DEMONSTRATION SOLIDIFICATION TESTS CONDUCTED ON RADIOACTIVELY CONTAMINATED ORGANIC LIQUIDS AT THE AECL WHITESHELL LABORATORIES

Marvin A. Ryz, Atomic Energy of Canada Limited, MB

Ward G. Brunkow, Richard Govers, The Chamberlain Group, Ltd., Lynchburg, VA

Dennis Campbell, Nochar Inc., Indiannapolis, IN

Donald Krause, BWXT Services, Inc., OH

ABSTRACT

The AECL, Whiteshell Laboratory (WL) near Pinawa Manitoba, Canada, was established in the early 1960's to carry out AECL research and development activities for higher temperature versions of the CANDU® reactor. The initial focus of the research program was the Whiteshell Reactor-1 (WR-1) Organic Cooled Reactor (OCR) that began operation in 1965. The OCR program was discontinued in the early 1970's in favour of the successful heavy-water-cooled CANDU system. WR-1 continued to operate until 1985 in support of AECL nuclear research programs.

A consequence of the Federal government's recent program review process was AECL's business decision to discontinue research programs and operations at the Whiteshell Laboratories and to consolidate its' activities at the Chalk River Laboratories. As a result, AECL received government concurrence in 1998 to proceed to plan actions to achieve closure of WL. The planning actions now in progress address the need to safely and effectively transition the WL site from an operational state, in support of AECL's business, to a shutdown and decommissioned state that meets the regulatory requirements for a licensed nuclear site. The decommissioning program that will be required at WL is unique within AECL and Canada since it will need to address the entire research site rather than individual facilities declared redundant.

Accordingly, the site nuclear facilities are being systematically placed in a safe shutdown state and planning for the decommissioning work to place the facilities in a secure monitoring and surveillance state is in progress. One aspect of the shutdown activities is to deal with the legacy of radioactively contaminated organic liquid wastes. Use of a polymer powder to solidify these organic wastes was identified as one possibility for improved interim storage of this material pending final disposition.

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INTRODUCTION

Stored at the WL Waste Management Area (WMA), are ~12,000 liters of radioactively contaminated organic liquid resulting from operational activities conducted at WL. The majority of this material is made up of used WR-1 reactor coolant (HB-40). Lesser amounts of xylene rinse solution, dielectric (EDM) fluid, vacuum pump oil and water are also being stored. The radioactivity of this liquid is above the allowable activity limit (15 Bq/ml) for incineration in the WL organic incinerator. Gamma activities on randomly selected samples of HB-40 and EDM fluid are shown in Table I.

Table I. Typical Gamma Activity on Organic Samples in Bq/ml

Sample I.D.	Nuclides	Activity
HB-40 (1)	C3-134 Cs-137	1.0E+01 3.4E+03
HB-40 (2)	Co-60 Cs-134 Cs-137	1.9E+00 9.6E+00 3.7E+03
HB-40 (3)	Co-60 Cs-134 Cs-137	1.6E+00 9.5E+00 3.1E+03
EDM-1	Co-60 Cs-134 Cs-137	1.4E+00 8.5E+00 3.0E+03
EDM-2	Co-60 Nb-94 Cs-134 Cs-137 Eu-154 Am-241	1.6E+00 4.0E-01 9.5E+00 3.3E+03 1.9E+00 5.0E+00

The drums storing this material are showing the effects of long-term storage through repeated freeze thaw cycles that are brought on by the local climate.

Repeated attempts to remove the activity from the organic by filtration proved to be only partially successful. Filtration was not seen as an effective means of dealing with this waste problem and continued storage, as a solid, was seen as the best interim option.

Demonstration solidification tests were conducted on bench-test samples of these liquids in an attempt to identify an improved storage method for this radioactive waste, pending final disposition. The solidification process used was to mix the organic with a specialty powder called Nochar PetroBond® supplied by Nochar Inc. Experts from Nochar Inc. and The Chamberlain Group Ltd., contracted by the U.S. Department of Energy (DOE), supervised the

work that was conducted in the WL Shielded Facilities (SF). Through sponsorship by the U.S. DOE Office of Science and Technology via the Transuranic and Mixed Waste, and the Decommissioning Focus Area, Nochar PetroBond® has been successfully used in the U.S. to solidify radioactive contaminated organic liquids. The final product has received Nevada Test Site approval for land disposal(1).

Nochar PetroBond® is a high-technology polymer solidification agent that can be designed to address the specific characteristics of a variety of organic liquids(2). It bonds the organic liquid into a soft, sponge rubber-like material. It is non-toxic, non-hazardous, non-corrosive and non-biodegradable. It produces no heat during the solidification process. It is incinerable to less than 0.02 % ash therefore allowing for possible final incineration at a later date. It has an absorbent capacity of up to 15:1 (ratio of oil to agent by weight) and up to 85:1 (ratio of water to agent) with minimal volume increase. It has been tested to 40 MRad gamma with limited evidence of radiological degradation. This product was seen as a possible solution to the continued storage requirements of the radioactively contaminated organic liquid at the WL WMA.

DEMONSTRATION TESTS

Approximately 20 liters of organic liquid was removed from six randomly selected drums for the demonstration solidification tests. A sample from each drum was analyzed for water and xylene content. The results of this analysis are shown in Table II.

Table II. Water and Xylene Analysis of Randomly Selected Samples

Sample I.D.	Drum Description	Water Content (% by Volume)	Density (g/ml)	Xylene Content (% by Volume)
#1	Volatiles	0.5-2	1.0055	21
#2	Organic	0.01	1.0021	23
#3	Organic, Water, Sludge	99.9	NA	NA
#4	Xylene Wash	0.02	1.0016	23
#5	Organic	0.05	1.0143	17

Twenty-eight samples, ranging from 300 ml to 20 liters, were prepared for the tests. Three specific Nochar PetroBond® polymers were used for the tests. Various weight ratios from 1/1 to 10/1 (liquid to polymer) were tested. To an empty 1000 ml glass beaker, a predetermined quantity of solidification agent was added by weight. Next, the various ratios (by weight) of organic were added to the beaker of solidification agent as shown in Figure 1.

Fig. 1: Photograph Showing the Addition of Organic to Polymer

The beakers were set aside in a fume hood, as shown in Figure 2, for 24 hours to allow the solidification process to be completed. Stirring was not required, however stirring greatly speeds up the solidification process and can also help eliminate the formation of a "skin" or barrier that can form during the stabilization process.

Fig. 2: Photograph of Test Samples During Solidification

After the 24-hour solidification period, the solidified mass was tested for free liquid. This is a simple test method to determine compliance with 40 CFR 264.314 and 265.314 for unabsorbed free liquids(3). The solidified mass was placed in a #60 mesh paint filter and allowed to sit for five minutes as shown in Figure 3. If no free liquid was observed passing through the paint filter, then the material was considered to have passed the test. At WL these paint filter tests were extended to 24 hours to further demonstrate the effectiveness of this process. Of all the tests conducted at WL, only two failed the free liquid test. These were due to a higher concentration of water in the organic in these samples than in the samples that passed the test. This was easily rectified by adding a small portion of water solidification agent to the organic solidification agent before the liquid was added to the polymer.

Fig. 3: Photograph of the Solidified Samples Being Tested for Free Liquid

Effective organic to polymer ratios varied for each liquid source, however the optimum ratio for HB-40 (the majority of the waste organic liquid in storage) was approximately 4 to 1, oil to polymer. Table III lists the results of the solidification tests.

Table III. Results of Solidification Tests

Sample #	Oil Type	Solidification Agent	Ratio* Oil/NOCHAR	Paint Filter Test		Comment
				Pass	Fail	
1	1	A-610	.6/1	√		
2	1	A-610	2/1	√		
3	1	A-610	3/1	√		
4	1	A-610	5/1		√	Higher Water Content
5	1	N-990	2/1	√		
6	1	A-610	1/1	√		
7	1	A-610/660	2/1	√		
8	2	A-610	2/1	√		
9	2	A-610	1.2/1	√		
10	2	A-610	5/1	√		
11	3	A-660	5/1	√		Aqueous
12	4	A-610	2/1	√		
13	5	A-610	1/1	√		
14	5	A-610	2/1		√	Higher Water Content
15	5	A-610	4/1	√		
16	5	N-990	2/1	√		
17	EDM	A-610	1/1	√		
18	EDM	A-610	2/1	√		
19	EDM	N-990	1/1	√		
20	EDM/Hyd.	A-610	1/1 2/1	√		

*Ratios By Weight

Following these small-scale solidification tests, larger (20 litre) volumes of HB-40 and dielectric fluid were solidified using these pre-determined ratios. These larger volumes solidified readily as well, however the process was greatly accelerated by stirring the mass as the organic was added. Without stirring, a skin tends to form at the organic/polymer interface. This creates a barrier that significantly delays the solidification process.

CONCLUSIONS AND FUTURE WORK

The WL demonstration tests were very successful. Samples of all organic liquid waste streams were readily solidified, and the resultant solidified product passed the required filter paper test

for free liquid. This method appears to be an efficient, safe, cost-effective way to treat the WL radioactively contaminated organic liquids for continued interim storage.

AECL is now seeking regulatory approval to deal with the remaining organic liquid wastes on a larger scale. Planning is underway to solidify these wastes in B-25 containers, as shown in Figure 4. To prevent the formation of a skin that can form as a barrier during the stabilization process and delay the completion of the solidification process, a stirring mechanism will be incorporated into the B-25 containers. When the B-25 containers are filled with solidified organic waste they will be placed into interim storage in existing above ground low-level bunkers at the WL WMA.

Fig. 4: Photograph of a Standard B-25 Storage Container

REFERENCES

1. U.S. Department of Energy, Nevada Operations Office, "Approval to Ship Mound Plant Low-Level Radioactive Waste to the Nevada Test Site".

2. NOCHAR Inc. Indianapolis, Indiana, Product Literature: "NOCHAR PetroBond® Absorbent Polymer Oil Solidification Agent".

3. Paint Filter Liquids Test, Method 9095.