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REACTOR EFFLUENT ACTIVITY REDUCTION
PROCESS WATER TREATMENT TESTS:
PART 2 - CORROSION EFFECTS

11

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E. J. Nesselson
Reactor Engineering Unit
Research and Engineering Section
IRRADIATION PROCESSING DEPARTMENT

October 9, 1961

HANFORD ATOMIC PRODUCTS OPERATION
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INTRODUCTION

The intent of the production test⁽¹⁾ that occasioned this study was to determine if overfeeding^(a) of a coagulant having little or no sulfur present as a main constituent or impurity and consequent lower levels of parent material - P^{31} and S^{32} - in process water would result in lower levels of radionuclides in reactor effluent water - e.g., P^{32} - that constitute a potential limitation to reactor operations. Aluminum nitrate nonahydrate - $Al(NO_3)_3 \cdot 9H_2O$ (ANN) - was the reagent chosen. Inasmuch as substitution of ANN for alum represented a change in the coagulant added to process water, provision was made to monitor fuel cladding and process tube corrosion rates. This report documents the corrosion data obtained during the test period. The radiological effects of this test have been separately reported.⁽²⁾

SUMMARY AND CONCLUSIONS

Twenty columns of weighed metal were charged at 105-H into twenty aluminum process tubes that were probologged before and after the in-reactor residence time of each charge. Four charges (two on each side) reached an exposure of 155 MWD/T, and six (three on each side) were irradiated to 560 MWD/T. After discharge of the six-column subplot, the test was terminated. The remaining ten columns (five on each side) were irradiated to variable goal (720 MWD/T). The water coagulant and/or process water turbidity were different for each of these irradiation periods.

Visual examination of the irradiated fuel indicated the following:

1. The presence of 0.012 ppm process water turbidity associated with ANN coagulation inhibited deposition of iron oxide film on test elements in-reactor for one month.
2. The presence of 0.006 ppm process water turbidity associated with alum coagulation did not inhibit deposition of film on control elements in-reactor for one month.
3. The presence of 0.005 ppm process water turbidity associated with either ANN or alum coagulation permitted iron oxide film to deposit and accumulate on the surface of both control and test elements at about the same rate.

- 1) Hall, RB and EJ Nesselson. Production Test IP-347-A, Use of Aluminum Nitrate in Place of Aluminum Sulfate for Water Treatment, HW-66295. 8/2/60. Unclassified.
- 2) Hall, RB. Reactor Effluent Activity Reduction Process Water Treatment Tests, Part 1 - Radiological Effects, HW-68521. 2/5/61. ~~CONFIDENTIAL~~.
- a) Overfeeding refers to a coagulant feed rate in excess of that required to produce a well-formed floc that will adsorb and agglomerate virtually all the colloidal and suspended matter present in raw water, e.g., 20 ppm when 5 ppm is sufficient for removal of turbidity.

Comparative weight loss data for metal located in both halves of the reactor for similar irradiation intervals and tube powers indicate little or no significant differences under the different water quality conditions that were obtained. Probolog measurements of the monitored process tubes indicated no real difference in tube corrosion rates as a function of process water coagulant or turbidity that prevailed during the test period.

EXPERIMENTAL DESIGN

Twenty columns of weighed metal from one lot of X-8001 Alcoa-clad OIIN fuel elements were charged in 105-H (ten on the near side and ten on the far side). Prior to charging, each of the twenty tubes was probologged; at the end of the irradiation period, each tube was reproblogged. Irradiation conditions for each column and water quality that prevailed during the testing interval are shown below.

TABLE I

Date Dis- charged*	No. of Col- umns	Columns from Each		Average Cumulative Exposure MWD/T	Coincident Process Water Treatment and Quality ⁽³⁾			
		Side of Reactor			Near (Control)		Far (Test)	
		Near	Far		Coagulant	Turbidity	Coagulant	Turbidity
		(Control)	(Test)		ppm	ppm	ppm	ppm
11/7/60	4	2	2	155	5 Alum	0.006	26 ANN	0.012 ⁽⁴⁾
12/26/60	6	3	3	560	5 Alum	0.005	20 ANN	0.005
Test terminated 12/27/60.								
1/24/61	10	5	5	720	Same on both sides			

RESULTS

A. Fuel Element Weight Loss

1. Exposure to 155 MWD/T

Tube operating conditions and slug weight loss for metal that was irradiated for 28 days are shown in Table II. It is noted that none of the fuel elements experienced a weight loss of, or in excess of, 1.00 gram. In general, weight losses of less than 1 gram are regarded as too small to permit conversion to a cladding corrosion rate. The averages for nine pieces of data from each column are from 0.43 to 0.50 gram/element on the control side of the reactor, and from 0.21 to 0.26 gram/element on the test side of the reactor.

* All metal charged 10/10/60.

3) Hall, RB. Reactor Effluent Activity Reduction Process Water Treatment Tests, Part 1 - Radiological Effects, HW-68521. 2/15/61. [REDACTED]

4) Ibid., estimated value, Footnote 2 in Table II.

DECLASSIFIED

-5-

HW-71280

TABLE II

Tube Operating Conditions & Fuel Element Weight Loss (Grams)
Production Test IP-347-A

Reactor Side and Coagulant	<u>Near: 5ppm Alum</u>		<u>Far: 26ppm ANN</u>	
Tube Number	<u>1659-H</u>	<u>2660-H</u>	<u>1690-H</u>	<u>2685-H</u>
Fuel Element Series	91T	2U	93T	1U
Average Tube Power, KW	780	920	790	905
Average Tube Outlet Temperature, °C*	97	102	87	98
Average Tube Inlet Temperature, °C*	15	15	15	15
Average Coolant Flow Rate, gpm	44	45	45	45
Exposure, MWD/T	158	163	141	157

<u>Piece No. from Rear</u>	<u>ΔW</u>	<u>ΔW</u>	<u>ΔW</u>	<u>ΔW</u>
1	0.29	0.46	0.06	0.12
3	0.51	0.60	0.16	0.63
5	0.25	0.58	0.28	0.42
7	0.37	0.52	0.34	0.34
9	0.48	0.62	0.26	0.11
11	0.91	0.64	0.54	0.23
13	0.32	0.32	0.01	+0.14
15	0.52	0.40	--	0.29
17	0.27	0.40	0.10	0.34
Aver.	0.43	0.50	0.21	0.26

* Temperature data available for 6 of the 28 days metal was irradiated.

DECLASSIFIED

HW-71280

2. Exposure to 560 MWD/T

Tube operating conditions and weight loss for metal that was irradiated for 76 days (the total elapsed time from charging of the metal to termination of the test) are shown in Table III. The averages for nine pieces of weight loss data from each column on the control (near) side of the reactor range from 2.14 to 2.47 grams/element; average weight loss data for an equal number of fuel elements on the test (far) side of the reactor range from 1.22 to 2.85 grams/element.

Heat flux and cladding surface temperature are two of the more important variables affecting fuel element corrosion rate; and this information is not reflected in the data contained in Table III. A more meaningful presentation of the data is given below.

3. Agreement Between Observed and Calculated Corrosion at 560 MWD/T

Figure 1 shows observed versus calculated corrosion for the six columns of metal (three on each side of 105-H) that were exposed to 560 MWD/T. Lines of 200, 100, and 50 percent of perfect agreement are indicated. The calculated weight losses were obtained as follows:

- a. Determine fuel element heat flux and surface temperature for both annulus and hole.
- b. Determine a cladding corrosion rate for both the annulus and hole.
- c. Convert corrosion rates to an equivalent weight loss, knowing fuel dimensions and irradiation period.

Generally, an observed corrosion loss that ranges within 1/2 to 2 times a predicted amount of corrosion is considered satisfactory agreement. It is noted that most of the values fall within the band encompassing this range. A total of 52 pieces of data are presented; two of them fall on the perfect agreement line. The remaining 50 are distributed as follows:

<u>Position with Respect to Perfect Agreement</u>	<u>Source of Data</u>		
	<u>Total</u>	<u>Control</u>	<u>Test</u>
Observed > Calculated	13	8	5
Observed < Calculated	37	18	19

Almost three times as many fuel elements (37 vs. 13) experienced less than a predicted amount of corrosion as those that sustained more than a predicted amount. Since the number of control pieces in each classification equals or exceeds the number of test pieces, one concludes that the ANN-cooled fuel elements did not corrode at a more rapid rate than the alum-cooled fuel elements. Sufficient data is not available, however, to conclude the opposite, i.e., that ANN-cooled cladding corroded less than alum-cooled cladding.

TABLE III

Tube Operating Conditions and Fuel Element Weight Loss (Grams)
Production Test IP-347-A

Reactor Side and Coagulant	Near: 5 ppm Alum			Far: 20 ppm ANN		
Tube Number	<u>1458-H</u>	<u>1663-H</u>	<u>3463-H</u>	<u>1483-H</u>	<u>1684-H</u>	<u>3486-H</u>
Fuel Element Series	92T	96T	4U	88T	94T	7U
Average Tube Power, KW	900	1000	975	935	900	990
Average Tube Outlet Temperature, °C*	97	98	97	94	98	99
Average Tube Inlet Temperature, °C*	12	12	12	12	12	12
Average Coolant Flow Rate, gpm	45	47	46	46	45	46
Exposure, MWD/T	542	574	565	544	559	579

<u>Piece No. from Rear</u>	<u>ΔW</u>	<u>ΔW</u>	<u>ΔW</u>	<u>Average, 3 Values</u>	<u>ΔW</u>	<u>ΔW</u>	<u>ΔW</u>	<u>Average, 3 Values</u>
1	0.81	1.19	0.65	0.88	0.73	1.62	0.77	1.04
3	0.96	1.34	1.59	1.30	--	3.67	1.10	2.39
5	1.82	2.36	3.25	2.48	1.92	4.74	1.88	2.84
7	2.45	3.23	3.12	2.94	2.00	2.79	6.84	3.87
9	3.12	2.78	3.02	2.97	1.58	2.08	5.12	2.92
11	4.27	3.82	3.77	3.95	1.45	1.30	4.36	2.37
13	3.08	2.90	3.13	3.04	1.13	1.13	3.17	1.81
15	2.03	2.34	2.23	2.20	0.44	0.60	1.00	0.68
17	0.79	2.35	1.23	1.46	0.53	0.07	1.47	0.69
Average	2.14	2.47	2.44		1.22	2.00	2.85	

* Temperature data available for 54 of the 76 days metal was irradiated.

DECLASSIFIED

HM-71280

DECLASSIFIED**B. Visual Examination of Irradiated Fuel****1. Exposure to 155 MWD/T**

Coincident with the discharge of metal that reached an exposure of 155 MWD/T, it was discovered that a microphotometer used for water plant control was out of calibration and had been providing "low" turbidity values; thus, filter runs were prolonged beyond a desirable operating period, and breakthrough of previously accumulated floc was taking place. The microphotometer was re-calibrated and water plant control re-established before further irradiation took place.

The author observed that upstream pieces of all four columns were clean; that is, no dark or colored film was present on the lateral surface of the fuel elements, and they had a dull, smooth aluminum oxide film similar to that of a pre-irradiated fuel element. Downstream pieces of the CONTROL columns had a characteristic, uniform, light-to-medium red film; however, downstream pieces of the TEST columns had NO film and were similar in appearance to the upstream pieces. As shown in Table I, the concentration of turbidity was twice as much (estimate of 0.012 ppm vs. 0.006 ppm) on the test side as on the control side of the reactor. A reasonable inference stemming from these values is that the more turbid coolant prevented deposition and accumulation of film on the downstream test metal. However, since the coolant was coagulated with aluminum nitrate, the possible influence of a high nitrate environment, or the combined effect of nitrate plus turbidity, could not be ruled out as a possible cause for this phenomenon.

2. Exposure to 560 MWD/T

Inspection of the six columns of metal that achieved this exposure revealed that ALL downstream pieces in EVERY charge were coated with SOME film. (This metal remained in-reactor for a seven-week period after process water turbidity control was re-established.) It was noted that the last five downstream fuel elements in the CONTROL columns had a THICKER layer of iron oxide film than the last five downstream pieces in the TEST columns.

These observations indicate that low concentrations of turbidity derived from filter breakthrough can prevent the accumulation of non-oxide film on fuel elements; and, if subsequently cooled with less turbid or turbid-free process water, iron oxide film deposits and accumulates at about the same rate that prevails on the control side of the reactor.

C. Probolog Measurements of Process Tubes

Table IV shows the results of probolog measurements that were made at the time weighed metal was charged, and again when the weighed fuel was discharged from those tubes. The data in Column 12 - the indicated corrosion rates - are subject to some definition and qualification before interpretation of the values is meaningful; i.e., an "0" means the reported value for the minimum wall thickness did not change between readings and so the rate is

DECLASSIFIED

-9-

HW-71280

reported as zero; a "?" means the reported value for the minimum wall thickness after fuel discharge is higher than the value obtained before charging, hence no reduction or a negative corrosion rate. Perhaps some even more important considerations are: 1) With what precision can an experienced probolog operator interpret a trace? 2) How reproducible is the trace? and 3) If a real difference in minimum wall thickness seems indicated from successive probologgings made after each metal cycle, how sure is one where the point of minimum wall thickness has remained at the same position? In general, a minimum wall thickness value can be ± 3 mils from the data reported.⁽⁵⁾

Keeping all these qualifications in mind, what do the tube corrosion rates in Column 12 signify? The first four values (28 calendar days between measurements) indicate that no detectable effect took place (corrosion rate either zero or negative). During this period, the two far side, if not all four, tubes were cooled with "turbid" process water.

The next six values (76 calendar days between measurements) indicate that little, if any, detectable effect took place (maximum wall thickness change of 3 mils on tube 1684-H, and the value reported could deviate from a true value by as much as 3 mils⁽⁵⁾). These six tubes were contacted with turbid-free coolant; three with aluminum sulfate-coagulated process water, and three with aluminum nitrate-coagulated process water. Hence, it would appear that no change in process tube corrosion rate is attributable to the coagulant substitution.

As further confirmation of this inference, reference to the last ten values may be enlightening. The value for tube 1464-H is so far afield from the others that the cause for this rapid corrosion has to be attributable to something other than the water; furthermore, it is located on the control side of the reactor and was cooled with alum-coagulated water only. Hence, three values remain to indicate tube corrosion on the control (near) side of the reactor, and four values are available on the test (far) side. It is noted that these figures range from 0 to 1 mil/year and 0 to 5 mils/year, respectively. Reference to Table I shows that water coagulant and quality for the latter part of this in-reactor interval (26 days of a total of 102 days) were the same. This would seem to indicate that there is no basis in fact for differences in tube corrosion rates on both halves of the reactor, or that the indicated difference stems from after-effects of the coagulant change. Since corrosion rate differences were small while the coagulants were dissimilar, the likelihood that terminating the difference would "shock" or accelerate corrosion of test tubes seems rather remote.

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⁵⁾ Private Communication, DE Strege.

DECLASSIFIED

EW-71280

TABLE IV

In-Reactor Measurements of Process Tube Corrosion, H Reactor
(All Tubes Charged with Weighed Metal on 10/10/60)
Production Test IP-347-A

Tube No. (1)	Location in Reactor (2)	Mo & Yr(6) In-stalled (3)	Date Probologged Prior to Test(6), if Applicable (4)		Probolog Measurements on Charging of PT IP-347-A (7)		Probolog Measurements on Discharging of PT IP-347-A Metal from That Tube (9)		Δ WT (7-9) (10)	Time Elapsed Yrs** (11)	CR mils/yr (10 ÷ 11) (12)
			WT,* mils (5)	Date (4)	WT,* mils (7)	Date(7) (6)	WT,* mils (9)	Date (8)			
1659	Near	1/59	N.A.	N.A.	10/10/60	55	11/8/60 ⁸	55	0	.077	0
2660	Near	5/57	N.A.	N.A.	10/10/60	43	11/8/60	45	-2	.077	?
1690	Far	1/59	N.A.	N.A.	10/10/60	50	11/8/60	53	-3	.077	?
2685	Far	7/56	N.A.	N.A.	10/10/60	32	11/8/60	33	-1	.077	?

1458	Near	7/56	N.A.	N.A.	10/10/60	50	12/28/60 ⁹	48	2	.21	9.3
1663	Near	7/56	6/60	45	10/10/60	42	12/28/60	41	1	.21	4.7
3463	Near	7/56	6/60	44	10/10/60	37	12/28/60	37	0	.21	0
1483	Far	7/56	N.A.	N.A.	10/10/60	37	12/28/60	36	1	.21	4.7
1684	Far	9/56	N.A.	N.A.	10/10/60	40	12/28/60	37	3	.21	14.0
3486	Far	2/59	N.A.	N.A.	10/10/60	43	12/28/60	42	1	.21	4.7

1464	Near	6/60	N.A.	N.A.	10/10/60	55	1/24/61 ¹⁰	42	13	.28	46(?)
1665	Near	10/57	N.A.	N.A.	N.A.	N.A.	1/24/61	45	N.A.	.28	N.A.
2664	Near	7/56	N.A.	N.A.	10/10/60	32	1/24/61	32	0	.28	0
2667	Near	4/56	N.A.	N.A.	10/10/60	28	1/24/61	27	1	.28	3.6
3465	Near	9/56	8/60	44	10/10/60	40	1/24/61	40	0	.28	0
1481	Far	6/60	N.A.	N.A.	10/10/60	55	1/24/61	53	2	.28	7.1
1678	Far	3/56	11/57	50	10/10/60	34	1/24/61	32	2	.28	7.1
2678	Far	3/57	N.A.	N.A.	10/10/60	33	1/24/61	28	5	.28	17.8
2681	Far	7/54	6/60	29	10/10/60	25***	1/24/61	53	N.A.	.28	N.A.
3480	Far	5/56	6/60	43	10/10/60	38	1/24/61	38	0	.28	0

* Minimum wall thickness at A point along the actively corroding length of the tube.

** Calendar days between probologging ÷ 365; 10/10 - 11/8 = 28; 10/10 - 12/26 = 76;
 10/10 - 1/24/61 = 102.

*** Replaced 10/60.

6) Production Scheduling. H Reactor Tube Corrosion Listing. 9/1/60. Unclassified.

7) Strege, DE. In-Reactor Measurements of Process Tube Corrosion, H-Reactor, Report 54, HW-67133. 10/20/60. Unclassified.

8) Strege, same title, HW-67369. 11/9/60. Unclassified

9) Strege, same title, Report 69, HW-67950. 12/29/60. Unclassified.

10) Strege, same title, Report 8, HW-68255. 1/24/61. Unclassified.

DECLASSIFIED

-11-

HW-71280

ALUM control	ANN test
○ 1458-H	● 1483-H
△ 1663-H	▲ 1684-H
□ 3463-H	■ 3486-H

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FIGURE 1

AGREEMENT BETWEEN OBSERVED AND CALCULATED CORROSION OF FUEL ELEMENT CLADDING COOLED WITH ALUM AND ANN - COAGULATED PROCESS WATER

**DATE
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8/22/94

END