THE NOCHAR PETROBOND® ABSORBENT POLYMER TRITIATED OIL SOLIDIFICATION DEMONSTRATION AT MOUND

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ABSTRACT

The DOE Office of Science and Technology, Decontamination and Decommissioning Focus Area is sponsoring the Large Scale Technology Demonstration and Deployment Project (LSDDP) at the DOE Mound Site near Dayton, Ohio. The goals of the LSDDP are to:

- Demonstrate existing developed technologies, that are unproven and/or unknown with respect to Tritium Decontamination and Decommissioning (D&D) applications, and validate that the technologies are superior (cost effective, safer and more ALARA) to currently used "baseline" technologies, and
- Communicate the demonstration results in such a manner that potential end users of the demonstrated technology can easily decide to adopt the demonstrated technology as one of their "baseline" tools for performing future D&D work.

Presently, the DOE Mound Site provides an ideal opportunity for the (LSDDP) since its closure requires the D&D of numerous tritium-contaminated facilities. Significant inventories of tritiated oil, which are the result of the operation of hundreds of tritium handling glove boxes and associated vacuum pumps, exist here and present a major challenge in meeting the Miamisburg Environmental Management Project (MEMP) goals which include strict adherence to ALARA principles while handling this hazardous tritiated waste. These vacuum and vane pumps, which were critical to the handling of tritium gas at MEMP (also known as the Mound DOE facility), required the use of oil in their operation. While in use the oil became contaminated with tritium. The tritium, which tends to replace the hydrogen in the oil hydrocarbons, is not easily separated. Hazardous chemicals and metals were introduced to the oil through normal operations and include lead, chromium, barium, mercury as well as other metals. Chemicals such as cyclohexane were added to the oil during the pump cleaning process to inhibit coagulation caused by tritium break down in the oil bonds. Under Resource Conservation and Recovery Act (RCRA) regulations the oil becomes mixed waste and is therefore handled as such for burial site disposal.

Three possibilities; long-term storage, incineration, and solidification using current technologies have been considered for mixed waste oil disposition in the baseline project. Even though incineration has been used in the past, none of these technologies were found to be viable at this time. The innovative technology that was demonstrated in the LSDDP is a polymer solidifying agent offered by the NOCHAR® Corporation of Indianapolis, IN. Using experience gained in major commercial oil spill operations, NOCHAR® has designed a product, Petrobond®, which

absorbs oil quickly upon contact and significantly reduces RCRA metal characteristics while aiding in ALARA concerns. One deployment of Nochar® polymers to solidify low level tritiated vacuum pump oil has been performed at the Mound Site. The Toxicity Characteristics Leaching Procedure (TCLP) analysis of oil solidified with NOCHAR® at Mound verified that the final waste form meets the LDRs for toxic metals given in 40CFR-268.40 and the Petrobond® polymer crystals are non-toxic, non-biodegradable and incinerable to less than 0.02% ash. The use of Nochar is simple. Waste oil is mixed with Petrobond® simply by pumping the oil into a drum containing the polymer. The oil is absorbed without any mechanical mixing. Superior ALARA benefits are achieved through the elimination of mixing and the capability of remote use, thus minimizing personnel contact with the oil itself or with tritium offgas. Deployments of Nochar® are now being planned at Mound to absorb inventories of high activity tritiated oil directly inside the gloveboxes where they were created. This will eliminate the need to transfer the oil to remote mixing stations, and therefore will provide additional ALARA benefits by reducing the amount of handling.

This paper provides details of the testing and initial deployment of the polymer. It describes the selection process of the polymer “formula” that was used at Mound and the TCLP results of the final waste product. Additionally, information will be presented that compares the performance, cost and ALARA capabilities of this technology to the baseline approach. Recommendations will be presented concerning the deployment of this technology at MEMP and other applicable sites.

The data obtained to-date from this technology in the Mound LSDDP shows promise in providing ALARA and burial solutions for this complex waste handling issue at the MEMP, the DOE complex, and the entire Nuclear Industry.

INTRODUCTION

A Large Scale Technology Demonstration and Deployment Project (LSDDP) has been initiated at the DOE Mound Site near Dayton, Ohio sponsored by the DOE Office of Science and Technology, Deactivation and Decommissioning Focus Area. This phase of the LSDDP involved the development and application selection of a Nochar Petrobond® solidification agent formula that will effectively solidify, significant inventories of tritiated mixed waste production oils for shipment to Nevada Test Site (NTS) under the NTS Waste Acceptance Criteria (WAC).

The Mound Site is a former component of the U.S. Department of Energy Nuclear Weapons Complex used for research and development, and production.

The tritium D&D operations in the T Building and SW/R Building complex are now the critical path for this D&D closure project. Any innovative technologies that can be inserted into a baseline toolbox is not only a prudent idea, but something that will help enable the project cost and schedule to be met.

LSDDP AT MOUND

As one of four major technology development focus areas in the DOE Office of Science and Technology (EM-50), the Deactivation and Decommissioning Focus Area (DDFA) is responsible for developing, demonstrating, and implementing cost-effective and safe technologies to deactivate and/or decommission buildings on DOE's list of surplus facilities. In order to fairly evaluate the cost and performance of new technologies, the DDFA has embarked upon a strategy, the Large Scale Demonstration and Deployment Project program, to sponsor these first time, full scale demonstrations within the DOE complex. The intent of the LSDDPs are to demonstrate potential advantages of the innovative D&D technologies. An Integrating Contractor Team (ICT) manages each LSDDP, oversees the demonstration, and evaluates the performance of both the innovative and baseline technologies. Results of the innovative and commercial technologies demonstrated in the LSDDP, will be published as Innovative Technology Summary Reports (ITSRs) and are available from the Federal Energy Technology Center (FETC) in Morgantown, West Virginia.

At Mound, the objective of the LSDDP is to identify, demonstrate, evaluate, and if successful, deploy improved technologies that are applicable to the deactivation and decommissioning of the Mound Tritium Facilities or Complex. D&D of Mound's surplus tritium facilities, the T and R/SW Buildings, provide the opportunity to compare, evaluate, and eventually deploy improved D&D technologies alongside baseline technologies in an ongoing site D&D project. The Mound LSDDP will identify and explore methods to improve worker safety while achieving cost and schedule savings. The project is expected to identify technologies that, when deployed in the Mound Tritium D&D project, will produce significant savings on the $128 million baseline. The results and successes of this demonstration project it is hoped, will benefit similar DOE facilities and projects.

The Mound LSDDP IC Team includes technical experts from the following organizations: B&W of Ohio, British Nuclear Fuels, Ltd., Foster Wheeler Environmental, Inc., IT Corporation, Westinghouse Savannah River Company, Florida International University, Los Alamos National Laboratory, Lawrence Livermore National Laboratory and Princeton Plasma Physics Laboratory (PPPL). It is anticipated that innovative technologies will be applied to the following decontamination tasks at the Mound Tritium Complex:

- Tritium contaminated glove boxes
- Tritium characterization techniques
- Productivity improvement technologies
- Tritium specialties decontamination
- Piping system removal and disposition

- Tritiated water treatment and disposal
- Miscellaneous radioactive and non-radioactive traditional building materials disposition, and
- Mixed waste treatment and disposal, which is primarily based on the vacuum pump oil that is being addressed by solidification with Nochar Petrobond®.

PROJECT DESCRIPTION

As described, the LSDDP project at Mound is a diverse ongoing venture that has led up to solidification of tritiated oil. The Phase I (non-tritiated oil phase) results, that have been obtained using RCRA heavy metal contaminated vacuum pump oils that have not been introduced to a radiological environment, will be discussed first.

Phase I: RCRA Oil Bench Tests

The primary purpose of phase I, was to:

- Establish TCLP factors of the solidified mass as examined by a certified laboratory
- Develop a specific formula of the Nochar® agent that will address the idiosyncrasies of waste oil generated at Mound.
- Perform “durability” tests on solidified samples. Verify that the solidified waste can meet Waste Acceptance Criteria of the chosen disposal site, in this case Nevada Test Site (NTS)
- Function as a dry run regarding the generation of solidified waste in a non-radioactive contaminated lab before the actual tritiated material is solidified, and
- “Clearing the way” by addressing a significant number of site regulatory and procedural issues for large scale tritiated oil solidification.

After technical review of the Nochar products and evaluation of the Mound waste oil, detailed planning was conducted before entering into the first full bench test. The selected formulas and mixtures are summarized in a chart as given in Table -1. Dealing with this waste oil presented many significant challenges that would not normally be encountered during standard application of this product. Some of them included:

- The possibility that 5% or greater water content could be in the oil, due to condensation after decades of operation.
- The potential for unique and non mineral oils mixed together in the waste reservoirs.
- Inability to bring in large mixing machines or perform hand mixing during the solidification operations, as is needed with the other industry accepted baseline products.
- The strict requirements for working in a nuclear facility, and
- Strict limitations on working with any material that has potential to produce fire hazard concerns.

Bench test work was conducted on bench tests in the R-166 Building Laboratory (non-radiological). All work was performed under the guidelines of a Health and Safety Plan (HASP) that has been established for excess chemical disposition at Mound. The permitting approval for this test or experimentation was granted through the Ohio EPA under hazardous waste regulation 3745-51-04 sections (F) & (9). Permission was granted for 30 gallons for each tritium level, that being low / medium / high, and 30 gallons of non-detectable pump oil.
Note: Activity levels established for low = <.10 Ci/L, medium = >.10 Ci/L but <10 Ci/L, high = > 10 Ci/L.

Bench Test Number One

Work began in the lab as outlined on the Bench Test Data Sheet provided in Table-1 of this report. To compensate for water content, the Nochar® A660 product was added at a specific ratio (total solidification agent volume by weight) on samples where oil was "spiked" with demineralized water to simulate water contaminated oil we may find in the tritium complex. The A660 product was developed for water absorption as well as solidifying acid mixtures. The primary products for solidifying oil are the A650 and A610 Petrobond® products. A650 is typically used on land based oil spills and has been known to have the ability to extract crude oil out of the top layer of soil and produce a rubbery compound in place. Limitations we found with A650 used here at Mound, is that it is highly dependent on a volatile material to prime or activate the polymers and solidify them, therefore, with the weight and texture of table salt it is better suited for land and/or water based oil spills which it was designed for. A high amount of volatility is actually found in crude oil and this need for a primer is a common requirement that is found in oil solidification products. A610 is a more "snow flake" type polymer product which will allow the priming or catalyst material to be "preloaded" at the factory due to its porous or lighter consistency. Because of this, the A610 Petrobond® can "pick up" or absorb up to 15 lbs. of liquid per pound of agent as compared with A650 which can absorb only 10 lbs. of liquid per pound of agent (Figure 1 illustrates the product in natural form). The vacuum pump mineral oils provide very little volatility, and in this situation, a volatile additive must be included in the mixture. Finding this additive was not an easy task as most commercial mineral spirit products have extensive amounts of impurities such as benzene and tolulene. This was unacceptable and would compound our problem of trying to dispose of mixed hazardous waste at a burial site. Through extensive research, a "clean" commercially sold mineral spirit product was found that is manufactured by Exxon Corporation. This product contains only trace amounts of hazardous chemicals as identified. As an added bonus, we found that the product had low odor and fewer flammability concerns.

Figure 1 – Nochar Petrobond® Crystals, Unabsorbed

Note: Table #1 below:

1. Pass/fail evaluations were performed by putting absorbed material in a paper filter and performing the standard paint filter test
2. Ratios of oil to Nochar in the mixture were calculated by weight.

Table I - Bench Scale Test #1, Data Form

Smpl.#	Container Size	Solidif. Agen	Oil Type	Container Weight (g)	Amount of Nochar Required (g)	Wt. Of container +	Oil Initial Volume (ml)	Nochar Initial Vol. (ml)	Wt. Of Oil (g)	Actual Amount of Nochar added (g)	Weight of Solid Mass (g)	Start Time	Stop Time	Final Volume (ml)	Ratio Used - Oil to Nocha	Pass/Fai	Commnts
1	125ml	A 650	DuoSea	11	43	15	50	10	43	43	8	092	[1]	12	1/1	P	Oil only
2	125ml	A65	DuoSea	11	7	11	50	2	43	7	5	093	[1]	60	6/1	F	Oil only
3	125ml	A65	Duo/Ca	111.2	43	154.2	50	10	43	43	8	111	[1]	12	1/1	P	Oil & Cat.
4	125ml	A65	Duo/Ca	11	7	11	50	1	4	7	5	111	[1]	60	6/1	F	Oil & Cat.
5	250ml	A61	Inland	18	42	22	50	18	4	42	8	094	[1]	25	1/1	P	Oil only
6	125ml	A 610	Inland	11	7	11	50	20	4	7	4	094	[1]	75	6/1	P	Oil only
7	250ml	A660/A61	Duo/Wat	181.5	7	188.	50	15	4	7	5	103	[1]	60	6/1	F	[2]
8	125ml	A660/A65	Du/W/C	111.2	7	118.2	50	15	43	7	5	103	[1]	70	6/1	F	[3
9	125ml	A660/A61	Duo/Wat	111.2	7	118.2	50	15	4	7	5	104	[1]	75	6/1	P	[4]
10	125m	A660/A65	Du/W/	111.2	7	118.2	50	15	43	7	5	105	[1]	70	6/1	P	[5]
1	250m	A	Inld/Cat	183.	4	226.5	50	18	4	4	86	112	[1]	240	1/1	P	10% Cat.
12	125m	A	Inld/Cat	11	7	11	50	20	4	7	4	123	[1]	75	6/1	P	10% Cat.
13	250m	A	Inld/Cat	18	4	22	50	18	4	4	8	124	[1]	240	1/1	P	20% Cat.
14	125m	A	Inld/Cat	11	7	11	50	2	4	7	49	125	[1]	6	6/1	P	20% Cat.
15	125m	A	Duo/Ca	11	4	15	50	10	4	4	8	130	[1]	125	1/1	P	30% Cat.
16	125m	A	Duo/Ca	11	7	11	50	1	4	7	5	131	[1]	60	6/1	F	30% Cat.
17	125m	A	Duo/Ca	11	4	15	50	10	4	4	8	131	[1]	125	1/1	P	50% Cat.
1	125m	A	Duo/Ca	11	7	11	50	1	4	7	50	132	[1]	6	6/1	P	50% Cat.
19	125m	A	Ultima	11	4	15	50	10	4	48	9	133	[1]	125	1/1	F	Ultima
20	250m	A	Ultima	18	4	23	50	19	4	48	9	133	[1]	23	1/	P	Ultima
21	250m	A	DuoSea	181	4	22	50	170	4	4	8	134	[1]	22	1/	P	10%Pre
22	250m	A	DuoSea	18	7	19	50	2	4	7	5	134	[1]	75	6/	P	10%Pre
23	250m	A	DuoSea	18	4	22	50	17	4	4	8	135	[1]	23	1/	P	20%Pre
24	250m	A	DuoSea	18	7	18	50	1	4	7	5	135	[1]	60	6/	F	20%Pre
25	250m	A	DuoSea	18	4	22	50	17	4	4	8	140	[1]	24	1/	P	30%Pre
26	125m	A	DuoSea	11	7	11	50	1	4	7	5	140	[1]	65	6/	F	30%Pre
27	250m	A	Inlan	18	4	22	50	8	4	4	8	141	[1]	15	1/	P	10%Pre
28	125m	A	Inlan	111	7	11	50	1	4	7	5	142	[1]	60	6/	F	10%Pre
29	125m	N	Inlan	11	N	N	5	N	42	N	N	N	N	N	N	N	N
30	125m	N	DuoSea	11	N	N	5	N	43	N	N	N	N	N	N	N	N

[1] - Stop time and examination time conducted 4 days after sample start time .
[2]- Oil + 10% water mixture , water/oil formula used 1 part A660 to 4 parts A610.
[3]- Oil + 10% water + 10% catalyst, water / oil formula used 1 part A660 to 4 parts A650.
[4]- Oil + 20% water, water/ oil formula used 1 part A660 to 4 parts A610.
[5]- Oil + 20% water + 10% catalyst. Water/ oil formula use 1 part A660 to 4 parts A650
* Final volumes as reported are only rough estimations.
** Pass / Fail evaluations are based only on visual observations and how well oil appeared to be absorbed.
*** Note: DuoSeal oil has a density of 43 (g) / 50 ml, Inland 19 Oil has a density of 42(g) / 50 ml.
C or Cat. = catalyst, PreL= Nochar product pre-loaded with catalyst (paint thinner) W or Wa = Water, Duo = Duoseal oil, Inld= Inland 19 Oil

Even with the implied excellent fire control properties of Nochar, it was determined that the use of a flammable liquid material would be greatly limited by site fire protection restrictions. Fire / explosion concerns are high safety priority items at Mound with 3000+ flammable liquids present at any one time and past production concerns with hydrogen gas. For this reason we turned to experimentation with a “pre-loading” of the catalyst or mineral spirit into the product and thus allowing proper ratio of Nochar to be staged in the solidification container without additional mixing or combining of ingredients in a hazardous environment. Therefore, upon adding the proper weight ratio of oil to the container, solidification would quickly begin and be completed in less than 30 minutes without any mixing by hand or large equipment, a requirement that the selected solidification agent must meet at Mound. Pre-loading of the catalyst was performed by hand in small quantities during the bench test here on Site. Too much of the mineral spirit causes the product to plasticize and invalidates its absorption capabilities, but up to that point, as more mineral spirits are added, a better / firmer consistency of solidified product is realized. Optimizing the primer, in turn, produces a better TCLP factor and a more conservative waste material mixture to assure meeting burial site WAC. [As given in Table –2, acceptable results were obtained, especially with the catalyst loaded absorbent]. Through experimentation at the Nochar production facility, a maximum mixture of 33% (weight ratio) mineral spirit catalyst was obtained. It was decided that would produce optimum absorption capabilities. The Nochar Corporation will be supplying pre-loaded material at the optimum amount established for the demonstration and future use.

As documented on the bench test data sheet (Table –1), the samples were produced with a 1 to 1 or 6 to 1 ratio (oil to Nochar). Time and materials available allowed for only a specific number of samples and the consensus of most cognizant site tritium waste personnel was that a conservative 1 to 1 ratio would provide a more realistic waste package to meet NTS criteria. PPPL had some success with up to 6 to 1 ratio regarding the physical solidified results, and therefore, we also experimented with this ratio. As illustrated in Table-1, the chosen formula using A610 product, A660 for water uptake, and preloaded low odor mineral spirits at the factory, was selected for the “custom” formula that would be used to dispose of Mound vacuum pump mineral oil in the Demo. This formula was developed strictly by bench tests performed under the Mound LSDDP. The sample photo (Figure –2) also illustrates stratification where a layer of non-absorbed Petrobond® is left at the bottom of the container thus providing a safety margin for absorbing oil in the unlikely event that is it released from the Nochar molecular matrix during transport or other external physical events.

Bench tests were also conducted with scintillation cocktail solvents used at Mound such as Ultimagold®. The results given for Ultimagold® in samples 19 & 20 on the test data sheet (Table –1) were quite successful. In each case the waste could be highly solidified without even adding catalyst due to the volatility of these waste products. This also allowed for use of the A650 agent which in turn produces a more rubbery solidified compound.

TABLE – II

Quanterra Environmental Services – St. Louis Laboratory

Bench Test #1 – TCLP Results

Client: B&W of Ohio, Inc.
One Mound Rd, Attn:Dr. Eugene Jendrek
Miamisburg, OH 45343
Project: DOE Mound

Sample Date: 03/11/99
Receipt Date: 03/17/99
Report Date: 04/01/99
Quanterra Project No.:145.04

Category: TCLP Metals

Matrix: Solid

Quanterr ID: 20899-001
Client ID: OO1

Analyte	Method:	Extraction Date:	Prep Date:	Analyses Date:	Result:	Units:	Detection Limit:	Reg. Limit:	Dilution:
Mercury	EPA7470	3/22/99	3/25/99	3/25/99	0.19	MG/L	0.0080	0.20	40
Arsenic	EPA6010	3/22/99	3/27/99	3/28/99	0.010	MG/L	0.040	5.0	4
Barium	EPA6010	3/22/99	3/27/99	3/28/99	0.24	MG/L	0.80	100.0	4
Cadimum	EPA6010	3/22/99	3/27/99	3/28/99	0.0042	MG/L	0.020	1.0	4
Chromium	EPA6010	3/22/99	3/27/99	3/28/99	0.085	MG/L	0.040	5.0	4
Copper	EPA6010	3/22/99	3/27/99	3/28/99	0.12	MG/L	0.10		4
Lead	EPA6010	3/22/99	3/27/99	3/28/99	0.14	MG/L	0.012	5.0	4
Selenium	EPA6010	3/22/99	3/27/99	3/28/99	0.013	MG/L	0.020	1.0	4
Silver	EPA6010	3/22/99	3/27/99	3/28/99	0.010	MG/L	0.040	5.0	4
Zinc	EPA6010	3/22/99	3/27/99	3/29/99	0.16	MG/L	0.080		4

Quanterr ID: 20899-017
Client ID: O25

Analyte	Method:	Extraction Date:	Prep Date:	Analyses Date:	Result:	Units:	Detection Limit:	Reg. Limit:	Dilution:
Mercury	EPA7470	3/22/99	3/25/99	3/25/99	0.040	MG/L	0.0080	0.20	4
Arsenic	EPA6010	3/22/99	3/27/99	3/29/99	0.010	MG/L	0.040	5.0	4
Barium	EPA6010	3/22/99	3/27/99	3/29/99	0.061	MG/L	0.80	100.0	4
Cadimum	EPA6010	3/22/99	3/27/99	3/29/99	0.0057	MG/L	0.020	1.0	4
Chromium	EPA6010	3/22/99	3/27/99	3/29/99	0.0072	MG/L	0.040	5.0	4
Copper	EPA6010	3/22/99	3/27/99	3/29/99	0.014	MG/L	0.10		4
Lead	EPA6010	3/22/99	3/27/99	3/29/99	0.052	MG/L	0.012	5.0	4
Selenium	EPA6010	3/22/99	3/27/99	3/29/99	0.012	MG/L	0.020	1.0	4
Silver	EPA6010	3/22/99	3/27/99	3/29/99	0.010	MG/L	0.040	5.0	4
Zinc	EPA6010	3/22/99	3/27/99	3/29/99	0.19	MG/L	0.080		

Quanterr ID: 20899-012 Matrix: Oil (raw, non-solidified)
Client ID: O30

Analyte	Method:	Extraction Date:	Prep Date:	Analyses Date:	Result:	Units:	Detection Limit:	Reg. Limit:	Dilution:
Mercury	EPA7470	3/18/99	3/30/99	3/30/99	7.60	MG/L	2.0	0.20	10000
Arsenic	EPA6010	3/18/99	3/27/99	3/29/99	0.25	MG/L	1.0	5.0	100
Barium	EPA6010	3/18/99	3/27/99	3/29/99	0.12	MG/L	20.0	100.0	100
Cadimum	EPA6010	3/18/99	3/27/99	3/29/99	0.16	MG/L	0.50	1.0	100
Chromium	EPA6010	3/18/99	3/27/99	3/29/99	0.18	MG/L	1.0	5.0	100
Copper	EPA6010	3/18/99	3/27/99	3/29/99	0.83	MG/L	2.50		100
Lead	EPA6010	3/18/99	3/27/99	3/29/99	3.60	MG/L	0.30	5.0	100
Selenium	EPA6010	3/18/99	3/27/99	3/29/99	0.29	MG/L	0.50	1.0	100
Silver	EPA6010	3/18/99	3/27/99	3/29/99	0.25	MG/L	1.0	5.0	100
Zinc	EPA6010	3/18/99	3/27/99	3/29/99	3.90	MG/L	2.0		100

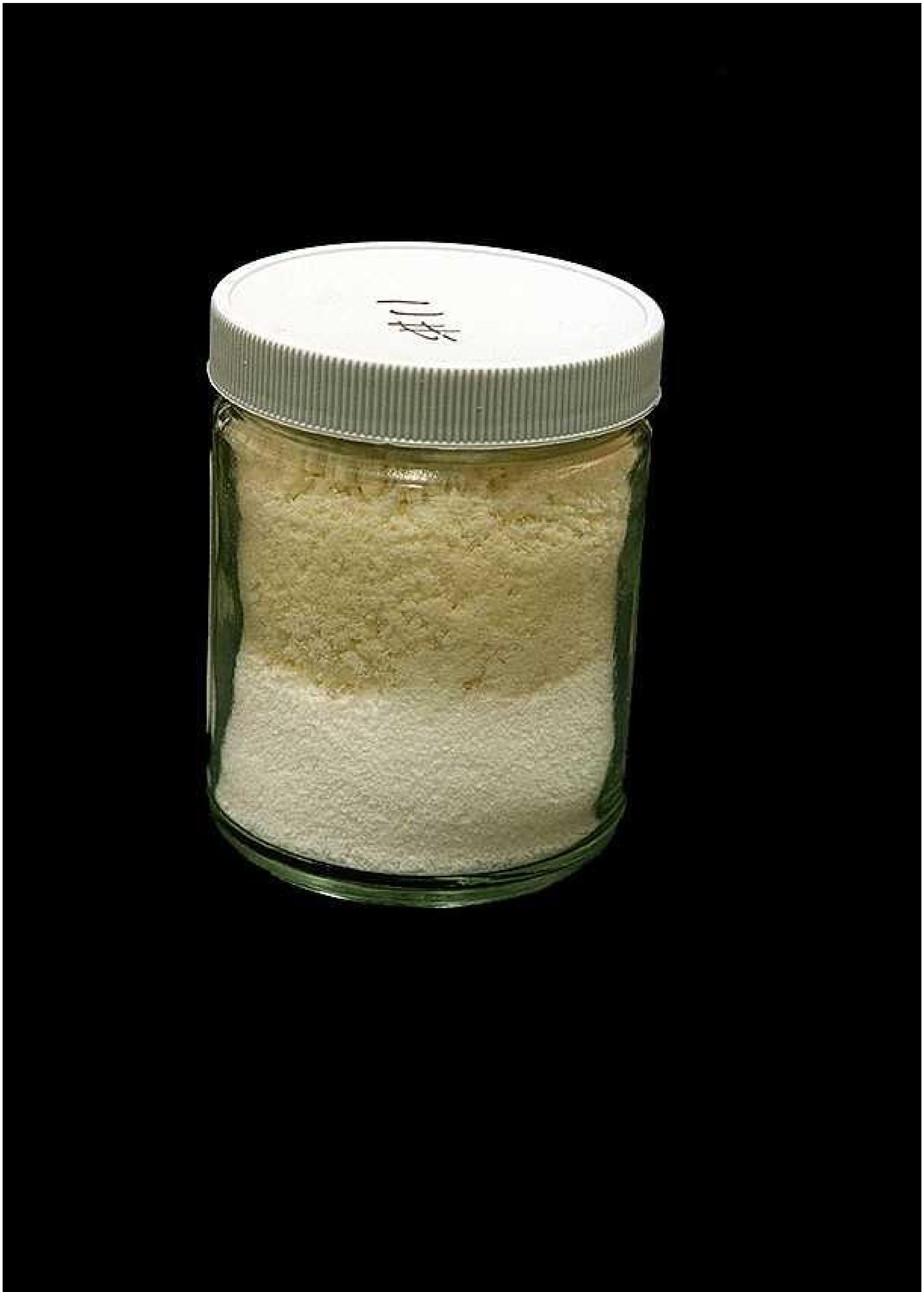

Figure 2: Solidified RCRA Oil Sample with Stratification

Bench Test Number Two

The second bench test with non-tritiated RCRA oil was conducted with the Mound formula that was developed from the first bench test and included ingredients for water uptake and catalyst or primer involvement. The following Tables – 3, 4 illustrate the improved results realized with the introduction of this “custom” solidification formula.

Table IV – Second Bench Test TCLP Lab Test

Sample Id	Mercury	Arsenic	Barium	Cadmium	Chromium	Copper	Lead	Selenium	Silver	Zinc	Dilution	Flash Point
Reg Limit mg/L	0.2	5.0	100	1.0	5.0		5.0	1.0	5.0			
Inst Det Limit	0.0001	0.0018	0.0042	0.0002	0.0016	0.0010	0.0012	0.0024	0.0009	0.0005		
B2 SAMPLE 01	0.0058	<0.0072	0.0200	0.0030	<0.0064	0.0420	0.0160	0.0130	0.0110	0.16	4X	>60 C
B2 SAMPLE 02												>60 C
B2 SAMPLE 03												>60 C
B2 SAMPLE 04	0.0063	<0.0072	0.0500	0.0017	<0.0064	0.0260	0.0210	0.0096	0.0077	0.19	4X	>60 C
B2 SAMPLE 05												>60 C
B2 SAMPLE 06	3.40	<0.18	<0.42	0.07	<0.16	1.00	1.20	<0.24	<0.09	3.00	100X	
	Sample 06 was diluted X1,000 for Hg											

TABLE –- III
MOUND BENCH TEST #2 DATA SHEET

Smpl.#	Container Size	Solidif. Agent	Oil Type	Container Weight (g)	Amount of Nochar Required (g)	Wt. Of container + Nochar	Oil Initial Volume (ml)	Nochar Initial Vol. (ml)	Wt. Of Oil (g)	Actual Amount of Nochar added (g)	Weight of Solid Mass (g)	Start Time	Stop Time	Final Volume (ml)	Ratio Used - Oil to Nochar	Pass/Fail	Commnts
	5/12/99																
1	125ml	Formula	Duo/Wat	121	24	145	NA	100	24	24	48	1445	1630	105	1/1	P	
2	125ml	Formula	Duo/Wat	120	17	137	NA	60	34	17	51	1452	1630	105	2/1	P	
3	125ml	Formula	Duo/Wat	121	18	139	NA	60	54	18	72	1501	1630	105	3/1	P	
4	250ml	610-V	Duo/Wat	193	44	237	NA	190	44	44	88	1522	1630	240	1/1	P	
5	250ml	610-V	Duo/Wat	193	36	229	NA	130	72	36	108	1536	1630	240	2/1	P	
6	125	NA	Duoseal	NA	NA	NA	125	NA	NA	NA	NA	NA	NA	NA	NA	NA	Baseline Oil Smpl
	5/19/99																
7	50ml	Formula	Synth	87	14	101	NA	50	28	14	42	1331	1700	50	2/1	P	
8	50ml	610-V	Synth	87	13	100	NA	50	13	13	26	1345	1700	50	1/1	P	
9	50ml	650-V	Synth	86	22	108	NA	50	22	22	44	1402	1700	50	1/1	P	
10																	
11																	

V= virgin product
Duo= Duoseal Oil
Synth.=Synthetic Oil
Wa=water

Phase II: Clean Oil Solidification In A DOT Liner / Quality Checks

The second phase of the demonstration involved the documentation of the performance of the Nochar® absorbed waste form in large quantities and in a certified waste shipment container. This operation was conducted with new (non-contaminated) vacuum pump oil with work being conducted in a "clean" area that did not require extensive controls. Observations were also made regarding the amount of expansion that took place with a container this size. At the conclusion of the solidification, four (4) ¼ inch diameter holes were drilled equal distance around the base of the drum and visual checks for moisture were made. A section of pipe was inserted down the middle of the solidified mixture with the upper end sealed to form a vacuum. Contents of the (core sample) were taken from the pipe and examined for liquid or non-absorbed oil. In each test, any absorbent that was released from the drum was subjected to the EPA paint filter test and the results documented. The final segment of the quality test and inspection involved observation of the overall solidification form. The liner was a translucent poly material and thus allowed the coloration of the oil and activated Nochar to be observed through the container walls and top bunghole cover. Thorough inspection of the process was made as allowed by the properties of the liner.

Phase III: Tritiated Oil Solidification

The third and last phase of the demonstration was performed to collect data from radiologically contaminated RCRA waste oil. The preceding phases could in some respects be viewed as dress rehearsals for the Phase III critical path tritiated oil evolution. This was a non-bench test, full-scale tritiated oil solidification process. Since this type of operation required work in a radiological area with significant safety controls and protocols, all reference to steps in the solidification process were made through procedures as identified in this section, including specific procedures written for this demo. Conduct of operations for this type of solidification task warranted and required extensive safety controls as referenced in the Site procedures and the HASP for the selected work areas.

In addition to the Demo procedures as referenced, the basic steps of the process included:

- 22.5 gallon poly liner and 30 gal. drum overpack inspection
- adding predetermined ratio of pre-mixed Nochar® to liner by weight
- moving liner into ventilated hood
- measuring out predetermined ratio of waste oil by weight that will be combined with Nochar
- slowly adding waste oil to the drum liner while observing and recording data
- verification that all ingredients have been properly combined, remove drum from hood and set aside to allow absorbent to "cure" for 15 minutes
- adding a shallow layer of additional Nochar® absorbent to the top of the drum liner at the end of solidification
- sealing drum, survey, weighing, mark and label drum in preparation for shipment or final disposition as specified by waste management
- notifying all cognizant department / individuals when Demo operations have been completed, initiate demobilization operations

All of these steps were carefully monitored and documented by the test engineer/data collector. The Table –5 data sheet documents all waste forms and ratios that were used.

Table V – Tritiated Oil Solidification In Liners, Data Sheet

Smpl.#	Container	Solidif.	Oil	Container Weight	Amount of Nochar Require	Wt. Of container + Nochar	Oil Initial Volume	Nochar Initial Vol	Wt. Of Oil	Actual Amount of Nochar adde	Weight of Solid Mass	Start Tim	Stop Tim	Final Volume	Ratio Used - Oil to Nocha	Pass/Fai	Commnts
	8-29-99 Bldg #23 Waste Facilit																
1	22.5 Gal	formula	1, 2, 3	25.5	40	65.5	3 gal.	15.5	23 lb.	40 lb.	63	133	144	20.4 gal	.6/1.0	P	.32
2	22.5 Gal	formula	1, 2, 3	25.5	40	65.5	3 gal.	15.5	23 lb.	40	63	134	151	20.4 gal	.6/1.0	P	.32
3	22.5 Gal	formula	1, 2, 3	25.5	40	65.5	3 gal.	15.5	23 lb.	40	63	140	160	20.4 gal	.6/1.0	P	.31
4																	
5																	
6	(1)= 3 gal. of Vacuum pump mineral oils (2)= 3 gal. of glycol waste lubricants (3)= 3 gal. Of polyphenyl ether Note: 1 gal. Of ea. Oil waste was used per liner																

Figure 3: Solidified Oil in Burial Liner – Top view

TCLP RESULTS

Paramount to meeting burial site WAC are the TCLP tests that a solidification agent will be subjected to. As given in Table-2, excellent reduction factors (reduced count of metal content in MG/L of oil) were realized, especially when the mineral spirit catalyst is added to the Nochar® product (as it was in sample #025). It should be mentioned that use of virgin material without the catalyst also met TCLP factor limits, but did not provide the reliability margin considered acceptable and preferred by the Mound Waste Management Department. Oils with high levels of mercury and lead were found and selected for the bench test. This oil provided most of the metallic contaminants that would exhibit RCRA concerns and worked well for establishing TCLP results. When using the recently established Land Disposal Restrictions (LDRs) for mercury, a clear pattern of comparison could be seen as illustrated in Table-2. Using the LDR limit of 0.2 Mg/L for mercury, sample #1 which contained no catalyst, passed by a narrow margin with a result of 0.19 Mg/L. Sample #25 which is indicative of the chosen solidification formula produced a result of 0.040 Mg/L, a factor of five less than sample #1 and well below limits. This is all based on the raw non-solidified oil sample, # 30 which showed a high mercury level of 7.6 Mg/L.

With the introduction of the Mound Formula and a pre-load with the Exxon mineral spirit catalyst/primer, TCLP factors improved significantly as seen in TCLP Data Table No. 4. The final results as seen in TCLP Table No. 6 with H3 oil were viewed as a great success; thus providing the greatest results yet as evidenced in the data for mercury or the most restrictive RCRA metals constituent.

TABLE VI: PHASE III, SOLIDIFIED TRITIATED RCRA OIL TCLP ANALYSIS – QUANTERRA LABS

Sample ID	Lab ID	Date Sampled	Prep Date	Analysis Date	Phs	Analye	Result	Units	Reg Limit mg/L	Fg	Detection Limits	Dilution	Blank	Method	SW-486
NC830	22033-001	8/30/99	9/9/99	9/9/99	Solid	Merc	.00092	MG/L	0.2		.0008	4	QCBLK206777-1	TCLP Metals	EPA 7470
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Arsenic	ND	MG/L	5	U	1.2	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Barium	ND	MG/L	100	U	0.8	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Cadmi	0.014	MG/L	1	B	0.02	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Chromi	0.0047	MG/L	5	B	0.04	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Copper	0.12	MG/L			0.1	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Lead	0.29	MG/L	5	B	0.4	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Seleni	ND	MG/L	1	U	1	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Silver	ND	MG/L	5	U	0.04	4	QCBLK206552-1	TCLP Metals	EPA 6010
NC830	22033-001	8/30/99	9/8/99	9/8/99	Solid	Zinc	0.071	MG/L		B	0.08	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/9/99	9/9/99	Solid	Merc	ND	MG/L	0.2	U	.0008	4	QCBLK206777-1	TCLP Metals	EPA 7470
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Arsenic	ND	MG/L	5	U	1.2	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Barium	ND	MG/L	100	U	0.8	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Cadmi	ND	MG/L	1	U	0.02	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Chromi	ND	MG/L	5	U	0.04	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Copper	ND	MG/L		U	0.1	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Lead	ND	MG/L	5	U	0.4	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Seleni	ND	MG/L	1	U	1	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Silver	ND	MG/L	5	U	0.04	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	EXTBLK206439-1	9/7/99	9/8/99	9/8/99	Solid	Zinc	0.012	MG/L		B	0.08	4	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Arsenic	102	%REC	5			1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Barium	102	%REC	100			1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Cadmi	100	%REC	1			1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Chromi	98	%REC	5			1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Copper	99	%REC				1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Lead	96	%REC	5			1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Seleni	99	%REC	1			1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Silver	99	%REC	5			1	QCBLK206552-1	TCLP Metals	EPA 6010
NA	QCLCS206552-1	9/8/99	9/8/99	9/8/99	Solid	Zinc	97	%REC				1	QCBLK206552-1	TCLP Metals	EPA 6010

CONCLUSIONS

In every mixture or test performed, the Nochar® product reacted quickly with the oil, or oil/water mixture. In most cases it had completely solidified in less than 30 minutes. As stated on the data sheet, samples were examined thoroughly four days after solidification took place. It is reasonable to say that during solidification operations, at least 4 days would transpire due to packaging and/or transportation to a burial site. Examination of the samples over this time span indicated a "curing" process took place and most samples appeared more dense and solidified than when observed at time of mixing, thus producing a better more solidified product at the shipping stage.

Nochar® is a very easy product to work with. Handling and measuring were easily accomplished. The material is a light non-hazardous material that can readily be dispensed and combined with oils. Due to the likely scenario of requiring all Nochar to be pre-measured so that oil can be added directly in the solidification container, all oil was added to selected pre-staged and measured Nochar® formulas. Once again, this facilitated ease of use and demonstrated the product will perform well in the selected Mound hazardous environment.

Different combinations and formulas of Nochar product and catalyst were experimented with. Experiments were also performed using various water content in some oils with the expectation that some condensation water will exist in tritiated vacuum pump oil at Mound that has been used over several decades. Expansion of material was minimal and in some cases showed a maximum of 10% as given in Table-1. In other instances, due to density of some of the solidification agents and the use of a catalyst, total volume actually declined slightly.

Due to considerable flammability concerns throughout Mound, the idea of pre-loading several Nochar products with a selected and approved catalyst was chosen over handling liquid bulk mineral spirits in the facility. The Nochar factory produced the pre-loaded product, while for test results here, the catalyst was added manually, in small quantities, with good success. The Nochar products are "primed" by the introduction of a volatile petrochemical such as paint thinner or mineral spirits where the end result is a solidified mass analogous to a piece of dense foam rubber. A threshold for this activation was found at between 30-50% (oil to mineral spirit weight ratio) catalyst added to the agent.

Further examination of the end product, which is a spongy foam rubber material in some instances, holds promise in that Nochar® in its solidified form, may have an ability to provide some attenuation of tritium when work does begin with the large amount of tritiated oils.

The innovative technology proved in the demonstration to be highly effective in handling and solving the mixed waste issue. It was found that it:

- Has a single step process – does not ever require mixing
- Minimized processing times by reducing handling, with minimal set up times
- Reduces worker exposure (ALARA)
- Increases productivity & improved project schedule
- Provides and overall cost savings for treatment and disposal of tritiated oil
- Can be developed into specific "custom" formulas for any situation

- Can be loaded in specific disposal/shipping containers right at the factory
- Requires virtually no processing equipment
- Is non toxic, non-biodegradable and incinerable to less than 0.02% ash
- Absorbs quickly with minimal increase in sorbate volume
- Is a free flowing easy to work with material that presents no safety hazards or issues except respiratory protection from dust when used in large quantities

The product is highly dependent though, on a volatile ingredient to act as a catalyst or primer in the activation of the polymer and its solidification process on the oil. This requirement along with the combination of ingredients that may be required for solidification of specific oil and water combinations, along with other additives or substances that can be found in waste oils, undoubtedly requires technical expertise from company representatives on the use and deployment of this product. Another issue of importance that would require this expertise is the speed at which Nochar will solidify some materials. If this is too fast, it can have the ability to 'crust over' and not allow the non-solidified liquid to filter down into the agent and complete the solidification process. This once again requires analysis and recommendations from a technical specialist to identify the proper formula or combination of agents needed based on the characterization and analysis of the waste oil to be solidified.

The basic cost of the Nochar solidification agent is approximately $18.00 per pound or $740.00 per 40 lb. drum, exclusive of any shipping or handling cost to transport. It is very likely that in a large oil solidification operation that this price would be further reduced to offer a volume discount price or agreement. Alternative oil handling methods, such as long term storage and incineration, would involve substantial costs as was revealed during analyses performed on "Baseline Technologies". Considerable savings can also be realized in many other cost related areas due to the fact that the Nochar product begins solidifying on the spot and does not require mixing or any involved use of processing equipment. This will provide cost savings in the areas of: total manhours required, ALARA and required personnel exposure to perform the work, improved productivity and ease of deployment and recovery.

After reviewing the results with the Site Chemical Analyst, we felt confident these results will meet our needs for substantiating our compliance with Land Disposal Regulatory Limits and providing a viable TCLP result along with meeting packaging requirements to comply with burial site High Moisture Content Waste (HMCW) criteria. Further review of this data by technical experts will take place as Mound continues into the tritiated oil solidification and disposal evolutions of this large D&D Project.

REFERENCES

1) PPPL Test Report "Performance of Oil Solidification Agents" (draft)
2) EPA Test Method 9095 Paint Filter Test
3) DOE Order 5820.2A Radioactive Waste Management
4) Waste Acceptance Criteria for the (NTS) Nevada Test Site
5) MD-10019 Radiological Control Manual
6) MD-10286 Mound Safety and Hygiene Manual
7) MD-10361 Mound Conduct of Operations
8) MD-70743 Waste Sampling Procedures

9) MD-70180 Mound Radioactive Liquid Waste Disposal
10) All applicable Site JSHAs
11) ML-287 Waste Disposal Daily Log
12) ML-7042X Low Level Radioactive waste Input Form
13) 29 CFR 1910: Occupational Safety and Health Administration – Department of Labor
14) DOE Order 5400.5: Radiation Protection of the Public and the Environment
15) DOE Order 414.1 Quality Assurance
16) PP-1007: Tritium Facility Conduct of Operations
17) PP-9326: Radiation Safety
18) Site (HASP) Health and Safety Plan, for Excess Chemical Disposition, Rev. 02
19) (OEPA) Ohio Environmental Protection Agency, Sections: 3745-51-04 (E), (F), "Treatability Studies"
20) Technical Manual, NOCHAR Petrobond® absorbent polymer, Nochar Corp.–Indianapolis Indiana
21) 10 CFR 835: Occupational Radiation Protection
22) MD – 80043:Radioactive Material Transfer and Unrestricted Release of Property/ Waste.
23) MSDSs for all materials being worked with in the Demo
24) SPA930055: Low Level radioactive waste prohibited materials list.
25) MD – 21358:Tritiated Liquid Waste Packaging Procedure
26) MD – 10463:Waste Management Quality Plan