

## **Understanding Contamination; Twenty Years of Simulating Radiological Contamination - 12430**

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### **ABSTRACT**

A wide variety of contamination simulant methods have been developed by researchers to reproducibly test radiological decontamination methods. Twenty years ago a method of non-radioactive contamination simulation was developed at the Idaho National Laboratory (INL) that mimicked the characteristics of radioactive cesium and zirconium contamination on stainless steel. This method involved baking the contaminants into the surface of the stainless steel in order to create a tenacious, tightly bound oxide layer. This type of contamination was particularly applicable to nuclear processing facilities (and nuclear reactors) where oxide growth and exchange of radioactive materials within the oxide layer became the predominant model for material/contaminant interaction. Additional simulation methods and their empirically derived basis (from a nuclear fuel reprocessing facility) are discussed.

In the last ten years the INL, working with the Defense Advanced Research Projects Agency (DARPA) and the U.S. Environmental Protection Agency (EPA), National Homeland Security Research Center (NHSRC), has continued to develop contamination simulation methodologies. The most notable of these newer methodologies was developed to compare the efficacy of different decontamination technologies against radiological dispersal device (RDD, or “dirty bomb”) type of contamination. There are many different scenarios for how RDD contamination may be spread, but the most commonly used approach to simulating RDD Contamination at the INL involves the application of an aqueous solution containing Cs-137 to selected surfaces. This contaminant application method was chosen during the DARPA projects and has continued to be used during most of the NHSRC series of decontamination trials and also gives a tenacious “fixed” contamination. Much has been learned about the interaction of cesium contamination with building materials, particularly concrete, throughout these tests. The effects of porosity, cation-exchange capacity of the material and the amount of dirt and debris on the surface are very important factors. The interaction of the contaminant/substrate with the particular decontamination technology is also very important. Results of decontamination testing from hundreds of contaminated coupons have lead to certain conclusions about the contamination and the type of decontamination methods being deployed.

In addition to the DARPA aqueous contamination method, recent work by the EPA at INL included the development and validation of an approach simulating the deposition of nuclear fallout. This contamination approach differs from previous tests in that it simulates a “loose” type of contamination. This may represent the first time that a radiologically contaminated “fallout” simulant has been developed to reproducibly test decontamination methods. While no contaminant/methodology may serve as a complete example of all aspects that could be seen in

the field, the study of this family of simulation methods provides insight into the nature of radiological contamination.

## INTRODUCTION

Over the past 50 years, the Idaho National Laboratory (INL), along with the many nuclear institutions around the world, have faced significant decontamination challenges. The various facilities have different decontamination problems, some of which are quite substantial. The INL has been home to over 50 nuclear reactors and many other nuclear facilities; most of these nuclear facilities have also undergone Decontamination and Decommissioning (D&D). One particularly difficult challenge is decontaminating facilities that treat spent nuclear fuel. The US has four primary locations where nuclear fuel reprocessing took place: a commercial site at West Valley, New York, and several large scale government owned facilities at Hanford, Washington, Savannah River, South Carolina, and the INL in Idaho.

These facilities are somewhat unique in their operation, products and their overall decontamination needs. Most modern spent fuel reprocessing facilities make use of a variant of the plutonium, uranium extraction methodology (PUREX). These facilities “pickle” or dissolve the spent fuel and recover the valuable nuclear material, while separating and disposing of the undesirable fission products as secondary waste. During this process, the complicated processing equipment, including hundreds of feet of stainless steel pipes, valves, vessels and other equipment, becomes highly contaminated [1]. This happens in part because the contamination adheres to the surface of the equipment, and also leaks through valves. The contamination quantities can be substantial because the entire inventory of fission products and actinides are released inside the process, and many tons of fuel may be processed each month. Thus the need for decontamination at these facilities may become very significant.

About twenty years ago the Idaho Chemical Processing Plant (ICPP), currently known as the Idaho Nuclear Technology Engineering Center (INTEC), began a search for decontamination processes which minimize secondary waste. At that time, the currently employed decontamination practices produced liquid waste containing large amounts of salt. This is a difficult and costly waste to treat. The search progressed over years of evaluating a large variety of different chemical and non-chemical methods, a few of which were adopted for use in the processes. In order to test the effectiveness of new decontamination technologies, a new simulated contamination methodology, termed SIMCON, was developed. SIMCON was designed to replicate the type of contamination that developed on stainless steel processing equipment [2].

Ten years later, INL began to develop methods of simulating urban contamination resulting from a radiological dispersal device (RDD). This work was sponsored by the Defense Advanced Research Projects Agency (DARPA) and sought to develop and test cleanup measures that could be deployed against an RDD. While there are a few similarities in the type and mechanisms of contamination produced by many of the processes, the simulants and substrates contaminated in the DARPA experiments are vastly different from those used in the INTEC processes. Additional efforts sponsored by the US Environmental Protection Agency (EPA) have led to the development of a contamination method that simulates fallout from an

Improvised Nuclear Device (IND). The IND work is particularly innovative as it diverges from creating tenacious contamination to creating a reproducible “loose” contamination. Examining these different types of contamination (and subsequent decontamination processes), particularly in regards to each other, sheds light on contamination processes that occur throughout the nuclear industry, and even beyond into the urban environment.

The primary rationale for testing these simulated contamination processes is to determine their applicability for use as contaminants during evaluation of decontamination methodologies. Different contamination processes have been provided by other researchers and all should be viewed within their context of what they were meant to achieve and what types of contamination they model. The INL contamination methods are the products of applications research to compare the performance of various decontamination methods (at a single condition); they tend toward applied rather than fundamental research. Their primary purpose has been to provide a platform with which to compare different decontamination methods on a level playing field. As such, conclusions about the nature of contamination are often framed within the results of the decontamination processes.

## NUCLEAR CONTAMINATION MODELS

For many years there has been an accepted model, called the “CRUD (chromium oxide) Model”, for the visualizing how nuclear contamination adheres to metal. Contamination within nuclear reactors may be thought of as occurring chiefly in the oxide formed on the inner surfaces of the metal piping in a reactor’s primary coolant system. While some activation of base metal within the core area of the reactor does occur, the majority of the contamination comes from activation products, e.g. metal that becomes suspended in the coolant and passes through the core where it becomes activated. These suspended materials are often a mixture of metal oxides (and even ions) however the longer lasting, highly energetic isotope of Co-60 typically becomes the most significant problem overall. The contaminated metal oxide layer, commonly known as “CRUD”, becomes a trap for the suspended and ionic contamination. In addition, some fission products may leak from the fuel and become lodged in this CRUD layer [3]. The visual representation of this contamination model, proposed by the Electric Power Research Institute, is shown in Figure 1 [3].

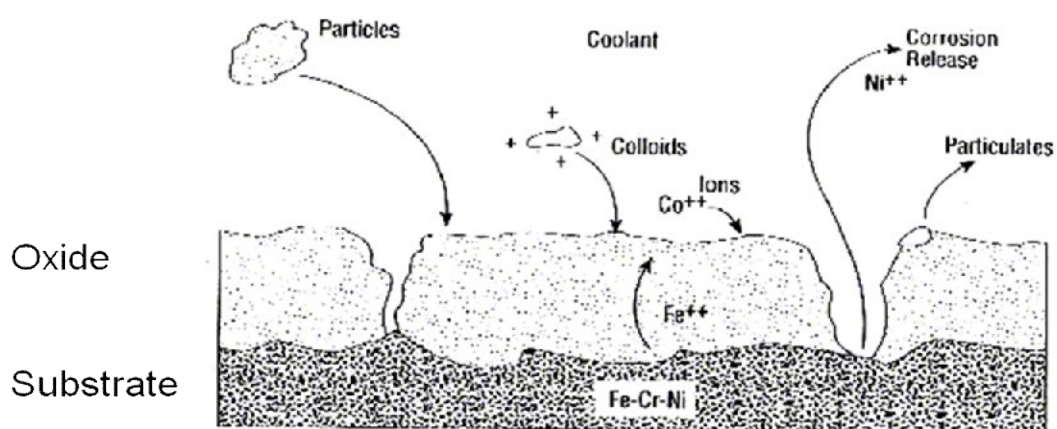


Fig. 1. EPRI “CRUD” Layer Contamination Model.

This model has served the nuclear decontamination industry well in terms of explaining contamination mechanisms and decontamination methods over the years. For instance, it was long known that an aggressive chemical decontamination process that utilizes a strongly oxidizing solution (such as alkaline permanganate) followed by a strongly reducing organic acid (such as oxalic acid) was highly effective at removing the tenacious oxide layer from stainless steel substrates. The mechanism is a two-step decontamination approach which removes the tenacious oxide layer and with it the contamination. Likewise, the INL has found that chemicals (such as nitric acid) that promote the growth of the oxide passivation layer on stainless steels are not highly effective at removing contamination. However, care must be made as to this generalization because some contamination is simply deposited as a solid on surfaces (such as a leak from a valve) and solutions that would not remove the oxide layer are sometimes highly effective at removing these deposits.

In the case of porous substrates, a different model may be utilized that describes the particular characteristics of the substrate. While similar in some respects to the EPRI CRUD model, this model also makes use of the porosity and cation-exchange capacity (CEC) of the material. Porous materials have a quality called “imbibition” that defines their ability to absorb contaminants beyond their surface. This imbibition depends on the porosity, the capillary action drawing the contamination further into the substrate pores, and the CEC that helps retain the contaminant in the material once it is drawn into the surface [4]. The main similarity between metal and urban (porous building material) contamination models is that the largest amount of contamination present typically resides in a “boundary” layer for porous materials (similar to the oxide layer). Removing this boundary layer, without causing additional imbibition is a significant task. For urban substrates, it is recognized that the boundary layer may consist of loosely bound, weathered material and grime. The INL porous material conceptual model is shown in Figure 2.

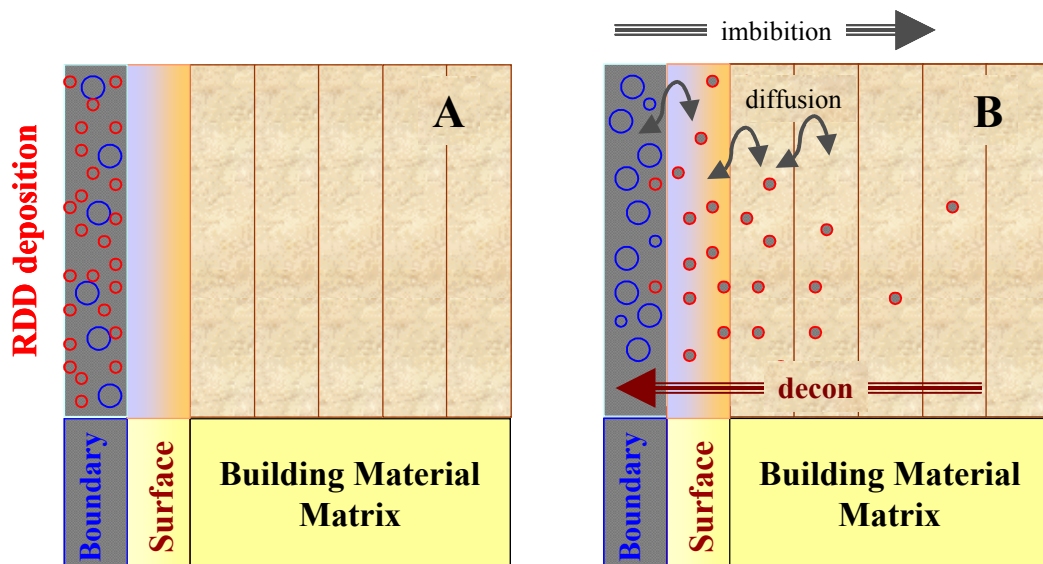


Fig. 2. INL RDD Deposition Model Showing Contaminant (in red) Diffusion.

## **NUCLEAR FACILITY STAINLESS STEEL CONTAMINATION**

Two contamination methods were developed at the INL to simulate the contamination found on stainless steel; they were termed SIMCON I and SIMCON II [2]. SIMCON I was simply a non-radioactive cesium or zirconium salt pipetted onto 25 mm diameter (7 mm thick) stainless steel disks and allowed to dry. This simulated a “loose” contamination that was relatively easily removed. The more tenacious and widely used simulant was SIMCON II. SIMCON II coupons have the non-radioactive cesium and zirconium salt baked onto the surface at 700° C to simulate fixed contamination. A sufficient quantity of cesium and zirconium was applied to give an approximate field (had it been radioactive) of 10 mSv/hr at 30 cm.. A wide variation exists in the initial quantity of salts adhered to the surface of the coupons. This is attributed to the vigorous (though inherently inconsistent) hand scrubbing they undergo to remove loose contaminants after baking. These coupons have been used at the INL to determine the effectiveness of many types of decontamination techniques (laser ablation, CO<sub>2</sub> pellet blasting, alternative chemicals, abrasives, strippable coatings, etc.) prior to using the techniques in radioactive environments. The amount of cesium and zirconium salts deposited on the surface of the coupons was determined using X-ray fluorescence, both before and after being treated by a decontamination process

Over 1000 coupons have been prepared and tested using various decontamination methods [5], [6], [7]. During these evaluations two observations have surfaced which were also noted in the tests detailed in this report. First, physical cleaning methods (non-chemical methods such as abrasive blasting) tend to preferentially remove the zirconium salts. This has been observed for several hundred coupons in all 15 non-chemical methods tested. Second chemical methods tend to preferentially remove cesium. Table I documents the results of selected physical and chemical decontamination methods. These results underscore the difference in the chemical nature and attachment of the different contaminants.

## **URBAN DECONTAMINATION**

In 2004 DARPA sponsored a project to develop new decontamination methods for urban environments after detonation of a RDD. The project supported several decontamination laboratories in their development programs. The INL was chosen for two aspects: developing and conducting urban contamination simulation tests and, in a separate effort, to develop a new decontamination methodology.

For testing of these newly developed decontamination technologies, three types of building materials were chosen as substrates, along with two common radionuclides as contaminants. The building materials were concrete, marble and granite. The two radionuclides were cesium-137 and cobalt-60. To produce a fairly uniform contamination, the radionuclides were sprayed on the building material surfaces. The use of these different contaminants and substrate resulted in significant differences in decontamination performance and in understanding different surface decontamination mechanisms.

Table I. SIMCON II Chemical and Non-chemical Decontamination Comparisons.

<b>Method</b>	<b>Cs</b>	<b>Zr</b>
Abrasive Grit Blasting	100%	100%
Abrasive Plastic Pellet Blasting	80%	93%
CO <sub>2</sub> Pellet Blasting	81%	100%
Bartlett TLC Stripcoat	42%	73%
Nd:YAG Laser Ablation	75%	99%
Hot Water	72%	17%
Alkaline Permanganate	60%	29%
Nitric/oxalic	50%	20%
NPOx Tests 3 & 7 –w/ ultrasonic bath and heat	100%	100%
NPOx Test 5 – w/ heat, no ultrasonic bath	95%	80%

In 2007 Environmental Protection Agency's (EPA) National Homeland Security Research Center (NHSRC) began conducting performance evaluations of commercial, off-the-shelf radiological decontamination technologies such as those originally developed for the nuclear power industry and the DOE complex using the protocols developed under the DARPA program. These tests focused on the decontamination of concrete contaminated with cesium-137 [8].

Concrete coupons, prepared during the DARPA project in 2005 were made from a single batch of concrete based on Type II Portland cement. The concrete was poured into 0.9 m square plywood forms and the surface was "floated" to bring the smaller aggregate and cement paste to the top, and then cured for 21 days. Following curing, square coupons were cut to the desired size with a laser-guided saw. For this evaluation, the "floated" surface was used as the working surface to minimize the possibility of chemical interferences due to mold release agents. The coupons were approximately 15 cm × 15 cm × 4 centimeters (cm) thick, with a surface finish that was consistent across all the coupons and that was representative of concrete structures typically found in an urban environment. The edges of the coupons were sealed with polyester resin and masked with tape to ensure that the contaminant would be applied only to the working surface of the coupon. These coupons were used for both the contaminated samples as well as the clean, uncontaminated, control samples.

The experiments initially used a contamination level of 50 µCi/ coupon. Later experiments used a much lower level (1 µCi/coupon), which was more appropriate for larger scale tests. For large

scale tests, we determined that health and safety concerns dictated lower levels and a new analysis of the contamination scenario agreed that 1  $\mu\text{Ci}/\text{coupon}$  was valid for urban contamination. Each coupon selected for contamination was spiked with 2.5 milliliters of an unbuffered, slightly acidic aqueous solution containing 0.4  $\mu\text{Ci}/\text{ml}$  Cs-137, which corresponds to an activity level of approximately 1  $\mu\text{Ci}$  per coupon. The liquid spike was delivered to each coupon using an aerosolization technique. The aerosol delivery device was constructed of two syringes. The first syringe had the plunger removed and a pressurized air line attached to the rear of the syringe. The second syringe contained the aqueous contaminant solution and was equipped with a 27 gauge needle which penetrated through the plastic housing near the tip of the first syringe. Air was supplied at a flow rate of approximately 1 - 2 liters per minute creating a turbulent flow through the first syringe. The liquid spike in the second syringe was introduced and became nebulized by the turbulent gas flow. The result was a very fine aerosol ejected from the tip of the first syringe, creating a controlled and uniform spray of fine liquid droplets deposited over the entire coupon working surface. These coupons are used within a short time frame; at least one week, and less than 21 days after contamination.

The level of gamma radiation emanating from the surface of the concrete coupons was measured both before and after application of the decontamination technologies to evaluate their decontamination efficacy. These measurements were made using an intrinsic, high purity germanium detector, which was regularly calibrated over the course of testing using standard instrument calibration procedures.

Ideally, decontamination technologies must not only be effective in removing threat contaminants from typical building materials, but must do so without being destructive to building surfaces. Due to the large areas likely to be affected by such an event, both the time required to perform effective decontamination and the cost of deployment are significant issues. An emphasis on “low-tech” methodologies led to the selection of simple, low cost, easy-to-use technologies which can be transported and deployed quickly, requiring only minimal support services or infrastructure.

These tests, using concrete contaminated with cesium solutions, give an idea of the degree of difficulty for removal of contamination resulting from a cesium-RDD. The results of the decontamination performance portion of those evaluation tests are summarized below in Table II. A detailed evaluation of each decontamination technology (performance, operability, costs, etc.) is available on the EPA website ([www.epa.gov/nhsr/pubs.html](http://www.epa.gov/nhsr/pubs.html)). For this contamination scenario it is interesting to note that the chemical techniques, while highly effective, were not quite as effective as a brute force removal of the surface (as in grit blasting). But the trade-off is more damage to the surface and more potential for transfer of contamination (i.e. imparting energy into the radioactive particles via pneumatic or rotary force). While no contamination was noted on the blanks, one air radioactivity monitor showed a slight elevation of contamination during mechanical tests, but not during chemical tests. Another conclusion is that this is a very tenacious type of contamination; that by intending to contaminate an item (as an intentional act) we created a very difficult problem to solve.

Table II. Chemical and Non-chemical Removal of Cesium Contamination from Concrete.

Method	% Removal	Std. Dev.
EAI Rad-Release II	85	± 2
Argonne SuperGel	73	± 5
EAI Rad-Release I	71	± 13
Decon Gel 1108	67	± 9
QDS Liquid	53	± 7
INTEK ND-600	52	± 12
QDS Foam	51	± 8
Decon Gel 1101	49	± 7
INTEK ND-75	47	± 6
Empire Blast Grit Blaster	96	± 3
Dust Director Diamond Flap Wheel	89	± 8
CS Unitech Sander	54	± 10
Dust Director Wire Brush	38	± 7
River Technology Rotating Water-jet	36	± 4

An additional series of tests was conducted to determine the effectiveness of common household cleaners on cesium contaminated indoor surfaces. These tests involved the use of the widely available Simple Green™ detergent side-by-side with water. The results are shown in Table III. In most cases the Simple Green™ removal efficacy was not significantly different from that of the water. Additionally, the porosity of the material being decontaminated had a large effect on the overall ability for both the Simple Green™ and the water to remove the contamination. In the case of granite, it is likely that the minerals that comprise the granite chemically bind the cesium reducing the ability of the both the detergent solution (Simple Green™) and the water to remove the contaminant.

Table III. Comparison of the Percent Removals for Simple Green and Water on Typical Interior Surfaces.

Material	%R (Simple Green)	%R (water)
Plastic laminate	97.6	93.4
Vinyl flooring	96.7	96
Granite	31.4	7.7
Poly coated wood	67.2	68.1
Painted wallboard	9.5	7.3
Stainless steel	97.5	94.8

## IMPROVISED NUCLEAR DEVICE FALLOUT SIMULANT

Some previous efforts had been made to characterize and simulate nuclear fallout for the purposes of detection and identifying biological pathways. Little has been done in terms of simulating fallout for the purpose of decontamination research. Fallout particle composition is



primarily made up of the local soil with the radionuclides precipitated and condensed on the surface of the particles. Local soil composition varies across the US. However both sand and clay are prevalent in the soils found in many US cities. Vaporized material forms small metallic oxide particles which become radioactive through inclusion of fission products, like Cs-137, into those particles. These particles agglomerate with particles of dirt that were swept into the fireball. Fission products, like Cs-137 also condense on the surface of these particles. Because of its ease of detection and ability to irreversibly bind with both clay and sand cesium offers an advantage for this kind of research.

Another aspect of fallout is that research shows that contaminants do not readily leach from the particles because of their spheroid, vitreous nature (due to the intense heat of the nuclear reaction) [10]. Clay and sand to simulate components of a non-leaching fallout were evaluated using various, known cesium retaining materials such as: AMBERLITE™ IRC-748 iminodiacetic acid chelating cation exchange resin; Diversified Industries cesium specific aluminosilicate/zeolite DT-30, a Kaolinite clay and a Montmorillonite clay. Two grams of each material was spiked with 2 mL of 0.4  $\mu\text{Ci/mL}$  of Cs-137 (yielding 0.8  $\mu\text{Ci}$  of Cs-137 on each of the materials), brought to a mixture volume of 15 ml, equilibrated in a test tube and allowed to separate for two hours. The supernate was decanted, an additional 15 ml of deionized water added, the tube shaken and the slurry allowed to separate for two hours. This process was repeated again and an aliquot of each rinsate was counted via liquid scintillation. After the first rinse, the DT-30 and Kaolinite clay each achieved very high rates of retention (measured as loss to the rinsate) of about 98%. These two materials were then used to create the solid, non-leaching cesium fallout simulant.

The target particle size distribution of typical nuclear fallout has been estimated to be between 44  $\mu\text{m}$  and 1000  $\mu\text{m}$  [11], with the majority less than 700  $\mu\text{m}$  [12]. The Kaolinite clay was already of a particle size that adequate yield passed through a #25, 300  $\mu\text{m}$  sieve. The DT-30 was crushed with a mortar and pestle and passed through a similar sieve, yielding particles less than 710  $\mu\text{m}$ . After the appropriate particle sizes were obtained, various proportions of DT-30 to clay were prepared. The 75%/25% ratio of DT-30/clay produced a powder that was easy to manipulate, free flowing, and gave an adequate representation of a two component “soil like” matrix, and used as the fallout simulant.

One batch of contaminated, simulated IND fallout was prepared by combining the appropriate amounts of DT-30 and kaolinite clay particles (sized as described above) to obtain the 75%/25% ratio. Approximately 40 g of simulated IND fallout material was prepared by weighing the desired amount of each material on an analytical balance, pouring them into a plastic bottle and mixing well by inverting repeatedly for approximately one minute. Then, 50 mL of aqueous Cs-137, with a total activity of 100  $\mu\text{Ci}$ , was added to the bottle containing the simulated IND fallout. The bottle was sealed and shaken for approximately 30 seconds and then poured into a plastic dish and allowed to dry overnight in a fume hood. The water evaporated off the particles leaving the Cs-137 on the surface of the particles at a concentration of approximately 1  $\mu\text{Ci/g}$  of simulated IND fallout material. The uniformity of the simulant was tested by removing an aliquot of both the DT-30 and clay (gravimetrically separated) particles and analyzing separately after

the overnight drying. Those aliquots showed that the cesium had uniformly distributed through both materials.

Because there was concern with the fallout particles aggregating after preparation, the Cs-137 tagged fallout material was placed in a mesh covered bottle and covered with a #50 Sieve sieve yielding particles less than 710  $\mu\text{m}$  for application onto the concrete coupons. The mass of the fallout simulant applied was determined by weighing an aliquot into each bottle. The target applied mass was 1.5 -2.0 g across the surface of the concrete coupons. Method development testing using untagged simulant gave the technician an idea about how thick to apply the particles to attain the target mass. A picture of a concrete coupon contaminated with DT-30, a component of the fallout simulant, on a concrete coupon is shown in Figure 3.

Tests were conducted to determine the ability of a sophisticated, high pressure rotary water jet system (RWJS) to remove this fallout from the concrete coupons. The RWJS is manufactured by River Technologies for the purpose of surface cleaning at nuclear power plants. It is composed of a rotating, double ended spray wand inside of a housing, along with a concentric outer housing that draws a very high vacuum. This unit is shown in Figure 4. The results of the tests are shown in Table IV [13]. In these tests the contaminant (Cs-137) was designed to be fully incorporated in the particle and not to be transferred from the particle to the surface (it is instead unavailable to the bulk of the concrete). In order to better simulate nuclear device blast conditions that have been shown to produce fallout particles composed of strongly bound fission products. The results of the preliminary tests demonstrate that this simulant performed that function quite well, with virtually all of it being removed by the water jet. Even the vacuum without the water jet was shown to be quite effective at removing this type of contamination.

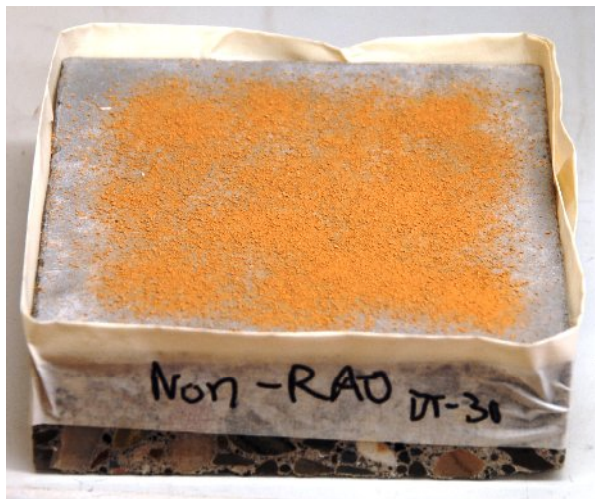


Fig. 3. Concrete Coupon Contaminated With the DT-30 Component.



Fig. 4. River Technologies Rotating Water Jet.

Table IV. Results of Decontamination using RWJS.

Method	Average %R	Std. Dev.
Dry Vacuum Only	95.4	1.6
Ambient Water (25 C) RWJ	97.5	0.7
Hot Water (82 C) RWJ	97.3	0.7

## CONCLUSIONS

After reviewing twenty years of efforts to simulate contamination several conclusions may be drawn:

- There are many ways to simulate contamination because there are a large variety of contamination scenarios.
- Some contamination will usually remain on or near the surface for a short duration. This contamination will often be termed “loose” or removable because it can be washed, vacuumed or brushed off. Loose contamination can also become “fixed” if it is allowed to remain on the surface for long periods of time and or to experience an event (such as rainfall) that mobilizes it further into the substrate. Therefore, removing contamination quickly with a “low impact” method (strippable coatings, vacuum cleaning, etc) can often be a cost effective method.
- Porous materials with high cation exchange capacity (like concrete) are typically the most difficult to decontaminate. Surfaces that have complex mineral or chemical interactions with the contaminant, as in the case of granite, can also be extremely difficult.
- The chemical nature of the contaminant may significantly impact its ability to be removed. Small, highly charged ions are typically much more difficult to remove than

large neutral species (e.g. precipitated solids). Mixed species (such as mixtures of actinides, metals and alkali fission products) can be among the most difficult to remove.

- Characterizing the profile (depth) of the contamination can be of great assistance in affecting decontamination. The barrier layer in building materials (e.g. “grime” layer), and the oxide layer on metals can trap and hold a significant fraction of the contaminants. Removing these layers without mobilizing the contaminant further into the substrate can be simple and often very effective.
- There is a tremendous difference between contamination that is simply deposited on the surface and contamination that is purposely, tenaciously fixed into the surface. Solid particles deposited onto the surface, particularly if they are loose (e.g. the IND case), are the easiest contamination to remove.
- Surface removal, though more destructive (e.g. abrasive blasting) can be far more effective at removing contamination than more sophisticated methods that “chemically” draw it away from the surface. However, there are many where non-destructive techniques can be more cost effective; particularly if a high value item (or structure) is sensitive to damage caused by mechanical techniques. There is no “one hammer” that satisfies every decontamination criteria.

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