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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA o LOS ALAMOS NEW MEXICO

PROCEDURES FOR DECONTAMINATION OF PLUTONIUM FROM VARIOUS SURFACES

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LOS ALAMOS SCIENTIFIC LABORATORY
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PROCEDURES FOR DECONTAMINATION OF PLUTONIUM FROM VARIOUS SURFACES

by

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ABSTRACT

Decontamination solutions, procedures, and their effectiveness in the removal of plutonium from various types of surfaces are reported.

CONTENTS

INTRODUCTION

A list of decontamination procedures and solutions was compiled from the sources listed in the Bibliography. Although the major portion of the work reported has been done with fission product contamination, the data are generally applicable to plutonium decontamination. Some of the solutions were used in the plutonium solvent extraction plant at Los Alamos and the results are noted in the section on decontamination procedures and solutions.

From these literature sources and the experiments at Los Alamos, a list of decontamination solutions in order of decreasing effectiveness and recommended procedures for decontamination have been prepared. It should be emphasized that there is no universal decontamination solution available. While one solution may decontaminate several types of materials readily, it may be too corrosive for some of them.

DISCUSSION

There are many factors which influence the effectiveness of a decontamination solution or procedure, all of which must be taken into consideration when planning any decontamination job.

The variables and some points for consideration are:

1. Type of material to be cleaned.
   a. Generally, there are one or two decontamination solutions which give better decontamination and less corrosion for one type of material than for any other type.
2. Previous history of surface.
   a. If the surface has been through several contamination-decontamination cycles, it will be more difficult to clean (and will require one of the more active decontaminants recommended for its material type) than if the contamination occurred for the first time. This is especially important if corrosive decontamination agents were used previously.
3. Whether contamination was dust or liquid.
   a. Dust should be removed with a vacuum cleaner followed by wiping with rags wet with a solution which will aid in collection of dust but will not dissolve dust or corrode the surface. A neutral solution of Na EDTA is satisfactory.
   b. In case of a liquid spill, excess solution should be removed by swabbing with cheesecloth or by vacuum removal to a tank followed by either H_2O and decontamination solutions

or by decontamination solutions alone. The radioactive solution should not be allowed to dry before use of a decontamination solution is started, if at all possible.

4. Type of liquid contaminant.

   a. If the radioactive solution is corrosive to the surface under consideration, then a decontamination solution which will dissolve a thin layer of surface should be chosen.

      Other contaminating solutions may dissolve a part of the surface and then precipitate a new compound; may form a complex with the surface, as is the case with many plastics; or may merely dry and be essentially a salt mechanically held in pits, scratches, etc., of surface under consideration, each requiring a different decontamination solution.

5. Age of contamination.

   a. Concerns mainly surfaces which can be deeply penetrated by contaminating solution, such as bare concrete by nitric acid solutions or painted surfaces by caustic or organic solutions. Obviously the length of time the contaminating solution has had to penetrate will determine how active a decontamination solution should be chosen.

6. Temperature of surface during drying of liquid contaminant.

   a. Temperatures over 300°C produce plutonium oxides which are hard to dissolve especially if the radioactive material has lodged in pits or scratches left by previous decontaminations.

The higher the temperature, the more active the decontaminating solution needed.

7. Type of decontamination desired.
   a. For example, if a small amount of corrosion cannot be tolerated, then it is obvious that a non-corrosive solution should be chosen.
8. Level of contamination.
   a. If level of contamination is low so that solutions and rags may be discarded, then this factor need not be considered. If level is high, however, then a solution should be chosen for first application that will give high decontamination factors and yet allow easy plutonium recovery. A water wash is one solution that fulfills these requirements.
9. Type of surface.
   a. Consideration should be given to whether the piece to be decontaminated could be dismantled and immersed in a decontamination solution, whether the surface could be soaked with decontamination solution, or whether decontamination has to be carried out by wiping. A less corrosive solution can be used for decontamination by immersion or soaking than for decontamination by wiping.
   b. Can surface stand abrasive action of steel wool, cleansing powders, etc., which enhance the effectiveness of most decontamination solutions?

10. Time available for decontamination.

a. The longer the time available for decontamination, the milder the decontamination solution needed.

SOME GENERAL CONSIDERATIONS

1. Type of surface to be cleaned is more important than type of decontamination solution handy.

2. Major portion of decontamination is usually accomplished in the first 15 minutes of application. In other words, usually the removal of the first solution and application of a new portion for 15 minutes will give better decontamination than if the first solution is left for 30 minutes.

3. The successive alternate application of two selected decontamination solutions usually is much more effective than either alone. Best results obtained if one solution is basic, other acidic. In some cases it appears to be important which of the two solutions is used first.

4. Each of the solutions should be rated in each case by consideration of the 10 factors listed previously before decontamination is attempted.

5. The initial step in each decontamination procedure should be in accordance with factor Number 3 listed under "Discussion." Obviously, use only as many steps in the procedure as is necessary to reduce count to desired level.

6. Decontamination factor (D.F.) is defined as initial activity in c/m divided by final activity in c/m.

The D.F.'s listed are rough estimates since variations in solution contact time, surface history and amount and type of abrasive action applied will change the value considerably.

7. Areas and equipment which will be subject to contamination should, where possible and desirable, be protected with a removable coating such as a plastic or paint. Rubber items have not, however, been successfully protected and subsequently decontaminated with this procedure.

DECONTAMINATION PROCEDURES AND SOLUTIONS

ALUMINUM

A. Recommended procedure.

1. Remove excess liquid by swabbing or transfer to storage tank. Do not allow surface to dry before proceeding to next step.

2. Rinse with water; do not allow to dry.

3. Wet with 10% citric acid solution and scrub with steel wool. Rinse with water.

4. Wet with 10% HNO_3 and scrub with steel wool. Rinse with water.

4a. Or wet with 5% NaOH-1% sodium tartrate-1.5% H_2O_2 and scrub with steel wool. Rinse with water. If surface is kept wet with solution for 15 minutes, the decontamination factor will be ~500. This solution has been used in the plutonium solvent extraction plant at Los Alamos and proved very effective. Decontamination factors of 100-500 were obtained with no visible corrosion.

B. Decontamination solutions in order of decreasing effectiveness.

	Solution	Remarks
1.	5% NaOH-1% sodium tartrate-1.5% H_2O_2	Very little corrosion, D.F. = 100-500
2.	10% HNO_3 + trace $Hg(NO_3)_2$	Corrosive
3.	10% HNO_3	Corrosive, D.F. = 100
4.	5% NaOH-10% sodium citrate	D.F. = 50
5.	0.3M citric acid-0.5M HCl-0.1% synthetic detergent	D.F. = 10
6.	10% citric acid	...
7.	2.5% sodium citrate-0.2% synthetic detergent, pH 7.0	D.F. ~1

BRASS

A. Recommended procedure.

1. Wipe with acetone or alcohol on cleansing tissue.
2. Rub lightly with emery cloth.
3. If wetting can be tolerated, use 5% ammonium citrate or 5% trisodium phosphate washes. Rinse with large amounts of water.

B. Decontamination solutions in order of decreasing effectiveness.

1. 2.5% sodium citrate-0.2% synthetic detergent, pH 7.0
2. 0.3M citric acid-0.5M HCl-0.1% synthetic detergent (corrosive)
3. 5% ammonium citrate
4. 5% trisodium phosphate
5. 1% sodium citrate-5% NaOH
6. Acetone or alcohol on cleansing tissue
7. H_2O

CONCRETE

Recommended procedure for bare concrete.

1. After removal of excess liquid, apply paste of commercial hand cleaner, scrub with stiff brush.

2. Remove paste with damp mop; do not flood with water.

3. If paste is not successful, apply concentrated HCl with ample ventilation for operators. Solution is corrosive.

4. If these are not successful, either coat surface with paint or remove layers of concrete. Flooding or solution decontamination makes activity penetrate deeper. Contamination has been known to seep through 6 inches of unpainted concrete.

GLASS

A. Recommended procedure.

1. For large areas or pieces:

   a. Rinse with water.

   b. Scrub with neutral solution of Na EDTA.

   c. Scrub with cheesecloth and concentrated HNO_3. Rinse with water.

   d. Scrub with 2% ammonium bifluoride. If possible, soak for 30 minutes. Very little corrosion claimed for this solution.

   e. Application of 20% HNO_3-3% HF. This solution very good, but extremely corrosive. Rinse with water.

2. For small pieces of glassware:

   a. Soak in concentrated HNO_3 for several days at room temperature, or several hours at boiling point. Rinse with water.

   b. Soak in 2% ammonium bifluoride for 30 minutes at room temperature. Rinse with water.

   c. Soak in 70% HNO_3-1% HF for 30 minutes. Rinse with water. Corrosive.

   d. Boil in 20% HNO_3-3% HF for 2 hr. Rinse with water. Very corrosive.

B. Decontamination solutions in order of decreasing effectiveness.

1.	20% HNO_3-3% HF	Corrosive, D.F. ~100
2.	70% HNO_3-1% HF	Corrosive
3.	2% ammonium bifluoride	Claimed to be non-corrosive
4.	Aqua regia	
4a.	Standard laboratory cleaning solution of dichromate-sulfuric acid	
5.	Concentrated HNO_3	Decontamination rate increases with increasing temperature
6.	1M tartaric acid	
7.	1M oxalic acid	
8.	a. 1% Na citrate-5% NaOH	About equally effective
	b. 2.5% Na citrate-1% synthetic detergent	
	c. 0.3M citric acid-0.1% synthetic detergent-0.5M HCl	D.F. ~4

9. 1M citric acid

10. 1M HNO_3

11. Versene with 1% synthetic detergent

12. 1M sodium carbonate

13. 1M sodium hydroxide

Increase in temperature will increase D.F. For the first 3 solutions an increase in temperature will increase corrosion.

IRON AND MILD STEEL

A. Recommended procedure.

1. Rinse with water.

2. Scrub with 10% citric acid-5% synthetic detergent; rinse with water.

3. Scrub with 0.3M citric acid-0.1% synthetic detergent-0.5M HCl. Rinse with water.

4. Scrub briefly with 6N HNO_3. Rinse with water.

5. Repeat number 4 as many times as necessary.

B. Decontamination solutions in order of decreasing effectiveness.

1.	6N HCL	Very corrosive
2.	6N HNO_3	Very corrosive
3.	10% citric acid-5% synthetic detergent	Mildly corrosive
4.	0.3M citric acid-0.5M HCl-0.1% synthetic detergent	D.F. ~20
5.	2.5% Na citrate-0.2% synthetic detergent	
6.	1% Na citrate-5% NaOH	
7.	Neutral solution of Na EDTA	

LUCITE, PLEXIGLASS, AND OTHER ACRYLIC PLASTICS

Use procedure and solutions as for glass. It should be remembered, however, that high HNO_3 concentrations will severely mar the surface of the plastic.

MONEL

A. Recommended procedure.

1. Rinse with water.

2. Scrub with 0.3M citric acid-0.5M HCl-0.1% synthetic detergent. Rinse with water.

3. Repeat if necessary.

B. Decontamination solutions in order of decreasing effectiveness.

1. 0.3M citric acid-0.5M HCl-0.1% synthetic detergent, D.F. ~10.

2. 1% Na citrate-5% NaOH.

3. Neutral solution of Na EDTA.

4. 2.5% Na citrate-0.2% synthetic detergent.

Nitric acid should be good but very corrosive; however, no data could be found in literature.

PAINT

A. Recommended procedure.

1. Wipe up excess solution, keeping area wet if possible.

2. Scrub with Na EDTA plus 2% synthetic detergent. Rinse with water.

3. Cover with wet Na EDTA rags and allow to soak for 1 hour. Remove rags and rinse with water. D.F.'s of 10-1000 have been obtained at Los Alamos with this procedure.

4. Scrub with 5% ammonium citrate, using steel wool as abrasive. Rinse with water.

5. If contamination still remains:

a. If there is no "swipe" count, apply new coat of paint.

b. If "swipe" count is present, apply paint remover.

B. Decontamination solutions in order of decreasing effectiveness.

1. Paint remover

2. Na EDTA with 5% synthetic detergent

3. a. 5% Na EDTA-1% synthetic detergent
   b. 0.3M citrate-0.5M HCl-0.1% synthetic detergent
   c. 2.5% Na citrate-0.2% synthetic detergent
   d. Trisodium phosphate, saturated solution
   e. 5% ammonium citrate
   f. 2% ammonium bifluoride
   g. 30 to 50% HNO_3
   h. 3 to 8N HCl

   (About equally effective; D.F. ~2 to 10)

PLASTICS OTHER THAN ACRYLIC BASE PLASTICS

Teflon, saran, polyethylene, etc., use procedures and solutions listed for glass or stainless steel.

PORCELAIN

A. Recommended procedure.

1. Rinse with H_2O.

2. Boil in saturated ammonium carbonate solution for 30 minutes.

3. Soak in 5% ammonium bifluoride solution for 30 minutes. Rinse with water.

B. Decontamination solutions listed in order of decreasing effectiveness.

1. Ammonium bifluoride
2. 20% HNO_3-3% HF
3. Ammonium carbonate
4. Water

RUBBER

Generally difficult to decontaminate.

A. Recommended procedure.

1. Wash with water.
2. Scrub with any one of the solutions listed in Part B.

B. Decontamination solutions listed in order of decreasing effectiveness.

1. 5% Na EDTA-1% synthetic detergent
2. Na EDTA-5% synthetic detergent
3. 1% Na citrate-5% NaOH
4. 2.5% Na citrate-0.2% synthetic detergent
5. 0.3M citric acid-0.1% synthetic detergent-0.5M HCl

SKIN

Immediate use of a synthetic detergent-sequestrant mixture has been reported to give better decontamination than if the use of the mixture is preceded by soap and water wash. However, no temperature was given for the water used and if the water was warm enough to cause the pores to open, the decontamination solution, normally used cold, would have a difficult time removing the activity. At Los Alamos steps

one and two of the recommended procedure normally give sufficient decontamination.

For decontamination of hair, omit the $KMnO_4$ treatment.

A. Recommended procedure.

1. Lather with liquid soap, using cold water, rinse thoroughly.

2. If count still remains, wash with synthetic detergent and sequestrant in a ratio of 1:2. Rinse with water.

a. Sequestrants such as:

1. Na EDTA

2. Citric acid

3. Sodium citrate

4. Sodium tartrate

5. Sodium phosphates

b. Do not use oxalates!

C.P. Cleaner, manufactured by Enley Products, Inc., is also satisfactory. Apply as label directs.

3. If count still remains, scrub with $KMnO_4$ crystals wet with just enough water to make thick paste. Rinse thoroughly. Repeat 3 times. Remove color with a 4% $NaHSO_3$ solution. (Use only as a last resort.)

4. Apply TiO_2 paste and rub thoroughly. Remove by swabbing. Rinse thoroughly with water.

B. Decontamination solutions in order of decreasing effectiveness.

1. TiO_2 paste (expensive)

2. $KMnO_4$ paste; color removed with 4% $NaHSO_3$

3. Synthetic detergent - sequestrant

4. C.P. Cleaner or similar hand cleaner

5. 3% trisodium nitrolotriacetate - synthetic detergent

6. 3% Na citrate, pH 7.0

7. 3% Na acetate, pH 2.0

8. 3% Na tartrate, pH 7.0

9. 3% Na lactate, pH 7.0

10. 3% glycine

11. 3% Na acetate, pH 7.0

12. Water with liquid soap

13. Isotonic saline solution

STAINLESS STEEL

A. Recommended procedure.

1. Remove excess liquid, flush with water, do not allow to dry.

2. Scrub with 30% HNO_3.

3. Scrub with 20% NaOH-10% Na tartrate, 30 minutes.

4. 10% oxalic acid, 1 hour contact time. Wash with water.

5a. If necessary, use 3% HF-20% HNO_3 at room temperature. Solution most effective if surface is kept wet for 15 minutes. This solution has been used at Los Alamos with excellent results. Decontamination factors of > 1000 were obtained in every case. This solution has a corrosion rate of < 0.01 inch per hour, does not pit, but will attack welds which have included flux. Good welds are as resistant as the stainless steel plate.

b. Scrubbing with 6 to 12N HCl is very good, but extremely corrosive. HCl pits metal, making next decontamination more difficult.

6. Wash with water.

7. If count remains, repeat 5a. If count is gone, wash with concentrated HNO_3 to passivate surface.

B. Decontamination solutions in order of decreasing effectiveness.

1. 6-12N HCl, corrosive with pitting of surface

2a. 3% HF-20% HNO_3, corrosive without pitting of surface. D.F. ~1000 at 15 minutes contact time. (Patent applied for by Bennet, ORNL)

b. Alternate use of 2.5% sodium tartrate-10% NaOH-1.5% H_2O_2 and 16N HNO_3

3. 3% HF-5% HNO_3, corrosive, more so than with 20% HNO_3

4a. Boiled 20 minutes in solution Turco*

b. 20% HNO_3-20% Na dichromate

5a. Boiled 20 minutes in 0.25 H_3PO_4

b. 20% oxalic acid at 80°C, 30 minutes contact time

6a. 10% oxalic at 80°C, 30 minutes contact time

b. 0.075M H_2O_2-0.5M H_2SO_4

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*Turco No. 4182, a special soap manufactured by Turco Products, Inc.

7a.	0.5M citric acid-0.5M HCl-0.1% synthetic detergent	About equally effective D.F. = 5 to 20 at 15 min. contact time
b.	Boiled 30 minutes in 1M HNO_3-0.003M periodic acid	
c.	Boiled 2 hours in 2M NaOH	
d.	Boiled 30 minutes in 2M citric acid	
e.	1% Na citrate-5% NaOH	
f.	2.5% Na citrate-0.2% synthetic detergent	
g.	1M oxalic acid-0.1M NaF	
h.	20% NaOH-10% Na tartrate	
8.	2% ammonium bifluoride	
9.	Ammonium fluosilicate	
10.	Boiled 20 minutes in Na_2CO_3 solution	
11.	60% HNO_3, room temperature	
12.	High pressure steam flush	
13.	Water	

TILE

If tile is used in an area which can become contaminated, the surface should be protected by paint.

If bare tile does become contaminated, clean first with paste of hand cleaner. Flooding or liquid decontamination merely spreads contamination to the cracks between tiles, which are extremely difficult to clean up. In such a case, removal of the tile or application of a coat of paint are the only remedies.

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