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J. F. Nesbitt
S. C. Slate
L. K. Fetrow

December 1980

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

Battelle

PNL-3514

PACIFIC NORTHWEST LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
Under Contract DE-AC06-76RLO 1830

Printed in the United States of America
Available from
National Technical Information Service
United States Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151

Price: Printed Copy $_______*; Microfiche $3.00

*Pages	NTIS Selling Price
001-025	$4.00
026-050	$4.50
051-075	$5.25
076-100	$6.00
101-125	$6.50
126-150	$7.25
151-175	$8.00
176-200	$9.00
201-225	$9.25
226-250	$9.50
251-275	$10.75
276-300	$11.00

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SUMMARY

This report presents evaluations of several methods for the in-process decontamination of metallic canisters containing any one of a number of solidified high-level waste (HLW) forms. The use of steam-water, steam, abrasive blasting, electropolishing, liquid honing, vibratory finishing and soaking have been tested or evaluated as potential techniques to decontaminate the outer surfaces of HLW canisters. Either these techniques have been tested or available literature has been examined to assess their applicability to the decontamination of HLW canisters.

Electropolishinq has been found to be the most thorough method to remove radionuclides and other foreign material that may be deposited on or in the outer surface of a canister during any of the HLW processes. Steam or steam-water spraying techniques may be adequate for some applications but fail to remove all contaminated forms that could be present in some of the HLW processes. Liquid honing and abrasive blasting remove contamination and foreign material very quickly and effectively from small areas and components although these blasting techniques tend to disperse the material removed from the cleaned surfaces.

Vibratory finishing is very capable of removing the bulk of contamination and foreign matter from a variety of materials. However, special vibratory finishing equipment would have to be designed and adapted for a remote process. Soaking techniques take long periods of time and may not remove all of the smearable contamination. If soaking involves pickling baths that use corrosive agents, these agents may cause erosion of grain boundaries that results in rough surfaces.

The report also includes a preconceptual design of a canister decontamination system using electropolishing along with some of the requirements of the system.

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INTRODUCTION

In most concepts for managing high-level wastes (HLW) from the nuclear-fuel cycle, the wastes in the form of spent fuel or solidified wastes are placed in metal containers or canisters for storage, transportation and disposal. These canisters serve the following purposes:

- to isolate and to contain the radioactive waste material
- to provide a barrier between the waste form and the environment
- to provide structural integrity for the waste form
- to provide the means to handle and transport the waste form.

One of the waste forms that has been under research and development for a number of years is glass. In the glass waste-form processes, as in most of the other waste-form processes, the outer surfaces of a canister may be contacted by the waste before and during the canister-filling step, or at least by air or gases, that could contain radioactive elements. Also, the outer surfaces of the canisters containing glass are heavily oxidized as a result of the high temperatures required to process the glass waste forms. This oxidation may trap or contain radioactive nuclides on the outer surfaces, making their removal more difficult.

Because this contamination can fall or flake off and will be transferred to other surfaces to which the canister is brought into contact as it is process or transported, it is a very important step in any waste-form process to decontaminate and clean all of the outer canister surfaces as soon as possible after the canister is filled and sealed.

To date, various methods to decontaminate canisters have been researched and developed. These can be classified in the general categories of spraying, soaking, and scrubbing. For years, the Pacific Northwest Laboratory (PNL) has been researching methods to decontaminate equipment and facilities that have been contaminated with radionuclides (Ayres 1971).

The best process for a particular application is dependent on several factors, such as the type and amount of contaminants present; the configuration of and the materials used in the object to be decontaminated; and the accessibility, size, and mass of the object as well as the time available to perform the decontamination.

This report includes descriptions of tests, data, and information pertinent to the decontamination of canisters similar to those identified by Simonen and Slate (1979) and which contain HLW applicable to both defense-waste processes identified by Stone, Goforth and Smith (1979) and commerical-waste processes identified by Treat (1980).

The report is divided into five major sections: spraying, soaking, scrubbing, an evaluation of all decontamination processes investigated, and a preconceptual design of a decontamination station using an electropolishing technique.

CONCLUSIONS

A number of cleaning techniques have been developed and used for the removal of radionuclides from various materials and items although only a limited number of these techniques are or may be adequate for and applicable to the in-process decontamination of metallic canisters containing nuclear HLW. Electropolishing appears to be the most effective and developed decontamination technique or method for removal of radionuclides and other foreign material from outer surfaces of a canister in a HLW process. Electropolishing:

- removes all contaminants and foreign material, such as oxides, from metallic surfaces
- removes layers of the metallic surface evenly and smoothly without preferentially attacking ground boundaries and other microstructural features
- controls and limits the spread of the contaminant removed from the item being cleaned
- functions as a system applicable to fully remote operations and maintenance
- generated secondary wastes of such a volume and compositions that these wastes can be incorporated into the overall HLW process.

Other decontamination techniques, such as steam or steam-water spraying, are fairly simple, have been found to be adequate for the quick removal of canister contamination in some HLW processes, and are compatible with remote operations and maintenance requirements. However, tests have shown that these techniques are not effective on contaminants strongly attached to the canister surface or that may have penetrated the surface. Also, these techniques are limited in their ability to remove foreign material, such as films and oxide coating, that may contain radionuclides.

Other decontamination methods summarized below either do not have the capability to reduce contamination levels as required in the available time or have other attributes that may make them less desirable for this particular application. These are listed sequentially in terms of their positive attributes:

- Abrasive Blasting
  - --is effective and fast
  - --removes contaminants, films and foreign material, as well as some base material
  - --has tendency to roughen surfaces
  - --requires additional development
  - --could generate large amounts of secondary wastes, although these may be incorporated in the HLW process.
- Liquid Honing
  - --effectively and quickly cleans small areas or components
  - --removes contaminants, films, and foreign material, as well as some base metal
  - --decontaminates nonmetallic surfaces
  - --may be time-consuming to decontaminate whole canister
  - --has tendency to roughen surfaces
  - --requires mechanical equipment in a remote zone
  - --could generate large amounts of secondary waste.
- Vibratory Finishing
  - --effectively and fairly quickly cleans small components
  - --removes contaminants, films and foreign material
  - --decontaminates nonmetallic surfaces
  - --requires specially designed mechanical equipment in a remote zone
  - --could generate large amounts of secondary wastes if a ceramic medium is used.
- Soaking Baths
  - --may require long periods of time to be effective
  - --decontamination factor may be small

--water offers limited removal of contaminants, films and foreign material unless special chemicals are used

--has special chemicals that may contain corrosive agents that will attack more than the item to be decontaminated

--has tendency to roughen surfaces

--requires little mechanical equipment in a remote zone.

SPRAYING

Spraying uses a high-pressure liquid or mixtures of liquids and solids to clean the surface of an object. The sprayed medium can be either water, steam, ice, an abrasive, or combinations of these. Two techniques investigated at PNL beginning in 1975 are steam and water spraying and liquid honing. In addition, the French have used a water spraying technique to decontaminate canisters filled with vitrified glass. Finally, researchers at the Savannah River Laboratory (SRL) have investigated abrasive blasting.

STEAM AND WATER SPRAYING

Under the sponsorship of the U.S. Energy Research and Development Administration (ERDA), considerable experimental efforts and tests were conducted at PNL using steam and water spray cycles to remove nuclear contaminants from metal surfaces and containers. In 1975, full-sized canisters containing high-level wastes were used in decontamination demonstrations. Tests were performed on seven different canisters that were filled with HLW during the Waste Solidification Engineering Prototype (WSEP) Program conducted by PNL.

PC-6 Canister Decontamination

The canister used for the first study was a nominal 8-in. dia x 8-ft-long 304L stainless steel (SS) canister that was filled with radioactive calcine in the WSEP program during the Sixth Pot Calcination Run (PC-6) conducted in August 1967. (The pot calcination process is similar to the in-can melting process.) The canister originally contained 1.3 mCi of radioactive material. The canister was stored in water from the date of filling until October 1969, then placed in air storage until December 1973, when it was again placed in water storage where it remained until these studies were started. Contamination of the canister occurred when it was filled with glass, and the contamination levels possibly modified some during storage.

All decontamination of the canister was performed remotely. The first method was to move the canister up and down through a fixed-position spray ring. The second method employed a commercial hydraulic jet cleaner (aqua-jet). The spray ring was connected to medium-pressure systems (90-psig process steam and 75-psig process water). The general decontamination procedure using the spray ring is shown below. Decontamination efforts with the aqua-jet were performed by a similar method.

General Procedure for Canister Decontamination Using Steam and Water Spray Ring (One Cycle)

A. Steam Spray
   1. Position canister 6 in. above and aligned with the center of spray ring.
   2. Turn on steam to full flow and allow the condensate to flush out.
   3. Lower the canister into the spray ring at a rate of 16 ft/min until top of canister is 6 in. below the spray ring.
   4. Raise the canister at a rate of 16 ft/min until canister is 6 in. above spray ring.
   5. Repeat Steps 3 and 4 for six complete cycles.

B. Water Spray
   1. Turn off the steam service to spray ring and turn on high-pressure water service.
   2. Lower the canister into the spray ring at a rate of 4 ft/min until top of canister is 6 in. below the spray ring.
   3. Raise the canister at a rate of 4 ft/min until canister is 6 in. above spray ring.
   4. Repeat Steps 2 and 3.

C. Repeat A above.

D. Repeat B above.

E. Obtain smear sample of canister.

Smear surveys of the canister were 1,290 counts/min/100 cm^2 (1.77×10^4 dis/min/100 cm^2) prior to start of the decontamination procedure. After five steam-water cycles (250 gal of water), the smear samples surveys were reduced to 97 counts/min/100 cm^2 (7.2×10^2 dis/min/100 cm^2). The canister was then recontaminated to 485 counts/min/100 cm^2 and subjected to five aqua-jet decontamination cycles. After these five cycles, the smear readings were down to about 95 counts/min/100 cm^2. Complete data on the smear samples are shown in Table 1.

Both water spray methods were found to be about equally effective in removing the contamination. Both old and new contamination was removed easily. The long period of time that the canister had been stored under water may have tended to soften the oxide layer. As indicated in Table 1, both of the final smear levels are higher than the previous ones. No explanation was given for this in the original literature.

Additional Canister Decontamination

Six other canisters filled in the WSEP program were used for additional decontamination tests. A summary of their histories is given in Table 2. As with the PC-6 canister, their contamination at the time of the tests was the result of the glass-filling process as well as long-term storage.

Four of the canisters (S-11, PG-3, PC-5 and PC-7) were cleaned using both steam and water at 100 psi and 70 psi, respectively, and the procedure described on page 8. This procedure as noted constitutes one cycle, and five such cycles were completed on each of the four canisters. The other two canisters were decontaminated with only steam to determine if it was possible to achieve similar cleaning with a simpler and shorter procedure. This procedure is listed below. Table 3 compares these two decontamination procedures.

Decontamination Procedure (Steam Only)

- Steam Spray (100-lb Pressure)
  1. Position canister 6 in. above and align with the center of spray ring.
  2. Turn on steam to full flow and allow the condensate to flush out.
  3. Lower the canister into the spray ring at a rate of 16 ft/min until top of canister is 6 in. below the spray ring.

TABLE 1. PC-6 Canister Decontamination Data

Sample Number	Gamma Energy Analysis										Geiger Müller Survey[e]			
	Ce^{141}	Ce^{144}	Cs^{134}	Cs^{137}	Nb^{95}	Ru^{103}	Ru^{106}	Zr^{95}	Eu^{154}	Eu^{155}	Total dis/min[d]	dis/min/100 cm^2	Total counts/min	counts/min/100 cm^2
S-9[a]	<1.50 x 10^3	3.79 x 10^5	<9.90 x 10^2	9.13 x 10^4	<1.11 x 10^2	<7.76 x 10^1	2.65 x 10^4	<7.77 x 10^3	1.70 x 10^4	3.32 x 10^4	5.47 x 10^5	1.77 x 10^4	40,000	1,291.67
S-10	<1.06 x 10^3	1.29 x 10^5	<9.66 x 10^2	5.55 x 10^4	<2.22 x 10^2	<3.23 x 10^2	<7.62 x 10^3	<3.47 x 10^3	7.00 x 10^3	1.56 x 10^4	2.07 x 10^5	6.69 x 10^3	17,500	565.02
S-11	<3.36 x 10^1	6.14 x 10^4	<5.80 x 10^2	3.07 x 10^4	<1.12 x 10^2	<5.37 x 10^2	1.26 x 10^4	<5.98 x 10^2	8.80 x 10^3	1.11 x 10^4	1.12 x 10^5	3.62 x 10^3	9,500	306.77
S-12	<8.40 x 10^1	2.06 x 10^4	<3.12 x 10^2	7.80 x 10^3	<1.11 x 10^2	<7.76 x 10^1	<5.97 x 10^3	<9.56 x 10^2	1.10 x 10^3	2.60 x 10^3	3.21 x 10^4	1.04 x 10^3	3,000	96.88
S-13	<3.02 x 10^2	<6.51 x 10^3	<5.55 x 10^2	5.81 x 10^3	1.11 x 10^2	<7.76 x 10^1	<2.82 x 10^3	3.59 x 10^2	--[c]	1.20 x 10^3	7.01 x 10^3	2.26 x 10^2	1,800	58,13
S-14	<2.18 x 10^2	1.41 x 10^4	<1.45 x 10^2	6.21 x 10^3	<1.11 x 10^2	<1.23 x 10^2	3.15 x 10^3	<2.29 x 10^2	--	2.00 x 10^3	2.23 x 10^4	7.20 x 10^2	3,000	96.88
S-15[b]	<3.36 x 10^1	1.15 x 10^5	<6.04 x 10^2	4.78 x 10^4	1.11 x 10^2	<7.76 x 10^1	<1.33 x 10^4	--	1.65 x 10^4	1.81 x 10^4	1.97 x 10^5	6.37 x 10^3	15,000	484.38
S-16	9.24 x 10^2	1.14 x 10^5	<7.97 x 10^2	3.77 x 10^4	<9.98 x 10^2	<1.20 x 10^3	9.12 x 10^3	<5.50 x 10^3	8.20 x 10^3	1.56 x 10^4	1.75 x 10^5	5.67 x 10^3	7,000	226.04
S-17	<1.34 x 10^2	<2.66 x 10^4	<4.35 x 10^2	4.71 x 10^4	<1.11 x 10^2	7.76 x 10^1	6.13 x 10^3	<3.35 x 10^3	2.90 x 10^2	4.40 x 10^3	5.44 x 10^4	1.76 x 10^3	5,000	161.46
S-18	<3.36 x 10^1	6.58 x 10^3	<1.45 x 10^2	1.76 x 10^3	<1.11 x 10^2	<7.76 x 10^1	9.94 x 10^2	2.39 x 10^2	--	6.00 x 10^2	7.18 x 10^3	2.32 x 10^2	1,400	45.21
S-19	<5.04 x 10^1	1.03 x 10^4	<1.45 x 10^2	5.52 x 10^3	1.11 x 10^2	7.76 x 10^1	1.99 x 10^3	7.17 x 10^2	--	1.30 x 10^3	1.17 x 10^4	5.53 x 10^2	2,900	93.65

(a) S-9 through S-14 are samples from spray-ring procedure.
(b) S-15 through S-19 are samples from aqua-jet procedure.
(c) Dash indicates not detectable.
(d) Isotopes with < signs not included in total disintegrations/min, dis/min.
(e) A Geiger-Mueller survey meter was used to obtain the counts per minute numbers.

TABLE 2. Summary of Canister Histories

Canister Specifics	Canister Number SS-11	 PG-3	 PC-5	 PC-7	 SS-6	 SS-5
Canister Material	304L	Mild Steel	304L	304L	304L	304L
Fill Date	3/70	1/68	7/67	8/70	11/68	10/68
Radioactivity Level at Fill, mCi	3.1	0.6	1.15	1.4	3.0	2.5
Total Heat Ratio, kW, at Fill Date	10.5	3.3	4.3	5.6	10.0	8.2
Heat-Rate Density, W/ℓ at Fill Date	175	95	78	93	168	127
Centerline Temperature, ^{o}C, at Fill Date	754	655	725	1012	767	633
Stored in Water	10/70- 5/71	7/68- 1/70	2/68- 4/70	1/71- 10/71	5/69- 10/69	4/69- 9/69
Stored in Air Since	5/71-	1/70-	4/70-	10/71-	10/69-	9/69-

4. Raise the canister at a rate of 16 ft/min until canister is 6 in. above the spray ring.
5. Steps 3 and 4 constitute a cycle. Repeat as needed.

The test conditions and results for the four canisters cleaned with steam and water and the two decontaminated with only steam are summarized in Table 4.

These tests indicate that the major portion of the contamination was removed early in the procedure. The canisters that had been stored for a number of years exhibited an outer layer of material that varied from a scale to a powder. This layer was mostly removed in the early decontamination cycles although a rather tenacious film remained. As indicated in Table 4, the steam cycle cleaned the canisters (SS-6 and SS-5) nearly as well as the steam and water cycle although the cycle was five times shorter in duration and required fewer services.

TABLE 3. Comparisons of Procedures

Procedures	Time Needed for Decontamination, min	Gallons Water Used	Pounds Steam Used
Steam and Water	140	310	40
Steam	30	0	20

After two of the six canisters had been decontaminated, they were recontaminated with actual high-level waste corresponding to a reactor burnup of about 54,000 MWD/MTU and an age of 2 yr. The waste was applied in two ways to simulate an "easy-" and a "worst-case" contamination. The "easy-case" used dry-waste calcine sprinkled on the canister; the "worst-case" involved a solution of dissolved calcine in nitric and hydrofluoric acids applied to the canister and allowed to dry. Table 5 summarizes the results of the steam-water decontamination tests on the two canisters recontaminated with HLW.

The analysis of the data on the doped canisters indicates that the dry calcine (Canister SS-5) was easily removed to very near the level prior to recontamination. However, the "worst-case" acid solution (Canister PC-5) was not easily removed, and the canister remained at a very high level of contamination. This high level is considered to be due to the absorption/penetration of the acid solution into the oxide on the surface of the canister.

These tests showed the ability of steam-water or steam sprays to remove surface contamination, but both techniques do not have the capability of removing all fixed surface contaminants or those contaminants that may have penetrated the surface.

Canister Material Decontamination Tests

Additional steam-water decontamination tests were performed on small 304L SS canisters and a flat Inconel bar. The canisters were 2.25 in. OD and 11 in. long; the flat bar was 1/4 in. thick, 2 in. wide and 11 in. long. These test pieces were exposed to conditions and contaminants that could be present while a canister is being filled with either calcine or glass containing high-level wastes. Basically, the test components were heated at 1050^{o}C for 1 h, cooled,

TABLE 4. Smear Tests Radiation Levels on WSEP Canisters

	dis/min/100 cm^2							
Canister	Initial	1st Cycle	2nd Cycle	3rd Cycle	4th Cycle	5th Cycle	Background	Decontamination Factor
S-11	2.80×10^6	8.63×10^4	4.39×10^4	4.43×10^4	1.04×10^4	3.93×10^3	1.86×10^3	712
PG-3	8.26×10^5			2.31×10^4		5.67×10^3	T(a)	147
PC-5	4.76×10^6			7.26×10^3		5.01×10^3	4.36×10^2	950
PC-7	3.45×10^6			2.65×10^4		9.97×10^3	2.36×10^2	346
SS-6	3.65×10^6	1.40×10^4(b)	8.83×10^3(c)				3.86×10^2	413
SS-5	3.13×10^6	2.4×10^4(b)	2.25×10^4(c)				1.41×10^3	139

(a) T = Trace < 100 dpm/100 cm^2.
(b) First test cycle consisted of 18 steam cycles.
(c) Second test cycle consisted of 12 additional steam cycles.

TABLE 5. Doped Canister Decontamination Data

Canister	dis/min 100 cm^2 Prior to Decontamination[a]	After Three Steam and Water Cycles	After Five Steam and Water Cycles	Background
SS-5	2.25×10^4	1.07×10^5	3.48×10^4	2.47×10^4
PC-5	3.09×10^3	3.17×10^6	1.27×10^6	6.04×10^2

(a) No smear was taken after contamination with HLW due to excessive radiation levels.

then exposed to the contaminants indicated in Table 6 and placed in a furnace at 1050°C for another hour.

These items were passed through a spray ring using 70 and 100 psig steam and water pressures, respectively. The procedure used for these tests was that described on page 8 except the pass-through rates were lower. As described, the procedure constitutes one cycle; 12 such cycles were used for each of these items. A summary of the test results is given in Table 7.

Several times during each of these tests, the level of contamination increased at the subsequent cycle. This increase was apparently due to incomplete decontamination and the exposure of new layers of contaminants which was caused by the removal of preceding layers. The decontamination factor varied from 0.2 to 1050, which indicates that steam-water spraying does not have the capability to remove all surface contaminants that may be present on canisters used in a glass waste-form process.

TABLE 6. Items and Types of Contamination

Contaminated Items	Type of Contamination
Canister #1	Old calcine dissolved in HNO_3
Canister #2	Crushed glass (5.74 g/50 ml H_2O)
Canister #3	Calcine in H_2O (0.74 g/50 ml H_2O)
Flat Bar	Calcine in H_2O (0.74 g/50 ml H_2O)

TABLE 7. Summary of Canister Material Smear Activity

Steam-Water Cycles	Counts/min/100 cm^2			
	Canister #1	Canister #2	Canister #3	Flat Bar
	47,000(a)	7,388,000(a)	6,044,000(a)	9,767,000(a)
1	51,500	1,041,000	1,869,400	837,000
2	672,000	353,000	582,100	348,800
3	10,500	430,000	353,700	125,600
4	6,900	403,000	369,400	279,000
5	20,100	884,300	347,000	176,700
6	11,200	284,000	107,000	511,600
7	15,700	47,700	83,900	227,900
8	9,800	71,600	100,700	167,400
9	42,500	120,900	134,300	325,600
10	24,600	83,300	67,200	83,700
11	4,700	16,800	56,000	162,800
12	223,800	35,800	60,500	9,300
Background:	200-4,000	400-4,500	400-1,500	

(a) Initial activity.

Marcoule Vitrification Plant (AVM) Canister Decontamination System

The operation of the continuous vitrification process in the Marcoule Vitrification Plant (AVM)(a) was initiated in 1978. The plant produces a container loaded with approximately 360 kg of vitrified glass each day of operation. At planned intervals, the glass is poured into the metallic containers or canisters which are 0.5 m in dia and 1 m high. Several hours after the canister is filled, it is transported to a weld area where it is sealed. Usually the next day, the canister is lowered into a tank located in a decontamination cell between the process and the storage cells where it is washed

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(a) Information source for this section: Casey 1978.

from top to bottom by a traveling spray ring. Each canister is washed with 450 ℓ of pure water at high pressure (200 bars or 2,900 psi) for 3 min. The final activity of the wash water has been shown to be less than 2×10^{-4} μCi/mℓ. Also, no activity has been detected in the air stream around the canister during the subsequent transfer to the storage area.

LIQUID HONING

Liquid honing[a] is a process used in the metal-finishing industry to remove burrs and machine marks, to relieve stress concentrations, and to clean surfaces prior to electroplating. Liquid honing combines a liquid containing 30-vol% abrasive particles with a stream of air at 90 psi. The air propels the abrasive and liquid mixture against the surface to be cleaned. During operation, the liquid and air are directed with a hand-held gun, which can be designed for specific applications. For example, a special gun can be used to facilitate cleaning of holes that have a large depth-to-diameter ratio. The thickness of metal removed during a typical decontamination cycle using liquid honing techniques equals approximately 0.01 mm. A typical honing system is shown schematically in Figure 1.

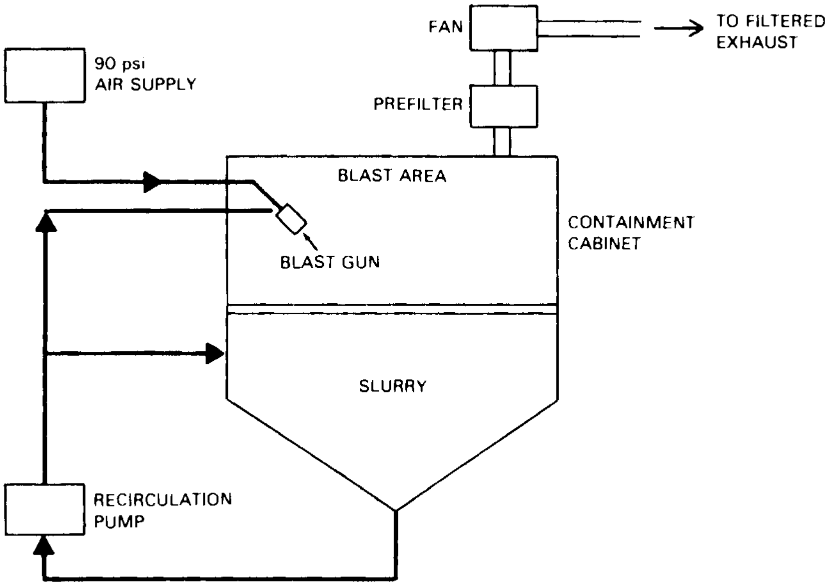

FIGURE 1. Schematic Drawing of a Liquid Honing System

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[a] Information source for this section: Arrowsmith and Allen (1978).

Liquid honing has been tested on a variety of nuclear reactor components contaminated with beta/gamma fission products. For example, the contamination level of a ball-channel inspection tool constructed of mild steel with inflatable rubber sides (Figure 2) was reduced from 900 to 80 mrem/h after 5 min of processing. A reactor process cap was decontaminated from 100 to <1 mrem/h after 3 min of processing (Figure 3). In these examples, liquid honing not only cleaned exterior metal surfaces but also decontaminated rubber surfaces as well as threaded areas, and deep into bolt holes. A summary of items decontaminated using liquid honing is presented in Table 8.

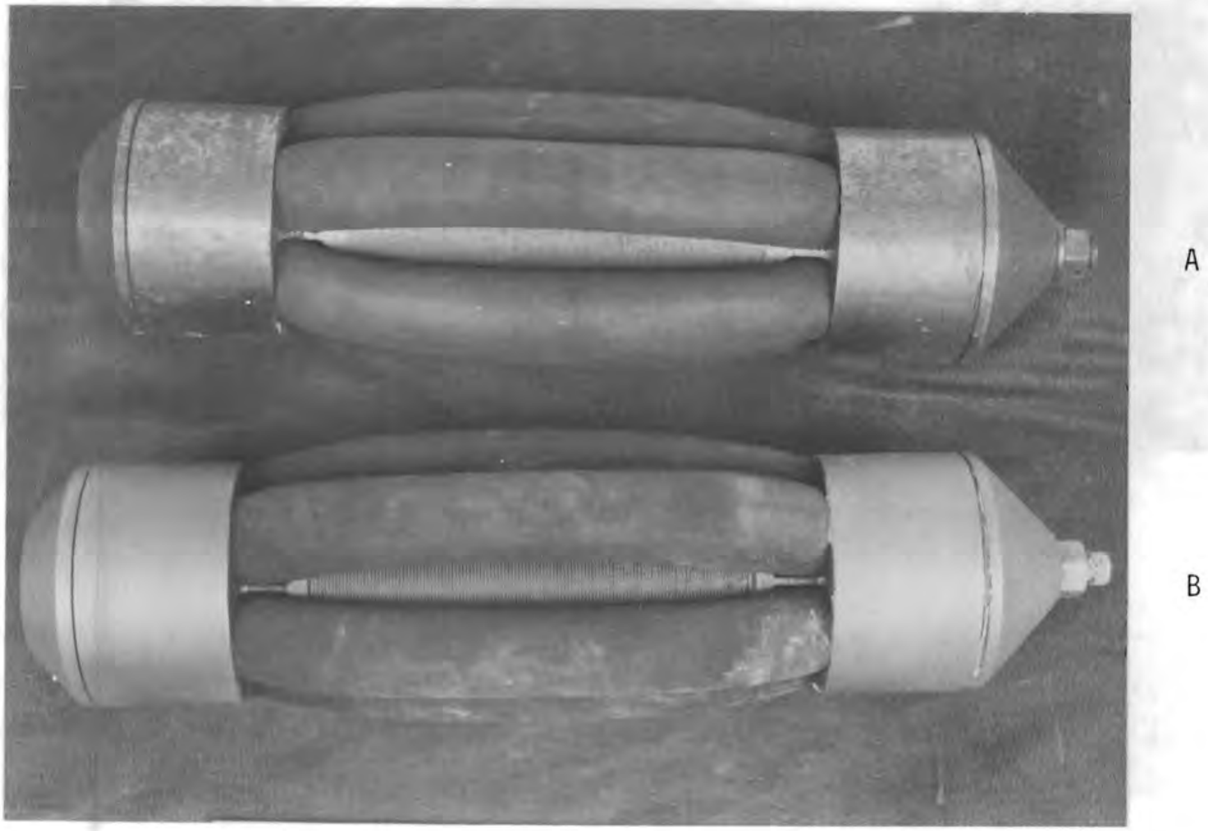

FIGURE 2. Stainless Steel Ball Inspection Tool: (A) Contaminated With Beta/Gamma Fission Product and (B) After Decontamination Using the Liquid Honing System

FIGURE 3. Reactor-Process Tube End-Caps After Decontamination by Liquid Honing

These tests show that liquid honing has the capability to remove quickly and effectively contamination from small items made of various materials and which have a wide range of configurations. A fully contained system to limit spread of the contaminants is required.

ABRASIVE BLASTING

Abrasive blasting or spraying techniques have been evaluated at SRL (Rankin 1980) as alternatives to chemical processing for decontamination of stainless steel canisters containing waste glass. Processes investigated have been dry abrasive blasting, wet (slurry) abrasive blasting, high-pressure water plus frit abrasive blasting, and high-pressure water blasting.

TABLE 8. Representative Items Decontaminated by Liquid Honing

Decontaminated Item	Radiation Levels Initial, mrem/h	Radiation Levels Final, mrem/h	Time
Ball-Channel Inspection Tool	900	70	5 min
Wheel Pulling Tool	3	0.1	5 min
Manipulator Tongs	50	0.4	5 min
Process-Tube End Cap	60	1	3 min
Process-Tube End Cap	100	5	3 min
Process-Tube Insert	200	5	2 min
Process-Tube Insert	200	26	45 sec
Process-Tube Insert	300	20	1.5 min
Reactor Fuel Spacer	300	20	2 min
Reactor Fuel Spacer	330	17	2 min

Laboratory-scale tests indicate that all processes, except high-pressure water blasting, would remove an oxide film from type-304L stainless steel similar to that expected on the outside of a HLW canister. Other laboratory-scale tests have shown that all processes, except high-pressure water blasting, would remove baked-on alpha contamination and alpha, beta and gamma contamination picked up from the vapor space inside a glass melter during production of waste glass containing actual radioactive sludge.

In addition, the major results of larger-scale tests carried out at equipment manufacturers' locations show that:

- all processes using glass frit as an abrasive would remove oxide from 304L stainless steel and would, therefore, be expected to remove radioactive contamination
- estimates of consumption rates made in laboratory-scale tests were confirmed.

Further development of larger-scale equipment for spot decontamination techniques is planned at SRL. This equipment will consist of wet (slurry), high-pressure water rinse, and high-pressure water plus slurry. The same glass frit used in the HLW process for the manufacturing of waste glass will be used as the abrasive in the decontamination techniques. All solid wastes from the decontamination process--the waste glass and the contaminated oxide removed from the 304L stainless steel--will be incorporated in the HLW process at the melter as a slurry. All liquid waste from the decontamination process will be evaporated. Provisions to decontaminate all of the canister's outer surfaces and to limit and control the spread of the contaminants are areas of concern.

SOAKING

Soaking is a very efficient and widely accepted method for decontamination. The contaminated part is placed in a tank of solvent where contaminants are removed principally by chemical action. The part is then flushed with clean water. Physical cleaning forces, such as agitation and spray, can be added to enhance the removal process. Two techniques useful in varying degrees for decontaminating contaminated materials that could be used in the HLW canisters are a mini-wet basin and a specific modified soaking adaptation called electropolishing.

ELECTROPOLISHING

Electropolishing is the opposite of electroplating. Instead of electrically adding a thin layer of a metal to an object as in electroplating, a thin layer of metal is removed. Electropolishing, as the name implies, is an electrochemical process used in both the laboratory and in a wide range of industrial applications to produce smooth, polished surfaces on a variety of metals and alloys.

Studies conducted at PNL under the Department of Energy (DOE) sponsorship have shown that electropolishing is also capable of rapidly and effectively removing radioactive contamination from metal surfaces. Mild steel, copper, aluminum, stainless steel, and highly alloyed corrosion-resistant and heat-resistant materials have been successfully decontaminated using this technique. Electropolishing can be used to decontaminate, without prior disassembly, relatively complex components and configurations, including assemblies with moving parts, the interior of tubing, and threaded sections. Whenever required, special electrodes and techniques can be used for the in-situ decontamination of components that cannot be accomodated in an electrolytic cell. Moreover, electropolishing effectively removes a variety of radionuclides including plutonium, uranium, radium, cobalt, strontium, cesium, and americium, as well as contamination that is baked on, ground in, or otherwise difficult to remove using conventional procedures (Allen and Arrowsmith 1979).

Electropolishing has been demonstrated to be an effective decontamination technique. The ability to rapidly clean even highly contaminated surfaces to background radiation levels is important both from an environmental and an economic standpoint. Complete decontamination controls the spread of contaminants or permits the repair and return to service of critical components with a minimum of time and personnel exposure. Decontaminated items can also be reused in nonradioactive applications, and metals and alloys can be recycled rather than buried (Allen et al. 1978).

Although the mechanisms of electropolishing are complex, its application as a decontamination technique is relatively simple as it is an intrinsically simple process with no moving parts except those required for circulation or agitation of the electrolyte. Thus, it is amenable to remote operation.

Most of the electropolishing decontamination studies have been conducted using immersion electropolishing systems of the type illustrated in Figure 4. The object to be electropolished serves as the anode in an electrolytic cell. The passage of electric current results in the anodic dissolution of the surface material and, under proper operating conditions, a progressive smoothing of the surface. Any radioactive contamination on the surface or entrapped within surface imperfections is removed and released into the electrolyte by the surface dissolution process. The amount of metal removed from the component surface to effect decontamination is usually less than 0.002 in. and is removed uniformly with no preferential attack of grain boundaries or other microstructural features (Allen et al. 1978).

Figure 5 illustrates the complex current-voltage relationship observed for many of the decontamination studies. At low voltages and current densities, the metal removal is nonuniform, resulting in etching rather than polishing. Conversely, at high voltages, the dissolution process is accompanied by excessive oxygen evolution resulting in severe pitting of the surface (Allen et al. 1978).

Typical decontamination times range from 5 to 30 min, corresponding to the removal of 0.0003 to 0.002 in. of surface material at a current density of

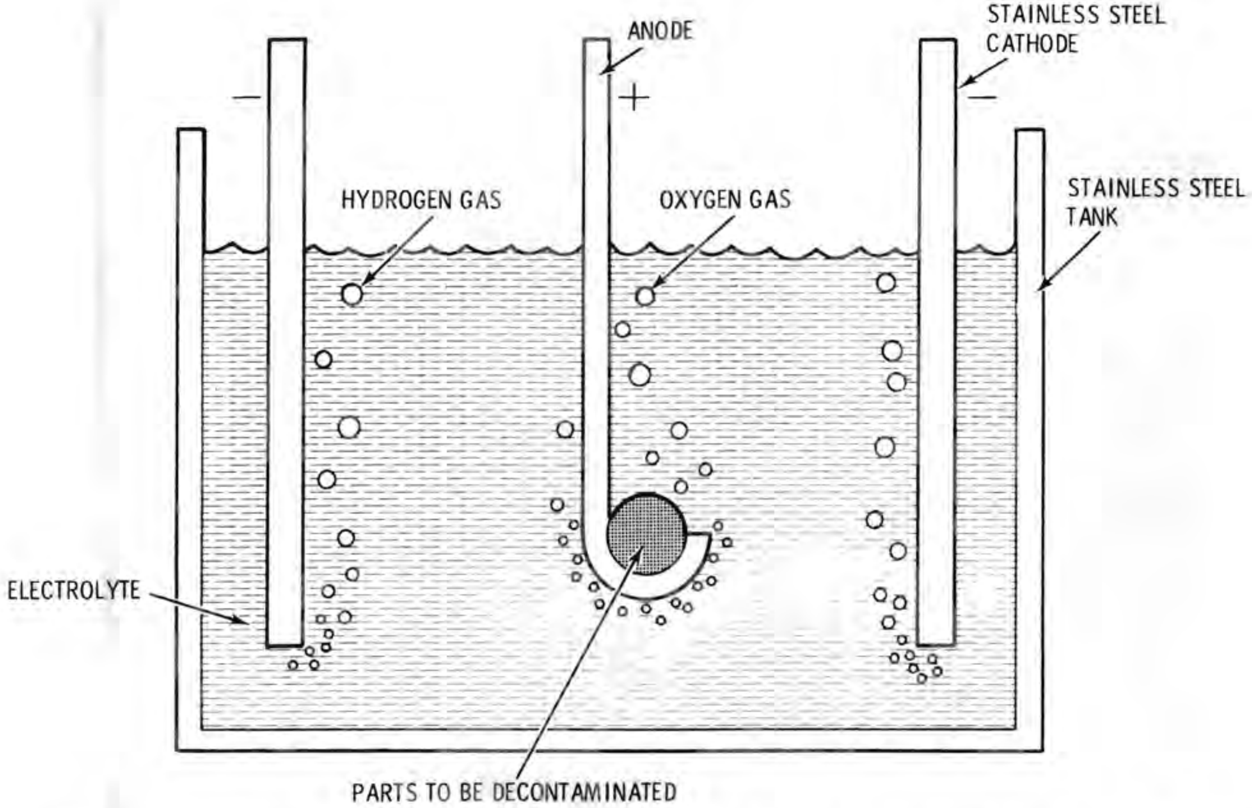

FIGURE 4. Schematic Drawing of Electropolishing Cell Used to Decontaminate Metal Surfaces

150 A/ft^2. It usually is necessary to move the anode contacts once during the electropolishing cycle to ensure decontamination of the area under the contacts.

Stainless steel electropolishing tanks are preferred for decontamination applications because the electropolishing system itself can be decontaminated whenever required by making the tank walls anodic. Also, the metallic tank walls can serve as the cathode for waste processing applications where maintenance of tolerances on the decontaminated component is not important. Otherwise, separate cathodes are used that are shaped and positioned either to ensure uniform polishing of the entire component or to minimize metal removal on threaded sections or other critical component areas.

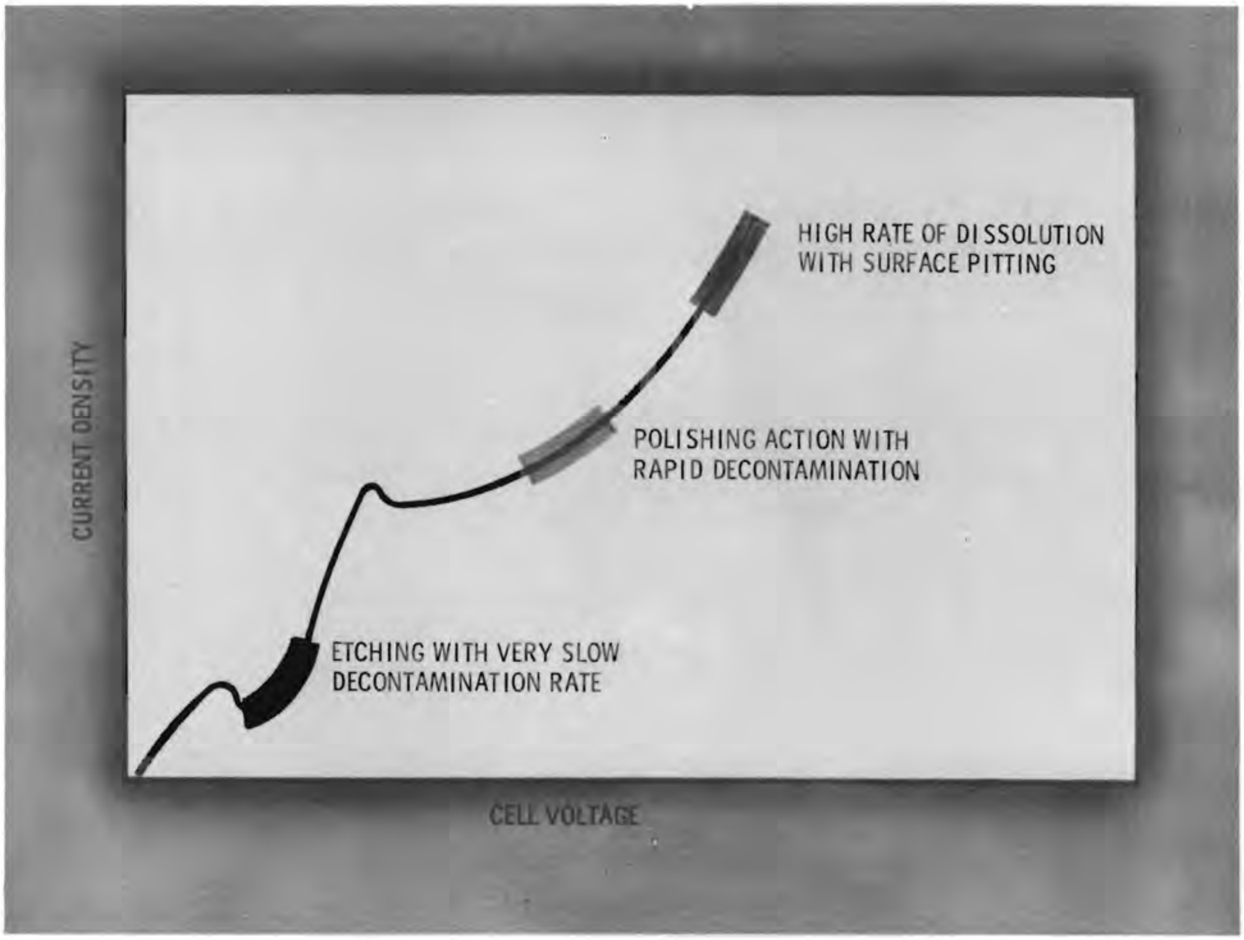

FIGURE 5. Relationship of Current Density and Cell Voltage for Electropolishing Showing Optimum Operating Region for Electrochemical Decontamination

Other components of an electropolishing decontamination system are dc and ac power supplies, rinse tanks, a ventilation system, and provision for cooling, heating and agitating the electrolyte and rinse solutions. (See Figure 21 for a proposed canister decontamination system using electropolishing.)

Process Parameters

Electropolishing does have some limitations as a decontamination technique. It is only effective for surface contamination and is not applicable to induced activity. Although the conductive power of the electropolishing solutions is quite good, the ability of the technique to remove contamination from deep cracks, crevices, holes, and other areas that are shielded from the cathode is limited unless the geometry of the object is favorable for the use of an internal cathode. The surfaces to be decontaminated must be electrically conductive, should be relatively clean, and in a geometry and size that can be accomodated in the electropolishing facility.

Also, although the surfaces produced by electropolishing usually exhibit better corrosion resistance and other properties than surfaces with standard as-received finishes (Zentler and Boyle 1961; Parlapanski 1970), the effect of the electropolishing decontamination treatment on the critical service properties of safety-engineered components must be determined before electropolishing can be used for this class of applications. However, electropolishing has great potential for the control of contamination as well as exposure-reduction applications because of its ability to rapidly remove beta/gamma surface contamination.

Decontamination Studies and Demonstrations

As indicated previously, a number of studies and demonstrations of decontamination using electropolishing have been conducted at PNL under DOE and other government agencies sponsorship. Highlights of previous activities pertinent to this subject and details of unreported work are included below.

Prepolishing Applications

Previous studies have shown that electropolished surfaces are more easily decontaminated using conventional decontamination methods than are metal surfaces with standard as-received finishes. This has been attributed to the removal by the electropolishing process of the microscopic surface imperfections capable of entrapping and retaining contamination (Platt and Powell 1979a). Figure 6, for example, shows electrochemically decontaminated stainless-steel laboratory ware used to transport radioactive solutions. The highly polished surfaces produced by the electropolishing treatment substantially

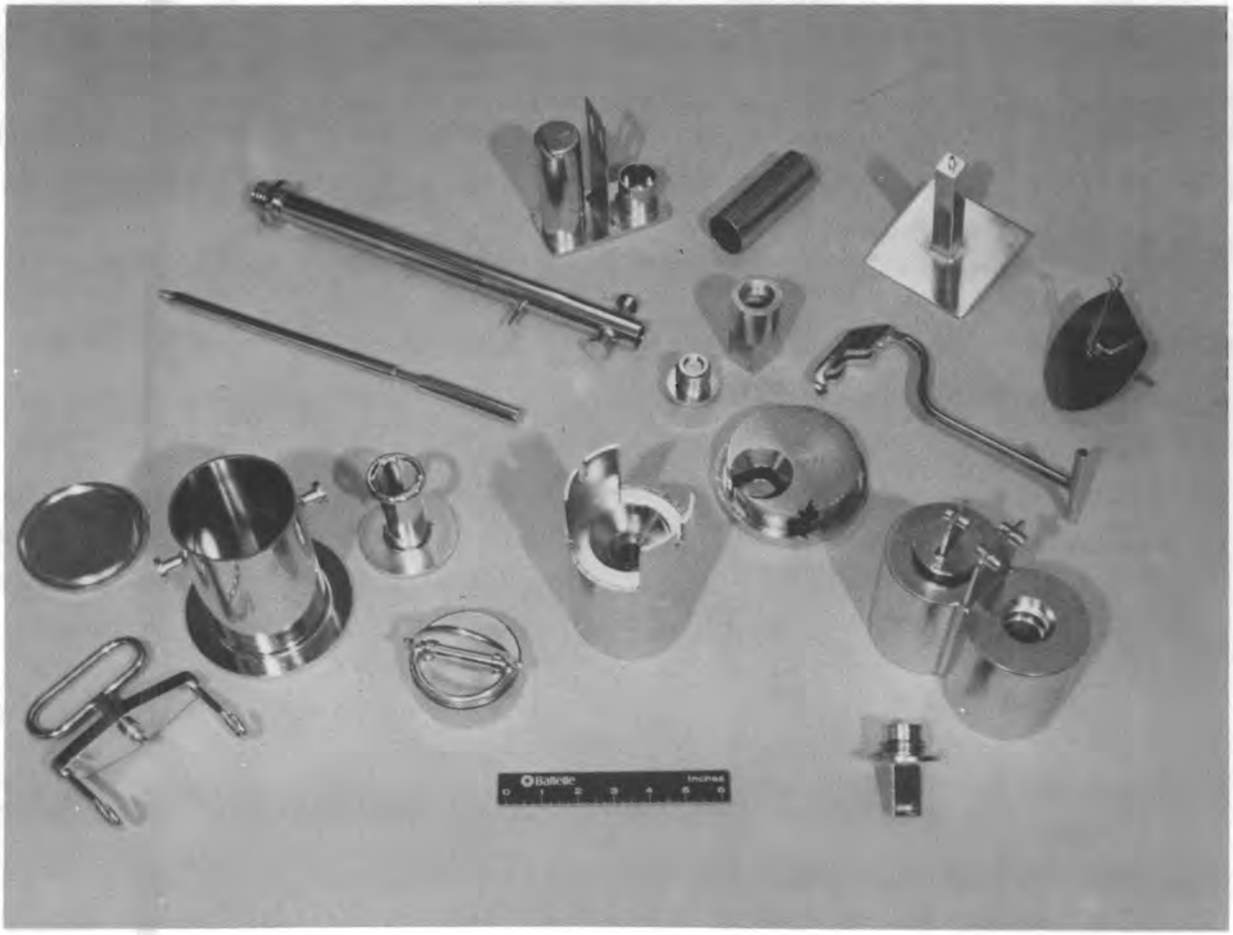

FIGURE 6. Stainless Steel Laboratory Ware Decontaminated by Electropolishing. The highly polished surface facilitated subsequent decontamination using standard swab techniques.

reduced the time and radiation exposure required for subsequent decontamination using swab and other conventional techniques (Allen and Arrowsmith 1979).

These observations suggest that electropolishing used to prepolish the surface of items that become contaminated in their normal service environment should facilitate subsequent decontamination and possibly even reduce the initial contamination level. This electropolishing application should be useful for large components and surfaces such as shipping casks and refueling pool walls, as well as for many types of smaller items, provided that the surfaces are not degraded by the service environment.

Contamination tests conducted in a BWR fuel-transfer channel during refueling operations confirmed that electropolished surfaces also are more resistant to contamination by radionuclides than are other surface finishes under identical service conditions. The results of tests comparing 18 electropolished 304 stainless-steel samples with equivalent samples representing standard surface finishes are presented in Table 9. The electropolished surfaces were substantially less contaminated by the refueling operation than were the other finishes. Subsequent treatment by a high-pressure water spray reduced the levels even further, producing a final contamination level for the electropolished surfaces lower than the minimum achievable levels for the other finishes by a factor of 5 to 13. These test results further demonstrate the value of prepolishing surfaces that will be contaminated in their normal service environment (Platt 1979).

Decontamination Demonstrations

Research studies have demonstrated the ability of electropolishing to reduce the radiation levels of steel tools and stainless steel vacuum system components, heavily contaminated with plutonium oxide, from 1 million dis/min/100 cm^2 to background in less than 10 min. Other examples of objects that have been decontaminated within minutes using electropolishing include hot-cell manipulator assemblies, analytical instrument components, laboratory transfer containers, offsite shipping containers, fission product storage capsules,

TABLE 9. Comparison of Contamination Levels for Electropolished and As-Received Surface Finishes Exposed in a BWR Fuel Transfer Channel During Refueling

	Contamination Level, counts/min	
Surface Finish	After Exposure During Refueling	After Decontamination With High-Pressure Water Spray
Electropolished	3,000 to 6,000	1,500
Superfinish	40,000	20,000
2B Sheet	40,000	20,000
No. 1 finish bead-blasted	15,000	7,500

laboratory animal cages, and nuclear-reactor process-tube components (Allen et al. 1978). These items included large components with more than 15 ft^2 of surface area, ducting, and threaded and precision-machined parts having critical tolerances.

Specific examples of items decontaminated in the 400-gal system located in PNL's Demonstration Decontamination Facility are (Allen et al. 1978):

- Stainless steel animal cages (Figure 7). Electropolishing for 20 min removed radium and lead-210 contamination that had resisted all previous decontamination efforts using standard techniques.

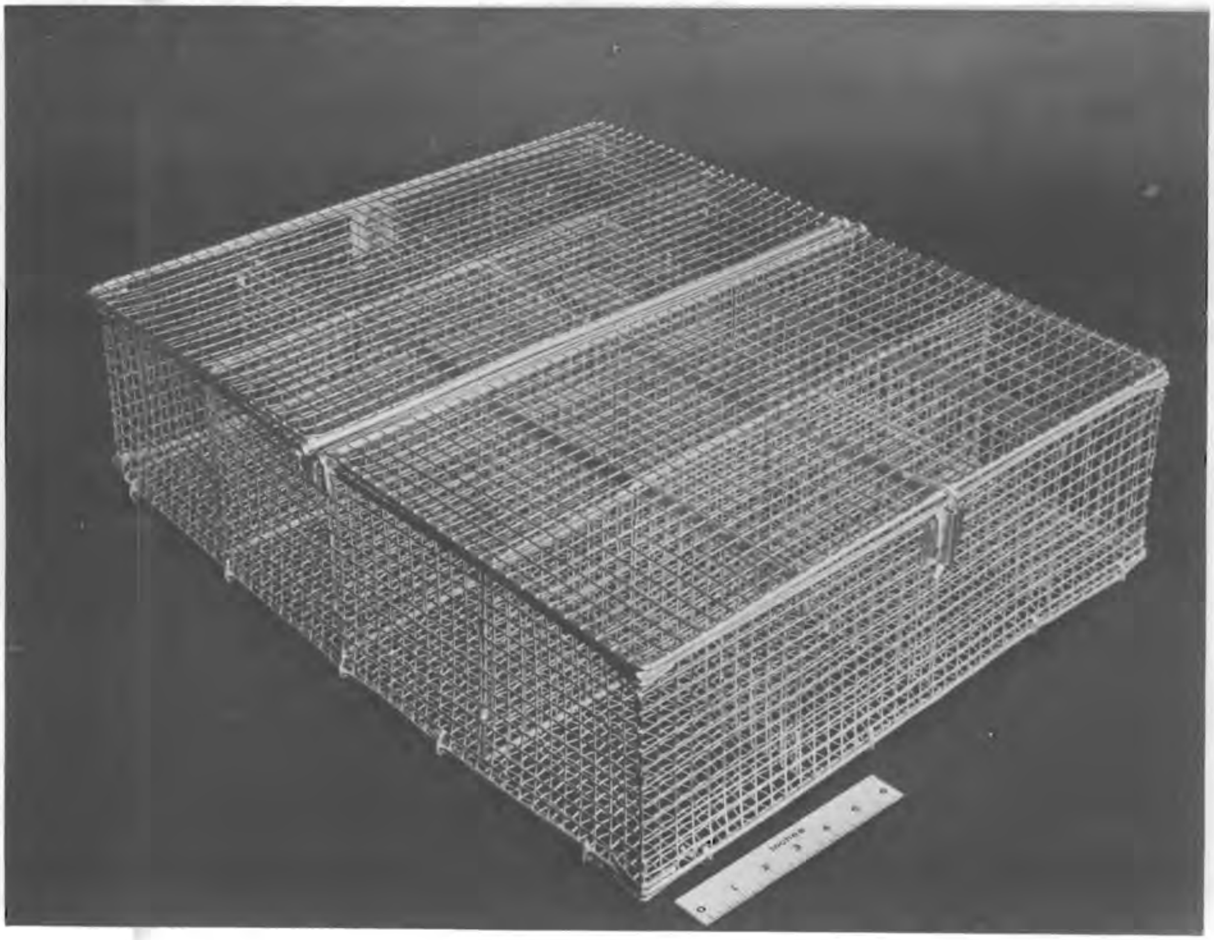

FIGURE 7. Stainless Steel Animal Cage Decontaminated by Electropolishing Within 20 min

- Mild-steel valve from the Hanford N-Reactor (Figure 8). The as-received unit was heavily corroded as well as contaminated with beta/gamma fission products. Electropolishing removed the corrosion layer and reduced the contamination level from 40 mrem/h to background.

A summary of the items decontaminated is shown in Table 10 while specifics related to some are noted in Table 11.

These tests indicate the ability of the electropolishing technique to remove effectively a variety of contaminants from items that vary considerably in size and configuration and that are made of various materials that could be used in HLW canisters. Therefore, the extension of this technology to HLW canisters appears to be very feasible.

Electrolyte Tests

Various combinations of phosphoric, sulfuric, nitric, hydrochloric, and hydrofluoric acids were tested as electrolytes for the removal of oxide layers from heavily oxidized 304L stainless steel canister material from the in-can melter solidification process. The best solutions and operating conditions found in the first test were:

- Electrolyte #1

55% H_3PO_4	Temperature - 80°C
45% H_2O	Current Density - 0.16 A/cm^2
	Time - 1 h

- Electrolyte #2

55% H_3PO_4	Temperature - 80°C
10% H_2SO_4	Current Density - 0.16 A/cm^2
35% H_2O	Time - 1 h.

Both of these electrolytes were effective in removing all of the exposed oxide. Figure 9 is an optical cross section showing the descaled metal and some of the original oxide that was masked to prevent its removal. Approximately 200 μm of base metal, or a thickness corresponding to that of the oxide layer, was removed by the descaling process. Although the resulting surface was microscopically rough, there was no pitting.

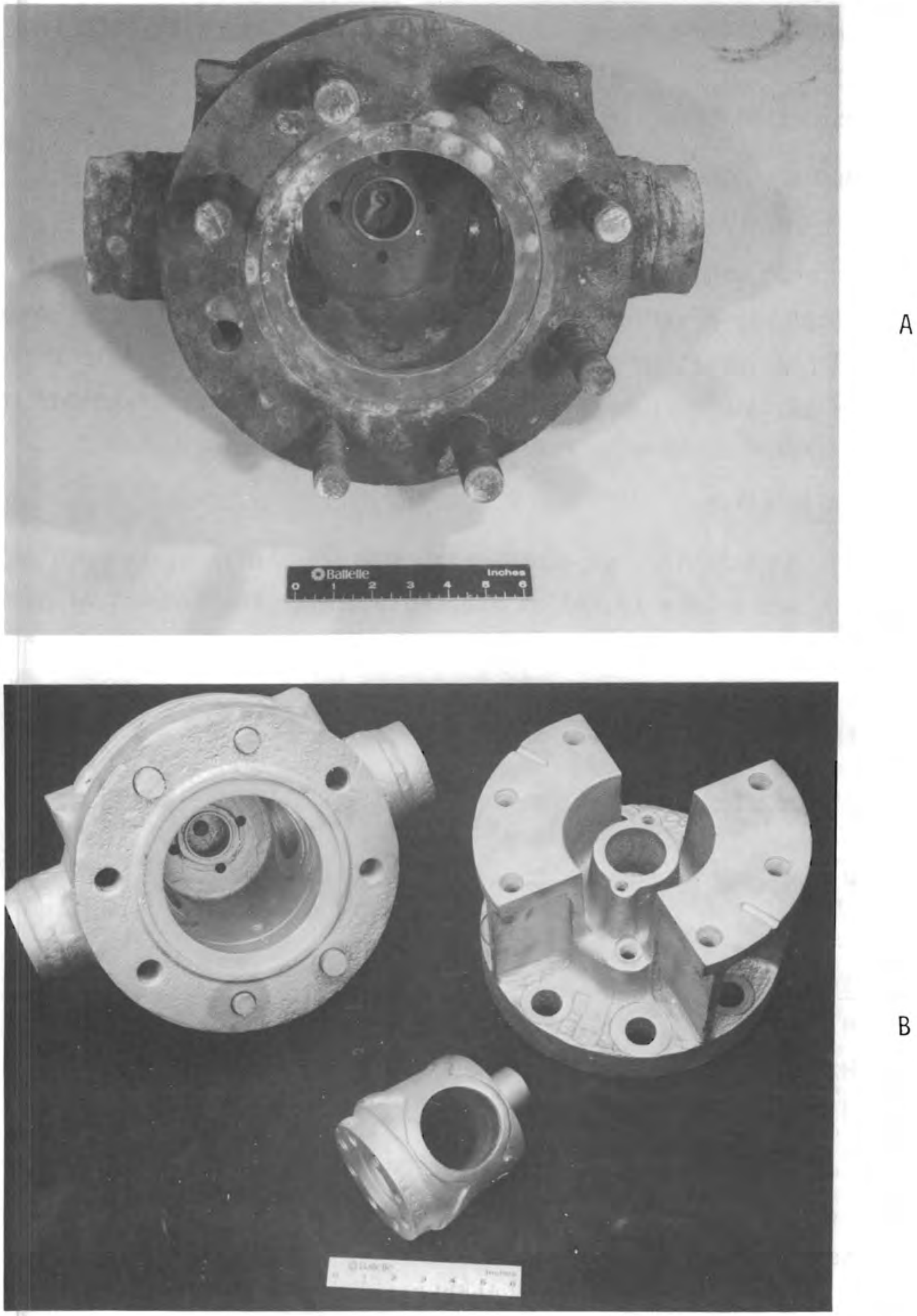

FIGURE 8. Mild-Steel Valve (A) Heavily Corroded and Contaminated Before Electropolishing and (B) Completely Decontaminated After Electropolishing

TABLE 10. Representative Surface-Contaminated Metal Items Decontaminated by Electropolishing

Item	Material	Contamination
Core drill bit	Mild steel	Beta/gamma
Animal cages and trays	Stainless steel	Radium; lead-210
Product receiving canister	Stainless steel	Plutonium
Standards capsules	Stainless steel	Plutonium
Traveling wire flux monitor	Stainless steel	Beta/gamma
Waste sampling tubes	Mild steel	Alpha/beta/gamma
Large reactor valves	Mild steel	Beta/gamma
Electropolishing tank	Stainless steel	Plutonium
Compressor blades	Aluminum	Uranium; beta/gamma
Ducting	Stainless steel	Plutonium
Pipe	Mild steel	Plutonium
Glove-box waste	Stainless and mild steel	Plutonium
Vacuum system parts	Stainless steel	Plutonium
Manipulator tong assemblies	Stainless, mild steel and aluminum	Beta/gamna
Analytical instrument components	Stainless steel	Alpha/beta/gamma
Laboratory ware	Stainless steel	Beta/gamma
Storage capsules	Hastelloy C Stainless steel	Strontium fluoride Cesium chloride
Pneumatic cylinder	Mild steel	Beta/gamma
Demister	Stainless steel	Plutonium
Connector rings	Mild steel	Beta
Pipe clamps	Mild steel	Beta/gamma

TABLE 10. (contd)

Item	Material	Contamination
Mass-spectrometer components	Stainless steel	Alpha/beta/gamma
Chemical vessel	Mild steel	Plutonium
End caps and inserts	Mild steel	Beta/gamma
Foot clamp	Mild steel	Cesium
Glove-box panels	Stainless steel	Plutonium
Power-reactor valve components	Stainless steel	Cobalt-60

Additional tests performed with various electrolytes for the decontamination of materials having an oxide layer as a result of being placed in a high-temperature furnace indicated that an electrolyte containing 63% of H_3PO_4, 15% H_2SO_4 and the balance of H_2O gave the best oxide removal rate.

These studies and others conducted for the PNL-administered Commercial High-Level Waste Fixation Program demonstrated that electropolishing using phosphoric acid electrolytes will remove oxide layers as thick as 200 µm from representative canister surfaces. However, longer electropolishing times (1 h versus 10 to 20 min) and more base metal removal (200 µm versus 25 to 50 µm) are required than would be needed for the decontamination of unoxidized stainless steel (Allen et al. 1978).

Thus, these test results show that 1) electropolishing techniques employing relatively mild electrolytes can be used to descale the surface of oxidized 304L canisters; 2) the optimum conditions for oxide removal correspond to those for optimum electropolishing; and 3) electrolyte compositions are known or can be developed to effectively remove not only contaminants but oxidations that will be generated on the surface of canisters in some of the HLW processes.

TABLE 11. Results of Electropolishing of Various Items

Item	Metal	Initial Contamination	Final Contamination	Electropolishing, Time, min	Contamination Type
Waste Sampling Tank	Mild Steel	1 mR/h	BG(a)	20	Alpha, beta, gamma
Valve (heavily corroded)	Mild Steel	40 mR/h	BG(a)	NR(b)	Beta, gamma
Corebit	Mild Steel	1 R/h	BG(a)	NR(b)	Beta, gamma
Tools	Stainless Steel and Mild Steel	10^6 dis/min/100 cm^2	BG(a)	5	Alpha, beta, gamma
Flux Monitor	Stainless Steel	2 mR/h	BG(a)	15	Beta, gamma
Ducting	Stainless Steel	15,000 dis/min/100 cm^2	BG(a)	5	Alpha
Standard Capsule	Stainless Steel	200,000 dis/min/100 cm^2	BG(a)	4	Alpha
Manipulator Tong	Stainless Steel-Mild Steel Aluminum	10 R/h	BG(a)	10	Beta, gamma
Compressor Blades	Aluminum	2,000 dis/min/100 cm^2 6,000 counts/min/100 cm^2	BG(a)	10	Alpha Beta, gamma
Storage Capsule	Hastelloy C-276	5 R/h	5 mR/h	20	Beta, gamma

(a) BG: Background.
(b) NR: Not Reported.

FIGURE 9. Optical Micrograph of Oxidized 304L Stainless Steel Descaled by Electropolishing With Electrolyte #1

Spent Electrolyte Solidification

As a result of studies[a] to determine the most effective means of converting spent phosphoric acid electrolyte into a neutralized solid suitable for onsite disposal, a direct caustic neutralization process has been selected and is now in use. In this process, 50 NaOH is added directly to the electrolyte, which causes a violent and instantaneous reaction, rapidly increasing the temperature from 25° to 105°C. After the pH reaches 9 to 10, addition of

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[a] Information source for this section: Platt and Powell (1979b).

NaOH is stopped and the solution cools. By the time the mixture reaches ambient temperature, it has become a solid. This process increases the waste volume by a factor of 2.1.

Two other alternative processes investigated were 1) adsorption of the electrolyte on an adsorbent clay followed by neutralization with 50 NaOH and 2) use of urea-formaldehyde to form a solid matrix to contain the electrolyte. The problems with the adsorption process were the difficulty in mixing the electrolyte, caustic, and adsorbent together and the large increase in waste volume (a factor of 3.8). The urea-formaldehyde process was simple and effective. However, since the electrolyte was not neutralized prior to mixing, the acid could be easily leached away from the solid. In addition, since the matrix is organic, it could evolve radiolytic gases when containing an alpha producer.

Canister Materials and Configuration Decontamination

Hastelloy Capsules

Double containment in Hastelloy C-276 capsules(a) is used to ensure the safe, long-term storage of radioactive strontium fluoride processed through the Hanford 225-B encapsulation facility. The inner storage capsule (5.7 cm OD by 0.484 cm long) is filled with strontium fluoride powder in a hot cell and sealed using remote-gas tungsten-arc welding. These remote filling, welding, and handling operations unavoidably contaminate the capsule surface with strontium fluoride in a form that is extremely difficult to remove using conventional scrubbing and spray decontamination procedures.

Therefore, electropolishing was developed as an alternative surface decontamination technique for the inner storage capsule. Initial feasibility studies using contaminated dummy capsules and capsule sections demonstrated the ability of electropolishing to rapidly and effectively remove the external strontium fluoride contamination. For example, the radiation levels of capsule surfaces smearing 4 rad/h were reduced to less than 200 counts/min by electropolishing for less than 30 min. Based on these test results, a 20-ℓ electropolishing system incorporating a cylindrical cathode to maintain

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(a) Information source for this section: Arrowsmith et al. (1977).

capsule dimensions, and special design features to facilitate remote manipulator operation, was developed for installation in the B-Plant encapsulation facility. Using this electrochemical capsule decontamination system, it is possible to produce the nonsmearable surface required for secondary encapsulation by electropolishing the capsules in <30 min with a total metal removal of <50 µm. Although this system is small, it has confirmed that capsules or canisters can effectively be decontaminated in a remote process without damage to the integrity of the canister.

Oxidized Inconel Canister Material

Another study(a) showed that the thinner oxide layer (<16 µm) and associated internally oxidized region formed on Inconel 601 by simulated in-can melter service is readily removed by the same type of electropolishing treatment used to decontaminate unoxidized metal surfaces. Figure 10 compares the electropolished Inconel-601 surface with the original oxidized surface (masked to prevent its removal). The figure shows that electropolishing removes material uniformly and does not preferentially attack grain boundaries or other microstructural features even in the heavily oxidized areas.

Canister Material

To compare results of different decontamination techniques and to determine if electropolishing might be an effective technique to be used following an initial steam-water spray decontamination process, electropolishing decontamination tests using a sulfuric-phosphoric acid mixture were performed on the small 304L stainless steel canisters and the Inconel bar discussed earlier. Figure 11 shows the electropolishing tank used and one of the mini-canisters.

After each of the test pieces had been exposed to a series of steam-water cleaning cycles as discussed in "Canister Material Decontamination Tests" (p. 12), they were processed through an electropolishing system. A summary of the test conditions and results are noted in Table 12.

(a) Information source for this section: Allen et al. (1978).

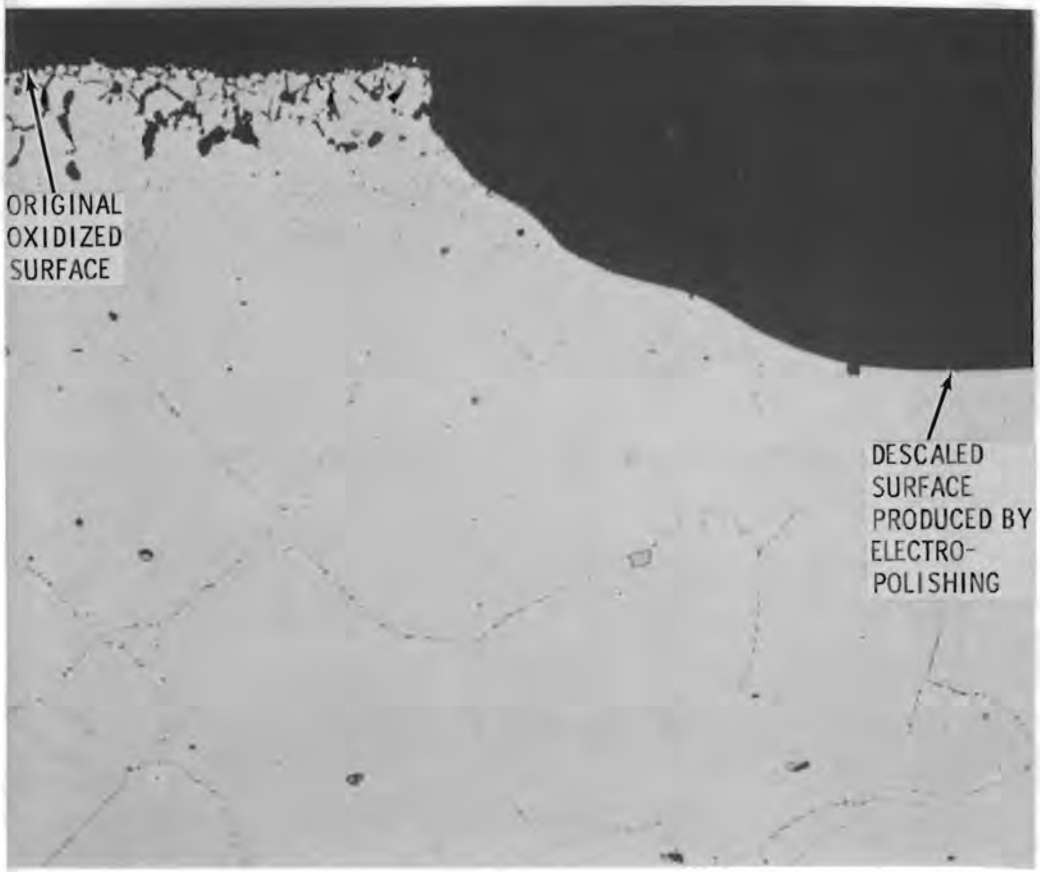

FIGURE 10. Cross Section of Oxidized Inconel 601 Canister Material, Comparing the Original Oxidized Surface and the Descaled Surface Produced by Electropolishing

These tests show that surfaces heavy with oxidation and contamination can be effectively decontaminated to a level which is equal to or below background by using a combination of steam-water washes followed by electropolishing. Also, the tests show that either 304L stainless steel or Inconel can be decontaminated and that the geometry of the contaminated item did not significantly affect the decontamination process. The use of electropolishing reduced the activity level of the canisters by a factor ranging from 24 to 1,000. Also, decontamination of all items using the same electrolyte showed the capability of contaminated electrolyte to still effectively decontaminate items.

FIGURE 11. Electropolishing Vessel and Mini-Canister

Since the steam-water wash cycles appear to be capable of removing only surface contamination in layers, and since electropolishing has the capability of removing contamination not only on the surface but also that trapped below the surface, as well removing material uniformly, it is felt that electropolishing alone could be used for decontamination.

As indicated by Allen et al. (1978), rapid progress has been made in the development of electropolishing as an effective decontamination technique for the removal of transuranics and other contaminants from a variety of metals and alloys. Along with other applications, electropolishing could be used as a decontamination technique to:

TABLE 12. Electropolishing Decontamination of Canisters and Flat Bar Materials

Item	Initial Contamination Level counts/min/100 cm^2	Electropolishing Time	Rinse Cycle	Final Contamination Level counts/min/100 cm^2
Canister #1	223,800	Preheated electrolyte, 1 h plus 5 min reversed polarity	20% nitric acid solution and then air-dry.	2,240
		30 min plus 5 min reversed polarity	Water, then 20% nitric acid. Then rinse in water and air-dry.	224
Canister #2	35,800	No preheat, 2 h and 30 min at various voltages and currents and intermediate rinses	Water, then 20% HNO_2, then water and air-dry.	1,500
		30 min at 5.6 VDC and 62 A	Same as above	190
Canister #3	60,500	No preheat, 90 min at various currents and voltages	Same as above.	630
		60 min at 5.6 VDC and 60 A then 30 min at various voltages and currents	Air-dry	750
		30 min at various currents and voltages	Same as Canister #2	190
Flat Bar	9,300	No preheat, 3 h and 15 min at various voltages and currents and intermediate rinses	Same as above	390

- Decontaminate canisters, equipment, shipping casks, and other components and surfaces contaminated by spent-fuel handling, transportation, encapsulation and storage operations.
- Decontaminate canisters, equipment, shipping casks, and other components and surfaces contaminated by the preparation, transportation and disposal of high-level and other defense wastes.

MINI-WET BASIN

In addition to electropolishing tests, canisters have been soaked in water for extended periods of time and the decontamination aspects evaluated. To demonstrate canister decontamination during storage, a mini-wet basin(a) (illustrated in Figure 12) was established to duplicate parameters of a water basin storage for canisters containing HLW.

The stainless steel-lined basin can hold up to four WSEP canisters. The water from the basin could be circulated through a 5-μm filter for particulate control and an ion exchange column to remove soluble radioactive elements. Cooling coils were provided for decay heat removal. Other operating variables, such as water velocity past the canisters, basin temperature, ion exchange resin used, and filtration, were similar to those designed for the Allied General Nuclear Service's Barnwell facility.

A total of three tests were performed on two canisters during the WSEP campaign. Table 13 shows the WSEP data and canister storage history. The canisters were prepared for these tests by removing all loose foreign material with a water spray ring.

The smearable contamination level of the canister surface was determined before and after each test by smearing a 1000-cm^2 section of the canister with a 100-cm^2 smear pad. A blank smear was used to determine the background level of A-Cell. Table 14 lists the results of the smear tests with the background levels subtracted.

(a) Information source for this section: McElroy (1978).

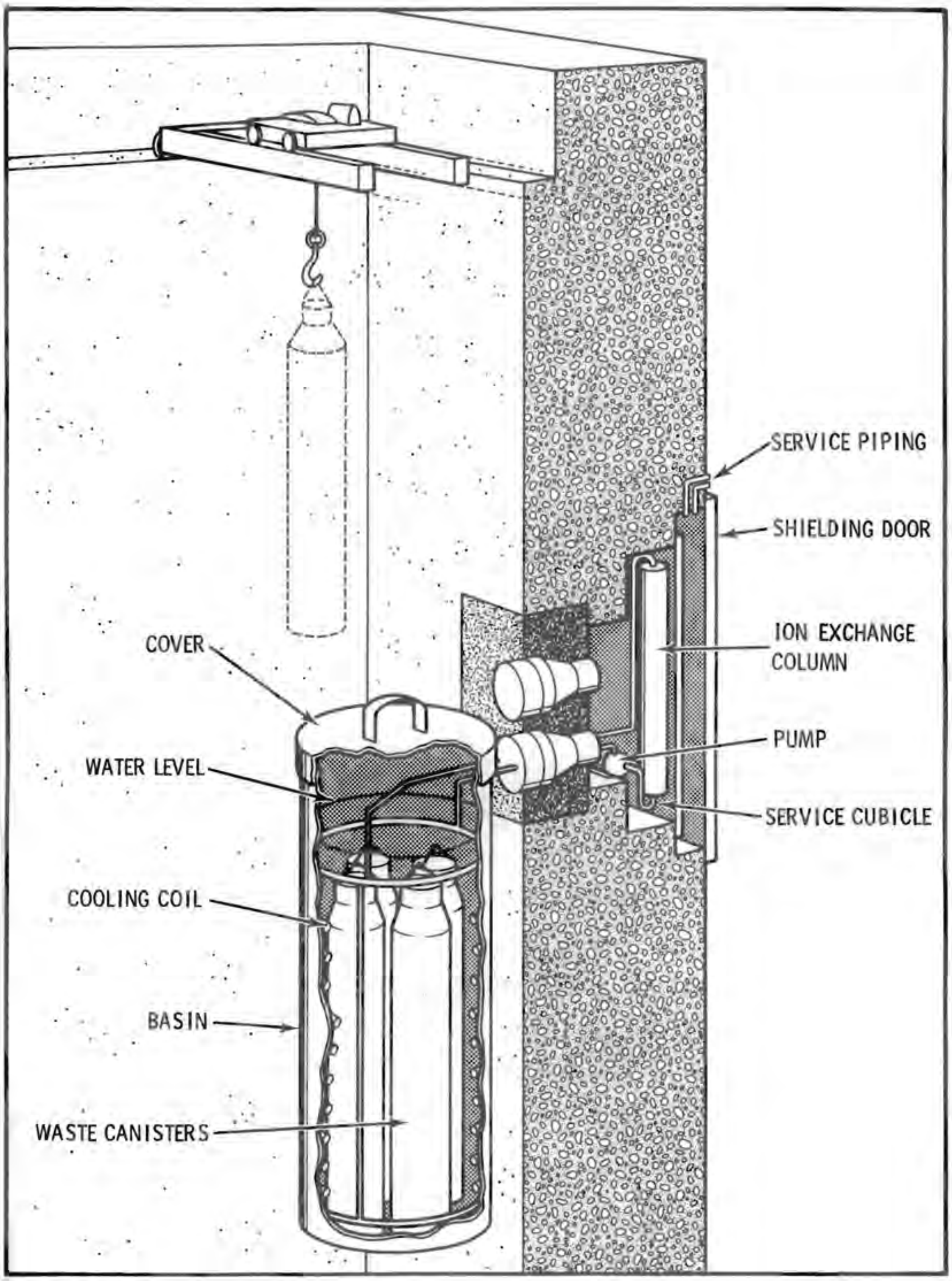

FIGURE 12. Mini-Wet Basin

TABLE 13. WSEP Canister Fill and Storage Data

	Canister	
	SS-7	PG-2
Fill History		
Fill date	December 1978	December 1978
Volume of solids, ℓ	62.0	35.0
Bulk Density, kg/ℓ	3.2	2.9
Fill height, in.	75.0	43.0
Total mCi	3.6	0.4
Total heat rate, kW	12.7	1.7
Heat rate density, W/ℓ	205.0	49.0
Centerline Temperature, °C	928.0	335.0
Wall Temperature, °C	475.0	220.0
Radiation at 8 ft in September 1976, rem/h	51.0	27.0
Canister Material	304L	304L
Storage History		
Water B-Cell, yr	2-1/2	1-3/4
Air B-Cell, yr	---	---
Air A-Cell, yr	5-1/2	5-1/4
Mini-wet basin, yr	1/2	1/2

Figure 13 is a graphical representation of the self-decontamination observed during the three tests. During the longest test, 86% of the initial smearable contamination was removed during storage in the mini-wet basin.

The basin water was periodically sampled and analyzed radiochemically for ^{137}Cs. Figure 14 shows the trend of ^{137}Cs concentration, dis/min/mℓ, in the basin water during each of the three tests. In Test A, a 66-d run, the basin water line was valved to bypass the deionizer. The effect of contamination diffusion to the ion-exchange bed is shown as a decreasing water contamination level. In Test B, a 31-d run, the basin water was valved to pass through the

TABLE 14. Radiochemical Analysis Results of Smear from 1000 cm^2 of Canister Surface

Canister No. and Days	Radioisotope Measured	Initial Smear, Ci	Final Smear, Ci
SS-7, 66	^{144}Ce	2.30×10^{-7}	1.65×10^{-7}
	^{106}Ru	(a)	5.08×10^{-9}
	^{134}Cs	2.17×10^{-9}	(a)
	^{137}Cs	1.89×10^{-7}	3.72×10^{-8}
	^{125}Sb	(a)	(a)
	^{154}Eu	1.58×10^{-8}	1.46×10^{-8}
	Total	4.37×10^{-7}	2.22×10^{-7}
SS-7, 31	^{144}Ce	1.65×10^{-7}	1.13×10^{-7}
	^{106}Ru	5.08×10^{-9}	5.21×10^{-9}
	^{134}Cs	(a)	(a)
	^{137}Cs	3.72×10^{-8}	2.17×10^{-8}
	^{125}Sb	(a)	1.40×10^{-9}
	^{154}Eu	1.47×10^{-8}	1.05×10^{-8}
	Total	2.22×10^{-7}	1.52×10^{-8}
PG-2, 145	^{144}Ce	7.52×10^{-7}	1.23×10^{-7}
	^{106}Ru	1.75×10^{-8}	(a)
	^{134}Cs	5.96×10^{-9}	(a)
	^{137}Cs	7.60×10^{-7}	7.11×10^{-8}
	^{125}Sb	5.41×10^{-9}	(a)
	^{154}Eu	1.25×10^{-7}	2.53×10^{-8}
	Total	1.67×10^{-6}	2.19×10^{-7}

(a) Below limits of detectability.

deionizer. In Test C, a 145-d run, the deionizer was valved out of the basin water recirculation system. The water recirculation was controlled at 0.5 gal/min.

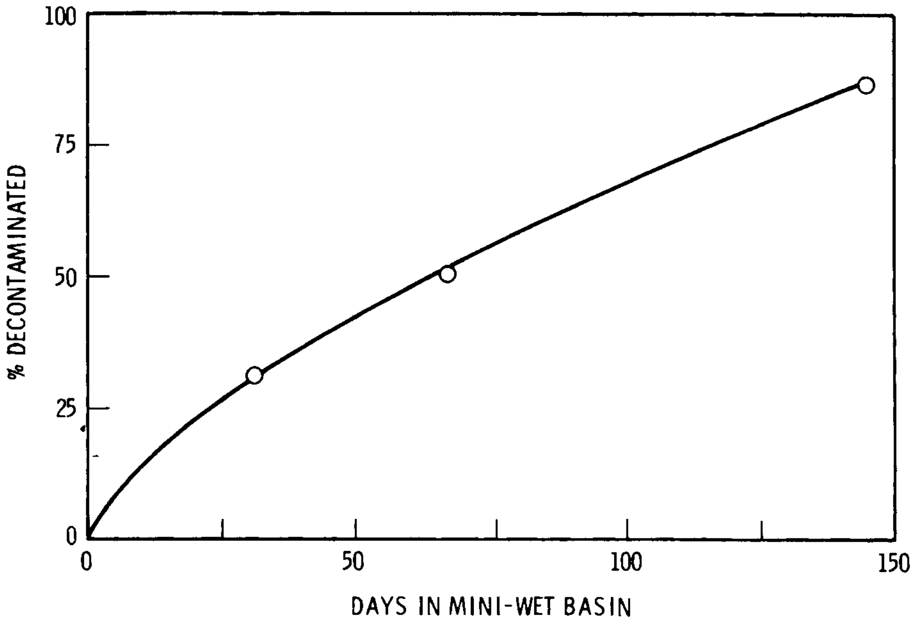

FIGURE 13. Self-Decontamination of HLW Canisters During Water-Basin Storage

Tests demonstrated that during storage of surface-contaminated canisters in deionized water, the radioactive contaminants will move from the canister to the water. This self-decontamination is not only time dependent but it is also dependent on chemical species, chemical structure and concentration, flow rates and canister wall films. Therefore, soaking for extended periods of time is a technique that may not result in the required decontamination factor or would not be compatible with the time requirements of the HLW process. Some studies indicate that a solution of nitric and hydroflouric acids will remove all surface oxides and contaminants. However, the treatment of the contaminated acids would be difficult.

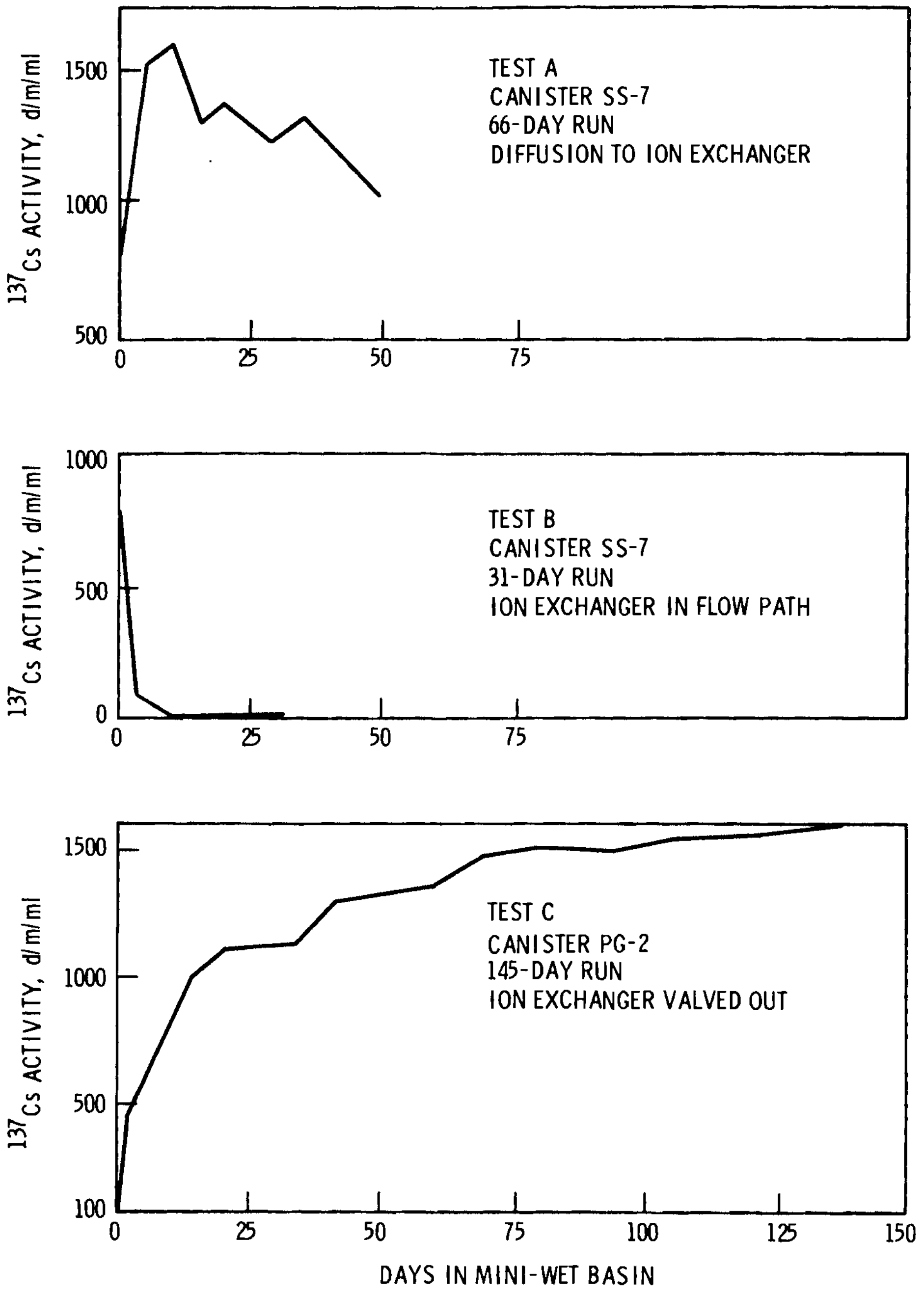

FIGURE 14. Radiochemical Analysis Results, Water Samples

SCRUBBING

Scrubbing is the mechanical removal of foreign material and contamination from a base surface. Scrubbing is typically done with a cloth, metal sponge, brush or abrasive in conjunction with a detergent or solvent. If an abrasive is used, it should not contain any materials that, if left on the surface of the item to be decontaminated, would degrade performance or affect the intended use of the item. In some forms of the scrubbing technique, all the physical and chemical force can be applied to a specific contaminated area. Scrubbing also tends to remove contamination that adheres to the surface, and is probably best applicable for removal of small areas of contamination.

Although scrubbing can be adapted for remote operation, it is typically used in a "hands-on" operation. The application of the technique over the entire exterior surface of a canister in a remote operation would require special equipment.

Vibratory finishing, a form of scrubbing, is described here.

VIBRATORY FINISHING

A vibratory finishing process usually combines mechanical scrubbing action with a chemical cleaning action. Vibratory techniques along with other pretreatment techniques, including high-pressure sprays, ultrasonic cleaning, vapor degreasing, and wet and dry abrasive blasting, were initially evaluated in PNL decontamination studies as possible means to remove paint, grease, corrosion layers or gross contamination from items prior to exposing them to other decontamination processes (Allen et al. 1978). Vibratory finishing was found to be the most promising of these techniques because it could accomplish all of these needs in a single step with a minimum of operator attention and at a high production rate.

A plastic or ceramic medium containing abrasive or nonabrasive metal particles is vibrated at a high frequency in the presence of a cleaning solution. The medium scours the surface of the metal components, and the cleaning solution flushes away the spent abrasive and the material removed from the surface. The composition of the solution can be varied as required to promote removal of grease and paint or other foreign material.

Virtually no solid secondary waste is produced by the process when metal particles are used. However, when a ceramic or plastic medium is used, an appreciable amount of secondary waste in the form of wet sludge is generated.

A vibratory finisher with a 4-ft^3 tub capacity that could hold >300 lb of metal (Figure 15) was used for these pretreatment studies. Other equipment with >50 ft^3 capacity is readily available.

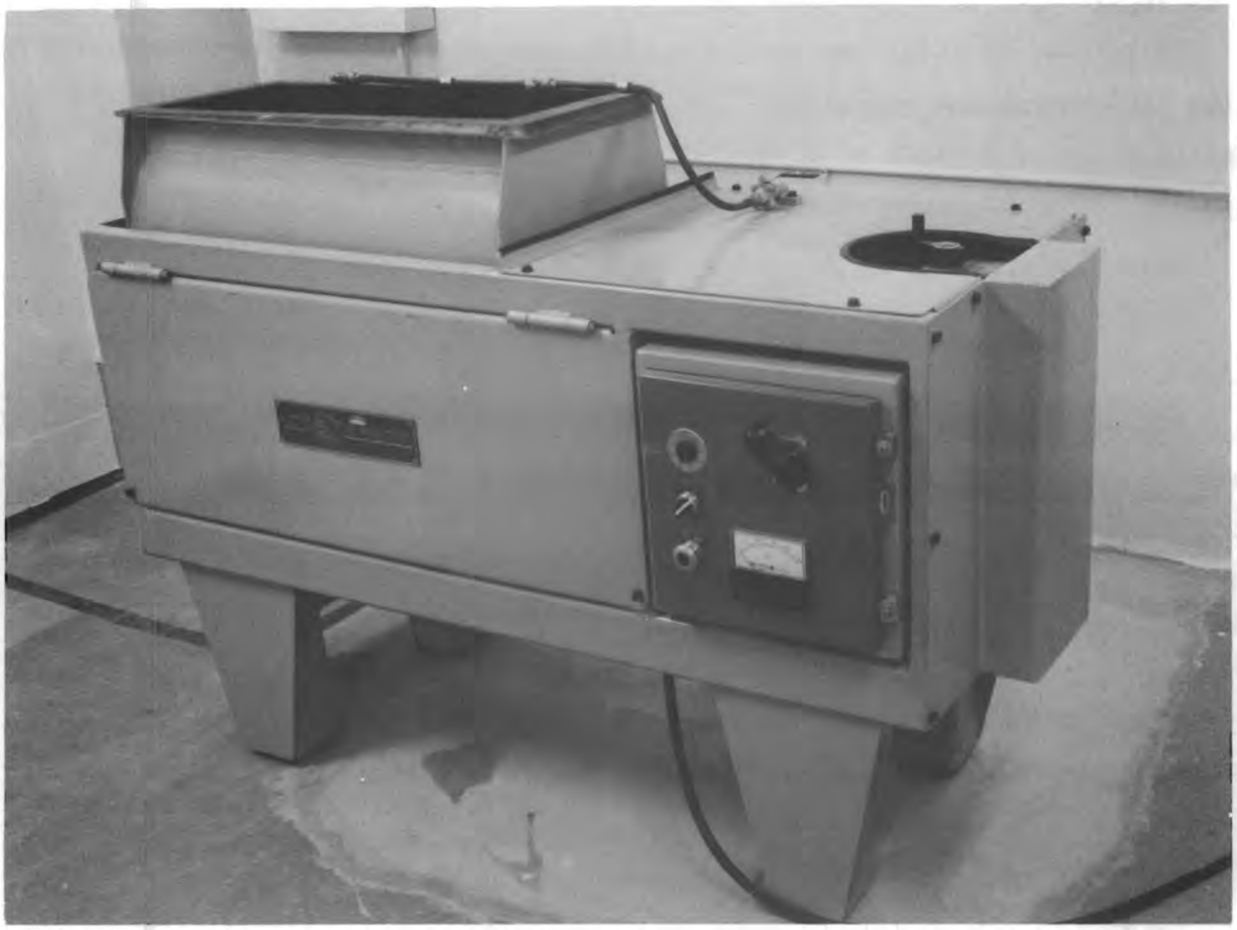

FIGURE 15. Vibratory Finisher With 4-ft^3 Tub Capacity Used in Pretreatment Studies

A variety of noncontaminated metal specimens were tested in the vibratory finisher using a conical-type plastic medium. The vibratory finisher successfully removed rust and epoxy-type paint from irregularly shaped objects (a pipe support bracket, a 2-in.-OD pipe, and a 1-in. hexagonal nut) in less than 10-h processing time. Tests with rectangular stainless steel samples of varying length-to-width ratios showed that although the sharp corners and edges are rounded by the abrasion process, the actual amount of metal removed is very small (less than 0.5 wt% for 31 h of processing) (Allen et al. 1978).

Similar encouraging results were obtained using different types of ceramic media and different cleaning solutions, including dilute phosphoric acid and dilute sodium hydroxide. The caustic solution was particularly effective in rapidly removing grease as well as latex and enamel paints. Epoxy paints required substantially longer processing times, but preliminary studies using special caustic additives and higher operating temperatures have indicated that just a few hours of vibratory finishing under these conditions may be adequate even for these paints.

Beta/gamma-contaminated mild steel and rusted clamps and connector rings from the Hanford N-Reactor were processed in the vibratory finisher. The initial radiation level of the clamps averaged about 100 mR/h, with most of the contamination associated with the heavy rust layer. Four hours of vibratory finishing were sufficient to remove this corrosion layer and leave a clean, bright surface ready for electropolishing as illustrated by the before-and-after picture in Figure 16. In addition, this pretreatment reduced the average radiation level to about 1 mR/h, with higher readings in a few small areas that were inaccessible to the medium.

A second test consisted of 150 contaminated carbon steel rings, 3 in. OD by 2 in. ID by 0.4 in. thick, processed in the same 2 ft^3 of ceramic medium used for the pipe clamps. The rings had an initial radiation reading of 3 to 6 mR/h, which was reduced to less than 0.5 mR/h with 4 h of processing. As with the clamps, most of the remaining contamination was in small inaccessible grooves (Allen et al. 1978).

FIGURE 16. Mild Steel Pipe Clamps (A) As-Received and (B) After Removal of Rust and Gross Contamination by 4 h of Vibratory Finishing

Laboratory-scale studies conducted by PNL in cooperation with Rockwell Hanford Operations Company and UNC Nuclear Industries have shown that vibratory finishing is a rapid and effective technique for removing plutonium and other radionuclide contamination from a variety of metallic and nonmetallic surfaces. For example, the contamination levels of steel tools and stainless steel glove-box panels and ventilation ducts heavily contaminated with plutonium oxide have been reduced from >4 x 10^5 dis/min/100 cm^2 to <10,000 dis/min/100 cm^2 in 1 h processing time. Other objects that have been decontaminated by vibratory finishing include Plexiglas panels, glass tanks, Neoprene gaskets, rubber gaskets, Hypalon gloves, PVC sheet, plastic gloves and bag-out bags (McCoy, Arrowsmith and Allen 1980).

Beta/gamma-contaminated mild steel fuel spacers, process-tube end-caps, and process-tube inserts were decontaminated by vibratory finishing in a steel medium for a total of 10 h using a commercial liquid compound. This test (Table 15) showed that average surface contamination levels of the components were substantially reduced and that most of this reduction occurred during

TABLE 15. Decontamination of Beta/Gamma-Contaminated Components Using Vibratory Finisher with Steel Medium

	Fixed Contamination, mrad/h							
	Initial		After 1 h		After 2 h		After 10 h	
Component	Outside	Inside	Outside	Inside	Outside	Inside	Outside	Inside
Fuel Spacer	800	10,000	20	100	25	75	15	70
Fuel spacer	300	1,500	35	45	25	25	20	80
End cap	200	5,000	20	700	10	100	10	120
End cap	200	1,000	10	30	10	28	8	15
Insert	500	--	300	--	250	--	200	--
Insert	150	--	100	--	130	--	100	--
Steel medium			2,000 counts/min		1,000 counts/min		400 counts/min	

the first hour of processing. Moreover, even after only 1 h of processing, all components were either nonsmearable or had such low levels of smearable contamination (<1000 counts/min) as to substantially facilitate subsequent handling and final decontamination operations (McCoy, Arrowsmith and Allen 1980).

Another demonstration was conducted using electropolishing to decontaminate material that had not been pretreated in the vibratory finisher. The pieces required repeated electropolishing with scraping and application of paint stripper between runs. These applications resulted in longer electropolishing times and a much longer handling time per unit of material (McCoy, Arrowsmith and Allen 1980).

Table 16 summarizes and compares the results of a three-part demonstration. It should be noted that these results represent averages and not optimum values for the entire demonstration. Both the time requirements and final contamination levels decreased as the demonstration progressed, reflecting the normal evolution of research and development-type procedures into production-oriented material handling methods. Moreover, these results were generated using small, manually operated vibratory finishing and electropolishing systems and do not reflect the substantial increase in production rate and decrease in labor requirements that could be realized through system scale-up and automation.

TABLE 16. Comparison of Demonstration Results

Specifics	Vibratory Finishing	Electropolishing With Vibratory Finishing	Electropolishing Without Vibratory Finishing
Number of runs	19	21	21
Total area processed, ft^2	315	131	118
Processing time, min/run	65	12	36
Production rate, ft^2/h	17	7	3
Labor, man-h/ft^2	0.14	0.37	0.72
Final contamination level, nCi/g	0.11	0.0006(a)	0.0014(a)

(a) Electropolishing can completely decontaminate metallic surfaces. The objective of this study was only to decontaminate material to well below the 10 nCi/g limit.

Furthermore, even after decontaminating this material, the medium and tub walls were found to be essentially uncontaminated. The same abrasive action that removes the contamination also keeps these surfaces clean. The contamination, rust, paint, spent abrasive, etc., are continuously washed out of the vibratory finisher into a waste container where they can be collected and concentrated for disposal. Thus, in addition to preparing surfaces for final decontamination by electropolishing, vibratory finishing is itself an effective decontamination technique that may be adequate for a number of applications. Its adaptation for use in full-scale canisters may be limited.

EVALUATION OF DECONTAMINATION PROCESSES

The application of a decontamination technique to HLW canisters must be considered on the basis of decontamination criteria to the degree that a selected technique is useful in meeting a specified and acceptable radiation level. In addition, a decontamination technique must be selected in terms of various factors, such as remote application, canister material, canister configuration, canister size and mass, and types and forms of contaminants. This section outlines possible decontamination criteria and evaluates the limitations and advantages of the various decontamination processes investigated, and concludes that electropolishing is the most effective process demonstrated.

DECONTAMINATION CRITERIA

Although the transportation and the repository requirements and limits of HLW canisters have not been fully identified or established yet, it is believed that the decontamination criteria for metallic canister holding HLW can be stated as follows: All significant removable radioactive nuclides, or foreign material containing radioactive material, or material that may fall or break off, shall be removed from the outer surfaces of a HLW canister as soon as practical after the canister is filled and sealed, and before the canister is moved or transported from the process cell where it was filled.

In a HLW solidification process, significant contamination levels should be determined in relationship to the facility and the existing background levels that may exist in the cells, the storage pools or for the internals of the shipping casks. The extreme lower significant level may be that identified in 49 CFR 173.397 and as shown in Table 17.

The specified level that the exterior of the canister is to be lowered to should assure that the filled waste canister will not cause significant radionuclide contamination of the downstream processing facilities by the shedding of surface contamination during normal operations.

TABLE 17. Limits for Removable Contamination

Radionuclides	Maximum Permissible Level for Any 300 cm^2 Surface Area Ci/cm^2	dis/min/cm^2
Natural or depleted uranium and natural thorium		
Beta-gamma	10^{-3}	2,200
Alpha	10^{-4}	220
All other beta-gamma-emitting radionuclides	10^{-4}	220
All other alpha-emitting radionuclides	10^{-5}	22

DECONTAMINATION PROCESSES LIMITATIONS AND ADVANTAGES

Steam and Water Spraying

The in-cell equipment for a steam-water decontamination process is rather simple and is of the type that is compatible with remote operations and replacement since it consists mainly of vessels and lines. A conceptual design of a spray decontamination facility is shown in Figure 17.

Tests have been performed on samples representing potential canister material, on small-scale canisters as well as on full-size canisters filled with HLW. Tests performed to date indicate that steam-water sprays remove most of the contamination early in the decontamination cycle but that there may be considerable contamination remaining even after fairly long cleaning cycles. Also, the sprays are not able to remove contaminants that may have penetrated the surface. Such contaminants can be expected in glass-waste processes where canisters oxidize and glass containing HLW may be deposited on the outer surfaces of the canister.

Steam Spraying

A steam decontamination station would be essentially the same as a steam and water station. Tests conducted with steam have shown results similar to those for steam and water cleaning of various materials and configurations

Also, since this blast technique tends to disperse the contaminants and the material removed from the surfaces being cleaned, controlling the spread of the contamination and maintaining a low background contamination level in the overall system are problems.

Abrasive Blasing

Certain abrasive blasting techniques are capable of removing radionuclides and foreign material (i.e., oxides) from stainless steel surfaces. Adapting a particular technique to a fully remote mode will involve considerable development and some in-cell mechanical components. Using glass frit as the abrasive medium and cycling it into the glass HLW process would be a major step in reducing the complexity of handling and processing the secondary wastes.

Soaking

Tests performed on full-scale canisters containing HLW show that with time (tens of days) a considerable reduction in radioactivity can occur due to self-decontamination in circulated and treated deionized water. However, this decontamination is not complete, and self-decontamination may not be adequate to prevent the spread of contaminants present on the outer surfaces of various HLW canisters during subsequent handling, transportation or storage.

Vibratory Finishing

Vibratory finishing has great potential for reducing contamination levels. The versatility of the operation combined with the minimal operator requirements make vibratory finishing particularly suitable for field decontamination installations and applicable to remote operations. Tests have shown that vibratory finishing is a fairly rapid and effective technique for removing plutonium and other radionuclide contamination as well as rust and other foreign material from the surfaces of a variety of metallic and nonmetallic items.

Although there presently are commercial vibratory units that have up to 60-ft^3 capacities, the size and mass of the HLW process canisters would limit the vibrating finishing system to the use of a ceramic medium rather than the metal medium which is normally used for decontamination applications.

Electropolishing

The in-cell electropolishing equipment will be more complicated and involved than that for a steam-water spray system, but still applicable to remote operation as well as remote removal and replacement. Also, the requirements of full-scale equipment are better identified and will probably be no more complex than the in-cell equipment required for vibratory finishing, liquid honing or the abrasive blasting processes.

Various tests performed on small-scale canisters and on various materials that might be used for HLW canisters have shown that electropolishing is an efficient and effective decontamination technique and produces cleaner surfaces much faster than either spraying or soaking. Figure 18 illustrates a comparison of the electropolishing, liquid honing, and vibratory finishing of 9 m^2 of the material, and shows how efficient electropolishing is compared to these processes. Also, for the time required for decontamination, the results of electropolishing are orders of magnitude better.

Unlike the various spraying or soaking techniques, electropolishing has the capability of removing loose and fixed contaminants from the surface as well as contaminants that may be trapped below the surfaces by applying uniform and controlled removal of the base material. Also, tests have shown electropolishing to be effective in removing other foreign material such as oxide layers that may hold or trap contaminants.

The electropolishing electrolyte contains the contamination removed from the surfaces being cleaned, and limits its dispersal. Liquid honing and abrasive blasting techniques do not guarantee this type of control. Also, this technique permits the maintaining of a low background contamination level in the system. Electropolishing also leaves a smoother surface on the decontaminated item without preferentially attacking grain boundaries or other microstructural features. The resulting electropolished surface exhibits better corrosion resistance than before, and if recontaminated can be decontaminated more easily.

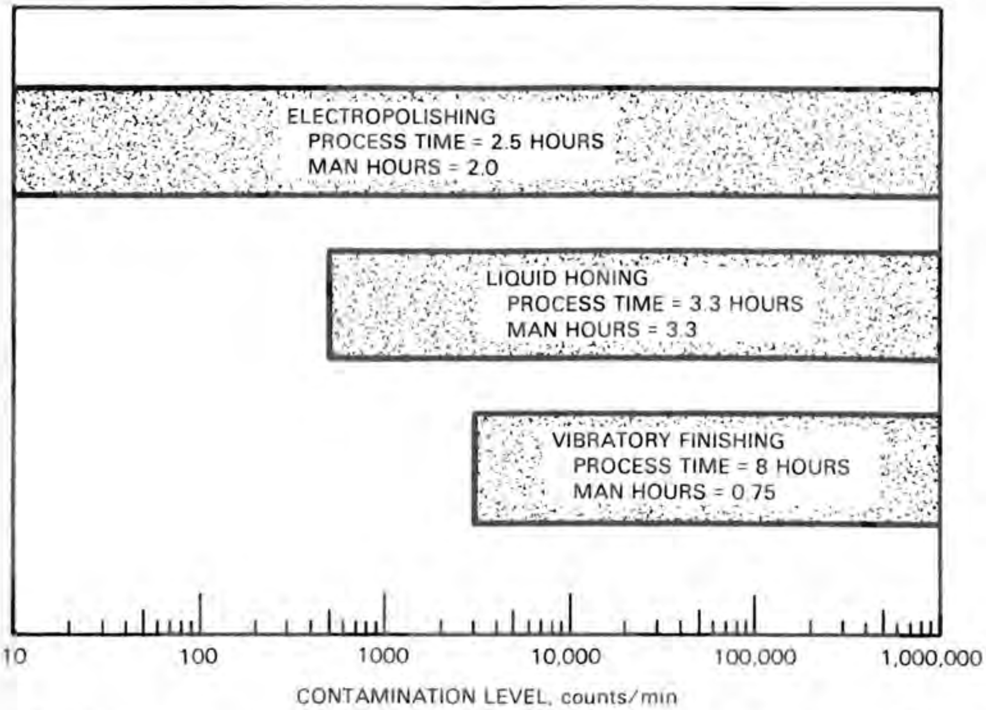

FIGURE 18. Comparison of Processing Time, Labor, and Final Contamination Level of 9 m^2 of Material Decontaminated Using Electropolishing, Vibratory Finishing, and Liquid Honing

Since electropolishing is so effective in decontaminating the materials used for canisters, and can remove the contaminants and the foreign material that could be deposited on the outside configuration of a canister in any of the HLW processes being considered, it should be the technique used for required canister decontamination.

PRECONCEPTUAL OUTLINE OF DECONTAMINATION STATION

A decontamination station or system to limit and to control the spread of nuclear contamination that may be deposited or present on the external surfaces of a canister will, by necessity, have to be designed to be compatible with a particular HLW solidification process. The decontamination system presented here must be considered preliminary and only as a concept that could be developed into a detailed design applicable to a given HLW process in a particular facility.

INTRODUCTION AND BACKGROUND

The first consideration in reducing contamination is preventing it in the first place. Radioactive contamination of the canister will occur principally as a result of the direct contact of the canister with HLW material such as calcine, contact with contaminated surfaces, or during exposure to the environment in the process cells. Means of potential contamination must be evaluated and provisions incorporated into the design of the HLW process, its equipment, the canister and the facility to prevent the spread of or at least minimize and control all radionuclides.

OBJECTIVES

Since the basic requirement for a decontamination system or station is to have the capability to reduce the contamination level of HLW canisters to a prerequisite level that could be dependent on the location in the HLW process, the objective of a decontamination system is to provide the equipment and the provisions to clean the external surfaces of HLW canisters as required as they are processed.

CRITERIA

The canister decontamination system shall have the capability to remove all significant radioactive nuclides and foreign material containing radioactive elements, as well as other material present that may fall or be broken off from all of the outer surfaces of a HLW canister without any harmful effects to the canister.

BASES AND ASSUMPTIONS

The following bases and assumptions are to be used for the HLW processes, the canisters and the decontamination system:

- The canisters will have only smooth surfaces and rounded corners, and all exterior surfaces will be accessible.
- The canisters are to be made of a material or materials that are compatible with the decontamination method or methods.
- The decontamination technique and procedure used will not affect the HLW product form or the integrity or quality of the canisters.
- The decontamination technique will limit and control the spread of contaminants removed from the canisters.
- The secondary waste produced during the decontamination process will be kept to a minimum.
- Decontamination secondary wastes will be of a form or forms that can be incorporated upstream in the HLW process, or easily solidified and disposed of.
- The decontamination technique will only be performed on closed and sealed canisters.
- The system must be remotely operable and reliable.

PROCESS DESCRIPTION

Canister decontamination will be initiated after a canister has been filled and sealed and is at such a temperature that the decontamination process will not have a detrimental effect on the waste product form. (The product and canister may need to be cooled to a steady state with the canister-wall temperature close to that of the decontamination solutions.)

The initial decontamination (which may be the only one) will be conducted in the primary process cell or during a transfer to a secondary cell. This first step will at least remove any loosely attached contaminated material that could become airborne or transferred by direct contact. Electropolishing, which is a special adaptation of soaking, is proposed for this decontamination system.

In the electropolishing technique, the object to be decontaminated serves as an anode in an electrolytic cell. The passage of electric current results in the anodic dissolution of the metal surface. Any radioactive contamination on the surface or entrapped within the surface imperfections or oxide layer is removed and released to the electrolyte during this process. The amount of metal normally removed from the component surface to effect decontamination is approximately 0.002 in.

A basic schematic of a canister electropolishing station is shown in Figure 19. The electrolyte, which may be any one of several acidic solutions, is held in a stainless steel tank. Provisions for heating, cooling, and agitating the solutions are included. The canister is lowered into the tank and electropolished. Afterwards, it is slowly removed while the electrolyte is rinsed off with hot water. Additional rinses may be performed in another tank.

The decontamination station, like all other operations in a remote process facility, should be as simple as possible and haVe a minimum number of controls and remote connections. A detailed design of an electropolishing tank for such a decontamination station, utilizing an overhead crane or hoist to install, relocate and move a canister, is shown in Figure 20.

This demonstration tank is designed for the decontamination of a canister 2 ft in dia and 3 ft tall. A 6-in.-wide cathode band is located in the upper, larger-diameter portion of the tank. Sections of the canister are lowered in line with the cathode band and decontaminated one at a time during successive cleaning cycles. After all of the contaminated surfaces of the canister have been exposed to the cathode band and cleaned, the canister is lifted out of the electrolyte and its outer surfaces rinsed with water or steam from sprays

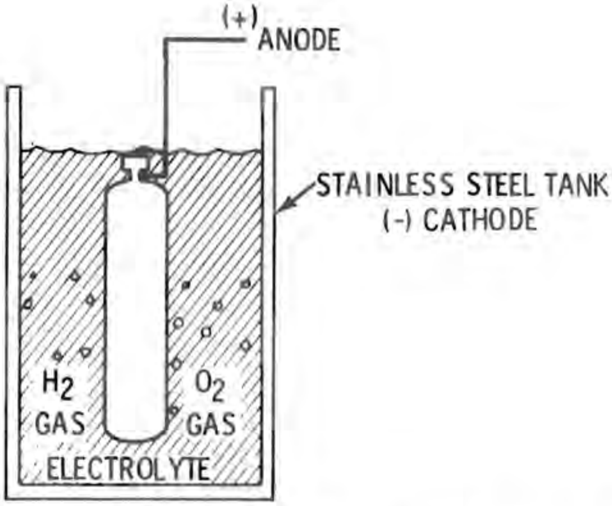

FIGURE 19. Schematic of Canister Decontamination

located around the upper portion of the tank. The condensate is collected in the tank. This condensate can replace the water that may have evaporated as a result of the heat loading of the canister.

If desired, the electrolyte can be drained from the tank prior to canister removal. The canister is then sprayed and removed from the tank. The resulting condensate is drained and collected for reuse in a separate holding tank. After the canister dries, a smear is taken and analyzed to determine if the decontamination was adequate.

A process flow diagram for this type of an electropolishing canister decontamination system is depicted by Figure 21. This diagram indicates the services and the auxiliary equipment required as well as their relationship to the canister in a remotely operated process decontamination system. For a system to decontaminate canisters 2 ft in dia and 10 ft tall, the electropolishing tank would have an approximate maximum dia of 4 ft and a height of ∿15 ft. If the canister were only 16 in. in dia, the maximum diameter of the tank could be reduced to ∿3 ft.

Assuming the canisters are fabricated of 304L stainless steel and have oxidized surfaces, the best electrolyte for decontamination application would be a phosphoric acid solution similar to one of those noted in "Electrolyte

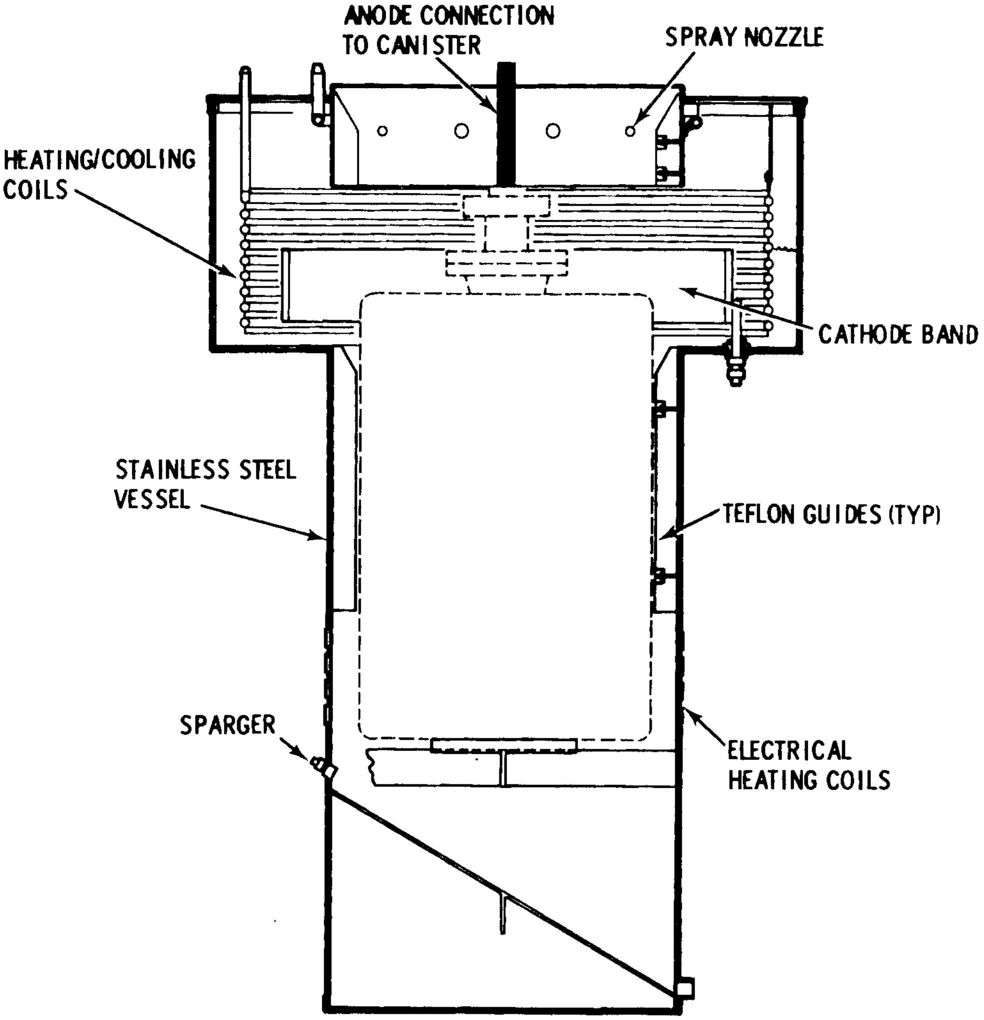

FIGURE 20. Electropolishing Tank Concept

Tests" (page 29). However, the electrolyte solution selection will depend on process safety and waste disposal requirements (including possible incorporation of the waste in the waste-form process). Approximately 300 gal of electrolyte would be the minimum amount required for a system to decontaminate 2-ft-dia by 10-ft-long canisters.

Although the electrolyte may be highly contaminated, it will remove contaminants from the surface of the canister. Also, when the activity of the solution is too high, the electrolyte, depending on its composition, can be reclaimed by fractionation, acid absorption, or solvent extraction, and then reused. The small amount of residual radioactive material can be then combined with other liquid wastes for treatments. It may also be possible to include all secondary wastes from electropolishing in the HLW stream. Initial assessments show that inclusion of typical electropolishing wastes would not adversely affect HLW solidification. The inside of the electropolishing tank may be cleaned by the use of installed spray nozzles or by reverse electropolishing.

The best methods to reclaim electrolyte or to handle and process the secondary wastes will be dependent on the particular HLW and decontamination process, the canister material and configuration, and the facility. Therefore, these methods are not identified further.

Immediately after the decontamination operation, the canister is transferred to the next cell or process station to prevent recontamination by the cell environment. As discussed above, the decontamination may be performed during transfer operations. This type of an arrangement is shown in Figure 22. If further decontamination operations are required for the canister, other decontamination stations located in adjacent process areas are to be utilized. These may be identical or similar to the first decontamination station, or an in-situ technique may be adequate if only selected areas on the canister will require decontamination.

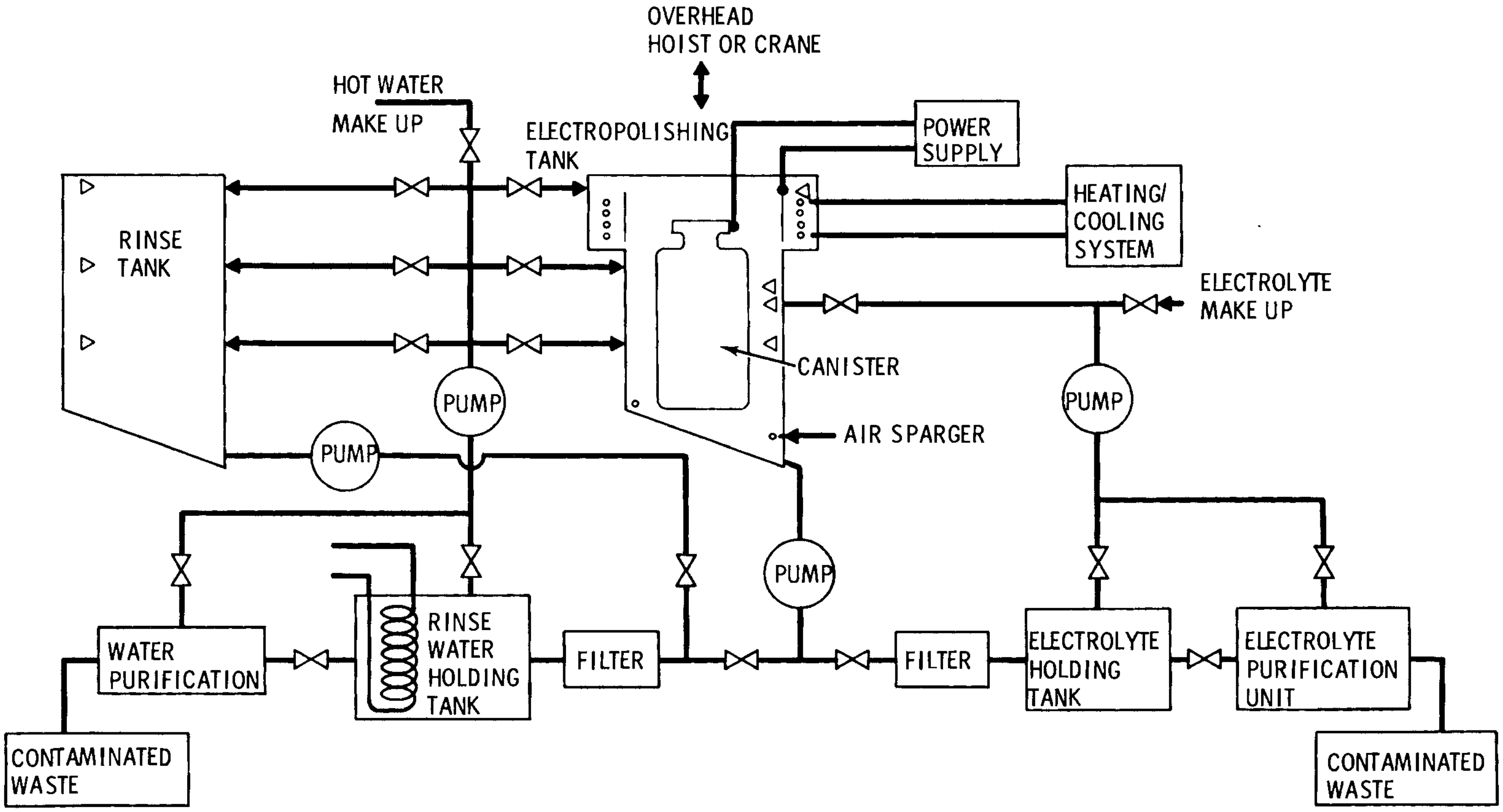

FIGURE 21. Electropolishing Process Flow Diagram

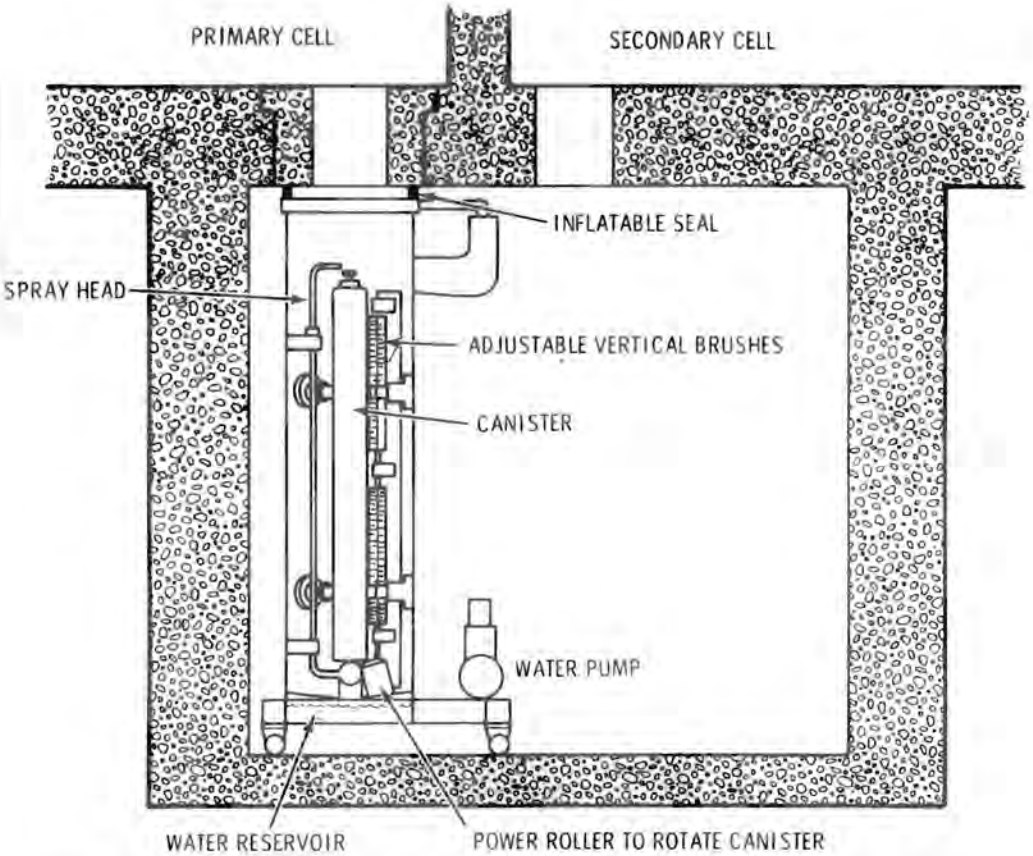

FIGURE 22. Scrubbing Decontamination Used in Conjunction With a Transfer Operation

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