Double Shell Slurry Low Temperature Corrosion Tests

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September 1983

**Prepared for the U.S. Department of Energy
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**Pacific Northwest Laboratory
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August 23, 1984

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Richland, Washington 99352

Dear Nick:

SUBJECT: MAXIMUM CAUSTIC CONCENTRATIONS - DILUTE NITRATE CONDITIONS

At 140°C our Mixture C-11 (5M OH⁻ and 0.3M NO₃⁻) caused cracking. No cracking occurred at or below 100°C for the mixture. Therefore, for the < 1M NO₃ solutions, OH⁻ should be kept below 5M in the 100-140°C range.

Nitrates at too low a concentration (specific level is not in the literature nor have we worked on this problem) can accelerate caustic cracking. For lack of better numbers, we should use data for sodium hydroxide. For stress-relieved steel you should not exceed 100°C or 10M caustic. For unrelieved tanks, and that concentration, temperatures above 70-80°C are not recommended. Because the tanks are stress-relieved the upper value of 80°C is probably a more reasonable value, particularly in light of the fact that tank walls are not a heating surface and the caustic should not be concentrated there.

Sincerely,

James R. Divine
Staff Engineer
Corrosion Research and Engineering

JRD:pl

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ABSTRACT

A series of year long tests have been completed on potential double shell slurry (DSS) compositions at temperatures up to 100°C. These tests have sought data on uniform corrosion, pitting, and stress corrosion cracking. No indication of the latter two types of corrosion were observed within the test matrix.

Corrosion rates after four months were generally below the 1 mpy (25 $\mu\text{m}/\text{y}$) design limit. By the end of twelve months all results were below this limit and, except for very concentrated mixtures, all were below 0.5 mpy.

Prediction equations were generated from a model fitted to the data. The equations provide a rapid means of estimating the corrosion rate for proposed DSS compositions.

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INTRODUCTION

Historically, interest in the corrosion of radwaste tanks goes back to the early fifties. Several 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 up to about pH 13 and, in the case of field tests, at temperatures estimated to be between 250 and 300°F (120-150°C). Uniform corrosion rates of 0.5 mpy (13 $\mu\text{m}/\text{y}$) or less were measured in both the liquid and vapor phases.

In 1980, an environmental impact statement (EIS) was prepared for the 241-AN and AW double shell waste storage tanks.⁽¹⁾ The available data, needed to prepare the EIS, garnered from the literature in general, from Savannah River experiments, and from Hanford work, were inadequate to describe the behavior of the waste proposed for storage in the new tanks. It was concluded that further corrosion rate development work was needed. It was possible, however, to state that, based on favorable but 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 relevant corrosion data meant initiating tests of tank construction material in an expanded OSS composition range and in other waste compositions with temperatures up to the tank design temperature, 350°F (177°C). The additional corrosion data are needed to allow safe operation of the waste storage tanks in a wide range of waste fluids.

This document provides a summary of the corrosion work performed on DSS waste at temperatures at or below 100°C. The prediction equations developed are still subject to revision as additional data are generated at higher temperatures and at other compositions and as improved models are developed. The basic conclusions are expected to remain valid.

The data are presented as a best fit to a multiparameter model. Because of the way the model is fit to the data (described in the text), it is not possible to conclude from the final prediction equation whether the presence or absence of a component is significant.

Theory

The mechanism of corrosion in the DSS mixtures has not been established though work is in progress. Potentially it is a very complex process because of all of the components as seen in Table 1.

Using only the major chemical components, reactions which are thermodynamically possible are:

Based strictly on visual observations of the oxide films it appears both equations (1) and (2), (3) and (4), or both sets can occur to produce both ferrous and ferric hydroxides. Then equations (5), (6), and (7) occur to produce both Fe_2O_3 and Fe_3O_4 .

Looking at solubility behavior, Lange's Handbook⁽²⁾ shows that the stable complexes are hydroxides and organic ligands. Fe(OH)_3 has a $\text{pK}_{\text{sp}} = 37.4$ and $\log K$'s (formation constants) of 11.87, 21.17, and 29.67 for $\log K_1$, $\log K_2$, and $\log K_3$, respectively. Similarly, the $\log K_1$ for

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
CO_3^{--} (2 fixed values)	0.2 or 0.25
PO_4^{--}	0.0 - 2.0
EDTA/HEDTA	0.0 - 1.0
Citrate (3 fixed values)	0.0, 0.2, or 0.6
SO_4^{--} (single value)	0.05

FIGURE 1. Weight Loss and U-Bend Specimens

ferric EDTA is 24.23 and for ferric citrate, 25.0. Other data are available on some of the remaining species, but the numbers are significantly smaller. They are not listed because no detailed calculations are being made. The numbers given above do suggest that the oxide/-hydroxides will be major components.

This general description is consistent with the types of deposits found. The deposits, depending on the particular waste composition, range in color from the red/brown of hematite (Fe_2O_3) to the black of magnetite (Fe_3O_4). In some cases there is an inner layer of hematite with a thin magnetite outer layer suggesting equation (5) is predominant and that equations (6) and (7) occur where intimate solution contact is possible.

Technical Approach

Determination of the corrosion behavior of the waste compositions towards the storage tank material was performed using laboratory coupon weight-loss and U-bend tests (Figure 1).^{*} The laboratory coupon tests were designed to cover the range of fluid compositions expected to be generated. These ranges are shown in Table 1. Not all combinations of composition are possible because of solubility constraints. Personnel at Rockwell Hanford Operations determined the solubility constraints using the computer code CORROS, also known by the name PREDICT-3. As a result of the solubility constraints and proposed test size, a computerized statistical test design was generated that required the use of 28 different solutions at four temperatures, 40, 60, 80, and 100°C. The compositions are shown in Table 2. This test matrix thus includes 112 tests. The test matrix was further expanded to include three exposure times, 4, 8, and 12 months for each of the 112 solution-temperature pairs. Each of the 336 tests had replicate coupons.

The corrosion rates obtained from the weight-loss tests were used to generate prediction equations. Both linear and non-linear equations were used to produce predictor expressions. The final result is a

^{*}The weight loss coupons were approximately 2 cm x 5 cm in dimension. The thickness was dependent on alloy type and heat but was about 0.5 or 0.3 cm. The U-bend stock was the same thickness but about 1.2 cm wide and 14 cm long; they were bent over a 1.9 cm diameter mandrel.

TABLE 2
TEST COMPOSITIONS
MOLARITY
100°C

Reference #	OH ⁻	AlO ₂ ⁻	NO ₃ ⁻	NO ₂ ⁻	CO ₃ ⁻	PO ₄ ⁻³	SO ₄ ⁻²	CMFX	Cit	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>4.6</u> +100 xs	1.0	<u>0.14</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>6.25</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> +100 xs	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*	<u>2.7</u> +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> +100 xs	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	1.303
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	1.196
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

series of equations to predict corrosion rate as a function of composition, temperature, alloy and time described later in the report.

Materials

The AN and AW double shell storage tanks are constructed of A-537, Grade 1, normalized carbon steel. Earlier single shell tanks were constructed of A-516 carbon steel. Thus tests included both alloys to provide continuity of operating experience between systems and waste types. Table 3 shows the nominal compositions of each alloy as well as their measured composition. The specified A-516 composition lies within the specified limits of the A-537 (no significant effect in corrosion rate is expected to be caused by the difference in carbon content). A-307 Grade A nuts and bolts were used in the U-bend tests. Because of potential dissolution and strength problems of insulation materials, no insulators were used.

TABLE 3. Alloy Compositions (wt w%)

	<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
Laboratory ^(a)	.15	.16	.20	1.27	<.01	.21	.025	.009	.43
Vendor Cert	.14	.14	.19	1.26		.18	.026	.006	.43
<u>ASTM A-516 Nominal</u>	.27M			.8-1.25			.035M	.04M	.13-.33
Laboratory	.13	.03	.10	.86	<.01	.05	.009	.019	.26
Vendor Cert	.12	#	#	.93	#	#	.01	.022	.25
<u>ASTM A-307 Nominal</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; #-not measured; ^(a) Corrosion Research & Engineering Section, PNL or E.M. Jorgensen Co., Seattle, Washington.

Analyses in the Corrosion Research & Engineering Section Laboratory, Table 3, for the two test alloys produced results in agreement with the standard values; carbon was not measured. Vendor certified compositions were consistent with these other analyses and the ASTM specification although the carbon contents were both about half the limits. The compositions of the nuts and bolts could render them slightly anodic to the other alloys; no sign of such behavior was noted under the conditions tested, however.

The major difference observed in the two alloys was the starting surface finish. In order to provide a representative corrosion surface machining was performed only on the edges and not on the original surfaces of the weight-loss coupons. The original mill scale was removed in an inhibited hydrochloric acid bath. The coupons were then annealed for one hour at 1100°F in air and weighed. The resulting actual surface of the A-537 coupons is rougher and the area is estimated, using profilometer measurements, at about 2.4 times as great as that of the A-516 coupons even though the geometric area (based on length, width, and thickness measurements) is only 1.1 times as large.

Coupons for the 8-month tests were sheared coupons compared to the edge milled coupons for the 4- and 12-month tests. The machining step was eliminated because the annealing step removes stresses set up during shearing. Both types of A-516 coupons had the same surface area of 26.5 cm². The A-537 coupons with milled edges had a surface area of 29.4 cm² while the sheared A-537 coupons had a geometric surface area of 29.1 cm².

Procedures

After cutting the specimens to size, each was numbered using a steel punch and then cleaned by one of the following methods:

- U-Bend Specimens: Scrubbed with soap and non-metallic brush, rinsed in deionized water, and then rinsed with acetone.
- Weight Loss Specimens: Coupons were immersed in an aqueous solution of 50 vol.% HCl and 1.25 vol.% formaldehyde for 10-15 minutes to remove scale, rinsed to remove solution, scrubbed with soap and a non-metallic brush, and then rinsed with deionized water and then acetone.

Following cleaning, each specimen was weighed to the nearest tenth of a milligram on a balance interconnected to a computer. This computerized technique reduced weighing and weight transcription errors, put the data in an accessible and, for safety, duplicated file, and provided a ready means of using the data for later calculations; a hard copy of the weight data was also kept.

After weighing, each specimen was heat treated to 1100°F (593°C) for one hour in air 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-bend specimens were weighed again after bending but before final assembly.

Reference specimens were also used which went through the entire procedure except testing in the "waste" mixtures. These were used to calibrate the metal lost in the acid cleaning bath and to estimate the amount of oxide produced in the annealing process. It was determined that the amount of iron lost because of the acid cleaning was negligibly small both relative to other weight changes and on an absolute scale, less than about 2 mg in a 15 minute cleaning period; cleaning of the actual test coupons requires less than 15 minutes. Thus, there were no corrections made for metal lost due to the cleaning procedure.

The oxide, formed during the annealing period, amounted to 1.112 ± 0.020 gm for the A-537 and 0.036 ± 0.029 g for the A-516 for coupons with machined edges. For coupons with sheared edges, the oxide formed amounted to 0.080 ± 0.012 gm for the A-537, and 0.070 ± 0.005 g for the A-516. These values are equivalent to 0.19, 0.007, 0.14, and 0.13 mils of corrosion; these were subtracted from the measured corrosion at the end of the tests.

The individual test containers were 16 oz., approximately 0.5 l, wide mouth, FEP or TFE Teflon® bottles with screw caps. The bottles were filled with the test mixture and then were put into constant temperature baths,

glycerin filled, at 100°C. The test mixtures were allowed to heat up and in some cases soften and liquify to facilitate specimen immersion. Prior to inserting the specimens, the bottles were rinsed off in deionized water to prevent the contamination of the test solution by glycerin.

The specimens were suspended off the bottoms of the bottles with Teflon® tape. After all specimens were immersed, the ends of the support tapes were secured around the top of the bottle with a wrap of Teflon® tape and the lid replaced. The baths were adjusted to the operating temperature of the test, 40, 60, 80, or 100°C, after specimens were put in. At least once a week the tank temperatures were checked and adjusted if needed.

Generally three weight-loss coupons and one U-bend specimen were placed in each bottle. For the 4-month test only two coupons (both A-516) plus the U-bend were used. For the 8-month test two A-537 weight-loss coupons were suspended with one near the bottom of the bottle and one suspended half-way down. The weight-loss coupon with the lowest number was nearest the bottom of the bottle. One A-537 U-bend was used in each bottle. One A-516 weight-loss coupon was used in each bottle and was suspended near the bottom. A-516 U-bends were added about 25 days after start up, one per bottle, to help facilitate the comparison with the other DSS tests. For the 12-month test, three weight-loss coupons (one A-537 and two A-516) plus an A-516 U-bend were placed in each bottle.

At the completion of the tests the corrosion coupons were removed from the test bottles, visually examined, photographed, rinsed with deionized water and acetone, dried, and then stored in a desiccator. They were again visually examined for pitting and cracking, and a description of each coupon was written. Next the weight-loss coupons were cleaned in inhibited hydrochloric acid (the composition as noted above), dried and weighed; the U-bends are not cleaned but are kept as archive references for color and appearance.

Chemical analyses of the waste composition for OH^- , NO_2^- , Cl^- , SO_4^{2-} , alumina and phosphate was performed at the end of 4-month tests. Similar analyses were also performed for seven solutions, representing most ranges, at the start and at the completion of the 8-month test. Of these seven solutions three of them had their solid phase analyzed.

Results

Examination of the coupons after exposure to the various waste fluid compositions, temperatures, and test durations revealed little variation in overall appearance. The coupons were all intact and most commonly covered with an even gray coating. Occasionally variations such as the presence of large, black crystals or scattered areas of rust color were present. The black crystals were analyzed from one test (4-month, Mixture #33) and were determined to be sodium ferrite (NaFeO_2). Figure 2 shows the appearance the samples displayed after exposure to the 28 test mixtures after 8 months at 100°C ; 4- and 12-month specimens were similar. The mixtures represented (see Table 2 for numbering system) begin with Number 1 at the top left and are ordered left to right, top to bottom with 59 at the bottom right. The color of the deposits on the U-bends from other related tests were compared to their corrosion rates; no significant trend was found between deposit color and corrosion rates.

Specimens from Mixture 30 were typical of high caustic tests and were selected for detailed surface examination at all four temperatures after 4-months exposure. In the scanning electron microscope (SEM) examination, no significant difference, other than surface roughness, could be seen between the two alloys used. In the 40°C test, small crystals (Figure 3), estimated at less than about $1\text{ }\mu\text{m}$ long by about $0.1\text{ }\mu\text{m}$ wide were seen. At 60°C , particles were present which were about $1\text{ }\mu\text{m}$ in diameter but without any definite crystalline appearance. At 80°C , however, regular shaped crystals, platelets, appeared which were about $2\text{ }\mu\text{m}$ across. By 100°C , the platelets measured about $5\text{ }\mu\text{m}$ across (Figure 3).

Mixtures 57, 38, 33 and 16 at 100°C and 4-months exposure were also selected for SEM examination. For each sample the crystal structures were basically the same. The only exceptions noted were in Mixture 33 (high concentrations of most components) where large crystals, about $120\text{ }\mu\text{m}$ across, formed local groups; these were in addition to the other crystals. They were, however, uniformly distributed over the surface and no special features were observed that might have triggered their nucleation. The composition of these large crystals also was different. Whereas the smaller crystals were

FIGURE 2. U-Bends After Exposure to the 28 Waste Solutions at 100°C for 8 Months

40°C, Specimen # 000247, ~0 mpy;
Photo Ref. # P-5142, 3000X

100°C, Specimen # 100026, 0.41 mpy;
Photo Ref. # P-5136, 3000X

FIGURE 3. SEM Photographs of Specimens Exposed to Mixture 30
for 4 Months at 40 and 100°C

found to contain iron and a trace of manganese (a constituent of the alloys), the large crystals also contained large amounts of sodium. X-ray diffraction analysis showed them to be NaFeO_2 . Figure 4 compares SEM photographs of the crystals from Mixtures 16 and 33. Mixture 33 which had the largest crystals also had the largest corrosion rate for all the 4-month tests.

All U-bend samples were examined for cracks. In no case was there either cracking of the U-bends or dissolution of the nuts and bolts. Thus, under these various test conditions, and based on a very limited number of tests, stress corrosion cracking does not appear to be a significant mode for failure.

Corrosion in terms of overall metal loss as mils per year ($1 \text{ mpy} = 0.001 \text{ inch/yr}$) was calculated from the weight-loss coupons. The results are summarized in Table 4. Comparing the corrosion rates of A-516 and A-537, one notes that they behaved very similarly as expected; as noted in the statistical discussion, there appears to be a difference in the two but the importance has not been clarified. For all temperatures, exposure durations and waste solutions, with the exception of Solutions 33 and 36, the uniform corrosion rates were below 1 mpy and typically less than 0.2 mpy . This is a very low rate and within the acceptable 1 mpy called out in the EIS. Corrosion rates in Solutions 33 and 36 slowed down to below 1 mpy by the end of the 12-month exposure.

Several trends were apparent. Corrosion rates generally increased with temperature, and decreased with exposure duration if one just compares the 8- and 12-month data. The 4-month data had lower corrosion rates than the 8-month data. This was not expected. As the iron coupons corrode they generally form a protective oxide coating resulting in decreased corrosion rates with time. The most likely explanation for the low corrosion rates determined for the 4-month tests is that the low total weight change is confused within the weight changes during the annealing process.

The corrosion rate is strongly influenced by the OH^- concentration. Table 5 lists solutions by decreasing corrosion rate determined at the end of 2952 hours (about four months). As the OH^- concentration decreases so does the corrosion rate. When the actual values of OH^- , which were determined

Mixture 16, Specimen # 100017, ~0.21 mpy;
Photo Ref. # P-5135, 300X

Mixture 33, Specimen # 100027, 0.95 mpy;
Photo Ref. # P-5134, 300X

FIGURE 4. SEM Photographs of Specimens Exposed at 100°C
for 4 Months to Mixtures 16 and 33

TABLE 4. Corrosion Rate Expressed in mpy for A-537 and A-516 Coupons Exposed to the 28 Waste Compositions at Four Temperatures for 4, 8 and 12 Months

DSS MPY AVERAGES FOR

40.00 DEG.C

	4 MONTHS		8 MONTHS		12 MONTHS	
	A537 ^a	A516	A537	A516	A537	A516
SOL# 1	.040 ^b	.088	.134	.147	-.050	.027
SOL# 3	-.080	.096	.173	.182	-.046	.021
SOL# 5	.460	.121	.145	.141	.001	.030
SOL# 7	.040	.087	.149	.139	-.005	.033
SOL# 8	-.132	.066	.155	.147	.022	.030
SOL# 9	-.152	.058	.168	.173	-.030	.031
SOL# 12	-.124	.074	.184	.134	-.013	.025
SOL# 13	.053	.086	.150	.166	.006	.037
SOL# 15	-.142	.106	.155	.268	.010	.011
SOL# 16	-.000	.077	.151	.166	.036	*****
SOL# 20	-.053	.096	.211	.182	.062	*****
SOL# 28	-.072	.059	.183	.172	.149	*****
SOL# 29	.074	.124	.182	.189	.043	*****
SOL# 30	-.033	.065	.184	.192	.004	*****
SOL# 33	-.053	.057	.236	.197	.020	*****
SOL# 36	.848	.225	.166	.162	.295	*****
SOL# 38	.416	.076	.205	.186	.097	*****
SOL# 44	.018	.107	.211	.204	.091	*****
SOL# 48	.111	.078	.137	.151	.093	*****
SOL# 49	-.118	.066	.132	.152	.139	*****
SOL# 50	-.089	.073	.146	.108	.033	*****
SOL# 51	-.033	.061	.128	.200	.084	*****
SOL# 52	-.051	.077	.113	.140	-.043	*****
SOL# 53	.085	.075	.128	.132	.008	*****
SOL# 55	.212	.088	.159	.078	.045	*****
SOL# 57	-.071	.088	.135	.141	.037	*****
SOL# 58	-.119	.073	.109	.128	.007	*****
SOL# 59	.021	.111	.120	.155	.059	*****

- a) Negative values indicate total corrosion rates less than the corrosion occurring during annealing which is based on ten reference specimens.
- b) Data are presented to three or more places throughout this report. For practical use, all numbers should be rounded to the nearest tenth of a mpy. Replicate samples suggest a variation of at least 0.2 mpy.

TABLE 4. (Continued)

		USS MPY AVERAGES FOR		50.00 DEG.C			
		4 MONTHS		8 MONTHS		12 MONTHS	
		A537	A516	A537	A516	A537	A516
SOL#	1	.061	.104	.216	.191	.022	.049
SOL#	3	.008	.083	.196	.190	.026	.043
SOL#	5	.078	.067	.273	.201	.000	.049
SOL#	7	-.025	.079	.159	.002	-.026	.038
SOL#	8	.076	.142	.224	.189	-.011	.037
SOL#	9	.031	.097	.245	.219	.013	.039
SOL#	12	-.085	.096	.238	.191	.016	.038
SOL#	12	-.073	.086	.186	.159	.016	.039
SOL#	15	.053	.087	.243	.272	-.002	.040
SOL#	16	-.020	.084	.177	.150	.043	.034
SOL#	20	.219	.298	.297	.267	.083	.073
SOL#	28	.048	.110	.233	.186	.001	.031
SOL#	29	.078	.112	.203	.179	.010	.036
SOL#	30	.231	.139	.258	.262	.108	.061
SOL#	32	.486	.192	.294	.232	.248	.087
SOL#	36	.160	.175	.263	.185	.130	.070
SOL#	38	.394	.126	.270	.187	.137	.061
SOL#	44	.300	.137	.264	.278	.117	.058
SOL#	48	.111	.068	.138	.163	-.028	.024
SOL#	49	-.041	.078	.192	.179	-.047	.023
SOL#	50	-.084	.070	.189	.139	.036	.011
SOL#	51	.401	.072	.175	.151	.063	.026
SOL#	52	.023	.072	.126	.140	-.043	.027
SOL#	53	-.140	.074	.229	.132	.016	.035
SOL#	55	-.140	.092	.142	.150	-.047	.034
SOL#	57	-.074	.079	.161	.153	-.020	.040
SOL#	59	-.091	.083	.176	.155	.017	.029
SOL#	59	.139	.091	.179	.152	-.026	.034

TABLE 4. (Continued)

DSS MPY AVERAGES FOR 60.00 DEG.C

	4 MONTHS		8 MONTHS		12 MONTHS	
	A537	A516	A537	A516	A537	A516
SOL# 1	.109	.076	.170	.162	.092	.039
SOL# 3	.131	.125	.160	.156	-.020	.032
SOL# 5	-.002	.077	.209	.158	.063	.020
SOL# 7	-.032	.044	.174	.154	.078	.024
SOL# 8	-.047	.038	.136	.126	-.008	.037
SOL# 9	-.007	.061	.184	.151	.008	.026
SOL# 12	-.184	.041	.157	.240	-.039	.020
SOL# 13	.181	.126	.130	.115	.092	.029
SOL# 15	.093	.043	.182	.164	.003	.024
SOL# 16	-.150	.072	.162	.140	-.007	.029
SOL# 20	-.127	.126	.219	.171	.025	.040
SOL# 28	-.045	.052	.169	.285	.084	.029
SOL# 29	.298	.411	.178	.223	.041	.035
SOL# 30	-.106	.092	.221	.175	.092	.043
SOL# 33	-.060	.132	.195	.203	.136	.043
SOL# 36	1.175	1.328	.239	.170	.404	.977
SOL# 38	.107	.114	.210	.185	.092	.052
SOL# 44	-.007	.111	.202	.188	.003	.037
SOL# 48	-.191	.095	.146	.252	-.028	.034
SOL# 49	-.006	.064	.181	.145	-.023	.016
SOL# 50	-.105	.071	.137	.151	.028	.017
SOL# 51	-.177	.061	.169	.155	.004	.014
SOL# 52	-.101	.106	.139	.216	.059	.022
SOL# 53	-.162	.041	.149	.160	.031	.021
SOL# 55	-.170	.078	.158	.154	-.022	.022
SOL# 57	.114	.110	.166	.101	-.007	.023
SOL# 58	.016	.038	.124	.128	-.015	.014
SOL# 59	-.145	.061	.232	.200	.008	.029

TABLE 4. (Continued)

ISS MPY AVERAGES FOR 100.00 DEG.C

		4 MONTHS		8 MONTHS		12 MONTHS	
		A537	A516	A537	A516	A537	A516
SOL# 1	*****		.215	.273	.235	.085	.091
SOL# 3	*****		.249	.256	.272	.044	.077
SOL# 5	*****		.183	.292	.275	.133	.086
SOL# 7	*****		.120	.210	.219	.108	.039
SOL# 8	*****		.212	.253	.325	.167	.092
SOL# 9	*****		.216	.392	.341	.156	.141
SOL# 12	*****		.110	.355	.283	.135	.120
SOL# 13	*****		.207	.286	.282	.092	.135
SOL# 15	*****		.082	.245	.222	.047	.054
SOL# 16	*****		.174	.274	.262	.259	.128
SOL# 20	*****		.384	.353	.450	.137	.100
SOL# 28	*****		.238	.409	.323	.154	.132
SOL# 29	*****		.150	.314	.278	.117	.058
SOL# 30	*****		.491	.360	.348	.175	.151
SOL# 33	*****		1.074	.350	.359	.489	.314
SOL# 36	*****		.489	.369	.305	.254	.123
SOL# 38	*****		.656	.482	.542	.676	.216
SOL# 44	*****		.414	.294	.298	.413	.567
SOL# 48	*****		.039	.214	.194	.019	.032
SOL# 49	*****		.001	.184	.182	.030	.036
SOL# 50	*****		-.009	.177	.146	-.031	.017
SOL# 51	*****		-.007	.187	.187	.099	.032
SOL# 52	*****		-.026	.164	.169	.027	.005
SOL# 53	*****		-.012	.187	.166	.023	.039
SOL# 55	*****		-.008	.184	.208	.018	.021
SOL# 57	*****		.019	.189	.177	.062	.067
SOL# 58	*****		.103	.211	.208	.054	.027
SOL# 59	*****		.076	.201	.178	.035	.029

*****No coupons exposed.

analytically, are examined, a secondary effect on the ranking of the solutions' corrosion potential is observed. Within each OH^- range (10, 7, 4 or 0.5M) the influence of other components can be seen. These effects may be seen in more detail in Figures 5 to 9 which are described later.

Seven solutions produced foaming during the tests. These are marked with an asterisk in Table 5. Solutions where foaming occurred are in the higher two-thirds of the corrosion rates. In all cases the solutions that foamed contained Complexant with 4.0M or greater OH^- .

Several solutions were analyzed before and at the completion of the tests to determine if the waste solutions varied with time. Seven solutions, representing most waste composition ranges, from the 100°C, 8-month tests, were chosen for this comparison. The analytical results are reported in Appendix A. Generally, Cl^- , SO_4^{2-} , NO_2^- and NO_3^- remained the same or decreased slightly. The opposite trend was observed for OH^- . It remained about the same or increased. Since the corrosion rates were determined to have a direct correlation with the OH^- concentration, the solutions where OH^- increased with exposure hours (example #38) might present a long term corrosion problem. The cause of the concentration increase, if real, has not been determined.

Conclusions for the Laboratory Experiments

Three potential types of corrosion were monitored using U-bends and weight-loss coupons: uniform corrosion, stress corrosion cracking, and pitting. All U-bend samples were examined for cracks and in all cases there were none observed. Under microscopic examination no potential pitting problems were discovered. For all combinations of solution chemistries and temperatures up to and including 100°C (212°F) during 12-months exposure, the uniform corrosion rates were below the 1 mpy called out as an upper limit in the EIS. Preliminary results of high temperature tests now in progress suggest this is not the case at temperatures approaching the design value of 350°F (177°C); this is consistent with reported data for high temperature caustic solutions.⁽³⁾

The corrosion behavior of the two carbon steels was very well behaved despite the "exotic" solutions. In the long term, the uniform corrosion rates decreased with exposure time. These corrosion rate data are used in

TABLE 5. Correlation of Corrosion Rates
to OH⁻ Concentration

Solution Mixture Number	mpy (X10 ³) at 100°C			Ideal OH ⁻ (M)	OH ⁻ (wt%) Analyzed at End of		Solution that Foamed
	2952 hrs.	6000 hrs.	8760 hrs.		4 months	8 months	
33	1074	353	373	10	13.11		*
38	656	502	369	10	10.99	11.36	
30	491	356	159	10	11.64	10.5	
36	490	348	166	10	11.81		*
44	414	295	516	10	10.05	10.2	
20	384	385	112	7	6.70	7.27	
28	239	380	139	7	9.40		*
1	216	260	89	4	6.04	6.9	
3	249	262	66	4	4.09		
9	216	376	146	4	3.94	5.25	
8	212	277	117	4	5.15		
13	207	285	121	4	5.66		*
16	174	270	172	4	3.85	5.1	
5	184	287	102	4	4.51		
29	151	302	78	7	10.67		*
7	120	213	62	4	6.34		*
12	110	331	125	4	4.33		
58	103	210	36	0.5	--		
15	82	237	52	4	7.84		*
59	76	194	31	0.5	0.72		
48	39	207	28	0.5	0.256		
57	20	185	65	0.5	0.097		
51	7	187	55	0.5	0.526	0.804	
49	5	183	34	0.5	0.280		
50	-9	167	1	0.5	--		
55	-10	192	20	0.5	0.217		
53	-13	180	34	0.5	1.09		
52	-27	165	6	0.5	--		

the next section to generate prediction equations for interpolating, within the bounds of existing chemistries and temperatures, the expected corrosion behavior of the storage tanks to new combinations of storage conditions.

STATISTICAL ANALYSIS OF THE DOUBLE SHELL SLURRY DATA

For all phases of the statistical analysis and model fitting described below, the data from the experiments at 40° and 60° with 4-month exposure time were omitted. This is because an analysis of the 4-month data showed that the observed corrosion rates at 40° and 60°, which were very small, had negligible correlation with the 28 compositions. Thus, when these data are included in the fitting of a prediction model for each alloy, the resulting fitted models do a poor job of predicting corrosion rates over the experimental region. However, with these data omitted, prediction models can be fitted to the remaining data which are quite useful for predicting corrosion rates over the remaining portion of the experimental region. It should also be recalled that no A-537 coupons were present at the 4-month, 100° experiments. Thus, the 4-month experimental data included in the statistical analysis are from the 80° experiments for A-537 and both the 80° and 100° experiments for A-516. The 8-month and 12-month experimental data for A-537 and A-516 are from experiments which include all 28 compositions at each of the four temperatures.

Preliminary Analysis of Variance

The experimental data for alloy A-537 and alloy A-516 were analyzed separately using the analysis of variance. For each alloy, this partitioned the total variability into components due to:

1. Exposure Time: For this preliminary analysis of variance the exposure times in hours were collapsed into three major groups, designated 4 months, 8 months, and 12 months. The effect of exposure time is then characterized by the variability due to differences among the three average corrosion rates corresponding to the three exposure time groups (averaging over the 28 compositions and four temperatures).
2. Composition: Differences among the 28 average corrosion rates corresponding to the 28 compositions (averaging over the three exposure times and the four temperatures).

3. Temperature: Differences among the four average corrosion rates corresponding to the four temperatures (averaging over the three exposure times and the 28 compositions).
4. Interaction of Exposure Time With Composition: If significant, the interpretation is that the effect of exposure time on corrosion rate is not the same for all compositions (or vice versa).
5. Interaction of Exposure Time With Temperature: If significant, the interpretation is that the effect of exposure time on corrosion rate is not the same for all temperatures (or vice versa).
6. Interaction of Composition With Temperature: If significant, the interpretation is that the effect of temperature on corrosion rate is not the same for all compositions (or vice versa).
7. Three-Way Interaction of Exposure Time, Composition, and Temperature: If significant, one interpretation is that the interaction of any two of the factors is not the same at all levels of the remaining factor. Another interpretation is that the effect of one of the three factors on corrosion rate is not the same at all combinations of the levels of the remaining two factors.
8. Experimental Error: In some exposure time/temperature combinations, two replicate coupons of alloy A-537 and one coupon of alloy A-516 were present at each composition. Similarly, in the remaining exposure time/temperature combinations, one coupon of alloy A-537 and two replicate coupons of alloy A-516 were present at each composition. For each alloy, the data from the replicate coupons were used to compute an estimate of the experimental error variance due to nonreproducibility of results when an experiment is repeated under prescribed similar conditions.

For each alloy, the estimate of experimental error variance (described in Item 8 above) was used to test whether any of the factors or interactions (specified in Items 1 through 7 above) contribute significantly to the overall variability of the observed corrosion rates. The results of the statistical tests are summarized in Tables 6 and 7, where the contribution of all factors and interactions are very highly statistically significant. This is a strong indication that it is possible to fit a mathematical model to the experimental data for each alloy which expresses corrosion rate as a function of exposure time, composition and temperature, and that if the form of the model is properly chosen, the fitted model will account for a significant portion of the total variability in the observed corrosion rates. That is, the fitted model for each alloy may be highly correlated with the observed penetration rates and could be useful for predicting corrosion rates over the experimental region.

Fitting a Prediction Model

The experiment was designed so that the data would accommodate fitting a 300 parameter polynomial model for each alloy which expresses corrosion rate as a function of exposure time in hours, temperature in °C, and the levels of the nine anions in a chemical solution. The 300 parameter model includes an intercept and 299 terms which are the 299 predictors defined in Table 8, each multiplied by a coefficient. The intercept and the 299 coefficients are unknown parameters in the model. It is of interest to estimate them by fitting the model to the experimental data, and then to perform statistical tests on the estimates to decide which of the unknown coefficients are not zero. That is, the statistical tests indicate which of the 300 terms are necessary in the model. However, this approach to the problem is not possible. As previously noted, part of the 4-month data are not utilized in the model fitting process. Thus, it is not possible to estimate all 300 of the unknown parameters. Deciding in advance which of the parameters to estimate and which to leave out of the model is an arbitrary decision, unless some specific assumptions can be made about which of the parameters are likely to be zero. However, there is no a priori basis for making such assumptions.

TABLE 6. Analysis of Variance Results for Alloy A-537

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>	<u>F-Ratio*</u>
Mean	1	4.375		
Time	2	79.791	35.896	722.9
Temp	3	25.733	8.578	184.7
Composition	27	53.213	1.971	42.4
Time X Temp	3	1.877	0.626	13.5
Time X Comp	53	26.788	0.505	10.9
Temp X Comp	81	27.222	0.336	7.2
Time X Temp X Comp	78	13.500	0.173	3.7
Experimental Error	108	5.016	0.046	

TABLE 7. Analysis of Variance Results for Alloy A-516

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>	<u>F-Ratio*</u>
Mean	1	0.041		
Time	2	67.489	33.744	162.6
Temp	3	34.342	11.447	55.1
Composition	27	86.813	3.215	15.5
Time X Temp	4	6.190	1.547	7.5
Time X Comp	53	53.465	1.009	4.9
Temp X Comp	81	67.385	0.832	4.0
Time X Temp X Comp	105	61.164	0.583	2.8
Experimental Error	176	36.516	0.207	

*Each F-ratio is the Mean Square for the factor of interest divided by the Mean Square for Experimental Error. Each F-ratio is compared with tabulated quantities of Snedecor's F distribution with numerator and denominator degrees of freedom equal to the degrees of freedom for the factor of interest and the Experimental Error, respectively. All of the above ratios are statistically significant, and there is less than a 0.0005 probability of being wrong in concluding that any of the factors affects penetration.

TABLE 8. Definition of the 299 Candidate Predictor Variables

<u>Column 1</u>	<u>Column 2</u>
OH	Temperature
A1	
NO ₃	(Temp) ²
NO ₂	
CPX	(Temp) ³
CO ₃	
PO ₄	Hours
SO ₄	
CRT	(Hours) ²
(OH) ²	
(A1) ²	Temp X Hours
(NO ₃) ²	
(NO ₂) ²	(Temp) ² x Hours
(CPX) ²	
OH x A1	(Temp) ³ x Hours
OH x NO ₃	
OH x NO ₂	Temp x (Hours) ²
OH x CPX	
A1 x NO ₃	(Temp) ² x (Hours) ²
A1 x NO ₂	
A1 x CPX	(Temp) ³ x (Hours) ²
NO ₃ x NO ₂	
NO ₃ x CPX	
NO ₂ x CPX	

The remaining 264 candidate predictors are generated by taking the products of each of the 24 predictors in Column 1 with each of the 11 predictors in Column 2.

The method used to select an adequate subset of predictors to include in each model is stepwise regression analysis. For each alloy, a model is built by forward stepping, which adds predictors to the model one-at-a-time by selecting the predictor (from those not in the model) at each step which contributes the most to the prediction ability of the model. Since some of the predictors are correlated, adding a predictor to the model can decrease the significance of other predictors in the model. Since some of the predictors are correlated, adding a predictor to the model can decrease the significance of other predictors in the model. The so called "F-Swap" procedure is used to help remedy this by removing predictors which are in the model but have become nonsignificant. The F-Swap procedure also allows a predictor which is not in the model to be exchanged for a predictor which is in the model but has become nonsignificant, if such an exchange improves the prediction ability of the model. Forward stepping terminates when no significant improvement in prediction ability can be achieved by adding more predictors to the model.

For the A-537 data, the forward stepping terminated with 55 predictors in the model and $R^2 = 0.89$. (R^2 is the percent of the variability in the observed corrosion rates accounted for by the fitted model.) The 55 selected predictors are listed in Table 9.

For the A-516 data, the forward stepping terminated with 74 predictors in the model and $R^2 = 0.87$. The 74 selected predictors are listed in Table 10.

Interpreting the Fitted Models

Most of the boundaries of the experimental region are determined by limitations on the "nominal levels" of the nine 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 experiment, and the 28 chemical solutions which are included in the experiment are specified in terms of the "nominal levels" of the anions. In addition, as noted in the earlier discussion, some discrepancy exists between nominal and

TABLE 9. Selected Predictors for the A-537 Model

T	$H \times OH \times NO_2$	$T \times H^2 \times OH \times Al$
OH	$H \times OH \times CPX$	$T \times H^2 \times OH \times NO_2$
CPX^2	$H \times Al \times NO_3$	$T \times H^2 \times OH \times CPX$
$T \times OH$	H^2	$T \times H^2 \times Al \times NO_2$
$T \times (NO_3)^2$	$H^2 \times CPX$	$T \times H^2 \times Al \times CPX$
T^2	$H^2 \times PO_4$	$T^2 \times H$
$T^2 \times OH$	$H^2 \times OH^2$	$T^2 \times H \times NO_3 \times CPX$
$T^2 \times CPX$	$H^2 \times NO_3 \times NO_2$	$T^2 \times H^2 \times OH^2$
$T^2 \times OH^2$	$T \times H \times OH$	$T^3 \times H$
$T^2 \times Al^2$	$T \times H \times (NO_2)^2$	$T^3 \times H \times OH$
$T^2 \times OH \times Al$	$T \times H \times OH \times NO_3$	$T^3 \times H \times OH \times NO_3$
$T^2 \times OH \times NO_3$	$T \times H \times Al \times CPX$	$T^3 \times H \times OH \times NO_2$
$T^3 \times NO_3$	$T \times H \times NO_2 \times CPX$	$T^3 \times H \times Al \times NO_3$
$T^3 \times SO_4$	$T \times H^2 \times NO_2$	$T^3 \times H \times Al \times NO_2$
$T^3 \times CRT$	$T \times H^2 \times PO_4$	$T^3 \times H \times Al \times CPX$
$T^3 \times Al^2$	$T \times H^2 \times SO_4$	$T^3 \times H^2 \times OH$
$T^3 \times (NO_3)^2$	$T \times H^2 \times (NO_3)^2$	$T^3 \times H^2 \times CO_3$
$H \times PO_4$	$T \times H^2 \times (NO_2)^2$	$T^3 \times H^2 \times OH \times NO_2$
$H \times OH \times NO_3$		
OH - hydroxide	CPX - complexant	T - temperature
NO_3 - nitrate	Al - aluminate	SO_4 - sulfate
CRT - citrate	PO_4 - phosphate	NO_2 - nitrite
H - hours	CO_3 - carbonate	

TABLE 10. Selected Predictors for the A-516 Model

H	$H \times A1 \times NO_2$	$T \times H^2 \times NO_3 \times CPX$
OH	$H \times NO_3 \times CPX$	$T \times H^2 \times NO_2 \times CPX$
CPX^2	H^2	$T^2 \times H \times OH$
$OH \times A1$	$H^2 \times A1$	$T^2 \times H \times CO_3$
$T \times NO_3$	$H^2 \times NO_2$	$T^2 \times H \times SO_4$
$T \times CO_3$	$H^2 \times CPX$	$T^2 \times H \times CRT$
T^2	$H^2 \times A1^2$	$T^2 \times H \times (NO_3)^2$
$T^2 \times NO_3$	$H^2 \times (NO_3)^2$	$T^2 \times H \times CPX^2$
$T^2 \times CO_3$	$H^2 \times CPX^2$	$T^2 \times H \times OH \times A1$
$T^2 \times OH \times A1$	$H^2 \times OH \times CPX$	$T^2 \times H \times OH \times NO_3$
$T^2 \times OH \times NO_2$	$H^2 \times A1 \times CPX$	$T^2 \times H \times OH \times NO_2$
$T^2 \times A1 \times NO_3$	$H^2 \times NO_3 \times NO_2$	$T^2 \times H \times A1 \times NO_3$
$T^2 \times A1 \times NO_2$	$H^2 \times NO_3 \times CPX$	$T^2 \times H^2 \times (NO_3)^2$
T^3	$H^2 \times NO_2 \times CPX$	$T^3 \times H \times OH$
$T^3 \times CPX$	$T \times H \times CO_3$	$T^3 \times H \times OH^2$
$T^3 \times OH \times NO_3$	$T \times H \times OH^2$	$T^3 \times H \times OH \times NO_3$
$T^3 \times OH \times NO_2$	$T \times H \times OH \times NO_3$	$T^3 \times H \times OH \times NO_2$
$T^3 \times NO_3 \times CPX$	$T \times H \times OH \times NO_2$	$T^3 \times H \times OH \times CPX$
$T^3 \times NO_2 \times CPX$	$T \times H \times OH \times CPX$	$T^3 \times H \times A1 \times NO_2$
$H \times CPX$	$T \times H \times A1 \times NO_3$	$T^3 \times H \times NO_2 \times CPX$
$H \times OH^2$	$T \times H \times A1 \times NO_2$	$T^3 \times H^2 \times OH$
$H \times (NO_2)^2$	$T \times H^2 \times OH$	$T^3 \times H^2 \times CRT$
$H \times OH \times NO_3$	$T \times H^2 \times NO_3$	$T^3 \times H^2 \times CPX^2$
$H \times OH \times NO_2$	$T \times H^2 \times NO_2$	$T^3 \times H^2 \times OH \times NO_3$
$H \times OH \times CPX$	$T \times H^2 \times A1 \times CPX$	
OH - hydroxide	CPX - complexant	T - temperature
NO ₃ - nitrate	A1 - aluminate	SO ₄ - sulfate
CRT - citrate	PO ₄ - phosphate	NO ₂ - nitrite
H - hours	CO ₃ - carbonate	

actual concentration. However, correlations among the 24 predictors listed in Column 1 of Table 8 are unavoidable. Taking cross-products of these predictors with the 11 predictors listed in Column 2 of Table 8 results in high correlations among some of the 299 possible predictors. Because of these correlations, straightforward interpretation of the terms in the fitted models is not possible.

To illustrate why this is so, consider a simple situation where there are only two predictors, say Predictor 1 and Predictor 2, but in the experimental data these predictors are highly correlated with each other. Now suppose it is of interest to fit a model to predict a response variable, such as corrosion rate, at given levels of the two predictors. Since the two predictors are highly correlated, it may only be necessary to include one of them in a fitted model to make predictions over the experimental region. However, it may be impossible to determine from the experimental data which of the predictors is having an effect on the response variable, or whether both predictors effect the response variable. Thus, if stepwise regression is implemented, it could terminate with only one of the predictors in the model. Suppose that, unknown to the experimenter, Predictor 2 has a strong effect on the response and Predictor 1 does not, but Predictor 1 is selected to be the only predictor in the model. In this case, the fitted model might give very good predictions of the response over the experimental region. However, the experimenter would be wrong to conclude that Predictor 1 has a strong effect on the response and Predictor 2 has no effect, just because Predictor 1 was selected to be in the fitted model.

This principle is amplified and much more complicated in selecting predictors from the 299 defined in Table 8. Thus, although fitted models which include the predictors listed in Tables 9 and 10 provide good predictions of penetration over the experimental region, an attempt to interpret the individual terms in the fitted models could be very misleading. The primary use of the fitted models should be to predict penetration for conditions inside the experimental region.

Using the Fitted Models to Predict Corrosion Rate

With R^2 values of 0.89 and 0.87, respectively, the A-537 and A-516 fitted models should be quite useful for predicting corrosion rates over various chemical solutions at given exposure times and temperatures within the ranges used in the experiment. Thus, the fitted models are presented and their implementation is illustrated by an example. Note, however, that the purpose of this section is to present only the mechanics of using the fitted models. The next section describes a portable, user-friendly computer program which utilizes the fitted models to predict corrosion rate. It is strongly recommended that this computer program be used rather than trying to implement the fitted models by hand.

To achieve maximum computing precision in the regression analysis, the corrosion rates, exposure times, temperatures, and chemical levels were standardized so that they are centered near zero and are in comparable units. Thus, the fitted models are in terms of the standardized variables listed in Table 11.

TABLE 11. Standardized Variables

Y	$= (\text{Corrosion Rate} - 0.134)/0.144$
X_1	$= (\text{Temp} - 70)/20$
X_2	$= (\text{Hours} - 5856)/2904$
X_3	$= (\text{OH} - 4)/3$
X_4	$= (\text{Al} - 0.9)$
X_5	$= (\text{NO}_3 - 3)/2$
X_6	$= (\text{NO}_2 - 3)/2.6$
X_7	$= (\text{CPX} - 0.3)/0.3$
X_8	$= (\text{CO}_3 - 0.15)/0.06$
X_9	$= (\text{PO}_4 - 0.2)/0.3$
X_{10}	$= (\text{SO}_4 - 0.05)/0.03$
X_{11}	$= (\text{CRT} - 0.2)/0.2$

To apply the fitted models, the standardized variables X_1 through X_{11} are computed for given levels of hours, temperature, etc., as specified in Table 11. These standardized values are then substituted into the equations given in Tables 12 and 13, to predict Y which is the standardized corrosion rate value. The predicted corrosion rate value in mpy is then obtained by applying the formula:

$$\text{Corrosion rate} = 0.144 Y + 0.134$$

As an example, consider predicting corrosion rates for both alloys with chemical Solution 30 at 100°C, exposed for 6,000 hours. Computation of the standardized variables X_1 through X_{11} is illustrated in Table 14. Substituting these standardized values into the prediction equations in Tables 12 and 13 gives predicted standardized corrosion rate:

$$\begin{aligned} \text{A-537: } Y &= 1.4974 \\ \text{and A-516: } Y &= 1.5598. \end{aligned}$$

The predicted corrosion rates are then computed as:

$$\begin{aligned} \text{A-537: cor.} &= 0.144(1.4974) + 0.134 = 0.350 \text{ mpy} \\ \text{and A-516: cor.} &= 0.144(1.5598) + 0.134 = 0.359 \text{ mpy} \end{aligned}$$

The observed mean corrosion rates are 0.360 mpy and 0.348 mpy for A-537 and A-516, respectively.

TABLE 12. Fitted Prediction Model for A-537

$$\begin{aligned}
Y = & 0.3052 + 0.4030 x_1 + 0.1681 x_3 + 0.0539 x_7^2 \\
& + 0.1162 x_1 x_3 - 0.1289 x_1 x_5^2 + 0.0736 x_1^2 + 0.1095 x_1^2 x_3 \\
& - 0.0351 x_1^2 x_7 - 0.0115 x_1^2 x_3^2 + 0.0149 x_1^2 x_4^2 \\
& + 0.0676 x_1^2 x_3 x_4 + 0.0253 x_1^2 x_3 x_5 + 0.0277 x_1^3 x_5 \\
& + 0.0318 x_1^3 x_{10} + 0.0155 x_1^3 x_{11} + 0.0812 x_1^3 x_4^2 \\
& - 0.0588 x_1^3 x_5^2 + 0.1153 x_2 x_9 + 0.1168 x_2 x_3 x_5 \\
& - 0.1013 x_2 x_3 x_6 + 0.0828 x_2 x_3 x_7 - 0.0625 x_2 x_4 x_5 \\
& - 1.3720 x_2^2 + 0.0871 x_2^2 x_7 - 0.1463 x_2^2 x_9 \\
& + 0.1381 x_2^2 x_3^2 - 0.1090 x_2^2 x_5 x_6 - 0.4430 x_1 x_2 x_3 \\
& - 0.5058 x_1 x_2 x_6^2 - 0.1821 x_1 x_2 x_3 x_5 \\
& - 0.0556 x_1 x_2 x_4 x_7 + 0.0700 x_1 x_2 x_6 x_7 \\
& + 0.1710 x_1 x_2^2 x_6 - 0.0835 x_1 x_2^2 x_9 + 0.0386 x_1 x_2^2 x_{10} \\
& - 0.1574 x_1 x_2^2 x_5^2 + 0.4034 x_1 x_2^2 x_6^2 - 0.5083 x_1 x_2^2 x_3 x_4 \\
& + 0.2914 x_1 x_2^2 x_3 x_6 - 0.0499 x_1 x_2^2 x_3 x_7 - 0.4630 x_1 x_2^2 x_4 x_6 \\
& + 0.3165 x_1 x_2^2 x_4 x_7 + 0.0573 x_1^2 x_2 - 0.0312 x_1^2 x_2 x_5 x_7 \\
& + 0.0622 x_1^2 x_2^2 x_3^2 + 0.1272 x_1^3 x_2 + 0.0866 x_1^3 x_2 x_3 \\
& + 0.0611 x_1^3 x_2 x_3 x_5 - 0.0903 x_1^3 x_2 x_3 x_6 - 0.0290 x_1^3 x_2 x_4 x_5 \\
& + 0.1250 x_1^3 x_2 x_4 x_6 - 0.0768 x_1^3 x_2 x_4 x_7 - 0.1057 x_1^3 x_2^2 x_3 \\
& - 0.1368 x_1^3 x_2^2 x_8 + 0.0429 x_1^3 x_2^2 x_3 x_6
\end{aligned}$$

TABLE 13. Fitted Prediction Model for A-516

$$\begin{aligned}
Y = & 0.1425 - 0.1810 X_2 + 0.2043 X_3 + 0.0507 X_7^2 \\
& +0.1372 X_3 X_4 - 0.1375 X_1 X_5 - 0.1314 X_1 X_8 + 0.2482 X_1^2 - 0.0271 X_1^2 X_5 \\
& -0.0468 X_1^2 X_8 - 0.1464 X_1^2 X_3 X_4 + 0.0298 X_1^2 X_3 X_6 - 0.0620 X_1^2 X_4 X_5 \\
& +0.0606 X_1^2 X_4 X_6 + 0.0851 X_1^3 + 0.0249 X_1^3 X_7 + 0.0287 X_1^3 X_3 X_5 \\
& -0.0144 X_1^3 X_3 X_6 + 0.0229 X_1^3 X_5 X_7 - 0.0176 X_1^3 X_6 X_7 + 0.0826 X_2 X_7 \\
& +0.1420 X_2 X_3^2 - 0.0907 X_2 X_6^2 + 0.2673 X_2 X_3 X_5 - 0.2876 X_2 X_3 X_6 \\
& +0.2049 X_2 X_3 X_7 + 0.2051 X_2 X_4 X_6 + 0.0771 X_2 X_5 X_7 - 0.7910 X_2^2 \\
& -0.2865 X_2^2 X_4 - 0.1722 X_2^2 X_6 + 0.0862 X_2^2 X_7 + 0.0537 X_2^2 X_4^2 \\
& +0.2468 X_2^2 X_5^2 - 0.2131 X_2^2 X_7^2 + 0.0631 X_2^2 X_3 X_7 - 0.2962 X_2^2 X_4 X_7 \\
& -0.2140 X_2^2 X_5 X_6 + 0.0845 X_2^2 X_5 X_7 - 0.2073 X_2^2 X_6 X_7 \\
& -0.0365 X_1 X_2 X_8 - 0.2167 X_1 X_2 X_3^2 - 0.4460 X_1 X_2 X_3 X_5 \\
& +0.4690 X_1 X_2 X_3 X_6 - 0.2930 X_1 X_2 X_3 X_7 + 0.0432 X_1 X_2 X_4 X_5 \\
& -0.4590 X_1 X_2 X_4 X_6 - 0.3390 X_1 X_2^2 X_3 - 0.1190 X_1 X_2^2 X_5 + 0.1172 X_1 X_2^2 X_6 \\
& +0.1451 X_1 X_2^2 X_4 X_7 - 0.0850 X_1 X_2^2 X_5 X_7 + 0.2296 X_1 X_2^2 X_6 X_7 \\
& -0.4392 X_1^2 X_2 X_3 - 0.0867 X_1^2 X_2 X_8 + 0.0399 X_1^2 X_2 X_{10} + 0.0596 X_1^2 X_2 X_{11} \\
& -0.0676 X_1^2 X_2 X_5^2 - 0.1066 X_1^2 X_2 X_7^2 + 0.0582 X_1^2 X_2 X_3 X_4 - 0.1232 X_1^2 X_2 X_3 X_5 \\
& +0.0751 X_1^2 X_2 X_3 X_6 - 0.0755 X_1^2 X_2 X_4 X_5 - 0.1228 X_1^2 X_2^2 X_5^2 + 0.1318 X_1^3 X_2 X_3 \\
& +0.0918 X_1^3 X_2 X_3^2 + 0.1727 X_1^3 X_2 X_3 X_5 - 0.1465 X_1^3 X_2 X_3 X_6 + 0.0551 X_1^3 X_2 X_3 X_7 \\
& +0.1061 X_1^3 X_2 X_4 X_6 - 0.0437 X_1^3 X_2 X_6 X_7 + 0.3900 X_1^3 X_2^2 X_3 \\
& -0.0224 X_1^3 X_2^2 X_{11} + 0.1160 X_1^3 X_2^2 X_7^2 - 0.0906 X_1^3 X_2^2 X_3 X_5
\end{aligned}$$

TABLE 14. Standardized Example Values

x_1	$= (100-70)/20 = 1.5$
x_2	$= (6000-5856)/2904 = 0.0496$
x_3	$= (10-4)/3 = 2.0$
x_4	$= (0.1-0.9) = -0.8$
x_5	$= (1-3)/2 = -1.0$
x_6	$= (0.2-3)/2.6 = -1.0769$
x_7	$= (0-0.03)/0.03 = -1.0$
x_8	$= (0.085-0.15)/0.06 = -1.0833$
x_9	$= (0.20-0.2)/0.3 = 0.0$
x_{10}	$= (0.05-0.05)/0.03 = 0.0$
x_{11}	$= (0.2-0.2)/0.2 = 0.0$

Appendices A and B include tables of observed means and predicted corrosion (from the fitted models) for all conditions present in the experimental data.

Computerized Models for Predicting Corrosion Rate

The fitted models displayed in Tables 12 and 13 have been incorporated into a computer program which predicts corrosion rate. The program is interactive and it prompts the user for alloy, exposure time, temperature, and a chemical composition. It then utilizes the fitted models to predict the corrosion rate, in mpy, which would be expected to occur under the specified conditions. The program also computes an estimate of the standard deviation of the predicted value which is then used to compute upper 90%, 95%, and 99% confidence bounds on the expected value. The program also outputs a heuristic criterion for deciding when a set of specified conditions is outside of the region of the experiment which makes the resulting predicted corrosion rate invalid.

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. Instead, user information with examples are given to illustrate implementation of the program.

Once the program is compiled on a computer, it is executed by giving the command "RUN DSSCORR" and then responding to the prompts for input. To illustrate this, Table 15 displays the prompts and the response to each of the prompts for a particular set of conditions. The first prompt asks whether the output is to be stored in a file. If the response is yes (Y), the next prompt is for the file name. In this example the output data is not to be stored in a file and the response is no (N). The next twelve prompts ask for alloy, exposure time, temperature, and the composition of interest expressed in moles/liter.

After completing the sequence of prompts and responses shown in Table 15, the program printed the results displayed in Table 16. The program first prints the specified set of conditions for which a prediction is to be made. The predicted corrosion rate in mpy and the standard deviation of the predicted value are then printed. This is followed by a comparison of the standard deviation of the predicted value with the square root of the residual mean square from the fitted model. The experiment was designed such that the standard deviation of the predicted value should be less than or equal to the square root of the residual mean square. Thus, if the standard deviation of a 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, in which case the prediction may be invalid. In this example, the ratio is 19%, which is well below 100%, and the prediction is probably valid. If the ratio exceeds 100%, a warning is printed indicating that the prediction may be invalid.

Finally, 90%, 95%, and 99% upper confidence bounds are printed for the expected corrosion rate at the specified set of conditions. For example, in Table 16 the upper 90% confidence bound is 0.20 mpy. The interpretation is that, based on the fitted model, the residual mean square of the fitted model, and the specified set of experimental conditions, the probability is 90% that the true corrosion rate will be less than 0.20 mpy. Another way of stating this is that if a large number of experiments were run under the specified conditions, we would expect 90% of the observed corrosion rates to be less than 0.20 mpy.

TABLE 15. Prompts & Responses for Example Run
of Prediction Program DSSCORR

Run DSSCORR

Do you want output to a file? Y/N

N

Input which alloy are you using. (516 or 537)

516

Input exposure time in hours.

6000

Input temperature in degrees centegrade.

80

Now inputing chemical contents in moles/liter.

Input Hydroxide

4

Input Aluminate

2.6

Input Nitrate

1

Input Nitrite

6

Input Complexant

.47

Input Carbonate

.15

Input Phosphate

1

Input Sulphate

.16

Input Citrate

.2

TABLE 16. Program Results From Example Conditions
Specified in Table 15

For alloy 516

With exposure time = 6,000 hours

Temperature = 80 degrees centigrade

Chemical contents in mole/liter

Hydroxide = 4.0000

Aluminate = 2.6000

Nitrate = 1.0000

Nitrite = 6.0000

Complexant = 0.4700

Carbonate = 0.1500

Phosphate = 1.0000

Sulphate = 0.1600

Citrate = 0.2000

The predicted corrosion rate is

0.18 mpy

The standard deviation of the predicted corrosion
rate is

0.01 mpy

The standard deviation of the predicted value is
19% of the experimental error standard deviation

Under these conditions, there is a 90% probability
that the actual corrosion rate will be less than

0.20 mpy

Under these conditions, there is a 95% probability
that the actual corrosion rate will be less than

0.20 mpy

Under these conditions, there is a 99% probability
that the actual corrosion rate will be less than

0.21 mpy

A word of caution is in order about the use of this prediction program to study the effects of one or more of the elements in the chemical compositions over the experimental region. It is tempting to hold the levels of all but one or two elements constant and then make predictions as the levels of the one or two elements are allowed to vary. A misapplication of the resulting predictions would be to plot them over the range of the elements which were varied, and then use these plots to make exploratory inferences about the effects of the individual elements on corrosion rate. The reason this is a misapplication of the fitted models is discussed in a previous section titled, "Interpreting the Fitted Model." However, if certain effects were hypothesized before the experiment based on scientific theory, then the type of plots discussed above may be useful for confirming such theory.

Conclusions

Graphical examples of the predictor equation for A-537, Table 12, are shown in Figures 5-9. The plotting code has not been developed to the point where points outside the test matrix (and, therefore, inappropriate for calculation) are omitted. In cases where the corrosion rate is zero or negative (discussed earlier), the plotting routine does not plot a line-implying zero but with a less cluttered graph.

The figures are useful for visualizing the data but, as has been stated throughout the report, it is only possible to use the predictor equations to predict corrosion rates, not to identify the components with a significant effect.

Corrosion rates generally increase with temperature and OH^- concentration, Figure 5. It is interesting to note that while this is observed, there is little effect until the higher temperatures and hydroxide concentrations are reached.

The variation in organic complexant concentration has little effect on the predicted corrosion rate--there may be a slight tendency for the predicted corrosion rate to decrease with increasing complexant concentrations for A-537, Figure 6. This observation is reasonable in the presence of the stronger complexant hydroxide.

NO_2^- , Figure 7, generally appears to increase the corrosion rate of A537 with increasing concentration. The NO_3^- , on the other hand, Figure 8 appears to have a strong influence on the corrosion rate with a maximum (in corrosion rate) occurring in the vicinity of the normal DSS NO_3^- concentration; the maximum corrosion rate is still far less than the design limit.

The $\text{Al}(\text{AlO}_2^-)$ behavior, Figure 9, is probably strongly influenced by other components because of their effects on its solubility. There does appear to be minimal effect in the central region of interest. The high corrosion rates at high AlO_2^- and low OH^- concentrations occur in a region physically impossible to reach; at this time the plotting code is not set up to delete physically impossible regions.

Overall, the corrosion rates for the conditions tested after one year are well below the design limit of 1 mpy. Only two components, OH^- and AlO_2^- , even indicate a tendency toward undesirable trends--that is, accelerating corrosion rates with changing concentration. These trends are limited by constraints of the test ranges and solubility.

No indication of pitting or stress corrosion cracking was observed. Based on previous work by Ondrejcin,⁽⁴⁾ this was not unexpected.

Consequently, no operating restrictions, within the test conditions, are needed for the waste tanks. Subsequent reports will examine other compositions, more dilute solutions, and higher temperatures.

FIGURE 5. Corrosion Rate as a Function of Temperature and Hydroxide

FIGURE 6. Corrosion Rate as a Function of Chelating Agent and Hydroxide

FIGURE 7. Corrosion Rate as a Function of Nitrite and Hydroxide

FIGURE 8. Corrosion Rate as a Function of Nitrate and Hydroxide

FIGURE 9. Corrosion Rate as a Function of Aluminate and Hydroxide

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Appendix A

Analytical results for OH^- , NO_3^- , NO_2^- , Cl^- , SO_4^{2-} , alumina and phosphate at the end of the 4-month tests plus analyses from several 8-month tests.

4 Months - 100°C

Soln. No.	wt. %	IC				ICP	
		wt. %				wt. %	
		OH ⁻	NO ₃ ⁻	NO ₂ ⁻	Cl ⁻	SO ₄ ⁼	Al
1	6.04	5.20	1.087	0.060	0.106	1.45	--
3	4.09	7.58	15.92	0.356	0.084	2.15	0.101
5	4.51	25.3	2.94	0.28	0.028	1.08	--
7	6.34	13.83	7.73	0.195	0.040	0.784	0.134
8	5.15	4.33	16.93	0.355	0.026	2.38	--
9	3.94	6.32	17.73	0.142	0.046	1.776	--
12	4.33	4.44	3.58	0.40	0.204	4.71	0.546
13	5.66	--	--	--	--	2.80	0.111
15	7.84	11.72	3.07	0.521	0.045	1.62	0.141
16	3.85	15.25	8.88	0.203	0.062	2.45	0.807
20	6.70	3.56	19.86	0.148	0.014	0.501	--
28	9.40	6.32	1.33	0.128	0.044	1.75	--
29	10.67	9.52	8.72	0.124	0.021	0.68	0.0264
30	11.64	4.32	0.502	0.353	0.167	0.143	0.145
33	13.11	2.73	9.46	0.238	--	0.135	0.043
36	11.81	5.10	0.278	0.099	0.018	0.050	0.024
38	10.99	9.44	6.68	0.295	0.066	0.041	0.015
44	10.05	3.55	15.13	0.180	0.076	0.1887	--
48	0.256	8.01	0.323	0.009	0.28	--	0.44
49	0.280	7.46	0.961	0.055	0.337	0.256	1.30
50	--	12.88	11.41	0.094	0.141	--	0.544
51	0.526	9.04	19.13	0.064	0.118	--	--
53	1.09	11.25	6.99	0.135	0.042	0.095	--
55	0.217	--	14.96	0.111	0.029	0.080	0.102
57	0.097	--	--	0.101	--	--	0.263
58	--	13.92	0.660	0.016	0.123	0.248	--
59	0.72	12.36	1.833	0.139	0.041	0.216	--

4 Months - 80°C

Soln. No.	wt. %	IC				ICP	
		wt. %				wt. %	
		OH ⁻	NO ₃ ⁻	NO ₂ ⁻	Cl ⁻	SO ₄ ⁼	Al
1	5.57	4.57	2.30	0.200	0.070	1.27	--
3	4.03	5.49	16.55	0.396	0.115	1.54	0.347
5	5.26	20.78	3.31	0.223	0.046	1.29	--
7	8.19	9.82	8.58	0.299	0.025	1.32	0.052
8	4.93	--	--	--	--	2.10	--
9	5.16	8.30	13.58	0.216	0.067	2.415	--
12	5.23	5.12	3.95	0.346	0.093	5.60	0.472
13	6.76	--	--	--	--	2.59	0.078
15	5.94	11.36	3.67	0.568	0.101	3.32	0.205
16	5.55	13.46	8.86	0.271	0.077	3.78	0.095
20	7.87	4.21	13.38	0.218	0.0165	0.57	--
28	9.97	6.54	1.50	0.109	0.0294	1.73	--
29	11.26	9.35	8.32	0.202	0.022	0.747	0.021
30	12.40	4.98	0.681	0.768	3.94	0.152	0.242
33	13.58	3.14	8.68	0.230	0.0024	0.083	0.023
36	15.61	8.69	0.435	0.194	0.014	0.082	0.025
38	11.66	13.34	4.29	0.415	0.112	0.054	0.047
44	11.55	4.11	7.71	0.214	0.119	0.218	--
48	0.196	8.71	0.800	0.021	0.350	--	0.453
49	0.442	8.41	0.769	0.053	0.357	0.187	1.385
50	--	12.94	10.31	0.088	0.120	--	0.881
51	0.480	--	--	--	--	--	--
52	0.766	--	--	--	--	--	--
53	1.45	12.34	8.81	0.154	0.020	0.106	--
55	0.214	--	17.15	0.127	0.069	0.104	0.601
57	0.084	--	--	--	--	--	0.520
58	--	19.54	1.01	0.029	0.171	0.344	--
59	1.13	18.03	2.97	0.213	0.049	0.311	--

4 Months - 60°C

Soln. No.	wt. %	IC				ICP	
		wt. %				wt. %	
		OH ⁻	NO ₃ ⁻	NO ₂ ⁻	Cl ⁻	SO ₄ ⁼	Al
1	5.43	4.56	2.80	0.133	0.045	1.260	--
3	4.19	5.53	17.75	0.253	0.046	1.467	0.483
5	5.30	20.08	3.173	0.166	0.027	1.295	--
7	7.10	11.96	10.70	0.281	0.033	1.458	0.047
8	5.52	4.20	12.92	0.275	0.027	2.261	--
9	5.28	14.778	24.121	0.476	0.170	2.307	--
12	5.17	3.959	3.111	0.504	0.057	5.146	0.584
13	7.13	5.09	11.29	0.400	0.021	1.927 (100X) 1.767 (10X)	0.1010 0.1057
15	6.62	13.29	4.80	0.643	0.027	2.322	0.1760
16	5.42	13.503	8.308	0.395	0.219	3.516 (100X) 3.222 (10X)	0.1995 0.1907
20	8.43	--	--	--	--	0.627	--
28	9.41	8.42	1.88	0.301	0.016	2.409	--
29	7.68	11.02	10.30	0.369	0.028	0.8067 (100X) 0.7527 (10X)	0.1023 0.0994
30	12.78	4.844	--	0.547	--	0.1526	0.3197
33	13.77	4.70	8.491	0.467	--	0.1313	0.020
36	11.55	11.55	0.457	0.32	0.081	0.0823	0.1531
38	12.09	10.993	4.115	0.550	--	0.0566	0.0521
44	11.38	2.852	8.137	0.419	--	0.2201	--
48	0.236	8.3909	0.6703	0.0874	0.3964	--	0.4430
49	0.098	7.135	0.672	0.104	0.480	0.2840	1.3988
50	--	12.372	1.489	0.126	0.163	0.0023 (100X) -- (10X)	0.8775 0.8431
51	0.496	11.9506	24.3356	0.125	0.3352	--	--
52	0.821	14.3147	13.528	0.2076	~0.0508	--	--
53	1.21	14.3632	11.1403	0.2076	46.169	0.1298	--
55	0.220	19.1354	18.2147	0.2053	~0.1656	0.106	0.7264
57	0.083	15.2917	19.8626	0.1456	~0.157	-- (100X) -- (10X)	1.0321 1.0276
58							
59	1.92	13.4085	0.5077	0.1549	~0.0715	0.2408	--

4 Months - 40°C

Soln. No.	wt. %	IC				ICP	
		wt. %				wt. %	
		OH ⁻	NO ₃ ⁻	NO ₂ ⁻	Cl ⁻	SO ₄ ⁼	Al
1	5.70	4.34	2.67	0.142	0.076	1.120	--
3	5.44	6.58	11.88	0.314	0.016	1.786	0.223
5	5.59	16.52	3.38	0.175	0.041	1.311	--
8	5.56	4.42	11.06	0.220	0.026	2.288	--
9	5.05	8.080	12.10	0.3065	0.093	2.234	--
12	5.80	5.078	3.816	0.603	0.037	0.555	0.258
16	5.56	10.679	7.238	0.399	0.222	3.392 (10X)	0.189
						3.659 (100X)	0.193
20	7.95	3.842	8.279	0.317	0.036	0.579	--
30	12.86	4.978	--	0.526	--	0.154	0.197
33	12.02	4.529	6.753	0.419	--	0.115	0.059
38	12.24	7.714	3.919	0.583	--	0.059	0.063
44	10.96	2.744	7.691	0.434	--	0.216	--
48	0.384	11.8505	1.0589	0.1463	0.5215	--	0.625
49	0.219	7.4699	0.7665	0.0874	0.4313	0.276	1.398
50	--	20.905	1.583	0.138	0.166	-- (10X)	0.442
						-- (100X)	0.463
51	0.552	12.793	22.277	0.1325	0.405	--	--
52	0.889	12.4324	11.8000	0.2218	~0.0519	--	--
53	1.38	6.7894	5.9312	0.0623	~0.0300	0.131	--
55	0.272	18.0705	20.1144	0.2216	~0.2884	0.117	0.450
57	0.092	14.9185	16.2518	0.1866	~0.1001	-- (10X)	0.755
						-- (100X)	0.762
58	--	8.6698	0.8428	0.1414	~0.2963	0.313	--
59	1.91	13.2446	0.4803	0.2049	~0.0706	0.238	--

8 Months - 100°C

Solution No.	wt. %					
	<u>OH⁻</u>	<u>NO₃⁻</u>	<u>NO₂⁻</u>	<u>Cl⁻</u>	<u>SO₄⁼</u>	<u>PO₄⁼</u>
1 before	6.9	4.25	3.39	.27	.32	
9 before	4.53	7.69	12.65	.62	.29	
9 after	5.25	8.18	13.87	.37	.14	
16 before	4.99	14.87	7.95	.33	.31	.55
16 after	5.1	12.27	7.97	.37	.17	.44
20 before	7.02	4.45	12.86	.28	.2	
20 after	7.27	4.18	12.45	.26	.17	
30 before	10.4	4.51	.73	.51	.22	1.08
30 after	10.5	4.12	.50	.43	.22	.98
38 before	8.95	13.65	11.64	.37	.071	.19
38 after	11.355	10.94	9.61	.52	.11	.17
44 before	10.146	4.10	8.18	.43	.15	
44 after	10.2	3.86	7.36	.38	.093	
51 before	.886	11.37	25.36	.11	.25	
51 after	.804	10.85	24.64	.093	.25	

Appendix B

A-516 & A-537

Tables of observed and predicted values,
tabulated by exposure time in months, tem-
perature in °C, and mixture.

NOTE: All data are presented to five
decimal places. For engineering
purposes, round to one decimal
place.

A-516 - 4-Month Data

Rows: Composition

Columns: Temperature

	80	100	ALL	
1	.10400 .11818	.28500 .30474	.21260 .23011	Observed Mean Predicted Value
3	.08300 .15517	.28867 .32117	.20640 .25477	
5	.06700 .11038	.23567 .29352	.16820 .22026	
7	.07850 .06993	.14200 .09136	.11660 .08279	
8	.14200 .13614	.27467 .28724	.22160 .22680	
9	.09750 .09131	.26267 .23129	.19660 .17530	
12	.09650 .11744	.14067 .14079	.12300 .13145	
13	.08650 .07177	.25367 .25950	.18680 .18441	
15	.08900 .12017	.09300 .08101	.09140 .09667	
16	.08400 .08803	.19833 .18875	.15260 .14846	
20	.29750 .16898	.54233 .47365	.44440 .35178	
28	.11000 .09048	.30133 .29948	.22480 .21588	
29	.11250 .08391	.17867 .20322	.15220 .15550	
30	.13850 .13002	.68067 .64107	.46380 .43665	

33	.19150 .20425	1.18800 1.16894	.78940 .78306	Observed Mean Predicted Value
36	.17500 .19188	.54333 .56350	.39600 .41485	
38	.12600 .11817	.62333 .58546	.42440 .39854	
44	.13650 .16315	.46367 .56056	.33280 .40160	
48	.06800 .06549	.06067 .08673	.06360 .07823	
49	.07750 .10569	.01867 -.01013	.04220 .03620	
50	.06950 .08869	.01167 -.00323	.03480 .03354	
51	.07250 .06516	.01433 .02503	.03760 .04108	
52	.07150 .08173	-.00167 .00359	.02760 .03484	
53	.07350 .04571	.01900 .05555	.04080 .05162	
55	.09200 .08538	-.00267 -.00571	.03520 .03073	
57	.07950 .06926	.03033 .01349	.05000 .03580	
58	.08300 .06426	.11500 .10965	.10220 .09150	
59	.09050 .11106	.09300 .07203	.09200 .08764	
ALL	.10689 .10756	.25193 .25151	.19391 .19393	

A-516 - 8-Month Data

Rows: Composition

Columns: Temperature

	40	60	80	100	ALL	
1	.14700 .18327	.16200 .16368	.19100 .17176	.23500 .28310	.18375 .20046	Observed Mean Predicted Value
3	.18200 .17539	.15600 .16298	.19000 .17041	.27200 .27503	.20000 .19595	
7	.13900 .21617	.15400 .17167	.00200 .15349	.21900 .23477	.12850 .19403	
8	.14700 .14732	.12600 .15190	.18900 .16979	.32500 .28126	.19675 .18756	
9	.17300 .14504	.15100 .15521	.21900 .17902	.34100 .29407	.22100 .19334	
12	.13400 .16991	.24000 .15528	.18100 .17039	.28300 .27289	.20950 .19212	
13	.16600 .16702	.11500 .15786	.15900 .17329	.28200 .29378	.18050 .19799	
15	.26800 .22441	.16400 .16795	.27200 .15078	.22200 .23819	.23150 .19533	
16	.16600 .13694	.14000 .14626	.15000 .18737	.26200 .33080	.17950 .20034	
20	.18200 .21372	.17100 .16698	.26700 .21517	.45000 .36966	.26750 .24138	
28	.17200 .18259	.28500 .20836	.18600 .21764	.32300 .29883	.24150 .22685	
29	.18900 .20253	.22300 .19397	.17900 .19173	.27800 .29002	.21725 .21956	
30	.19200 .19192	.17500 .18716	.26200 .22247	.34800 .35861	.24425 .24004	

33	.19700 .21693	.20300 .22779	.23200 .23976	.35900 .38194	.24775 .26660
36	.16200 .12140	.17000 .22221	.18500 .19290	.30500 .34503	.20550 .22038
38	.18600 .15435	.18500 .19855	.18700 .22342	.54200 .45374	.27500 .25752
44	.20400 .23593	.18800 .17324	.27800 .24445	.29800 .40947	.24200 .26577
48	.15100 .14817	.25200 .15673	.16300 .15136	.19400 .19936	.19000 .16390
49	.15200 .13246	.14500 .14178	.17900 .13967	.18200 .19069	.16450 .15115
50	.10800 .13920	.15100 .16067	.13900 .14834	.14600 .12925	.13600 .14437
51	.20000 .15130	.15500 .16404	.15100 .14570	.18700 .21907	.17325 .17003
52	.14000 .16674	.21600 .17020	.14000 .14933	.16900 .11205	.16625 .14958
53	.13200 .15703	.16000 .17641	.13200 .16585	.16600 .13132	.14750 .15765
55	.07800 .14053	.15400 .15925	.15000 .13645	.20800 .21801	.14750 .16356
57	.14100 .16130	.10100 .15136	.15300 .13158	.17700 .16151	.14300 .15144
58	.12800 .15509	.12800 .15469	.15500 .13969	.20800 .19204	.15475 .16038
59	.15500 .11313	.20000 .17155	.15200 .16663	.17800 .17865	.17125 .15749
ALL	.16263 .16851	.17296 .17103	.17937 .17587	.26515 .26456	.19503 .19499

A-516 - 12-Month Data

Rows: Composition

Columns: Temperature

	40	60	80	100	ALL
1	.02700 -.01807	.03950 .03741	.04900 .03321	.09100 .06566	.05163 .02956
3	.02050 .00999	.03150 .06879	.04350 .04670	.07750 .06901	.04325 .04862
5	.03000 -.00006	.02000 .00002	.04800 -.00858	.08600 .01832	.04571 .00400
7	.02300 .07088	.02450 .07806	.03800 .03955	.03850 .02317	.03100 .05291
8	.03050 .02307	.03700 .07369	.03700 .03430	.09200 .10536	.04913 .05910
9	.03150 .08558	.02600 .06336	.03900 .01809	.14100 .10533	.05938 .06809
12	.02450 .03646	.02000 -.02621	.03750 .04369	.12000 .10602	.05050 .04004
13	.03750 .02717	.02850 .02430	.03950 -.01125	.13500 .15515	.06013 .04884
15	.01150 .01421	.02400 .02607	.04000 .05348	.05450 .04814	.03250 .03548
16	.02400 .04111	.02950 .00494	.03450 .02804	.12800 .16493	.05829 .06241
20	.03100 -.03475	.04000 -.04644	.07300 .06245	.10000 .26535	.06529 .07542
28	.04400 .06067	.02950 .10472	.03100 .03865	.13250 .13470	.06143 .08811
29	.07300 -.01243	.03450 .07723	.03600 .00266	.05800 .06387	.04714 .03930
30	.04300 .03420	.04250 .11948	.06100 .01671	.15100 .21523	.07886 .10529

33	.04100 .05401	.04300 .05279	.08700 .09443	.31450 .32377	.13286 .14228
36	.24200 .33551	.97650 .70209	.07800 .16545	.12250 .10361	.37086 .32540
38	.05800 -.01786	.05250 .26896	.06150 .02676	.21550 .26131	.10243 .15660
44	.03800 .03135	.03700 .00160	.05800 .07196	.56750 .39749	.19471 .13906
48	.02500 -.00230	.03450 .05849	.02350 .02291	.03200 .00640	.02929 .02476
49	.02100 .01174	.01550 .07439	.02350 .05683	.03650 .03787	.02457 .04999
50	.03200 .03695	.01750 .04034	.01100 .06969	.01700 .06124	.01757 .05421
51	.02100 .01278	.01400 .09149	.02600 -.00586	.03250 .06022	.02371 .04350
52	.03200 .03671	.02200 -.02084	.02700 .09201	.00500 -.00358	.02000 .02455
53	.03600 -.01240	.02050 -.03311	.03450 .01751	.03950 .02437	.03214 .00073
55	.02500 .01167	.02200 -.00554	.03400 .01101	.02050 .06005	.02543 .02039
57	.02400 -.02656	.02300 .05463	.04000 .00324	.06700 .02431	.04057 .01969
58	.03000 .03878	.01350 -.00564	.02800 -.00564	.02750 .04226	.02400 .01439
59	.01400 .05983	.02850 .01983	.03350 .02279	.02900 .02236	.02800 .02711
ALL	.03584 .03128	.06239 .06803	.04176 .03801	.10470 .10578	.06363 .06364

A-537 - 4-Month Data

Rows: Composition

Columns: Temperature

	80	ALL	
1	.06100 .05569	.06100 .05569	Observed Mean Predicted Value
3	.00800 .03972	.00800 .03972	
5	.07800 .05344	.07800 .05344	
7	-.02500 -.04058	-.02500 -.04058	
8	.07600 .09348	.07600 .09348	
9	.03100 .01015	.03100 .01015	
12	-.08500 -.08904	-.08500 -.08904	
13	-.07800 -.05215	-.07800 -.05215	
15	.05300 .04276	.05300 .04276	
16	-.02000 -.01363	-.02000 -.01363	
20	.21800 .19824	.21800 .19824	
28	.04800 .13315	.04800 .13315	
29	.07800 .14500	.07800 .14500	
30	.23100 .25154	.23100 .25154	

33	.48600 .45309	.48600 .45309
36	.16000 .18895	.16000 .18895
38	.39400 .29211	.39400 .29211
44	.30000 .33141	.30000 .33141
48	.11100 -.02137	.11100 -.02137
49	-.04100 -.00809	-.04100 -.00809
50	-.08400 -.19938	-.08400 -.19938
51	.40100 .22583	.40100 .22583
52	.02300 -.05813	.02300 -.05813
53	-.14000 .01079	-.14000 .01079
55	-.14000 -.10651	-.14000 -.10651
57	-.07400 .06811	-.07400 .06811
58	-.09100 .03774	-.09100 .03774
59	.13300 .12218	.13300 .12218
ALL	.07543 .07730	.07543 .07730

A-537 - 8-Month Data

Rows: Composition

Columns: Temperature

	40	60	80	100	ALL	
1	.13350 .14075	.17000 .16763	.21650 .20486	.27300 .25672	.19825 .19249	Observed Mean Predicted Value
3	.17250 .16875	.16000 .17375	.19600 .20753	.25650 .27697	.19625 .20675	
7	.14900 .15214	.17400 .16702	.15950 .19588	.21000 .24472	.17313 .18994	
8	.15500 .15004	.13600 .16436	.22350 .19807	.25250 .26709	.19175 .19489	
9	.16800 .13807	.18350 .16290	.24500 .21595	.39250 .31244	.24725 .20734	
12	.18400 .19809	.15750 .17643	.23800 .21514	.35550 .32282	.23375 .22812	
13	.15000 .15606	.13000 .17017	.18550 .20405	.28650 .27564	.18800 .20148	
15	.15450 .18791	.18250 .17105	.24300 .19370	.24500 .25168	.20625 .20108	
16	.15100 .15281	.16200 .16299	.17750 .21813	.27400 .32897	.19113 .21572	
20	.21100 .21971	.21900 .18677	.29700 .23796	.35300 .29670	.27000 .23529	
28	.18250 .16859	.16900 .18167	.23300 .24625	.40850 .39574	.24825 .24806	
29	.18200 .20354	.17800 .18413	.20350 .23555	.31350 .33911	.21925 .24058	
30	.18450 .20139	.22050 .20547	.25750 .26873	.36000 .34962	.25563 .25630	

33	.23550 .21204	.19500 .23603	.29450 .30024	.35000 .34084	.26875 .27229
36	.16550 .20926	.23900 .21076	.26350 .26713	.36950 .38027	.25938 .26686
38	.20550 .14899	.21000 .20738	.27050 .27991	.48150 .46687	.29188 .27579
44	.21050 .21494	.20150 .20171	.26450 .26866	.29400 .33966	.24263 .25624
48	.13650 .14884	.14600 .14677	.13800 .17282	.21400 .21371	.15863 .17054
49	.13200 .10912	.18150 .13635	.19250 .16355	.18350 .20530	.17238 .15358
50	.14550 .12398	.13700 .14671	.18900 .18691	.17700 .18588	.16213 .16087
51	.12750 .18295	.16850 .15072	.17500 .17281	.18700 .17738	.16450 .17097
52	.11300 .10611	.13950 .17271	.12550 .16457	.16350 .16705	.13538 .15261
53	.12850 .13672	.14950 .16318	.22950 .20419	.18700 .18416	.17363 .17206
55	.15900 .13619	.15800 .16123	.14150 .15234	.18400 .17437	.16063 .15603
57	.15500 .12971	.16600 .14104	.16050 .16695	.18900 .18435	.16763 .15551
58	.10900 .12417	.12400 .13830	.17650 .17600	.21100 .20889	.15513 .16184
59	.12050 .12239	.23200 .16653	.17900 .20336	.20150 .19719	.18325 .17237
ALL	.16004 .16086	.17369 .17236	.21020 .21190	.27307 .27200	.20425 .20428

A-537 - 12-Month Data

Rows: Composition

Columns: Temperature

	40	60	80	100	ALL
1	-.05000 -.03480	.09200 .00955	.02200 .00380	.08500 .04899	.03725 .00689
3	-.04600 -.03133	-.02000 .01404	.02600 -.00431	.04400 .04913	.00100 .00688
5	.00100 -.00425	.06300 .00496	.00000 .01518	.13300 .13973	.04925 .03891
7	-.00500 -.01046	.07800 .00245	-.02600 -.01285	.10800 .05734	.03875 .00912
8	.02200 .01636	-.00800 .06008	-.01100 -.00108	.16700 .12149	.04250 .04921
9	-.03000 -.02744	.00800 .01952	.01300 .00155	.15600 .18471	.03675 .04459
12	-.01300 -.04966	-.03900 -.00300	.01600 .01168	.13500 .13122	.02475 .02256
13	.00600 .02613	.09200 .06461	.01600 -.00204	.09200 .12492	.05150 .05341
15	.01000 .03031	.00300 -.01650	-.00200 .00248	.04700 .05893	.01450 .01881
16	.03600 .06654	-.00700 .00715	.04300 -.00181	.25900 .24568	.08275 .07939
20	.06200 -.00261	.02500 -.02058	.08300 .05253	.13700 .19987	.07675 .05730
28	.14900 .13778	.08400 .09355	.00100 .03991	.15400 .09399	.09700 .09131
29	.04300 .09184	.04100 .04856	.01000 .02525	.11700 .17626	.05275 .08548
30	.00400 .05487	.09200 .09207	.10800 .05469	.17500 .22440	.09475 .10651

33	.02000 .02882	.13600 .11026	.24800 .27855	.48900 .47184	.22325 .22236
36	.29500 .27425	.40400 .31127	.13000 .12091	.25400 .29905	.27075 .25137
38	.09700 .09803	.09200 .17519	.13700 .16372	.67600 .59832	.25050 .25882
44	.09100 .05455	.00300 .02857	.11700 .12199	.41300 .39942	.15600 .15113
48	.09300 .10630	-.02800 .00603	-.02800 -.02963	.01900 -.00367	.01400 .01976
49	.13900 .05652	-.02300 -.00403	-.04700 -.03719	.03000 .01207	.02475 .00684
50	.03300 .05468	.02800 .01740	.03600 -.02363	-.03100 -.02148	.01650 .00674
51	.08400 .06319	.00400 .02175	.06300 .06121	.09900 .08508	.06250 .05781
52	-.04300 -.02520	.05900 .00858	-.04300 -.02860	.02700 .00968	-4.7E-10 -.00889
53	.00800 .00768	.03100 .02903	.01600 .03354	.02300 .08165	.01950 .03798
55	.04500 .06725	-.02200 -.01427	-.04700 -.01913	.01800 .04434	-.00150 .01955
57	.03700 .01331	-.00700 .01748	-.02000 .02068	.06200 .05650	.01800 .02699
58	.00700 .02405	-.01500 -.02462	.01700 .01246	.05400 .08511	.01575 .02425
59	.05900 .07151	.00800 .04032	-.02600 -.00577	.03500 .01147	.01900 .02938
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