25 + [25 (molds) X 7 (Vicat)] + [25 X 5 (Viscosity)] + [25 X 1(mechanical strength)] = 350 tests. The total here is the maximum for a waste stream and is expected to be lower, since some molds may cure to Vicat value in the first few days of curing.

5.9 Venue of Tests

All bench scale tests will be conducted in laboratory 113 of building 637, INTEC. Scale up tests will be done in the low bay area of building 637 where the mixer is housed in an enclosed cell.

6. EXPERIMENTAL PLAN FOR WASTE ABSORPTION IN SILICA GEL

The method of waste immobilization here involves mixing commercial grade silica gel with the acidic waste effluent solutions. The resulting mass must be devoid of free liquid for meeting the disposal criteria at the WIPP site. In this method the waste species are likely to be contained by physical adsorption and cationic exchange [14] at the surface of the silica gel. Therefore, the waste loading is expected to depend on (1) the surface area of the original silica gel powder and (2) the wetting

characteristics of the silica particle surface by the waste solution. While the surface area would be determined by the hydrated silica gel particle size distribution in the commercial powder, the adherence of the waste to the particle surface would depend on the conditions leading to favorable wetting interactions between the particle surface and the ionic species characteristics of the waste. Silica gel testing will focus on neutralization and waste loading. Latest WIPP regulations require the waste to be partially neutralized to above a pH of 2. Each waste stream will be neutralized with sodium hydroxide, calcium hydroxide and magnesium oxide. This will generate titration curves for each waste with each neutralizing agent. With the waste neutralized to pH of 2, the waste will be absorbed on silica gel. The waste loading will be varied to determine the exact loading where free liquid develops. This will be checked with freeze/thaw cycling and vibration testing. The results will establish an upper bound for operation loading limits.

7. EXPERIMENTAL PLAN FOR CONTAINING LET&D WASTE SOLUTION IN GROUT

As mentioned earlier, LET&D waste solution is highly acidic and additionally contains RCRA toxic metals. Previous studies have indicated the need for adding slag to the cement matrix to immobilize the RCRA metals such as mercury [3]. The present experimental plan is to prepare three batches of LET&D simulants with each batch varying in mercury concentration as 1X, 2X and 3X (where X = original mercury concentration). Additional batches will be prepared where individual components will be varied up to ±5 wt% while maintaining the other components at the formulation amounts. This will help to establish an operational envelope for the LET&D grout formulation. The results will be transformed into a formulation map composed of three independent variables cement-slag and LET&D waste solution. Presumably, the map will provide venue for directional changes in the relative proportions of the three components to formulate compositional regions of optimum waste loading and waste form properties. The retention of mercury will be evaluated by TCLP tests after 28 day curing of the grout.

8. DRUM COMPATIBILITY STUDY

There is a question concerning the material of construction for the drums or containers of solidified waste. Can the drums be made of carbon steel or is stainless steel needed? There are no specific requirements for containers for low-level waste forms; however, for local purposes, the drums must maintain their integrity during interim storage and shipping to the repository. In order to determine the material of construction for the waste drums, a corrosion study will be completed for each of the waste types.

The drum compatibility study will consist of preparing grout and silica gel samples and placing corrosion coupons in the samples. The coupons will be type 1020 carbon steel and 304 stainless steel representing standard drum materials of construction. It is planned to test the coupons after 3 months and after 12 months of exposure. The lower half of each coupon will be placed in the solidified, simulated waste with the upper half remaining in the headspace of the sample bottle. Three similar coupons will be placed in each sample to provide minimum statistical values. Grouted samples will be prepared for LET&D acid, NWCF scrub, steam reformer scrub, SBW-180, and SBW-189. Similar samples will be prepared for the neutralized wastes absorbed on silica gel (except LET&D acid which will be grouted only).

In summary using SBW-180 as a example, there will be 4 grouted samples (2 containing 3 carbon steel coupons each and 2 containing 3 stainless steel coupons each) and 4 silica gel samples (2 containing 3 carbon steel coupons each and 2 containing 3 stainless steel coupons each). One set of carbon steel coupons and 1 set of stainless steel coupons will be examined at after 3 months for both grout and silica gel. The remaining sets will be examined after 12 months. The corrosion coupons will be visually and

microscopically examined. Each coupon's mass will be compared from pre- and post-test to determine any mass loss and thus the corrosion rate. NOTE: Samples for this study were prepared in July 2003. No results are available this fiscal year.

9. DELIVERABLES

1. Formulation map for each waste stream with property values, expressing relations among properties, waste loading and grout composition
2. A select group of formulations with tolerable ranges in composition, waste loading and properties for testing on a larger scale
3. A detailed display of experimental procedure and results to the public domain in the form of an external report.

10. LABORATORY SETUP AND EQUIPMENT

The small-scale laboratory setup for this study is currently housed in the laboratory 113 of 637 INTEC. This laboratory is equipped with hood space for grout preparation, viscometer, Vicat tester, pH meter and other essential accessories. The mechanical strength tester is located in the high bay area of building 637, INTEC. The mixer for grout preparation on a gallon scale is located in the high bay area and is currently operational.

11. RESULTS AND DISCUSSION

11.1 Waste Stream Fixation in Grout

11.1.1 The Effects of $Ca(OH)_2$ and NaOH Neutralizers on Grout Formation

Fixation of SBW-180, SBW-189 and NWCF Scrub, as a function of neutralizing agents, and cement/slag ratio was investigated. Of the three neutralizing agents, ammonium hydroxide, sodium hydroxide and calcium hydroxide, the last one has yielded more effective results. The results of $Ca(OH)_2$ addition to cement-slag and SBW-189 are shown in Figures 3 and 4. The $Ca(OH)_2$ was varied from 3.5 to 11 weight% as shown by horizontal boundaries on the diagram. The blocks indicated on Figures 3 and 4, delineate the compositional region for forming stable grouts with desirable processing properties of viscosity, grout setting time, waste loading and waste form shrinkage. Grout compositions outside of the marked region, were observed to be either very low in waste loading (below 50 wt%) or showed large shrinkage and registered 50 on the Vicat hardening scale. In the range of waste loadings above 52 wt% and under 71 wt%, the grouts also appear to remain soft with increase of slag/cement ratio beyond the 50% vertical boundary line on the diagram. Within the compositional block, the setting time was noted to increase with increase in waste loading towards higher $Ca(OH)_2$. Yet these grouts were noted to set to Vicat value of 1 within fifteen days. Rapid setting within three to five days seemed to occur for grout compositions occurring under the 7 weight% $Ca(OH)_2$ boundary line. About this boundary the waste loading of SBW-189 ranges from 60 to 68 weight%. The viscosity for these grouts ranges from 20000 to 35000 cp. The viscosity was noted to increase to about 40,000 cp in the directions of 4 wt% and 11 wt% $Ca(OH)_2$, from the 7 weight% $Ca(OH)_2$ median position. While the increase in viscosity towards 4 wt% $Ca(OH)_2$ boundary line is attributed to lower waste loading, a similar increase towards higher waste loading seems to be caused by increase in the $Ca(OH)_2$ to about 11 weight%. On the basis of these results

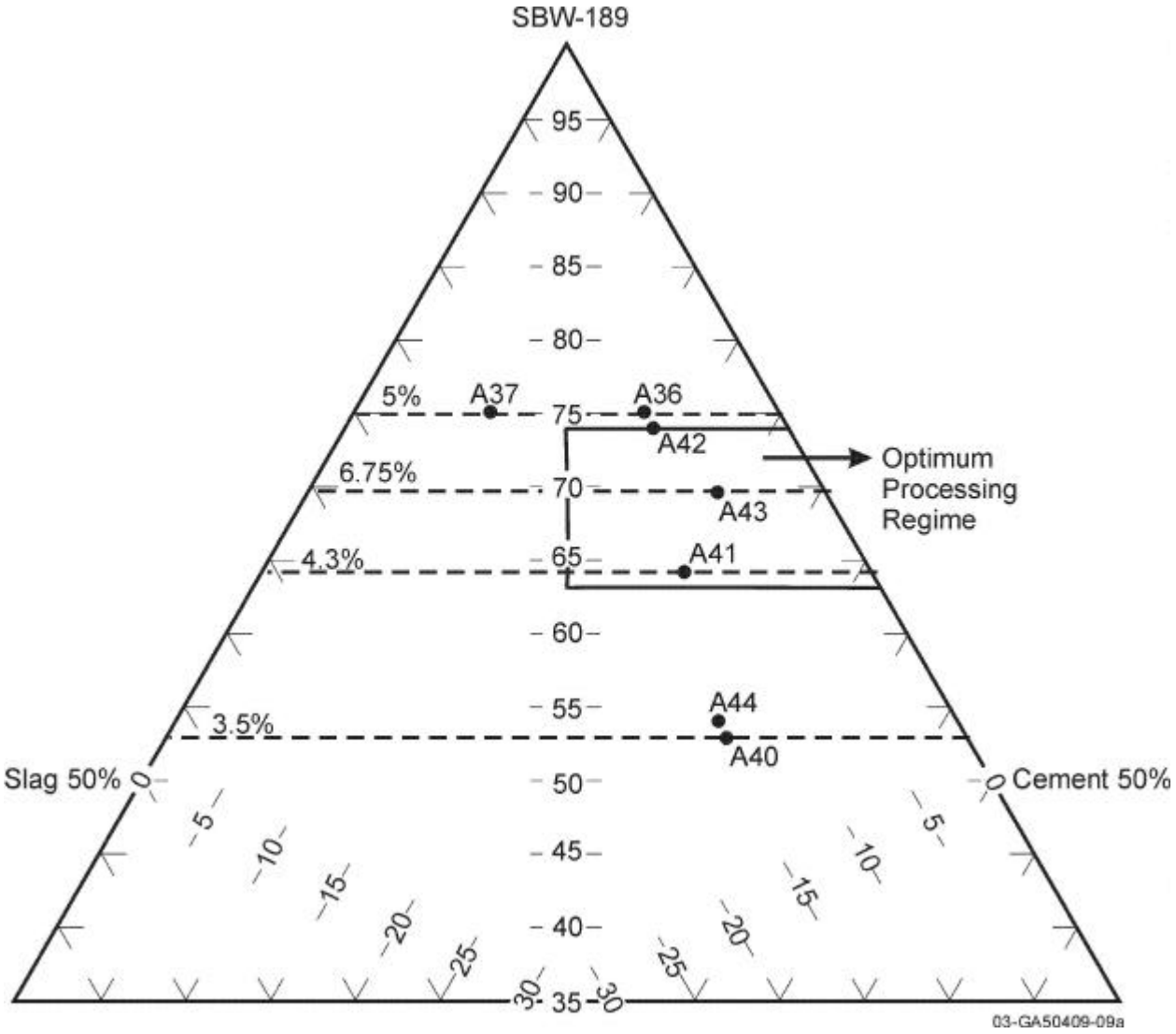

Figure 3. Grout formulation diagram with SBW-189 waste, slag, cement and $Ca(OH)_2$ as independent variables. $Ca(OH)_2$ weight% values are projected onto the ternary surface from the quaternary apex, at 5 wt%, 6.75wt%, 4.3 wt% and 3.5 wt% horizontal boundaries. The upper limit of slag/cement ratio is marked by the vertical boundary at nearly 50% slag:50% cement weight ratio. The upper and lower limits of waste loadings in the optimum processing regime are calculated as [%waste /(100% total solids - % $Ca(OH)_2$)] , thus the upper limit = 75% /(100 - 5) = 71.25 wt%, and lower limit = 65% /(100 - 4.0) = 62.4 wt%. SBW-180 are also processed within the same block.

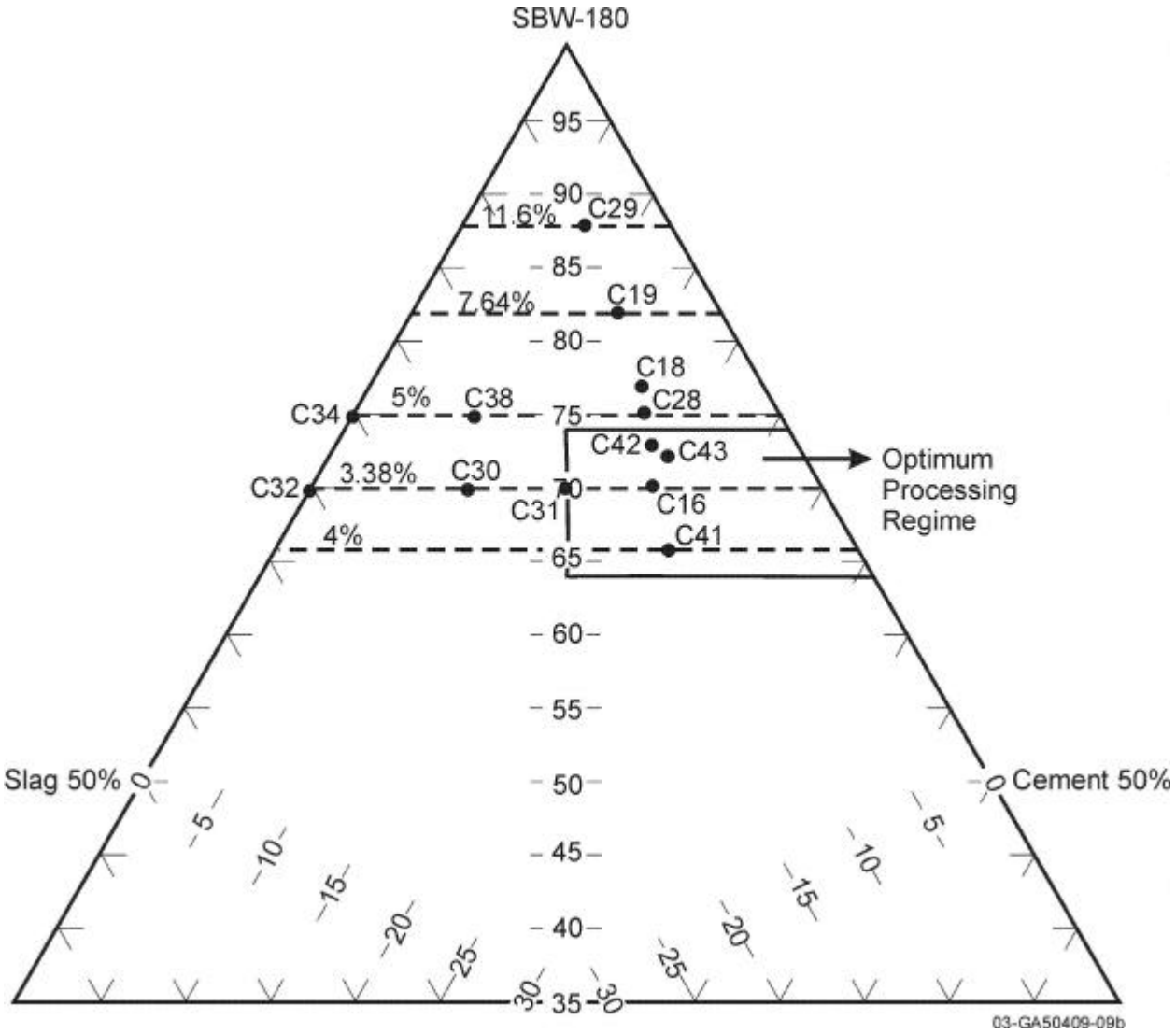

Figure 4. Grout formulation diagram with SBW-180 waste, slag, cement and $Ca(OH)_2$ as independent variables. $Ca(OH)_2$ weight% values are projected onto the ternary surface from the quaternary apex, at 11.6 wt%, 7.64 wt%, 5 wt%, 3.38 wt%, and 4.0 wt% horizontal boundaries. The upper limit of slag/cement ratio is marked by the vertical boundary at nearly 50% slag:50% cement weight ratio. The upper and lower limits of waste loadings in the optimum processing regime are calculated as [%waste /(100% total solids - % $Ca(OH)_2$)] , thus the upper limit = 75% /(100 - 5) = 71.25 wt%, and lower limit = 65% /(100 - 4.0) = 62.4 wt%. SBW-189 are also processed within the same block.

an optimum compositional field for grout formation appears to fall within 7 and 5 wt% $Ca(OH)_2$ and also in the range of slag/cement ratio varying from 0 to 50 wt% slag. These results also apply to SBW-180 grout with differences caused by lower acid molarity in the latter waste stream. Because of low acid molarity of 1.0 when compared to nearly 3.0 (Table 1) for SBW-189, a higher loading of SBW-180 results under the same formulation ratio as for SBW-189. Thus, the region of stable grout formation for SBW-180 (Figure 4) also falls within the block outlined for SBW-189 (Figure 3). This difference in waste loading between SBW-180 and SBW-189 as a function of acid molarity is more evident in Figures 4 and 5. In these figures, the transformation of the liquid waste stream to grout through stages of gellation, liquefaction and slurry formation are noted with dependence of pH on total solids (cement+slag+$Ca(OH)_2$) addition to the waste stream. At low acid molarity as in SBW-180, variation in $Ca(OH)_2$ / ($Ca(OH)_2$ + Waste) from 10 wt% in C42 grout to 5.9 wt% in C41 grout, has a smaller influence on gellation, as a result the spread in the mass of total solids needed to achieve gellation is much narrower, from 43 to 50 grams for neutralizing 215 grams of SBW-180 to a pH of about 3 (Figures 5 and 6). Whereas for SBW-189 with acid molarity of 3.0 the spread in total solids mass ranges from 58 to 80 grams for the same variation in $Ca(OH)_2$ /waste ratio and similar neutralization pH of about 3. For both waste streams, liquefaction is reached at pH 7 with vigorous stirring and no further addition of total solids (Figures 5 and 6). The residence time for this reaction is on the order of ten minutes. This step is followed by subsequent addition of solids to form a stable grout with viscosity in the range of 10,000 to 20,000cp for both waste streams. The net result of these differences in the interaction with the total solids and $Ca(OH)_2$ is reflected in the lower waste loading for SBW-189 by about 3 to 5 wt% compared to that of SBW-180. From this exercise it may be concluded that a uniform formulation can be applied to waste streams showing variations in acid molarity with corresponding changes in waste loading. It remains to be observed how the RCRA metals will be immobilized by these formulations, the general results from preliminary studies have shown slag to be instrumental in the retention of RCRA components and hence slag was introduced to form the formulation block in Figure 3. These experiments were not extended to evaluate the behavior of NWCF Scrub solution. At present it is presumed that Scrub waste stream with molarity of 2.0 will show fixation behavior in grout that is intermediate between SBW-180 and SBW-189, and therefore, the formulation diagram in Figure 3 is predicted to apply to Scrub waste stream as well.

The results from the original matrix studies (pages 1 and 4 of Appendix A) show the high amounts of NaOH as a neutralizer did not yield good grouts for SBW-189 while keeping the waste loading high (70 wt% and above). In these experiments, liquid NaOH (50 weight to weight %) was used; thus, excess water is added to the waste. The grouts formed by NaOH neutralization considerably degrade or do not set up. The degrading characteristics are like large shrinkage, large cracking, and evolution of sodium nitrate fibers (Figure 7). Wastes that are fully neutralized to caustic and grouted at about 35 wt% are very stable, such as the NWCF scrubber grout. Also of note is that one process option being considered is to use solid NaOH to pre-neutralize the SBW prior to CsIX. This option is a recent development and has not been explored from a grouting standpoint.

Both $Ca(OH)_2$ and NaOH neutralized grouts were subjected to identical accelerated curing conditions (forced air drying). While the $Ca(OH)_2$ grouts have stably resisted the accelerated curing (Figures 8 & 9) for both SBW-180 and SBW-189, the rapid degradation of NaOH neutralized grouts became apparent in Figure 7. Accelerated curing was introduced by placing the grout cubes in an evacuating hood, where the suction of air from the hood possibly enhances the escape of vapor phase from the grout, resulting in the formation of sodium nitrate fibers that could be characterized by X-ray powder diffraction shown in Figure 10. At present it is speculated that such fibers should not be allowed to form, as they may potentially contain other alkalis like the radioactive cesium, or may even contain RCRA metals as occluded elements in the lattice that could be released with the dissolution of nitrate on contact with moisture. It has been possible to retard the formation of these crystalline fibers by curing the NaOH neutralized waste forms in an enclosed atmosphere, so that the vapor phase is contained within the waste

form during its curing period. This impeded escape of vapor phase is accomplished by a simple method of enclosing the waste form in a sealed plastic bag. It remains to be tested how these waste forms will stabilize upon subsequent exposure to ambient conditions. Alternatively, it will be noted from Figures A and B (in the Executive Summary) that sodium nitrate layer formation is totally eliminated by introduction of slag and simultaneous decrease of waste loading by about 5 wt% in grouts A12 and A13 (Figure 8 and Table 6).

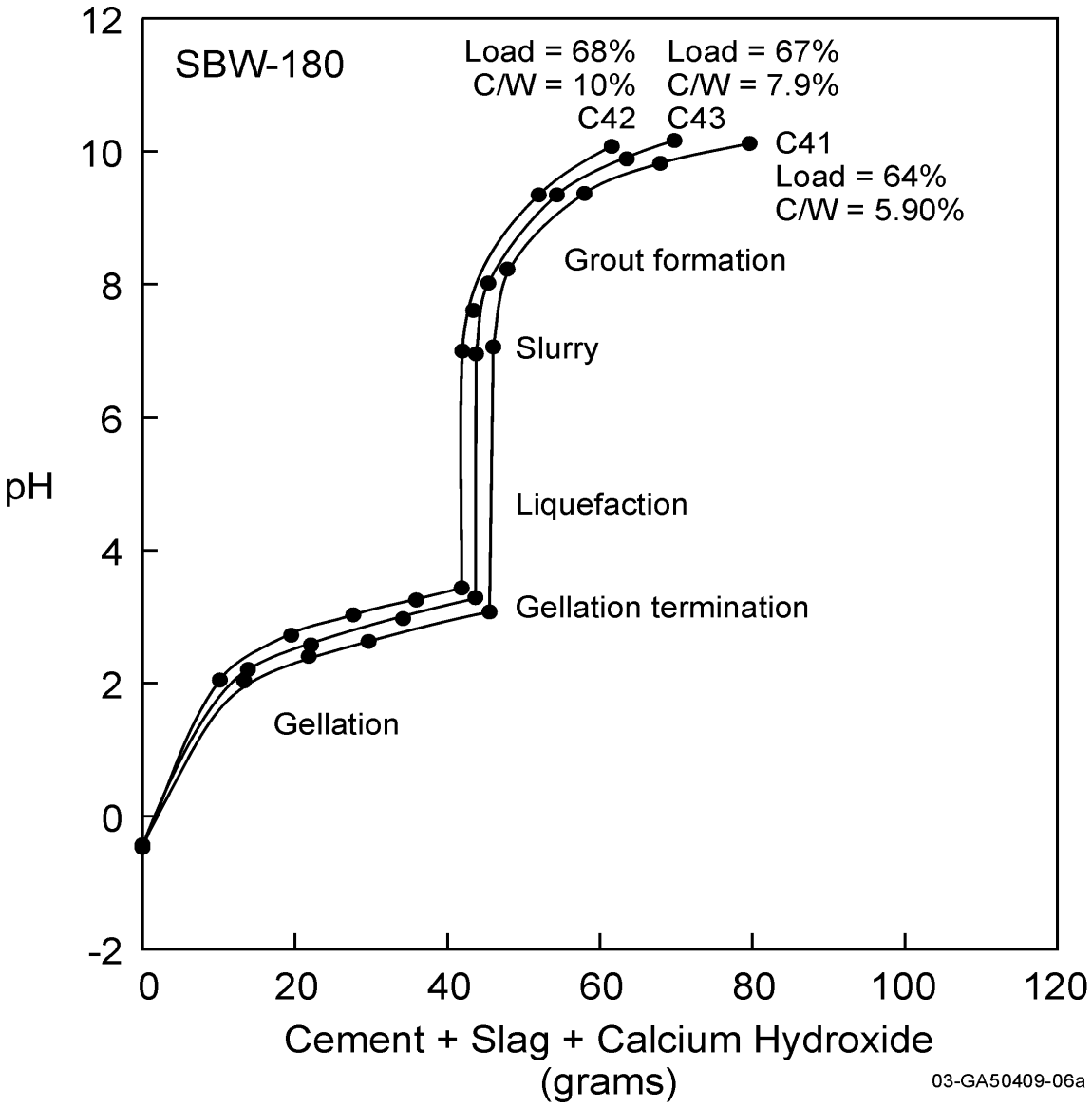

Figure 5. pH variations as a function of total solids addition to the SBW-180 waste solution. The first order phase change from gel to slurry is marked by a sharp vertical rise in pH and occurs with rigorous stirring of the gelatinous mass with no further addition of total solids. Subsequent additions of total solids lead to transformation of slurry to grout. Because of lower acid molarity of nearly 1 for SBW-180, these phase changes are less sensitive to variations in the neutralizer $Ca(OH)_2$, causing the trends to be closely spaced for SBW-180 fixation in the grout. C/W = $Ca(OH)_2$ / waste + $Ca(OH)_2$; Load = Waste Loading; C42, C43, C41 = SBW-180 grouts.

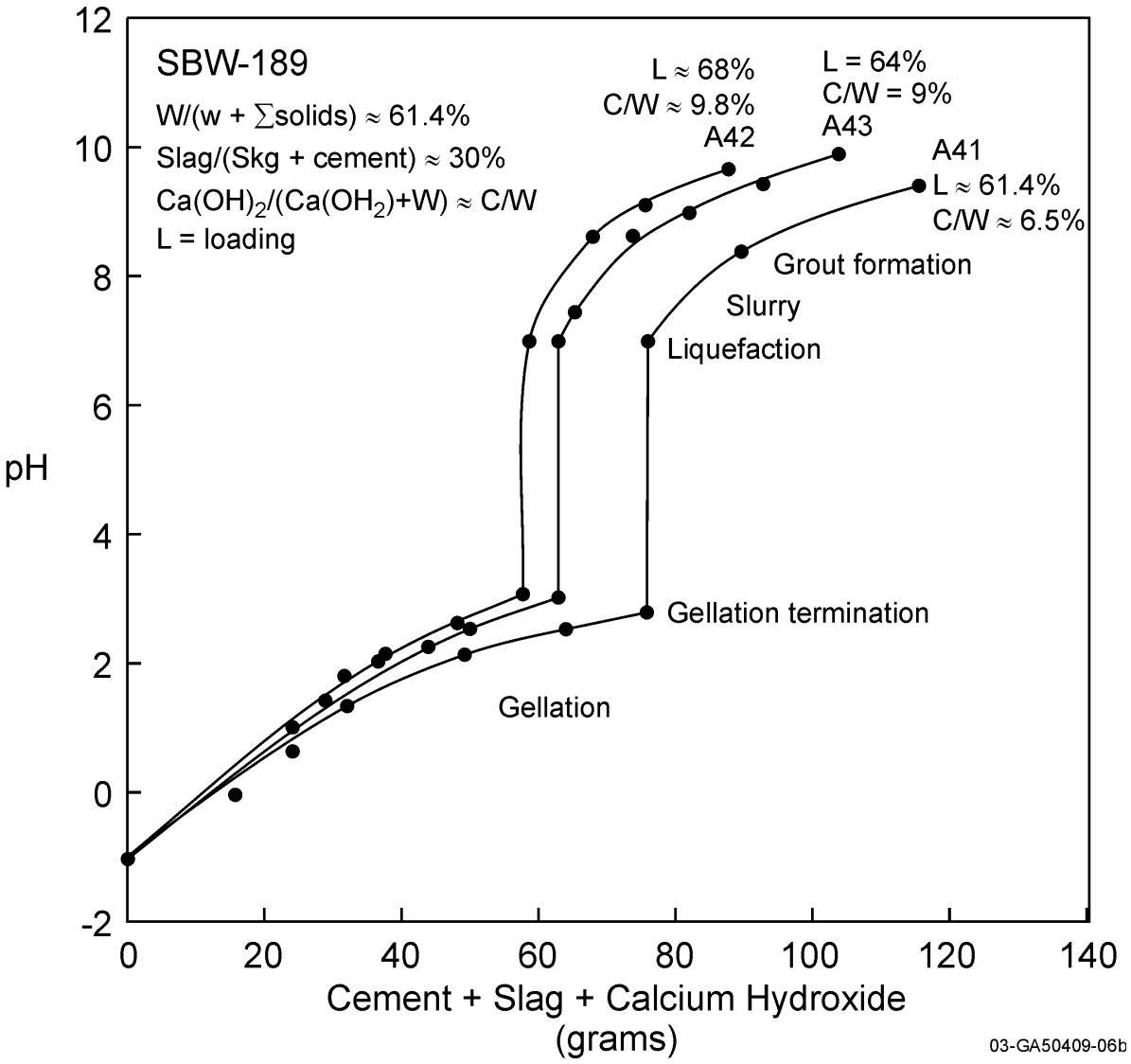

Figure 6. pH variations as a function of total solids addition to the SBW-189 waste solution. The trends are similar to the one shown in Figure 4. But due to higher acid molarity of nearly 2.86 for SBW-189, the phase changes are more sensitive to variations in the neutralizer $Ca(OH)_2$, causing the trends to be more widely spaced with respect to total solids addition for SBW-189 fixation in the grout. Both Figure 4 and Figure 5 trends correspond to 30 w% slag / 70 wt% cement ratio in Figure 3. A42, A43, A41, = SBW-189 grouts.

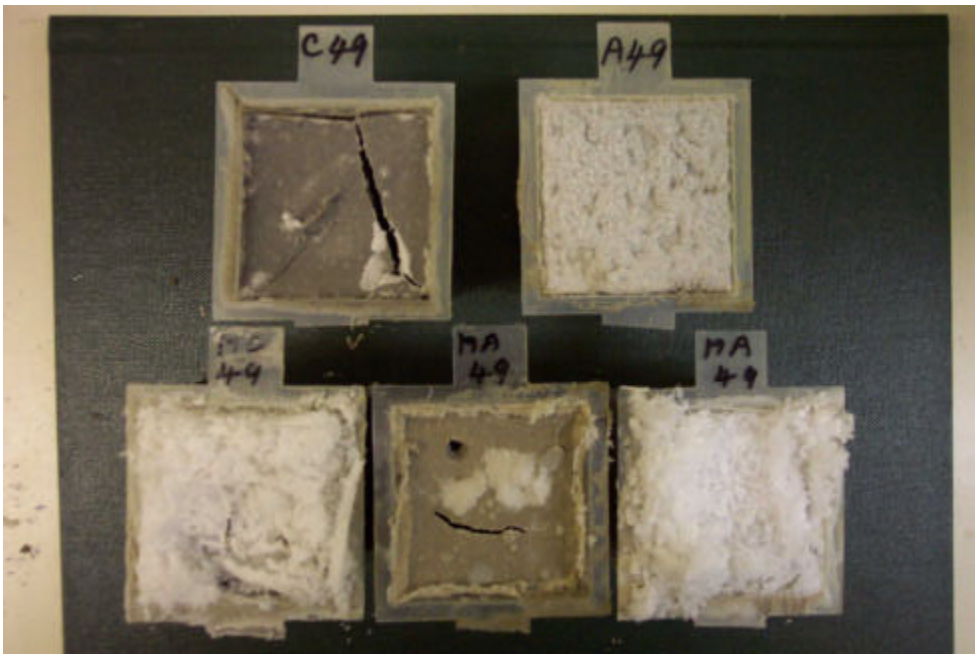

Figure 7. Grout cubes of SBW-189, (A49) and SBW-180 (C49) after neutralization by NaOH. Note the large dimensional changes due to shrinkage, cracking and white sodium nitrate crystallite formation on the surface.

Figure 8. Cured grouts of SBW-189. Left to Right : Top row: A12, A13, A40, Bottom row: A41, A42, A43, and A44. Surface whitening to varying degrees, caused by formation of about 1 mm thick sodium nitrate crystallite layer due to differences in the formulation. Compositions are shown on the formulation diagram Figure 3 and Table 6. Introduction of higher slag content (25 wt%) eliminated sodium nitrate vaporization and crystallization in grouts A12 and A13, and also imparted a brownish color to the grout matrix. Properties of these grouts are shown in Tables 7 and 8, and Figure 6.

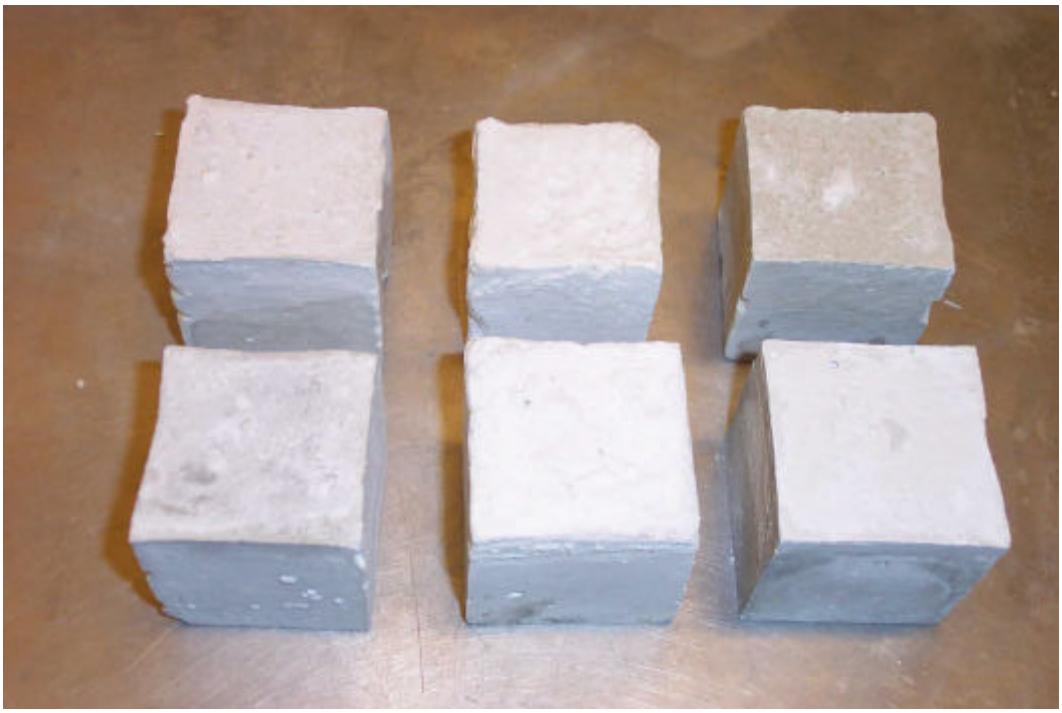

Figure 9. Cured grout of SBW-180. left to right: Top row: C16, C28 and C31, Bottom Row: C41, C43, C44. Properties of these grouts are shown in Tables 7 and 8, and Figure 5. Their relative compositions are shown on the formulation diagram, Figure 4 and Table 6.

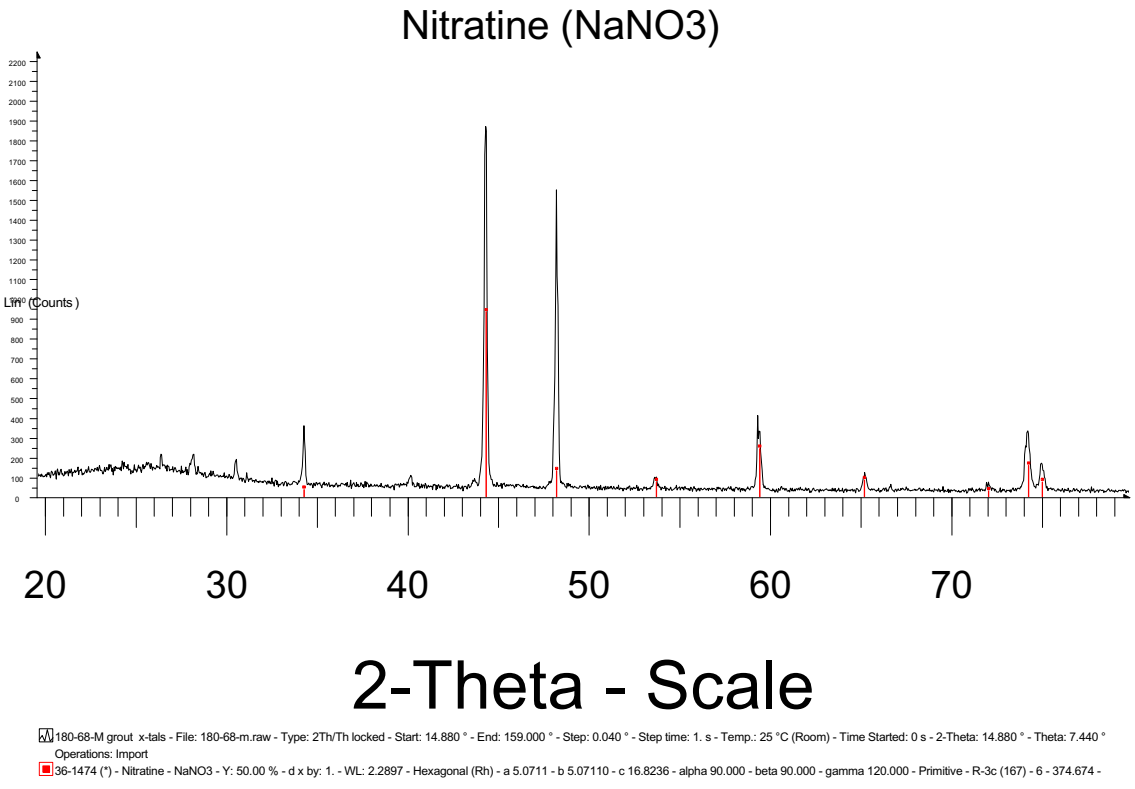

Figure 10. X-ray powder diffraction of fibrous crystallites forming on the surface of grouts in Figure 7.

11.1.2 Mechanical Properties of Grout

Exploratory tests were conducted to evaluate the quality of the waste form by yet another easily measurable property, the mechanical strength. The sensitivity of this property to chemical component additives has been documented in the commercial concrete industry [18], accordingly in the present study it is speculated that the changes in the formulation and waste loading would induce possible variations in the mechanical strength that may provide yet another parameter for assessing the stability of the grout. This contention is supported by the results gathered from preliminary tests shown in Table 8. In these tests, the cubes measuring 5cmX5cmX5cm were enclosed in a plastic bag and subjected to a dynamic uniaxial compression as shown in Figure 11. The force was increased at a steady rate of 267 lbs/sec, until

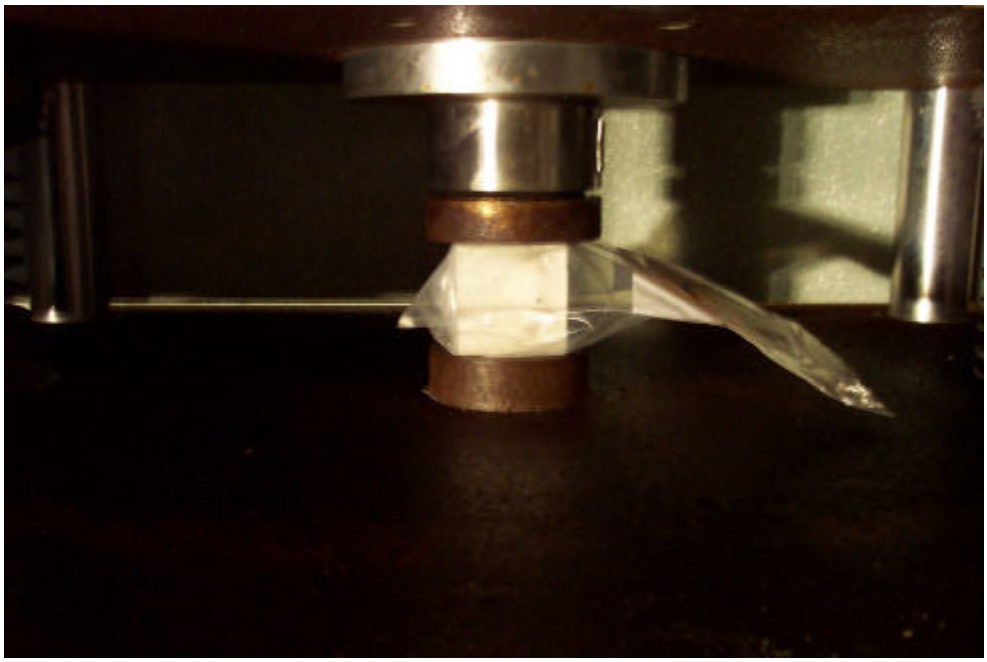

Figure 11. Position of the grout sample in the strength tester. The grout cube is placed in a plastic bag and then held between the discs for compression test in ambient atmosphere. The load is ramped at the rate of 150 lbs/sec until the breaking point of the sample.

failure of the sample. The force at the failure point and the time to failure were recorded. The mechanical strength was calculated as force / unit area and an approximate dependence of this property on formulation and waste loading is noted in Tables 7 and 8. As would be expected a general result emerges suggesting an increase in the mechanical strength with decrease in waste loading, for example, A40 and C41 grouts of SBW-189 and 180 record a higher mechanical strength of 2500 PSI (Table 8). At the low waste loadings of 51 wt% SBW-189 (A40) and 63 wt% SBW-180 (C41) in these grouts, the variations in the formulation (Tables 7 and 8) have little influence on the mechanical properties. However, the sensitivity to changes in formulation is apparent at higher waste loading of 68 wt%. For example the mechanical strengths of grout C28 and C16 are 1400 and 1500 PSI, and of A42 and A12 are 880 and 1190 PSI respectively. A noticeable change in the formulation of these grouts is in the $Ca(OH)_2$ neutralizer content, indicating decrease of mechanical strength with increase in $Ca(OH)_2$, although grout hardening is accelerated by increased addition of calcium hydroxide. From this difference in the calcium hydroxide effect it may be tentatively concluded that lowering of $Ca(OH)_2$ to 3.4 wt% (C16) from 10.20 wt% (C28) or to 3.2 wt% (A12) from 7.4 wt% (A42), prolongs the curing time, but also enhances the

mechanical strength. It may seem appropriate to consider decreasing the $Ca(OH)_2$ to a lower value of 3.5 wt% since, this component is already a part of Portland cement. It is present in the cement as CH in conjunction with C-S-H, where CH and C-S-H are abbreviations for calcium hydroxide and calcium silicate hydrate in the hydrated cement. During the curing period both these components interact to determine the modulus of elasticity of Portland cement [19]. Since, CH has a lower critical stress intensity factor (K_c) than Portland cement [19], excess addition of $Ca(OH)_2$ would very likely have a degrading effect on the mechanical behavior of the grout, as is observed in formulations C28 and A42 in Tables 7 and 8. Thus the testing of mechanical strength would not only contribute to evaluating the stability of the grout, which is a useful property for safe transportation of this nuclear material but it may also form the basis for introducing beneficial changes to formulations.

Table 7. Formulations, Viscosity, and Vicat Numbers for SBW-189 and SBW-180

Sample#	SBW-180 Wt%	SBW-189 Wt%	Ca(OH)2 Wt%	Cement Wt%	Slag Wt%	Vicat 7 day	Viscosity Centipoises X 10^3	Nitrate Layer Thickness
C16	67.98		3.4	20.03	8.58	0	20-25	0
C17	68.13		6.81	17.53	7.5	0	20-25	1 –2 mm
C18	71.66		7.16	14.81	6.35	0	20-25	10 mm
C19	76.43		7.64	11.14	4.77	50	8-12	
C27	67.62		3.38	20.29	8.69	0	12-20	1-2 mm
C28	68		10.20	15.23	6.52	0	25-30	0
C29	77		11.63	7.67	3.70	0	530-35	10 mm
C30	67.62		3.38	8.69	20.29	2	20-25	0
C31	67.62		3.38	14.49	14.49	2	15-20	0
C32	67.62		3.38	0	29	50	20-25	
C34	71.54		5.00	0	23.45	50	20-25	
C36	71.54		5.00	16.41	7.0	2	20-25	0
C38	71.54		5.00	7.03	16.41	10	20-25	0
C41	63.92		4.01	22.44	9.62	1	20-25	0
C42	68.4		7.59	16.76	7.20	0	25-30	0
C43	67.01		5.77	19.04	8.16	0	35-40	0
C44	60.17		1.8	26.60	11.43	1	30-35	0
A12		68	3.2	3.2	25.6	50	20-25	0
A13		68	3.2	6.4	22.4	30	25-30	0
A36		71.54	5.00	16.41	7.03	40	5-10	
A37		71.54	5.00	7.03	16.41	40	20-25	
A40		51	3.56	31.84	13.64	2	30-35	0
A41		61.36	4.29	24.03	10.30	1	30-35	0
A42		68	7.4	17.0	7.60	7	30-35	0
A43		64.2	6.75	22.30	6.68	1	35-40	0
A44		53.52	1.34	31.63	13.53	1	15-20	0

Table 8. Mechanical Properties of Grout Cubes

Grout #	Waste loading wt%	Density g/cm^3	Area inches^2	Force lbf	Strength psi	Breaking Time sec
C16	67.98	1.43	4.0	6000	1500	49.6
C28	68	1.51	2.25	3130	1390	62.2
C31	67.2	1.38	4.0	4080	1020	30.3
C41	63.92	1.58	3.0	7680	2500	85.1
C43	67.01	1.32	4.0	5010	1250	35.1
C44	60	1.44	4.0	6430	1600	36.4
A40	51	1.76	4.0	10060	2510	47.4
A41	61.36	1.64	4.0	4940	1230	30.6
A42	68	1.44	4.0	3550	880	34.8
A43	64	1.54	4.0	4640	1160	30.0
A12	68	1.61	4.0	4780	1190	69.5
A13	68	1.66	4.0	4460	1110	32.0

11.1.3 Grouting of Pre-Neutralized Sodium-Bearing Waste

Towards the end of the fiscal year, it was noted that the separations research was finding that the sodium-bearing waste needed to be pre-neutralized in order to reduce or prevent degradation to the cesium ion-exchange sorbent. The latest intent is to use sodium hydroxide to partially neutralize the waste streams to 0.5 Molar. For the SBW-189 stream simulant, it takes 114 mL sodium hydroxide to bring 1 L to 0.5 Molar. With both the SBW-180 and SBW-189 wastes now at 0.5 Molar, the question becomes, "Can a common grout formulation be found for both wastes?" A limited number of grout samples were prepared at the end of the year to determine if this is possible. The short answer is, "Yes." Table 9 shows the preliminary common formulation used for SBW. In the case of SBW-189 grout, 66 wt% is SBW and 9 wt% sodium hydroxide to partially neutralize; thus, 75 wt% would be the incoming liquid to be solidified. In the case of SBW-180 grout, 72 wt% is SBW and 3 wt% is sodium hydroxide, again for 75 wt% to be grouted. The remaining powders are 7 wt% calcium hydroxide, 6 wt% slag, and 12 wt% Portland cement. These formulations were successfully used in the 55-gallon drum pilot plant mixer tests [22]. The formulations proved to be self-leveling in the drums and readily thickened to a clay-like compound. It is thought that the blast furnace slag promotes the self-leveling property. Without slag, the grout is much thicker. A detailed formulation study is needed to confirm these preliminary findings and determine the operational envelop.

Table 9. Pre-Neutralized SBW Grout Formulation from CsIX Process

Component	SBW-180 (wt%)	SBW-189 (wt%)
Sodium-Bearing Waste	72	66
Sodium Hydroxide (50%)	3	9
Calcium Hydroxide	7	7
Blast Furnace Slag	6	6
Portland Cement	12	12

11.2 Waste Absorbed on Silica Gel

For the SBW-180 and SBW-189, the silica gel could be sprinkled on the surface of the liquid waste simulant. The silica gel would readily absorb the liquid and fall to the bottom of the sample bottle (Figure 12). For the calciner scrubber solution, mixing is required due to the higher viscosity of the solution. Without mixing, the silica gel will not fall to the bottom of the solution. Detailed results of the silica gel matrices for SBW-180 and SBW-189 are found in Appendix B. Also included is a limited set of samples for scrubber waste. In general, all three wastes can be directly absorbed on silica gel in the range of 65 wt% to 75 wt%. Samples at 65 wt% to 70 wt% usually exhibited excess silica gel remaining on top of the sample. From 70 wt% and above the waste approached saturation to the point where free liquid remained in the sample.

One finding of concern was the acidic vapor coming off the post-treatment silica gel. When litmus paper was placed in the headspace of the sample bottle, it readily turned red in a matter of seconds, indicating acidic conditions. This presents a serious problem for this waste form in that the acid vapor in the headspace could condense and readily corrode the waste drum. To eliminate or reduce this problem, simulants were pre-neutralized to a pH over 2 and then mixed with the silica gel. For the samples made with pre-neutralized simulants, the acidic vapors were greatly reduced. Pre-neutralization reduced one problem, but presented a second one, i.e. the waste becomes more viscous when the acid is reduced. Thus, for wastes pre-neutralized to a pH over 2, mixing is required due the increase in viscosity and some precipitation. One other advantage for pre-neutralization is that the RCRA corrosive code can be removed from the waste form.

Figure 12. Typical grout (left) and silica gel (right) bottled samples. Note: Both are with SBW-189 simulant. The silica gel was directly loading with no pre-neutralization. The grout contains high slag with calcium hydroxide.

The paint filter test was used only once in this test. In all cases, it was very easy to observe free liquid on the surface of the silica gel. For the one test case, a gel that physically looked like it was saturated, but exhibited no free liquid, was placed in the filter. No free liquid fell from the filter. It was only when physical pressure was exerted on the silica gel, that some liquid was squeezed from the saturated gel.

Several silica gel samples underwent thermal cycle testing. Most samples passed the test with no free liquid. These samples had waste loadings less than 74 wt%. Samples, from 74 wt% to 76 wt% that were near saturation but showed no free liquid when prepared, did exhibit free liquid. In these latter cases, the free liquid "floated" to the top surface. Further, a vibration test (not originally planned) was completed on the silica gel samples. A vibrator was placed in a plastic bin that contained the silica gel sample bottles. The samples were vibrated for 8 hours per day for 3 days. Again, as in the thermal cycle tests, the saturated samples showed free liquid. Samples that had excess silica gel on the top did not show free liquid.

11.3 Mercury Retention for Grout Made from Direct Evaporation Condensate

One other treatment alternative for sodium-bearing waste is to evaporate the waste to a solid. In this case the off-gas contains considerable acidic and water vapors. These vapors would be further processed by the Process Equipment Evaporator (PEW) and by the Liquid Effluent Treatment and Disposal (LET&D) facilities. This reduces the waste to a highly acidic solution of minimum volume. The estimated composition is noted in Table 1. From the table, it can be noted that the concentrated waste solution contains mercury and chromium. Since this solution would be a low-level waste, the waste form would need to pass RCRA hazardous, toxic metal leach requirements for land disposal. From previous studies [4], it was found that the LET&D acid solution could be grouted at 35 wt% loading. Using this grout formulation, 5 grout samples were prepared for leach testing. The grout contained 35 wt% LET&D acid, 12.4 wt% calcium hydroxide, 13.1 wt% blast furnace slag, and 39.5 wt% Portland cement. For one sample, 2 times the mercury was added and in another sample, 3 times the mercury was added. In a third sample, 5% extra simulant was added to suggest a process variation. The final sample was made with nitric acid with no added mercury to provide a "blank" sample. Following a 28 day cure period, the grout samples were crushed and leached in accordance with the toxicity characteristic leaching procedure (TCLP) and the resulting leachate was analyzed for mercury and chromium. In all cases, the mercury and chromium leach values were less than the Land Disposal Restrictions Universal Treatment Standards (Table 10). Thus, grout is a viable waste form for this effluent.

Table 10. Mercury and Chromium Leach Results for LET&D Acid Grout

Sample	Test	----------Mercury (mg/L) ----------			
		In Simulant	In Grout	After TCLP	UTS Limit
LTD-35-9	Normal Grout	702.07	361.25	0.005	0.025
LTD-35-10	2 X Mercury	1412.52	726.82	0.009	0.025
LTD-35-11	3X Mercury	2122.98	1092.38	0.003	0.025
LTD-35-12	+5% Simulant	737.17	379.31	0.008	0.025
LTD-35-13	12M Nitric Acid	0.00	0.00	0.002	0.025
Sample	Test	----------Chromium (mg/L) ----------			
		In Simulant	In Grout	After TCLP	UTS Limit
LTD-35-9	Normal Grout	103.99	53.51	0.008	0.60
LTD-35-10	2 X Mercury	103.99	53.51	0.007	0.60
LTD-35-11	3X Mercury	103.99	53.51	0.006	0.60
LTD-35-12	+5% Simulant	109.19	56.18	0.006	0.60
LTD-35-13	12M Nitric Acid	0.00	0.00	0.005	0.60

11.4 Absorption of NWCF Scrubber Solution on Calcine

If the New Waste Calciner Facility were used for sodium-bearing waste treatment, one candidate method for disposal of the calciner scrubber solution is absorption onto the calcine. The calcine would then be sent to the Waste Isolation Pilot Plant as transuranic waste. Laboratory scale testing was completed to evaluate the placement of scrubber solution on calcine. Varying amounts of scrub simulant were absorbed on pilot-plant calcine to observe calcine absorbency and morphology and any reactions. It was noted that the acidic scrub caused an exothermic reaction and observable gas was evolved. A hard crust was formed where the calcine and scrub reacted. It was concluded that up to 10 wt% scrub may be added to the calcine before free liquid occurs. Details of this study are presented in Appendix C.

11.5 Grout Radiation Levels Due to SBW Undissolved Solids

Calculations were completed to investigate the effect of undissolved solid (UDS) breakthrough on the grout radiation levels for SBW. In the CsIX process, it is planned to have a solids filter system followed by the ion exchange sorbent. The grouted waste must remain under 200 millirem per hour (mR/hr) on contact to meet the WIPP acceptance criteria. The calculations show that the UDS have minimal impact. It turns out in the worst case that if 100% of the SBW UDS were to pass through the filters and ion exchange sorbent, the resulting radiation levels only increase about 10 mR to 15 mR per drum.

Appendix D shows the calculation spreadsheets and the MicroShield data sheets. The raw data was taken from Reference 23. The MicroShield data sheets have not been independently verified; however, the calculations were set up as in previous grout drum calculations. The results are summarized as follows:

	SBW-180	SBW-189
Grout without UDS	29.0 mR/hr	118.3 mR/hr
Grout with 100% UDS	39.6 mR/hr	132.1 mR/hr

The large difference between the two tank waste can be attributed to cobalt and europium. If the Co-60, Eu-152, Eu-154,and Eu-155 for SBW-189 are placed at SBW-180 values, the radiation level drop to about 35 mR/hr.

In summary, the UDS does not negatively impact the radiation levels of the grouted waste. There is sufficient margin for operational safety to avoid the 200 mR/hr contact limit.

11.6 SBW Thermal Analysis Results

Thermophysical Properties Research Laboratory of West Lafayette, Indiana, completed a thermal analysis study of the neutralization of sodium-bearing waste [24]. The thermal conductivity and specific heat as a function of pH were measured for both SBW-180 and SBW-189 non-hazardous simulants using sodium hydroxide, calcium hydroxide, and a combination of both neutralization agents. The thermal conductivity does not vary significantly (0.525 to 0.625 W/m·K) over the pH range from 0 to 11 as noted in Figures 13 and 14. The specific heat for both raw simulants is about 3.2 W·s/g·K; then, as the solution is neutralized, the specific heat lowers to about 2.9 W·s/g·K (Figures 15 and 16). In the latter neutralization for both sodium hydroxide and calcium hydroxide, sodium hydroxide was used to neutralize to a pH of about 0.5 molar and then calcium hydroxide was used the remainder of the experiment.

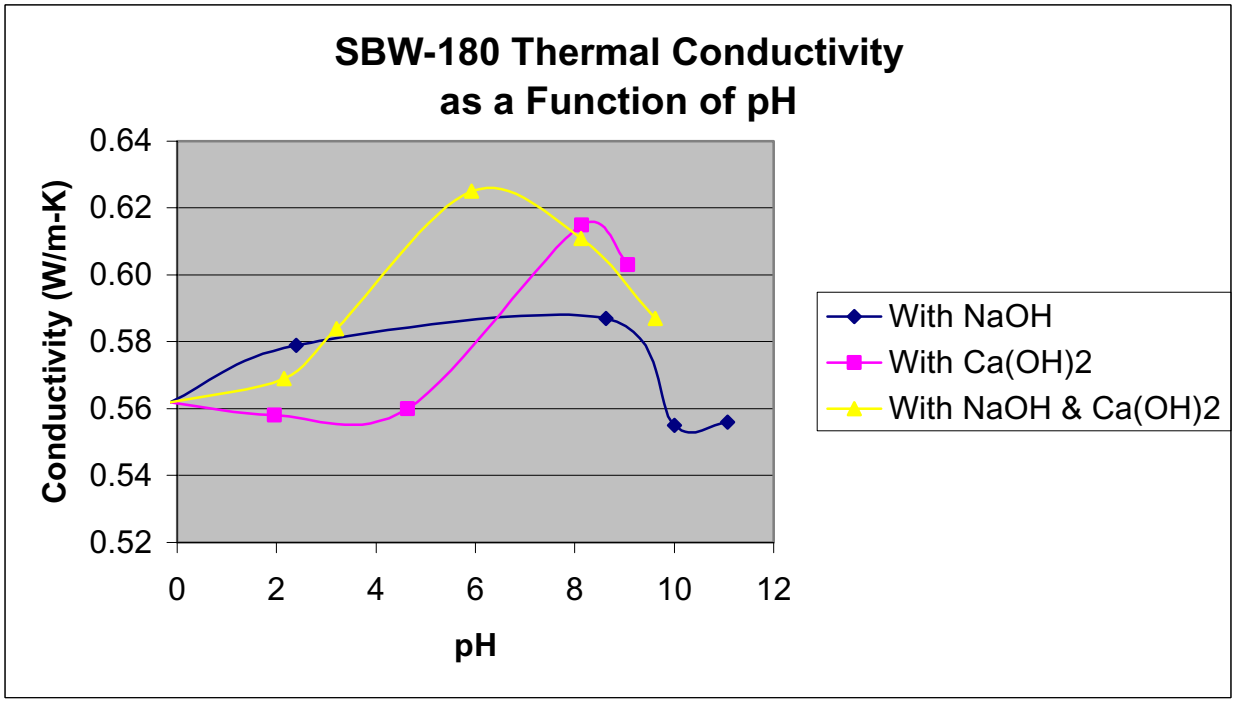

Figure 13. SBW-180 thermal conductivity as a function of pH and neutralization agent (data from TPRL [24]).

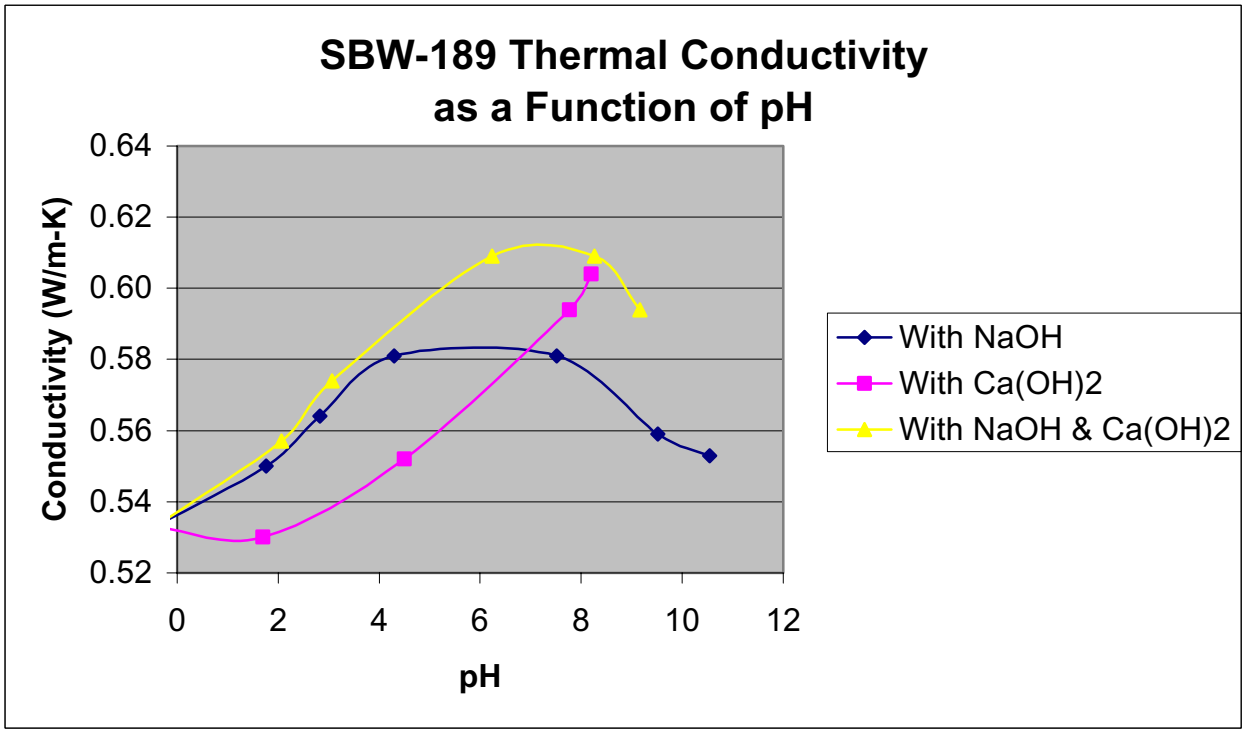

Figure 14. SBW-189 thermal conductivity as a function of pH and neutralization agent (data from TPRL [24]).

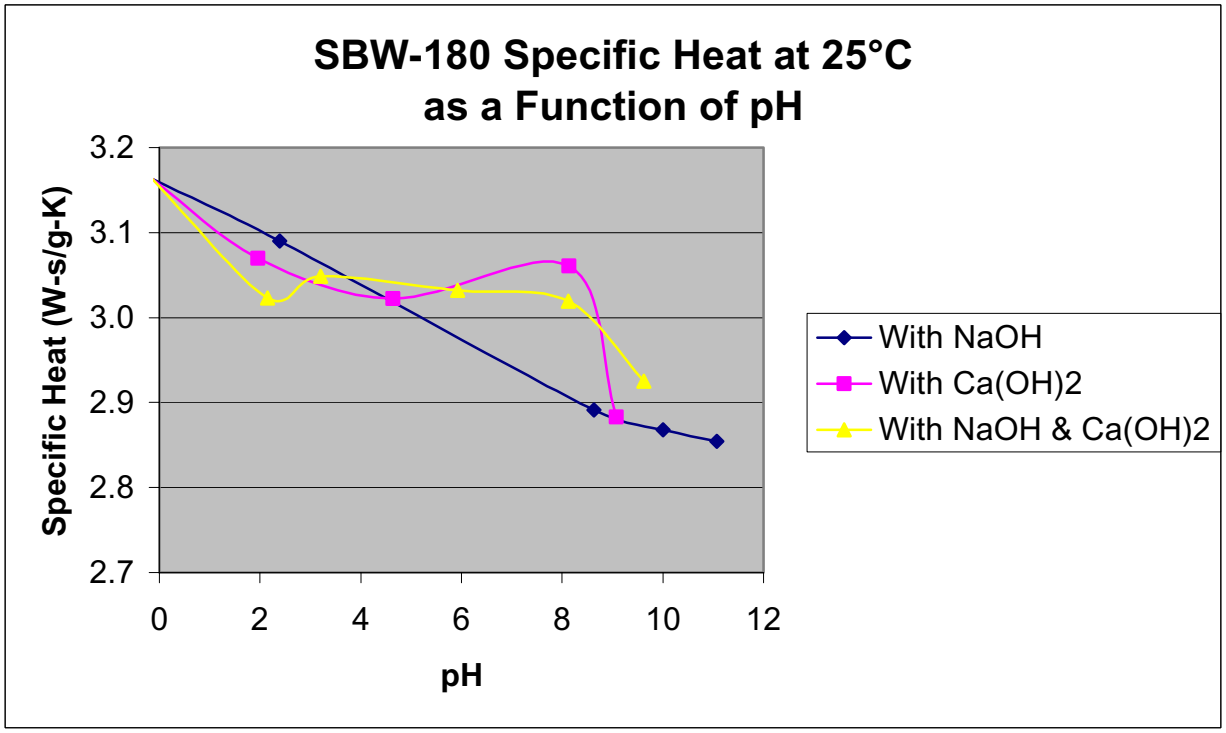

Figure 15. SBW-180 specific heat as a function of pH and neutralization agent (data from TPRL [24]).

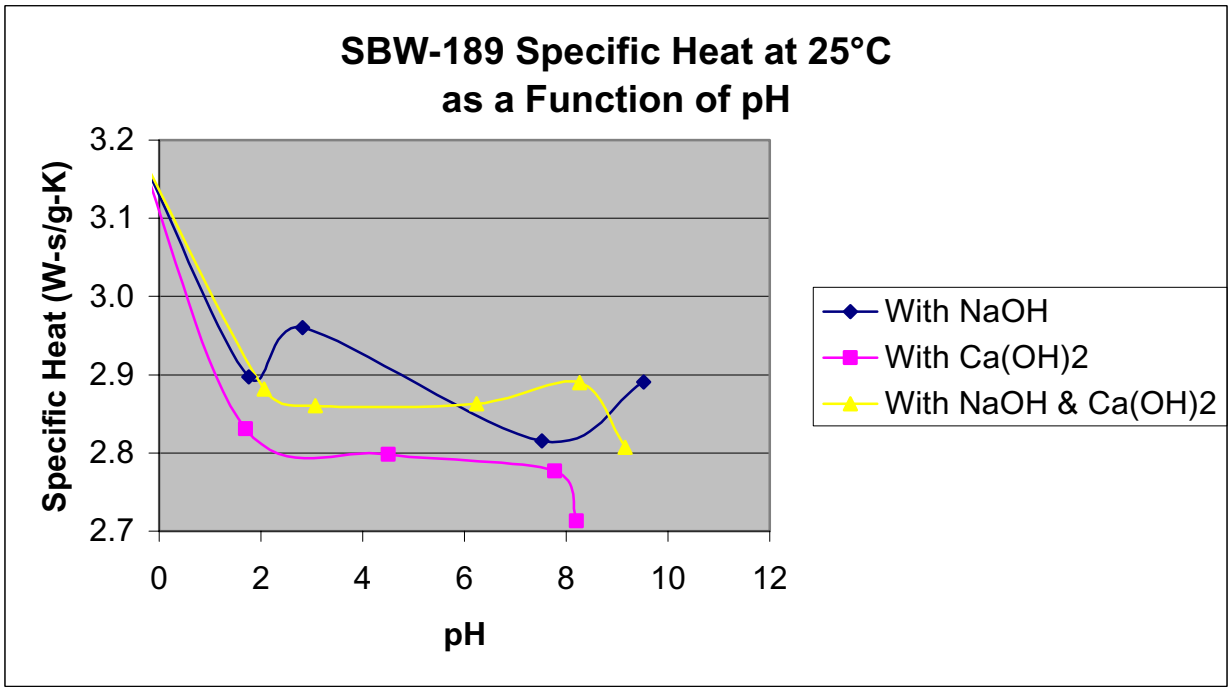

Figure 16. SBW-189 specific heat as a function of pH and neutralization agent (data from TPRL [24]).

12. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

A large part of the report has concentrated on the methodology for developing grouts that are compositionally compatible with the various waste effluents of INTEC. At the hindsight, the problem may appear to be very simple because the only qualifying criterion for disposal in the waste isolation pilot plant is that the grouts be devoid of any bleed liquid. A simple empirical approach of mixing Portland cement and waste streams may then appear to meet the demands for forming a suitable grout. The project was commenced with this intent, where both ad-hoc tests and tests based on statistical design led to results ranging from hardened grouts to unsetting grouts and to grouts with discolored bleed liquids. Owing to uncertainties in the results, the method was progressed towards examining the roles of the various components like the waste stream composition, the slag/cement ratio, and the total proportions of acid neutralizing agents. In view of the nearly four independent components involved in the determination of the grout stability and waste loading, the statistically designed component proportions were plotted on a triangular composition net, which then was used to determine the future course of compositional directions for introducing desirable changes in the grout properties. This method has led to minimizing production of secondary wastes by enabling preparation of grouts with predictable properties. The compositional boundaries for formation of stable grouts are thus determined to span from 100% cement to 50% cement / 50% slag for waste loadings ranging from 55 wt% to 70 wt% (Figures 3 and 4). Within these boundaries the stability of the grout is influenced by yet another factor the acid molarity of the waste effluents.

Noting that the grout properties in the construction industry are related to water / cement ratio, [18, 20, 21] where the liquid, in this case water has a neutral pH, it is natural to consider neutralizing the acidic waste effluents. The three commonly available neutralizing agents NH_4OH, NaOH or $Ca(OH)_2$ were tested for this purpose (Table 4). As the compositions would indicate, ammonium hydroxide is perhaps the most suitable agent, for there are no common cationic effects involved between the neutralizer, and the waste or cement+slag. However, its odor and high vapor pressure, placed stringent restrictions on its use in a normal laboratory setting. As a result the use of this reagent was considered only to explore the mechanism of acidic waste fixation in a grout. Through this important basic experimental exercise, the steps of gellation, liquefaction, and neutral (pH=7) slurry formation became apparent for eliminating the bleed liquid. Of the remaining two neutralizing agents, experiments showed $Ca(OH)_2$ to be more effective than NaOH in the elimination of bleed liquid and in the hardening of grout. Two problems were noted with the use of NaOH, 1. gels formed by direct addition of NaOH to the waste rapidly peptized to become liquid again, which could be an important cause for the occurrence of bleed liquid, and 2. sodium nitrate fibers appeared to grow on the surface during the curing period, due to additional introduction of sodium ions to the already sodium nitrate enriched waste effluents, these nitrate fibers in several grouts seemed to diminish curing, leading to large dimensional changes, cracking and softness. The third approach of $Ca(OH)_2$ addition to the liquid waste effluent seemed to eliminate the second part of the problem but bleed liquid continued to persist in several grouts, once again indicating incomplete hydration reactions due to the interaction of the cement with $Ca(OH)_2$ enriched gel. This problem was eliminated by changing the addition of $Ca(OH)_2$ from directly to the waste effluent to blending it with the cement or cement + slag in powdered form. Subsequent addition of the blended powder to the waste effluent revealed all the mechanistic steps for grout formation (Figures 5 and 6), total elimination of bleed liquid and rapid setting of the grout with minimal dimensional change during curing. Since, the Portland cement itself contains calcium hydroxide, a limited addition of calcium hydroxide was considered necessary to avoid potential prolongation in the hardening of the grout. Preliminary experiments have pointed to an upper limit of about 10 wt% $Ca(OH)_2$. The compressive strength tests (Tables 6 and 7) have also pointed to weakening of the grout with increases in $Ca(OH)_2$. Thus the optimum $Ca(OH)_2$ addition is on the order of 3.5 wt% for formation of desirable grouts.

In order to reduce waste/grout mixing operational complexity, it is desirable to develop a uniform grout formulation. By using the formulation diagrams (Figures 1 - 4) and improving on them in the future, it should be possible to find a common formulation for all SBW effluents, especially after pre-neutralization. Two uniform formulations may be considered: 1) An common general formulation or 2) a common cement powder formulation. For example, the general formulation would be where following neutralization, all SBW wastes would be grouted at a certain waste loading using the same calcium hydroxide to slag to cement ratio. In the second case, the waste loading may change for each waste, but the calcium hydroxide to slag to cement ratio would remain constant. The first method would be preferable, but even the second would reduce complexity. A cost advantage may be found in adopting a uniform formulation technology based on the development of formulation maps (Figures 1 - 4). The results obtained from these formulation maps have successfully been applied for the pilot plant scale up process.

Table 11 summarizes the formulations recommended for further evaluation. The formulations provide a range of processing options such as pre-neutralization and pre-blending all powders prior to mixing with the liquids. Such processes will need to be "tailored" for the process, mixer, and equipment selected. It is noted that blast furnace slag may be left out of SBW grouts; however, when doing so, the grout was thicker. With slag, the SBW grouts were observed to be self-leveling.

Table 11. Formulations recommended for further evaluation.

Waste Steam	Mixing Method	Loading wt%	50% NaOH wt%	$Ca(OH)_2$ Wt%	Slag wt%	Portland Cement wt%	Silica Gel wt%	Viscosity cP	Density g/cm^3
SBW-180	1	72	0	5	0	23		38200	1.42
SBW-180	2	75	0	8.5	0	16.5		20500	1.43
SBW-180	3	72	3	7	6	12			1.47
SBW-180	4	65	6.6				28.4		1.10
SBW-189	1	68	0	5	0	27		27500	1.61
SBW-189	2	68	0	12.4	0	19.6		28900	1.51
SBW-189	3	66	9	7	6	12			1.53
SBW-189	4	57.1	12.9				30.0		1.28
Scrub	2	35	18	1	41.3	4.7		12000	1.70
Scrub	3	54.4	15.6				30.0		1.21
LET&D Acid	2	35		12.4	13.1	39.5			1.96

Mixing Methods: 1 – combine calcium hydroxide and cement and mix in one step
2 – neutralize with calcium hydroxide and then mix in cement and slag
3 – pre-neutralize with sodium hydroxide and mix remaining powders
4 – neutralize with sodium hydroxide and mix with silica gel

Summary of Conclusions and Recommendations:

- SBW-180 can be grouted at 72 wt% to 75 wt%
- SBW-180 can be absorbed on silica gel at 65 wt% following partial neutralization
- SBW-189 can be grouted at 66 wt% to 68 wt%

- SBW-189 can be absorbed on silica gel at 57 wt% following partial neutralization
- Calcine Scrub can be grouted at 35 wt%
- Calcine Scrub can be absorbed on silica gel at 54 wt% after partial neutralization
- The grout formulations were satisfactory in the pilot scale continuous mixer
- Partial neutralization prior to ion exchange followed by grouting is possible
- Vibration and thermal cycling can cause free liquid in near saturated samples; thus, waste loading must be 5% to 10% lower and allow excess silica gel.
- Need to verify formulations "work" with full simulant including RCRA metals
- Need to investigate drum corrosion for the selected formulations

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APPENDIX A

GROUT TEST DATA

50% NaOH wt %	$Ca(OH)_2$ wt %	Slag wt %	Cement wt %	Solution pH	Mix pH	Mix Viscosity at 2.5 rpm cp	Mix Viscosity at 10 rpm cp	7 Day Vicat Depth mm	> 28 Day Vicat Depth mm	Cured Cube Mass g	Cured Density g/cm3	Free Liquid mL	Comp. Strength psi	
	5.0%		27.0%	2.72	9.02	4,900	1,840	50	17	168.9	1.69	0		Concav
	5.0%		17.0%	2.48	8.33	NT	NT	50	50	159.5	1.48	0		Stratifie
	14.0%		18.0%	10.36	10.73	666,000	302,000	3	0	163.1	1.48	0		Very th
	14.0%		8.0%	10.90	11.16	NT	548,000	7	17	145	1.4	0		Extrem
5.0%	5.0%		22.0%	3.28	8.42	142,000	5,120	18	50	180.1	1.56	0		Pulled f
5.0%	5.0%		12.0%	3.08	8.92	NT	1,920	50	50	147.7	86.2	0		Shrunk
	5.0%	9.0%	8.0%	2.54	7.14	NT	1,080	50	50	163.4	1.49	0		
	14.0%	10.0%	8.0%	10.78	11.15	4,800,000	2,060,000	7	0	159.1	1.6	0		Very th
5.0%	5.0%	4.0%	8.0%	3.11	8.89	9,600	2,590	50	50	141.6	1.38	0		Cracks
5.0%	9.0%		8.0%	8.52	8.96	65,900	22,400	20	5	165.1	1.43	0		Pulled f
	5.0%	16.0%	11.0%	2.63	8.66	3,300	1,280	50	50	175.2	1.63	0		
5.0%	9.0%		18.0%	8.92	10.09	204,800	80,500	3	0	161.6	1.52	0		Shrunk
5.0%	9.0%	10.0%	8.0%	8.12	9.40	134,400	53,000	6	0	164.5	1.52	0		Pulled a
5.0%	5.0%	13.7%	8.3%	3.32	8.76	765	5,024	50	50	161.3	1.59	0		Shrunk
5.0%	5.0%	13.3%	8.0%	7.60	9.06	NT	1,040	50	50	162.2	1.51	0		Shrunk
5.0%	5.7%	13.3%	8.0%	6.41	8.40	16,220	6,160	50	50	175.9	1.59	0		Pulled a
	5.0%	13.3%	8.0%	2.54	7.63	NT	1,016	50	50	158.9	1.59	5		Free liq
	5.0%	16.0%	9.6%	2.62	8.65	NT	1,056	50	50	165.2	1.61	0		Layer c
	10.7%	13.3%	8.0%	9.08	10.60	1,587,000	NT	0	0	170.5	1.61	0		Shrunk
	6.4%	16.0%	9.6%	3.94	8.13	9,020	3,520	50	50	170.8	1.59	0		Crack g
1.4%	5.0%	16.0%	9.6%	3.20	8.04	6,750	1,912	50	50	178.1	1.62	6		Free liq
	8.1%	8.5%	12.0%	7.49	8.19	NT	19,400	50	13	172.4	1.5	0		Pulled a
2.5%	8.3%		16.2%	8.53	9.13	134,400	50,400	32	23	164.8	1.49	0		Ideal co
2.2%	7.8%	9.0%	13.0%	8.65	9.30	NT	51,700	21	0	152.6	1.61	0		Sample
2.5%	7.2%	2.2%	10.1%	7.57	8.90	5,630	1,920	50	50	161.4	1.43	0		Starting
2.2%	5.0%	8.5%	12.3%	2.88	8.11	NT	2,440	50	50	168.4	1.57	0		Starting
5.0%	6.4%	6.0%	11.2%	7.47	9.21	8,200	4,000	22	10	140.8	1.39	0		Pulled f
2.5%	8.2%	8.6%	8.1%	8.70	9.22	130,600	44,000	50	5	158.7	1.57	0		Pourab
0.3%	5.3%	16.0%	10.1%	2.61	8.60	NT	1,240	50	50	172	1.6	0		
2.5%	11.5%	3.3%	11.4%	9.31	NT	NT	NT	0	0	146.9	1.48	0		Too thi
2.0%	6.0%	14.4%	8.6%	2.97	8.89	7,620	3,120	50	31	168	1.53	0		Pulled a
2.2%	7.2%	7.8%	11.6%	7.82	8.44	21,500	18,240	50	35	175	1.49	0		Starting
2.2%	7.2%	7.8%	11.6%	7.54	8.01	6,400	20,300	50	50	176.9	1.47	0		
2.2%	7.2%	7.8%	11.6%	8.28	9.12	25,500	13,180	50	27	159.6	1.65	0		Pulled f
2.2%	7.2%	7.8%	11.6%	6.63	8.35	27,140	2,048	22	5	162.5	1.6	0		Shrunk

surement Not Taken

50.0% NaOH wt %	$Ca(OH)_2$ wt %	Slag wt %	Cement wt %	Water wt %	Solution pH	Mix pH	Mix Viscosity at 2.5 rpm cp	Mix Viscosity at 10 rpm cp	7 Day Vicat Depth mm	> 28 Day Vicat Depth mm	Cured Cube Mass g	Cured Density g/cm3	Free Liquid mL	Comp. Strength psi	
	7.6%		23.0%	20.1%	NT	NT	NT	5,760	0	0	121.1	1.17	0	NT	
	3.5%		31.3%	13.1%	NT	NT	NT	6,620	0	0	152.0	1.56	0	NT	
	8.0%		26.0%	10.0%	NT	NT	NT	40,000	0	0	139.6	1.37	0	NT	
	3.5%		27.0%	12.4%	NT	NT	NT	2,240	0	0	126.6	1.33	0	NT	
	1.8%	11.4%	26.6%		NT	NT	NT	35,000	0	1	162.7	1.44	0	1600	
	5.1%		26.2%	6.7%	NT	NT	NT	31,000	0	0	136.0	1.50	0	NT	
	5.0%		30.0%	5.0%	NT	NT	NT	4,320	0	0	NT	NT	0	NT	Cracke
	4.0%	9.6%	22.5%		NT	NT	NT	25,000	0	1	147.2	1.58	0	2500	
	3.0%		30.0%	3.0%	NT	NT	NT	8,800	50	1	NT	NT	0	NT	
	5.8%	8.2%	19.0%		NT	NT	NT	40,000	0	0	142.6	1.32	0	1250	
	3.4%	8.7%	20.3%		NT	NT	NT	20,000	2	0	141.8	1.26	0	NT	
	3.4%	20.3%	8.7%		NT	NT	NT	25,000	0	0	141.0	1.39	0	NT	
	3.4%	14.5%	14.5%		NT	NT	NT	20,000	0	0	1.43.6	1.38	0	1020	
	3.4%	29.0%			NT	NT	NT	25,000	NT	0	NT	NT	0	NT	
	3.0%		29.0%		NT	NT	NT	18,000	5	0	NT	NT	0	NT	Cracke
	3.2%		28.8%		n/a	9.09	49,000	15,200	50	12	193.4	1.48	0	10	Combir
	5.0%		27.0%		n/a	9.38	NT	NT	1	0	189.2	1.44	0	470	Combir
	3.4%	8.6%	20.0%		NT	NT	NT	25,000	0	0	NT	NT	0	1500	
	10.2%	6.5%	15.3%		NT	NT	NT	30,000	0	0	119.1	1.15	0	1390	
	6.8%	7.5%	17.5%		NT	NT	NT	25,000	2	0	120.4	1.21	0	NT	
	7.6%	7.2%	16.8%		NT	NT	NT	30,000	0	0	NT	NT	0	NT	
	5.0%		25.0%		n/a	9.26	NT	NT	6	0	183.5	1.40	0	110	Combir
3.0%	6.0%	9.0%	12.0%		-0.4	9.12	85,800	56,400	50	10	189.7	1.52	0	30	
	5.0%	23.5%			NT	NT	NT	25,000	NT	NT	NT	NT	0	NT	
	5.0%	7.0%	16.5%		NT	NT	NT	25,000	0	0	145.7	1.38	0	NT	
	5.0%	16.5%	7.0%		NT	NT	NT	25,000	0	0	141.7	1.52	0	NT	
	7.2%	6.3%	14.8%		NT	NT	NT	25,000	10	0	NT	NT	0	NT	
10.0%	0.0%		18.0%		2.54	8.89	NT	1,080	50	50	NT	NT	0	NT	
	5.0%		23.0%		n/a	9.16	92,800	38,200	42	15	185.6	1.42	0	10	Combir
	10.0%		18.0%		8.24	8.79	NT	NT	50	6	177.5	1.35	0	20	
	10.0%	9.0%	9.0%		8.66	9.22	NT	NT	2	0	167.2	1.28	0	300	
	10.0%	9.0%	9.0%		NT	NT	NT	NT	0	0	152.4	1.16	0	260	
	10.0%	9.0%	9.0%		8.67	9.22	NT	NT	4	0	173.4	1.19	0	250	
3.0%	6.0%	8.0%	11.0%		-0.10	9.04	61,800	37,500	36	1	190.0	1.49	0	20	
3.0%	10.0%	7.0%	8.0%		0.19	11.05	NT	NT	1	0	172.3	1.31	0	100	
3.0%	10.0%		15.0%		0.42	10.84	NT	NT	4	0	174.6	1.17	0	60	
3.0%	6.0%	7.0%	12.0%		0.29	9.26	87,260	NT	50	50	NT	NT	5	NT	Free liq
	8.2%	19.8%	0.0%		4.98	8.37	14,200	7,870	50	50	178.5	1.36	0	NT	Sponge
	8.2%	17.8%	2.0%		7.32	8.48	9,700	5,020	50	50	174.6	1.33	0	NT	Sponge
	8.4%		17.6%		7.23	8.78	NT	16,880	50	29	183.5	1.40	0	NT	
8.4%			17.6%		2.40	8.47	3,870	1,392	50	50	NT	NT	0	NT	
9.3%			16.7%		2.56	8.69	3,300	1,472	50	50	NT	NT	0	NT	

surement Not Taken

50% NaOH	$Ca(OH)_2$	Slag	Cement	NH_4OH	Solution	Mix	Mix Viscosity at 2.5 rpm	Mix Viscosity at 10 rpm	7 Day Vicat Depth	> 28 Day Vicat Depth	Cured Cube Mass	Cured Density	Free Liquid	Comp. Strength	
wt %	wt %	wt %	wt %	wt %	pH	pH	cp	cp	mm	mm	g	g/cm3	mL	psi	
10.0%			16.0%		2.71	8.90	NT	NT	50	50	NT	NT	0	NT	
0.0%	10.0%		16.0%		8.57	8.92	NT	NT	50	0	181.0	1.38	0	200	
0.0%	10.0%	8.0%	8.0%		8.53	9.06	NT	NT	50	0	170.3	1.30	0	170	
0.0%	10.0%	8.0%	8.0%		NT	NT	NT	NT	6	0	151.2	1.15	0	260	
0.0%	10.0%	8.0%	8.0%		8.65	9.04	NT	NT	10	0	173.4	1.19	0	190	
3.0%	9.2%	6.4%	7.4%		0.10	10.03	NT	NT	1	0	172.3	1.32	0	90	
0.0%	10.1%		15.9%		8.85	9.28	291,800	87,700	46	22	177.6	1.35	0	NT	
	8.5%		16.5%		8.39	8.97	NT	20,506	47	3	187.9	1.43	0	40	
	8.5%	16.5%	0.0%		7.67	8.47	12,370	4,600	50	50	148.6	1.13	0	NT	Small b
	8.5%	14.8%	1.7%		5.64	7.82	15,600	9,500	50	22	176.7	1.35	0	40	Mix thic
	10.3%		13.8%		8.43	8.99	101,500	54,400	50	29	168.6	1.29	0	NT	
	10.3%	10.3%	3.5%		8.56	8.98	90,600	64,200	50	33	175.2	1.34	0	NT	
	8.6%		15.4%		6.98	8.81	NT	16,880	50	36	182.6	1.39	0	NT	
	7.6%	4.8%	11.2%		NT	NT	NT	12,000	50	NT	NT	NT	0	NT	
	8.7%		14.3%		7.42	8.92	30,100	11,520	50	49	185.4	1.41	0	NT	
	11.6%	3.7%	7.7%		NT	NT	NT	35,000	0	NT	NT	NT	NT	NT	
			13.9%	16.1%	NT	NT	NT	7,000	50	NT	NT	NT	NT	NT	
			63.1%	6.9%	NT	NT	NT	40,000	0	NT	NT	NT	NT	NT	

surement Not Taken
nended Formulation

50% NaOH	$Ca(OH)_2$	Slag	Cement	Solution	Mix	Mix Viscosity at 2.5 rpm	Mix Viscosity at 10 rpm	7 Day Vicat Depth	> 28 Day Vicat Depth	Cured Cube Mass	Cured Density	Free Liquid	Comme
wt %	wt %	wt %	wt %	pH	pH	cp	cp	mm	mm	g	g/cm3	mL	
	5.0%		27.0%	-0.24	7.88	NT	NT	50	NT	NT	NT	52	
	5.0%		17.0%	-0.60	5.44	NT	NT	50	NT	NT	NT	79	
	14.0%		18.0%	7.70	9.10	183,000	94,100	1	0	199.1	1.73	0	Pulled fr
	14.0%		8.0%	6.98	7.55	340,000	14,660	50	16	180.3	1.53	0	
5.0%	5.0%		22.0%	1.91	8.13	3,680	1,240	50	NT	NT	NT	26	
5.0%	5.0%		12.0%	1.84	6.02	NT	4,450	50	NT	NT	NT	26	
	5.0%	9.0%	8.0%	-0.68	3.63	NT	4,350	50	NT	NT	NT	31	
	14.0%	10.0%	8.0%	7.59	8.16	100,000	53,300	35	0	194.4	1.62	0	Pulled fr
5.0%	5.0%	4.0%	8.0%	1.70	2.91	NT	NT	50	NT	NT	NT	45	1 day ble
5.0%	9.0%		8.0%	2.70	8.01	3,620	944	50	NT	NT	NT	63	
	5.0%	16.0%	11.0%	-0.44	7.12	4,060	1,600	50	NT	NT	NT	68	
5.0%	9.0%		18.0%	5.28	8.34	14,780	5,360	50	12	190.3	1.55	0	
5.0%	9.0%	10.0%	8.0%	5.71	8.20	12,510	4,720	50	NT	NT	NT	24	
5.0%	5.0%	13.7%	8.3%	1.92	6.62	4,130	2,104	50	NT	NT	NT	31	
5.0%	5.0%	13.3%	8.0%	2.11	6.60	NT	NT	50	NT	NT	NT	50	
5.0%	5.7%	13.3%	8.0%	1.88	6.82	5,890	2,360	50	NT	NT	NT	39	
	5.0%	13.3%	8.0%	-0.59	4.79	7,680	3,500	50	NT	NT	NT	39	
	5.0%	16.0%	9.6%	-0.33	5.31	4,770	2,344	50	NT	NT	NT	60	
	10.7%	13.3%	8.0%	4.34	8.42	NT	NT	50	NT	NT	NT	39	
	6.4%	16.0%	9.6%	1.50	5.64	4,420	1,400	50	NT	NT	NT	68	
1.4%	5.0%	16.0%	9.6%	0.33	5.76	7,100	2,608	50	NT	NT	NT	66	
	8.1%	8.5%	12.0%	1.96	7.35	6,370	2,080	50	NT	NT	NT	58	
2.5%	8.3%		16.2%	2.35	8.34	NT	NT	50	NT	NT	NT	52	
2.2%	7.8%	9.0%	13.0%	2.13	8.00	14,730	3,600	50	NT	NT	NT	21	
2.5%	7.2%	2.2%	10.1%	1.72	5.45	4,000	1,184	50	NT	NT	NT	66	
2.2%	5.0%	8.5%	12.3%	0.46	5.26	5,760	2,736	50	NT	NT	NT	60	
5.0%	6.4%	6.0%	11.2%	2.25	7.86	NT	NT	50	NT	NT	NT	42	1day ble
2.5%	8.2%	8.6%	8.1%	2.38	6.97	3,840	1,440	50	NT	NT	NT	52	
0.3%	5.3%	16.0%	10.1%	-0.23	5.99	4,480	1,360	50	NT	NT	NT	73	
2.5%	11.5%	3.3%	11.4%	7.81	8.56	64,600	20,300	50	0	171.4	1.72	0	
2.0%	6.0%	14.4%	8.6%	1.57	6.98	8,990	2,248	50	NT	NT	NT	66	
2.2%	7.2%	7.8%	11.6%	1.99	6.70	4,210	2,224	50	NT	NT	NT	50	
2.2%	7.2%	7.8%	11.6%	1.91	7.45	5,630	2,112	50	NT	NT	NT	34	
2.2%	7.2%	7.8%	11.6%	1.99	7.32	NT	NT	50	NT	NT	NT	66	
2.2%	7.2%	7.8%	11.6%	2.16	6.98	6,590	2,712	50	NT	NT	NT	60	

surement Not Taken

50% NaOH	$Ca(OH)_2$	Slag	Cement	Solution	Mix	Mix Viscosity at 2.5 rpm	Mix Viscosity at 10 rpm	7 Day Vicat Depth	> 28 Day Vicat Depth	Cured Cube Mass	Cured Density	Free Liquid	Comp. Strength	
wt %	wt %	wt %	wt %	pH	pH	cp	cp	mm	mm	g	g/cm3	mL	psi	Commer
	3.6%	13.6%	31.8%	NT	NT	NT	35,000	2	NT	203.2	1.73	0	2510	
	1.4%	13.5%	31.6%	NT	NT	NT	20,000	1	NT	213.1	1.70	0	NT	
	4.3%	10.3%	24.0%	NT	NT	NT	35,000	1	NT	189.2	1.64	0	1230	
8.0%	14.0%	9.0%	7.0%	-0.28	10.54	NT	NT	0	0	198.3	1.51	0	360	Combine
8.0%		10.0%	20.0%	-0.38	7.80	5,280	1,928	50	50	NT	NT	NT	NT	Free liqu
	6.8%	6.7%	22.3%	NT	NT	NT	40,000	0	NT	NT	NT	NT	NT	
8.0%	12.0%	9.0%	7.0%	-0.46	9.67	NT	NT	0	0	195.8	1.52	0	230	Combine
	12.1%		22.0%	7.45	8.24	74,200	32,300	50	2	202.8	1.55	0	590	some fla
	12.1%	22.0%	0.0%	7.74	7.67	36,800	NT	50	50	199.7	1.52	0	NT	very thic
	12.1%	19.8%	2.2%	7.64	8.11	32,100	15,330	50	50	192.1	1.46	0	NT	Thin flim
9.0%	10.0%	8.0%	7.0%	-0.66	9.34	NT	NT	1	0	195.4	1.50	0	220	Combine
9.0%	10.0%	7.0%	8.0%	-0.34	10.20	NT	NT	1	0	184.4	1.41	0	170	Clay-like
9.0%	10.0%		15.0%	-0.51	9.82	NT	NT	1	0	191.3	1.28	0	60	Clay-like
9.0%	6.0%	7.0%	12.0%	-0.54	8.64	NT	NT	30	1	2020.6	1.30	0	170	
9.0%	7.0%	6.0%	12.0%	-0.55	8.85	171,500	141,600	1	0	191.7	1.53	0	120	Pre-neut
	12.4%		19.6%	7.87	8.55	47,700	28,960	50	0	197.6	1.51	0	350	
	12.4%		19.6%	6.65	8.13	41,600	24,240	50	2	199.3	1.52	0	390	some fla
	12.4%	19.6%	0.0%	7.48	8.31	49,000	33,000	50	50	199.7	1.52	0	NT	some fla
	12.4%	17.6%	2.0%	7.64	8.06	27,400	12,000	50	50	181.5	1.38	0	NT	Thin flim
	3.2%	14.4%	14.4%	NT	6.32	20,200	8,160	50	40	181.4	1.39	0	NT	even sur
	9.6%	11.2%	11.2%	NT	5.19	NT	9,680	50	50	184.4	1.33	0	NT	soft
	3.2%	3.2%	25.6%	NT	8.31	NT	20,400	12	1	204.0	1.57	0	NT	even sur
	3.2%	6.4%	22.4%	NT	7.94	NT	3,000	30	1	207.9	1.60	0	NT	even sur
	3.2%	9.6%	19.2%	NT	7.64	NT	7,100	50	2	193.3	1.48	0	NT	even sur
	6.4%	3.2%	22.4%	NT	8.24	NT	65,760	4	1	194.5	1.49	0	NT	whitish s
	12.8%	19.2%		NT	NT	NT	27,000	7	0	NT	NT	NT	NT	
	0.0%	16.0%	16.0%	NT	4.38	20,500	12,560	50	50	181.0	1.40	0	NT	even sur
	12.8%		19.2%	NT	NT	NT	35,360	0	0	NT	NT	NT	NT	
	12.8%	9.6%	9.6%	NT	8.45	72,600	52,400	24	1	172.6	1.34	0	NT	No visibl
	12.8%		19.2%	NT	8.41	NT	35,360	13	1	168.1	1.29	0	NT	grey grou
	7.4%	7.6%	17.0%	NT	NT	NT	35,000	7	NT	NT	NT	0	880	
	0.0%		32.0%	NT	8.02	NT	3,000	33	1	192.5	1.47	0	NT	concave
	3.2%		28.8%	NT	8.36	NT	12,880	4	1	192.5	1.48	0	NT	Grey Gro
	3.2%	19.2%	9.6%	NT	7.29	NT	15,000	50	50	193.2	1.48	0	NT	very soft
	12.8%	14.4%	4.8%	NT	7.58	NT	14,000	50	50	174.3	1.34	0	NT	paste lik
	6.4%	12.8%	12.8%	NT	5.84	NT	12,800	50	50	180.0	1.38	0	NT	harder th
	8.0%	8.0%	16.0%	NT	NT	NT	NT	4	0	184.2	1.41	0	270	Combine
	8.0%	8.0%	16.0%	1.34	7.64	6500	1808	50	NT	NT	NT	25	NT	Added C
	8.0%	8.0%	16.0%	NT	8.17	176300	NT	1	1	199.4	1.52	0	420	Combine
	12.4%	14.7%	4.9%	8.20	8.68	34,600	18,800	50	0	196.3	1.50	0	920	
	3.2%		28.8%	NT	8.15	45800	21600	10	1	208.6	1.59	0	130	
	5.0%		27.0%	NT	8.31	68800	27520	4	0	210.1	1.60	0	250	
9.0%	9.2%	6.4%	7.4%	-0.48	8.62	NT	NT	0	0	189.4	1.44	0	160	Clay-like

50% NaOH	$Ca(OH)_2$	Slag	Cement	NH_4OH	Solution	Mix	Mix Viscosity at 2.5 rpm	Mix Viscosity at 10 rpm	7 Day Vicat Depth	> 28 Day Vicat Depth	Cured Cube Mass	Cured Density	Free Liquid	Comp. Strength	
wt %	wt %	wt %	wt %	wt %	pH	pH	cp	cp	mm	mm	g	g/cm3	mL	psi	Commer
	12.8%	17.2%			7.78	7.97	49,900	NT	50	50	185.0	1.41	0	NT	
	12.8%	15.5%	1.7%		7.64	8.05	27,100	13,410	50	50	188.2	1.44	0	NT	
9.0%	7.0%	6.0%	7.0%		-0.54	7.99	58,200	40,240	37	10	193.4	1.46	0	10	Combine
	5.0%	7.0%	16.4%		NT	NT	NT	10,000	40	NT	NT	NT	NT	NT	
	5.0%	16.4%	7.0%		NT	NT	NT	25,000	40	NT	NT	NT	NT	NT	
			7.6%	22.4%	NT	NT	NT	6,000	50	NT	NT	NT	NT	NT	
			60.4%	9.6%	NT	NT	NT	40,000	0	NT	226.7	1.81	NT	NT	

surement Not Taken
nended Formulation

50% NaOH wt %	$Ca(OH)_2$ wt %	Slag wt %	Cement wt %	NH_4OH wt %	Solution pH	Mix pH	Mix Viscosity at 2.5 rpm cp	Mix Viscosity at 10 rpm cp	7 Day Vicat Depth mm	> 28 Day Vicat Depth mm	Cured Cube Mass g	Cured Density g/cm3	Free Liquid mL	Comp. Strength psi	Comme
			58.0%	12.0%	NT	NT	NT	40,000	0	0	205.6	1.75	NT	NT	
16.0%	1.0%	46.7%	5.3%		12.79	13.28	NT	NT	1	0	234.0	1.78	0	1640	
17.0%	1.0%	44.1%	4.9%		12.16	12.68	48,000	28,880	1	0	229.5	1.75	0	1520	
17.5%	1.0%	42.7%	4.8%		12.13	12.67	53,400	16,400	7	0	227.1	1.73	0	1440	
18.0%	1.0%	41.3%	4.7%		12.03	12.44	35,200	12,000	7	0	223.4	1.70	0	1280	
18.0%	1.0%	41.3%	4.7%		NT	NT	NT	NT	NT	0	198.2	1.92	0	1080	Added H
19.3%	1.0%	38.0%	4.2%		11.98	12.56	26,600	12,220	9	0	217.5	1.66	0	1090	Good m
20.7%	1.0%	34.5%	3.8%		10.70	11.34	NT	140,000	14	1	183.5	1.48	0	20	
20.7%	1.0%	0.0%	38.3%		10.15	10.51	1,459,000	NT	1	1	192.4	1.47	0	750	Thick --
23.2%	1.0%	32.3%	3.5%		12.82	13.00	NT	NT	23	1	193.3	1.47	0	NT	Good m
			45.6%	14.4%	NT	NT	NT	26,000	1	0	182.6	1.46	NT	NT	
		45.6%		14.4%	NT	NT	NT	19,600	5	0	NT	NT	NT	NT	
		31.9%	13.7%	14.4%	NT	NT	NT	8,960	50	NT	NT	NT	NT	NT	
		13.7%	31.9%	14.4%	NT	NT	NT	12,000	1	0	136.3	1.72	NT	NT	
22.0%	1.0%	31.1%	3.5%		10.50	NT	38,100	7,600	18	17	193.6	1.48	0	NT	Thixotro
22.0%	1.0%	0.0%	34.6%		10.17	10.65	1,730,000	584,000	6	0	204.2	1.56	0	470	Thick --
22.0%	1.0%	31.0%	3.5%		11.44	12.27	NT	NT	22	16	200.7	1.53	0		Good m
22.2%	1.0%	30.4%	3.4%		9.97	11.18	NT	NT	50	49	201.5	1.54	0	NT	Thicken
22.7%	1.0%	29.0%	3.2%		10.44	11.14	4,300	10,080	50	50	197.2	1.50	0	NT	
22.7%	1.0%	0.0%	32.3%		11.80	12.40	23,230	3,710	5	1	211.6	1.61	0	50	Pourabl
22.7%	1.0%	16.1%	16.1%		12.01	12.49		816	40	2	199.8	1.52	10	490	Very liq
23.3%	1.0%	27.7%	3.1%		10.80	11.53	7,940	2,944	50	35	191.7	1.46	0	NT	
23.3%	1.0%		30.7%		11.71	12.41	7,680	5,560	15	3	210.1	1.60	0	40	
23.3%	1.0%	15.4%	15.4%		11.46	12.32	NT	NT	44	2	195.6	1.49	10	410	Very flu
	14.0%	9.0%	7.0%		NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	Unable
			2.0%	28.0%	NT	NT	NT	3,000	50	NT	NT	NT	NT	NT	

surement Not Taken
nended Formulation

APPENDIX B

SILICA GEL TEST DATA

CsIX Silica Gel Variation Tests using WM-180 SBW Non-Hazardous Simulant

Matrix Sample	Liquid Waste wt %	50% NaOH wt %	Silica Gel wt %	pH after NaOH & before gel	Final Density g/cm3	Free Liquid mL	Comments
SG-V-1	72.0%	0.0%	28.0%	-0.53	1.17	0	
SG-V-2	72.0%	5.0%	23.0%	1.92	NT	37	Sample turned orange color
SG-V-3	76.0%	0.0%	24.0%	-0.53	1.24	0	
SG-C1-1	76.0%	5.0%	19.0%	NT	NT	73	Sample turned orange color
SG-C1-2	74.0%	0.0%	26.0%	-0.53	1.21	0	
SG-C1-3	74.0%	5.0%	21.0%	1.92	NT	47	Sample turned orange color
SG-C1-4	72.0%	2.5%	25.5%	-0.23	1.21	0	
SG-C1-5	76.0%	2.5%	21.5%	-0.29	NT	42	
SG-C2-1	74.0%	2.5%	23.5%	-0.27	NT	27	
SG-C2-2	74.0%	2.5%	23.5%	-0.28	NT	33	
SG-C2-3	74.0%	2.5%	23.5%	-0.23	NT	27	
SG-C2-4	74.0%	2.5%	23.5%	-0.22	NT	30	
Samples -- Neutralized over pH of 2							
SG-65-1 *	65.0%	6.6%	28.4%	2.01	1.10	0	

NT = Measurement Not Taken
* Recommended Formulation

CsIX Silica Gel Variation Tests using WM-189 SBW Non-Hazardous Simulant

Matrix Sample	Liquid Waste wt %	50% NaOH wt %	Silica Gel wt %	pH after NaOH & before gel	Final Density g/cm3	Free Liquid mL	Comments
S89-V-1	72.0%	0.0%	28.0%	-0.19	1.16	0	
S89-V-2	72.0%	5.0%	23.0%	0.44	NT	30	
S89-V-3	76.0%	0.0%	24.0%	-0.12	1.42	50	Free liquid during thermal cycle
S89-C1-1	76.0%	5.0%	19.0%	-0.50	NT	65	
S89-C1-2	74.0%	0.0%	26.0%	0.17	1.23	0	
S89-C1-3	74.0%	5.0%	21.0%	-0.13	NT	50	
S89-C1-4	72.0%	2.5%	25.5%	0.57	1.16	0	
S89-C1-5	76.0%	2.5%	21.5%	-0.01	NT	50	
S89-C2-1	74.0%	2.5%	23.5%	-0.02	NT	30	
S89-C2-2	74.0%	2.5%	23.5%	-0.80	NT	80	
S89-C2-3	74.0%	2.5%	23.5%	-0.67	NT	65	
S89-C2-4	74.0%	2.5%	23.5%	-0.73	NT	5	
Samples -- Neutralized over pH of 2							
S89-S70-1	53.8%	16.2%	30.0%	4.28	1.37	0	
S89-S70-2 *	57.1%	12.9%	30.0%	2.55	1.28	0	

NT = Measurement Not Taken
* Recommended Formulation

Silica Gel Absorption Tests using NWCF Scrubber Non-Hazardous Simulant

Sample	Liquid Waste wt %	50% NaOH wt %	Silica Gel wt %	pH after NaOH & before gel	Final Density g/cm3	Free Liquid mL	Comments
NS-65-2	65.0%	0.0%	35.0%	NT	0.94	0	
NS-70-2	70.0%	0.0%	30.0%	NT	1.02	0	
NS-75-2	75.0%	0.0%	25.0%	NT	1.28	0	
Samples -- Neutralized over pH of 2							
NS-65-3	50.5%	14.5%	35.0%	2.46	0.95	0	
NS-70-3 *	54.4%	15.6%	30.0%	2.43	1.21	0	
NS-75-3	58.2%	16.8%	25.0%	2.30	1.40	0	

NT = Measurement Not Taken
* Recommended Formulation

APPENDIX C

ABSORPTION OF NWCF SCRUBBER SOLUTION ONTO CALCINE

Absorption of NWCF Scrubber Solution onto Calcine

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1. Executive Summary

If the New Waste Calciner Facility were used for sodium-bearing waste treatment, one candidate method for disposal of the calciner scrubber solution is absorption onto the calcine. The calcine would then be sent to the Waste Isolation Pilot Plant. Laboratory scale testing was completed to evaluate the placement of scrubber solution on calcine. Varying amounts of scrub simulant were absorbed on pilot-plant calcine to observe calcine absorbency and morphology and any reactions. It was noted that the acidic scrub caused an exothermic reaction and observable gas was evolved. A hard crust was formed where the calcine and scrub reacted. It was concluded that up to 10 weight percent scrub may be added to the calcine before free liquids occur. Due to these effects, process engineers and designers must evaluate the results and determine feasibility and whether further testing is warranted.

2. Table of Contents

3. Introduction/Background

3.1. Previous history and prior data

Operating the calciner to treat the remaining one million gallons of sodium-bearing waste (SBW) liquid is one of the options being considered for removing all radioactive liquid waste from the Idaho Nuclear Technology and Engineering Center (INTEC) Tank Farm. In the past the calciner scrubber blow down solutions were recycled back to the tank farm without any other treatment for disposal. Absorption on calcine for waste disposal of calciner scrubber blow down solutions that would be produced from running the calciner to process the liquid SBW stored at the tank farm is one of the alternatives being studied during FY03. The scrub volume is estimated to be small compared to the overall volume of calcine. The advantage of this volume difference would be the ability to take time to disperse small quantities (almost "noise level" both as it affects composition / characterization and hopefully rheology) of scrub solution onto much larger volumes of calcine. In this sense we would be more interested in the "low end" tests vs. information at the edge of visible free liquid.

It is assumed that this treatment option incorporating calciner scrub solution absorbed onto calcine would be performed only during the successful operation of the NWCF calciner to treat the remaining liquid stored at INTEC Tank Farm. It is also assumed that the scrub solution would be accumulated or stored until calcine product is available for batch absorption into the shipping containers.

3.2. Purpose and scope

One of the candidate technologies for disposal of calciner scrub solutions is absorption onto calcine. This initial evaluation simply involves determining the absorbency and morphology of different calcines for possible disposal of New Waste Calcining Facility (NWCF) scrubber blow down solutions. In this case, the final solid product would be remote-handled transuranic (TRU) waste, destined for disposal at the Waste Isolation Pilot Plant (WIPP).

The main purpose of this laboratory scale testing is to determine the amount of calciner scrub simulant that can be absorbed onto calcine if the calcine is packaged for transportation to WIPP without any other treatment. The composition of the calciner scrub simulant is shown in Table 1. This testing will primarily determine if this is a feasible means for disposal of this proposed waste stream. The morphology of the calcine after loading with scrub solution and the maximum waste loading without free liquid breakthrough will be monitored. Other observations such as volume changes, gas and heat generation, that are useful in evaluating the results of these laboratory scale tests will be documented. Because the waste is expected to be classified as TRU and can be disposed of to WIPP, it does not need to meet RCRA limits for toxic metals. Therefore, non-hazardous simulants will be used for this testing and toxicity characteristic leaching procedures (TCLP) will not be performed before disposal of any wastes.

Tasks for later consideration, pending the outcome of the initial testing, include evaluation of gas generated from scrub solution absorption onto calcine, the effects of pre-neutralization of scrub before absorption onto calcine, different methods of scrub application (e.g. spraying

vs. a small stream or drops), and the effects of mechanical mixing the calcine and scrub solution together.

3.3. Test objectives/Evaluation criteria

1. The immediate objective of this laboratory scale testing is to demonstrate how much calciner scrub solution could be absorbed if combined with calcined solids. Testing will be conducted to determine a maximum waste loading without any free liquid breakthrough and the affect on the calcine morphology.

2. Determine whether absorption on calcine is technically feasible.

3. Document the calcine morphology (e.g. agglomeration, flow properties, swelling) during absorption.

4. Measure heat generation with a thermocouple.

5. Determine whether additional testing, such as measurement of gas generation rate is needed.

Besides the limitations of any laboratory-scale testing, it is believed that the levels of success realized in this testing can at least be proven for a full-scale system.

4. Theory/Approach

The general understanding of the calcine for this testing is that it is porous to some unknown extent. The macropores are likely to be connected, while the micropores are probably discrete. So the extent of absorption is unknown and the affect on the calcine solid is also unknown. It may dissolve the calcine. It may react chemically to release gas as in an acid-base reaction. Chemical reactions may cause gas evolution (i.e. H_2) or reactions near the surface could cause swelling, (volume changes), and hence pore closure. Absorption reactions could release heat that would cause waste evaporation (gas generation). When subjected to heat or pressure, the liquid may be ejected from the pores due to gas formation in the particles.

For the purpose of this test plan, it is expected that the calcine solids will be able to absorb and retain the liquid calciner scrubber solutions. Also, it is assumed the absorbed liquid solution will permeate uniformly throughout the solid calcine absorbate.

The initial testing will involve measuring the initial diameter and image analysis using microscopes equipped with digital cameras. The scrub simulant will be added to a flask or graduated cylinder containing the calcine for each test. Waste loadings of the liquid simulant will range from 3 wt%, 5 wt%, then in 5 wt% increments up to 25 wt%, or as needed to push the upper waste loading range to find the free liquid breakthrough point. For the initial test with a waste loading of 25 wt% a thermocouple will be in place to measure any heat generation and the container will be equipped with a detection system by water displacement to observe any gas evolution. At the end of testing, the calcine will be removed and measured to compare

with the initial images and diameter size. Any other visual observations of the calcine morphology (e.g. agglomeration, flow properties) will also be made.

No analysis of each test will be needed to determine the maximum waste loading. The visual observations of each test will be used to determine the morphology of the calcine after absorbing the calciner scrub solution.

5. Experimental

5.1. Facility/Equipment Description

The projected test composition of NWCF scrubber blow down solution, not including the radioisotopes, is presented in Table 1.[1] Also included in Table 1 is the composition of the simulant that will be used in this testing. After making up the simulant it will be analyzed by INTEC-Analytical Laboratory Department (ALD) to ensure that it adequately represents the targeted NWCF scrubber solution composition. Table 2 contains the minimum and maximum expected concentrations of the various simulated SBW calcine compositions for this testing. The absorption of scrub solution by calcine will be conducted in INTEC laboratories 111 or 113. Laboratory equipment required for this testing includes: flasks, beakers, graduated cylinders, thermocouples, tubing, scales, wash bottles, etc.

A typical experimental setup is shown as Figure 1. About 300 ml of Run SBW-HT-10 pilot plant calcine simulant will be used as the absorbent for each test. For all of the tests, the calciner scrub simulant of one liter will be made up in a laboratory hood and stored in a poly carboy. After the flask is filled with the right amount of calcine and the scrub simulant is loaded on top, a stopper containing a thermocouple will seal the top of the flask. The thermocouple needs to be located in the flask where it can record any heat generated during testing. The flask should also contain a connection near the top for some small clear tubing. The other end of the tubing is placed inside a supported upside down graduated cylinder that is set inside a beaker of water. The graduated cylinder needs to be filled as full as possible with water by pulling the air out through the tubing. Then pull the end of the tubing down near the opening of the cylinder so that any gas generated during testing will become trapped inside the cylinder to indicate any gas evolution.

For testing not concerned with measuring gas evolution, a simpler setup using just a graduated cylinder, that is more representative of the proposed shipping canisters, would be used to absorb the scrub solution onto the calcine.

Following the addition of simulant to the calcine, the samples will be allowed to set for about 3 days or until any chemical reactions are complete. If free liquid is noted in any sample, that sample will be judged unacceptable. Acceptable samples will then undergo the thermal cycle test [2] followed by the paint filter test,[3] again to watch for the development of any free liquid.

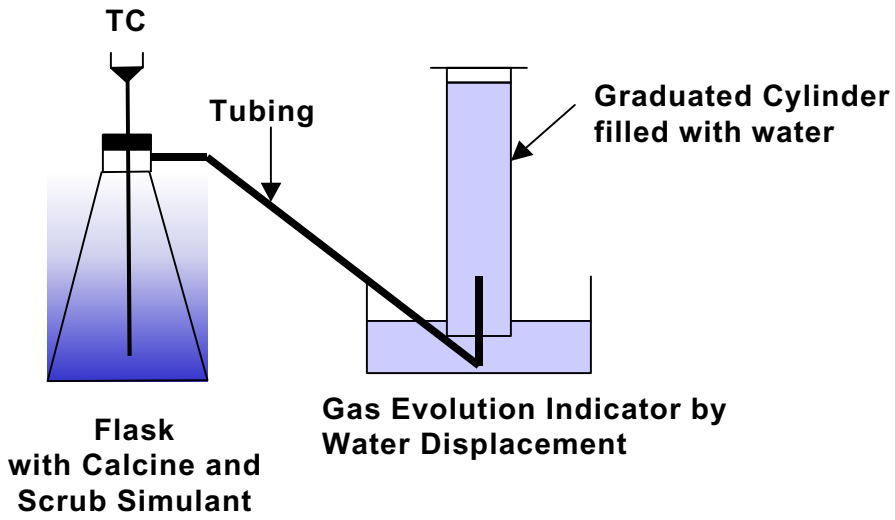


Figure 1. Scrub Absorption onto Calcine Test Apparatus

Table 1. INTEC NWCF Scrubber Blow Down Solution from EDF-3387 and Simulant for Testing

Element	Projected Composition (M)	Test Simulant Concentration (M)
Acids (H+)	2.33000	2.3300
Aluminum (Al)	1.56000	1.5600
Arsenic (As)	0.00009	0
Barium (Ba)	0.00002	0
Boron (B)	0.00542	0.0054
Cadmium (Cd)	0.00097	0
Calcium (Ca)	0.05040	0.0504
Chloride (Cl)	0.03810	0.0381
Chromium (Cr)	0.00173	0
Cobalt (Co)	0.00001	0
Copper (Cu)	0.00016	0.0002
Fluoride (F)	0.08880	0.0888
Gadolinium (Gd)	0.00003	0
Iron (Fe)	0.01220	0.0122
Lead (Pb)	0.00035	0
Lithium (Li)	0.00008	0
Magnesium (Mg)	0.00432	0.0043
Manganese (Mn)	0.00428	0.0043
Mercury (Hg)	0.21000	0
Molybdenum (Mo)	0.00042	0.0004
Nickel (Ni)	0.00073	0.0007
Nitrate (NO_3)	8.24000	8.2400
Phosphate (PO_4)	0.03100	0.0310
Potassium (K)	0.07900	0.0790
Ruthenium (Ru)	0.00003	0
Selenium (Se)	0.00002	0

Element	Projected Composition (M)	Test Simulant Concentration (M)
Silicon (Si)	0.03680	0.0368
Silver (Ag)	0.00004	0
Sodium (Na)	0.60300	0.6030
Strontium (Sr)	0.00003	0
Sulfate (SO_4)	0.02410	0.0241
Vanadium (V)	0.00001	0
Zinc (Zn)	0.00025	0.0003
Zirconium (Zr)	0.01860	0.0186

Table 2. Sodium Bearing Waste Simulant Calcine Compositions (mass%).

Species	Minimum Expected Conc.	Maximum Expected Conc.	SBW-HT-10 Conc.		Species	Minimum Expected Conc.	Maximum Expected Conc.	SBW-HT-10 Conc.
Al_2O_3	45.49	90.90	61.2		HgO	0.00	2.50	0.0
B_2O_3	0.24	8.65	0.3		MoO_3	0.00	0.04	0.0
CdO	0.02	0.36	0.1		NiO	0.00	0.06	0.0
CaF_2	0.35	5.30	2.8		KNO_3	0.38	4.17	1.6
CaO	0.74	4.66	2.3		K_2O	0.53	4.40	3.0
$Ca_3O_8P_2$	0.01	1.04	0.2		NaCl	0.16	0.91	0.8
CrO_3	0.07	0.27	0.2		$NaNO_3$	2.97	27.73	10.0
Fe_2O_3	0.30	1.19	0.9		Na_2O	2.36	25.10	13.2
MnO_2	0.00	0.86	0.7		Na_2SO_4	0.31	3.55	2.3
$HgCl_2$	0.00	0.70	0.0		ZrO_2	0.00	2.69	0.6

6. Results

Initial testing involved selecting a simulated calcine to use for NWCF scrub absorption. SBW-HT-10 a pilot-plant calcine is expected to represent any future calcine compositions and is shown in Table 2. This calcine was made up from a WM-185/ANN blend and has an aluminum to alkali metal ratio (Al/Na=K mol/mol) of 1.86.[4] This run of SBW-HT-10 also had a product to fines wt. ratio of 2.13. Digital images of several SBW-HT-10 product particles were taken using either a Zeiss Axioplan or Olympus SZH10 microscope with Olympus DP11 digital camera system. Figures 2 and 3 show the images taken of the SBW-HT-10 product and fines material used for testing. The color in Figure 2 is not correct, because of the microscope used to take the digital image. The color in Figure 3 is correct with light tan fines and dark brown calcine particles. Appendix A contains additional images to illustrate size and several product particles whole and sectioned to illustrate the structure of the calcine before and after testing.

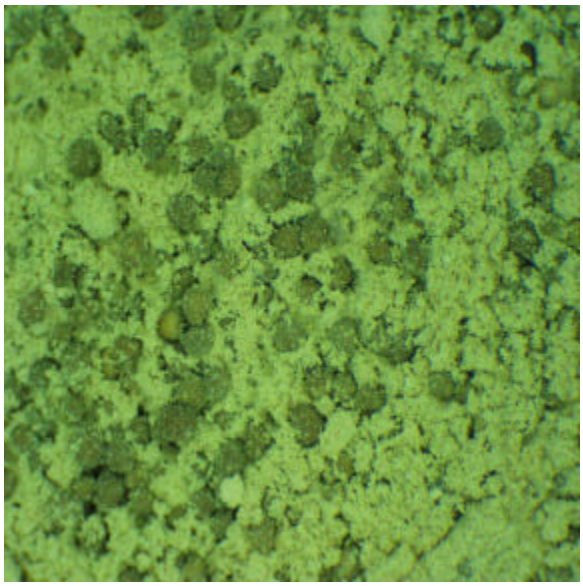


Figure 2. Product and Fines at 10X

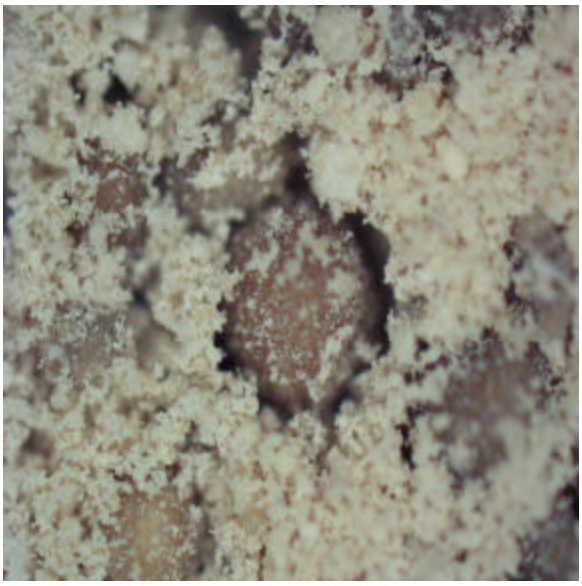


Figure 3. Product and Fines at 50X

Some small samples were tried first to see the effects of adding the scrub solution on top of the calcine or adding the calcine on top of the scrub solution. In either case the scrub solution could only be absorbed so far before a hardened crust developed and prevents any more solution to be absorbed by the calcine. Bubbling occurred in the solution as it started to seep down into the calcine. The absorption rate was very slow and at a high waste loading, mixing was needed to permit absorption of the free liquid. Figure 4 illustrates two cases of scrub addition. On the left scrub solution was added on top of the calcine. At the right, calcine was added to the scrub. Notice that a void formed between the calcine and scrub when the calcine was added on top of the scrub solution. When the scrub solution and calcine solids were completely mixed together to remove them from the flask and exposed to air, they readily dried.

Figure 4. 100 ml Flasks with Scrub absorbed onto Calcine (left) and Calcine absorbed onto Scrub (right).

The next set of tests involved using 100-ml beakers with 3, 5, 10, 15, and 20 wt% loadings of simulated NWCF scrub solution absorbed onto SBW-HT-10 calcine. The 100-ml beakers with a 2-inch diameter are representative of the shipping canisters with a 2-foot diameter. About 20 grams of calcine were used for all of the samples in this test. As noted in Figure 5, the 3, 5, and 10 wt% loaded samples of scrub did not completely cover the top of the calcine in the beakers. The 15 and 20 wt% loaded samples contained enough scrub to completely cover the top surface of the calcine, but had to be tilted to spread out the thick liquid solution of scrub. The beakers were then covered and sealed with a wax parafilm to prevent any evaporation effects. After two and a half hours there was not any free liquid remaining on the 3 and 5 wt% loaded samples, while the rest of the samples still contained free liquid especially around the edges of the beakers. After twenty-four hours only the 20 wt% loaded sample still had a little free liquid around the edges of the beaker. Mass balances were recorded and are shown in Table 3. Also digital images where taken of each sample from this test and are shown in Figure 5 and in Appendix B. The fines of the calcine seem to dissolve into the scrub solution, while the product particles are unaffected as shown in the microscopic digital images.

Table 3. Mass balances from different waste loadings of scrub absorbed onto calcine.

	Scrub Solution (g)	SBW Calcine (g)	Waste Loading (wt%)	Mass Loss (%)
3wt%Scrub/97wt% Calcine	0.672	20.838	3.12	0.6
5wt%Scrub/95wt% Calcine	1.047	20.023	4.97	0.5
10wt%Scrub/90wt% Calcine	2.240	20.010	10.07	0.8
15wt%Scrub/85wt% Calcine	3.554	20.213	14.95	0.8
20wt%Scrub/80wt% Calcine	5.156	20.346	20.22	0.7

Figure 5. 100-ml beakers of 3, 5, 10, 15 and 20 wt% loading of scrub absorbed onto calcine.

A test to measure the gas evolution from any chemical reactions was setup with a 500-ml Erlenmeyer flask containing about 200-ml of SBW-HT-10 calcine. A 20-wt% loading of simulated NWCF scrub solution was added on top of the calcine and then the flask was capped and connected to the 500-ml water displacement indicator for any gas evolution. A visible yellow vapor formed inside the flask, but did not displace any water in the graduated cylinder.

A type-K thermocouple placed through the rubber stopper capping the flask was used to stir and mix the scrub solution and calcine completely together. An exothermic chemical reaction with foaming occurred as the mixture was stirred together and only lasted for a short time of less than a minute. This foaming from the chemical reaction was quite vigorous for large waste loadings of material that are mixed quickly together. Figure 6 shows an image of the test as the calcine and scrub are being mixed together with some foaming and the NOx gas being generated. The gas evolution captured in the graduated cylinder displaced an amount of water equal to about 525 to 550 milliliters. The temperature increase in the flask above ambient temperature was about 10°C on the average. Some condensate was visible in the flask and tubing as the temperature returned back to ambient.

Figure 6. 500-ml Flask showing foaming and NOx when scrub solution and calcine are mixed together.

7. Discussion and Analysis

The immediate objective of this testing was to demonstrate how much NWCF scrub solution could be absorbed if combined with calcine solids. This testing showed that up to a waste loading of 10-wt% there would not be any free liquid remaining after a short period of absorption time. The higher waste loadings, above 10-wt%, increased the likelihood of foaming and off gas evolution that could adversely affect this as a feasible means for disposal of this proposed waste stream. The simple effects of evaporation on the NWCF scrub solution may even account for the presence of no free liquids in the samples after any long period of time. Some of the samples with the higher waste loadings contained a soft waxy layer at the interface between the scrub solution and the calcine.

The one major problem with this type of waste treatment is the hard crust or clumps of material that is created from combining the scrub solution and calcine solids together. An interesting discovery is that the solid calcine product material contains a hard shell with a center filled with small crystals. Analysis of the calcine product material after being soaked with scrub solution indicates that the shell is not as hard and is easier to break the particles apart.

8. Conclusions

The absorption of scrubber solution on calcine causes an exothermic reaction, foaming, and gas generation. At loading levels greater than 10 wt%, free liquid remains. In all cases a hard crust forms where the calcine reacts with the scrub. At loading levels less than 10 wt%, the effects of the chemical reactions, may be sufficiently low to permit absorption of the scrub on calcine.

Due to crust formation in the reaction vicinity, the calcine is not longer free flowing; thus, any subsequent treatment of the calcine may be prevented.

9. Recommendations

If the process loading requirements are less than 10 wt%, the absorption of scrub onto calcine could be possible. Process engineers and designers must evaluate the effects noted from these experiments and determine the feasibility of such a process and if any further testing is warranted.

10. References

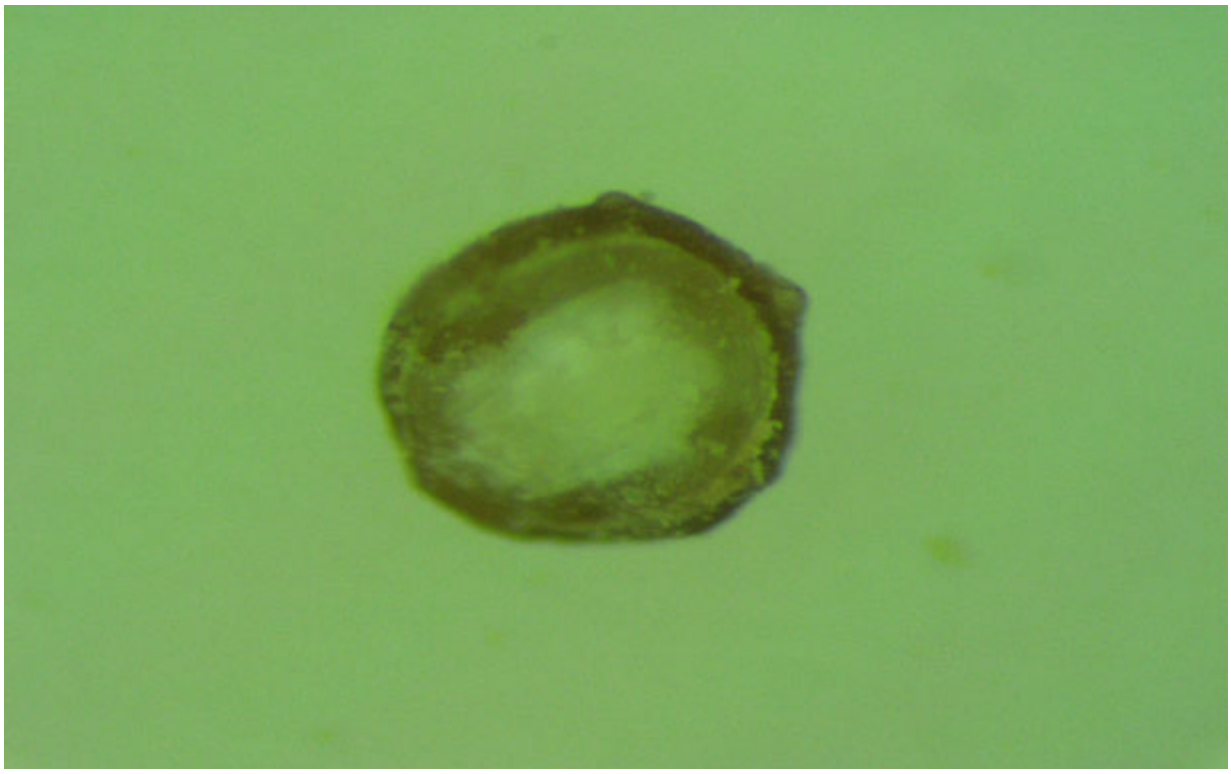
1. Engineering Design File 2373, "Process Design of SBW Treatment-Alternatives" INEEL Project File NO. 22681 Rev. 0, Sept 2002.

2. U.S. Nuclear Regulatory Commission Technical Branch of the Low-Level Waste Management and Decommissioning Division, "Technical Position on Waste Form," Revision 1, January 1991.

3. U.S. Environmental Protection Agency, "Paint Filter Liquids Test," Method 9095 A, Revision 1, December 1996.

4. J. A. Nenni, D. R. Marshall, "Flowsheet Development for Calcination of WM-185 Waste During NWCF Operations in March Through June 1999," INEEL/EXT-01-00832, June 2001.

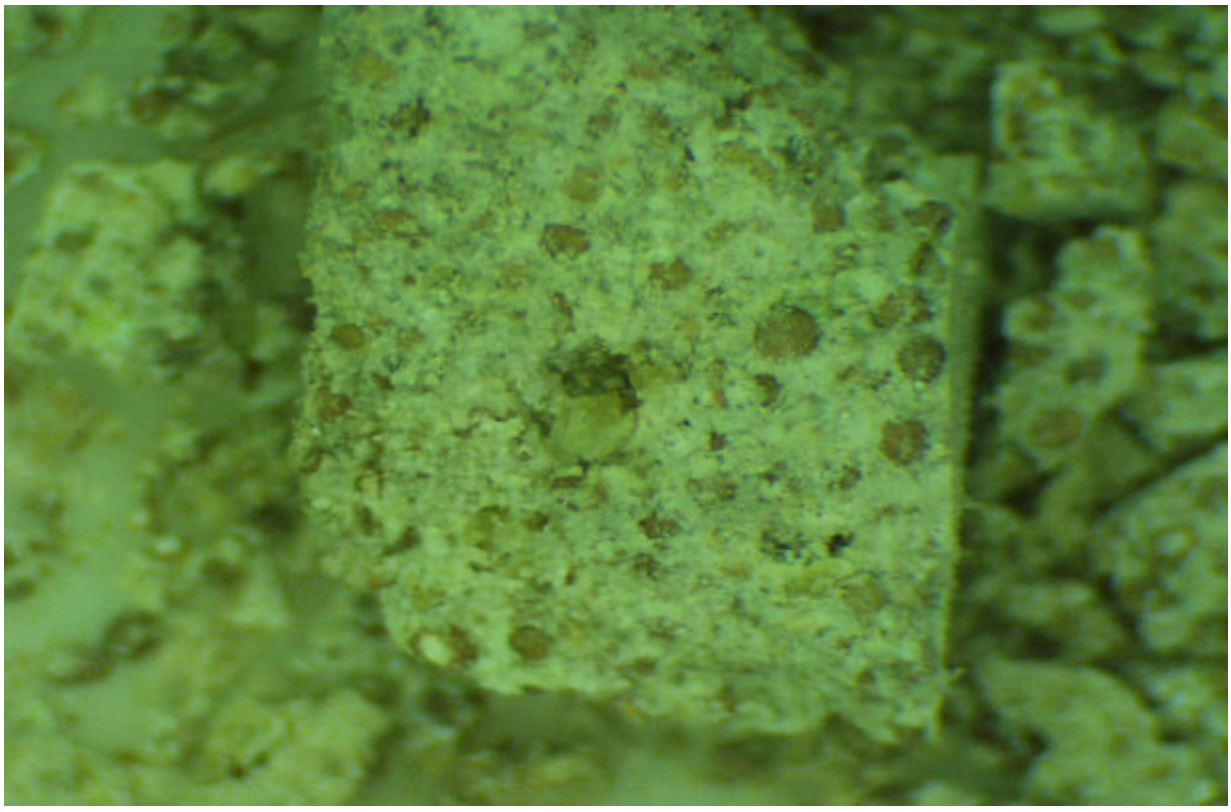
Digital Imaging Analysis of SBW-HT-10 Calcine Before and After NWCF Scrub Absorption Tests



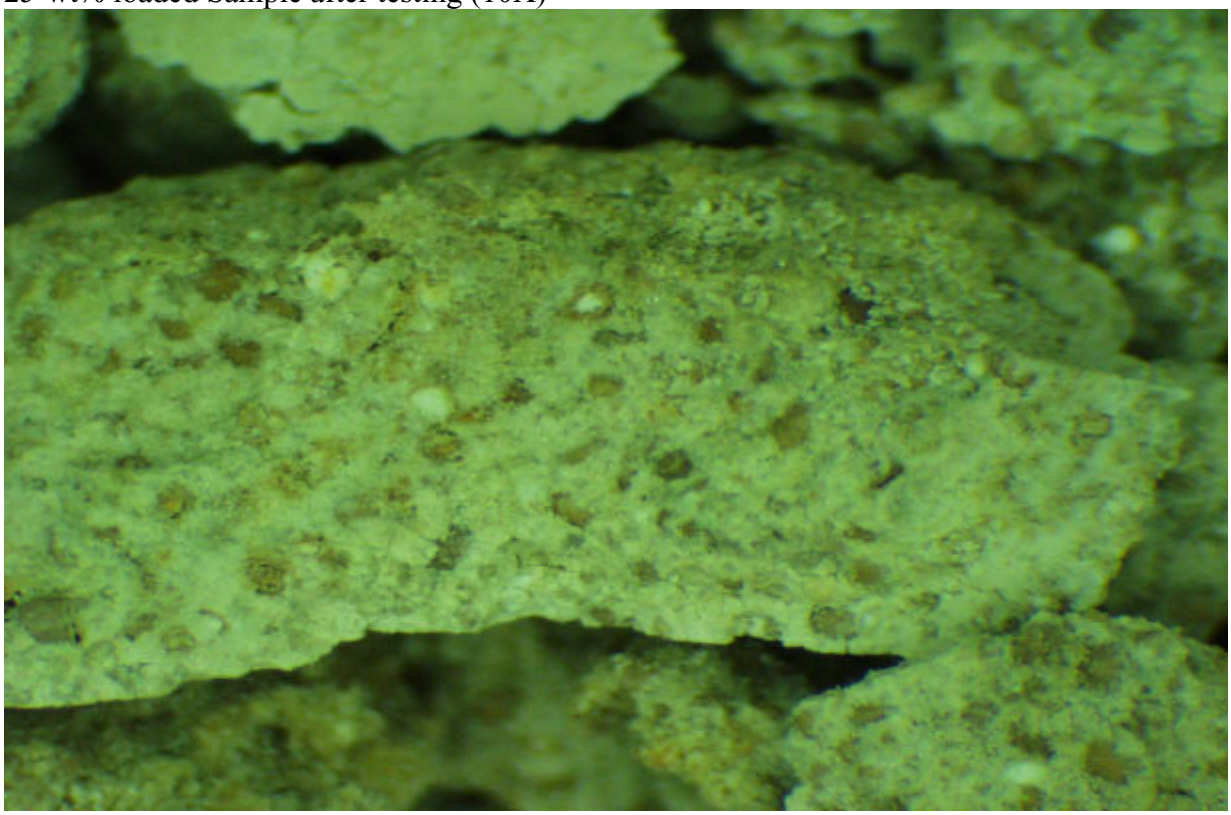
Product Particle Cross-Section Before Testing (70X)

Whole Product Particles at 50X

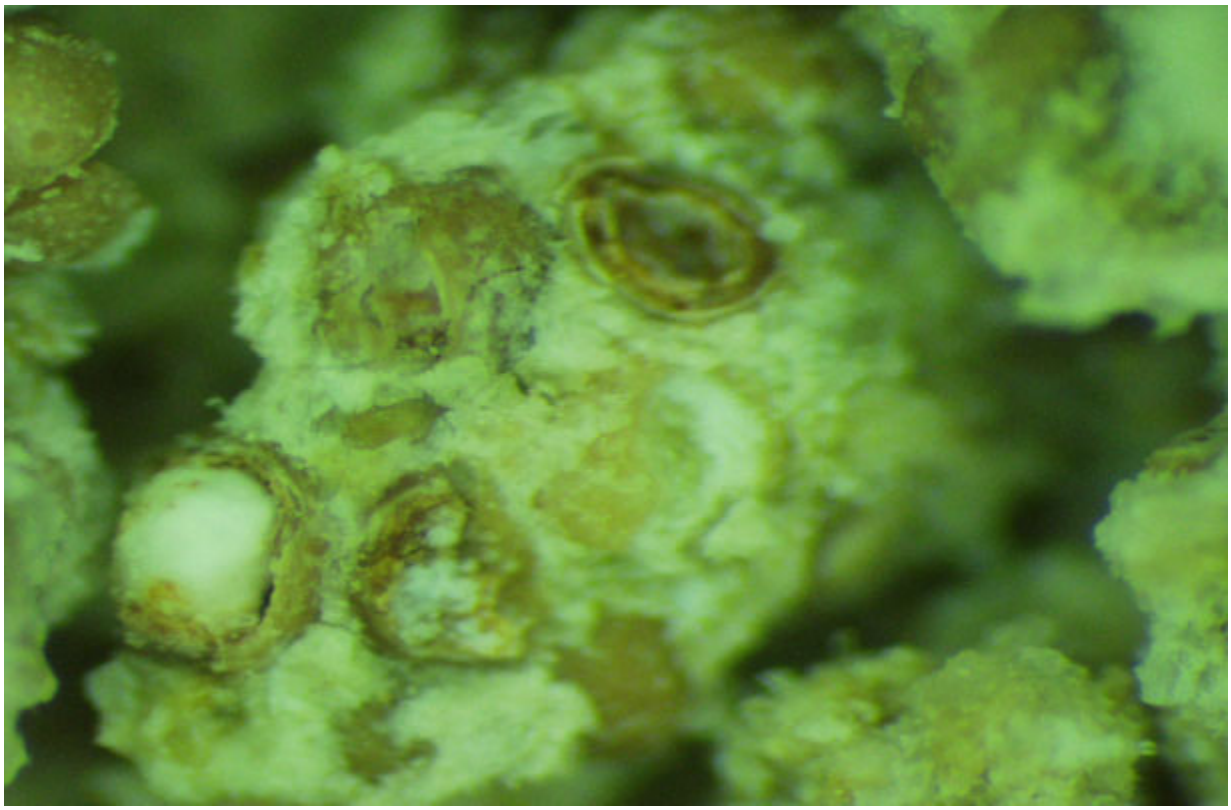
Product Particles Cross-Sectioned at 50X



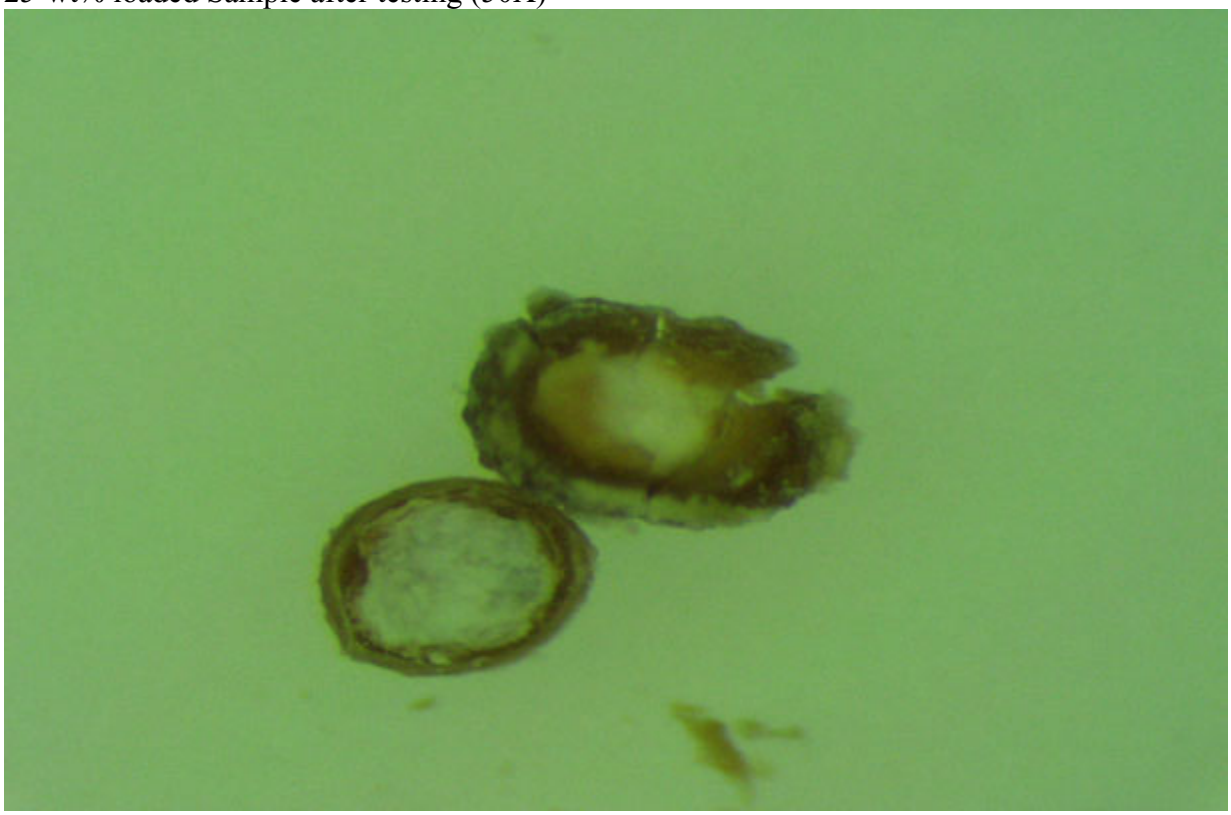
25-wt% loaded Sample after testing (10X)



25-wt% loaded Sample after testing (10X)



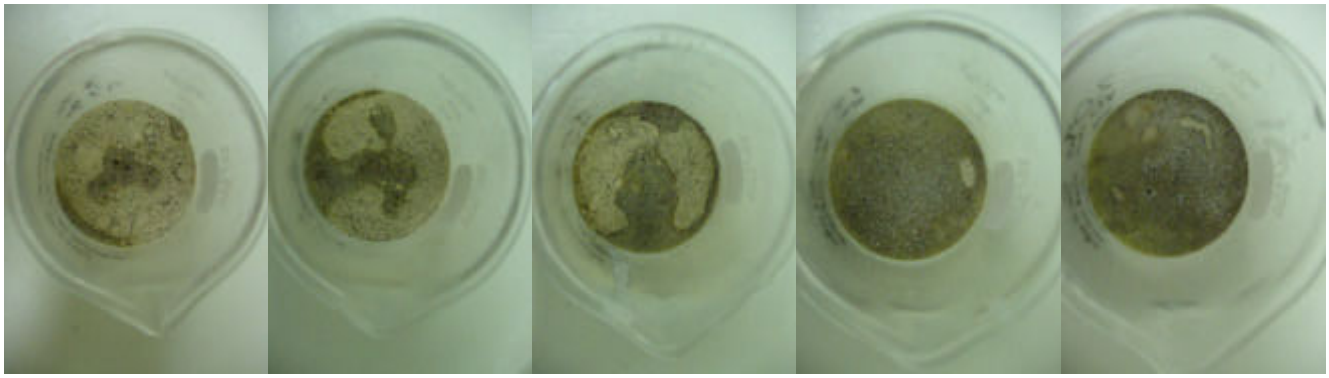
25-wt% loaded Sample after testing (50X)



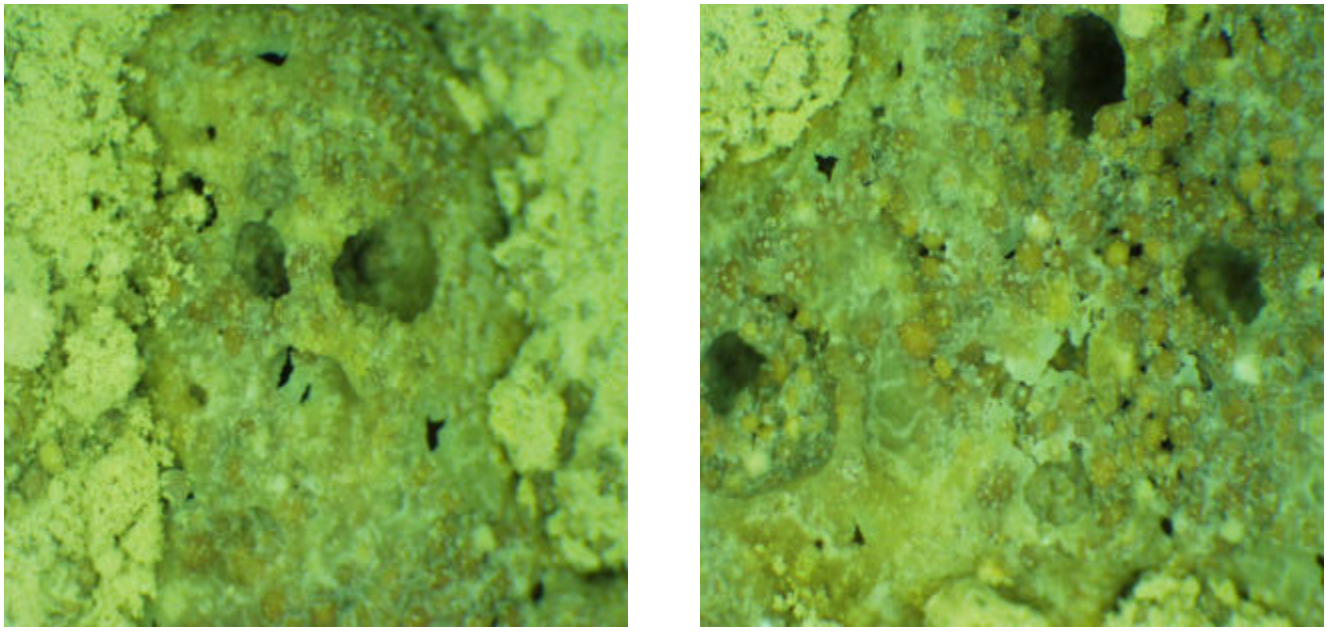
25-wt% loaded Sample after testing (50X)

Digital Imaging Analysis of SBW-HT-10 Calcine with Various Waste Loadings of NWCF Scrub

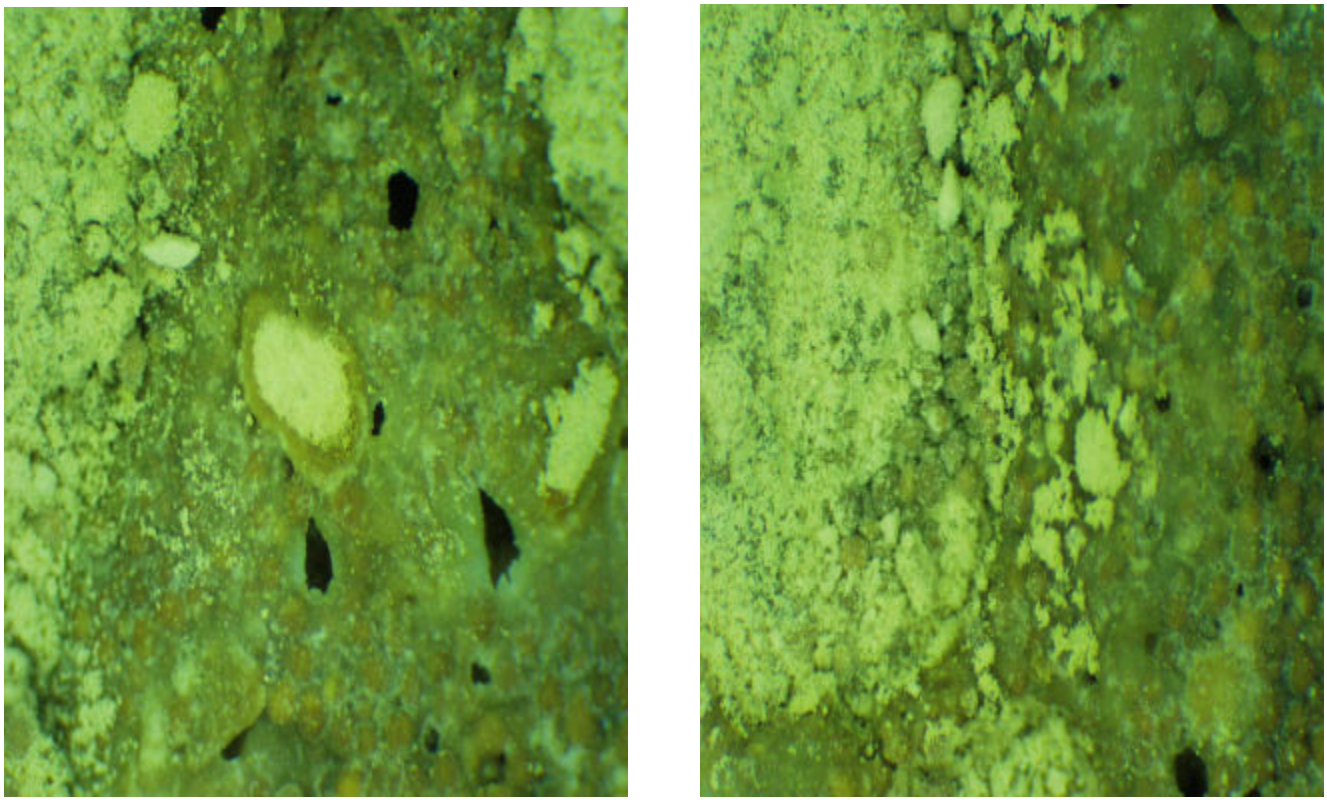
100-ml beakers of 3, 5, 10, 15 and 20 wt% loading of scrub absorbed onto calcine before testing.



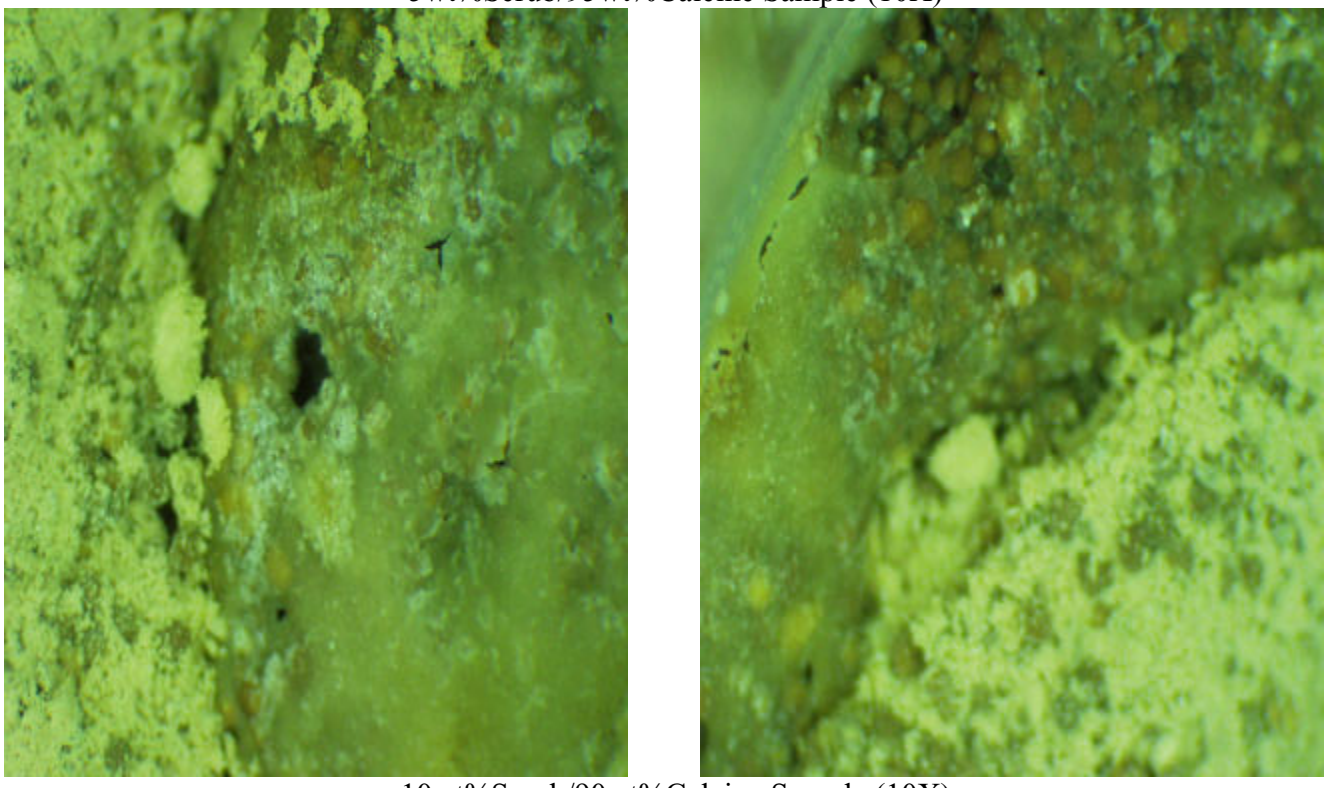
100-ml beakers of 3, 5, 10, 15 and 20 wt% loading of scrub absorbed onto calcine after testing.



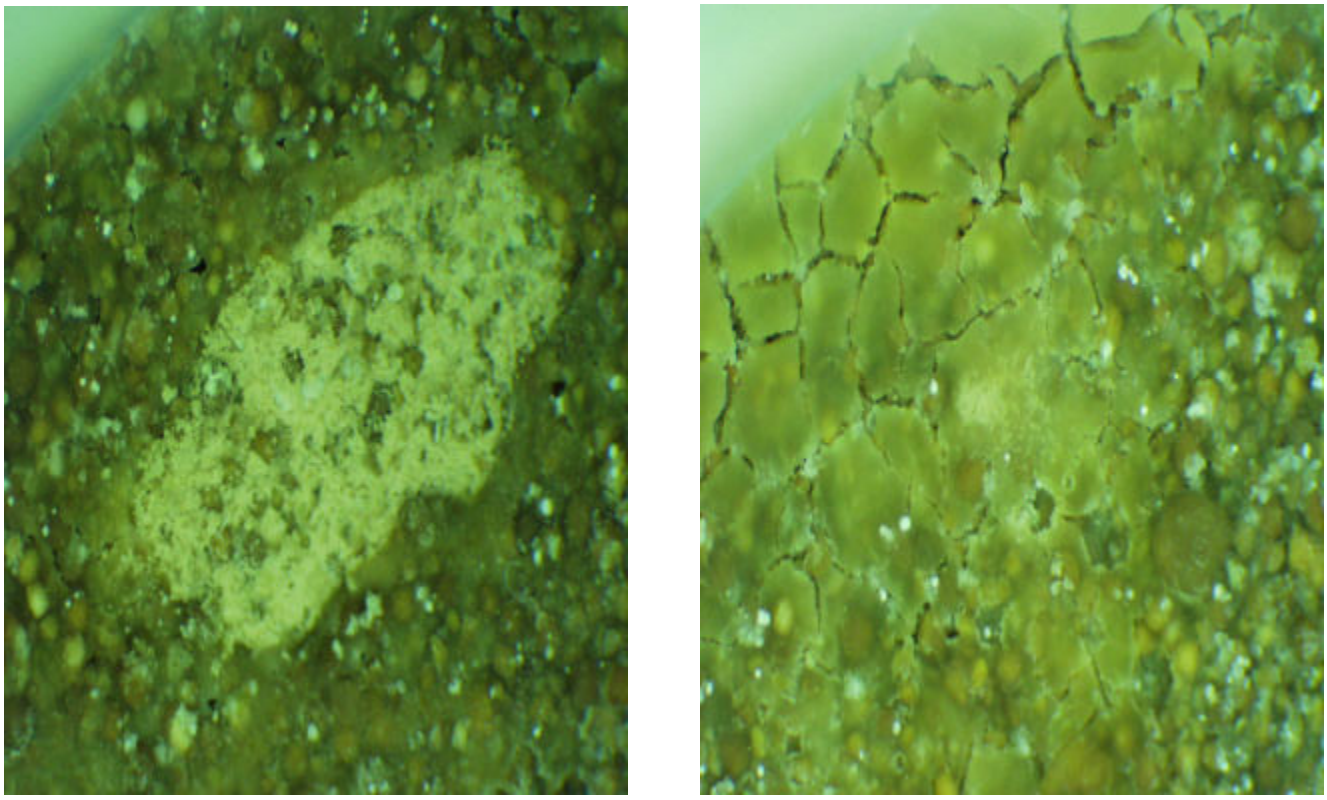
3wt%Scrub/97wt%Calcine Sample (10X)



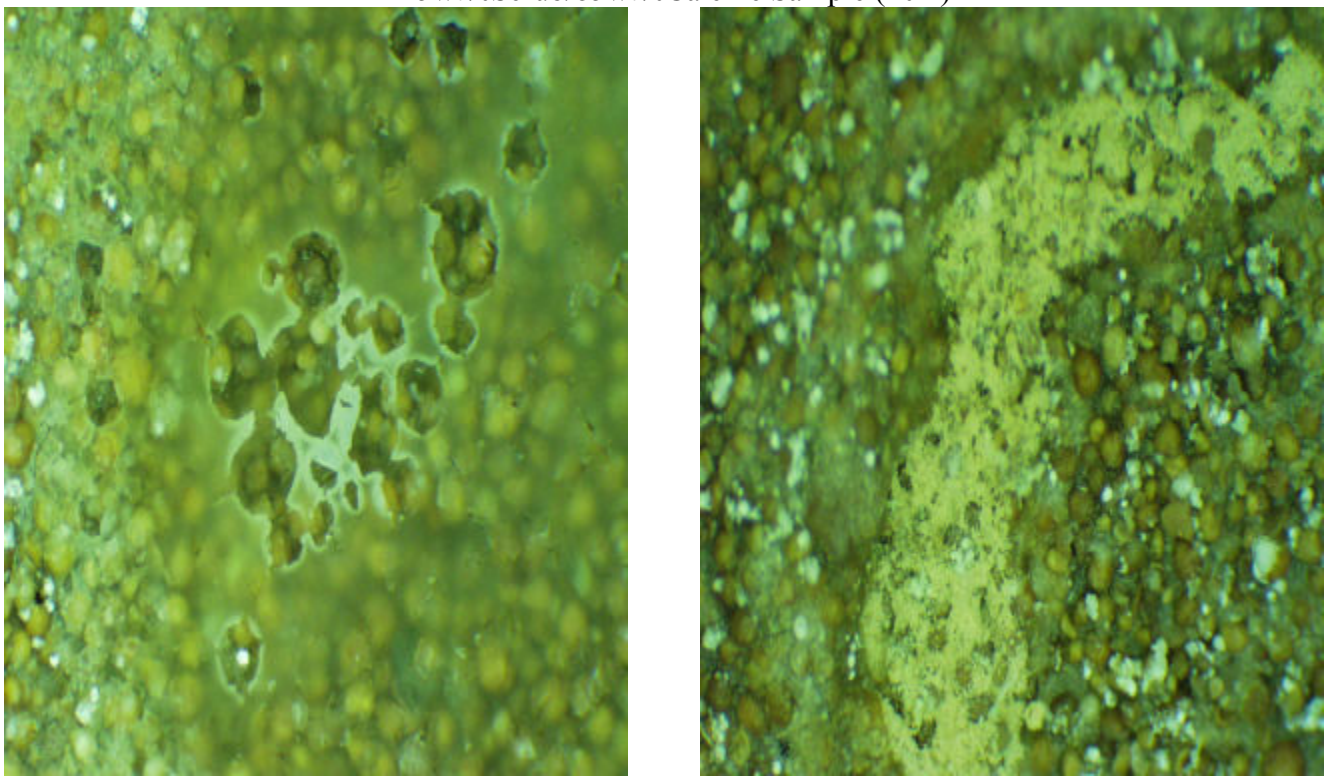
5wt%Scrub/95wt%Calcine Sample (10X)



10wt%Scrub/90wt%Calcine Sample (10X)



15wt%Scrub/85wt%Calcine Sample (10X)



20wt%Scrub/80wt%Calcine Sample (10X)

APPENDIX D

GROUT RADIATION LEVELS DUE TO SBW UNDISSOLVED SOLIDS CALCULATION AND DATA SHEETS

Filter Solids That Breakthrough To Grout
WM-180 SBW

Assumed Solids Breakthrough = 100%
Assumed Grout Waste Loading = 70%

Nuclide	Liquid Ci/L	Solids Ci/kg	Liquid uCi/mL	Solids uCi/mL	Combined uCi/mL	Grouted Liquid uCi/mL	Grouted with solids uCi/mL
Am-241	7.07E-05	3.13E-04	7.07E-02	7.70E-05	7.08E-02	4.95E-02	4.95E-02
Am-243	1.11E-08		1.11E-05		1.11E-05	7.77E-06	7.77E-06
Ba-137m	2.48E-05	2.45E-01	2.48E-02	6.04E-02	8.52E-02	1.74E-02	5.96E-02
Cd-113m	1.72E-06		1.72E-03		1.72E-03	1.20E-03	1.20E-03
Ce-144	3.25E-07		3.25E-04		3.25E-04	2.28E-04	2.28E-04
Cm-243	1.47E-08		1.47E-05		1.47E-05	1.03E-05	1.03E-05
Cm-244	9.15E-07		9.15E-04		9.15E-04	6.41E-04	6.41E-04
Co-60	4.14E-06	3.55E-05	4.14E-03	8.73E-06	4.15E-03	2.90E-03	2.90E-03
Cs-134 *	5.36E-09	2.59E-04	5.36E-06	6.37E-05	6.91E-05	3.75E-06	4.84E-05
Cs-135 *	4.46E-10		4.46E-07		4.46E-07	3.12E-07	3.12E-07
Cs-137 *	2.62E-05	2.61E-01	2.62E-02	6.42E-02	9.04E-02	1.83E-02	6.33E-02
Eu-152	1.31E-06		1.31E-03		1.31E-03	9.17E-04	9.17E-04
Eu-154	4.64E-05	4.30E-04	4.64E-02	1.06E-04	4.65E-02	3.25E-02	3.26E-02
Eu-155	8.52E-05		8.52E-02		8.52E-02	5.96E-02	5.96E-02
H-3	1.82E-05		1.82E-02		1.82E-02	1.27E-02	1.27E-02
I-129	2.39E-08		2.39E-05		2.39E-05	1.67E-05	1.67E-05
Nb-93m	8.86E-07		8.86E-04		8.86E-04	6.20E-04	6.20E-04
Nb-94	5.91E-07		5.91E-04		5.91E-04	4.14E-04	4.14E-04
Ni-63	2.46E-05		2.46E-02		2.46E-02	1.72E-02	1.72E-02
Np-237	1.22E-06	3.37E-06	1.22E-03	8.29E-07	1.22E-03	8.54E-04	8.55E-04
Np-239	1.11E-08		1.11E-05		1.11E-05	7.77E-06	7.77E-06
Pa-233	1.52E-06		1.52E-03		1.52E-03	1.06E-03	1.06E-03
Pm-146	2.64E-08		2.64E-05		2.64E-05	1.85E-05	1.85E-05
Pm-147	8.84E-05		8.84E-02		8.84E-02	6.19E-02	6.19E-02
Pr-144	3.25E-07		3.25E-04		3.25E-04	2.28E-04	2.28E-04
Pu-238	5.71E-04	8.75E-02	5.71E-01	2.15E-02	5.93E-01	4.00E-01	4.15E-01
Pu-239	8.27E-05	1.30E-02	8.27E-02	3.20E-03	8.59E-02	5.79E-02	6.01E-02
Pu-240	5.26E-06		5.26E-03		5.26E-03	3.68E-03	3.68E-03
Pu-241	1.36E-04		1.36E-01		1.36E-01	9.52E-02	9.52E-02
Rh-106	4.83E-07		4.83E-04		4.83E-04	3.38E-04	3.38E-04
Ru-106	4.83E-07		4.83E-04		4.83E-04	3.38E-04	3.38E-04
Sb-125	6.70E-06	3.36E-03	6.70E-03	8.27E-04	7.53E-03	4.69E-03	5.27E-03
Sb-126	2.99E-08		2.99E-05		2.99E-05	2.09E-05	2.09E-05
Se-79	2.27E-07		2.27E-04		2.27E-04	1.59E-04	1.59E-04
Sm-151	1.74E-04		1.74E-01		1.74E-01	1.22E-01	1.22E-01
Sn-121m	3.47E-08		3.47E-05		3.47E-05	2.43E-05	2.43E-05
Sn-126	2.13E-07		2.13E-04		2.13E-04	1.49E-04	1.49E-04
Sr-90	2.03E-02	6.16E-02	2.03E+01	1.52E-02	2.03E+01	1.42E+01	1.42E+01
Tc-99	9.38E-06	2.35E-05	9.38E-03	5.78E-06	9.39E-03	6.57E-03	6.57E-03
Te-125m	1.63E-06		1.63E-03		1.63E-03	1.14E-03	1.14E-03
Th-234	1.07E-08		1.07E-05		1.07E-05	7.49E-06	7.49E-06
U-234	1.07E-06	4.31E-06	1.07E-03	1.06E-06	1.07E-03	7.49E-04	7.50E-04
U-235	3.95E-08	8.88E-08	3.95E-05	2.18E-08	3.95E-05	2.77E-05	2.77E-05
U-236	5.84E-08	1.67E-07	5.84E-05	4.11E-08	5.84E-05	4.09E-05	4.09E-05
U-238	2.34E-08	3.79E-08	2.34E-05	9.32E-09	2.34E-05	1.64E-05	1.64E-05
Y-90	2.03E-02	6.16E-02	2.03E+01	1.52E-02	2.03E+01	1.42E+01	1.42E+01
Zr-93	1.15E-06		1.15E-03		1.15E-03	8.05E-04	8.05E-04

UDS 0.246 g/L

Nuclides listed are those greater than 1.0E-8 Ci/L
* Cesium values after ion exchange

Data Source: C. M. Barnes and C. B. Millet, "Feed Composition for the Sodium-Bearing Waste Treatment Process," INEEL/EXT-2000-01378, Revision 2, January 2003.
Column B data from Table 23
Column C data from Table 31

Sample Calculations

Liquid SBW from Ci/L to uCi/mL

$$\frac{7.34E\text{-}5\ Ci}{L} \times \frac{1000000\ uCi}{Ci} \times \frac{L}{1000\ mL} = \frac{7.34E\text{-}2\ uCi}{mL}$$

UDS from Ci/kg to uCi/mL

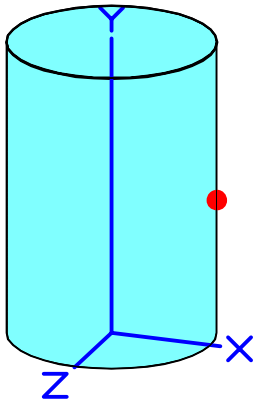
$$\frac{8.40E\text{-}6\ Ci}{kg} \times \frac{2.5\ g}{L} \times \frac{kg}{1000\ g} \times \frac{L}{1000\ mL} \times \frac{1000000\ uCi}{Ci} = \frac{2.10E\text{-}5\ uCi}{mL}$$

MicroShield v6.00 (6.0-00013)
INEEL

Page : 1
DOS File : Grout SBW-180.ms6
Run Date: April 29, 2003
Run Time: 1:33:07 PM
Duration : 00:00:36

File Ref: _____
Date: _____
By: _____
Checked: _____

Case Title: TRU Waste Drum
Description: WM-180 SBW Grouted After CsIX at 70% Loading
Geometry: 7 - Cylinder Volume - Side Shields



Source Dimensions

Height	86.36 cm	2 ft 10.0 in
Radius	29.21 cm	11.5 in

Dose Points

	X	Y	Z
# 1	31.37 cm	43.18 cm	0 cm
	1 ft 0.4 in	1 ft 5.0 in	0.0 in

Shields

Shield Name	Dimension	Material	Density
Source	2.31e+05 cm³	Concrete	1.5
Transition	1.0 cm	Air	0.00122
Air Gap		Air	0.00122
Wall Clad	.16 cm	Iron	7.86
Top Clad	.16 cm	Iron	7.86

Source Input
Grouping Method : Standard Indices
Number of Groups : 25
Lower Energy Cutoff : 0.015
Photons < 0.015 : Included
Library : ICRP-38

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Am-241	1.1459e-002	4.2397e+008	4.9500e-002	1.8315e+003
Am-243	1.7986e-006	6.6550e+004	7.7700e-006	2.8749e-001
Ba-137m	4.0279e-003	1.4903e+008	1.7400e-002	6.4380e+002
Cd-113m	2.7778e-004	1.0278e+007	1.2000e-003	4.4400e+001
Ce-144	5.2779e-005	1.9528e+006	2.2800e-004	8.4360e+000
Cm-243	2.3843e-006	8.8219e+004	1.0300e-005	3.8110e-001
Cm-244	1.4838e-004	5.4902e+006	6.4100e-004	2.3717e+001
Co-60	6.7131e-004	2.4838e+007	2.9000e-003	1.0730e+002
Cs-134	8.6807e-007	3.2119e+004	3.7500e-006	1.3875e-001
Cs-135	7.2224e-008	2.6723e+003	3.1200e-007	1.1544e-002
Cs-137	4.2362e-003	1.5674e+008	1.8300e-002	6.7710e+002
Eu-152	2.1227e-004	7.8541e+006	9.1700e-004	3.3929e+001
Eu-154	7.5233e-003	2.7836e+008	3.2500e-002	1.2025e+003
Eu-155	1.3797e-002	5.1047e+008	5.9600e-002	2.2052e+003
H-3	2.9399e-003	1.0878e+008	1.2700e-002	4.6990e+002
I-129	3.8658e-006	1.4304e+005	1.6700e-005	6.1790e-001
Nb-93m	1.4352e-004	5.3103e+006	6.2000e-004	2.2940e+001
Nb-94	9.5835e-005	3.5459e+006	4.1400e-004	1.5318e+001
Ni-63	3.9816e-003	1.4732e+008	1.7200e-002	6.3640e+002
Np-237	1.9769e-004	7.3145e+006	8.5400e-004	3.1598e+001
Np-239	1.7986e-006	6.6550e+004	7.7700e-006	2.8749e-001
Pa-233	2.4538e-004	9.0789e+006	1.0600e-003	3.9220e+001
Pm-146	4.2825e-006	1.5845e+005	1.8500e-005	6.8450e-001
Pm-147	1.4329e-002	5.3017e+008	6.1900e-002	2.2903e+003
Pr-144	5.2779e-005	1.9528e+006	2.2800e-004	8.4360e+000
Pu-238	9.2595e-002	3.4260e+009	4.0000e-001	1.4800e+004
Pu-239	1.3403e-002	4.9591e+008	5.7900e-002	2.1423e+003
Pu-240	8.5187e-004	3.1519e+007	3.6800e-003	1.3616e+002
Pu-241	2.2038e-002	8.1539e+008	9.5200e-002	3.5224e+003

DOS File : Grout SBW-180.ms6
Run Date: April 29, 2003
Run Time: 1:33:07 PM
Duration : 00:00:36

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Rh-106	7.8242e-005	2.8950e+006	3.3800e-004	1.2506e+001
Ru-106	7.8242e-005	2.8950e+006	3.3800e-004	1.2506e+001
Sb-125	1.0857e-003	4.0170e+007	4.6900e-003	1.7353e+002
Sb-126	4.8381e-006	1.7901e+005	2.0900e-005	7.7330e-001
Se-79	3.6806e-005	1.3618e+006	1.5900e-004	5.8830e+000
Sm-151	2.8241e-002	1.0449e+009	1.2200e-001	4.5140e+003
Sn-121m	5.6251e-006	2.0813e+005	2.4300e-005	8.9910e-001
Sn-126	3.4491e-005	1.2762e+006	1.4900e-004	5.5130e+000
Sr-90	3.2871e+000	1.2162e+011	1.4200e+001	5.2540e+005
Tc-99	1.5209e-003	5.6272e+007	6.5700e-003	2.4309e+002
Te-125m	2.6389e-004	9.7641e+006	1.1400e-003	4.2180e+001
Th-234	1.7338e-006	6.4152e+004	7.4900e-006	2.7713e-001
U-234	1.7338e-004	6.4152e+006	7.4900e-004	2.7713e+001
U-235	6.4122e-006	2.3725e+005	2.7700e-005	1.0249e+000
U-236	9.4678e-006	3.5031e+005	4.0900e-005	1.5133e+000
U-238	3.7964e-006	1.4047e+005	1.6400e-005	6.0680e-001
Y-90	3.2871e+000	1.2162e+011	1.4200e+001	5.2540e+005
Zr-93	1.8635e-004	6.8948e+006	8.0500e-004	2.9785e+001

Buildup
The material reference is : Source

Integration Parameters

Radial	34
Circumferential	24
Y Direction (axial)	34

Results

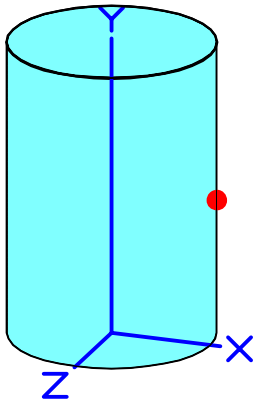
Energy MeV	Activity photons/sec	Fluence Rate MeV/cm²/sec No Buildup	Fluence Rate MeV/cm²/sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	1.082e+09	4.892e-33	1.158e-23	4.196e-34	9.935e-25
0.02	1.753e+08	7.884e-16	9.913e-16	2.731e-17	3.434e-17
0.03	5.319e+07	7.774e-06	1.298e-05	7.705e-08	1.287e-07
0.04	1.668e+08	3.174e-02	7.156e-02	1.404e-04	3.165e-04
0.05	4.617e+07	2.032e-01	5.941e-01	5.413e-04	1.583e-03
0.06	1.579e+08	3.656e+00	1.276e+01	7.261e-03	2.535e-02
0.08	1.605e+08	1.952e+01	7.941e+01	3.089e-02	1.257e-01
0.1	2.244e+08	6.128e+01	2.573e+02	9.376e-02	3.936e-01
0.15	1.117e+06	7.962e-01	3.179e+00	1.311e-03	5.234e-03
0.2	2.289e+07	2.707e+01	1.008e+02	4.777e-02	1.780e-01
0.3	7.224e+06	1.613e+01	5.314e+01	3.061e-02	1.008e-01
0.4	1.586e+07	5.474e+01	1.633e+02	1.067e-01	3.182e-01
0.5	6.153e+06	2.973e+01	8.203e+01	5.836e-02	1.610e-01
0.6	1.671e+08	1.063e+03	2.741e+03	2.075e+00	5.350e+00
0.8	1.136e+08	1.118e+03	2.603e+03	2.126e+00	4.951e+00
1.0	1.148e+08	1.589e+03	3.430e+03	2.929e+00	6.323e+00
1.5	1.357e+08	3.485e+03	6.599e+03	5.863e+00	1.110e+01
2.0	2.654e+04	1.049e+00	1.836e+00	1.622e-03	2.839e-03
3.0	4.910e+02	3.486e-02	5.513e-02	4.730e-05	7.479e-05
TOTALS:	2.651e+09	7.469e+03	1.613e+04	1.337e+01	2.904e+01

MicroShield v6.00 (6.0-00013)
INEEL

Page : 1
DOS File : Grout SBW-solids-180.ms6
Run Date: May 1, 2003
Run Time: 4:31:39 PM
Duration : 00:00:38

File Ref: _____
Date: _____
By: _____
Checked: _____

Case Title: TRU Waste Drum
Description: WM-180 SBW With Solids Grouted After CsIX at 70% Loading
Geometry: 7 - Cylinder Volume - Side Shields



Source Dimensions

Height	86.36 cm	2 ft 10.0 in
Radius	29.21 cm	11.5 in

Dose Points

	X	Y	Z
# 1	31.37 cm	43.18 cm	0 cm
	1 ft 0.4 in	1 ft 5.0 in	0.0 in

Shields

Shield Name	Dimension	Material	Density
Source	2.31e+05 cm³	Concrete	1.5
Transition	1.0 cm	Air	0.00122
Air Gap		Air	0.00122
Wall Clad	.16 cm	Iron	7.86
Top Clad	.16 cm	Iron	7.86

Source Input
Grouping Method : Standard Indices
Number of Groups : 25
Lower Energy Cutoff : 0.015
Photons < 0.015 : Included
Library : ICRP-38

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Am-241	1.1459e-002	4.2397e+008	4.9500e-002	1.8315e+003
Am-243	1.7986e-006	6.6550e+004	7.7700e-006	2.8749e-001
Ba-137m	1.3797e-002	5.1047e+008	5.9600e-002	2.2052e+003
Cd-113m	2.7778e-004	1.0278e+007	1.2000e-003	4.4400e+001
Ce-144	5.2779e-005	1.9528e+006	2.2800e-004	8.4360e+000
Cm-243	2.3843e-006	8.8219e+004	1.0300e-005	3.8110e-001
Cm-244	1.4838e-004	5.4902e+006	6.4100e-004	2.3717e+001
Co-60	6.7131e-004	2.4838e+007	2.9000e-003	1.0730e+002
Cs-134	1.1204e-005	4.1455e+005	4.8400e-005	1.7908e+000
Cs-135	7.2224e-008	2.6723e+003	3.1200e-007	1.1544e-002
Cs-137	1.4653e-002	5.4216e+008	6.3300e-002	2.3421e+003
Eu-152	2.1227e-004	7.8541e+006	9.1700e-004	3.3929e+001
Eu-154	7.5465e-003	2.7922e+008	3.2600e-002	1.2062e+003
Eu-155	1.3797e-002	5.1047e+008	5.9600e-002	2.2052e+003
H-3	2.9399e-003	1.0878e+008	1.2700e-002	4.6990e+002
I-129	3.8658e-006	1.4304e+005	1.6700e-005	6.1790e-001
Nb-93m	1.4352e-004	5.3103e+006	6.2000e-004	2.2940e+001
Nb-94	9.5835e-005	3.5459e+006	4.1400e-004	1.5318e+001
Ni-63	3.9816e-003	1.4732e+008	1.7200e-002	6.3640e+002
Np-237	1.9792e-004	7.3231e+006	8.5500e-004	3.1635e+001
Np-239	1.7986e-006	6.6550e+004	7.7700e-006	2.8749e-001
Pa-233	2.4538e-004	9.0789e+006	1.0600e-003	3.9220e+001
Pm-146	4.2825e-006	1.5845e+005	1.8500e-005	6.8450e-001
Pm-147	1.4329e-002	5.3017e+008	6.1900e-002	2.2903e+003
Pr-144	5.2779e-005	1.9528e+006	2.2800e-004	8.4360e+000
Pu-238	9.6067e-002	3.5545e+009	4.1500e-001	1.5355e+004
Pu-239	1.3912e-002	5.1476e+008	6.0100e-002	2.2237e+003
Pu-240	8.5187e-004	3.1519e+007	3.6800e-003	1.3616e+002
Pu-241	2.2038e-002	8.1539e+008	9.5200e-002	3.5224e+003

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Rh-106	7.8242e-005	2.8950e+006	3.3800e-004	1.2506e+001
Ru-106	7.8242e-005	2.8950e+006	3.3800e-004	1.2506e+001
Sb-125	1.2199e-003	4.5138e+007	5.2700e-003	1.9499e+002
Sb-126	4.8381e-006	1.7901e+005	2.0900e-005	7.7330e-001
Se-79	3.6806e-005	1.3618e+006	1.5900e-004	5.8830e+000
Sm-151	2.8241e-002	1.0449e+009	1.2200e-001	4.5140e+003
Sn-121m	5.6251e-006	2.0813e+005	2.4300e-005	8.9910e-001
Sn-126	3.4491e-005	1.2762e+006	1.4900e-004	5.5130e+000
Sr-90	3.2871e+000	1.2162e+011	1.4200e+001	5.2540e+005
Tc-99	1.5209e-003	5.6272e+007	6.5700e-003	2.4309e+002
Te-125m	2.6389e-004	9.7641e+006	1.1400e-003	4.2180e+001
Th-234	1.7338e-006	6.4152e+004	7.4900e-006	2.7713e-001
U-234	1.7361e-004	6.4237e+006	7.5000e-004	2.7750e+001
U-235	6.4122e-006	2.3725e+005	2.7700e-005	1.0249e+000
U-236	9.4678e-006	3.5031e+005	4.0900e-005	1.5133e+000
U-238	3.7964e-006	1.4047e+005	1.6400e-005	6.0680e-001
Y-90	3.2871e+000	1.2162e+011	1.4200e+001	5.2540e+005
Zr-93	1.8635e-004	6.8948e+006	8.0500e-004	2.9785e+001

Buildup
The material reference is : Source

Integration Parameters

Radial	34
Circumferential	24
Y Direction (axial)	34

Results

Energy MeV	Activity photons/sec	Fluence Rate MeV/cm²/sec No Buildup	Fluence Rate MeV/cm²/sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	1.118e+09	5.054e-33	1.196e-23	4.335e-34	1.026e-24
0.02	1.771e+08	7.963e-16	1.001e-15	2.758e-17	3.468e-17
0.03	7.742e+07	1.132e-05	1.890e-05	1.121e-07	1.873e-07
0.04	1.725e+08	3.281e-02	7.399e-02	1.451e-04	3.272e-04
0.05	4.623e+07	2.034e-01	5.948e-01	5.419e-04	1.584e-03
0.06	1.579e+08	3.656e+00	1.276e+01	7.261e-03	2.535e-02
0.08	1.605e+08	1.952e+01	7.942e+01	3.089e-02	1.257e-01
0.1	2.248e+08	6.138e+01	2.577e+02	9.391e-02	3.943e-01
0.15	1.134e+06	8.081e-01	3.226e+00	1.331e-03	5.313e-03
0.2	2.331e+07	2.756e+01	1.027e+02	4.865e-02	1.813e-01
0.3	7.249e+06	1.619e+01	5.332e+01	3.071e-02	1.011e-01
0.4	1.743e+07	6.016e+01	1.795e+02	1.172e-01	3.497e-01
0.5	6.675e+06	3.225e+01	8.899e+01	6.331e-02	1.747e-01
0.6	4.939e+08	3.142e+03	8.101e+03	6.133e+00	1.581e+01
0.8	1.143e+08	1.125e+03	2.618e+03	2.139e+00	4.980e+00
1.0	1.151e+08	1.593e+03	3.439e+03	2.936e+00	6.338e+00
1.5	1.360e+08	3.494e+03	6.616e+03	5.878e+00	1.113e+01
2.0	2.656e+04	1.049e+00	1.837e+00	1.622e-03	2.840e-03
3.0	4.910e+02	3.486e-02	5.513e-02	4.730e-05	7.479e-05
TOTALS:	3.050e+09	9.576e+03	2.155e+04	1.748e+01	3.962e+01

Filter Solids That Breakthrough To Grout
WM-189 SBW

Assumed Solids Breakthrough = 100%
Assumed Grout Waste Loading = 70%

Nuclide	Liquid Ci/L	Solids Ci/kg	Liquid uCi/mL	Solids uCi/mL	Combined uCi/mL	Grouted Liquid uCi/mL	Grouted with solids uCi/mL
Am-241	7.34E-05	8.40E-06	7.34E-02	2.10E-05	7.34E-02	5.14E-02	5.14E-02
Am-242m	1.51E-08		1.51E-05		1.51E-05	1.06E-05	1.06E-05
Am-243	2.13E-08		2.13E-05		2.13E-05	1.49E-05	1.49E-05
Ba-137m	4.74E-05	3.10E-02	4.74E-02	7.76E-02	1.25E-01	3.32E-02	8.75E-02
Cd-113m	3.30E-06		3.30E-03		3.30E-03	2.31E-03	2.31E-03
Ce-144	6.23E-07		6.23E-04		6.23E-04	4.36E-04	4.36E-04
Cm-242	2.98E-08	3.02E-08	2.98E-05	7.55E-08	2.99E-05	2.09E-05	2.09E-05
Cm-243	2.82E-08		2.82E-05		2.82E-05	1.97E-05	1.97E-05
Cm-244	1.05E-06		1.05E-03		1.05E-03	7.35E-04	7.35E-04
Co-60	3.62E-05	1.31E-04	3.62E-02	3.28E-04	3.65E-02	2.53E-02	2.56E-02
Cs-134 *	4.03E-08	2.77E-05	4.03E-05	6.93E-05	1.10E-04	2.82E-05	7.67E-05
Cs-135 *	8.54E-10		8.54E-07		8.54E-07	5.98E-07	5.98E-07
Cs-137 *	5.01E-05	3.30E-02	5.01E-02	8.25E-02	1.33E-01	3.51E-02	9.28E-02
Eu-152	2.50E-06		2.50E-03		2.50E-03	1.75E-03	1.75E-03
Eu-154	1.84E-04	1.23E-04	1.84E-01	3.08E-04	1.84E-01	1.29E-01	1.29E-01
Eu-155	1.63E-04		1.63E-01		1.63E-01	1.14E-01	1.14E-01
H-3	9.66E-06		9.66E-03		9.66E-03	6.76E-03	6.76E-03
I-129	5.30E-08		5.30E-05		5.30E-05	3.71E-05	3.71E-05
Nb-93m	1.70E-06		1.70E-03		1.70E-03	1.19E-03	1.19E-03
Nb-94	1.13E-06	6.07E-05	1.13E-03	1.52E-04	1.28E-03	7.91E-04	8.97E-04
Ni-63	3.14E-05		3.14E-02		3.14E-02	2.20E-02	2.20E-02
Np-237	4.59E-07	4.90E-07	4.59E-04	1.23E-06	4.60E-04	3.21E-04	3.22E-04
Np-239	2.13E-08		2.13E-05		2.13E-05	1.49E-05	1.49E-05
Pa-233	2.91E-06		2.91E-03		2.91E-03	2.04E-03	2.04E-03
Pm-146	5.05E-08		5.05E-05		5.05E-05	3.54E-05	3.54E-05
Pm-147	1.69E-04		1.69E-01		1.69E-01	1.18E-01	1.18E-01
Pr-144	6.23E-07		6.23E-04		6.23E-04	4.36E-04	4.36E-04
Pu-238	3.87E-04	1.23E-03	3.87E-01	3.08E-03	3.90E-01	2.71E-01	2.73E-01
Pu-239	4.35E-05	1.39E-04	4.35E-02	3.48E-04	4.38E-02	3.05E-02	3.07E-02
Pu-240	1.01E-05		1.01E-02		1.01E-02	7.07E-03	7.07E-03
Pu-241	1.33E-02	7.35E-02	1.33E+01	1.84E-01	1.35E+01	9.31E+00	9.44E+00
Rh-106	9.24E-07		9.24E-04		9.24E-04	6.47E-04	6.47E-04
Ru-106	9.24E-07		9.24E-04		9.24E-04	6.47E-04	6.47E-04
Sb-125	1.28E-05		1.28E-02		1.28E-02	8.96E-03	8.96E-03
Sb-126	5.71E-08		5.71E-05		5.71E-05	4.00E-05	4.00E-05
Se-79	4.34E-07		4.34E-04		4.34E-04	3.04E-04	3.04E-04
Sm-151	3.33E-04		3.33E-01		3.33E-01	2.33E-01	2.33E-01
Sn-121m	6.64E-08		6.64E-05		6.64E-05	4.65E-05	4.65E-05
Sn-126	4.08E-07		4.08E-04		4.08E-04	2.86E-04	2.86E-04
Sr-90	3.88E-02	1.60E-02	3.88E+01	4.00E-02	3.88E+01	2.72E+01	2.72E+01
Tc-99	9.96E-06	2.21E-03	9.96E-03	5.53E-03	1.55E-02	6.97E-03	1.08E-02
Te-125m	3.13E-06		3.13E-03		3.13E-03	2.19E-03	2.19E-03
Th-234	2.06E-08		2.06E-05		2.06E-05	1.44E-05	1.44E-05
U-234	1.74E-06	5.47E-07	1.74E-03	1.37E-06	1.74E-03	1.22E-03	1.22E-03
U-235	6.01E-08	4.98E-09	6.01E-05	1.25E-08	6.01E-05	4.21E-05	4.21E-05
U-236	7.81E-08	8.02E-08	7.81E-05	2.01E-07	7.83E-05	5.47E-05	5.48E-05
U-238	4.35E-08	2.69E-09	4.35E-05	6.73E-09	4.35E-05	3.05E-05	3.05E-05
Y-90	3.88E-02	1.60E-02	3.88E+01	4.00E-02	3.88E+01	2.72E+01	2.72E+01
Zr-93	2.20E-06		2.20E-03		2.20E-03	1.54E-03	1.54E-03

UDS	2.5 g/L	Nuclides listed are those greater than 1.0E-8 Ci/L * Cesium values after ion exchange

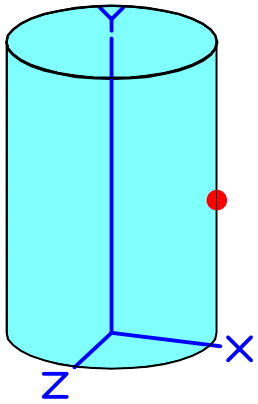
Data Source: C. M. Barnes and C. B. Millet, "Feed Composition for the Sodium-Bearing Waste Treatment Process," INEEL/EXT-2000-01378, Revision 2, January 2003.
Column B data from Table 23
Column C data from Table 33

MicroShield v6.00 (6.0-00013)
INEEL

Page : 1
DOS File : Grout SBW-189.ms6
Run Date: May 1, 2003
Run Time: 2:05:00 PM
Duration : 00:00:38

File Ref: _____
Date: _____
By: _____
Checked: _____

Case Title: TRU Waste Drum
Description: WM-189 SBW Grouted After CsIX at 70% Loading
Geometry: 7 - Cylinder Volume - Side Shields



Source Dimensions

Height	86.36 cm	2 ft 10.0 in
Radius	29.21 cm	11.5 in

Dose Points

	X	Y	Z
# 1	31.37 cm	43.18 cm	0 cm
	1 ft 0.4 in	1 ft 5.0 in	0.0 in

Shields

Shield Name	Dimension	Material	Density
Source	2.31e+05 cm³	Concrete	1.5
Transition	1.0 cm	Air	0.00122
Air Gap		Air	0.00122
Wall Clad	.16 cm	Iron	7.86
Top Clad	.16 cm	Iron	7.86

Source Input
Grouping Method : Standard Indices
Number of Groups : 25
Lower Energy Cutoff : 0.015
Photons < 0.015 : Included
Library : ICRP-38

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Am-241	1.1898e-002	4.4024e+008	5.1400e-002	1.9018e+003
Am-242m	2.4538e-006	9.0789e+004	1.0600e-005	3.9220e-001
Am-243	3.4491e-006	1.2762e+005	1.4900e-005	5.5130e-001
Ba-137m	7.6854e-003	2.8436e+008	3.3200e-002	1.2284e+003
Cd-113m	5.3473e-004	1.9785e+007	2.3100e-003	8.5470e+001
Ce-144	1.0093e-004	3.7343e+006	4.3600e-004	1.6132e+001
Cm-242	4.8381e-006	1.7901e+005	2.0900e-005	7.7330e-001
Cm-243	4.5603e-006	1.6873e+005	1.9700e-005	7.2890e-001
Cm-244	1.7014e-004	6.2953e+006	7.3500e-004	2.7195e+001
Co-60	5.8566e-003	2.1669e+008	2.5300e-002	9.3610e+002
Cs-134	6.5279e-006	2.4153e+005	2.8200e-005	1.0434e+000
Cs-135	1.3843e-007	5.1219e+003	5.9800e-007	2.2126e-002
Cs-137	8.1252e-003	3.0063e+008	3.5100e-002	1.2987e+003
Eu-152	4.0510e-004	1.4989e+007	1.7500e-003	6.4750e+001
Eu-154	2.9862e-002	1.1049e+009	1.2900e-001	4.7730e+003
Eu-155	2.6389e-002	9.7641e+008	1.1400e-001	4.2180e+003
H-3	1.5648e-003	5.7899e+007	6.7600e-003	2.5012e+002
I-129	8.5881e-006	3.1776e+005	3.7100e-005	1.3727e+000
Nb-93m	2.7547e-004	1.0192e+007	1.1900e-003	4.4030e+001
Nb-94	1.8311e-004	6.7749e+006	7.9100e-004	2.9267e+001
Ni-63	5.0927e-003	1.8843e+008	2.2000e-002	8.1400e+002
Np-237	7.4307e-005	2.7494e+006	3.2100e-004	1.1877e+001
Np-239	3.4491e-006	1.2762e+005	1.4900e-005	5.5130e-001
Pa-233	4.7223e-004	1.7473e+007	2.0400e-003	7.5480e+001
Pm-146	8.1946e-006	3.0320e+005	3.5400e-005	1.3098e+000
Pm-147	2.7315e-002	1.0107e+009	1.1800e-001	4.3660e+003
Pr-144	1.0093e-004	3.7343e+006	4.3600e-004	1.6132e+001
Pu-238	6.2733e-002	2.3211e+009	2.7100e-001	1.0027e+004
Pu-239	7.0603e-003	2.6123e+008	3.0500e-002	1.1285e+003

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Pu-240	1.6366e-003	6.0555e+007	7.0700e-003	2.6159e+002
Pu-241	2.1551e+000	7.9740e+010	9.3100e+000	3.4447e+005
Rh-106	1.4977e-004	5.5416e+006	6.4700e-004	2.3939e+001
Ru-106	1.4977e-004	5.5416e+006	6.4700e-004	2.3939e+001
Sb-125	2.0741e-003	7.6742e+007	8.9600e-003	3.3152e+002
Sb-126	9.2595e-006	3.4260e+005	4.0000e-005	1.4800e+000
Se-79	7.0372e-005	2.6038e+006	3.0400e-004	1.1248e+001
Sm-151	5.3936e-002	1.9956e+009	2.3300e-001	8.6210e+003
Sn-121m	1.0764e-005	3.9827e+005	4.6500e-005	1.7205e+000
Sn-126	6.6205e-005	2.4496e+006	2.8600e-004	1.0582e+001
Sr-90	6.2964e+000	2.3297e+011	2.7200e+001	1.0064e+006
Tc-99	1.6135e-003	5.9698e+007	6.9700e-003	2.5789e+002
Te-125m	5.0696e-004	1.8757e+007	2.1900e-003	8.1030e+001
Th-234	3.3334e-006	1.2334e+005	1.4400e-005	5.3280e-001
U-234	2.8241e-004	1.0449e+007	1.2200e-003	4.5140e+001
U-235	9.7456e-006	3.6059e+005	4.2100e-005	1.5577e+000
U-236	1.2662e-005	4.6851e+005	5.4700e-005	2.0239e+000
U-238	7.0603e-006	2.6123e+005	3.0500e-005	1.1285e+000
Y-90	6.2964e+000	2.3297e+011	2.7200e+001	1.0064e+006
Zr-93	3.5649e-004	1.3190e+007	1.5400e-003	5.6980e+001

Buildup
The material reference is : Source

Integration Parameters

Radial	34
Circumferential	24
Y Direction (axial)	34

Results

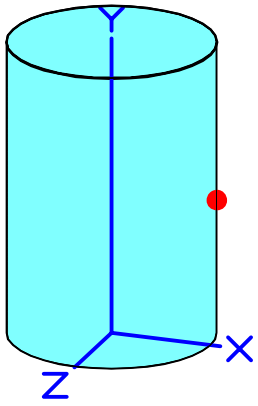
Energy MeV	Activity photons/sec	Fluence Rate MeV/cm²/sec No Buildup	Fluence Rate MeV/cm²/sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	8.515e+08	3.849e-33	9.111e-24	3.301e-34	7.815e-25
0.02	1.686e+08	7.581e-16	9.531e-16	2.626e-17	3.302e-17
0.03	9.081e+07	1.327e-05	2.217e-05	1.315e-07	2.197e-07
0.04	4.433e+08	8.433e-02	1.902e-01	3.730e-04	8.410e-04
0.05	1.192e+08	5.247e-01	1.534e+00	1.398e-03	4.087e-03
0.06	1.695e+08	3.924e+00	1.370e+01	7.793e-03	2.721e-02
0.08	3.055e+08	3.716e+01	1.512e+02	5.881e-02	2.392e-01
0.1	6.605e+08	1.804e+02	7.572e+02	2.759e-01	1.158e+00
0.15	2.700e+06	1.924e+00	7.681e+00	3.168e-03	1.265e-02
0.2	8.298e+07	9.812e+01	3.656e+02	1.732e-01	6.452e-01
0.3	1.507e+07	3.365e+01	1.108e+02	6.383e-02	2.102e-01
0.4	3.489e+07	1.204e+02	3.593e+02	2.347e-01	7.002e-01
0.5	1.432e+07	6.918e+01	1.909e+02	1.358e-01	3.747e-01
0.6	3.564e+08	2.267e+03	5.846e+03	4.426e+00	1.141e+01
0.8	4.329e+08	4.261e+03	9.919e+03	8.104e+00	1.887e+01
1.0	5.666e+08	7.839e+03	1.692e+04	1.445e+01	3.120e+01
1.5	6.528e+08	1.677e+04	3.175e+04	2.821e+01	5.342e+01
2.0	6.238e+04	2.465e+00	4.314e+00	3.811e-03	6.671e-03
3.0	9.459e+02	6.716e-02	1.062e-01	9.112e-05	1.441e-04
TOTALS:	4.968e+09	3.168e+04	6.640e+04	5.615e+01	1.183e+02

MicroShield v6.00 (6.0-00013)
INEEL

Page : 1
DOS File : Grout SBW-solids-189.ms6
Run Date: May 1, 2003
Run Time: 1:58:51 PM
Duration : 00:00:38

File Ref: _____
Date: _____
By: _____
Checked: _____

Case Title: TRU Waste Drum
Description: WM-189 SBW With 100%Solids Grouted After CsIX at 70% Loading
Geometry: 7 - Cylinder Volume - Side Shields



Source Dimensions

Height	86.36 cm	2 ft 10.0 in
Radius	29.21 cm	11.5 in

Dose Points

	X	Y	Z
# 1	31.37 cm	43.18 cm	0 cm
	1 ft 0.4 in	1 ft 5.0 in	0.0 in

Shields

Shield Name	Dimension	Material	Density
Source	2.31e+05 cm³	Concrete	1.5
Transition	1.0 cm	Air	0.00122
Air Gap		Air	0.00122
Wall Clad	.16 cm	Iron	7.86
Top Clad	.16 cm	Iron	7.86

Source Input
Grouping Method : Standard Indices
Number of Groups : 25
Lower Energy Cutoff : 0.015
Photons < 0.015 : Included
Library : ICRP-38

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Am-241	1.1898e-002	4.4024e+008	5.1400e-002	1.9018e+003
Am-242m	2.4538e-006	9.0789e+004	1.0600e-005	3.9220e-001
Am-243	3.4491e-006	1.2762e+005	1.4900e-005	5.5130e-001
Ba-137m	2.0255e-002	7.4944e+008	8.7500e-002	3.2375e+003
Cd-113m	5.3473e-004	1.9785e+007	2.3100e-003	8.5470e+001
Ce-144	1.0093e-004	3.7343e+006	4.3600e-004	1.6132e+001
Cm-242	4.8381e-006	1.7901e+005	2.0900e-005	7.7330e-001
Cm-243	4.5603e-006	1.6873e+005	1.9700e-005	7.2890e-001
Cm-244	1.7014e-004	6.2953e+006	7.3500e-004	2.7195e+001
Co-60	5.9261e-003	2.1926e+008	2.5600e-002	9.4720e+002
Cs-134	1.7755e-005	6.5694e+005	7.6700e-005	2.8379e+000
Cs-135	1.3843e-007	5.1219e+003	5.9800e-007	2.2126e-002
Cs-137	2.1482e-002	7.9483e+008	9.2800e-002	3.4336e+003
Eu-152	4.0510e-004	1.4989e+007	1.7500e-003	6.4750e+001
Eu-154	2.9862e-002	1.1049e+009	1.2900e-001	4.7730e+003
Eu-155	2.6389e-002	9.7641e+008	1.1400e-001	4.2180e+003
H-3	1.5648e-003	5.7899e+007	6.7600e-003	2.5012e+002
I-129	8.5881e-006	3.1776e+005	3.7100e-005	1.3727e+000
Nb-93m	2.7547e-004	1.0192e+007	1.1900e-003	4.4030e+001
Nb-94	2.0764e-004	7.6828e+006	8.9700e-004	3.3189e+001
Ni-63	5.0927e-003	1.8843e+008	2.2000e-002	8.1400e+002
Np-237	7.4539e-005	2.7579e+006	3.2200e-004	1.1914e+001
Np-239	3.4491e-006	1.2762e+005	1.4900e-005	5.5130e-001
Pa-233	4.7223e-004	1.7473e+007	2.0400e-003	7.5480e+001
Pm-146	8.1946e-006	3.0320e+005	3.5400e-005	1.3098e+000
Pm-147	2.7315e-002	1.0107e+009	1.1800e-001	4.3660e+003
Pr-144	1.0093e-004	3.7343e+006	4.3600e-004	1.6132e+001
Pu-238	6.3196e-002	2.3382e+009	2.7300e-001	1.0101e+004
Pu-239	7.1066e-003	2.6295e+008	3.0700e-002	1.1359e+003

Nuclide	curies	becquerels	µCi/cm³	Bq/cm³
Pu-240	1.6366e-003	6.0555e+007	7.0700e-003	2.6159e+002
Pu-241	2.1852e+000	8.0854e+010	9.4400e+000	3.4928e+005
Rh-106	1.4977e-004	5.5416e+006	6.4700e-004	2.3939e+001
Ru-106	1.4977e-004	5.5416e+006	6.4700e-004	2.3939e+001
Sb-125	2.0741e-003	7.6742e+007	8.9600e-003	3.3152e+002
Sb-126	9.2595e-006	3.4260e+005	4.0000e-005	1.4800e+000
Se-79	7.0372e-005	2.6038e+006	3.0400e-004	1.1248e+001
Sm-151	5.3936e-002	1.9956e+009	2.3300e-001	8.6210e+003
Sn-121m	1.0764e-005	3.9827e+005	4.6500e-005	1.7205e+000
Sn-126	6.6205e-005	2.4496e+006	2.8600e-004	1.0582e+001
Sr-90	6.2964e+000	2.3297e+011	2.7200e+001	1.0064e+006
Tc-99	2.5001e-003	9.2502e+007	1.0800e-002	3.9960e+002
Te-125m	5.0696e-004	1.8757e+007	2.1900e-003	8.1030e+001
Th-234	3.3334e-006	1.2334e+005	1.4400e-005	5.3280e-001
U-234	2.8241e-004	1.0449e+007	1.2200e-003	4.5140e+001
U-235	9.7456e-006	3.6059e+005	4.2100e-005	1.5577e+000
U-236	1.2685e-005	4.6936e+005	5.4800e-005	2.0276e+000
U-238	7.0603e-006	2.6123e+005	3.0500e-005	1.1285e+000
Y-90	6.2964e+000	2.3297e+011	2.7200e+001	1.0064e+006
Zr-93	3.5649e-004	1.3190e+007	1.5400e-003	5.6980e+001

Buildup
The material reference is : Source

Integration Parameters

Radial	34
Circumferential	24
Y Direction (axial)	34

Results

Energy MeV	Activity photons/sec	Fluence Rate MeV/cm²/sec No Buildup	Fluence Rate MeV/cm²/sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	8.592e+08	3.883e-33	9.193e-24	3.331e-34	7.886e-25
0.02	1.688e+08	7.591e-16	9.545e-16	2.630e-17	3.306e-17
0.03	1.190e+08	1.739e-05	2.904e-05	1.723e-07	2.878e-07
0.04	4.500e+08	8.560e-02	1.930e-01	3.786e-04	8.537e-04
0.05	1.192e+08	5.247e-01	1.534e+00	1.398e-03	4.087e-03
0.06	1.695e+08	3.924e+00	1.370e+01	7.793e-03	2.721e-02
0.08	3.055e+08	3.716e+01	1.512e+02	5.881e-02	2.392e-01
0.1	6.605e+08	1.804e+02	7.572e+02	2.759e-01	1.158e+00
0.15	2.703e+06	1.926e+00	7.689e+00	3.171e-03	1.266e-02
0.2	8.298e+07	9.812e+01	3.656e+02	1.732e-01	6.452e-01
0.3	1.507e+07	3.365e+01	1.108e+02	6.383e-02	2.102e-01
0.4	3.489e+07	1.204e+02	3.593e+02	2.347e-01	7.002e-01
0.5	1.432e+07	6.921e+01	1.910e+02	1.358e-01	3.748e-01
0.6	7.744e+08	4.927e+03	1.270e+04	9.617e+00	2.480e+01
0.8	4.351e+08	4.282e+03	9.970e+03	8.145e+00	1.896e+01
1.0	5.692e+08	7.874e+03	1.700e+04	1.451e+01	3.134e+01
1.5	6.554e+08	1.683e+04	3.188e+04	2.832e+01	5.363e+01
2.0	6.241e+04	2.466e+00	4.316e+00	3.813e-03	6.674e-03
3.0	9.460e+02	6.717e-02	1.062e-01	9.113e-05	1.441e-04
TOTALS:	5.436e+09	3.447e+04	7.351e+04	6.156e+01	1.321e+02

APPENDIX E

REFERENCE LITERATURE FOR LOW-LEVEL WASTE SOLIDIFICATION PROGRAM

REFERENCE LITERATURE
FOR THE LOW LEVEL WASTE SOLIDIFICATION PROGRAM

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