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LABORATORY EVALUATION OF DECONTAMINATION AGENTS
FOR INCONEL PIPING CONNECTORS

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LABORATORY EVALUATION OF DECONTAMINATION AGENTS
FOR INCONEL PIPING CONNECTORS

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INTRODUCTION

This document reports results of laboratory tests of three decontaminating agents for Inconel piping connectors. During recent decontaminations of Hanford single-pass reactors, it was difficult to reduce the radioactivity of the Inconel connectors between the supply headers and the process tube nozzles. The aluminum and carbon steel components of the flow system were successfully decontaminated; however, very low decontamination factors (<2) were obtained on the connectors.

SUMMARY AND CONCLUSIONS

Laboratory tests were conducted to evaluate three candidate solutions for decontaminating Inconel piping connectors. The reagents tested included:

- An inhibited sulfuric-oxalic acid solution ($0.3M$ H_2SO_4 , $0.1M$ $H_2C_2O_4$)
- Sulfam-3*, a proprietary sulfamic acid base decontaminant
- Bisulf-16*, a proprietary sodium bisulfate base decontaminant.

The sulfuric-oxalic mixture proved to be the most effective; decontamination factors were 4.7 and 11.5 for solution temperatures of 70 and 80 °C at 30 min exposure. The decontamination factors for Sulfam-3 and Bisulf-16 at 70 °C and 30 min were only 2.9 and 1.8. Lower temperatures and reduced acid concentrations in the sulfuric-oxalic mixture were less effective.

The results of corrosion tests of sulfuric-oxalic mixtures on reactor components have been reported previously.⁽¹⁻³⁾ For a temperature of 70 °C and 30 min contact, the corrosion of aluminum would be <0.05 mil, of carbon steel <0.05 mil, and of stainless steel and Inconel

* Sulfam-3 and Bisulf-16 are local code names for two proprietary preparations.

<0.01 mil. These corrosion penetrations are not excessive, and no major corrosion problems are expected from the use of a properly inhibited 0.3M–0.1M, sulfuric-oxalic acid mixture.

DISCUSSION

Sample Preparation and Testing Procedures

The decontaminants were evaluated using 1 in. segments of contaminated Inconel connectors. The samples were brushed and rinsed to remove loose contamination, counted in a small lead shielded chamber, and stored in deionized water to prevent drying of the corrosion product film. Orientation markings, made on the samples before initial counting, insured that measurements of radioactivity were always taken at the same spot.

After initial radiation measurements were made, the samples were suspended on fine stainless steel wires in beakers of the decontaminating agent. The solutions were vigorously stirred during the testing period.

The samples were removed from solution at intervals of 5, 10, 20, and 30 min and rinsed to remove loosened material; the radiation level was then measured near each end of the sample. The radiation measurement data are presented in Table I.

Three decontaminating agents were evaluated in the laboratory studies; Sulfam-3, Bisulf-16, and various inhibited mixtures of sulfuric and oxalic acids. These agents were selected because they are approved for reactor use. The test sequence is shown in Table I.

Tests 1 through 3 were initial comparison tests of the three decontaminating agents. Tests 4 through 7 determined the effect of temperature on the efficiency of the sulfuric-oxalic mixture; Tests 8 and 9 determined the effects of reduced acid concentrations.

Test Results

The efficiency of a decontaminating agent is determined by calculating a decontamination factor or DF $\left(DF = \frac{\text{activity before decontamination}}{\text{activity after decontamination}} \right)$. Tests results are in Table I.

TABLE I
TEST CONDITIONS, RADIATION MEASUREMENTS, AND DECONTAMINATING AGENTS

Test	Decontamination Agent	Concentration and Temperature	Time, min	Sample Readings ^(a)				Average DF, 30 min Exposure
				counts/min	DF	counts/min	DF	
1	Sulfam-3 No Inhibitor	6 oz/gal 70 °C	0	20,000	-	28,000	-	2.86
			5	15,000	1.33	20,000	1.4	
			10	14,500	1.38	19,000	1.48	
			20	10,000	2.0	14,000	2.0	
			30	7,250	2.76	9,500	2.95	
2	Bisulf-16 No Inhibitor	6 oz/gal 70 °C	0	21,000	-	23,000	-	1.42
			5	21,000	1	20,000	1.15	
			10	19,000	1.1	20,000	1.15	
			20	14,000	1.5	17,000	1.35	
			30	16,000	1.3	15,000	1.53	
3	Sulfuric-Oxalic No Inhibitor	0.3M-0.1M 70 °C	0	7,000	-	19,000	-	18.2
			5	3,000	2.33	7,500	2.54	
			10	800	8.75	5,200	3.66	
			20	550	12.7	1,500	12.7	
			30	500	14	850	22.4	
4	Sulfuric-Oxalic 0.1% Inhib 21 ^(b)	0.3M-0.1M 80 °C	0	20,000	-	26,000	-	11.5
			5	14,000	1.43	18,000	1.44	
			10	8,300	2.41	9,700	2.68	
			20	4,300	4.65	5,500	4.72	
			30	1,800	11.1	2,200	11.8	
5	Sulfuric-Oxalic 0.1% Inhib 21	0.3M-0.1M 70 °C	0	20,000	-	23,000	-	4.82
			5	15,000	1.33	20,000	1.15	
			10	9,700	2.08	8,200	2.9	
			20	6,700	2.94	7,200	3.2	
			30	4,000	5	4,800	4.7	
6	Sulfuric-Oxalic 0.1% Inhib 21	0.3M-0.1M 60 °C	0	25,000	-	20,000	-	3.0
			5	17,000	1.47	17,000	1.18	
			10	16,000	1.56	15,000	1.25	
			20	14,000	1.79	8,100	2.47	
			30	9,000	2.7	6,000	3.33	
7	Sulfuric-Oxalic 0.1% Inhib 21	0.3M-0.1M 50 °C	0	25,000	-	24,000	-	1.69
			5	25,000	1	21,000	1.14	
			10	18,000	1.39	15,000	1.6	
			20	17,000	1.47	15,000	1.6	
			30	15,000	1.66	14,000	1.71	
8	Sulfuric-Oxalic 0.1% Inhib 21	0.3M-0.05M 70 °C	0	25,000	-	30,000	-	2.39
			5	17,000	1.47	17,000	1.76	
			10	15,000	1.67	17,000	1.76	
			20	14,000	1.78	15,000	2	
			30	11,000	2.27	12,000	2.5	
9	Sulfuric-Oxalic 0.1% Inhib 21	0.2M-0.1M 70 °C	0	25,000	-	19,000	-	2.11
			5	20,000	1.25	16,000	1.19	
			10	14,000	1.79	14,000	1.36	
			20	10,000	2.5	14,000	1.36	
			30	10,000	2.5	11,000	1.73	

(a) A reading was taken near each end of the sample.

(b) Inhib-21 is a local code name for a proprietary inhibitor.

The DF's observed for Sulfam-3 and Bisulf-16 were well below 4 and were considered unsatisfactory. The high DF for the sulfuric oxalic mixture dictated further investigation. Tests 4 through 7 demonstrated a very strong dependence on temperature, indicating that a successful in-reactor decontamination using this mixture would require careful temperature control. The decrease in decontamination factors observed between Test 3 and Tests 4 and 5 is explained by the presence of the proprietary thiourea-base inhibitor (Inhib-21).

Reactor decontaminations require large quantities of chemicals and generally represent a substantial investment. To reduce chemical costs, lower acid concentrations were evaluated to determine the minimum acceptable sulfuric-oxalic concentration. Tests 8 and 9 showed that lower concentrations at the same exposure and temperature are unsatisfactory.

The results of laboratory tests are shown on a McMillan chart⁽⁴⁾ of DF, time, solution concentration, and temperature (Figure 1). Results for any given condition can be determined from Figure 1. Starting with contact time on the lower right margin, a line is drawn vertically to the solution concentration, horizontally to the temperature, and vertically to the resulting DF. An important relationship between temperature and time (for a given solution) is indicated by the breaks in the higher-temperature lines. The relative efficiency of the solutions can be determined from the slope of the solution lines; a steep slope indicates an effective decontaminating solution.

The data in Figure 1 and Table I show that the 0.3M-0.1M sulfuric-oxalic acid mixture is the most effective of the solutions tested for decontamination of Inconel connectors. For reactor use, a minimum temperature of 70 °C should be maintained.

Corrosion

It is obvious that excessively corrosive decontaminating agents are undesirable. Data from both laboratory and pilot plant studies⁽¹⁻³⁾ indicate the following maximum corrosion rates would be expected for a 30 min decontamination at 70 °C using an inhibited 0.3M sulfuric-0.1M oxalic acid mixture:

Aluminum	0.04 mil
Zircaloy-2	nil
Carbon Steel	0.05 mil
Stainless Steel	0.008 mil
Inconel X	0.004 mil

These corrosion rates are not considered excessive.

FIGURE 1
McMillan Chart of Laboratory Decontamination Factors

REFERENCES

1. J. E. Mendel. Laboratory Investigation of Decontaminating Solutions for Present Reactors, HW-67037. September, 1960.
2. J. A. Ayres, T. F. Demmitt, A. P. Larrick, L. D. Perrigo, R. B. Richman, and R. D. Weed. Decontamination Studies for HAPO Water-Cooled Reactor Systems: Progress Report - September 1, 1960 through September 1, 1961, HW-71259. February 16, 1962.
3. J. A. Ayres, T. F. Demmitt, A. P. Larrick, G. E. Neivaaur, L. D. Perrigo, R. B. Richman, and R. D. Weed. Decontamination Studies for HAPO Water-Cooled Reactor Systems: Progress Report - January 1, 1960 through September 1, 1960, HW-67937. December 27, 1960.
4. McMillan. "Fuels Steam Power, Trans. ASME, vol. 51, p. 349. 1929.

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