

Decontamination Methods Testing for the Waste Isolation Pilot Plant - 15691

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ABSTRACT

On February 14, 2014, americium and plutonium contamination was released in the Waste Isolation Pilot Plant (WIPP) salt caverns. Several practical, easily deployable methods of decontaminating WIPP salt, using a surrogate contaminant and americium (^{241}Am), were developed and tested. The effectiveness of the methods is evaluated qualitatively, and to the extent practical, quantitatively. Of the methods tested (dry brushing, vacuum cleaning, water washing, strippable coatings, and mechanical grinding), the most practical seems to be water washing. Effectiveness is very high, and water washing is easy and rapid to deploy. The amount of wastewater produced (2 L/m^2) would be substantial and may not be easy to manage, but the method is the clear winner from a usability perspective. Removable surface contamination levels (smear results) from water washed coupons found no residual removable contamination. Thus, whatever contamination is left is likely adhered to (or trapped within) the salt. The other option that shows promise is the use of a fixative barrier. Bartlett Nuclear, Inc.'s Polymeric Barrier System proved the most durable of the coatings tested. The coatings were not tested for contaminant entrapment, only for coating integrity and durability.

INTRODUCTION

On February 14, 2014, a release of contamination occurred within the Department of Energy's (DOE) Waste Isolation Pilot Plant (WIPP) underground transuranic (TRU) waste repository near Carlsbad, New Mexico. The WIPP is a deep geologic repository carved out of a salt bed. Rooms interconnected by drifts (i.e., corridors) are mined out of the salt. Containerized TRU waste is stored in the rooms. It has been determined that one or more of the waste containers breached and released americium and plutonium, contaminating the mine, the ventilation system, and 21 site personnel.[1]

The operating contractor, Nuclear Waste Partnership, LLC, contracted the Idaho National Laboratory (INL) to determine the relative effectiveness of various methods of decontamination for the WIPP mine. The decontamination of salt surfaces had not been well described in the literature. New processes, described in this report, have been designed and tested using actual WIPP salt coupons, as well as other materials, and both surrogate contaminants and americium (^{241}Am) contamination. The INL has extensive experience in simulating different kinds of contamination, including that from nuclear fallout and dirty bombs.[2-6]

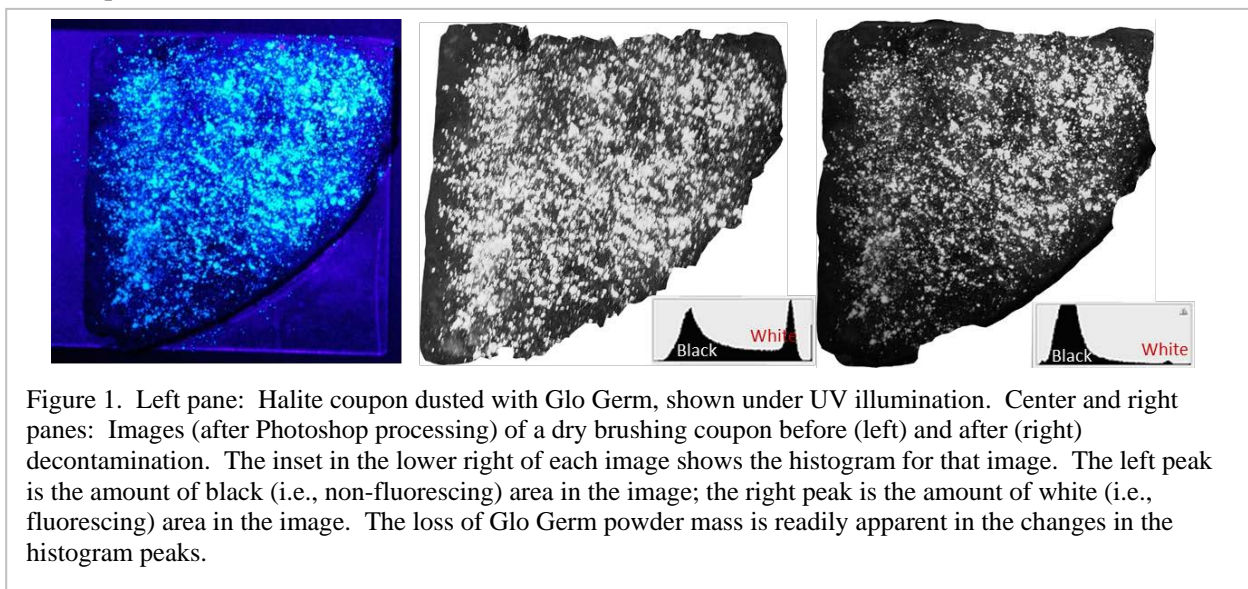
A good case can be made that the contamination in the WIPP mine is loosely attached to surfaces. Fixed, tenacious contamination usually arises from species that are liquid and corrosive in nature, penetrating the surface of a material. Materials that are dry (dusty) are generally less tenacious. For non-radioactive tests, an insoluble powder called Glo Germ was chosen to model this loose contamination. Glo Germ is visible when irradiated with ultraviolet (UV) light. The brightness of the surface can be quantified and counted. A tracer solution containing americium (^{241}Am) is used in the radioactive tests. The slightly acidic tracer solution penetrates the salt surface more than a loose particulate contaminant would, but it provided the best way to apply a homogeneous layer of contamination on the samples.

To simulate the surface conditions in the WIPP mine drifts, solid chunks of rock salt (halite) were used as an analog for the wall and ceiling surfaces in the mine. The halite was cut from a WIPP mine core taken before the contamination event. Loose salt rubble removed during mining activities at WIPP was employed to simulate the floor surfaces in the mine. The rubble was made into a salt rubble bed ~4 cm thick. Solid samples measured ~10 cm square. Rubble samples measured ~30 cm square. The rock salt core was cut into squares approximately 10 cm on a side. However, the core was cylindrical, so there were a number of irregularly shaped pieces leftover from around the edges after the 10 cm × 10 cm coupons were cut out. Those irregular scraps were used for surrogate contamination tests. The square cut coupons were used for the radioactive tests. It was decided that the best data (in terms of consistency between coupons) can be gathered with relatively smooth coupons, despite the fact that the actual salt rock is quite irregular. Thus all solid coupons were cut ~3 cm thick.

The coupons, contaminated with either Glo Germ or americium, were subjected to a number of different decontamination methods: brushing, vacuuming, mechanical grinding, water washing, and strippable coatings. The use of fixative barriers to immobilize contamination was also investigated. Of primary importance was determining how effective each of these methods is at removing (or fixing) contamination from (or to) a halite surface. More qualitative aspects of the methods were also evaluated: ease of use, potential for contaminant re-suspension, volume and type of secondary waste, and (relative) rate of application/removal.

LOOSE SURROGATE CONTAMINANT REMOVAL TECHNIQUES

Dry brushing, vacuuming, water washing, strippable coatings – and later, mechanical grinding – were tested as decontamination methods on solid WIPP halite coupons. UV sensitive Glo Germ powder was selected as the surrogate contaminant because it is safe, non-toxic, and easy to detect. Three coupons were used for each test. The coupons used in these tests are the irregular scraps, approximately 100 cm². Nonetheless, because every coupon is analyzed individually, the data stays consistent and reproducible to that coupon.



Semi-quantitative decontamination results were obtained by the following method: One hundred milligrams of Glo Germ powder is applied to each coupon. The 100 mg is distributed on the surface

using a small bottle with six holes in the top (similar to a salt shaker). Using a manually adjusted digital camera, pictures are taken before and after decontamination. The images are processed through Adobe Photoshop to determine the amount of white light, thus the quantity of Glo Germ powder. The coupon shown in the left pane of Figure 1 is illuminated under ultraviolet (UV) light. A purple/blue (long wave UV) haze can be seen. The bright spots are the Glo Germ surrogate contaminant. The coupon area in the image is selected (cut), changed to a black and white image (Figure 1), then the area of the white peak of the histogram (insets in Figure 1) is quantified. This method does not give high precision results; however, the amount of light is proportional to the mass of fluorescing powder. Therefore, the histogram discriminates between large piles and small smudges of powder. The collection of results from the five tests (excluding grinding) performed on these WIPP salt coupons is shown in Table I.

Table I. Relative Decontamination Levels for Surrogate Contaminant Tests

Method	% Effectiveness	Std. Dev.
Dry Brush	21.7	12.2
Water Wash	98.0	2.0
DeconGel Strippable Coating	91.3	6.0
Vacuuming	23.5	7.8
Stripcoat Strippable Coating	25.0	8.9

As can be seen in Table I, two of the methods, water washing and DeconGel 1108, were highly successful at decontaminating the powder from the salt surface. In the case of water washing, the 98% reported is not an absolute value. There was obviously some residual material, apparent in Figure 2; however, the residual is so slight that it became only noise in the histogram. Based on removal efficacy, both water washing and DeconGel are candidates for use on the salt. However, removing the DeconGel strippable coating took significant time compared with the other methods, rendering it quite inefficient. In the case of the Stripcoat material, removal of the coating from the ~100 cm² coupon took over 15 minutes – extremely long for a strippable coating. The salt seems to interact with the coatings adhering them to the surface. Based on these results, brushing, vacuuming, and Stripcoat have minimal effectiveness.

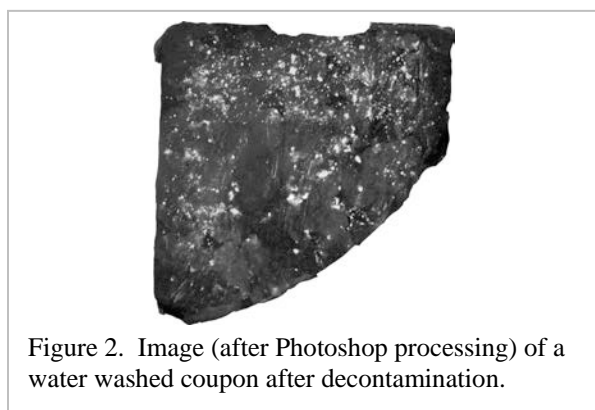


Figure 2. Image (after Photoshop processing) of a water washed coupon after decontamination.

Complementing the evaluations previously done of dry brushing, water washing, et al., surface grinding was tested. Three coupons were each dusted with 100 mg of Glo Germ fluorescent powder in the same manner as the previous removal tests. The sample surfaces were then ground down with a Makita side arm grinder at 1200 rpm with a wire cutter head and a vacuum dust collection system. The dust collection cowl was connected to a Minuteman HEPA vacuum.

Grinding efficiently removes the surrogate contaminant, as well as a layer of the salt surface; however, some contaminant is retained in the pores and grain boundaries of the sample. Unfortunately, as can be seen in Figure 3, the removed contaminant and salt is widely dispersed in the working area, despite the

grinder head employing a dust collection system. While some contaminant is captured in the vacuum during removal, this method would disperse contamination that is settled and adhered to a surface. Under UV illumination, significant contamination could be seen on the operator and on the surrounding surfaces. (See Figure 3.) It seems that this method would make the situation worse, not better, especially in an actively ventilated mine drift. Based on these results, it was not considered worthwhile to quantify the removal efficiency using the white light histogram method.



Water Washing Considerations

The water washed Glo Germ coupons were submitted to microscopic evaluation to determine why contaminant removal was not complete (though photo-processing shows that it is substantially complete). Figure 4 shows side-by-side comparisons of two microscopic photographs taken at a site that brightly fluoresces white/green. The purple/blue in the photo on the left is given by the UV light source. The photo on the right shows the same site under visible (i.e., white) light, showing the natural look of the salt surface. In the right hand photo, it is clear that the source of fluorescence is a contaminant particle in an inclusion within the salt. These tiny surface irregularities, visible under magnification, can act as contaminant traps, and are equally important to proper decontamination as the more obvious cracks, crevices, and general surface irregularities visible to the naked eye.

If water washing were pursued as a decontamination method, there are some considerations that should be understood. With the application of water or a significant increase in humidity, the salt may become “sticky” on the surface. As an example, vacuum cleaning was

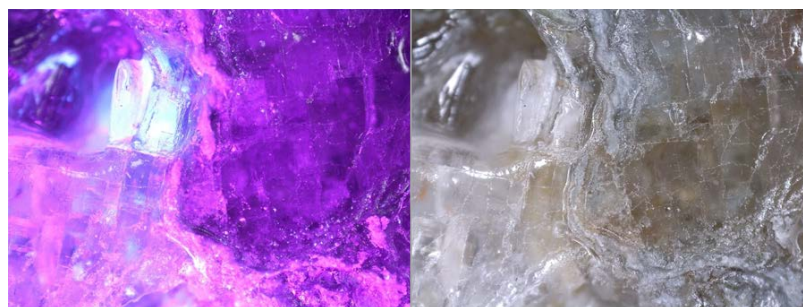


Figure 4. Contaminant inclusion in a halite sample, shown under UV (left) and visible (right) illumination.

predicted to be a more effective decontamination method than it proved to be in testing. It is thought that higher than typical ambient humidity (due to rain that week) may have caused the powder to be more adherent to the surface. This could become a factor as large amounts of water are applied in the mine drifts: As the humidity rises, the surface may hold contaminants more tenaciously. A second consideration may be that contaminants successfully removed from the wall during wash down may re-suspend into the air after drying out. Thus, contaminants remaining on a humid wall or ceiling surface and those entrained in wet floor rubble may be prone to aerosolization once moisture levels return to salt cavern norms.

As a corollary to testing the wash down process, a test was conducted to determine if surrogate contamination is transported significantly into a bed of WIPP salt rubble upon wash down. The floor surface in the WIPP mine consists of several inches of salt rubble – residue from mining activities to open up the drifts and rooms of the mine. The test showed that rinsing of the surface did push contamination under the surface (peaking around 2 cm deep). In

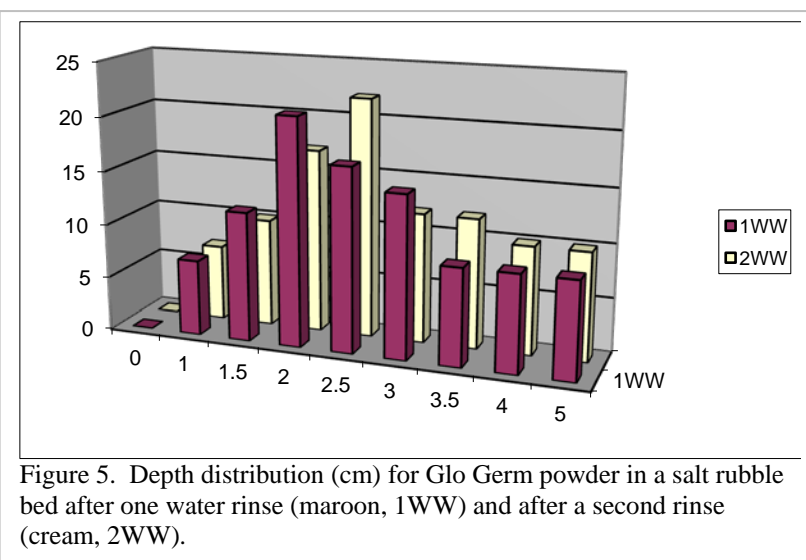


Figure 5. Depth distribution (cm) for Glo Germ powder in a salt rubble bed after one water rinse (maroon, 1WW) and after a second rinse (cream, 2WW).

the test, a 100 cm² salt rubble surface contaminated with 100 mg of Glo Germ powder was washed with a common hand pump sprayer that delivered about 20 ml of water over 15 seconds. Figure 5 shows the depth distribution of the powder within the rubble (red/maroon bars). Salt rubble was removed in a 5 cm diameter circle (~20 cm²) and each layer was examined. Powder was deposited throughout the 5 cm column, but the majority was in the 1.5 – 3 cm range. That distribution may be adequate to remove the threat of re-suspension, but it is still quite close to the surface. Further irrigation pushes the contamination lower; however, the figure shows that some fraction of the contamination is likely to remain in the upper portion of the salt rubble bed (yellow/cream bars).

RADIOACTIVE COUPON DECONTAMINATION

Surrogate contaminant tests determined that water washing and DeconGel 1108 strippable coating were effective methods for removing surrogate contamination from the surface of the salt, while other methods were decidedly less capable. Radioactive tracer tests were conducted with the two more effective methods.

An americium tracer solution (^{241}Am) at a concentration of approximately 8 nCi/ml was applied to salt coupons and steel plates in a stippling fashion. Stippling consists of placing small drops, in this case 0.025 ml each, of contaminant on the surface of the target material. This level of tracer yielded alpha contamination levels of approximately 21,000 disintegrations per minute (dpm) for the steel plates (used as a standard/baseline) and about 2,700 dpm for the salt coupons. Stippling is an established technique for preparing standards to determine matrix effects with radiometric instruments. A stippled steel plate is shown in Figure 6. The stippling was confined to an area the size of the radiometric detector probe being used for these tests.

The americium tracer was applied to two steel plates and six salt coupons. The salt coupons used were the most regular of the twelve, $\sim 100\text{ cm}^2$, 3 cm thick coupons. As the tracer was applied to the surface of the salt, it was observed that it did not bead, like in Figure 6, rather it wicked into the surface pores, cracks, and imperfections. The structure of the salt appears to have $\sim 1\text{ cm}$ grains, which allows solution to imbibe into the intergranular areas. This explanation for the observed behavior is supported by the results of pre-decontamination measurements: The same amount of tracer returned $\sim 13\%$ of the radiometric counts that were found on the steel plates. The tracer had likely penetrated into the salt matrix, attenuating its detectable activity.

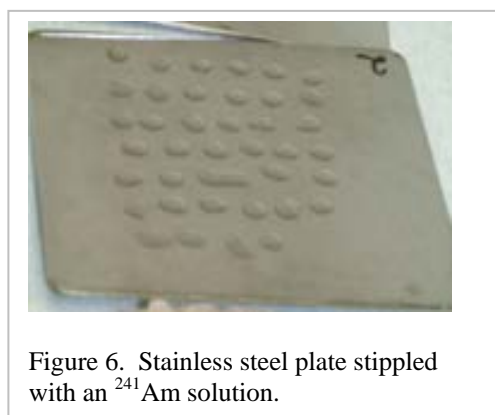


Figure 6. Stainless steel plate stippled with an ^{241}Am solution.

Radiometric counting was done by a Ludlum 2224 "scaler" handheld meter, using a 60 second count. This meter has a 20% efficiency for alpha and beta/gamma activity. Analysis showed typically 2500 – 3000 dpm/100 cm^2 alpha before decontamination and 70 – 195 dpm/100 cm^2 alpha, post decontamination, using either decontamination method. See Table II. The alpha activity data shows that removal efficiency averaged 96% and was consistently $\geq 93\%$.

Two different quantification methods were attempted for the gamma radiation portion of the test. Unfortunately, neither could provide an alternative method for quantifying the decontamination results. A portable, high purity germanium gamma scan unit, the ORTEC Detective, found insufficient radiation signature from the ^{241}Am spike levels to permit good quantification, although it did provide ready identification of the spike material as ^{241}Am . The Ludlum 2224 unit used for alpha detection was also employed, this time in beta/gamma mode. It did not provide acceptable results. The Ludlum beta/gamma readings averaged 746 dpm before decontamination and 674 dpm after, with a background of ~ 640 dpm (general background activity in the hood).

Table II. Alpha Contamination Quantification for Americium Tracer Tests

Decon Method	Sample #	Alpha Before Decon corrected		Alpha After Decon corrected		Alpha Removal [%]
		[cpm]	[dpm]	[cpm]	[dpm]	
Water wash	W103	579	2895	29	145	94.99
Water wash	W101	526	2630	22	110	95.82
Water wash	W102	658	3290	14	70	97.87
None	Steel coupon #2	4322	21610			
Strippable Coating	W105	713	3565	20	100	97.19
Strippable Coating	W106	561	2805	39	195	93.05
Strippable Coating	W104	475	2375	23	115	95.16
None	Steel coupon #1	4188	20940			
Water wash	WB201 blank*	0	0	3	15	
Strippable Coating	WB202 blank*	0	0	11	55	

* Alpha background determined to be ~27.5 dpm.

Water washing was by far the easier method of decontaminating these coupons and was also highly effective. The process used was the same as that previously established during the non-radioactive testing: a 15 second water rinse using a spray bottle. A photograph of this method is shown in the left pane of Figure 7. The rinsate was collected and found to amount to about 20 ml from each coupon, which is essentially complete recovery of the solution (as measured in earlier experiments). Scaled to practical use, the volume used for water washing is ~2 L/m² of decontaminated surface. One ml of each 20 ml volume was counted using liquid scintillation to determine the amount of radioactivity recovered. The measured activity averaged 6,533 dpm alpha per coupon, while only ~2700 dpm alpha surface



Figure 7. Left: Water washing americium from a salt coupon. Right: Removing DeconGel strippable coating from a salt coupon.

contamination was detected by the Ludlum meter prior to decontamination. This result indicates that washing removed virtually everything from the surface, but only ~31% of the total applied (the steel control plates registered 21,000 dpm). The remaining contamination is thought to be entrained in the porosity of the coupon.

While the DeconGel strippable coating was also highly effective at removing contamination, it was time consuming and difficult to remove. It took, on average, 15 minutes to remove approximately 95% of the coating. Complete removal was not possible. Vetting what was seen with the non-radioactive tests, the strippable coatings became somewhat incorporated into the salt surface and were very difficult to remove – much more difficult to remove from the salt surface than from stainless steel or aluminum. A photograph of this portion of the test is seen in the right pane of Figure 7. The surface of the coating was scored with a plastic knife to give a place to begin peeling the coating. The coating materials were found to work better as fixatives rather than strippable coatings.

FIXATIVE COATINGS PERFORMANCE

Based on how the strippable coatings bonded with the halite surface, fixative coatings were tested as a method of immobilizing contamination. The envisioned use of these coatings would be to affix and seal contamination in place on WIPP cavern ceilings and walls, and within the salt rubble that covers the drift floors. Ease of application and coating quality were assessed, and coating durability was tested.

BHI Energy's Stripcoat TLC Free, a water based solution, is primarily intended as a strippable coating for removing contamination; however, it is promoted as a barrier to, or fixative of, contamination as well. It was tested alongside two other coatings designed as barriers: Bartlett Nuclear's Polymeric Barrier System (PBS) and Minova's Tekflex PM. The Polymeric Barrier System is a water based polymeric solution designed for fixing contamination and forming an impermeable barrier. Tekflex PM is a cement modified polymer coating designed for mining applications to consolidate and solidify mined surfaces. Once cured, Stripcoat and PBS form an elastomeric membrane. Tekflex forms a more brittle, cementitious coating. The flexible coatings performed better. The elastic nature of those barriers sustained abuse much better than the more brittle Tekflex coating. Of the two elastic coatings, PBS appears to be the better candidate – demonstrating a decided advantage in the degree of deformation and abuse necessary to compromise the coating.

The PBS and Stripcoat coatings are less viscous during application than Tekflex, which is designed for use on mine drift walls and ceilings. Thus, application of PBS to a ceiling may be challenging. If it proves too difficult to deposit on the drift ceilings, Tekflex could be substituted. The ceilings being less likely to sustain damage or wear than the walls and rubble floor, Tekflex's more brittle nature would not be a significant drawback. As the ceilings deform and settle over time, though, the coating will start to develop cracks sooner than would a PBS coating.

Coating Application

Each coating was sprayed on two types of salt samples – solid halite coupons and halite rubble beds. The rubble samples were built up in trays constructed of wood 2×4 boards arranged in a square and screwed to a plywood backing. The sample area is 30 cm × 30 cm × 4 cm deep. WIPP salt rubble with a particle size distribution centered at approximately 5 mm in diameter was placed in the sample trays.¹ The samples were then compacted by repeatedly driving over them until the salt rubble ceased to compact.

The coatings were applied at room temperature (~20°C) through a 3 mm nozzle on a HVLP (high volume low pressure) paint sprayer operating at 30 psi. In the first round of testing each coating was sprayed onto

¹ The material was not sieved or screened. It was simply hand sorted to eliminate large chunks.

samples positioned horizontally. In a second trial, the coatings were applied with the samples propped against the wall nearly vertically.

Four coatings or coating combinations were created for the first set of tests:

1. PBS (red)
2. Stripcoat (yellow) – Referred to as “TLC” in the sample labels visible in photographs.
3. Tekflex PM (gray)
4. Stripcoat + PBS (reddish orange) – Stripcoat was applied first, allowed to dry, followed by a coat of PBS.

In the second test set, four additional coating combinations were tried:

1. Tekflex + PBS – Tekflex was applied, allowed to dry, then PBS was applied as an overcoat.
2. PBS + Tekflex – PBS, then Tekflex.
3. Tekflex + Stripcoat – Tekflex, then Stripcoat.
4. Stripcoat + Tekflex – Stripcoat, then Tekflex.

On the rubble samples, PBS and Stripcoat tended to infiltrate rather than coat the material during application. This issue was overcome if a sufficiently thick coating of the solutions was applied. When these materials were sprayed on the vertically orientated samples, running was observed, but a continuous coating was still achieved.

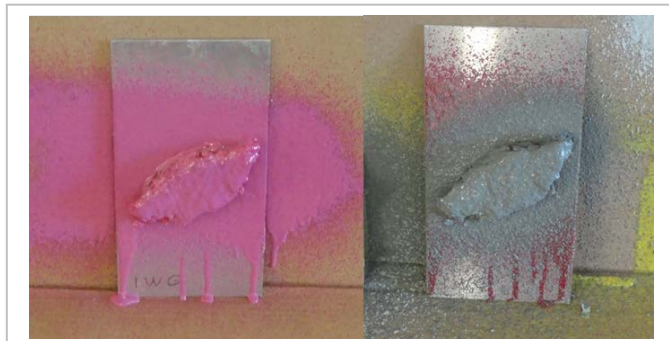


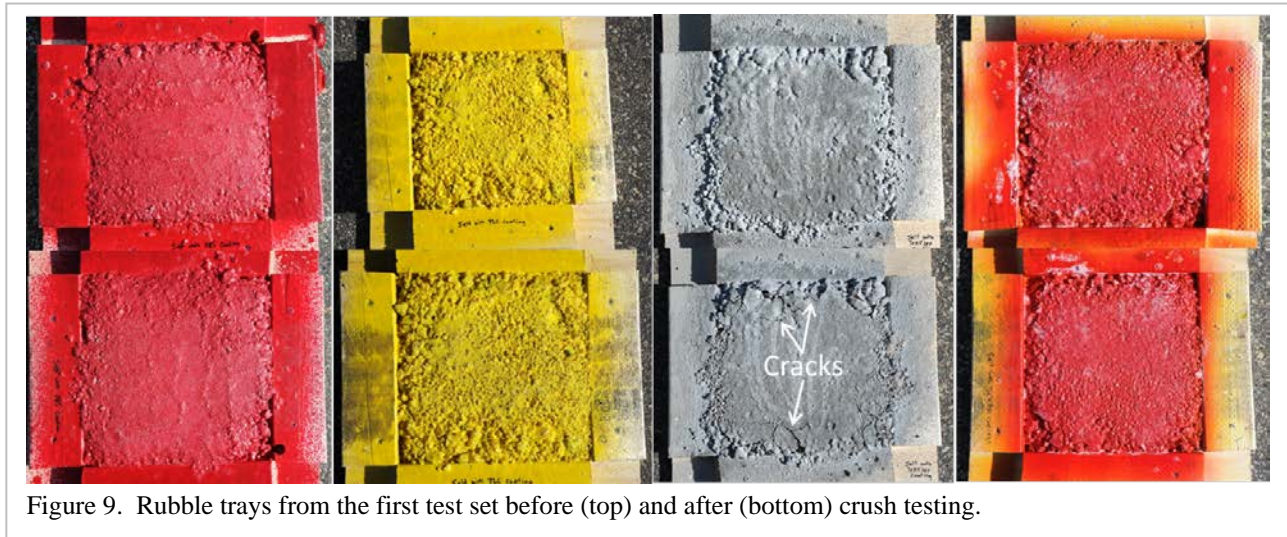
Figure 8. Application of PBS (left) and Tekflex (right) to a vertical surface. Note the propensity of the PBS coating to run during application.

Applying either material overhead may be quite messy. The Tekflex, on the other hand, stayed where it was applied. See Figure 8.

Once applied and dry, all coatings formed a good barrier on the sample surfaces. White spots appeared on the surface of the coatings (see the leftmost coupon in Figure 11), likely fine salt crystals that dissolved in the wet solution and then recrystallized. The white spots are most evident on the samples coated with PBS, probably because of the higher color contrast between the red coating and the white crystals. The Stripcoat and PBS barriers formed fine bubbles upon application. The bubbles were most likely formed from entrained air in the pneumatic sprayer, or they may have been produced due to a chemical reaction between the solution and the underlying salt.

Coating Durability

To test the strength and durability of the coatings, each sample was subjected to a crush test. The test consisted of driving over each sample with one tire of a ½ ton pickup (curb weight ≈ 2300 kg). In the first round of tests, each sample was subjected to four passes under the truck tire. After each pass, the sample was photographed and a qualitative visual inspection was conducted. In the second round of tests, each sample was subjected to ten passes under the truck tire.



Rubble Beds

Each of the rubble samples would compress ~6 mm as the tire pressed down on it, then relax once the load passed. In the first round of testing, the Tekflex sample developed cracks after the first pass. The cracks grew more extensive with each pass, but the sample largely stayed intact. The PBS, Stripcoat, and Stripcoat + PBS samples showed no cracking, retaining a largely intact barrier over the rubble. Figure 9 shows the samples before testing and after four passes were completed.

In round two of testing, the coatings with a top layer of Stripcoat or PBS proved quite durable, with no failure of the membrane even after ten passes under the truck tire. See Figure 10. The coatings with a top layer of Tekflex sloughed Tekflex powder with each pass under the tire, but the underlying elastic coating maintained its integrity. Based on these results it does not appear that Tekflex enhances coating strength or stability. When applied as an undercoat, it simply adds another, superfluous step in the process. When applied as an overcoat, it proves to be a dust source.

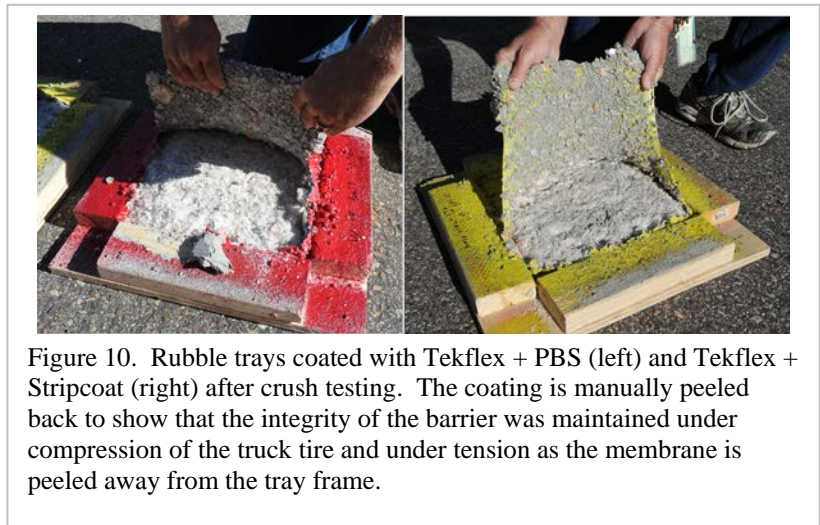
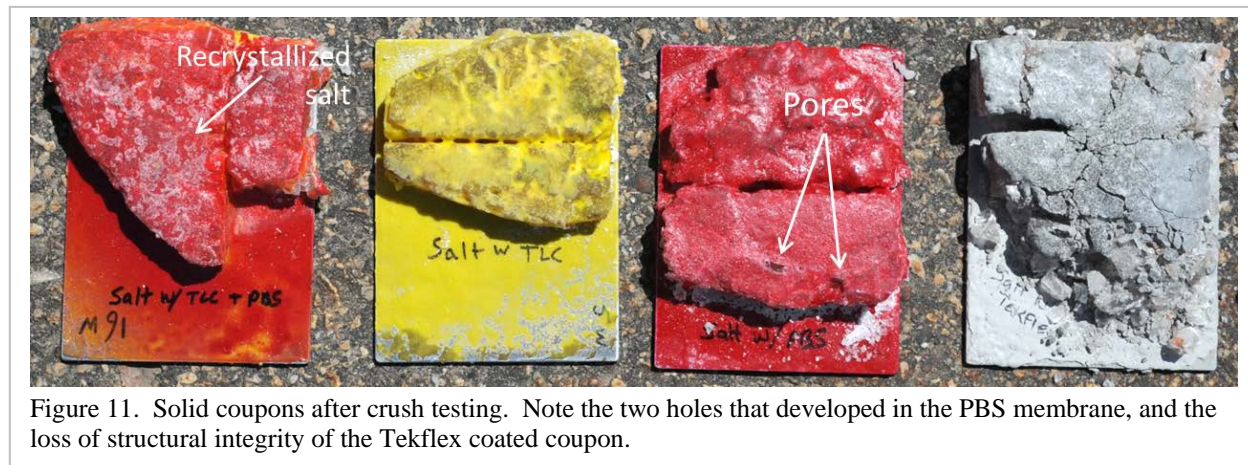


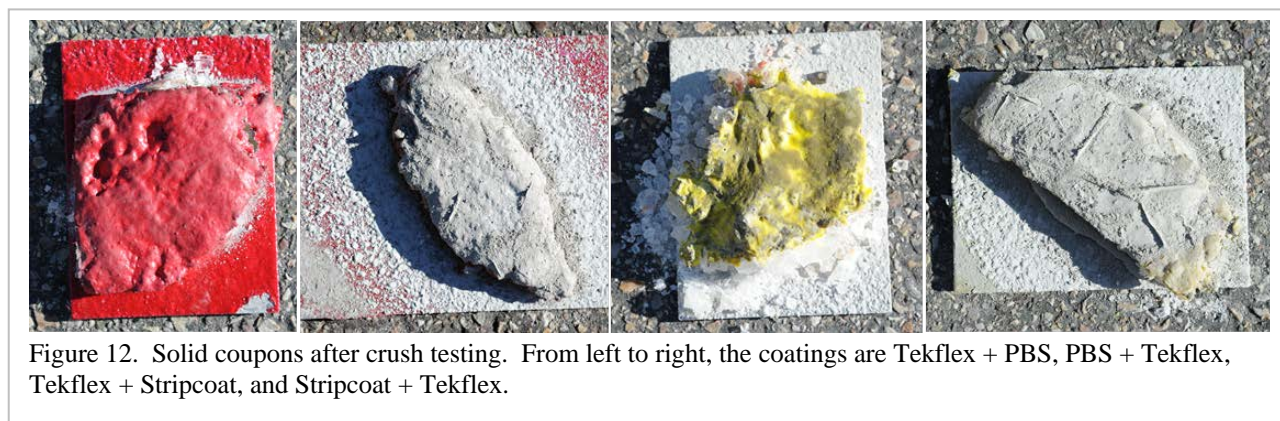
Figure 10. Rubble trays coated with Tekflex + PBS (left) and Tekflex + Stripcoat (right) after crush testing. The coating is manually peeled back to show that the integrity of the barrier was maintained under compression of the truck tire and under tension as the membrane is peeled away from the tray frame.

Solid Coupons

Unlike the rubble beds, the solid coupons did not have a frame to support their edges. As a result, all of the solid coupons exhibited some crumbling and fracturing along the fore and aft edges. *In situ*, edge effects may not be a significant issue, other than perhaps at wall corners. Aside from those edge effects, in test round one the more brittle Tekflex coating again fared the worst, fracturing extensively. The PBS and Stripcoat coated coupons showed virtually no wear or damage, other than along the edges. Two holes/pores were exposed in the PBS sample. A second spray coat of material would seal up the exposed porosity. Figure 11 shows the coupons after four passes under the truck tire.



In round two of testing solid coupons, all four coating combinations survived ten passes under the truck tire with their membranes intact; however, the sample with an outer membrane of Stripcoat (third pane from the left in Figure 12) lost much of its structural integrity around the perimeter of the coupon. The sample with an under layer of Stripcoat (rightmost pane in Figure 12) fared much better. It is not clear why the Tekflex + Stripcoat sample fared so much worse than the others. The Tekflex outer coating (the gray colored coupons in Figure 12) was ground down under tire wear, providing a powdery surface and a dust source – a result seen also in the rubble beds. The ground, powdery surface is most evident in the Stripcoat + Tekflex sample (rightmost image in Figure 12), where the tire tread is impressed into the loose coating. Like the rubble beds, the addition of the Tekflex layer does not appear to enhance coating integrity, except perhaps when combined with Stripcoat.



SUGGESTED FUTURE WORK

Based on what was learned in these initial experiments, several avenues for additional investigation are logical. One investigative path would be to perform a scientific study of the interaction of americium with halite under a variety of systematically varied conditions (e.g., humidity, pH). This line of inquiry would serve to benchmark the results of the radioactive tracer tests already conducted, as well as provide insight into the potential effect of various atmospheric conditions – during dispersal and decontamination – in the mine.

Further evaluation of halite's ability to hold contamination in place once it is rinsed down into the rubble or into pores in the solid halite is another logical area of investigation. The potential for re-release after water in the aggregate or porosity dries out, and after the surface is subjected to traffic or wear is of particular interest.

A third avenue of investigation would be to evaluate application of water washing or a barrier coating at a larger scale, perhaps a ~20 m³ room. Criteria such as application time, material consumption, waste water and secondary waste generation, and safety concerns could be evaluated more realistically at this scale. Other factors that could be evaluated include the ease of vertical and overhead application of the PBS coating, and the potential benefits of applying a mixture of Tekflex and PBS. Is such a mixture more viscous, leading to better application performance? Once cured, does it provide the same barrier elasticity and durability as the PBS coating alone?

A fourth area of investigation that is warranted is evaluation of fogging as a decontamination and fixative method. The INL has extensive experience implementing the fogging method as a decontamination tool.

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