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ELECTRODECONTAMINATION OF STAINLESS STEEL

M. R. Bennett

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CHEMICAL TECHNOLOGY DIVISION

Chemical Development Section

ELECTRODECONTAMINATION OF STAINLESS STEEL

M. R. Bennett

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1.0 ABSTRACT

In experimental studies, decontamination factors of 1000 to 3000 were obtained by electrostripping deposited radioactivity from stainless steel surfaces, used as the anode, in 2% sulfuric acid at current densities as low as 0.01 amp/in.^{2}. Stainless steel cathodes were used. The method was successfully applied to contaminated equipment.

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2.0 INTRODUCTION

Stainless steel and other metallic equipment used in radiochemical environments can be decontaminated by corrosive reagents which dissolve and remove a layer of metal. Corrosive-reagent techniques are often successful on initial decontaminations, but subsequent attempts to decontaminate surfaces previously corroded are usually more difficult and less effective owing to the pitting and etching of the surface in the earlier treatment.

Scouting investigations had indicated that electrolytic techniques were promising as a means of decontaminating metal. The experimental program reported here included laboratory-scale electrodecontamination and corrosion studies of stainless steel samples that had been contaminated during 2.5 years of pilot plant operation with processes including the Redox and 25 processes. The effects of the following variables on the decontamination achieved and on the corrosion rate of the samples were studied: current density, time, cathode material, agitation, temperature, throwing power of the solution, and shape of the cathode. Field test decontaminations were carried out on three tanks from the No. 200 RaLa modification cubicle in the Isotopes Building and on a stainless steel evaporator cover plate that had been used for three years in the 23 process, all of which were recovered from the burial ground.

3.0 DESCRIPTION OF EQUIPMENT AND PROCESS

The electrolytic cells used for the experimental decontamination studies (see Fig. 3-1) were fabricated from 3-in.-diam. pyrex glass pipe; they were designed with side arms to permit the introduction of electric-motor-driven agitators, thermometers, and electrodes. The volume capacity of each cell was 350 ml.

The cathodes were stainless steel, and the surface area was varied to give the desired current density. The anodes consisted of the material to be decontaminated. Test specimens were samples of type 347 stainless steel that had been contaminated during 2.5 years of pilot plant operation. Each specimen had a surface area of 5.0 to 6.0 in.^{2}. These specimens were suspended in the electrolyte, current was passed through the cell for the time indicated, and the electrodes were removed before the current was turned off. The contamination remaining on the electrode was determined by counting techniques.

The composition of the electrolyte used for the decontamination studies was selected primarily on the basis of corrosion, conductance, and cost. Preliminary tests showed that a 2% solution of H_2SO_4, with a relatively high equivalent conductance of 213 mhos at 25°C, corroded stainless steel only very slowly at low current densities (approximately 0.14 mil/hr at 0.03 amp/in.^{2}). Since the cost of such a dilute acid mixture would be relatively low on a large scale, further investigation of electrolyte compositions seemed unwarranted.

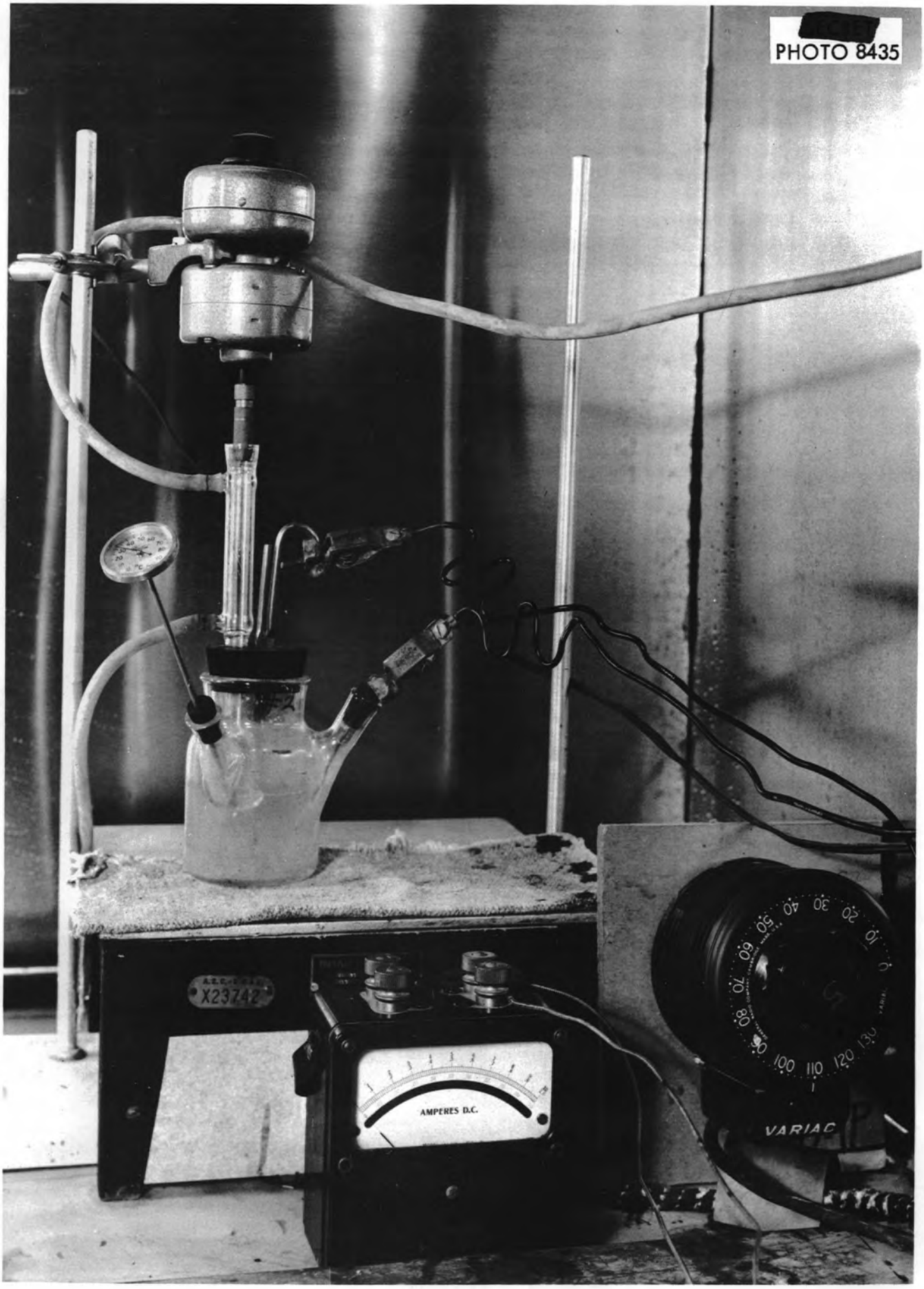

Fig. 3-1. Equipment Used in Laboratory Electrodecontamination Experiments

4.0 EFFECT OF VARIABLES

4.1 Current Density and Time

The use of low current densities for electrodecontamination of large stainless steel vessels is of practical importance since stainless steel is a relatively poor conductor of electricity and tends to overheat locally when high current densities are used. The decontamination rate was found to increase as the current density was increased over the range 0.01 to 0.03 amp/in.^{2} (see Table 4-1 and Fig. 4-1), but gross beta decontamination factors of 1000 or more were obtained over the entire range.

Decontamination was achieved in minimum time and with minimum electrical current at 0.03 amp/in.^{2}, although the corrosion rate was slightly higher than at 0.01 amp/in.^{2} (see Table 4-2). The time required for removal of more than 99% of the contamination (decontamination factor of 1000) varied from 20 to 24 hr at 0.01 amp/in.^{2} to about 3 hr at 0.03 amp/in.^{2}.

The corrosion rate increased between 0.01 and 0.1 amp/in.^{2} by a factor of 50 (see Table 4-2 and Fig. 4-2). Six type 347 stainless steel disks, 6 in.^{2} in area, were used as anodes in the tests. A current density of 0.01 to 0.1 amp/in.^{2} was used for an equivalent quantity of electricity, and the resulting metal losses were obtained by weighing. In another series of experiments on similarly contaminated samples, the total metal lost from the samples was somewhat less at 0.03 amp/in.^{2}, but the time required to give a decontamination factor of 1000 was also somewhat less (see Table 4-3).

The electrolytic treatment did not appear to harm the stainless steel surface. Three clean disks of type 347 stainless steel, 6 in.^{2}

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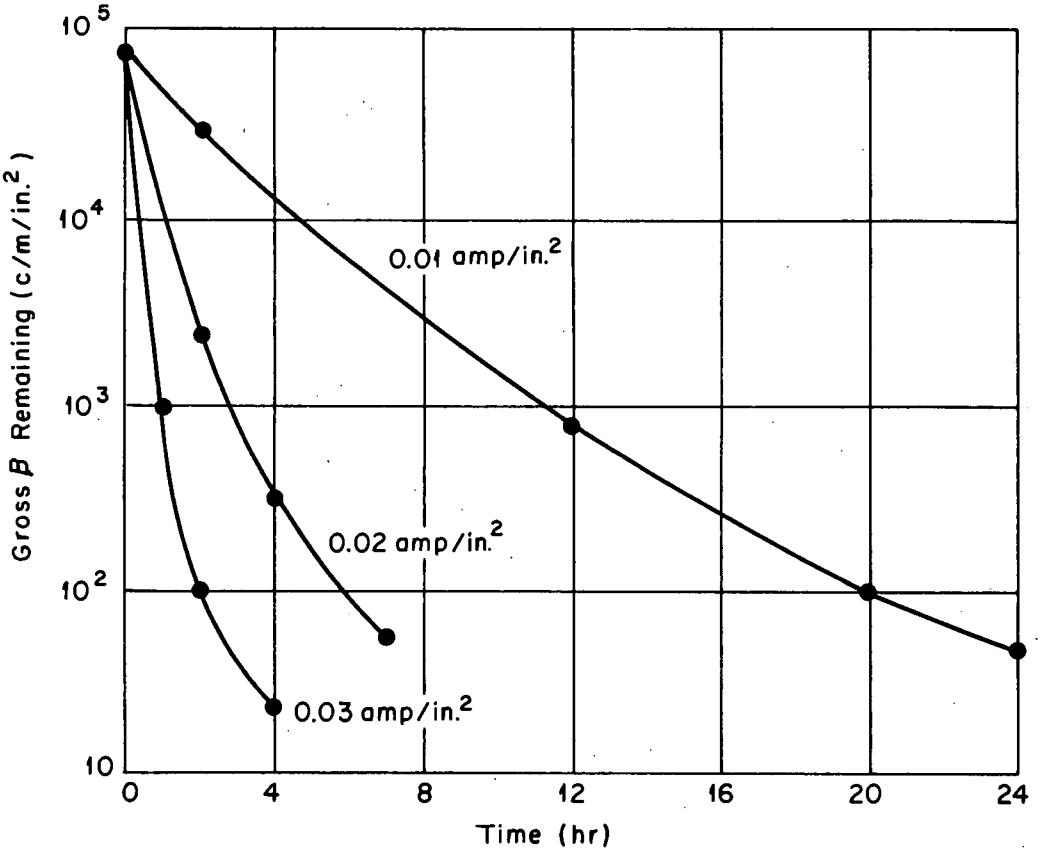

Electrolyte: 2% H_2SO_4
Temperature: 27°C
Initial gross β contamination of sample: 10^5 c/m/in.²
Cathode: stainless steel
Anode: samples of contaminated type 347 stainless steel
No agitation

Fig. 4-1. Effect of Current Density on Decontamination Rate of Contaminated Stainless Steel Samples.

Table 4-1

Effect of Current Density on Decontamination of Contaminated Stainless Steel

Electrolyte: 2% H_2SO_4
Temperature: 25°C
Cathode: stainless steel
Anode: samples of contaminated type 347 stainless steel
No agitation

Anode Current Density (amp/in.^{2})	Time (hr)	Electrical Current (coulombs/in.^{2})	Activity on Sample (c/m/in.^{2})	Cumulative Activity Removed (% of total)
0.01	0	---	5.0×10^4	---
	1.0	36	3.5×10^4	30.0
	2.0	72	2.7×10^4	46.0
	3.0	108	2.0×10^4	59.0
	4.0	144	1.6×10^4	68.0
	5.0	180	1.2×10^4	75.0
	10.0	360	3.0×10^4	94.0
	15.0	540	800	98.4
	20.0	720	200	99.6
	25.0	900	50	99.9
0.02	0	---	4.0×10^4	---
	0.5	36	1.8×10^4	53.8
	1.0	72	8.5×10^3	78.8
	1.5	108	3.9×10^3	91.2
	2.0	144	2.2×10^3	94.5
	3.0	216	700	99.0
	4.5	324	260	99.2
	5.0	360	200	99.5
	7.0	504	50	99.9
0.03	0	---	4.0×10^4	---
	0	36	1.6×10^4	58.8
	0.66	72	7.0×10^3	82.5
	1.0	108	3.5×10^3	91.0
	2.0	216	400	99.0
	3.0	324	60	99.8

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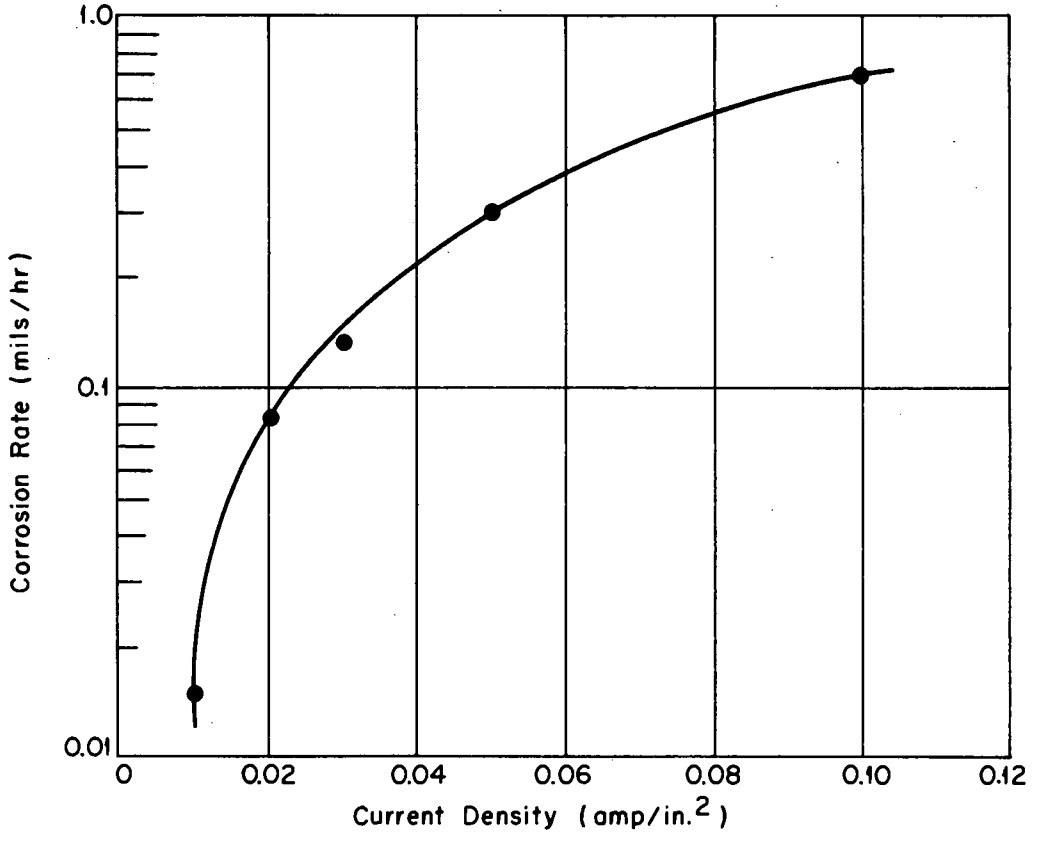

Electrolyte: 2 % H_2SO_4
Temperature: 27°C
Electrical energy: 300 coulombs/in.^{2}
Cathode: Stainless Steel
No agitation

Fig. 4-2. Effect of Current Density on Corrosion Rate of Contaminated Stainless Steel Samples.

Table 4-2

Effect of Current Density on Corrosion Rate of Stainless Steel Samples

Electrolyte: 2% sulfuric acid
Temperature: 30°C
Cathode: stainless steel
Anode: type 347 stainless steel disks, approximately 6 in.^{2}
Electrical current: 300 coulombs/in.^{2}
No agitation

Anode Current Density (amp/in.^{2})	Time (hr)	Corrosion Rate (mils/hr)
0	25	0
0.01	8.33	0.015
0.02	4.16	0.085
0.03	2.77	0.136
0.05	1.67	0.300
0.10	0.83	0.718

Table 4-3

Effect of Current Density and Time on Total Metal Loss from Contaminated Stainless Steel Samples

Electrolyte: 2% sulfuric acid
Temperature: 25°C
Cathode: type 347 stainless steel
Anode: contaminated type 347 stainless steel disks, approximately 6 in.^{2}, average β activity 5.0×10^4 c/m/in.^{2}
No agitation
Final decontamination factor of each sample: 1000

Anode Current Density (amp/in.2)	Time (hr)	Total Metal Loss (mils)
0.01	20-24	0.30-0.360
0.02	7-8	0.616-0.704
0.03	3-5	0.408-0.544

in area, were passivated by being used as anodes and were then electrolyzed at 0.02 amp/in.^{2} for 16 hr. Three other similar disks were passivated in the usual way by being held in 20% HNO_3 at 60°C for 30 min. All six disks were then suspended in 60% HNO_3 for 24 hr, and the weight loss of each was determined. There was no significant difference in the weight losses of the six samples; the electrolyzed samples were therefore just as resistant to 60% HNO_3 as the passivated samples (see Table 4-4).

Several samples of a welded section of 3/16-in. type 347 stainless steel which had been subjected to a pitting treatment in ferric chloride solution were used as anodes and electrolyzed over a current density range of 0.01 to 0.03 amp/in.^{2} for 8 hr. Microscopic examination of the sample showed that the electrolysis had caused no change either in the weld or in the size of the pits.

4.2 Cathode Material

If buildup of activity on the cathode during electrolytic decontamination can be prevented, frequent replacement of the cathode can be avoided. The amount of activity deposited on a stainless steel cathode was found to be approximately 1 to 5% of the total activity removed from the anode, while that deposited on a copper cathode was 30 to 35% of the activity removed (see Table 4-5). The only apparent disadvantage in using stainless steel as cathode material in preference to copper is its higher electrical resistance, which is approximately 53 times that of copper; however, the use of large-diameter stainless steel tubing filled with copper or aluminum or the use of copper or aluminum rod sprayed with stainless steel as the cathode might overcome this difficulty. These possibilities have not been tried.

It is thought that the reason for deposition of larger amounts

Table 4-4

Effect of Electrolysis on Resistance of Type 347 Stainless Steel to 60% Boiling HNO_3

Uncontaminated samples pretreated as indicated, weighed, suspended in 60% HNO_3 at room temperature for 24 hr, and reweighed; average penetration calculated from weight

Sample Pretreatment	Weight (g)			Penetration (mils/month)
	Initial	Final	Loss	
Electrolyzed at 0.02 amp/in.^{2} for 16 hr	10.9857	10.9491	0.0366	
	9.8993	9.8676	0.0317	
	10.6305	10.6002	0.0303	1.280 (avg.)
Passivated by holding in 20% HNO_3 at 60°C for 30 min	10.0761	10.0414	0.0347	
	11.1213	11.0822	0.0391	
	11.6530	11.6233	0.0297	1.340 (avg.)

Table 4-5

Effect of Cathode Material on Deposition of Activity on Cathode

Electrolyte: 2% H_2SO_4
Temperature: 25°C
Anode: samples of contaminated type 347 stainless steel average β activity 5.0×10^4 c/m/in.^{2}; activity removed from anode by end of experiment, >99%
No agitation

Cathode Material	Current Density (amp/in.^{2})	Time (hr)	No. of Runs	Electrical Current (coulombs/in.^{2})	Activity on Cathode (% of total removed from anode)
Copper	0.01	20.0	3	720	30-40
	0.03	3.0	2	324	30-40
Type 347 stainless steel	0.01	20.0	1	720	<2
	0.03	3.0	2	324	<1

activity on copper than on stainless steel might be due to the hydrogen overvoltage of the copper. Possibly, also, some of the dissolved copper was redeposited, carrying along some of the activity.

4.3 Agitation

The decontamination rate decreased somewhat when the electrolyte was agitated (see Table 4-6). No explanation is offered for this, but it is conceivable that lack of agitation may enhance ionic movement through the electrode films.

4.4 Temperature

The effect of temperature on the decontamination rate was found to be negligible (see Table 4-6), although slightly more rapid decontamination was obtained at higher temperatures. However, since an increase in the temperature tends to increase the corrosion rate and also necessitates heating the solution prior to electrolysis, it is considered preferable to use solutions at ambient temperatures.

4.5 Throwing Power of Solution and Shape of Cathode

The decontamination obtained in a given time was shown to decrease as the distance of the contaminated surface from the cathode increased (see Table 4-7). Electrolytic decontamination of the inner surface of a contaminated stainless steel dip leg, 24 in. long and 0.5 in. in diameter, was attempted. It was placed inside of and parallel to a steel pipe of larger diameter (see Fig. 4-3) in such a way that the larger pipe could be used as both the cell and the cathode. The dip leg was made the anode at a current density of 0.03 amp/in.^{2}. A 2% H_2SO_4 solution was used as the electrolyte. The dip leg was marked off in four 6-in.-long

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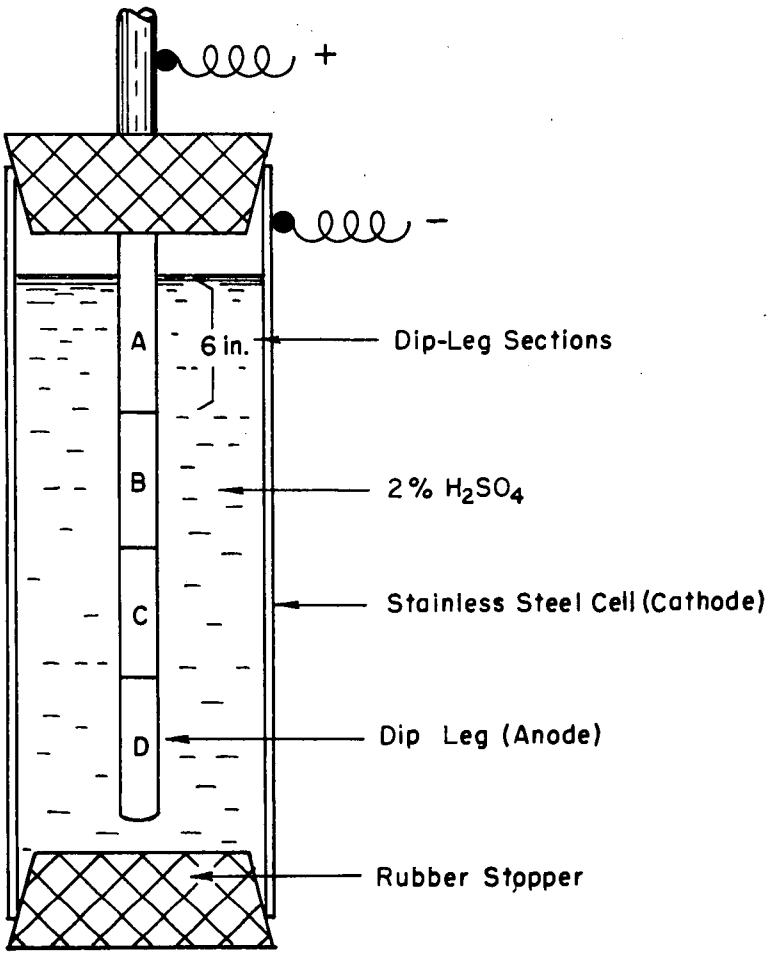

Fig. 4-3. Apparatus for Studying Throwing Power of Solution and Shape of Cathode.

Table 4-6

Effect of Temperature and Agitation on Decontamination Rate of Contaminated Stainless Steel Samples

Electrolyte: 2% H_2SO_4
Current density: 0.02 amp/in.^{2}

Conditions	Temp. (°C)	Time (hr)	Electrical Current (coulombs/in.^{2})	Activity on Sample (c/m/in.^{2})	Cumulative Activity Removed (% of total)
No agitation	25	0	---	8.0×10^4	---
		0.5	36	2.5×10^4	68.8
		1.0	72	1.1×10^4	86.3
		2.0	144	1.8×10^3	97.8
		3.0	216	500	99.4
		5.0	360	90	99.9
		7.0	504	50	99.96
Agitation	25	0	---	4.0×10^4	---
		0.5	36	2.1×10^4	47.5
		1.0	72	1.2×10^4	70.0
		2.0	144	2.9×10^3	92.8
		3.0	216	1.0×10^3	97.5
		5.0	360	450	98.9
		7.0	504	200	99.5
		9.0	648	90	99.8
Agitation	70	0	---	4.0×10^4	---
		0.5	36	1.9×10^4	52.5
		1.0	72	1.0×10^4	75.0
		2.0	144	2.6×10^3	93.5
		3.0	216	850	97.9
		5.0	360	300	99.3
		7.0	504	110	99.7
		9.0	648	60	99.9

Table 4-7

Effect of Throwing Power in 2% H_2SO_4 on Decontamination Factor

Initial activity of each section of dip-leg surface: ~200 mr/hr
Electrolyte: 2% H_2SO_4
Current density: 0.03 amp/in.^{2}
Time: 4 hr
Temperature: 25°C
No agitation

Pipe Section[a]	Approximate Final Activity of Section (mr/hr)	Decontamination Factor of Inner Surface
A	175	1.15
B	160	1.25
C	115	1.75
D	60	3.35

(a) Each section was about 6 in. long. Section A was the one at the top of the dip leg, i.e., the farthest from the cathode.

sections, and, at the end of 4 hr, readings at contact were made with a Cutie Pie on each section. Lead shielding was used to ensure that each reading gave the activity for the section desired. The decontamination factor of the inner surface of the section nearest the cathode was about 3 times that of the section farthest away.

The best decontamination of a surface would thus be obtained when all parts of the contaminated surface were exposed to the cathode. A cylindrical or rod-shaped cathode would be preferable for a cylindrical tank, particularly for a tank containing dip legs. For a permanent installation, which might be considered in design of new equipment, such cathodes might be permanently placed, or retractable electrodes might be used.

5.0 LARGE-SCALE TESTING

5.1 RaLa Equipment

Fairly successful electrodecontamination was obtained on three tanks from the RaLa No. 200 cubicle (see Table 5-1) which was reclaimed from the burial ground. The test was carried out in cooperation with the Operations Division at ORNL. The quantitative data are considered somewhat unreliable owing to the erratic background readings at various parts of the entire unit. These background readings are apparently due to activity held up in the piping system and valves.

The cubicle consisted of five tanks ranging from about 18 to 74 liters in volume, which were connected with numerous pipes and columns to a height of about 9 ft. Electrolytic decontamination was attempted on three. All lines, tanks, and columns were contaminated, and gave a high background reading at any point, 40 to 7000 mr/hr. Owing to the complexity of design of the unit, it was necessary to carry out decontamination operations on one tank at a time. The waste tank was treated first, it being the largest. Since cutting of pipe lines was not advisable, the maximum diameter of the cathode was limited to 1/8 in. Therefore, for conductivity purposes (see Sect. 4.2) copper was used for the cathode instead of stainless steel. The tank was filled with the electrolyte, 2% H_2SO_4. A 50-amp rectifier was used to obtain direct current.

Decontamination was carried out on the second and third tanks with the same 1/8-in.-diameter copper cathode. Other conditions were the same except the current densities.

Table 5-1

Electrolytic Decontamination of Three Tanks from No. 200 RaLa Cubicle

Electrolyte: 2% H_2SO_4
Temperature: 25°C
Cathode: type 347 stainless steel

Tank	Surface Area (in.^{2})	Anode Current Density (amp/in.^{2})	Current (amp)	Voltage (volts)	Cathode Current Density (amp/in.^{2})	Time (hr)	Avg. Activity Remaining (mr/hr)	Electrical Current (coulombs/in.^{2})
Waste	2200	0.015	33	9	6.8	0	1200	---
						20	30-40[a]	1080
Feed I	800	0.03	24	6.5	6.0	0	110	---
						5	65[a]	540
Feed II	800	0.03	24	6.5	6.0	0	450	---
						5	200	540
						9	180[b]	1026

(a) Probably background.

(b) Very high background in immediate area because of lines and valves attached to tank.

5.2 23 Process Evaporator Cover Plate

A stainless steel evaporator cover plate that had been used for three years in the 23 process was electrolytically decontaminated to approximately background (see Table 5-2). The plate was approximately 200 in.^{2} in area, and it had an activity of 5×10^3 mr/hr. One small spot that had been badly damaged physically was not decontaminated, but, when this spot was covered with lead, the total cover plate activity reading was that of background.

Table 5-2

Electrolytic Decontamination of Evaporator Cover Plate from 23 Process

Electrolyte: 2% H_2SO_4
Temperature: 25°C
Cathode: type 347 stainless steel
Surface area of cover plate: ~200 in.^{2}

Current Density (amp/in.^{2})	Electrical Current (coulombs/in.^{2})	Time (hr)	Activity (mr/hr)
0.01	---	0	5.0×10^3
0.01	36	1	3.45×10^3
0.01	72	2	2.6×10^3
0.01	144	4	1.55×10^3
0.01	216	6	650
0.01	288	8	400
0.01	360	10	300
0.01	432	12	250
0.01	504	14	250
0.03	720	16	225[a]
0.03	936	18	225[b]
---	---	18	30[c]

(a) Activity only in one small area.

(b) Electrolyte replaced with fresh.

(c) Radioactive spot shielded with lead.