**Prediction Equations for
Corrosion Rates of A-537 and
A-516 Steels in Double Shell
Slurry, Future Purex, and
Hanford Facilities Wastes**

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June 1985

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Pacific Northwest Laboratory
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PREDICTION EQUATIONS FOR CORROSION RATES
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ABSTRACT

Even though the interest in the corrosion of radwaste tanks goes back to the mid-1940's when waste storage was begun, and a fair amount of corrosion work has been done since then, the changes in processes and waste types have outpaced the development of new data pertinent to the new double shell tanks. As a consequence, Pacific Northwest Laboratory (PNL) began a development of corrosion data on a broad base of waste compositions in 1980.

The objective of the program was to provide operations personnel with corrosion rate data as a function of waste temperature and composition. The work performed in this program examined A-537 tank steel in Double Shell Slurry and Future PUREX Wastes, at temperatures between 40 and 180°C as well as in Hanford Facilities Waste at 25 and 50°C.

In general, the corrosion rates were less than 1 mpy (0.001 inch/yr) and usually less than 0.5 mpy. Excessive corrosion rates (> 1 mpy) were only found in dilute waste compositions or in concentrated caustic compositions at temperatures above 140°C. Stress corrosion cracking was only observed under similar conditions.

The results are presented as polynomial prediction equations with examples of the output of existing computer codes. The codes are not provided in the text but are available from the authors.

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INTRODUCTION

The interest in the corrosion of radwaste tanks goes back to the mid-1940's when waste storage was begun. Test programs related to the bismuth phosphate and PUREX processes were conducted both in the laboratory and in the field (in-tank). These early tests were conducted in solutions with a pH of 6 to 13 and, in the case of field tests, at temperatures estimated to be between 250 and 300°F (120-150°C). Uniform corrosion rates up to 2.5 mpy (65 $\mu\text{m}/\text{y}$) were measured in both the liquid and vapor phases; pitting was also observed.

In 1980, an environmental impact statement (EIS) was prepared for the 241-AN and AW double shell waste storage tanks.⁽¹⁾ The data used to prepare the EIS were taken from the corrosion literature from Savannah River experiments and from Hanford work. During preparation of the EIS, the data were found to be inadequate for describing the corrosion behavior of the waste proposed for storage in the new tanks. It was concluded that further corrosion rate data were needed to properly define the operating limitations on the tanks. It was possible, however, to state that, based on limited data, the double shell slurry (DSS) specified in the EIS could be put in the tanks without additional testing, provided the temperatures were to be kept close to or below the proposed operating values.

The development of more relevant corrosion data required initiating tests of tank construction material over a range of compositions and temperatures. The major emphasis was placed on temperature at or below 100°C because that was the primary operating range. Tests up to at least 350°F (177°C) were called for because that was the nominal design temperature at which corrosion rates were not expected to exceed one mil per year.

This document provides a summary of the corrosion work performed in DSS waste at temperatures at or below 180°C (356°F). The prediction equations developed are subject to revision as additional data are

generated. However with over 3000 data points in over 60 waste compositions, no changes are expected which will significantly affect the results.

The report is divided into three parts:

- Part 1 - Experimental Considerations
- Part 2 - Data Analysis Methodology
- Part 3 - Results

PART 1 - EXPERIMENTAL CONSIDERATIONS

The double shell slurry (DSS), Future PUREX (FP), and Hanford Facilities (HF) wastes contain radioactive components. Under the normal operating conditions, the radiation level at the tank walls is expected to be less than about 1000 R/h. Dose rates this low are not expected to significantly impact the corrosion rate in these wastes. Therefore, for simplicity, all tests were performed using synthetic waste materials prepared in accordance with instructions from Rockwell Hanford Operations. The compositions used fell into the ranges given in Tables 1, 2, and 3.

As a consequence of their radioactive constituents, the wastes are stored in nominal one million gallon (3.8 Ml) underground tanks. The new generation of tanks are double wall construction using ASTM A-537 Class I carbon steel plate. The tanks are 76 feet (22.9 m) in diameter (inner tank) and are designed for a maximum waste temperature of 350°F (177°C). The tank walls are expected to have a maximum temperature of 200°F (93°C). After construction, the tanks are stress relieved at 1100°F (593°C) for one hour.

The steels used in the test program included both the A-537 currently used in tank construction and A-516 used in the older tanks. A-516 was included in the testing to provide continuity with past work. The steel compositions are given in Table 4.

This part of the report incorporates a discussion of materials, chemistry, and the methodology of experimental design. It also contains general commentary on the state of knowledge of the corrosion mechanism, the decomposition processes of the organic components in the waste, and stress corrosion cracking in the waste compositions.

WASTE COMPOSITIONS

The composition ranges given in Tables 1 and 2 were used to design test matrices. Constraints placed on the test design were that three time periods, four, eight, and twelve months, and four (for DSS), three (for FP), or two (for HF) temperatures would be used. The resulting waste composition test matrices, designed by the methods described below in EXPERIMENTAL DESIGN, are given in Tables 5 to 8.

TABLE 1. Double Shell Slurry Composition Range

	COMPOSITION Range (MOLARITY) <u>Low - High</u>
OH^-	0.5 - 10.0
NO_3^-	1.0 - 8.0
NO_2^-	0.2 - 10.0
AlO_2^-	0.0 - 5.0
$\text{CO}_3^{=}$ (2 fixed values)	0.2 or 0.25
$\text{PO}_4^{=}$	0.0 - 2.0
EDTA/HEDTA	0.0 - 1.0
Citrate (3 fixed values)	0.0, 0.2, or 0.6
$\text{SO}_4^{=}$ (single value)	0.05

TABLE 2. Range of Composition of PUREX Related Wastes

<u>Waste</u>	<u>OH^-</u>	<u>Al^a</u>	<u>NO^-</u>	<u>NO_2^-</u>	<u>$\text{CO}_3^{=}$</u>	<u>$\text{SO}_4^{=}$</u>	<u>$\text{Cit}^{=b}$</u>	<u>F^-</u>	<u>K^+</u>
PUREX Sludge	.2-.8	-	.02-.1	.01-.05	.01-.05	s	-	1-1.5	.2-.5
Declad. Slurry	3.-8.	-	.2-.5	-	s	s	-	s(<1.)	2.-3
CAW (Opt. A)	.5-1.	.5-1.	2.-4.	1.-2.	-	s	-	.1-.2	-
CAW (Opt. B)	.001-.1	.5-1.	3.-s	1.-2.	-	s	1.-2.	.15-.3	-
CAW (Opt. C)	.5-1.	.5-.1	3.-s	1.-2.	s	s	-	.15-.3	-

^aAl may be aluminum hydroxide or sodium aluminate, depending on alkalinity.

^bCit = Citrate.

^sSaturated, i.e., < 0.5 M for $\text{CO}_3^{=}$ or $\text{SO}_4^{=}$, < 4 M for NO_2^- .

TABLE 3. Hanford Facilities Waste
Composition Range

OH^-	0.0001 - 2 M
NO_2^-	0 - 0.01
$\text{PO}_4^{=}$	0 - 5
$\text{SO}_4^{=}$	0 - 2.9

TABLE 4. Alloy Compositions (wt %)

	<u>C</u>	<u>Cr</u>	<u>Cu</u>	<u>Mn</u>	<u>Mo</u>	<u>Ni</u>	<u>P</u>	<u>S</u>	<u>Si</u>
<u>ASTM A-537 Nominal</u>	.24M*	.25M	.35M	.65-1.4	.08M	.25M	.035M	.04M	.13-.55
Heat 1**	.15	.15	.20	1.26	<.01	.20	.026	.007	.43
Heat 2	.15	.19	.26	1.20	.04	.13	.008	.008	.25
<u>ASTM A-516 Nominal</u>	.27M			.8-1.25			.035M	.04M	.13-.33
Heat 1	.13	.03	.10	.89	<.01	.05	.009	.020	.26
<u>ASTM A-307 Nominal (Used in U-bends)</u>									
Bolt							.06M	.15M	
Laboratory	0.10	.024	.11	.43	.01	.07	.019	.020	.19
Nut							.13M	.23M	
Laboratory	.067	.005		.44		.01		.10	.08

* M - Maximum.

** Laboratory Measurements.

TABLE 5. RHO Tank Corrosion Data Base
(Number of Coupons)

Experiment	Months of Exposure	Temperature [C]	Alloy		
			A-516	A-537	Total
Hanford Facilities Waste (HF) 22 Compositions	4	25	22	43	65
		50	22	44	66
	8	25	22	44	66
		50	22	44	66
	12	25	22	44	66
		50	22	44	66
	4	140	27	55	82
		180	28	56	84
	8	140	27	56	83
		180	29	58	87
	12	140	28	55	83
		180	27	53	80
Double Shell High Temperature (DSS HT) 28 Compositions	4	40	56	28	84
		60	56	28	84
		80	56	28	84
		100	56	0	56
	8	40	28	56	84
		60	28	56	84
		80	28	56	84
		100	28	56	84
	12	40	18	28	46
		60	56	28	84
		80	56	28	84
		100	56	28	84

TABLE 5. RHO Tank Corrosion Data Base
(Number of Coupons) [continued]

Experiment	Months of Exposure	Temperature [C]	Alloy		
			A-516	A-537	Total
Double Shell Remake Test (DSS RT) 15 Compositions	6	100	15	30	45
	8	100	15	30	45
	12	100	15	30	45
	4	140	23	46	69
		180	22	46	68
Future PUREX High Temperature (FP HT) 23 Compositions	8	140	22	46	68
		180	23	46	69
	12	140	22	43	65
		180	23	45	68
	4	40	36	72	108
		70	36	71	107
		100	36	72	108
Future PUREX Low Temperature (FP LT) 36 Compositions	8	40	36	72	108
		70	36	71	107
		100	36	72	108
	12	40	36	72	108
		70	36	72	108
		100	36	72	108
Future PUREX Extra High Temp 2 Compositions	4	140	0	12	12
		180	0	12	12
Total			1324	2048	3372

TABLE 6. Double Shell Slurry Test Compositions

Reference No.	Molarity									
	OH ⁻	AlO ₂ ⁻	NO ₃ ⁻	NO ₂ ⁻	CO ₃ ⁻	PO ₄	SO ₄	CMPX	Cit	ρ g/ml
1	4.0	1.0	1.0	1.0	0.2	0	0.05	0.6	0	1.446
3	4.0	1.0	1.0	<u>5.97</u>	<u>0.18</u>	0.6	0.05	0	0.6	1.790
5	4.0	1.0	<u>4.8</u> +100xs*	1.0	<u>1.14</u>	0	0.05	0	0	1.479
7	4.0	1.0	5.5	4.0	<u>0.14</u>	<u>0.39</u>	<u>0.015</u>	<u>0.43</u>	0.60	1.728
8	4.0	2.0	1.0	<u>6.26</u>	<u>0.16</u>	0	<u>0.017</u>	<u>0.29</u>	<u>0.20</u>	1.678
9	4.0	2.0	2.0	<u>4.47</u> +100xs	0.11	0	0.05	0	0	1.596
12	4.0	<u>3.7</u>	1.0	1.0	0.2	0.6	0.05	0	0.6	1.678
13	4.0	<u>2.6</u>	1.0	6.0	<u>0.15</u>	0.1	<u>0.16</u>	<u>0.47</u>	0.2	1.725
15	4.0	<u>2.5</u>	<u>5.7</u>	1.0	<u>0.15</u>	<u>0.41</u>	<u>0.16</u>	0.2	0.6	1.673
16	4.0	<u>2.95</u>	<u>4.0</u> +50xs*	2.7 +50xs	<u>0.007</u>	0.1	0.05	0	0.2	1.678
20	7.0	0.5	1.0	<u>4.1</u> +100xs	0.09	0	0.05	0	0	1.550
28	7.0	<u>1.97</u>	2.0	0.6	<u>0.16</u>	0	<u>0.017</u>	<u>0.49</u>	0.20	1.592
29	7.0	<u>1.0</u>	<u>4.8</u>	<u>5.2</u>	<u>0.11</u>	<u>0.20</u>	<u>0.012</u>	0.25	0.6	1.803
30	10	0.1	1.0	0.2	<u>0.085</u>	0.2	0.05	0	0.2	1.459
33	10	0.1	1.0	<u>5.3</u>	<u>0.11</u>	0.07	<u>0.12</u>	<u>0.21</u>	0.2	1.675
36	10	0.1	<u>5.0</u>	0.2	<u>0.12</u>	<u>0.25</u>	<u>0.013</u>	<u>0.36</u>	0.2	1.608
38	10	<u>0.05</u>	<u>4.63</u> +50xs	<u>1.36</u> +50xs	0	0.07	0.05	0	0.2	1.633
44	10	<u>0.20</u>	1.0	<u>2.6</u> +100xs	0	0	0.05	0	0.2	1.548
48	0.5	0	1.5	0.2	0.2	0.2	0.05	0	0.2	1.198
49	0.5	<u>0.2</u> +10xs	1.5	0.2	0.2	0.2	0.05	0.25	0.2	1.254
50	0.5	0	3.0	3.0	0.2	<u>1.2</u>	0.05	0.75	0.2	1.725
51	0.5	0	3.0	<u>8.2</u> +50xs	0.2	0	0.05	0	0.2	1.609
52	0.5	0	<u>6.5</u> +50xs	<u>5.2</u> +50xs	<u>0.14</u>	0	0.05	0.85	0	1.72
53	0.5	0.1	3.0	3.0	0.2	0	0.05	0.98	0.6	1.625
55	0.5	0.1	<u>6.4</u> +50xs	<u>5.2</u> +50xs	<u>0.14</u>	0.2	0.05	0	0.2	1.637
57	0.5	<u>0.2</u>	3.0	<u>8.0</u> +50xs	0.2	<u>0.82</u>	0.05	0.25	0	1.772
58	0.5	<u>0.2</u>	3.0	0.2	0.2	0	0.05	0	0	1.199
59	0.5	<u>0.2</u>	3.0	0.2	0.2	0	0.05	1.0	0.2	1.416

* Excess salt, in grams
 Complexant: 50% EDTA 50% HEDTA (Na form)
 Underlined components are saturated

TABLE 6 (continued). Future PUREX Compositions (Low Temperature)

PNL Ident	RHO Ident	Composition of Liquor, M							
		OH ⁻	F ⁻	NO ₃ ⁻	NO ₂ ⁻	Aluminum ^{a)}	Citrate	Zirconium	Iron
60	A1	0.001	0.1	2.5	0.01	<u>0.5</u>	0.6	-	0
61	A3	0.001	0.1	1.5	1.0	<u>0.5</u>	0.6	0	0
62	A4	0.001	0.1	1.5	1.0	<u>1.1</u>	1.3	0	0
63	A5	0.001	0.1	3.0	2.0	<u>0.5</u>	0.6	0	0
64	A6	0.001	0.1	3.0	2.0	<u>1.1</u>	1.3	0	0
65	A7	0.001	0.2	2.5	0.01	<u>1.1</u>	1.3	0	0
66	A9	0.001	0.2	5.0	0.1	0.5	0.6	0	0
67	A11	0.01	0.1	<u>4.77</u>	0.01	1.0	1.3	0	0
68	A21	0.1	0.2	<u>4.74</u>	0.01	<u>1.1</u>	1.3	-	0.4
69	B1	0.2	0.1	0.05	0.01	0.0	0.0	0	0
70	B3	0.2	1.0	0.05	0.01	0.0	0.0	0	0
71	B5	0.2	0.1	2.0	0.01	0.0	0.0	0	0
72	B8	0.2	1.0	5.0	0.01	<u>0.05</u>	0.0	0	0
73	B9	0.2	1.0	3.0	2.0	<u>0.05</u>	0.0	0	0
74	B15	0.8	0.1	0.05	0.02	<u>0.51</u>	0.0	0	0
75	C7	1.0	<u>0.74</u>	0.2	0.01	<u>0.8</u>	0.0	0	0
76	C9	5.0	0.3	0.2	0.01	0.0	0.0	0	0
77	C10	5.0	<u>0.53</u>	0.5	0.01	0.0	0.0	0	0
78	C11	5.0	<u>0.49</u>	0.3	0.2	1.0	0.0	0	0
79	C12	9.0	0.2	0.2	0.01	0.0	0.0	0	0
80	C13	9.0	0.2	0.5	0.01	1.0	0.0	0	0
81	D6	0.2	0.2	5.0	0.1	<u>0.10</u>	0.09	0	0
82	D7	0.2	0.1	3.0	2.0	<u>0.10</u>	0.09	0	0
83	D19	0.8	0.1	5.0	0.01	<u>0.73</u>	0.27	0	0
84	D20	0.8	0.2	3.0	2.0	0.9	0.72	0	0
85	E12	1.0	<u>0.72</u>	<u>1.2</u>	<u>0.8</u>	<u>0.5</u>	<u>0.05</u>	0	0
86	1S	0.3	0.5	0.02	0.0	0.0	0.0	0	0
87	2C	0.001	0.3	<u>4.0</u>	2.0	1.0	2.0	0	0
88	2H	0.5	0.3	<u>4.0</u>	2.0	0.3	0.0	0	s
89	3C	0.1	0.15	<u>4.0</u>	2.0	1.0	2.0	0	0
90	3S	0.8	0.5	0.05	0.02	0.0	0.0	s	0
91	4C	0.1	0.3	2.0	0.2	1.0	2.0	0	0
92	4H	1.0	0.3	2.0	0.2	0.5	0.0	0	s
93	4S	0.8	<u>0.93</u>	0.02	0.0	0.0	0.0	s	0
94	5H	1.0	0.3	2.0	1.0	0.5	0.0	0	0
95	6H	1.0	0.3	<u>4.0</u>	2.0	0.5	0.0	0	0

^aAl is present as Al(citrate) or NaAlO₂

*W. M. Bowen and H. J. Eding, Statistical Design of the Corrosion Study of PUREX Related Wastes. To be published.

TABLE 7. Future PUREX Compositions (High Temperature)

PNL Ident.	RHO Ident.	OH	F	NO ₃	NO ₂	Al	Citrate	Zirconium	Iron
96	A17	.1	.1	5.0	.01	.5	.6	.05	0.2
97	A19	.1	.2	2.5	.01	.5	.6	.05	0.2
98	A23	.1	.2	3.0	2.0	1.1	1.3	.05	0.2
69	B1*	.2	.1	.05	.01	0	0	.05	0.2
70	B3*	.2	1.0	.05	.01	0	0	.05	0.2
99	B4	.2	1.0	.05	.02	.05	0	.05	0.2
72	B8*	.2	1.0	5.0	.01	.05	0	.05	0.2
73	B9*	.2	1.0	3.0	2.0	.05	0	.05	0.2
100	B20	.8	.62	3.0	2.0	0	0	.05	0.2
101	C3	1.0	.4	.12	.08	.80	0	.05	0.2
76	C9*	5.0	.3	.2	.01	0	0	.05	0.2
77	C10*	5.0	.53	.5	.01	0	0	.05	0.2
78	C11*	5.0	.49	.3	.2	1.0	0	.05	0.2
80	C13*	9.0	.2	.5	.01	1.0	0	.05	0.2
82	D7*	.2	.1	3.0	2.0	.1	.09	.05	0.2
102	D10	.5	.01	3.0	.01	.027	.09	.05	0.2
89	3C*	.1	.15	4.0	2.0	1.0	2.0	.05	0.2
91	4C*	.1	.3	2.0	.2	1.0	2.0	.05	0.2
202	New	1.2	.1	4.0	2.0	1.1	.6	.05	0.2
203	Points	1.2	.1	5.0	.01	1.1	.6	.05	0.2
220	"	1.2	1.0	3.0	.01	1.1	.6	.05	0.2
221	"	1.2	1.0	3.0	2.0	1.1	.6	.05	0.2
223	"	1.2	1.0	5.0	.01	1.1	.6	.05	0.2

* From Low Temperature Design.
The presence of zirconium and iron are not expected to have an effect.

TABLE 8. Hanford Facilities Waste Compositions

25°C Molar Concentrations					50°C Molar Concentrations				
Soln#	OH ⁻	PO ₄ ⁼	SO ₄ ⁼	NO ₂ ⁻	Soln#	OH ⁻	PO ₄ ⁼	SO ₄ ⁼	NO ₂ ⁻
1	0.0001	0.0	0.0	0.01	1	0.0001	0.0	0.0	0.01
2	"	"	0.8	"	2	"	"	0.6	"
4	"	"	<u>1.7</u>	0.002	3	"	"	<u>2.9</u>	0.0
5	"	"	"	0.01	4	"	"	"	0.002
6	"	0.4	0.0	0.0	5	"	"	"	0.01
7	"	"	"	0.01	6	"	2.0	0.0	0.0
12	"	<u>0.8</u>	"	0.002	12	"	<u>4.6</u>	"	0.002
13	"	"	"	0.01	13	"	"	"	0.01
15	"	<u>0.5</u>	<u>1.6</u>	0.0	15	"	<u>3.9</u>	<u>1.3</u>	0.0
16	"	"	"	0.01	16	"	"	"	0.01
20	0.6	0.0	<u>1.7</u>	0.0	20	0.6	0.0	<u>2.9</u>	0.0
23	0.5	0.4	0.8	.002	23	0.5	2.0	0.6	0.002
25	0.9	<u>0.8</u>	0.0	0.0	25	0.9	<u>4.6</u>	0.0	0.0
29	"	<u>0.5</u>	<u>1.6</u>	0.01	29	"	<u>3.9</u>	<u>0.8</u>	0.01
30	0.01	0.0	0.0	0.002	30	0.01	0.0	0.0	0.002
31	"	"	"	0.01	31	"	"	"	0.01
32	0.2	"	0.8	0.0	32	0.2	"	0.6	0.0
35	1.2	"	<u>1.7</u>	0.01	35	1.2	"	<u>2.9</u>	0.01
36	0.8	0.4	0.0	0.0	36	0.8	2.0	0.0	0.0
37	"	"	"	0.01	37	"	"	"	0.01
41	2.0	<u>0.8</u>	"	"	41	2.0	<u>5.0</u>	"	"
45	"	<u>0.5</u>	<u>1.6</u>	0.002	45	"	<u>4.2</u>	<u>0.8</u>	0.002

Underlined concentration denotes saturated mixture.
Mixture 3 occurs only at 50°C and Mixture 7 only at 25°C.

Several other small tests were run in addition to this basic set. These were labeled Double Shell Slurry and Future PUREX Oven Tests (DSS OT and FP OT), the Double Shell Slurry Remake Test (DSS RT), and some extra tests for Future PUREX at the higher temperatures (FP XHT).

CORROSION SPECIMENS

Both weight-loss coupons and U-bend specimens were used in the tests, Figure 1. U-bends were used despite being a severe and uncalibrated test method. They are particularly useful because they can provide a rapid and inexpensive comparison of the behavior of stressed material in different waste compositions.

No specific tests were established to examine crevice corrosion. However, the coupons were supported in the solution with Teflon[®] tape which formed crevices.

After cutting the specimens to size, each was numbered and cleaned for 10-15 min in a solution of 200 ml concentrated HCl + 200 ml H₂O + 5 ml formaldehyde and rinsed in deionized water. The specimens were then scrubbed using a nonmetallic brush, rinsed in deionized water and then rinsed with acetone.

Following cleaning, each specimen was weighed on a balance and the data transmitted electronically to a computer. This computerized technique reduced weighing and weight transcription errors, put the data in an accessible file and provided a means for extracting the data for later calculations and incorporation into the corrosion data base.

After weighing, each specimen was heat treated at 1100°F (593°C) for one hour to simulate the stress relief treatment received by the tanks. The nuts and bolts used in the U-bend specimens were only heated ten minutes to oxidize the surface. Each specimen was reweighed after heat treatment; the U-bends were weighed again after the first bending step but before final assembly.

Because no cleaning was performed after the annealing in order to better simulate the surface condition of the actual tanks (in actual practice the tanks are hydrotested after annealing and are covered with

Teflon[®] - Registered Trademark of E.I. du Pont de Nemours.

FIGURE 1. Weight Loss and U-Bend Specimens

a loose hematite layer), the actual starting weight of the base metal in the coupons is unknown. To estimate the starting weight, a number of representative specimens were selected and cleaned after the annealing process. These are used to calculate an average weight loss due to annealing. As a consequence, however, any specimen with a low corrosion rate and a below average degree of oxidation may have a nonrepresentatively low (negative) corrosion rate calculated.

TEST PROCEDURES

Bottles of TFE Teflon[®] were used as the primary containment for all tests. In the low temperature tests, 40 to 100°C, glycerin baths were used to maintain the temperature; the 25°C tests were simply allowed to follow ambient laboratory conditions.

For the high temperature, 140 and 180°C, tests, two procedures were used. The first, and unsuccessful, method was to place the test bottles in ovens at the appropriate temperature. However, because the seals were not sufficiently tight, some bottles lost moisture. In addition, those compositions containing complexant (a mixture of HEDTA and EDTA) foamed severely, ruining the bottles and, in some cases, the ovens.

As a result, two large vessels were constructed to contain the test bottles. They were operated at the vapor pressure of water at the appropriate temperature. This minimized the water loss and reduced the effect of foaming.

EXPERIMENTAL DESIGN

The computer-aided design of regression experiments is a significant advance over the more restrictive classical experimental designs (for example, factorial designs and special purpose response surface designs). The ADDEXC computer program at PNL incorporates the methods described by Wynn⁽²⁾ and St. John and Draper⁽³⁾ for constructing optimal regression experimental designs. This is especially useful in designing corrosion experiments, where solubilities or other factors require the use of variable composition levels. This process was used to design the weight loss tests only.

Hypothesized Model Form

A hypothesized prediction model with unknown parameters is required for the construction of a computer-aided design. This study includes "major" and "minor" chemicals. The major chemicals are those which are expected to influence the corrosion rate. The effective concentration levels of the major chemicals appear in the hypothesized model as linear (or first order) terms, quadratic (or second order) terms, and mixed quadratic terms (products of all possible pairs). The minor chemicals are expected to have little or no effect on the corrosion rate. The effective concentration levels of the minor chemicals appear in the hypothesized model only as linear terms.

The FP system is used as an example of the design process. For the FP waste, there are five major chemicals and three minor chemicals. Thus, the hypothesized polynomial model for a specified composition is given by

$$y = a + \sum_{i=1}^8 b_i X_i + \sum_{i=1}^5 c_i X_i^2 + \sum_{i=1}^5 \sum_{j < i} d_{ij} X_i X_j + e, \quad (1)$$

where:

- y is the measured value of the corrosion indicator (e.g., corrosion rate in mils/year) for a specified composition,
- $X_1, X_2, \dots, X_5$ are the effective concentration levels of the major chemicals,
- X_6, X_7, X_8 are the effective concentration levels of the minor chemicals,
- $a, b_i, c_i,$ and d_{ij} are 24 unknown constants (parameters) that will be estimated from the experimental data,
- e is a random component due to the nonreproducibility of y when replicate experiments are performed using a specified composition.

Justification of Computer-Aided Designs

The objective of the experimental design process is to determine which compositions should be included in the study. The chosen set of compositions must provide sufficient information to accommodate estimation of the 24 unknown constants in Equation 1, thus yielding a "prediction equation" that predicts corrosion for compositions in the study region defined in Table 2. Moreover, the compositions should be selected in such a way that corrosion predictions (made from the prediction equation) over the study region will have the smallest possible prediction variances. The experiment should also include more compositions than the number of unknown constants in Equation 1 (i.e., more than 24 compositions), so that the validity of the hypothesized model can be tested. With these considerations in mind, a list of possible compositions must be developed that adequately covers the pertinent concentration levels of the eight chemicals in combination with each other.

Because the range of concentrations of each chemical is continuous, the number of possible compositions is infinite. The classical factorial design approach would be to define three concentration levels (low, medium, and high concentrations) for each major chemical and two concentration levels (low and high concentrations) for each minor chemical. With five chemicals at three levels and three chemicals at two levels, the number of possible compositions is $3^5 \cdot 2^3 = 1944$. However, since there are only 24 unknown constants in Equation 1, it is not necessary to include 1944 compositions in the study. Also, practical considerations such as available facilities, manpower, time and budget dictate an upper limit of 40 compositions. This is the motivation for considering fractional factorial designs and special purpose response surface designs such as discussed by Mitchell and Bayne⁽⁴⁾ or Webb.⁽⁵⁾

A fractional factorial design would include a subset (fraction) of the 1944 compositions, selected under quite restrictive assumptions about possible higher order terms and cross product terms that do not appear in Equation 1. A special purpose response surface design would include a set of compositions chosen specifically to provide adequate information for fitting Equation 1. A response surface design would not

be a subset of the 1944 compositions but would be more efficient for fitting Equation 1 than a fractional factorial design and would possess desirable statistical properties. Unfortunately, certain properties of the chemicals preclude the use of factorial and response surface designs. For example, some of the chemicals interact in such a way that a high (or low) concentration of one lowers the saturation levels of others. Thus, many of the compositions required for a factorial or response surface design are not possible. That is, the "effective concentration levels" of the chemicals for such compositions are different than the levels specified by the design. If included in the study, these "impossible" compositions would tend to be quite similar to other compositions already in the design. Such redundancies are not only inefficient from the experimenter's point of view, but they would cause serious problems with attempting to fit Equation 1 to the experimental data. This situation can be satisfactorily dealt with through computer-aided experimental design.

The computer-aided design of the corrosion study was an iterative procedure. First, a list of "candidate compositions" was prepared that thoroughly covered the ranges of all chemicals of interest. These compositions were then evaluated using the Rockwell CORR-3 computer code which calculates the solubilities of the salts to give the effective levels of the chemicals. The list of compositions was then reviewed to determine whether additional compositions were needed. Before a composition could be added to the list, it was first evaluated by CORR-3 to determine whether it would actually fill a void.

When the list of candidate compositions was complete, it was processed by the ADDEXC program which chose the best subset (of size less than 40) of the candidate compositions for fitting Equation 1. This subset of compositions was then reviewed, and any noticeable voids gave guidance in determining which compositions should be added to the list of candidates. After adding such compositions, the list of candidates was again submitted to the ADDEXC program to select a subset. This procedure was repeated until a set of fewer than 40 compositions was defined which adequately covered the ranges of the eight chemicals and would be as good as or better than any other subset of equal size

selected from the candidates for fitting Equation 1 and making predictions of corrosion over the study region.

Chemical Designations

Equation 1 expresses corrosion as a function of the effective levels of five major chemicals and three minor chemicals. The major chemicals are expected to have noticeable effects on the corrosion, while the minor chemicals are expected to have small or negligible effects. Thus, Equation 1 includes linear, quadratic, and cross product terms for the effective levels of the major chemicals and only linear terms for the effective levels of the minor chemicals.

The selection of the major and minor chemicals was based on background information on which chemicals were most likely to influence the corrosion rate. The literature and background did not allow any chemicals to be completely eliminated from consideration as having a possible effect on corrosion. However, OH^- , F^- , NO_3^- , NO_2^- , and AlO_2^- were considered to be the most important chemicals and are the major chemicals for this study. Citrate is believed to have only a minor (linear) effect on corrosion and not to interact with the other chemicals. Insoluble solids such as $\text{Zr}(\text{OH})_4$ and $\text{Fe}(\text{OH})_3$, carbonate and sulfate, have low concentrations in solution with little effect expected on corrosion in these wastes. Of these, $\text{Zr}(\text{OH})_4$ and $\text{Fe}(\text{OH})_3$ were used as minor chemicals and the others dropped from consideration.

In summary, the five major chemicals for the design of this study are OH^- , F^- , NO_3^- , NO_2^- , and AlO_2^- , and the three minor chemicals are citrate, zirconium and iron.

Composition Selection

As noted, there are conditions that preclude the application of classical experimental designs to corrosion studies. Computer-aided experimental design is introduced above as a workable alternative, and the development of a list of "candidate compositions" is briefly described. Ideally, the set of candidate compositions should include all compositions that would be included in the study if there were no restrictions on time, cost, available facilities, etc.. These compositions should thoroughly cover the five types of PUREX related wastes.

Although factorial designs cannot be used explicitly for this purpose, some factorial design concepts give guidance in developing a set of candidate compositions from which the ADDEXC program selected the final design.

In a factorial design, the chemical levels would be fixed throughout the design. The low, medium and high levels of the major chemicals would be coded -1, 0 and 1, and the low and high levels of the minor chemicals would be coded -1 and 1. In this manner, each of the 1944 resulting compositions would have a distinct code. For example, the composition given by (0, 0, 0, 0, 0, 1, 1, 1) includes the medium level of each major chemical and the high level of each minor chemical. It was pointed out above that many of these compositions are not possible due to varying saturation levels. However, by letting s denote the saturated concentration level of any chemical in a composition, the three levels of each major chemical can be coded $.33s$, $.67s$, and s , and the two levels of each minor chemical can be coded 0 and s . Thus, the composition (0, 0, 0, 0, 0, 1, 1, 1) would be expressed as $(.67s, .67s, .67s, .67s, .67s, s, s, s)$. It is then possible, using CORR-3, to transform the 1944 factorial design points into a list of compositions that more thoroughly covers the effective levels of the chemicals in combination with each other. However, the transformation of all 1944 factorial points, via the CORR-3 code, would be quite tedious and time-consuming. Thus, an alternative approach was taken.

Mitchell and Bayne⁽⁴⁾ and Webb⁽⁵⁾ give two and three level fractional factorial designs for fitting specific response surface models. Several of their designs were combined, and redundant points were omitted, resulting in 210 candidate compositions. After transforming to fractions of the actual saturation levels via CORR-3, the 210 compositions were reviewed. Nearly half of the points were discarded, and some were replaced by points which filled identifiable voids in the range of compositions. Selection of the "replacement points" was also based on the Mitchell and Bayne⁽⁴⁾ and Webb⁽⁵⁾ designs.

A final set of 120 candidate compositions was produced. This set of compositions was judged to provide good coverage of the five types of PUREX-related waste and mixtures of them. The ADDEXC program selected

the best subset of these 120 candidate compositions for fitting Equation 1. The 36 selected compositions are displayed in Table 6.

This process was repeated to select compositions for each waste type. It was also used together with results from the 100°C tests to select compositions for the high temperature tests.

CORROSION PROCESSES

An attempt was made in the laboratory to define the corrosion mechanism. By having this information, the number of "bottle" tests conducted could be reduced. The laboratory work was unsuccessful in establishing the mechanism although some groundwork was laid. The following discussion describes some of the reactions believed to be involved in the corrosion process.

In the OSS and FP wastes, the first formed product is most likely some form of Fe^{2+} , for example, $\text{Fe}(\text{OH})_2(\text{s})$ or FeY^{n-} (where Y = complexant, moiety, citrate, EDTA, or HEDTA). Mixed complexes such as $\text{FeY}(\text{NO}_3)^{n-}$ are also possible but not likely to be very important. The HF type wastes are a special case and are not specifically discussed.

The Fe(II) product(s) all tend to be oxidized to the corresponding Fe(III) product(s) at normal room temperature and slightly above (up to ~ 80-90°C). At higher temperatures, species like $\text{Fe}^{3+}\text{EDTA}$ begin to decompose in such a way as to destroy the EDTA.

At very high OH^- concentrations (4-10 M), the most likely forms of Fe(III) are $\text{Fe}(\text{OH})_3$ [or Fe_3O_4] and $\text{Fe}(\text{OH})_4^-$ [or FeO_2^-]. This will be true independent of the presence or absence of chelants.

As OH^- concentration goes down, the oxidizing effectiveness (kinetically speaking) of NO_3^- and NO_2^- increases. Thus, oxidizing agents other than O_2 (including but less importantly H^+ or H_2O) must be considered.

At the concentration levels of NO_3^- and NO_2^- found in the compositions studied, these oxidants are corrosion inhibitors due to the reaction:*

This reaction becomes more and more facile as OH^- concentration decreases. The importance of FeY^- (solubilized iron) increases as pH is lowered in the range of DSS compositions. However, the formation of solubilized iron is still relatively unimportant compared to the formation of Fe(OH)_3 or Fe_3O_4 as seen by the magnitude of the equilibrium constant for the solubilization reaction:

As a representative calculation based on this reaction and its equilibrium constant, consider a worst case (lowest expected OH^- concentration) example:

$$\begin{aligned} [\text{Y}^{4-}] &= 0.5, \quad (\text{OH}^-) = 10^{-3} \\ \frac{[\text{FeY}^-] [\text{OH}^-]^3}{[\text{Y}^{4-}]} &= 10^{-12.3} \\ \text{max } [\text{FeY}^-] &= \frac{(10^{-12.3})(0.5)}{(10^{-3})^3} \approx 2.5 \times 10^{-4} \text{ M} \end{aligned}$$

Because the K_{sp} of Fe(OH)_3 is 4×10^{-38} (Ref. 6), the activity of Fe^{3+} in this situation is $\sim 4 \times 10^{-29}$ M. Consequently, the solubilized iron

* NO_3^- and NO_2^- can, at low OH^- concentrations, promote the oxidation (corrosion) of iron. If present in sufficient concentration they cause the iron to passivate, i.e., cease to corrode at a high rate. Thus, though they tend to drive the corrosion reaction, they can act as "inhibitors" for metals such as iron.

from all forms may reach a value of $\approx 10^{-3}$ M for this case of DSS compositions; note that at higher pH, the potential for solubilized iron decreases, hence, corrosion rates would be expected to decrease as pH increases as long as the insoluble iron forms protective films. At very high pH (e.g., 10 M OH^-), solubilization again becomes important due to $\text{Fe}(\text{OH})_4^-$ (FeO_4^{2-}) formation and leads to increased corrosion. The formation of Na_2FeO_2 , identified by its X-ray diffraction pattern and examined by SEM, was reported in an earlier report.⁽⁷⁾

The primary anodic (corrosion) reaction is believed to be

In oxygenated wastes, the primary cathodic reaction is the reduction of O_2 followed by conversion of $\text{Fe}(\text{OH})_2$ to either Fe_3O_4 or $\text{Fe}(\text{OH})_3$. In anoxic wastes, the primary cathodic reaction is

The reduction of NO_3^- and/or NO_2^- is of only minor importance in contributing to the direct cathodic corrosion reaction. NO_2^- and, to a lesser extent NO_3^- , exerts its major role in the noncorrosion redox reaction

or

The rates of redox reactions involving NO_2^- and NO_3^- are pH dependent, becoming extremely slow in solutions whose pH corresponds to high caustic waste.

In complexant (HEDTA/EDTA) free compositions, one decomposition product of $\text{NO}_3^-/\text{NO}_2^-$, detected by odor, is ammonia or related volatile compounds. In compositions with HEDTA/EDTA, other reactions, discussed

further below, occur, including the production of oxalate, without the apparent generation of ammonia or its analogs.

The protection of Fe from corrosion in these wastes is due to the production of a protective Fe_3O_4 film. However, the exact mechanisms of formation and the critical agents have not been worked out. Recent, unpublished work by Smialowska and her students at Ohio State University appears to be on the way to this goal, albeit only at dilute (~ 0.05 M) hydroxide levels. Because satisfactory information is being obtained from the empirical studies there are no plans to delve further into the mechanism.

ORGANIC DECOMPOSITION

DSS waste containing HEDTA/EDTA has a tendency to foam, with the degree and rate of foaming increasing with temperature and caustic content. The compositions with greatest action were Nos. 33 and 36 with 10 M OH^- and .21 M and .36 M total complexant, respectively. In one set of samples, Composition 33 was found to contain 211 ppm oxalate after 8 months at 100°C and 3.7 wt.% after less than two days at 180°C. In this latter test, the volume increased an estimated 30-50%. Similarly, No. 36 was found to contain 166 ppm after 8 months at 100°C and 5.3 wt.% after less than two days at 140°C.

Other analyses on selected compositions are given in Table 9. Being at lower caustic levels, little foaming occurred in them; less oxalate was formed. Beyond consuming various chemicals, the action of the decomposition products on corrosion is not expected to be greater than the original complexants which themselves have not proven strongly influential.

STRESS CORROSION CRACKING

Due to the limited amount of stress corrosion cracking (SCC) that occurred in these tests, no statistical analysis was made as was done by Ondrejcin⁽⁸⁾ in his studies. Indeed, though U-bend tests are considered to be a severe test,⁽⁹⁾ less SCC was observed than expected from previous work.^(8,10)

In the FP compositions, only Numbers 71, 72, and 81 produced cracking at temperatures at or below 100°C and Numbers 78, 79, and 102 at 140° temperatures. HF compositions, tested at 25°C and 50°C, produced no cracking. The DSS compositions produced no cracking except at 180°C in Compositions 30 and 36. Thus, two regimes are found which cause cracking--low temperature, low concentration (LTLC) and high temperature, high concentration (HTHC).

Based on the corrosion behavior at various $\text{NO}_2^-/\text{OH}^-$ levels, the SCC in the LTLC is believed to be occurring in the active/passive transition region. For example, in FP Compositions 61 and 65 and HF Compositions 2 and 7, corrosion rates were nonuniform and high, over 2 mpy, and the pH relatively low (≤ 11) with low oxidant concentrations (0.01 NO_2^-). In these active conditions no SCC occurred. With slightly higher (intermediate) $\text{OH}^-/\text{NO}_2^-$ levels, cracking occurred. At still higher caustic levels, no further cracking or significant corrosion occurred--until the HTHC conditions were reached. In the low concentrations the behavior resembles the corrosion in the active region, whereas at the higher concentrations the steel was passive.

CREVICE CORROSION

Although no specific crevice corrosion tests were established, crevices were present between the coupons and the Teflon[®] tape which was used to suspend them in the test bottles. No crevice attack was observed.

NONUNIFORM CORROSION

In general the corrosion was uniform. The only observed instances of nonuniform attack were, as noted above, in the dilute compositions FP 61 and 65 and HF 2 and 7. In these cases the major attack was along corners or at the identification numbers. The stressed oxide at these regions allowed corrosion to initiate and proceed rapidly.

TABLE 9. Analysis^(a) of Organic Chelating Agents^(b) in Double Shell Slurry Samples^(c)

	Concentration ^(d) , ppm (mM) ^(e)						Number Of Unknowns ^(g)
	Oxalic Acid	Citric Acid	NTA ^(f)	ED3A ^(f)	EDTA	HEDTA	
Solution 1*, 140°C			10,439 (44)	5,365 (22)	38,881 (111.7)		
Solution 1, 180°C	Trace			25.16			
Solution 3*, 140°C							> 30
Solution 3*, 180°C							> 10
Solution 5*, 140°C							> 17
Solution 5*, 180°C							> 17
Solution 15, 140°C	Trace		720	63	2,440		5
Solution 15, 180°C	Trace			85			> 20
Solution 15*, 100°C, 4 months	Trace	1,396 (6.0)		111 (0.5)	226 (0.8)	618 (2.15)	~ 3
Solution 15, 100°C, 8 months	Trace		1,890	3,180		42,200	~ 4

a) Qualitative and quantitative identifications based solely on gas chromatographic (GC) analysis.

b) Identified as methyl esters (BF₃/Methanol).

c) Samples consist of liquids(*) and solids.

d) No entry indicates compound is below detection level.

e) Numbers in () are mM concentration of species in liquid samples.

f) These species are products of chelator degradation.

g) Some samples contained numerous unknowns, many of which are degradation products of chelating agents.

PART 2 - STATISTICAL ANALYSIS OF THE TANK CORROSION DATA

Statistical comparisons were made between results of all tests, where possible, to see if they differed greatly. The conclusion of this analysis was that the two sets of Oven Test data gave very different corrosion rates values in comparison to the remaining data base. This could be explained by the fact that the Oven Tests had experienced a significant loss of water during the test; the results are similar to data generated by Maness⁽¹¹⁾ in moistened waste. Consequently, the DSS OT and FP OT data were not used in the prediction modeling.

A summary of the data that were actually used for model development is given in Table 5 of Part 1.

DEVELOPMENT OF UNIFIED STATISTICAL PREDICTION MODELS

The objective of the initial modeling effort was to develop a unified model for predicting corrosion rates that makes use of all available Tank Corrosion data. This unified model can be expected to give estimates that are not as precise for regions of chemical compositions where a specific subset of the data had been used to develop a model previously, but does give a prediction procedure that has much broader applicability. The unified models are based on a wider range of compositions than the models developed previously for the individual waste types. Thus, they can be used to predict corrosion for compositions that fall outside the individual waste ranges but are inside the overall combined composition range.

The chemical compositions used in the Tank Corrosion tests were selected to accommodate fitting a polynomial model that expresses corrosion rate data as a function of powers and cross products of exposure time, temperature, and the different levels of chemicals in the compositions for each waste type. The same strategy was used for the unified models.

Prior to actual model development, the data were transformed and coded in a manner similar to previous models developed for subsets of this data. Specifically, a transformation was made on observed corrosion rates (expressed as mils/year [mpy]) of the form

$$\ln mpy = \ln (mpy + 1.25)$$

The natural log transformation is a variance stabilizing procedure that was deemed necessary for this data due to an observed increase in corrosion variability with increased corrosion rates. The modeling procedure assumes that the variability in corrosion rates is approximately constant over all test conditions. The addition of 1.25 made all transformed values greater than zero.

The chemical compositions were coded by subtracting the approximate mid-range of the levels that occurred in the tests from each value and dividing by approximately one-half of the range. This helps protect against computer round-off error which can be a significant problem, especially with a data base of this size, the range of compositions that were used, and the use of polynomial models that require many multiplications. The resulting coded values are basically in the interval of -1 to 1. For the unified model, the actual codings for each composition are:

$$c0 = (\log_{10}(\text{OH}) + 1) / 3$$

$$c1 = (A1 - 1.8) / 1.8$$

$$c2 = (\text{NO}_3 - 3.75) / 3.75$$

$$c3 = (\text{NO}_2 - 4) / 4$$

$$c4 = (\text{Complex} - .5) / .5$$

$$c5 = (F - .5) / .5$$

$$c6 = (\text{CO}_3 - .1) / .1$$

$$c7 = (\text{PO}_4 - 2.5) / 2.5$$

$$c8 = (\text{SO}_4 - 1.5) / 1.5$$

$$c9 = (\text{Citrate} - 1)$$

Exposure temperature and hours of exposure were also coded so that the values have minimum and maximum of approximately -1 and 1, respectively. The codings for the unified models are:

$$t1 = (\text{Temperature} - 100) / 75$$

$$h1 = (\text{Hours} - 5800) / 3000.$$

Due to the fact that some chemical constituents did not appear in each waste type it was not possible to use every cross product term in the model. A synopsis of which waste type had which chemical constituent is given below.

Waste type	Chemical Constituent									
	OH	Al	NO ₃	NO ₂	Complex	F	CO ₃	PO ₄	SO ₄	Citrate
	c0	c1	c2	c3	c4	c5	c6	c7	c8	c9
HF	x			x				x	x	
DSS	x	x	x	x	x		x	x	x	x
FP	x	x	x	x		x				x

If a particular chemical constituent did not appear in the compositions for a particular waste type it was given a value of zero prior to coding for all data of that waste type.

The initial polynomial model consisted of the following 372 terms:

- Intercept - 1 term
- Chemical Composition Terms - 3D terms:
 - Simple Linear Terms - c0 c1 c2 c3 c4 c5 c6 c7 c8 c9 - 10 terms
 - Pure Quadratic Terms - c0c0 c1c1 c2c2 c3c3 c4c4 c5c5 - 6 terms
(considered to be major constituents)
 - Cross Product Terms - c0c1 c0c2 c0c3 c0c4 c0c5 - 14 terms
c1c2 c1c3 c1c4 c1c5
c2c3 c2c4 c2c5
c3c4 c3c5
[Complex(c4) and F(c5) never occurred together]

- Temperature and Time Terms - 11 terms:
 - Simple Linear Terms - $t_1 h_1$ - 2 terms
 - Pure Quadratic Terms - $h_2 t_2 t_3$ - 3 terms
 - Cross Product Terms - $t_1 h_1 t_2 h_1 t_3 h_1 t_1 h_2 t_2 h_2 t_2 h_3$ - 6 terms
- Cross Products of Composition With Temperature and Time - 330 terms:
 - Each of the 30 chemical composition terms multiplied by each of the 11 temperature and time terms.

A model with so many terms in it can be expected to include some that do not add significant information and could be discarded without degrading the predictive capability of the equation; that is, a smaller subset of terms is probably adequate. To select an adequate subset of the terms, a stepwise regression procedure was used. The BMDP Statistical Software Program P2R - Stepwise Regression with the FSWAP option was used for this purpose. In this procedure, two options are set which control the program's progress. One is F-to-enter which defines the minimum F-statistic a term must have to enter into the model, while F-to-remove defines the minimum F-statistic a term must maintain to remain in the model. For these models, F-to-enter and F-to-remove, both were set at 1, which is approximately equal to a significance level of .25. The stepwise program then proceeds by selecting the term, not already in the model, that has the highest F-statistic and meets the F-to-enter criteria. Since many of the terms are correlated, adding a term to the model changes the F-statistic for each of the terms already in the model. Because of this, the program then removes the term in the model with the lowest F-statistic if it does not meet the F-to-remove criteria; this is repeated until all "nonsignificant" terms are removed from the model. The program then repeats all of these steps until none of the terms not in the model meet the F-to-enter criteria needed to enter the model.

This procedure was applied separately to each of the alloys. For the A-516 data, a model with 90 terms was chosen with an R^2 value of .49 (R^2 is the percent of the variability in the observed corrosion rates accounted for by the selected model). For the A-537 data, a model with

75 terms and an R^2 of .56 was obtained. The actual prediction models for these two alloys are given in Tables 10 and 11.

TABLE 10. Prediction Equation for the Unified Model for A-516

$$\ln (\text{mpy}+1.25) =$$

0.44689	- 0.33268 * c0
+ 0.51193 * c1	- 0.25037 * c3
+ 0.11644 * c4	+ 0.07253 * c5
+ 0.05771 * c8	- 0.15436 * c9
- 0.04278 * h1	+ 0.25251 * c0c0
- 0.93999 * c0c1	+ 0.16392 * c0c2
+ 0.98505 * c0c3	- 0.35255 * c0c5
+ 0.07049 * c1c1	- 0.13554 * c1c3
- 0.28557 * c2c2	+ 0.23369 * c2c3
+ 0.18810 * c3c3	+ 0.12739 * c3c4
+ 0.11878 * c4c4	- 0.11630 * c5c5
+ 0.29212 * t3h2	+ 0.34029 * t1c0c0
+ 0.09469 * t1c0c1	+ 0.28007 * t1c0c4
- 0.09158 * t1c1c1	+ 0.05127 * t1c1c2
- 0.09365 * t1c2c4	+ 0.17116 * t1c4c4
- 0.47770 * t2c0	- 0.26765 * t2c1
+ 0.27385 * t2c2	+ 0.33650 * t2c4
+ 0.22040 * t2c9	+ 1.52390 * t2c0c0
+ 1.11735 * t2c0c1	- 0.49871 * t2c0c2
- 0.90439 * t2c0c3	+ 0.13918 * t2c1c3
+ 0.46569 * t2c1c4	+ 0.16832 * t2c2c4
+ 0.05944 * t2c2c5	- 0.39237 * t2c3c4
+ 0.23374 * t2c5c5	- 0.96299 * t3c0
+ 0.27445 * t3c3	+ 0.12148 * t3c7
+ 1.46157 * t3c0c0	- 0.89585 * t3c0c3
- 0.19815 * t3c0c5	+ 0.13113 * t3c1c3
+ 0.14051 * t3c2c4	+ 0.19550 * t3c5c5
+ 0.02724 * h1c6	- 0.02390 * h1c2c5
- 0.04625 * h2c2	- 0.29232 * h2c0c3
+ 0.20210 * h2c0c4	+ 0.09265 * h2c1c4
+ 0.16275 * h2c2c2	- 0.15945 * h2c2c3
- 0.05443 * h2c3c3	- 0.06936 * h2c3c4
+ 0.04473 * t1h1c5	+ 0.04759 * t2h1c9
+ 0.06355 * t2h1c1c4	+ 0.05665 * t2h1c2c5
+ 0.03705 * t2h1c3c4	+ 0.05342 * t3h1c1c5
+ 0.03814 * t3h1c2c5	+ 0.30509 * t1h2c0c0
+ 0.51128 * t2h2c3	- 0.83544 * t2h2c4
+ 0.32411 * t2h2c5	- 0.12741 * t2h2c9
+ 0.27206 * t2h2c0c2	+ 0.23257 * t2h2c0c4
+ 0.36282 * t2h2c1c3	- 0.84023 * t2h2c1c4
+ 0.38234 * t2h2c1c5	- 0.08692 * t2h2c2c5
+ 0.26535 * t2h2c3c4	- 0.14146 * t2h2c4c4
- 0.08363 * t2h2c5c5	+ 0.16914 * t3h2c7
- 0.34490 * t3h2c0c0	+ 0.40998 * t3h2c0c3
+ 0.15827 * t3h2c1c1	- 0.09016 * t3h2c2c3
- 0.26224 * t3h2c5c5	

TABLE 11. Prediction Equation for the Unified Model for A-537

$$\ln(\text{mpy}+1.25) =$$

0.43205	+ 0.55970 * c1
- 0.34198 * c3	+ 0.18315 * c4
+ 0.24974 * c5	+ 0.11096 * c7
- 0.12772 * c9	+ 0.30340 * t1
+ 0.63840 * c0c0	- 0.67956 * c0c1
+ 0.20703 * c0c2	+ 1.31491 * c0c3
- 0.34562 * c0c5	+ 0.12923 * c1c1
- 0.03165 * c1c2	- 0.16298 * c1c3
+ 0.08573 * c1c4	+ 0.13896 * c1c5
- 0.33832 * c2c2	+ 0.31082 * c2c3
+ 0.28202 * c3c3	+ 0.07159 * c3c4
+ 0.05832 * c3c5	+ 0.11298 * c4c4
- 0.08185 * c5c5	- 0.43004 * t1c0
+ 0.11316 * t1c8	+ 0.69320 * t1c0c0
- 0.10608 * t1c0c3	- 0.06173 * t1c1c4
- 0.07806 * t1c2c3	- 0.13423 * t1c3c3
- 0.69605 * t2c0	+ 0.42856 * t2c3
- 0.42572 * t2c7	+ 0.22014 * t2c8
+ 0.07859 * t2c9	+ 0.16308 * t2c0c0
- 0.29343 * t2c0c2	- 1.00524 * t2c0c3
- 0.11869 * t2c0c5	+ 0.25478 * t2c1c3
- 0.10346 * t2c2c3	- 0.13387 * t2c3c3
+ 0.19910 * t2c5c5	- 0.08787 * t3c1
+ 0.17871 * t3c7	+ 0.09917 * t3c9
- 0.26151 * t3c0c3	- 0.13895 * t3c0c4
- 0.22433 * t3c0c5	+ 0.21617 * t3c2c2
+ 0.14096 * t3c3c3	- 0.12292 * t3c3c4
+ 0.01768 * h1c6	- 0.11417 * h1c0c0
+ 0.04434 * h1c2c2	- 0.03892 * h1c3c3
- 0.05244 * h2c6	- 0.08303 * h2c0c0
- 0.09040 * h2c0c4	+ 0.04020 * h2c1c5
- 0.08524 * h2c4c4	+ 0.03395 * h2c5c5
- 0.25629 * t1h1c0c0	+ 0.03848 * t1h1c1c4
- 0.01958 * t1h1c1c5	+ 0.04262 * t1h1c3c4
- 0.11474 * t2h1c0c0	- 0.02753 * t3h1c1c1
- 0.08666 * t1h2c5	- 0.05076 * t2h2c1c2
+ 0.10346 * t3h2c5	- 0.15302 * t3h2c0c0
+ 0.19643 * t3h2c0c3	- 0.26171 * t3h2c0c4

DEVELOPMENT OF SPECIFIC WASTE TYPE MODELS

The R^2 values for the two unified prediction equations indicate that they could be useful for estimating corrosion rates under a variety of conditions. However, the R^2 values are low enough that further modeling is desirable within each waste type with the objective of developing models with better prediction capabilities for each waste type. The strategy used to develop these models is identical to that

used for the unified models presented in the previous section. The details for each of the specific waste type models are presented below.

The initial variable coding used for the Hanford Facilities Waste model is as follows:

$$\begin{aligned} \ln mpy &= \ln(mpy + 1.25) \\ c0 &= (\log_{10}(OH) + 2) / 2 \\ c3 &= (NO_2 - .005) / .005 \\ c7 &= (PO_4 - 2) / 2 \\ c8 &= (SO_4 - 1.3) / 1.5 \\ t1 &= (Temperature - 37.5) / 12.5 \\ h1 &= (Hours - 5800) / 3000. \end{aligned}$$

The initial polynomial model consisted of the following 90 terms:

- Intercept - 1 term
- Chemical Composition Terms - 14 terms
 - Simple Linear Terms - $c0\ c3\ c7\ c8$ - 4 terms
 - Pure Quadratic Terms - $c0c0\ c3c3\ c7c7\ c8c8$ - 4 terms
 - Cross Product Terms - $c0c3\ c0c7\ c0c8$ - 6 terms
 $c3c7\ c3c8$
 $c7c8$
- Temperature and Time Terms - 5 terms
 - Simple Linear Terms - $t1\ h1$ - 2 terms
 - Pure Power Terms - $h2$ - 1 term
 - Cross Product Terms - $t1h1\ t1h2$ - 2 terms
- Cross Products of Composition With Temperature and Time - 70 terms
 - Each of the 14 chemical composition terms multiplied by each of the 5 temperature and time terms.

The previously described stepwise regression procedure was run for each alloy. For the A-516 alloy, a model with 23 terms was selected with a resulting R^2 of .73, while the A-537 alloy yielded a model with 27 terms and an R^2 of .77. The resulting prediction equations are given in Tables 12 and 13.

TABLE 12. Prediction Equation for the Hanford Facilities
Waste Model for A-516

$$\ln(\text{mpy}+1.25) =$$

0.49120	- 0.69672 * c8
+ 0.19042 * t1	+ 0.12453 * c0c0
+ 0.18201 * c0c7	- 0.05918 * c0c8
- 0.05657 * c3c7	+ 0.43338 * c7c7
- 0.68786 * c7c8	- 0.94744 * c8c8
- 0.04710 * t1h2	- 0.12716 * t1c0
+ 0.02976 * t1c3	- 0.62066 * t1c7
+ 0.31747 * t1c8	- 0.07964 * t1c0c7
- 0.37539 * t1c7c7	- 0.18118 * t1c8c8
- 0.04065 * h1c0c0	+ 0.03648 * h1c0c8
- 0.02780 * h2c3c8	- 0.01941 * t1h1c3
- 0.05492 * t1h2c0c7	

TABLE 13. Prediction Equation for the Hanford Facilities
Waste Model for A-537

$$\ln(\text{mpy}+1.25) =$$

0.48513	- 0.17665 * c0
- 0.47249 * c8	- 0.02997 * h1
+ 0.19403 * c0c0	- 0.19290 * c0c8
- 0.05796 * c3c7	+ 0.49664 * c7c7
- 0.55030 * c7c8	- 0.94142 * c8c8
+ 0.02795 * t1c3	- 0.80442 * t1c7
+ 0.02402 * t1c0c8	+ 0.13320 * t1c3c3
- 0.39373 * t1c7c7	- 0.28026 * t1c7c8
- 0.38944 * t1c8c8	- 0.02145 * h1c0c3
+ 0.03236 * h2c0	- 0.06337 * h2c8
- 0.01760 * h2c0c3	+ 0.07216 * h2c0c8
- 0.01564 * t1h1c3	+ 0.01788 * t1h1c3c7
- 0.02461 * t1h2c3	+ 0.02254 * t1h2c0c3
+ 0.01981 * t1h2c3c7	

The initial variable coding used for the Future PUREX model is as follows:

$$\begin{aligned} \ln mpy &= \ln(mpy + 1.25) \\ c0 &= (\log_{10}(OH) + 1) / 2 \\ c1 &= (A1 - .5) / .5 \\ c2 &= (NO_3 - 2.5) / 2.5 \\ c3 &= (NO_2 - 1) \\ c5 &= (F - .5) / .5 \\ c9 &= (Citrate - 1) \\ t1 &= (Temperature - 110) / 70 \\ h1 &= (Hours - 5800) / 3000. \end{aligned}$$

The initial polynomial model for the Future PUREX model consisted of the following 264 terms:

- Intercept - 1 term
- Chemical Composition Terms - 21 terms
 - Simple Linear Terms - $c0\ c1\ c2\ c3\ c5\ c9$ - 6 terms
 - Pure Quadratic Terms - $c0c0\ c1c1\ c2c2\ c3c3\ c5c5$ - 5 terms
(Considered major constituents)
 - Cross Product Terms - $c0c1\ c0c2\ c0c3\ c0c5$ - 10 terms
 $\quad\quad\quad c1c2\ c1c3\ c1c5$
 $\quad\quad\quad c2c3\ c2c5$
 $\quad\quad\quad c3c5$
- Temperature and Time Terms - 11 terms
 - Simple Linear Terms - $t1\ h1$ - 2 terms
 - Pure Power Terms - $h2\ t2\ t3$ - 3 terms
 - Cross Product Terms - $t1h1\ t2h1\ t3h1\ t1h2\ t2h2\ t3h2$ - 6 terms
- Cross Products of Composition With Temperature and Time - 231 terms
 - Each of the 21 chemical composition terms multiplied by each of the 11 temperature and time terms.

The previously described stepwise procedure was run for each alloy. For the A-516 alloy, a model with 60 terms was selected with a resulting R^2 of .72, while the A-537 alloy yielded a model with 73 terms and an R^2 of .74. The resulting prediction equations are given in Tables 14 and 15.

TABLE 14. Prediction Equation for the
Future PUREX Model for A-516

$$\ln(\text{mpy}+1.25) =$$

0.09874	+ 0.15015 * c1
- 0.21357 * c3	+ 0.10738 * c5
- 0.05644 * c9	+ 0.26211 * t1
+ 0.29901 * c0c0	- 0.22256 * c0c1
+ 0.20756 * c0c2	+ 0.31140 * c0c3
- 0.08517 * c0c5	- 0.11109 * c1c1
+ 0.06044 * c1c5	- 0.28940 * c2c2
+ 0.06175 * c2c3	+ 0.02466 * c2c5
+ 0.47303 * c3c3	- 0.37378 * t3
+ 0.05881 * t1c3	- 0.25776 * t1c5
- 0.42528 * t1c0c0	- 0.20299 * t1c0c2
- 0.26580 * t1c0c3	- 0.53571 * t1c1c1
+ 0.08984 * t1c1c2	- 0.22856 * t1c1c5
+ 0.15640 * t1c2c2	- 0.18035 * t2c1
+ 0.09189 * t2c3	- 0.08397 * t2c5
+ 0.11644 * t2c9	+ 0.21485 * t2c0c1
- 0.21997 * t2c0c2	- 0.18551 * t2c0c3
+ 0.11821 * t2c0c5	+ 0.06958 * t2c1c1
- 0.04807 * t2c1c5	+ 0.16304 * t2c2c2
- 0.03975 * t2c2c3	- 0.14207 * t2c3c3
+ 0.21814 * t3c5	+ 0.45216 * t3c0c0
+ 0.22375 * t3c0c3	+ 0.56366 * t3c1c1
- 0.07471 * t3c1c2	+ 0.18801 * t3c1c5
+ 0.02949 * h1c3	- 0.04462 * h1c0c0
+ 0.04914 * h1c2c2	- 0.06199 * h1c3c3
+ 0.05390 * h2c0c1	- 0.10045 * h2c0c3
- 0.02181 * t1h1c1	+ 0.08614 * t1h1c0c2
+ 0.05395 * t2h1c0c2	+ 0.06054 * t1h2c0c3
+ 0.10626 * t1h2c1c1	+ 0.14529 * t2h2c0c3
- 0.13203 * t3h2c1c1	- 0.03384 * t3h2c1c3

TABLE 15. Prediction Equation for the
Future PUREX Model for A-537

$$\ln(\text{mpy}+1.25) =$$

-0.02747	+ 0.13656 * c1
- 0.31590 * c3	+ 0.12089 * c5
- 0.05652 * h1	+ 0.56172 * c0c0
- 0.17505 * c0c1	+ 0.26634 * c0c2
+ 0.44921 * c0c3	- 0.11591 * c1c1
+ 0.04517 * c1c5	- 0.37008 * c2c2
+ 0.10497 * c2c3	+ 0.04898 * c2c5
+ 0.62019 * c3c3	+ 0.13328 * t2
- 0.08502 * t1c2	+ 0.18695 * t1c3
- 0.41623 * t1c5	- 0.48947 * t1c0c0
- 0.52049 * t1c0c2	- 0.11358 * t1c0c3
+ 0.25372 * t1c0c5	- 0.21018 * t1c1c1
+ 0.08104 * t1c1c2	- 0.05342 * t1c1c3
- 0.28878 * t1c1c5	+ 0.44402 * t1c2c2
- 0.12472 * t1c2c3	+ 0.04974 * t1c2c5
- 0.10873 * t1c3c3	- 0.06194 * t2c1
+ 0.14930 * t2c3	- 0.09779 * t2c5
- 0.24522 * t2c0c0	+ 0.15501 * t2c0c1
- 0.41169 * t2c0c2	- 0.19426 * t2c0c3
+ 0.06582 * t2c1c1	+ 0.04498 * t2c1c3
+ 0.15209 * t2c2c2	- 0.10993 * t2c2c3
- 0.36470 * t2c3c3	+ 0.16313 * t2c5c5
- 0.07786 * t3c3	+ 0.35635 * t3c5
+ 0.53624 * t3c0c0	+ 0.21225 * t3c0c2
+ 0.11317 * t3c0c3	- 0.34402 * t3c0c5
+ 0.21271 * t3c1c1	+ 0.20321 * t3c1c5
- 0.30664 * t3c2c2	- 0.04929 * t3c3c5
+ 0.03847 * h1c3	+ 0.04555 * h1c1c1
+ 0.09184 * h1c2c2	- 0.05916 * h1c3c3
+ 0.02404 * h2c3	- 0.08787 * h2c0c0
- 0.07987 * h2c0c3	- 0.04798 * h2c0c5
- 0.02125 * h2c1c3	- 0.06984 * t1h1c1
- 0.03439 * t1h1c1c1	+ 0.09911 * t1h1c5c5
- 0.03140 * t2h1c1	- 0.03621 * t2h1c2c2
- 0.02658 * t2h1c2c3	- 0.03844 * t2h1c2c5
+ 0.06462 * t3h1c1	+ 0.02163 * t3h1c3c5
+ 0.04126 * t3h2c0c1	

The initial variable coding used for the Double Shell Slurry model is as follows:

```

lnmpy = ln ( mpy + 1.25 )
c0 = ( OH - 5 ) / 5
c1 = ( A1 - 1.8 ) / 1.8
c2 = ( NO3 - 3.75 ) / 3.75
c3 = ( NO2 - 4 ) / 4
c4 = ( Complexant - .5 ) / .5
c6 = ( CO3 - .1 ) / .1
c7 = ( PO4 - .6 ) / .6
c8 = ( SO4 - .08 ) / .08
c9 = ( Citrate - .3 ) / .3
t1 = ( Temperature - 110 ) / 70
h1 = ( Hours - 5800 ) / 3000.

```

The initial polynomial model consisted of the following 300 terms:

- Intercept - 1 term
- Chemical Composition Terms - 24 terms
 - Simple Linear Terms - c0 c1 c2 c3 c4 c6 c7 c8 c9 - 9 terms
 - Pure Quadratic Terms - c0c0 c1c1 c2c2 c3c3 c4c4 - 5 terms
(Considered major constituents)
 - Cross Product Terms - c0c1 c0c2 c0c3 c0c4 - 10 terms
c1c2 c1c3 c1c4
c2c3 c2c4
c3c4
- Temperature and Time Terms - 11 terms
 - Simple Linear Terms - t1 h1 - 2 terms
 - Pure Power Terms - h2 t2 t3 - 3 terms
 - Cross Product Terms - t1h1 t2h1 t3h1 t1h2 t2h2 t3h2 - 6 terms
- Cross Products of Composition With Temperature and Time - 264 terms
 - Each of the 24 chemical composition terms multiplied by each of the 11 temperature and time terms.

The previously described stepwise procedure was run for each alloy. For the A-516 alloy, a model with 138 terms was selected with a resulting R^2 of .87, while the A-537 alloy yielded a model with 123 terms and an R^2 of .93. The resulting prediction equations are given in Tables 16 and 17.

It is clear from a review of the R^2 values for the individual waste type models that a much better prediction will be obtained for data that closely match the conditions of that waste type in comparison to predictions from the unified model. Therefore, the strategy to be applied in utilizing these predictive models is to use a waste type specific model whenever possible for the best prediction, but to use the unified model for conditions that do not match a particular waste type but are within the overall combined range of compositions.

INTERPRETING THE FITTED MODELS

Several precautionary statements need to be made about attempts to interpret the values of coefficients for terms in the models. First, most of the boundaries of the experimental region are determined by limitations on the "nominal" levels of the ten anions in a chemical solution. That is, because of solubilities and saturation, some of the anions which may be added to a solution at their highest levels may be effective at much lower levels due to the presence of other components at high or low levels. These relationships were taken into account in designing the experiments for each one of the waste types and the chemical compositions which are included in each experiment are specified in terms of the "nominal" levels of the anions. Second, all terms appear as coded values and not raw values. Third, and most important, the reader should be aware of the correlations among the terms in the predictor equations and the effect this has on interpretation. Although the fitted models provided in this report will produce reasonably good predictions of corrosion rates in their respective experimental regions, the values of the individual estimated coefficients should not be used to infer the effect or importance of the corresponding terms relative to the corrosion rate. The primary use of these models is for predicting corrosion rates and not for corrosion process modeling.

TABLE 16. Prediction Equation for the
Double Shell Slurry Model for A-516

$$\ln(\text{mpy}+1.25) =$$

0.35623	+ 0.06738*c0	- 0.02347*c6
+ 0.25686*t1	- 0.04862*h1	+ 0.08390*c0c2
+ 0.05145*c0c3	- 0.05637*c1c3	- 0.03443*c2c4
- 0.05326*c3c3	+ 0.03322*c4c4	+ 0.07015*t2
- 0.17715*t3	- 0.17600*t3h1	- 0.23498*t2h2
- 0.22804*t3h2	+ 0.37881*t1c0	+ 0.19508*t1c1
- 0.08595*t1c2	+ 0.05473*t1c8	+ 0.03973*t1c9
+ 0.49876*t1c0c1	- 0.15824*t1c0c4	+ 0.08228*t1c1c4
- 0.23792*t1c2c2	- 0.08699*t1c2c4	- 0.07762*t1c3c3
+ 1.27783*t2c0	+ 0.30020*t2c1	+ 0.22079*t2c3
- 0.41118*t2c4	+ 0.27982*t2c6	+ 0.09121*t2c8
+ 0.04623*t2c9	- 0.14225*t2c0c0	+ 1.30946*t2c0c1
- 0.80931*t2c0c2	- 0.33061*t2c0c3	- 0.17411*t2c0c4
+ 0.05522*t2c1c2	+ 0.80180*t2c1c3	- 0.39727*t2c1c4
- 0.37731*t2c2c2	+ 0.22213*t2c2c3	+ 0.56886*t2c3c3
- 0.15096*t2c3c4	+ 0.90473*t3c0	+ 0.19095*t3c3
- 0.46926*t3c4	+ 0.23861*t3c6	- 0.18521*t3c0c0
+ 0.77224*t3c0c1	- 0.78476*t3c0c2	- 0.30361*t3c0c3
+ 0.76046*t3c1c3	- 0.55555*t3c1c4	- 0.12974*t3c2c2
+ 0.15545*t3c2c3	+ 0.61573*t3c3c3	- 0.13841*t3c3c4
- 0.05843*h1c0	+ 0.02624*h1c1	- 0.04375*h1c8
+ 0.13921*h2c0	- 0.03647*h2c2	+ 0.02789*h2c4
- 0.01718*h2c7	+ 0.02971*h2c8	+ 0.09750*h2c0c1
- 0.07650*h2c0c2	+ 0.07466*h2c3c3	+ 0.04368*t1h1c4
+ 0.06753*t1h1c6	- 0.09846*t1h1c8	- 0.02562*t1h1c9
- 0.05626*t1h1c1c1	- 0.08697*t1h1c1c2	- 0.09872*t1h1c2c2
+ 0.13825*t2h1c6	+ 0.09068*t2h1c8	+ 0.01973*t2h1c9
- 0.06424*t2h1c0c1	- 0.21248*t2h1c0c2	- 0.20911*t2h1c0c3
+ 0.19216*t2h1c1c3	- 0.20282*t2h1c2c2	+ 0.06628*t2h1c2c3
+ 0.09573*t2h1c2c4	+ 0.30310*t3h1c1	+ 0.30948*t3h1c2
+ 0.35245*t3h1c3	- 0.34298*t3h1c4	+ 0.32392*t3h1c6
+ 0.14172*t3h1c8	- 0.18050*t3h1c0c0	- 0.28486*t3h1c0c2
- 0.20337*t3h1c0c3	- 0.29758*t3h1c0c4	+ 0.32737*t3h1c1c1
+ 0.44818*t3h1c1c2	+ 0.48974*t3h1c1c3	+ 0.36291*t3h1c2c3
- 0.10353*t1h2c3	- 0.09584*t1h2c7	- 0.13128*t1h2c1c3
- 0.79964*t2h2c0	- 0.73167*t2h2c1	- 0.18738*t2h2c3
- 0.27765*t2h2c6	- 0.08778*t2h2c7	- 0.17980*t2h2c0c0
- 0.95796*t2h2c0c1	+ 0.75500*t2h2c0c2	- 0.10604*t2h2c0c3
+ 0.56810*t2h2c0c4	+ 0.40366*t2h2c1c1	- 0.24157*t2h2c1c3
- 0.36621*t2h2c1c4	+ 0.34287*t2h2c2c2	- 0.43098*t2h2c2c3
- 0.60234*t2h2c3c3	+ 0.11074*t2h2c3c4	- 0.20131*t2h2c4c4
- 0.92076*t3h2c0	- 0.19490*t3h2c1	+ 0.34814*t3h2c2
+ 0.26132*t3h2c3	- 0.05672*t3h2c9	- 0.17718*t3h2c0c0
- 1.13872*t3h2c0c1	+ 0.66634*t3h2c0c2	+ 0.18220*t3h2c0c4
+ 0.57041*t3h2c1c1	+ 0.41926*t3h2c1c2	+ 0.35630*t3h2c2c2
- 0.60755*t3h2c3c3	+ 0.13393*t3h2c3c4	- 0.10743*t3h2c4c4

TABLE 17. Prediction Equation for the
Double Shell Slurry Model for A-537

$$\ln(\text{mpy}+1.25) =$$

0.37056	+ 0.11927*c0	- 0.02342*c7
+ 0.01044*c8	+ 0.12178*t1	- 0.03829*h1
+ 0.02270*c0c0	+ 0.04895*c0c4	- 0.02715*c1c4
- 0.01474*c2c4	- 0.11119*t1h1	- 0.03053*t2h1
+ 0.16028*t1h2	- 0.05020*t2h2	- 0.14079*t3h2
+ 0.08045*t1c0	+ 0.07652*t1c1	+ 0.10885*t1c0c0
- 0.09139*t1c0c2	- 0.02482*t1c0c4	- 0.03900*t1c2c2
+ 0.28135*t2c0	+ 0.07072*t2c1	+ 0.06746*t2c2
+ 0.15747*t2c3	- 0.09862*t2c4	+ 0.07945*t2c7
+ 0.13908*t2c0c1	- 0.19892*t2c0c2	+ 0.09085*t2c0c3
- 0.09023*t2c0c4	+ 0.04596*t2c1c1	+ 0.27585*t2c1c3
- 0.15524*t2c1c4	+ 0.06598*t2c2c3	+ 0.33739*t2c3c3
- 0.15406*t2c3c4	+ 0.11057*t2c4c4	+ 0.23344*t3c0
+ 0.07075*t3c2	+ 0.17134*t3c3	+ 0.06021*t3c7
- 0.05317*t3c0c0	- 0.11893*t3c0c2	+ 0.09767*t3c0c3
+ 0.05595*t3c0c4	+ 0.27565*t3c1c3	- 0.15817*t3c1c4
+ 0.09030*t3c2c3	+ 0.35537*t3c3c3	- 0.13883*t3c3c4
+ 0.16408*t3c4c4	- 0.02109*h1c0	- 0.01327*h1c0c3
- 0.02982*h1c1c2	+ 0.02044*h1c2c3	+ 0.01231*h1c3c4
- 0.04678*h2c6	+ 0.05438*h2c0c0	+ 0.06485*h2c0c2
+ 0.03366*h2c0c3	- 0.02918*h2c0c4	+ 0.03675*h2c1c2
- 0.05080*h2c1c3	- 0.02821*h2c2c3	- 0.02586*h2c4c4
- 0.04527*t1h1c1	+ 0.02480*t1h1c6	- 0.09625*t1h1c0c0
- 0.03815*t1h1c0c3	+ 0.04588*t1h1c3c3	- 0.07023*t2h1c0
+ 0.01817*t2h1c7	- 0.05497*t2h1c0c0	- 0.07411*t2h1c0c2
+ 0.08310*t2h1c1c2	+ 0.03018*t2h1c2c4	+ 0.02867*t2h1c3c3
- 0.03306*t3h1c0	+ 0.06009*t3h1c3	- 0.08355*t3h1c0c0
- 0.04943*t3h1c0c2	+ 0.09693*t3h1c1c1	+ 0.07707*t3h1c1c3
- 0.06387*t3h1c2c4	+ 0.05534*t3h1c3c4	+ 0.02716*t3h1c4c4
- 0.13971*t1h2c0	- 0.12221*t1h2c6	+ 0.04816*t1h2c9
+ 0.07451*t1h2c0c2	+ 0.09468*t1h2c2c3	+ 0.03766*t2h2c0
- 0.08216*t2h2c1	+ 0.18219*t2h2c6	- 0.07950*t2h2c7
- 0.01675*t2h2c9	- 0.13014*t2h2c0c0	+ 0.13433*t2h2c0c2
- 0.21682*t2h2c0c3	+ 0.07601*t2h2c0c4	+ 0.07516*t2h2c1c1
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+ 0.04656*t3h2c3	+ 0.46753*t3h2c6	- 0.07208*t3h2c7
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- 0.26186*t3h2c0c4	+ 0.25929*t3h2c1c2	- 0.18256*t3h2c1c3
+ 0.33627*t3h2c1c4	- 0.50403*t3h2c3c3	+ 0.21108*t3h2c3c4

PART 3 - USING THE FITTED MODELS TO PREDICT CORROSION RATES

The corrosion rate prediction models developed in this report should be useful in producing estimates of corrosion rates for various chemical compositions at selected exposure times and temperatures within the ranges of the data used for each model. An example of implementing the prediction equations is given below. A computer program has been developed which will do these calculations and is recommended over calculating a predicted value by hand. This program is described in the next section. In addition to examining single compositions, a range can be examined; this procedure is reviewed in the final section.

To achieve maximum computing precision in the regression analysis, the exposure times and temperatures and chemical constituents were coded so that they are centered near zero and are in comparable units. Thus, the fitted models presented are in terms of these coded variables. To apply a particular fitted model, the input variables (time, temperature and composition) must first be coded in the same way as the variables used in fitting the model. These coded values are then substituted into the appropriate equation and a predicted value of $\ln(\text{mpy} + 1.25)$ is calculated. This value is then transformed as follows :

$$\text{mpy} = \exp(\text{predicted value}) - 1.25,$$

which is the predicted corrosion rate.

As an example, consider predicting the corrosion rate for an A-537 alloy exposed to 50°C temperature for 8760 hours in a waste composition similar to Composition 16 in the Customer Waste experiment. The coded variables are as follows:

$$\begin{aligned} c0 &= (\log_{10}(.0001) + 2) / 2 &= -1 \\ c3 &= (.01 - .005) / .005 &= 1 \\ c7 &= (3.9 - 2) / 2 &= .95 \\ c8 &= (1.3 - 1.3) / 1.5 &= 0 \\ t1 &= (50 - 37.5) / 12.5 &= 1 \\ h1 &= (8760 - 5800) / 3000 &= .99. \end{aligned}$$

These coded variables are substituted into the prediction equation for A-537 alloy exposed to Hanford Facilities Waste type of compositions. The resulting value is .2416. The predicted corrosion rate is then $\exp(.2416) - 1.25 = 0.023$ mpy. This compares with an observed corrosion rate of .068 mpy for an actual specimen in the study.

A COMPUTER PROGRAM FOR PREDICTING CORROSION RATES

The fitted models developed in this report for predicting corrosion rates have been incorporated into an interactive computer program. The program requests the user to state the alloy, waste type, exposure time, temperature, and the chemical constituents appropriate to the selected waste type. It then utilizes the fitted models to predict the average corrosion rate, in mpy, that would be expected to occur under the specified conditions. The program also computes an estimate of the standard deviation of the predicted value. The program uses these results to compute upper confidence limits for the true average corrosion rate for that set of conditions at the 90, 95, and 99% confidence levels. It also computes upper 90/90, 95/90 and 99/90 tolerance limits for the average corrosion rates expected to occur in repeated experiments under this set of conditions. For example, an upper 95/90 tolerance limit indicates that we are 95% confident that 90% of average corrosion rates observed under this set of conditions will fall below the limit. The FORTRAN code for the program is not presented in this report but is available from the authors either on tape or as a hard-copy listing.

Once the program is compiled on a computer it is executed by giving the command "RUN CORR" and then responding to the prompts for input. To illustrate this, Table 18 displays the prompts and the response to each of the prompts for a particular set of conditions. The first prompt asks whether the resulting output should be stored in a file. If the response is Y (yes), the next prompt is for the file name. In this example, the results are not to be stored in a file and the response is N (no). The program then prompts for the alloy, waste type, exposure time, temperature, and the constituents appropriate to the selected waste type expressed in moles/liter.

After completing the sequence of prompts and responses shown in Table 18, the program printed the results displayed in Table 19. The program first outputs the values of all conditions that were input by the user for the prediction. It then gives the predicted corrosion rate in mpy and the standard deviation of the predicted value. This is followed by a comparison of the standard deviation of the predicted value with the square root of the residual mean square for that model. If the standard deviation of the predicted value is more than 100% of the square root of the residual mean square, the set of conditions specified may be outside of the experimental region used to develop the model, in which case the predicted corrosion rate may be invalid. The program then prints the upper confidence limits and tolerance limits described above. In the example, there is 99% confidence that the true average corrosion rate is less than .35 mpy and that 90% of the corrosion values that might occur under the given conditions will be less than .50 mpy.

TABLE 18. Prompts and Responses for Example Run
of Prediction Program CORR

RUN CORR

DO YOU WANT OUTPUT TO A FILE? Y/N
N

ENTER WHICH WASTE MODEL TO USE

- 1 --• DOUBLE SHELL SLURRY
- 2 --• FUTURE PUREX
- 3 --• HANFORD FACILITIES WASTE
- 4 --• ALL OF THEM TOGETHER (COMBINED MODEL)

1

INPUT WHICH ALLOY YOU ARE USING: (516 OR 537)
537

INPUT EXPOSURE TIME IN HOURS
8898

INPUT TEMPERATURE IN DEGREES CENTIGRADE
140

NOW INPUTTING CHEMICAL CONTENTS IN MOLES/LITER:

INPUT HYDROXIDE
4

INPUT ALUMINATE
3.7

INPUT NITRATE
1

INPUT NITRITE
1

INPUT COMPLEXANT
0

INPUT CARBONATE
.2

INPUT PHOSPHATE
.6

INPUT SULPHATE
.05

INPUT CITRATE
.6

TABLE 19. Output From Prediction Program CORR

Using the Double Shell Slurry Model for Alloy 537
With Exposure Time = 8898 Hours
Temperature = 140 Degrees Centigrade

Chemical Contents in Mole/Liter:

Hydroxide	=	4.0000
Aluminate	=	3.7000
Nitrate	=	1.0000
Nitrite	=	1.0000
Complexant	=	0.0000
Fluoride	=	0.0000
Carbonate	=	0.2000
Phosphate	=	0.6000
Sulphate	=	0.0500
Citrate	=	0.6000

[illegible]

The Standard Deviation of the Predicted Corrosion Rate Is04 MPY

The Standard Deviation of the Predicted Value is. 42%
of the Experimental Error Standard Deviation

Under These Conditions, There is 90% Confidence That

The True Average Corrosion Rate Is Less Than.30 MPY

And That 90% of the Corrosion Rates That Might Occur Will Be $<.45$ MPY

Under These Conditions, There is 95% Confidence That

The True Average Corrosion Rate Is Less Than.32 MPY

And That 90% of the Corrosion Rates That Might Occur Will Be $<.47$ MPY

Under These Conditions, There is 99% Confidence That

The True Average Corrosion Rate Is Less Than.35 MPY

And That 90% of the Corrosion Rates That Might Occur Will Be <.50 MPY

DATA PRESENTATION

Presentation of the fitted models in graphical form provides an opportunity to visualize expected trends as a function of multiple variables. However, it is very important to be aware of the danger of assuming that a given selected variable actually influences the corrosion rate because of the possible correlations among the terms (components) in the equations. Nevertheless, a general idea of what the behavior will be combined with graphical displays provides an opportunity to

predict the effects of possible changes which can be confirmed with a limited series of experiments.

Figure 2 is a plot of corrosion rate as a function of temperature and hydroxide concentration at high nitrate, nitrite, and aluminate concentrations using the combined model. The curve looks not unreasonable. However, by taking the data of Reynolds and Herting⁽¹²⁾ into account, the system is found to be compositionally unattainable; therefore, the plotting code prints a grid at the zero corrosion rate level, Figure 3.

By reducing the (fixed) concentrations of the nitrate, nitrite, and aluminate, the shape of curve, Figure 4 (comparable to Figure 2), is changed slightly. The blank region at the left side of the graph is where the predictor model predicts a zero or negative corrosion rate. If the solubility effect is again considered, Figure 5 is the result.

The effect of solubility, as presently handled in the graphical display, is overly conservative. Though correct in calling out the solubility effect, it does not take into consideration compositions beyond those limits where solids may be present which may have some effect on the corrosion rate, for example by preventing free convection or slowing diffusion.

Therefore for the user who needs accurate corrosion data, the proper procedure is to obtain the data as illustrated in Tables 18 and 19. Although information presented in that format may contain regions in which the solubility is exceeded, it is based on actual tests, some of which contained excess salts.

FIGURE 2. Unrestricted Plot of Concentrated DSS Corrosion Rates

FIGURE 3. Solubility Limited, Variation of Figure 2

FIGURE 4. Unrestricted Low Concentration DSS Plot

FIGURE 5. Solubility Limited, Variation of Figure 4

SUMMARY

A series of tests using Double Shell Slurry, Future PUREX, and Hanford Facilities wastes were completed during a four year period. Except for very dilute and high temperature concentrated conditions, the corrosion rates of A-537 and A-516 steels were low, less than 1 mpy (25 $\mu\text{m}/\text{y}$) and generally less than 0.5 mpy.

Administrative controls are used to remain out of the very dilute region and operating procedures limit progress to the other extreme. Thus, the normal operating conditions for the wastes tested do not lead to significant corrosion rates.

Localized and stress corrosion cracking were only observed under the extreme conditions noted above. Stress corrosion cracking appears to be less prevalent than in related work performed at Savannah River Laboratory.

The data gathered has been analyzed and combined into several predictor models, based on polynomial equations. These equations are presented in the text of the report. The authors can also provide the information in computerized format to interested persons.

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