Sorbent Testing for the Solidification of Unidentified Rocky Flats Laboratory Waste Stored at the Idaho National Laboratory

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ABSTRACT

At the request of the U.S. Department of Energy (DOE), MSE Technology Applications, Inc. (MSE) evaluated various commercially available sorbents to solidify unidentified laboratory liquids from Rocky Flats that are stored at the Idaho National Laboratory (INL). The liquids are a collection of laboratory wastes that were generated from various experiments and routine analytical laboratory activities carried out at Rocky Flats. The liquids are in bottles discovered inside of buried waste drums being exhumed from the subsurface disposal area at the Radioactive Waste Management Complex (RWMC) by the contractor, CH2M Hill Washington International (CWI). Free liquids are unacceptable at the Waste Isolation Pilot Plant (WIPP), and some of these liquids cannot be returned to the retrieval pit. Stabilization of the liquids into a solid mass will allow these materials to be sent to an appropriate disposal location. The selected sorbent or sorbent combinations should produce a stabilized mass that is capable of withstanding conditions similar to those experienced during storage, shipping, and burial. The final wasteform should release less than 1% liquid by volume per the WIPP Waste Acceptance Criteria (WAC). The absence or presence of free liquid in the solidified wasteforms was detected when tested by SW-846, Method 9095B, Paint Filter Free Liquids, and the amount of liquid released from the wasteform was determined by SW-846, Method 9096, Liquid Release Test. Reactivity testing was also conducted on the solidified laboratory liquids.

INTRODUCTION

At the request of the U.S. Department of Energy (DOE), MSE Technology Applications, Inc. (MSE) evaluated various sorbents to solidify unidentified liquids from the Rocky Flats facility that are presently stored at the Idaho National Laboratory (INL). The liquids are a collection of laboratory wastes that were generated from various experiments and routine analytical laboratory activities carried out at Rocky Flats. The liquids are in bottles discovered inside of buried waste drums being exhumed from the subsurface disposal area at the Radioactive Waste Management Complex (RWMC) by the contractor, CH2M Hill Washington International (CWI). Ultimate disposal of the materials is planned for the Waste Isolation Pilot Plant (WIPP). However, free liquids are unacceptable at WIPP, and the liquids cannot be returned to the retrieval pit. Stabilization of the liquids into a solid mass will allow these materials to be sent to an appropriate disposal location. The selected sorbent combinations should produce stabilized masses that are capable of withstanding conditions similar to those experienced during storage, shipping, and burial (such as overburden pressure). Other performance criteria included testing and observation to determine sorbent capacity, sorption rate, curing time, and reactivity. The final wasteform should release less than 1% liquid by volume per the WIPP Waste Acceptance Criteria (WAC). The absence or presence of free liquid was tested for by the use of SW-846, Method 9095B, Paint Filter Liquids [1], and the amount of liquid released from the wasteform was determined by the use of SW-846, Method 9096, Liquid Release Test (LRT) [2].

Several sorbents were considered for this test sequence. After discussions with INL, it was decided to use clay sorbents (not polymer sorbents) for the solidification of the unknown liquids since some of the chemicals have the potential for violent polymerization.

Accordingly, MSE tested and evaluated a number of appropriate sorbents with selected chemicals from the list provided by CWI. Three sorbents were initially identified as having the required capabilities. These sorbents are identified as Aquaset, Petroset II Granular (Petroset II-G), and Aquaset II Granular (Aquaset II-G). The initial test sequence called for specific liquids to be tested against specific sorbents or sorbent combinations. Subsequent to the initial test sequence, it was determined that information regarding the character of the liquids would not be available in the field prior to solidification. As such, it was determined that all of the liquids would need to be solidified with a single, robust combination of the sorbents. This paper contains the results of all of the solidification testing conducted by MSE using neat chemicals and surrogate rinsate recipes developed for the Rocky Flats liquids.

TEST OBJECTIVES

Specific objectives of this sorbent testing and evaluation in fiscal year (FY) 2006 were:

- identify the chemical categories, the neat chemicals and rinsates, and surrogate recipes to be used for sorbent testing from the list provided by CWI;
- identify the sorbent materials;
- identify the initial sorbent combinations and waste-loading ratios based on historical data from MSE sorbent testing;
- verify the presence/absence of free liquid in the final wasteforms using the Paint Filter Test (PFT) according to SW-846, Method 9095B [1] at ambient temperature;
- verify the amount of liquid released during the LRT according to SW-846, Method 9096, Liquid Release Test [2] at ambient temperature for selected chemical/sorbent combinations;
- identify sorbent addition and mixing methods after verifying the waste-loading ratios;
- identify analytical test methods to determine reactivity for the different groups of chemicals tested; and
- test the solidified wasteforms by the chemical reactivity test methods identified.

MATERIAL DESCRIPTIONS

To perform the testing, a representative group of neat chemicals and rinsate solutions were selected from the list provided by CWI, and sorbents were identified. Information pertaining to the CWI chemical list, the reasons the chemicals were chosen from the different chemical categories on the CWI listing, and the sorbents that were tested is contained in the following sections.

CWI Listed Chemicals

CWI provided MSE with a list of neat chemicals that may be present in the unidentified Rocky Flats bottles recovered from the exhumation of barrels at the RWMC. The unidentified bottles are contained in barrels of sludge from Rocky Flats and were packaged several years ago prior to burial at the RWMC. It is expected that approximately 10% of the liquids within the Rocky Flats bottles will be in the form of neat chemicals and 90% in the form of rinsates from the different laboratory procedures conducted at Rocky Flats. The entire list of potential neat chemicals included approximately 500 compounds. As testing that number of compounds was beyond the scope of the project, a subset of the original Rocky Flats list of chemicals was identified by CWI as the species that are considered to be problematic due to flammability and/or chemical reactivity. The subset of chemicals is shown in Table I.

Table I. Chemical list received from CWI.

Reactive Chemicals	Flammable	Reactive	Notes
1,4-dioxane	X	X	Flash point 12.2 °C (54 °F); explosive; peroxidizable
2-ethoxyethanol		X	Explosive; can form peroxides
Acetic anhydride		X	Reacts violently with water
Alkyl polyoxyalkylene glycol ether	X	X	Peroxidizable
Ammonium nitrate		X	Explosive and strong oxidizer; flash point 38.9 °C (102 °F)
Bromine		X	Strong oxidizing agent
Chromic acid		X	Strong oxidizing agent
Chromium nitrate		X	Strong oxidizing agent
Cumene hydroperoxide		X	Explosive; strong oxidizing agent
Diethyl ether	X	X	Flash point -45 °C (-49 °F); peroxidizable compound
Diethylene ether	X	X	Synonym for 1,4-dioxane
Glycol methylene ether	X	X	Flammable; peroxidizable; explosive
Hydrazine	X	X	Flash point 37.2 °C (99 °F); air reactive; strong reducing agent
Hydrogen peroxide (various concentrations)		X	Strong oxidizing agent
Hydroxylamine hydrochloride		X	Strong reducing agent
Hydroxylamine nitrate		X	Highly reactive decomposition
Isopropyl alcohol, isopropanol	X	X	Flash point 11.7 °C (53 °F); peroxidizable; explosive
Lithium hydride		X	Water reactive; air reactive; strong reducing agent
Magnesium perchlorate		X	Strong oxidizing agent
Nitric acid, various concentrations		X	Strong oxidizing agent
Nitrobenzene		X	Strong oxidizing agent
Nitrocellulose, collodion		X	Explosive; flash point -42.8 °C (-45 °F); lower explosive level 2%
Nitromethane	X	X	Flash point 35 °C (95 °F); strong oxidizing agent
Perchloric acid		X	Strong oxidizing agent
Picric acid		X	Explosive; shock sensitive
Potassium chromate		X	Strong oxidizing agent
Potassium dichromate		X	Strong oxidizing agent
Potassium permanganate		X	Strong oxidizing agent
Silver nitrate		X	Strong oxidizing agent
Sodium, elemental		X	Water reactive; air reactive
Sodium-potassium (NaK) alloy		X	Water reactive; air reactive
Sodium dichromate		X	Strong oxidizing agent
Sodium nitrate		X	Strong oxidizing agent
Sodium nitrite		X	Strong oxidizing agent
Sodium perchlorate		X	Strong oxidizing agent
Sodium peroxide		X	Reacts vigorously with air or water; strong oxidizing agent
Tetrahydrofuran	X	X	Flash point 14.4 °C (6 °F); peroxidizable; explosive
Toluene 2,4-diisocyanate,		X	Water reactive
Cyanide salts (Na, K)		X	
Magnesium powder		X	Flammable
NaK alloy		X	Water reactive
Molybdenum sulfide		X	

Each of the chemicals shown in Table I can be classified into 1 or more of 12 categories of chemical hazard types. Those categories include air reactive chemicals, water reactive chemicals, oxidizing/ reducing agents, acids, bases, flammable chemicals, explosive chemicals, peroxidizable chemicals, chlorinated solvents, sulfide salts solutions, and cyanide salt solutions. For testing purposes, a limited number of representative chemicals from each of the categories was selected. The air reactive chemical category was eliminated from the testing sequence as solidification of air reactive materials will not eliminate the hazard; as such, there was no reason to evaluate the sorption process for air reactive compounds. In field practice, if a liquid is observed to react with air after its bottle is opened, the reaction will be allowed to proceed to completion. If any residue remained after the material had reacted completely, that residue was combined with the sorbent blend. The explosive category was also eliminated as no liquid-phase, neat chemicals meeting the U.S. Environmental Protection Agency (EPA) or U.S. Department of Transportation definitions of "explosives" were identified on the original list of Rocky Flats chemicals. Nitromethane is a liquid at room temperature and with pressure and can be detonated by a strong initiator (e.g., blasting cap). However, such conditions should not occur during the exhumation of barrels at the RWMC and subsequent handling of the barrel contents. Consequently, this chemical category was also eliminated from testing. In field practice, potentially explosive solid chemicals, which were derived from or contained in liquids (e.g., crystallized picric acid or nitrocellulose), will be set aside for special handling.

The representative chemicals that were chosen for testing from each of the remaining chemical categories are listed in Table II with an explanation as to why they were selected. Some of the chemicals fall into more than one category of chemicals and were tested by chemical reactivity methods used for each of the chemical categories.

Table II. Neat chemicals selected by MSE and CWI for solidification and reactivity testing.

Chemical Category	Selected Chemical(s)	Explanations
Flammables	Methyl ethyl ketone and hydrazine	These materials were commonly used flammable liquids. Methyl ethyl ketone has a relatively low flash point [-21.1 °C (-6 °F)], and hydrazine undergoes very rapid oxidation under suitable conditions.
Acids	Nitric acid – 2 strengths	Nitric acid is commonly used at DOE facilities.
Bases	Ammonium hydroxide – 2 strengths	Ammonium hydroxide is a common base that is readily available.
Oxidizing agents	Nitric acid and sodium perchlorate solution	Nitric acid is commonly used at DOE facilities. Sodium perchlorate is among the stronger oxidizers on the Rocky Flats list of chemicals of which several perchlorate compounds are included.
Reducing agents	Hydrazine	Hydrazine is a strong reducing agent.
Water reactive	Acetic anhydride	This is the only water reactive liquid that is on the list of Rocky Flats chemicals.
Peroxidizables	1,4-dioxane and cumene hydroperoxide	1,4-dioxane readily forms peroxides upon exposure to air or sunlight. Cumene hydroperoxide is a viable surrogate for a peroxidizable component.
Sulfide salts	Sodium sulfide solution	Sodium sulfide is a common sulfide salt, and it can be used as a "sulfide standard."
Cyanide salts	Sodium cyanide solution	Potassium cyanide was specifically known to be included in some drums of Rocky Flats chemicals.
Chlorinated solvents	TCE, PCE, and 1,1,1-TCA*	These are common chlorinated solvents that were on the list of Rocky Flats chemicals.

*TCE = trichloroethylene, PCE = perchloroethylene (tetrachloroethene), 1,1,1-TCA = trichloroethane

Rinsate Descriptions

After consulting with the client and reviewing the Rocky Flats laboratory documentation [3], it was expected that approximately 90% of the liquids within the Rocky Flats bottles would be in the form of rinsates produced from the different laboratory procedures conducted at Rocky Flats. Consequently, several rinsate formulas were developed for inclusion as liquid surrogates in the sorbent testing matrix. A listing of the rinsate formulas tested for solidification is included in Table III.

Table III. Rinsate solutions selected by MSE and CWI for solidification testing.

Rinsate Category	Selected Chemical(s)	Recipe
Inorganic acids	Nitric acid	Volumetric dilution of concentrated nitric acid with deionized (DI) water at a ratio of 1:999.
Organic acids	Acetic acid – 2 strengths	1) Volumetric dilution of glacial acetic acid with DI water at a ratio of 1:1. 2) Volumetric dilution of glacial acetic acid with DI water at a ratio of 6:94.
Inorganic bases	Ammonium hydroxide	Volumetric dilution of concentrated ammonium hydroxide with DI water at a ratio of 1:999.
Organic bases	Hydrazine	Volumetric dilution of hydrazine with DI water at a ratio of 6:94.
Oxidizing agents	Sodium perchlorate solution	12.24 grams per liter (g/L) in DI water; therefore, a 0.1-molar (M) solution.
Water-miscible compounds	Isopropyl alcohol – 2 strengths	1) Volumetric dilution of isopropyl alcohol with DI water at a ratio of 1:1. 2) Volumetric dilution of isopropyl alcohol with DI water at a ratio of 6:94.
Water-immiscible compounds	Various chlorinated solvents – 2 strengths	1) Chloroform, di-chloromethane, PCE, TCE, and 1,1,1-TCA at solubility limits of each in DI water. 2) Volumetric dilution of above solution with DI water at a ratio of 12.5:87.5.
Neutralized acids	2 inorganic acids and a salt	950 milligrams per liter (mg/L) of NO_3^-, 35,000 mg/L of Cl^-, and 10-15 mg/L of F^-, with a pH between 8.0 and 9.0 in DI water.
Decontamination solution	2 strengths based on Kennedy Weber (KW) formula	3.17 g of Versene 100 solution; 3.38 g of anhydrous citric acid; and 2.80 g of Igepal CA-630 solution diluted to 1 L with DI water then diluted at a ratio of 1:99 with DI water.

Sorbent Descriptions

As stated earlier, candidate sorbents were identified by MSE from experience gained through previous sorbent testing and solidification projects. The sorbents selected for testing and a description of each is listed below. All of the sorbents listed are granular clay products that eliminate the need for mixing and ensure a uniform wasteform. As mentioned previously, no polymer sorbents were used for this test sequence due to the characteristics of some of the listed Rocky Flats laboratory chemicals.

- **Petroset II-Granular** – A modified clay-based, granular-form stabilizing agent that is manufactured by Fluid Tech, Inc. Petroset II-G is a solidification agent used for liquids that are essentially 100% water-immiscible organics.

- **Aquaset** – A water-activated, granular-form solidification agent used for aqueous liquids that can contain a small amount of dissolved salts and/or suspended solids, detergents, chelating agents, resins, and up to 5% oils. Aquaset is composed of clays modified by a proprietary process and is manufactured by Fluid Tech, Inc.
- **Aquaset II-Granular** – A granular solidification agent used for the solidification of aqueous solutions that are high in dissolved salts, such as neutralized acids and bases, and those organic liquids that are water soluble or miscible. Aquaset II-G is manufactured by Fluid Tech, Inc.

EXPERIMENTAL ACTIVITIES

Initially, the goal of the experimental work conducted in this project was to identify specific optimum sorbents or blends of sorbent materials that would solidify the individual bottles of unidentified liquids from Rocky Flats. In this scenario, each sorbent or blend of sorbents would be specialized dependent on the chemical characteristics of each type of liquid. In addition, the initial sorbent/surrogate addition method called for the sorbent to be added to the surrogate within a sealable container with other sorbent addition methods to be subsequently investigated. However, after discussions with the client, it was determined that the chemical characteristics of the various waste materials would not be known prior to the solidification process. As such, solidification of all the neat chemical and rinsate surrogate wastes would need to be accomplished using the same robust blend of sorbents. The selected blend of sorbents was identified as Recipe A. Additionally, all the sorbent/surrogate combinations should be prepared using the "tray preparation method" to better mimic the assigned field methods. In the tray preparation method, an appropriate quantity of sorbent is first spread on a flat tray, and the quantity of surrogate required to produce the assigned weight-based, waste-loading ratio was then poured onto the surface of the sorbent blend. The two compounds were then lightly folded together and scooped into a sealable container to allow the mixture to complete the process of solidification and curing.

In general, the tray preparation method of sample development was conducted within a laboratory hood. In those cases where the neat chemical being solidified was deemed to be hazardous, the solidification process and those process steps where contact with the neat chemical could occur were conducted in a sealed, pressurized, and vented glove box. Photographs showing the use of the glove box when generating the hydrazine wasteforms are shown in Fig. 1a and 1b.


a) b)

Fig. 1. Hydrazine sample generation with the selected sorbent blend in the glove box.

The sorbent/surrogate combinations were required to pass bench-scale stability and reactivity evaluations and remain stable under conditions that may be encountered during solidification, storage, shipping, and burial. Therefore, various stability evaluations were made on the sorbent/surrogate combinations including curing behavior, sorption capacity, sorption rate, and presence of free liquids in the final wasteform. Analytical test procedures were employed to test the reactivity of the solidified combinations that passed the bench-scale stability experiments. The solidified chlorinated solvents were not subjected to specific bench-scale reactivity experiments due to the lack of reactivity of these compounds.

BENCH-SCALE STABILITY EXPERIMENTS

The overall objective of the bench-scale stability experiments was to determine a waste-loading ratio for the selected sorbent blend that was capable of solidifying the unidentified Rocky Flats liquids and could maintain stability under a variety of conditions that may occur during solidification, storage, shipping, and burial of the solidified waste. From previous MSE experience and historical data, weight-based, waste-loading ratios of 2:1 and 3:1 (sorbent to surrogate) were initially identified for testing. As such, the majority of the surrogates within the neat chemical and rinsate categories were solidified at ratios of 2:1 and 3:1 using the selected blend of sorbents and allowed to cure. In those cases where both of these ratios failed to pass the initial testing regime, ratios of 4:1 and 5:1 were used for solidification.

Observations made during preparation of the selected chemical/sorbent combinations included any evidence of reaction between the surrogate and sorbent (i.e., fuming, fizzing, heat evolution, precipitation products, etc.), the volume of the mixture, the behavior of the tilted mixtures during curing, the pH of the surrogate and the mixture, any loss of stability of the combinations over time (i.e., separation of chemical-free liquid from the sorbent), and the consistency of the mixtures during curing. Observational information that was recorded during the preparation of the rinsate/sorbent combinations is shown in Table IV. Observational information that was recorded during the preparation of the neat chemical/ sorbent combinations is shown in Table V. The mixture consistencies reported in the two tables correspond to the consistency immediately after combining the liquids with the sorbent blend. As stated earlier, the liquid was poured onto the sorbent blend and folded into the sorbent with little mixing in an attempt to mimic field conditions. The descriptions for the consistency of the mixtures were dry, damp, moist, and wet. The dry condition corresponds to a combination that is dry to the touch and appears dusty, damp corresponds to a product that has no free liquid and is slightly more wet than the dry condition, moist corresponds to the condition where a film of liquid covers the sorbent and the mixture glistens slightly, and wet corresponds to some free liquid remaining in the combinations after slight mixing.

The samples were checked 2 hours after sample generation, and the consistency of all the samples was a dry to damp paste. All the liquids had been sorbed after 2 hours and, in general, the separate grains of sorbent had merged to form a paste. The sample consistency remained the same or slightly drier throughout the 2-week curing period as after the 2-hour sample consistency check.

The pH was taken for the neat chemicals and rinsate solutions prior to solidification with the selected sorbent blend. The pH of the liquid sorbent combinations was taken 2 hours after sample generation and every third day during the 14-day curing period. The mixture pH values shown in Table IV and V correspond to the 2-hour value and the 14-day value, respectively. The 2-hour and 14-day pH values vary only slightly, indicating that the pH values for the combinations remain reasonably constant after adding the liquid wastes to the sorbent blend.

Table IV. Observations made during surrogate rinsate/sorbent sample generation.

Rinsate Name and Description	Waste-Loading Ratio	Reaction	Surrogate Rinsate pH	Mixture pH	Mixture Consistency
Inorganic Acid					
Dilute nitric acid 1:999 with DI water	2:1	None	1.93	7.29 – 7.56	Damp paste
	3:1	None	1.93	7.88 - 7.92	Damp paste
Organic Acids					
Acetic acid, 1:1 with DI water	3:1	Foaming	1.45	3.10 - 3.13	Damp paste
	4:1	Foaming	1.45	3.21 - 3.30	Dry paste
Acetic acid, 6:94 with DI water	2:1	None	2.39	3.80 - 3.90	Damp paste
	3:1	None	2.39	3.85 - 3.92	Damp paste
Inorganic Base					
Ammonium hydroxide, 1:999 with DI water	2:1	None	10.40	8.39 - 8.56	Damp paste
	3:1	None	10.40	8.50 - 8.55	Damp paste
Organic Base					
Hydrazine, 6:94 with DI water	3:1	Exothermic	7.26	8.31 - 8.35	Damp paste
	4:1	Exothermic	7.26	8.56 - 8.60	Dry paste
Oxidizing Agent					
Sodium perchlorate solution - 0.01 M	2:1	None	5.47	9.31 - 9.37	Damp paste
Sodium perchlorate solution - 0.01 M	3:1	None	5.47	9.33 - 9.40	Damp paste
Water-Miscible Compounds					
Isopropyl alcohol, 1:1 with DI water	3:1	None	7.46	10.00 - 10.08	Damp paste
Isopropyl alcohol, 1:1 with DI water	4:1	None	7.46	10.08 - 10.10	Dry paste
Isopropyl alcohol, 6:94 with DI water	2:1	None	6.22	9.50 - 9.57	Damp paste
Isopropyl alcohol, 6:94 with DI water	3:1	None	6.22	9.61 - 9.70	Damp paste
Water-Immiscible Compounds					
Chloroform, di-chloromethane, PCE, TCE, and 1,1,1-TCA solubility limits in DI water	2:1	None	5.85	8.45 - 8.52	Damp paste
Chloroform, di-chloromethane, PCE, TCE, and 1,1,1-TCA solubility limits in DI water	3:1	None	5.85	8.51 - 8.57	Damp paste
Above diluted 12.5:87.5 with DI water	2:1	None	6.35	8.41 - 8.49	Damp paste
Above diluted 12.5:87.5 with DI water	3:1	None	6.35	8.51 - 8.56	Damp paste
Neutralized Acids					
950 mg/L NO$_3^-$, 35,000 mg/L Cl$^-$, and 12.5 mg/L DI water	2:1	None	9.13	9.25 - 9.29	Moist paste
	3:1	None	9.13	9.31 - 9.38	Moist paste
	4:1	None	9.13	9.46 - 9.52	Damp paste
	5:1	None	9.13	9.54 - 9.59	Dry paste
KW Decontamination Solutions					
3.17 g/L Versene 100, 3.38 g/L citric acid; and 2.80 g/L Igepal CA-630	2:1	None	1.52	7.84 - 7.90	Damp paste
3.17 g/L Versene 100, 3.38 g/L citric acid; and 2.80 g/L Igepal CA-630	3:1	None	1.52	7.85 - 7.90	Damp paste
Above diluted 1:99 with DI water	2:1	None	3.53	8.71 - 8.79	Damp paste
Above diluted 1:99 with DI water	3:1	None	3.35	8.72 - 8.79	Damp paste

Table V. Observations made during neat chemical/sorbent sample generation.

Neat Chemical Name and Description	Waste-Loading Ratio	Reaction	Chemical Surrogate pH	Mixture pH	Mixture Consistency
Flammables					
Methyl ethyl ketone	2:1	None	2.16	7.84 – 7.88	Moist grains
Methyl ethyl ketone	3:1	None	2.16	7.96 – 8.00	Moist grains
Acids					
Nitric acid – 11N[*]	2:1	Strong acid gas and minor foaming	-0.9	0.36 – 0.45	Moist grains
Nitric acid – 11N	3:1	Strong acid gas and minor foaming	-0.9	0.60 – 0.66	Damp paste
Nitric acid – 1N	2:1	Moderate foaming	0.60	2.17 – 2.20	Moist paste
Nitric acid – 1N	3:1	Moderate foaming	0.60	2.38 – 2.42	Damp paste
Bases					
Ammonium hydroxide – 11N	2:1	None	14.13	11.8 – 11.82	Moist grains
Ammonium hydroxide – 11N	3:1	None	14.13	11.97 – 11.95	Moist grains
Ammonium hydroxide – 5N	2:1	None	12.70	11.41 – 11.42	Moist grains
Ammonium hydroxide – 5N	3:1	None	12.70	11.73 – 11.75	Moist grains
Oxidizing Agents					
Sodium perchlorate solution – 0.1 M	2:1	None	5.97	9.74 – 9.80	Moist grains
Sodium perchlorate solution – 0.1 M	3:1	None	5.97	9.95 – 9.97	Moist grains
Sodium perchlorate solution – 0.1M	4:1	None	5.97	10.13 – 10.16	Moist grains
Sodium perchlorate solution – 0.1 M	5:1	None	5.97	10.06 – 10.12	Moist grains
Reducing Agents					
Hydrazine	3:1	Exothermic	7.41	8.61 – 8.63	Damp grains
Hydrazine	4:1	Exothermic	7.41	8.73 – 8.77	Damp grains
Water Reactive					
Acetic anhydride	3:1	None	5.61	3.39 – 3.43	Moist grains
Acetic anhydride	4:1	None	5.61	3.66 – 3.71	Moist grains
Peroxidizables					
1,4-dioxane	2:1	None	4.35	9.15 – 9.20	Moist grains
1,4-dioxane	3:1	None	4.35	9.35 – 9.40	Moist grains
Cumene hydroperoxide	3:1	None	7.83	8.94 – 8.94	Wet grains
Cumene hydroperoxide	4:1	None	7.83	8.86 – 8.95	Moist grains
Chlorinated Solvents					
TCE	2:1	None	9.58	7.35 – 7.44	Damp grains
TCE	3:1	None	9.58	7.40 – 7.47	Damp grains
1,1,1-TCA	2:1	None	9.31	7.41 – 7.49	Damp grains
1,1,1-TCA	3:1	None	9.31	7.50 – 7.54	Damp grains
PCE	2:1	None	8.74	7.95 – 8.01	Damp grains
PCE	3:1	None	8.74	8.01 – 8.05	Damp grains
TCE, PCE, and 1,1,1-TCA (1:1:1)	2:1	None	8.52	8.37 – 8.41	Damp grains
TCE, PCE, and 1,1,1-TCA (1:1:1)	3:1	None	8.52	8.40 – 8.46	Damp grains
Sulfide Salts					
Sodium sulfide solution	2:1	None	11.86	10.79 – 10.84	Moist grains
Sodium sulfide solution	3:1	None	11.86	10.91 – 10.95	Moist grains
Sodium sulfide solution – oxidized	2:1	None	12.04	10.95 – 11.00	Moist grains
Sodium sulfide solution – oxidized	3:1	None	12.04	10.93 – 10.98	Moist grains
Cyanide Salts					
Sodium cyanide solution	2:1	None	12.20	10.94 – 11.00	Moist grains
Sodium cyanide solution	3:1	None	12.20	10.95 – 11.00	Moist grains
Sodium cyanide solution – oxidized	2:1	None	12.35	10.98 – 11.04	Moist grains
Sodium cyanide solution – oxidized	3:1	None	12.35	11.10 – 11.15	Moist grains

[*] N = Normal

The selected sorbent blend had a buffering effect on most of the neat chemicals and rinsate liquids. Upon inspection of Table IV, the buffering effect is apparent. The rinsate surrogate pH values ranged from 1.45 to 10.40. The pH values for the solidified rinsate solutions ranged from 7.29 to 10.10 with the exception of the acetic acid rinsate samples. However, the sorbent blend did raise the pH for the acetic acid samples above the corrosive threshold, which is less than 2. The neat chemicals had pH values ranging from -0.9 for the 11N nitric acid sample up to 14.13 for the 11N ammonium hydroxide sample. The sorbent blend had a significant buffering effect on all of the solidified neat chemical wasteforms with the exception of the 11N nitric acid samples; however, the blend did raise the pH of the 1N nitric acid out of the corrosive range. The sorbent blend had a surprising effect on the strong base sorbent combinations by lowering the pH values to below the corrosive threshold of 12. The pH values for the rest of the solidified neat chemicals ranged from 7.35 to 11.15.

After the 14-day curing period, PFT and LRT were performed according to SW-846, Method 9095B, Paint Filter Free Liquids Test [1] and SW-846, Method 9096, Liquids Release Test Procedure [2] to determine if free liquids exist and what the quantity of liquids were in the final wasteforms. Only samples that passed the PFT were subjected to LRT since any loaded sorbent that fails the PFT is assumed to release liquids if subjected to pressure during the LRT. Any samples that failed the LRT were prepared again with higher ratios of the sorbent blend. The PFT and LRT results for the surrogate rinsate sorbent combinations are presented in Table VI, and the results for the neat chemical sorbent combinations are presented in Table VII. The LRT values that are in bold print in Tables VI and VII represent samples that did not pass the WIPP WAC of 1% liquid release by volume.

As shown in Tables VI and VII, all of the solidified neat chemicals and rinsate solutions wasteforms passed the PFT and the LRT WAC of 1% release by volume at the ratio of 3:1 except the neutralized acid rinsate sample and the sodium perchlorate rinsate solution. These two rinsate solutions were solidified using a 4:1 and a 5:1 ratio of sorbent to surrogate, and both rinsates passed the PFT and the LRT WAC for both of those ratios. Because the selected sorbent blend (Recipe A) was able to control the liquid released from the samples at the tested ratios, the testing proceeded to the reactivity testing phase. The reactivity samples were tested at the waste-loading ratios determined during the stability experiments.

BENCH-SCALE REACTIVITY EXPERIMENTS

The objective of the bench-scale reactivity experiments was to determine whether the chemical and sorbent combinations were still reactive according to test methods specific to the chemical category for each of the solidified chemicals and rinsate solutions. Flammability, ignitability, and cyanide and sulfide testing was performed on the solidified wasteforms from the specific chemical category. Each of the chemical categories and the test method that was used to test the chemical reactivity of the solidified chemical and sorbent wasteforms are discussed below.

Flammable Chemical Reactivity Testing

The first test that was performed on the solidified flammable wasteforms was American Society for Testing and Materials (ASTM) D4982-95, Standard Test Methods for Flammability Potential Screening Analysis of Waste, Test Method A, Test Sample Exposed to Heat and Flame [4]. This test method can be applied to waste liquids, sludges, or solids and was used to indicate the fire-producing or fire-sustaining potential of the wasteforms. The second test that was performed was SW-846, Method 1030, Ignitability of Solids [5]. This method is suitable for the determination of the ignitability of solids and is appropriate for pastes, granular materials, solids that can be cut into strips, and powdery substances. This method may also be used (but is not required) to determine whether a solid waste, "when ignited burns so vigorously and persistently that it creates a hazard[5]."

Table VI. PFT and LRT results for the surrogate rinsate/sorbent samples.

Rinsate Name and Description	**Weight-Based, Waste-Loading Ratio (sorbent to liquid)**	**PFT Pass/Fail**	**LRT* Percent Liquid Released by Volume**
Inorganic Acid			
Dilute nitric acid 1:999 with DI water	2:1	Pass	**1.578**
Dilute nitric acid 1:999 with DI water	3:1	Pass	0.085
Organic Acid			
Acetic acid, 1:1 with DI water	3:1	Pass	0.031
Acetic acid, 1:1 with DI water	4:1	Pass	0.026
Acetic acid, 6:94 with DI water	2:1	Pass	**1.627**
Acetic acid, 6:94 with DI water	3:1	Pass	0.089
Inorganic Base			
Ammonium hydroxide, 1:999 with DI water	2:1	Pass	**1.592**
Ammonium hydroxide, 1:999 with DI water	3:1	Pass	0.190
Organic Base			
Hydrazine, 6:94 with DI water	3:1	Pass	0.051
Hydrazine, 6:94 with DI water	4:1	Pass	0.049
Oxidizing Agent			
Sodium perchlorate solution - 0.1 M	2:1	Pass	**1.688**
Sodium perchlorate solution - 0.1 M	3:1	Pass	0.048
Water-Miscible Compounds			
Isopropyl alcohol, 1:1 with DI water	3:1	Pass	0.031
Isopropyl alcohol, 1:1 with DI water	4:1	Pass	0.029
Isopropyl alcohol, 6:94 with DI water	2:1	Pass	**1.518**
Isopropyl alcohol, 6:94 with DI water	3:1	Pass	0.145
Water-Immiscible Compounds			
Chloroform, di-chloromethane, PCE, TCE, and 1,1,1-TCA solubility limits in DI water	2:1	Pass	**1.482**
Chloroform, di-chloromethane, PCE, TCE, and 1,1,1-TCA solubility limits in DI water	3:1	Pass	0.031
Above diluted 12.5:87.5 with DI water	2:1	Pass	**1.513**
Above diluted 12.5:87.5 with DI water	3:1	Pass	0.075
Neutralized Acids			
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	2:1	Pass	**3.582**
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	3:1	Pass	**2.178**
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	4:1	Pass	0.029
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	5:1	Pass	0.034
KW Decontamination Solutions			
3.17 g/L Versene 100, 3.38 g/L anhydrous citric acid; and 2.80 g/L Igepal CA-630	2:1	Pass	**1.683**
3.17 g/L Versene 100, 3.38 g/L) anhydrous citric acid; and 2.80 g/L Igepal CA-630	3:1	Pass	0.040
Above diluted 1:99 with DI water	2:1	Pass	**1.709**
Above diluted 1:99 with DI water	3:1	Pass	0.041
*WIPP Criteria - 1% Release by Volume			

Table VII. PFT and LRT results for the neat chemical/sorbent samples.

Neat Chemical Name and Description	**Weight-Based, Waste-Loading Ratio (sorbent to liquid)**	**PFT Pass/Fail**	**LRT * Percent Liquid Released by Volume**
Flammables			
Methyl ethyl ketone	2:1	Pass	0.015
Methyl ethyl ketone	3:1	Pass	0.018
Hydrazine	3:1	Pass	0.047
Hydrazine	4:1	Pass	0.030
Acids			
Nitric acid – 11N	2:1	Pass	0.032
Nitric acid – 11N	3:1	Pass	0.017
Nitric acid – 1N	2:1	Pass	**1.695**
Nitric acid – 1N	3:1	Pass	0.035
Bases			
Ammonium hydroxide – 11N	2:1	Pass	**1.874**
Ammonium hydroxide – 11N	3:1	Pass	0.040
Ammonium hydroxide – 5N	2:1	Pass	**1.638**
Ammonium hydroxide – 5N	3:1	Pass	0.035
Oxidizing Agents			
Nitric acid – 11N	2:1	Pass	0.032
Nitric acid – 11N	3:1	Pass	0.017
Nitric acid – 1N	2:1	Pass	**1.695**
Nitric acid – 1N	3:1	Pass	0.035
Sodium perchlorate solution – 0.1N	2:1	Pass	**1.656**
Sodium perchlorate solution – 0.1N	3:1	Pass	**1.276**
Sodium perchlorate solution – 0.1N	4:1	Pass	0.028
Sodium perchlorate solution – 0.1N	5:1	Pass	0.035
Reducing Agents			
Hydrazine	3:1	Pass	0.047
Hydrazine	4:1	Pass	0.030
Water Reactive			
Acetic anhydride	3:1	Pass	0.029
Acetic anhydride	4:1	Pass	0.025
Peroxidizables			
1,4-dioxane	2:1	Pass	**1.037**
1,4-dioxane	3:1	Pass	0.024
Cumene hydroperoxide	3:1	Pass	0.008
Cumene hydroperoxide	4:1	Pass	0.008
Chlorinated Solvents			
TCE	2:1	Pass	0.003
TCE	3:1	Pass	0.004
1,1,1-TCA	2:1	Pass	0.003
1,1,1-TCA	3:1	Pass	0.003
PCE	2:1	Pass	0.003
PCE	3:1	Pass	0.003
TCE, PCE, and 1,1,1-TCA (1:1:1)	2:1	Pass	0.005
TCE, PCE, and 1,1,1-TCA (1:1:1)	3:1	Pass	0.004

Neat Chemical Name and Description	**Weight-Based, Waste-Loading Ratio (sorbent to liquid)**	**PFT Pass/Fail**	**LRT * Percent Liquid Released by Volume**
Sulfide Salts			
Sodium sulfide solution	2:1	Pass	**2.026**
Sodium sulfide solution	3:1	Pass	0.254
Sodium sulfide solution – oxidized	2:1	Pass	**1.981**
Sodium sulfide solution – oxidized	3:1	Pass	0.309
Cyanide Salts			
Sodium cyanide solution	2:1	Pass	**1.847**
Sodium cyanide solution	3:1	Pass	0.241
Sodium cyanide solution – oxidized	2:1	Pass	**1.693**
Sodium cyanide solution – oxidized	3:1	Pass	0.262
* WIPP Criteria - 1% Release by Volume			

The chemicals selected for flammable testing were methyl ethyl ketone (2-butanone) and hydrazine. Quantities of each of these chemicals were solidified during the bench-scale stability experiments at waste-loading ratios of 2:1 and 3:1 for methyl ethyl ketone and 3:1 and 4:1 for hydrazine. Subsequent to the completion of the curing period, samples of each solidified wasteform were sent for reactivity testing.

All four samples were initially screened for flammability using ASTM D4982-95. The two hydrazine-based samples would not ignite and therefore were not deemed to be flammable under the conditions of this test. The solidified hydrazine samples were then tested for ignitability by SW-846, Method 1030, and determined to be noncombustible. The two methyl ethyl ketone-based samples ignited and were deemed to be flammable under ASTM D4982-95. These two samples were therefore subjected to SW-846, Method 1030, to determine if the materials were capable of propagating combustion and, secondarily, if propagation occurred, the rate at which such propagation progressed would be determined. Both of the methyl ethyl ketone-based samples were deemed to be capable of propagating combustion and therefore were deemed to be ignitable under SW-846, Method 1030. In addition, both materials propagated combustion along a 250-millimeter (mm) (9.843-inch) length strip of sample in less than 2 seconds. Both methyl ethyl ketone-based samples exhibited a propagation rate of approximately 40 mm (1.575 inches) per second.

(Note: Propagation of combustion in SW-846, Method 1030, does not mean the solid waste meets the definition of a Resource Conservation and Recovery Act (RCRA) characteristic ignitable waste as described in 40 Code of Federal Regulations (CFR) 261.21 (a)(2) [6].)

The sorbent blend seems to limit the flammable reactivity for one of the chemicals tested but not the other, indicating that the solidified wasteforms should be containerized separately after solidification to ensure there is no contact with other solidified wasteforms.

Acid and Base Chemical Reactivity Testing

MSE performed all of the acid and base reactive testing on the solidified chemical acidic and basic wasteforms. The tests were conducted according to ASTM D4980-89, Standard Test Methods for Screening of pH in Waste [7]. Test Method B, pH Screening by Electrometric Measurement was used to determine the pH of the solidified chemical wasteforms. As such, the pH of the solidified surrogate/ sorbent mixture was used as the determining factor relative to the reactivity for the acidic and basic solidified samples.

Three strengths of nitric acid were used to generate the solidified acidic wasteforms, while hydrazine and two strengths of ammonium hydroxide were used to generate the solidified basic wasteforms. The pH of all of the prepared samples (not only the acidic and basic samples) were tested during the bench-scale stability experiments by mixing 2 g of the solidified material with 20 g of DI water to form a 10% slurry. The pH of the slurry was then determined using a calibrated pH meter and electrode. The samples were tested immediately after solidification, 2 hours after solidification, and every 3 days during the curing period. The range of pH values measured for each sample is shown in Tables IV and V. The first value shown in the tables is the pH taken immediately after solidification, while the second value is the pH taken on the last day of the curing period. For the most part, the pH values changed only slightly throughout the curing period, usually rising by less than 0.10 pH units.

The pH values for the rinsate solutions made with the inorganic acid solutions plus the inorganic and organic base solutions were buffered into the neutral range after solidification with the sorbent blend. The organic acid rinsate solution made using acetic acid was the only acid or base rinsate that was not buffered to the neutral range after solidification. However, the sorbent blend did raise the pH for those two rinsate solutions out of the corrosive threshold ($pH < 2$).

The pH values for the neat base solutions of 11N and 5N ammonium hydroxide were decreased from initial values of 14.13 and 12.70, respectively, to pH values in the range of 11.41 to 11.97, just out of the corrosive threshold of above 12. The sorbent blend did raise the pH values for the 1N nitric acid samples from 0.60 to a range of 2.17 to 2.42; however, the blend had little effect on the 11N acid samples.

In general, the pH values for the rest of the cured mixtures were buffered in the range of approximately 7.0 to 10.0 except for the neat acidic and basic samples and the organic acid rinsate samples generated with acetic acid.

After the nitric acid and ammonium hydroxide wasteforms were tested for PFT and LRT, they were placed in individual waste buckets outside the laboratory. It was observed that the gases from the two solidified wasteforms seemed to be commingling in the airspace between the two waste buckets to form a gas and that the solidified nitric acid samples were giving off much more gas than the solidified ammonium hydroxide samples. Photographs were taken of the samples and are presented in Fig. 2. The ammonium hydroxide samples were then added to the nitric acid samples in the acid waste bucket, and the resulting gas production is shown in Fig. 3. The temperature raised 22.8 °C (73 °F) when the basic waste samples were added to the waste acid samples. This will be discussed later in the paper.

Because gas production resulted when the samples were in close proximity with each other, it was decided to test the other solidified wasteforms for proximity and contact reactivity, and this will be discussed later in this paper.

Oxidizing Agent Reactive Chemical Reactivity Testing

MSE performed the oxidizing agent chemical reactive testing on the solidified chemical wasteforms generated at several waste-loading ratios during the bench-scale stability experiments. The tests were conducted according to ASTM D4981-95, Standard Test Method for Screening Oxidizers in Waste [8]. This test method is applicable to the analysis of waste liquids, sludges, and solids.

Fig. 2. Proximity chemical reactivity test of solidified nitric acid (lower bucket) and solidified ammonium hydroxide (upper bucket) showing fumes produced from acid bucket.

Fig. 3. Contact chemical reactivity test of solidified nitric acid and solidified ammonium hydroxide showing fumes produced due to contact of wasteforms.

Sodium perchlorate (0.01 and 0.1 M) and three concentrations of nitric acid (rinsate, 1N, and 11N) solutions were solidified with the selected blend of sorbents to generate the oxidizing chemical wasteforms during the bench-scale stability experiments. The oxidizer content of those solidified wasteforms was tested during the bench-scale stability experiments by mixing 2 to 3 g of the solidified material with 3 g of DI water to form a slurry. A drop of the slurry was then applied to a strip of potassium iodide starch paper, and any color change to the paper was noted. A blue color change indicated the presence of potentially hazardous oxidizing compounds in the tested slurry. This test was performed on the samples immediately after solidification, 2 hours after solidification, and every 3 days during the curing period. No color was shown for any of the samples prepared with sodium perchlorate at the four waste-loading ratios tested. Also, no color was seen in the samples of the 1N or rinsate concentrations of the nitric acid wasteforms. Color was only shown for the samples prepared with 11N nitric acid at both 2:1 and 3:1 waste-loading ratios. This indicates that the selected sorbent blend limits the oxidizing capacity of the chemicals tested with the exception of the very strong acid. Since the sorbent blend does not limit the oxidizing potential for all of the chemicals tested, the solidified wasteforms should be containerized separately before disposal.

Reducing Agent Reactive Chemical Reactivity Testing

MSE performed a test for reducing agent chemical reactivity that used ammonium molybdate [9] to produce molybdenum blue as an indicator of the reactivity of the solidified wasteforms. MSE’s literature review indicated that no particular qualitative ("spot") test existed for all organic functional (e.g., aldehyde, amine) groups that can serve as reducing agents. As such, the test using ammonium molybdate was used to evaluate the presence of reducing agents in the solidified samples. Testing was conducted on four samples formed by solidifying hydrazine and a hydrazine rinsate at two waste-loading ratios with Recipe A.

The reducing agent content of the solidified wasteforms was tested during the bench-scale stability experiments by mixing 2 to 3 g of the solidified material with 5 g of DI water to form a slurry. The slurry was then mixed with the appropriate quantity of ammonium molybdate to form molybdenum blue. Any color change to the slurry was noted. A blue color change indicated the presence of reducing compounds in the tested slurry. This test was performed on the samples immediately after solidification, 2 hours after solidification, and every 3 days during the curing period. No color was shown for any of the four samples prepared with either neat hydrazine or hydrazine rinsate at any time during the testing sequence, indicating the selected sorbent blend inhibits the reduction potential for hydrazine solidified with the selected sorbent blend.

Water Reactive Chemical Reactivity Testing

MSE conducted water reactive chemical testing on two samples of acetic anhydride solidified at waste-loading ratios of 3:1 and 4:1 with the selected blend of sorbents during the bench-scale stability experiments. The tests were conducted according to ASTM D5058-90, Standard Test Methods for the Compatibility of Screening Analysis of Waste, Test Method C, Water Reactivity [10]. This test method was used to determine whether the samples had the potential to generate extreme heat or violent reactions and produce fumes, dusts, or other products when mixed with water. The test was performed by mixing 1 g of the solidified material with 10 g of DI water and by noting any reactions that occurred. As no violent or obvious reactions occurred during the mixing, a temperature probe was used to determine any rise in temperature that occurred during the test. As in the previous tests, this test was performed on the samples immediately after solidification, 2 hours after solidification, and every 3 days during the curing period. No reactions or temperature increases occurred during any of the tests conducted during the water reactive chemical testing sequence, indicating the selected sorbent blend inhibits the water reactivity for acetic anhydride, which was the only water reactive chemical on the CWI list.

Peroxidizable Chemical Reactivity Testing

MSE used peroxide test strips to determine the amount of organic peroxide present in the samples of 1,4-dioxane and cumene hydroperoxide prepared during the bench-scale testing regime [11]. Samples of each neat chemical were solidified with the selected blend of sorbents at waste-loading ratios of 2:1 and 3:1 for dioxane and 3:1 and 4:1 for cumene hydroperoxide. Per the manufacturer's instructions, a slurry of each solidified chemical was prepared at a ratio of 2 g of solid to 10 g of DI water. The slurries were then allowed to settle for approximately 2 to 3 minutes. The test strip was then wetted with the liquid portion of the slurry. The color generated on the test strip was then compared to a series of known colors provided with the test strips to determine the concentration of organoperoxide in the slurry. Two types of test strips were used in the testing. The first type of strip presents semi-quantitative readings between 100 and 1,000 mg/L peroxide and provides warning coloration for peroxide levels greater than 2,000 mg/L. The second type of strip identifies quantitative readings between 1 and 100 mg/L peroxide. After the initial test group was completed, only the second type of strip was used as the quantity of peroxide did not reach the higher levels indicated for the first strip. As in the previous tests, this test was performed on the samples immediately after solidification, 2 hours after solidification, and every 3 days during the curing period.

The tests conducted on both samples of solidified 1,4-dioxane immediately after solidification did not indicate the presence of any peroxide in the sample slurries. However, tests for both of the 1,4-dioxane samples performed 2 hours after solidification indicated the presence of 1-mg/L peroxide. The samples tested at periods of time greater then 2 hours after solidification throughout the curing period did not indicate the presence of any peroxide in the slurries prepared from the 1,4-dioxane samples.

The tests conducted immediately after solidification on the samples prepared with cumene hydroperoxide at both the 3:1 and 4:1 waste-loading ratios indicated the presence of 40 mg/L of peroxide. This value decreased to 30 mg/L for both samples 2 hours after solidification. Twenty-four hours after solidification, the sample solidified at a waste-loading ratio of 3:1 contained 20 mg/L of peroxide. This sample contained 15 mg/L peroxide 96 hours after solidification and remained at that concentration throughout the curing period. The sample of cumene hydroperoxide solidified at a waste-loading ratio of 4:1 contained 15 mg/L 24 hours after solidification and dropped to 10 mg/L in the sample tested at 96 hours after solidification and remained at that value throughout the remainder of the curing period. This indicates the selected sorbent blend decreases the peroxide concentration in the solidified cumene hydroperoxide wasteforms over the short term.

Sulfide Salt Chemical Reactivity Testing

Four samples of sodium sulfide were solidified with the sorbent blend: two were oxidized, and two were not oxidized, and both sets of samples were generated at the waste-loading ratios of 2:1 and 3:1 (sorbent to surrogate). The initial concentration of the sodium sulfide solution used to generate the solidified samples was 770 mg/L. Two samples were solidified without oxidizing the sodium sulfide. Initial sulfide concentration for the unoxidized solidified sample at the 2:1 waste-loading ratio corresponded to a concentration of 257 milligrams per kilograms (mg/kg), and the initial sulfide concentration for the unoxidized solidified sample at the 3:1 waste-loading ratio corresponded to a concentration of 193 mg/kg. The initial concentrations for the sodium sulfide solution oxidized with potassium permanganate was unknown; consequently, the initial sodium sulfide concentration for the solidified samples was unknown as well.

MSE used ASTM D4978-95, Standard Test Methods for Screening of Reactive Sulfides in Waste [12], to determine the concentration of sulfide in those four samples immediately after solidification, 2 hours after solidification, and every 3 days throughout the curing period. After completion of the curing period, samples of all four of the solidified materials were sent for analysis to determine the concentration of sulfide and sulfate in the solidified wasteforms.

The use of ASTM D4978-95 for a spot test during the curing period involved preparing a slurry of 5 to 10 g of the solid material with 15 mL of DI water in a small beaker. A strip of lead acetate paper was dampened and adhered to the wall of the beaker above the slurry. The slurry was then acidified with hydrochloric acid to a pH of less than 2.0, which causes the reactive sulfides present in the slurry to form hydrogen sulfide gas. This gas reacts with the lead present in the paper to produce a silvery-gray coloration due to lead sulfide being formed on the paper. The presence of the gray color indicates the presence of reactive sulfides in the solidified material. The following two paragraphs explain the results for the spot tests and the analytical results for the oxidized and unoxidized samples.

The two samples of solidified, unoxidized sodium sulfide solution showed positive indications of the presence of sulfide in the test slurry in both of the samples tested using the ASTM spot test during curing. The two samples of solidified, unoxidized sodium sulfide solution submitted to the analytical laboratory for quantitative analysis contained concentrations of 29.1 and 28.2 mg/kg of sulfide in the 2:1 and 3:1 waste-loaded samples, respectively, compared to the initial concentrations of 257 mg/kg and 193 mg/kg. It should be noted that both of the sulfide concentrations for the solidified samples were lower than the sulfide destruction limits of less than 56 mg/kg stated in the project test plan. This indicated that the sulfide might have reacted with the sorbent blend to form sulfates. Therefore, the samples were analyzed for sulfates; the sulfate concentration for the sample generated at the waste-loading ratio of 3:1 had a sulfate concentration of 1,500 mg/kg, and the sample generated at the 2:1 waste-loading ratio had a sulfate concentration of 1,700 mg/kg. This seems to indicate that the sulfide solution reacts with the clay sorbents to produce sulfate and therefore limits the sulfide content in the solidified wasteforms.

The two samples of solidified sodium sulfide solution that had been oxidized with potassium permanganate did not indicate the presence of sulfide in the test slurry using the ASTM spot test during the curing period. The two oxidized sodium sulfide solution samples that were submitted to the analytical laboratory for quantitative analysis contained concentrations of 13.8 and 3.5 mg/kg of sulfide in the 2:1 and 3:1 waste-loaded samples, respectively. These two samples contained concentrations that were also well below the concentration limits called out in the project test plan. This shows that both the oxidized and unoxidized sodium sulfide solution samples were below the accepted final concentration values. This indicates that the sulfide samples may not need to be oxidized before the solidification process.

Cyanide Salt Chemical Reactivity Testing

Four cyanide-bearing samples were solidified with the selected blend of sorbents using a cyanide solution with a concentration of 900 mg/L cyanide. Two of these samples were prepared by solidifying unoxidized sodium cyanide solution at waste-loading ratios of 2:1 and 3:1. The other two samples were oxidized with potassium permanganate prior to solidification at the same waste-loading ratios. After the curing period, the four solidified cyanide samples were submitted to the analytical laboratory to quantify the cyanide concentration levels in the samples. The two unoxidized, solidified cyanide samples contained concentrations of cyanide that were below the detection limits of the analytical technique while the two oxidized, solidified cyanide samples contained concentrations of 120 and 180 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively. The cyanide destruction limit used for this testing was less than or equal to 22 mg/kg of cyanide for the solidified cyanide wasteforms.

As can be seen from these analytical results, the cyanide concentrations found in the oxidized samples were greater than expected while the concentrations found in the unoxidized samples were much less than expected. After determining that the analyses were correct for the specific samples, MSE determined that the oxidation process for cyanide by potassium permanganate did not lower the cyanide concentration to the level required and therefore would have to be increased. In addition, MSE hypothesized that cyanide was being broken down in the pale-colored, unoxidized cyanide solution wasteforms by the light under which the samples were stored throughout the curing period and the waiting period before analysis in the analytical laboratory. MSE also hypothesized that the cyanide in the solidified samples oxidized with the potassium permanganate was not being further broken down by light exposure due to the darker color of the treated samples. In addition, the smaller sample container used for the analytical laboratory samples was more conducive to the permeation of light into the solid materials. Fig. 4 illustrates the variation in color between the oxidized samples and the unoxidized samples. Fig. 5 shows the types of containers used for samples during the curing period and for the period of time awaiting laboratory analysis.

To test this hypothesis, two additional groups of four samples each were prepared with the same 900-mg/L cyanide solution and the same waste-loading ratios of 2:1 and 3:1 as the first sample group. In the second group, the four samples were prepared in the same manner as the first group of samples except the amount of potassium permanganate used in the oxidation process was more than tripled, and the amount of time allowed to pass during the oxidation process prior to solidification was doubled. In the third group, the four samples were prepared in the same manner as the second except the sample containers were wrapped in aluminum foil to prevent light exposure during the 14-day curing period. All samples from groups two and three were wrapped in aluminum foil after the curing period prior to being submitted to the analytical laboratory to eliminate any further destruction by light before sample analysis.

Fig. 4. An oxidized sample of solidified cyanide solution (left) and an unoxidized sample of solidified cyanide solution (right) from the first sample group.

Fig. 5. Sample containers for the curing period (right) and the period awaiting laboratory analysis (left).

The results of the laboratory analyses of the samples from the second and third groups of cyanide samples are: 1) the two unoxidized, solidified cyanide samples from group number two contained concentrations of 179 and 132 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively, 2) the two oxidized, solidified samples from that sample group contained concentrations of 0.276 and 0.202 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively. The results show that the additional potassium permanganate and longer oxidation time destroys the cyanide before solidification with the selected sorbent blend.

The two unoxidized, solidified samples from group number three contained concentrations of 270 and 177 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively. The two oxidized, solidified cyanide samples from that sample group contained concentrations of 4.39 and 0.369 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively. Such results again demonstrate that the higher dosage of the oxidizer and longer oxidation times destroy the cyanide prior to solidification. These results also indicate that the samples exposed to light have a smaller concentration of cyanide in the wasteforms.

As can be seen from these results, the analytical data from the second and third groups of samples represent the concentrations that should theoretically be present in the specific samples. That is, there is more cyanide in the unoxidized, solidified cyanide solution samples than the samples oxidized by the larger quantities of potassium permanganate. In addition, higher concentrations of cyanide are present in those samples solidified at ratios of 2:1 (sorbent to liquid) compared to the samples solidified at ratios of 3:1 due to simple dilution. Also, the group three samples that were protected from light contain more cyanide than those samples that were exposed to light.

The oxidation of cyanide by potassium permanganate is not simple as a number of interfering reactions can occur during the process. All of these interfering reactions serve to rob the process of the permanganate ion, allowing cyanide to remain unoxidized in the solidified material. However, as cyanide is a relatively unstable compound, it can be destroyed by oxidation with potassium permanganate, but the amount of permanganate that is required should be three to four times the stoichiometric value.

The oxidized samples in groups two and three contained concentrations of cyanide that were well below the concentration value deemed to be adequate for the cyanide destruction limits called out in the test plan of less than or equal to 22 mg/kg of cyanide for the solidified cyanide wasteforms.

Proximity Chemical Reactivity Testing

The objective of the bench-scale, proximity chemical reactivity testing was to determine whether any of the solidified wasteforms were capable of reacting with any of the other solidified wasteforms while simply being placed in the proximity of each other and/or coming in contact with each other. The method used for this testing was to place a small (approximately 5 g) quantity of each of the selected wasteforms close to each other on a ceramic plate and determine if a reaction was occurring. The occurrence of a reaction was defined as fuming, fizzing, spitting, and/or heat evolution. If no reaction occurred by placing the wasteforms in the vicinity of each other, the materials were brought into contact with each other and again observed to see if a reaction occurred.

In the case of the solidified nitric acid and solidified ammonium hydroxide, the wasteforms were brought into proximity with each other during the process of disposing of the samples after the LRT testing as previously shown in Fig. 2. As such, significantly larger quantities (1 to 2 L) of these two solidified wasteforms were used for proximity testing as compared to the tests conducted with the other wasteforms. Fig. 2 shows the gas evolving from the nitric acid sample but not from the ammonium hydroxide sample; however, a faint odor of ammonia was detected. Fig. 3 shows the gas produced when the ammonium hydroxide and the nitric acid wasteforms were combined. A distinct odor of ammonium was noticed when the two wasteforms were combined. The wasteform temperature was increased by 15 °C (59 °F) as a result of the reaction mentioned when the wasteforms were combined.

The pairs of solidified wasteforms that were tested at the small scale as well as the proximity and contact reactions that were produced in this series of tests are shown in Table VIII.

Table VIII. Proximity chemical reactive testing results.

Reaction Pair	Proximity Reaction	Contact Reaction
nitric acid and ammonium hydroxide	No discernable reaction	Fumes produced, temperature increase 15 °C (59 °F).
nitric acid and hydrazine	Fuming by hydrazine	Fumes produced, temperature increase 2.8 °C (37 °F).
nitric acid and cumene hydroperoxide	No discernable reaction	No discernable reaction
nitric acid and methyl ethyl ketone	No discernable reaction	No discernable reaction
nitric acid and acetic anhydride	No discernable reaction	No discernable reaction
ammonium hydroxide and hydrazine	Fuming by hydrazine	No discernable reaction
ammonium hydroxide and cumene hydroperoxide	No discernable reaction	No discernable reaction
ammonium hydroxide and methyl ethyl ketone	No discernable reaction	No discernable reaction
ammonium hydroxide and acetic anhydride	No discernable reaction	Fumes produced, no temperature increase
hydrazine and cumene hydroperoxide	Fuming by hydrazine	No discernable reaction
hydrazine and methyl ethyl ketone	Fuming by hydrazine	No discernable reaction
hydrazine and acetic anhydride	Fuming by hydrazine	Fumes produced, temperature increased 26.1 °C (79 °F)
cumene hydroperoxide and methyl ethyl ketone	No discernable reaction	No discernable reaction
cumene hydroperoxide and acetic anhydride	No discernable reaction	No discernable reaction
methyl ethyl ketone and acetic anhydride	No discernable reaction	No discernable reaction

CONCLUSIONS

MSE performed bench-scale, solidification experiments on 10 categories of neat chemicals, each representing a different chemical hazard type that could be found in the unidentified liquids from the Rocky Flats facility that are presently stored at the INL. The hazard types tested were water reactive chemicals, oxidizing agents, reducing agents, acids, bases, flammable chemicals, peroxidizable chemicals, chlorinated solvents, sulfide salts solutions, and cyanide salt solutions. In addition, solidification experiments were conducted on nine categories of rinsate formulas.

The field-scale solidified materials will be required to remain stable under conditions that may be encountered during solidification, storage, shipping, and burial. Therefore, the bench-scale sorbent/ surrogate combinations were required to remain stable during three forms of testing that included stability testing, reactivity testing, and proximity reactivity testing. The stability testing incorporated curing behavior, sorption capacity, sorption rate, and presence of free liquids in the final wasteform as determined by the use of PFT and LRT methods. The reactivity testing included flammability tests; ignitability tests; pH screening; screening for oxidizing agents; determination of reducing agents; water reactive chemical testing; testing for the presence of organic peroxides; and determining the concentration of sulfide and cyanide in oxidized and unoxidized samples. Proximity and contact reactivity testing was also conducted with the solidified wasteforms.

The results of the stability testing showed that all of the solidified neat chemical and rinsate solutions wasteforms passed the PFT and the LRT WAC of 1% release by volume at the ratio of 3:1 except the neutralized acid rinsate sample and the sodium perchlorate rinsate solution. These two rinsate solutions were solidified using a 4:1 ratio of sorbent to surrogate, and both rinsates passed the PFT and the LRT WAC at that ratio.

The results of the chemical reactivity testing can be explained by taking each test sequence separately. The results of the flammability and ignitability testing showed that the two hydrazine-based samples would not ignite and therefore were not deemed to be flammable and were determined to be noncombustible. The two methyl ethyl ketone-based samples were deemed to be flammable and ignitable with combustion propagation rates of approximately 40 mm (1.575 inches) per second.

(Note: Propagation of combustion in SW-846, Method 1030, does not mean the solid waste meets the definition of an RCRA characteristic ignitable waste as described in 40 CFR 261.21 (a)(2) [6].)

The pH was taken for the neat chemicals and rinsate solutions prior to solidification with the selected sorbent blend. The selected sorbent blend had a significant buffering effect on the neat chemicals and rinsate liquids; in a number of cases, the pH of the solidified mixture was buffered to a value between 7 and 10 or the pH was buffered to value not in the corrosive range. To that end, the pH of the solidified surrogate/sorbent mixture was used to define a form of the reactivity for the solidified samples. In a number of cases, the reactivity of the samples was reduced or limited by the buffering action of the solidification material. A compilation of the reactivity results for all chemical and rinsate categories is shown in Tables IX and X.

Of the solidified chemicals tested for the presence of oxidizing agent chemical reactivity, only the samples of solidified 11N nitric acid indicated the presence of potentially hazardous oxidizing compounds. None of the solidified chemicals that were tested for the presence of reducing agents indicated the existence of those materials after solidification.

Water reactive chemical testing was conducted on two samples of solidified acetic anhydride. This testing did not indicate the presence of any water reactivity in either of those solidified samples.

Table IX. Reactivity results and pH buffering effects for the surrogate rinsate/sorbent samples.

Rinsate Name and Description	Weight-Based, Waste-Loading Ratio (sorbent to liquid)	pH Buffering	Limits the Reactivity for Chemical Category
Inorganic Acid			
Dilute nitric acid 1:999 with DI water	2:1	Yes	Yes
Dilute nitric acid 1:999 with DI water	3:1	Yes	Yes
Organic Acid			
Acetic acid, 1:1 with DI water	3:1	Out of corrosive range	No
Acetic acid, 1:1 with DI water	4:1	Out of corrosive range	No
Acetic acid, 6:94 with DI water	2:1	Out of corrosive range	No
Acetic acid, 6:94 with DI water	3:1	Out of corrosive range	No
Inorganic Base			
Ammonium hydroxide, 1:999 with DI water	2:1	Yes	Yes
Ammonium hydroxide, 1:999 with DI water	3:1	Yes	Yes
Organic Base			
Hydrazine, 6:94 with DI water	3:1	Yes	Yes
Hydrazine, 6:94 with DI water	4:1	Yes	Yes
Oxidizing Agent			
Sodium perchlorate solution - 0.1 M	2:1	Yes	Yes
Sodium perchlorate solution - 0.1 M	3:1	Yes	Yes
Water-Miscible Compounds			
Isopropyl alcohol, 1:1 with DI water	3:1	No	NA
Isopropyl alcohol, 1:1 with DI water	4:1	No	NA
Isopropyl alcohol, 6:94 with DI water	2:1	No	NA
Isopropyl alcohol, 6:94 with DI water	3:1	No	NA
Water-Immiscible Compounds			
Chloroform, di-chloromethane, PCE, TCE, and 1,1,1-TCA solubility limits in DI water	2:1	Yes	NA
Chloroform, di-chloromethane, PCE, TCE, and 1,1,1-TCA solubility limits in DI water	3:1	Yes	NA
Above diluted 12.5:87.5 with DI water	2:1	Yes	NA
Above diluted 12.5:87.5 with DI water	3:1	Yes	NA
Neutralized Acids			
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	2:1	No	No Effect
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	3:1	No	No Effect
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	4:1	No	No Effect
950 mg/L NO_3^-, 35,000 mg/L Cl^-, and 12.5 mg/L F^-, pH 8.5 in DI water	5:1	No	No Effect
KW Decontamination Solutions			
3.17 g/L Versene 100, 3.38 g/L anhydrous citric acid; and 2.80 g/L Igepal CA-630	2:1	Yes	NA
3.17 g/L Versene 100, 3.38 g/L anhydrous citric acid; and 2.80 g/L Igepal CA-630	3:1	Yes	NA
Above diluted 1:99 with DI water	2:1	Yes	NA
Above diluted 1:99 with DI water	3:1	Yes	NA
NA = not applicable since no reactivity testing was conducted			

Table X. Reactivity results and pH buffering effects for the neat chemical/sorbent samples.

Neat Chemical Name and Description	**Weight-Based, Waste-Loading Ratio (sorbent to liquid)**	**pH Buffering**	**Limits the Reactivity for Chemical Category**
Flammables			
Methyl ethyl ketone	2:1	Yes	No
Methyl ethyl ketone	3:1	Yes	No
Hydrazine	3:1	Yes	Yes
Hydrazine	4:1	Yes	Yes
Acids			
Nitric acid – 11N	2:1	No	No
Nitric acid – 11N	3:1	No	No
Nitric acid – 1N	2:1	Out of corrosive range	No
Nitric acid – 1N	3:1	Out of corrosive range	No
Bases			
Ammonium hydroxide – 11N	2:1	Out of corrosive range	No
Ammonium hydroxide – 11N	3:1	Out of corrosive range	No
Ammonium hydroxide – 5N	2:1	Out of corrosive range	No
Ammonium hydroxide – 5N	3:1	Out of corrosive range	No
Oxidizing Agents			
Nitric acid – 11N	2:1	No	No
Nitric acid – 11N	3:1	No	No
Nitric acid – 1N	2:1	Out of corrosive range	Yes
Nitric acid – 1N	3:1	Out of corrosive range	Yes
Sodium perchlorate solution – 0.1N	2:1	Yes	Yes
Sodium perchlorate solution – 0.1N	3:1	Yes	Yes
Sodium perchlorate solution – 0.1N	4:1	Yes	Yes
Sodium perchlorate solution – 0.1N	5:1	Yes	Yes
Reducing Agents			
Hydrazine	3:1	Yes	Yes
Hydrazine	4:1	Yes	Yes
Water Reactive			
Acetic anhydride	3:1	No	Yes
Acetic anhydride	4:1	No	Yes
Peroxidizables			
1,4-dioxane	2:1	Yes	Yes
1,4-dioxane	3:1	Yes	Yes
Cumene hydroperoxide	3:1	Yes	No
Cumene hydroperoxide	4:1	Yes	No
Chlorinated Solvents			
TCE	2:1	Yes	NA
TCE	3:1	Yes	NA
1,1,1-TCA	2:1	Yes	NA
1,1,1-TCA	3:1	Yes	NA
PCE	2:1	Yes	NA
PCE	3:1	Yes	NA
TCE, PCE, and 1,1,1-TCA (1:1:1)	2:1	Yes	NA
TCE, PCE, and 1,1,1-TCA (1:1:1)	3:1	Yes	NA
Sulfide Salts			
Sodium sulfide solution	2:1	Out of corrosive range	Yes
Sodium sulfide solution	3:1	Out of corrosive range	Yes
Sodium sulfide solution – oxidized	2:1	Out of corrosive range	Yes
Sodium sulfide solution – oxidized	3:1	Out of corrosive range	Yes

Neat Chemical Name and Description	Weight-Based, Waste-Loading Ratio (sorbent to liquid)	pH Buffering	Limits the Reactivity for Chemical Category
Cyanide Salts			
Sodium cyanide solution	2:1	Out of corrosive range	No
Sodium cyanide solution	3:1	Out of corrosive range	No
Sodium cyanide solution – oxidized	2:1	Out of corrosive range	Yes
Sodium cyanide solution – oxidized	3:1	Out of corrosive range	Yes
NA = not applicable since no reactivity testing was performed			

MSE used test strips to determine the amount of organic peroxide present in the solidified samples of 1,4-dioxane and cumene hydroperoxide. Only one of the tests conducted on the samples of solidified 1,4-dioxane indicated the presence of any organic peroxide; a value of 1-mg/L peroxide was detected in that sample. Organic peroxide values as high as 40 mg/L were detected in the solidified samples of cumene hydroperoxide. However, these high values decreased during the curing period to values of 10 to 15 mg/L.

Four samples were tested to determine the concentration of sulfide in the solidified materials. Of these samples, two contained oxidized sulfide, and two contained unoxidized sulfide. The two unoxidized sulfide samples contained concentrations of 29.1 and 28.2 mg/kg of sulfide in the 2:1 and 3:1 waste-loaded samples, respectively, compared to the initial concentrations of 257 mg/kg and 193 mg/kg. The two oxidized sulfide samples contained concentrations of 13.8 and 3.5 mg/kg of sulfide in the 2:1 and 3:1 waste-loaded samples, respectively. It should be noted that the sulfide concentrations for all four of the solidified samples were lower than the sulfide destruction limits of less than 56 mg/kg stated in the project test plan.

Three sets of four solidified, cyanide-bearing samples were tested to determine the concentration of cyanide in the solidified materials. For each of these sample sets, two samples were unoxidized, and two were oxidized. The results of testing of the first set of samples showed that the two unoxidized, solidified cyanide samples contained concentrations of cyanide that were below the detection limits of the analytical technique while the two oxidized, solidified cyanide samples contained concentrations of 120 and 180 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively. The results of testing of the second set of samples showed that the two unoxidized, solidified cyanide samples contained concentrations of cyanide that were 179 and 132 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively, while the two oxidized, solidified cyanide samples contained concentrations of 0.276 and 0.202 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively. The results of testing of the third set of samples showed that the two unoxidized, solidified cyanide samples contained concentrations of cyanide that were 270 and 177 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively, while the two oxidized, solidified cyanide samples contained concentrations of 4.39 and 0.369 mg/kg for the 2:1 and 3:1 waste-loaded samples, respectively. Again, it should be noted that both of the cyanide concentrations for the oxidized, solidified samples were lower than the cyanide destruction limits of less than 22 mg/kg stated in the project test plan.

The results of the proximity reactivity testing showed that four combinations of solidified chemicals exhibited reaction processes when placed in near proximity to each other. These pairs of chemicals were nitric acid/ammonium hydroxide; nitric acid/hydrazine; ammonium hydroxide/acetic anhydride; and hydrazine/acetic anhydride.

RECOMMENDATIONS

The first option for final disposal is the broad-spectrum approach in which all of the unidentified Rocky Flats laboratory waste is treated by solidifying with the selected sorbent blend proven to control the liquid release aspects of the solidified wasteforms, limit the reactivity of some of the wasteforms, and provide some pH buffering effects. If this option is taken, MSE recommended that each solidified laboratory wasteform be containerized separately before disposal to eliminate any potential reactivity issues that may occur in the disposal pit.

The second option for final disposal calls for more upfront chemical analysis with commercially available techniques for the individual laboratory waste bottles followed by individual treatment schemes using appropriate sorbents based on the chemical characteristics of the waste. Disposal of the similar solidified wasteforms could then be done in the same container for burial. If this option is selected, MSE recommended additional testing to prove the compatibility of the solidified wasteforms.

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