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7. Abstract

This report documents the results of the corrosion tests that were performed to aid in the selection of the construction materials for multi-function waste tanks to be built in the U.S. Department of Energy Hanford Site. Two alloys were tested: 304L and Alloy 20 austenitic stainless steel. The test media were aqueous solutions formulated to represent the extremes of the chemical compositions of waste to be stored in the tanks.

The results summarized by alloy are as follows:

For 304L the tests showed no stress-corrosion cracking in any of the nine test solutions. The tests showed pitting in one of the solutions. There were no indications of any weld heat-tint corrosion, nor any sign of preferential corrosion in the welded areas.

For Alloy 20 the tests showed no general, pitting, or stress-corrosion cracking. One crevice corrosion coupon cracked at the web between a hole and the edge of the coupon in one of the solutions. Mechanical tests showed some possible crack extension in the same solution.

Because of the failure of both alloys to meet test acceptance criteria, the tank waste chemistry will have to be restricted or an alternative alloy tested.

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12/13/93

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(Phase 1)

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Yes No N/A

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☐	☐	☒	The stated task will accomplish the objective clearly.
☒	☐	☐	Corrosion tests modes being evaluated are appropriate.
☐	☐	☐	Test methods are satisfactory.
☒	☐	☐	Test temperature is appropriate.
☒	☐	☐	Test solutions are pertinent.
☒	☐	☐	Test matrix is acceptable.
☒	☐	☐	Test standards are sufficient.
☒	☐	☐	Test criteria are acceptable.
☒	☐	☐	Test criteria are sufficient.
☒	☐	☐	Test acceptance criteria are stated clearly.
☐	☐	☒	Test period is acceptable for scoping or screening test.
☒	☐	☐	Test specimens are acceptable and appropriate.
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Remarks

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(Phase 1)

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Reviewer: RE Westerman

Date: 12-16-93

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GLOSSARY

ABBREVIATIONS, ACRONYMS, AND INITIALISMS

ASME	American Society of Mechanical Engineers	LOM	Light optical microscope
ASTM	American Society for Testing and Materials	LTS	Low temperature sensitization
CC	Crevice corrosion	M	Molarity
CERT	Constant rate extension test	MWTF	Multi-Function Waste Tank Facility
Cl ⁻	Chloride	NACE	National Association of Corrosion Engineers
DOS	Degree of sensitization	NO ₃ ⁻	Nitrate
DST	Double-shell tank	NO ₂ ⁻	Nitrite
EDS	Energy dispersive	OH ⁻	Hydroxide
EPR	Electrochemical potentiokinetic reactivation	PNL	Pacific Northwest Laboratory
F ⁻	Fluoride	RA	Reduction of area
FCAW	Flux-cored arc welding	SAW	Submerged arc welding
FDC	Functional Design Criteria	SCC	Stress-corrosion cracking
GMAW	Gas metal arc welding	SEM	Scanning electron microscope
GTAW	Gas tungsten arc welding	SS	Stainless steel
HAZ	Heat affected zone	SST	Single-shell tank
HF	Hydrofluoric acid	tup	The "anvil" on which the tip of the stressing bolt in the WOL specimen rests
HTS	High temperature sensitization	UTS	Ultimate tensile strength
HWVP	Hanford Waste Vitrification Project	WHC	Westinghouse Hanford Company
IG	Intergranular	WOL	Wedge-opening-load
IGSCC	Intergranular stress corrosion cracking	XRD	X-ray diffraction
IPM	Intermediate processing module	YS	Yield strength

1.0 INTRODUCTION

1.1 OBJECTIVE

The corrosion test program described in this report was designed to provide initial (scoping) corrosion data needed to select the materials of construction for the Multi-Function Waste Tank Facility (MWTF) project. The primary purpose of the scoping test program was to test the suitability of 304L (UNS S30403) stainless steel (SS). A secondary objective was to test a backup alloy, Alloy 20 (UNS N08020), for use as a tank material should 304L SS prove unsatisfactory.

The functional design criteria (FDC) document, WHC-SD-W236-FDC-001 (Kendall 1991), designated 304L SS as the construction material of choice. However, a corrosion-evaluation program was required to validate selection of materials for the primary tanks because the existing database for the MWTF operating conditions is not sufficient to support materials selection. Type 304L SS and Alloy 20 were selected for testing. Test specimens of Alloy 625 (UNS N06625) were also procured.

Alloy 20 was selected as the preferred backup material because of expected corrosion resistance and relatively low cost. Alloy 625 specimens had been procured as secondary backup if early data indicated that neither the 304L SS nor Alloy 20 alloy was adequate.

1.2 ORGANIZATION

The corrosion testing program was directed and planned by Westinghouse Hanford Company (WHC). The testing was performed by Pacific Northwest Laboratory (PNL). The test results obtained were documented by PNL. The corrosion test plan, WHC-SD-W236A-001 (Carlos 1993), was written by WHC, with the support of an in-house corrosion consultant, with concurrence and support of the PNL corrosion laboratory scientists. In addition, it was reviewed independently by an outside consultant. The corrosion testing program personnel are listed below.

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In-house Consultant	Dr. J. R. Divine, PE
Welding Engineer	J. P. Hauptmann
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Metallographers	N. T. Saenz, L. B. Priest

W. C. Carlos was the principal author of this report, with written contributions also supplied by W. F. Brehm, R. E. Westerman, and M. J. Danielson. B. E. Sartoris provided editorial assistance and K. J. Stamey led the document preparation effort.

1.3 GENERAL APPROACH

The general approach to the corrosion test for the Hanford MWTF tanks was to review and augment the information already developed by U.S. Department of Energy (DOE) contractors at the Savannah River and Oak Ridge sites. Both sites had recently tested carbon steel for corrosion for their new waste tanks. Both sites had chosen 304L SS (Pawel 1992 and Wiersma 1991). Wastes at Savannah River and Oak Ridge are similar to Hanford wastes, but the Hanford wastes have a broader range of chemical compositions and wider temperature ranges.

The Hanford MWTF corrosion test plan (Carlos 1993) is similar to the Savannah River and Oak Ridge corrosion test plans (Pawel 1992 and Wiersma 1991). All of the plans included weight-loss tests, cyclic polarization tests, crevice-corrosion tests, and stress-corrosion cracking (SCC) U-bend tests. The Hanford test plan included SCC tests using pre-cracked wedge-opening-load (WOL) specimens.

The Hanford corrosion-test solutions (simulated wastes) are based entirely on the expected worst-case compositions of Hanford wastes.

2.0 SUMMARY

The two candidate alloys examined in the corrosion test program were evaluated for their resistance to general corrosion, pitting corrosion, and SCC. The alloys performed well in the great majority of the tests. Some deficiencies observed, however, make it necessary either to (1) define acceptable solution-composition limits for the alloys or (2) to use materials with greater corrosion resistance.

The general corrosion of 304L SS exceeded the FDC requirement of < 0.0004 in./y in one solution (solution 4 - 6M NaOH, high chloride, low fluoride, low nitrite, low nitrate). This behavior by itself is not sufficient grounds for rejecting the material, because the corrosion penetration rate observed was small in absolute terms and the corrosion allowance could easily be increased to account for the short time during which the tank would experience conditions typical of the test solution.

A more serious problem with 304L SS is the pitting observed in solution 3 (pH 5, high chloride/fluoride, high nitrate, low nitrite). Pits were observed in seven of the 25 specimens tested in that solution, including U-bend specimens and crevice-corrosion specimens. Pitting was observed in immersed specimens and specimens in the vapor space and at the waterline. These pits were not observed in an interim examination of these materials after two months' test time. Solution 3 had been expected to be the most likely, of the nine tested solutions, to show pitting behavior. Electrochemical potentiodynamic scan tests of 304L SS did not show pitting tendencies in any of the solutions. The absence of a tendency to pit in the short-term (several hours) potentiodynamic scan tests shows the limitations of the current understanding and application of this test method in determining pitting susceptibility in these solutions.

U-bend specimens of 304L SS did not crack in any of the solutions. Slow-strain-rate tests of 304L SS in selected solutions did not show decreased strength or ductility or fractographic evidence of brittle behavior. Pre-cracked WOL compact-tension specimens showed no tendency for crack elongation in any of the solutions.

The Alloy 20 specimens showed good resistance to general corrosion and pitting attack, and no cracking was observed on any U-bend specimen of this material. However, a crevice-corrosion coupon of Alloy 20 cracked through the web between a bolt hole and the specimen edge in solution 1 (6M sodium hydroxide, low chloride/fluoride, high nitrate, low nitrite). A WOL specimen tested in the same solution showed signs of crack extension during the test.

The deficiencies in performance noted would eliminate the two candidate materials from consideration as primary tank materials unless tank operating conditions can be changed to accommodate the use of one of the materials. In the case of 304L SS the tendency to pit and the possibility of SCC can be effectively eliminated if the pH levels in the tanks are kept above a value of ≈ 10.5 and temperatures are kept below ≈ 60 °C (140 °F). The values of 10.5 pH minimum and 60 °C (140 °F) maximum were stated by a panel of experts convened by the DOE-sponsored Tank Structural Integrity Panel (TSIP) after a presentation and review of our data. We have no basis at the present to recommend lower pH or significantly higher temperature boundaries than those

specified by the experts' panel. In the case of the Alloy 20 material, any unacceptable operating conditions in caustic solutions would have to be determined before guidelines for tank operation could be determined.

If use of either of the two candidate alloys with their attendant difficulties is not considered justifiable, other candidates are available. The most attractive among these are the so-called 6% Mo, or "super-austenitic" steels. Two of these that should be considered are AL-6XN¹ (UNS N08367) and 254 SMO² (UNS S31254). Both alloys have low carbon, nitrogen additions, and about 20% nickel, 20% chromium, and 6% molybdenum. They are much stronger than the 300 series stainless steels, and much more resistant to SCC and pitting. Both are ASME Code-approved in recent Code cases. Whether they can easily be fabricated is unknown (at the moment); Kaiser Engineers Hanford (KEH) staff are seeking information regarding their capability to be fabricated into large tanks. The alloys are reportedly slightly less expensive than Alloy 20. As they are stronger than 300 series stainless steel, perhaps the tank wall could be thinner, saving material and expense, and resulting in lower welding stresses. We anticipate that their acceptable behavior in the range of anticipated Hanford waste solutions used in the present study would be relatively easy to demonstrate.

If a material more corrosion resistant than the 6% Mo alloys is ever deemed necessary, the NiCrMo alloy Hastelloy³ C-276 would be the alloy of choice.

The material of choice for the secondary tank and inter-connecting piping should remain 304L SS provided it can be shown (1) that galvanic effects are not serious and (2) that it will not come into long-term contact with any unanticipated solution that could cause unacceptable corrosion damage.

¹AL-6XN is a registered trademark of Allegheny Ludlum Corporation, Pittsburgh, Pennsylvania.

²254 SMO is a registered trademark of Avesta Sheffield AB, Schaumburg, Illinois.

³Hastelloy is a registered trademark of Haynes International, Inc., Kokomo, Indiana.

3.0 CRITERIA

The test parameters for the initial corrosion tests were based on the WHC-generated criteria for the MWTF. The following design criteria are listed in the FDC.

3.1 DESIGN CRITERIA

3.1.1 Design Pressure

The FDC assumes the solution density for pressure considerations is 2.0. Therefore, the maximum static pressure at the bottom may be 2 atmospheres.

3.1.2 Design and Operating Temperatures

The design temperature is 205 °F (120 °C). The probable operating temperature is about 150 °F (66 °C).

3.1.3 Reference Material

The reference material is 304L SS. All other alloy choices (regarding strength, corrosion resistance, etc.) are compared to 304L SS.

3.1.4 Reference Waste

The reference Hanford waste composition limits are given in Table 1. This table is discussed in more detail in Section 5.2 of this report.

3.1.5 Design Life of Facility

The design life of the MWTF is 50 years.

3.2 TEST CRITERIA

Materials were tested for several different types of corrosion: general (uniform) corrosion, pitting corrosion, stress-corrosion cracking (SCC), crevice-corrosion, and preferential corrosion in the welded area. The following criteria were used to evaluate the different types of corrosion.

- General corrosion - less than 0.0004 in. per year (0.01 mm/y)
- Pitting corrosion - less than 0.0004 in. per year (0.01 mm/y)
pitting rate based on deepest pit
- Stress corrosion - no cracks seen with 20X magnification; no cracks
cracking observed in metallographic examination(s); no
evidence of SCC susceptibility from CERT and
WOL tests
- Crevice-corrosion - less than 0.0004 in. per year (0.01 mm/y)
based on deepest pit
- Preferential corrosion in welded area - less than 0.0004 in. per year (0.01 mm/y)
based on visual examination similar to
determination of pitting corrosion

Table 1. Waste Compositions Based on Review of FDC.

	FDC (mole/liter)		Unprocessed Waste				I P M Expected (mole/liter)		Recommended Changes to FDC (mole/liter)
	min	max	DST (mole/liter)		SST (mole/liter)		min	max	
Ag			0	0.0013	0	0			0.0013
Al	0.01	0.51	0.24	1.1	0.029	0.5			1.1
As			0	0.0066					0.0066
B			0	0.0536					0.054
Ba	0	0.13	0	0.0016	0	0.0014			
Bi					0	0.076			0.076
Ca	0	0.35	0.0014	0.099	0	0.17			
Cd	0	0.077	0	0.0074					
Cr	0	0.026	0.0084	0.28	0.0001	0.091			0.28
Cu			0	0.02					0.02
Fe	0.025	0.75	0.0004	0.26	0.0057	0.89			0.89
Hg			0	2.8E-05					2.79E-05
K			0.044	0.73	0.0002	0.0095			0.73
La, Nd	0	0.049	0	0.0066	0	0.0010			
Mg			0.0004	0.046					0.046
Mn	0	0.23	0.0003	0.16	0.0009	0.41			0.41
Mo	0	0.055	0.0002	0.0029					
Na	0.037	0.71	3.5	10.7	1.6	7.1			10.7
Ni	0	0.11	0.0002	0.0080	0.0000	0.042			
Pb			0	0.0040	0	0.12			0.12
Pd, Rh	0	0.005	0	0.0063					0.0063
Si (SiO2)	0	0.29	0.0024	0.1007	0.0004	0.46			0.46
Ti	0	0.05	0	0.0020					
U	0	0.11	0	0.0460					
Zr (ZrO2)	0	0.32	0	1.3	0	0.065			1.3
Acetate					0	0.0055			0.0055
Citrate					0.0042	0.06			0.06
Oxalate							0	0.34	0.34
Formate							0	0.34	0.34
EDTA					0	0.022			0.022
HEDTA									
Fe(CN)6					0	0.025			0.025
Cl	0	0.008	0.003	0.17	0	0.022			0.17
CO3	0.01	0.5	0.087	0.69	0.0144	0.38	0	2.7	2.7
F	0	0.36	0.014	5.	0.0010	0.71			5.
FP (Ca)	0	0.037	0	0.0001					
NO2			0.43	1.8	0.0086	0.83	0	1.	1.8
NOx (NO3)	0	0.58	0.77	3.6	0.64	5.1	0	4.7	5.1
OH (free)			0.51	4.4	0.25	6.9	2.2	6.9	6.9
PO4	0	0.056	0	0.4	0.0007	3.8			3.8
SO4	0	0.025	0.0128	0.16	0.0103	0.22			0.22
TOC	0	0.912	0	0.17			0	2.1	2.1
pH	5	14							delete 14

FDC = Functional Design Criteria (Kendall 1991)
IPM = Intermediate Processing Module

SST = single-shell tank
DST = double-shell tank

4.0 TEST STANDARDS

All testing in the program was carried out where applicable in accordance with the most current standards established by the American Society for Testing and Materials (ASTM) and test procedures established by the National Association of Corrosion Engineers (NACE); these standards include the following:

- ASTM A 262-81, *Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels*, Practice A, "Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels"
- ASTM A 262-81, *Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels*, Practice B, "Ferric Sulfate-Sulfuric Acid Test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels"
- ASTM E 399-90, *Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials*
- ASTM E 647-83, *Standard Test Method for Constant-Load-Amplitude Fatigue Crack Growth Rates Above 10^{-8} m/cycle*
- ASTM E 8-88, *Standard Methods of Tension Testing of Metallic Materials*
- ASTM E 813-88, *Standard Test Method for J_{IC} , A Measure of Fracture Toughness*
- ASTM G 1-88, *Practice for Preparing, Cleaning and Evaluating Corrosion Test Specimens*
- ASTM G 3-89, *Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing*
- ASTM G 5-87, *Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements*
- ASTM G 15-89a, *Terminology Relating to Corrosion Testing*
- ASTM G 16-88, *Practice for Applying Statistics to Analysis of Corrosion Data*
- ASTM G 28-72, *Standard Method of Detecting Susceptibility to Intergranular Attack in Wrought Nickel-Rich, Chromium-Bearing Alloys*
- ASTM G 30-79, *Standard Practice for Making and Using U-Bend Stress-Corrosion Test Specimens*
- ASTM G 31-72, *Practice for Laboratory Immersion Corrosion Testing of Metals*
- ASTM G 46-76, *Standard Practice for Examination and Evaluation of Pitting Corrosion*

- ASTM G 49-85, *Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens*
- ASTM G 58-85, *Practice for Preparing of Stress-Corrosion Test Specimens for Weldments*
- ASTM G 59-78, *Practice for Conducting Potentiodynamic Polarization Resistance Measurements*
- ASTM G 61-86, *Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Local Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys*
- NACE RP-0173-73, *Collection and Identification of Corrosion Products*
- NACE TM-0169-76, *Laboratory Corrosion Testing of Metals for the Process Industries*
- NACE TM-0171-71, *Autoclave Corrosion Testing of Metals in High Temperature Water.*

5.0 TEST ENVIRONMENTS

The test environments consisted of simulated waste solutions, with attention given to the liquid zone, the vapor zone above the liquid solutions, and the interface zone between the liquid and vapor zones.

The test plan also addressed the potential corrosive environments associated with condensates from the test solutions.

5.1 SOLUTIONS

The range of the proposed waste composition for the MWTF tanks is given in the FDC. These values were selected because they were the feed criteria for the Hanford Waste Vitrification Project (HWVP).

At the start of the MWTF materials selection program it was realized that though the tanks might provide the feed to the HWVP, they would not necessarily always contain waste maintained within these specifications. Not only would the ranges be expected to be different, but other constituents, currently in the existing storage tanks, could also be present.

5.2 SELECTION OF INDIVIDUAL TEST SOLUTIONS

A group of WHC process chemists and chemical engineers reviewed the waste compositions believed to exist in the single-shell (SST) and double-shell (DST) tank farms. They recommended changes to the FDC waste composition (Appendix G). The results of these recommendations are detailed in Table 1 (Section 3.1.4). The table gives the original FDC values on the left (expressed in molarity), the expected maximum and minimum concentrations in the SSTs, DSTs, and the Intermediate Processing Module (IPM), then the recommended changes to the FDC on the right.

5.3 MAKEUP OF SOLUTIONS

Because of its influence on pitting and stress-corrosion cracking, chloride is one of the major constituents in a corrosion study on stainless steels. In addition, fluoride may also play a similar role, though a lesser one. Even if less effective, fluoride is important because of the quantities present in the Hanford waste. Other important components include nitrates, nitrites, and hydroxides. Constraints on the size of the test matrix made certain assumptions necessary on the relationship between chloride and fluoride, thus the chloride-to-fluoride ratio was fixed at 2.

Selection of the ratio of 2 was somewhat arbitrary. For SSTs the chloride-to-fluoride ratio is typically about 0.03 and ranges from 0 to 1. In the DST waste, the ratio is most frequently around 0.01 to 0.05, i.e., similar to the SST waste. However, in the complex concentrate, the ratio rises to about 1.5 and in the DST slurry it can reach about 13. In addition, some washed wastes are predicted to have a ratio of about 2. The concentration of chloride in the tanks ranges from 0 to nearly 0.2M. The solution matrix shown in Table 2 was selected.

For some species the measured concentrations were as planned. However, solubility constraints caused the original target compositions of some of the nine test solutions to undergo precipitation. Based on results of solution make-up experiments at PNL, a revised set of solution compositions was proposed that conformed to the intent of the original matrix of test solutions while taking into account the limited solubilities resulting from the Na^+ common-ion effect. An attempt was made to retain the 6M NaOH in the concentrated solutions while keeping the Cl^- and F^- concentrations as close to their desired maximums as possible. The revised compositions are shown in Table 3.

In the revised procedure the Cl^- and F^- concentrations were determined by "amount added," rather than by analysis, because of the difficulty of analyzing small quantities of these species in concentrated solutions.

An intermediate coupon inspection in solution 3 to determine the solution pH in the two test vessels showed that the pH had increased significantly from the initial pH of 5. Vessel 5 had a pH of 6.2; vessel 6 had a pH of 9.2. Thin films of hydrated iron oxide (rust) were observed on the stressing bolt and on the top of the WOL specimen in vessel 6, as well as on the laser-cut edges of the metal washers of the crevice specimens from both vessels. The formation of these corrosion products is believed to be primarily responsible for these increases in pH. The pH of the solutions was adjusted to 5 with HCl, the specimens were replaced in the Teflon⁴ vessels, and the vessels were replaced in the constant-temperature bath. The entire examination process took about 4 hours. A week later all of the solution 3 vessels were checked for pH. Vessels 5 and 6 showed pH values of 5.8 and 6.1, respectively; vessels 23 and 24, containing specimens of Alloy 20, showed pH values of 6.4 and 7.9, respectively. Thereafter, the pH was adjusted periodically to maintain it at the desired levels.

5.4 SOLUTION LEVEL CONTROL

A total of 36 2-L Teflon vessels were used to contain the specimens in the general corrosion test. Accurate control of the liquid level was required in these tests. Use was made of switches that signaled unacceptable deviations from the normal level.

The level-sensing vertical float switches (maximum service temperature of 105 °C) are constructed of polypropylene on all wetted surfaces except for the O-ring, which is made of ethylene propylene. The switches have a dry-reed switch in the float stem, actuated by a magnet in the float which actuates/deactuates over approximately 1/4-in travel. At the start of the tests, after the vessels had reached their operating temperature (97 °C), all switches were lowered 1/4-in. below the point at which the LED indicators shut off. The LED indicators were monitored daily, and when an indicator was lit, 50 ml of deionized water was added through the condenser (35-40 ml would raise the liquid level in the vessels approximately 1/4-in.). Most of the test vessels needed no additions over the 4-month test duration, and none of the switches failed or exhibited any problems.

⁴Teflon is a registered trademark of E. I. du Pont de Nemours and Company, Wilmington, Delaware.

5.5 pH ADJUSTMENT

The pH adjustments made to the low-pH vessels are summarized in Table 4. No attempt was made to measure or adjust the pH of the high NaOH tests (solutions 1, 4, 5, 6).

5.6 VAPOR (CONDENSATE)

It was thought that in some cases certain components, primarily fluoride and chloride, might selectively volatilize from the solution and enhance the corrosivity of the vapor phase. To resolve this issue, several solutions were selected as sources of "condensate." These low-pH solutions, containing several high concentrations of F^- and Cl^- , were distilled and the resulting distillate analyzed. The condensate collection system is shown in Figure 1; the results of the distillation are shown in Table 5. On the bases of the composition of the distillate, we decided not to test coupons in condensate environments.

5.7 EXPOSURE TIMES

The coupons were tested for 120 days. The constant rate extension test (CERT) specimens were tested to failure. The electrochemical tests were performed at a scan rate of 0.2 mV/s. A typical test would run for about 4 hours.

5.8 AERATION

All liquid test solutions in the Teflon vessels were air sparged with atmospheric air at a rate of 1 L/day. The CERT test solutions were sparged with 2 to 3 L/day of air during the test.

5.9 TEMPERATURES

The temperature of the bulk liquid was $97\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ ($207\text{ }^{\circ}\text{F}$). The goal temperature was $97\text{ }^{\circ}\text{C}$ ($207\text{ }^{\circ}\text{F}$). Practical testing considerations mandated a temperature definition of $97\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$.

Table 2. Original Proposed Test Solution Compositions (mol/L).

Solution	1	2	3	4	5	6	7	8	9
OH ⁻ (free)	6.0	10 ⁻⁹	10 ⁻⁹	6.0	6.0	6.0	10 ⁻⁹	10 ⁻⁹	10 ⁻⁶
Cl ⁻ + F ⁻ (a)	0.01	0.01	0.30	0.30	0.01	0.30	0.30	0.01	0.01
NO ₂ ⁻	0.001	0.001	0.001	0.001	1.00	1.00	1.00	1.00	0.001
NO ₃ ⁻	4.00	0.01	4.00	0.01	0.01	4.00	0.01	4.00	0.01
pH	14.8	5.0	5.0	14.8	14.8	14.8	5	5	8

(a) The Cl⁻ to F⁻ ratio is 2:1

Entries are based on solution preparation data and are conservatively estimated to be within ±5% of values listed.

Table 3. Test Solution Compositions (mol/L).

Solution	1	2	3	4	5	6	7	8	9
OH ⁻ (free)(a)	6.0	10 ⁻⁹	10 ⁻⁹	6.0	6.0	6.0	10 ⁻⁹	10 ⁻⁹	10 ⁻⁶
Cl ⁻	0.007	0.007	0.20	0.20	0.007	0.20	0.20	0.007	0.007
F ⁻	0.003	0.003	0.10	0.01	0.003	0.01	0.10	0.003	0.003
NO ₂ ⁻	0.001	0.001	0.001	0.001	1.00	1.00	1.00	1.00	0.001
NO ₃ ⁻	2.00	0.01	2.00	0.01	0.01	2.00	0.01	2.00	0.01
pH	NM(b)	5.0	5.0	NM	NM	NM	5	5	8

(a) Free hydroxide entries given for solutions 2, 3, 7, 8, and 9 were calculated from measured pH values. The pH values are considered accurate to ± 0.02 units.

Entries are based on solution preparation data and are conservatively estimated to be within ± 5% of values listed.

(b) NM = not measured

Table 4. History of pH Adjustments, General Corrosion Test.

Solution	Vessel	Material	Adjustment Date	Solution pH	Adjusted pH
3	5 6	304L 304L (WOL)	9-7-93	6.22 9.20	5.03 ^(a) 5.01 ^(a)
3	5 6 23 24	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	9-14-93	5.78 6.08 6.43 7.90	no adjustment
3	5 6 23 24	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-5-93	6.04 6.24 6.61 7.81	5.07/5.81 ^(b) 4.94/6.00 ^(b) 4.97/5.84 ^(b) 4.98/5.89 ^(b)
2	3 4 21 22	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-5-93	7.26 8.45 6.79 9.32	3.87/5.33 ^(b) 5.36/6.31 ^(b) 4.83/6.15 ^(b) 5.23/6.58 ^(b)
7	13 14 31 32	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-5-93	8.52 8.98 6.90 9.05	5.02/6.24 ^(b) 5.00/6.32 ^(b) 5.04/6.06 ^(b) 4.97/6.19 ^(b)
8	15 16 33 34	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-5-93	8.68 8.69 8.44 8.52	4.70/6.53 ^(b) 4.85/6.39 ^(b) 5.03/6.48 ^(b) 5.04/6.49 ^(b)
3	5 6 23 24	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-26-93	6.00 6.44 6.09 6.45	4.86 ^(c) 4.90 ^(c) 4.86 ^(c) 5.46 ^(c)
2	3 4 21 22	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-26-93	6.53 6.90 6.36 6.99	5.15 ^(c) 4.42 ^(c) 4.66 ^(c) 4.53 ^(c)
7	13 14 31 32	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-26-93	7.10 7.61 6.49 6.65	4.91 ^(c) 4.75 ^(c) 4.71 ^(c) 4.70 ^(c)
8	15 16 33 34	304L 304L (WOL) Alloy 20 Alloy 20 (WOL)	10-26-93	7.34 7.41 7.29 7.18	5.18 ^(c) 4.94 ^(c) 5.08 ^(c) 5.06 ^(c)

- (a) pH measured only in pH-adjusted aliquot, not in entire vessel. Acid (HCl) addition made to vessel on basis of aliquot.
- (b) pH in adjusted aliquot determined 10-5-93/pH in entire vessel determined 10-13-93. Acid (HNO₃) addition was made to entire vessel on basis of aliquot on 10-5-93.
- (c) pH of entire vessel determined 10-26-93 after pH adjustment of vessel based on aliquot. Acid used for pH adjustment was HNO₃.

Table 5. Solution Compositions Used for Condensate Experiments (mol/L) and Analysis of Condensate Obtained From Them.

Solution		Concentration (mol/L)			
		F ⁻	CL ⁻	NO ₂ ⁻	NO ₃ ⁻
3	Bulk solution	0.10	0.20	0.001	1.9
	Condensate	0.000057	<0.000011	0.001	0.000082
4	Bulk solution	0.01	0.20	0.001	0.010
	Condensate	0.000049	<0.000011	0.023	0.00016
7	Bulk solution	0.10	0.20	0.95	0.05
	Condensate	0.00052	0.002	0.17	0.061
8	Bulk solution	0.003	0.007	1.00	2.0
	Condensate	0.000041	<0.0011	0.078	0.017
Deionized Water	Bulk solution	0.000052	0.000011	0.00067	0.000063
	Condensate	0.000049	0.00037	NM	0.000058
0.1M HF	Bulk solution	0.1	NM	NM	NM
	Condensate	0.016	NM	NM	NM

NM = not measured

Figure 1. Condensate Collection Test Using a Lachat⁵ Microdistillation Tube

⁵Lachat® is a trademark of Lachat Instruments, Milwaukee, Wisconsin.

6.0 TEST TYPES, CONFIGURATIONS, AND SIZES

The corrosion-test plan attempted to address all of the anticipated types of corrosion and/or material degradation mechanisms that could affect austenitic stainless steels. The test addressed stress-corrosion cracking (SCC), crack growth, pitting, crevice, general (uniform), preferential, interface, intergranular, and heat-tint corrosion (a form of crevice corrosion) in the condensate (tank dome), vapor zone, vapor-liquid interface, and liquid zones.

SCC was addressed by the U-bend and CERT tests. The U-bend coupons were examined visually for cracks and the CERT specimens were examined for the presence of ductile or brittle fracture at failure.

SCC crack growth was addressed by the WOL test and by measuring any crack growth during the test.

General corrosion was addressed by measuring any weight loss of the coupons during the test.

Crevice corrosion was addressed by fastening a insulated washer onto the surface of the weight-loss test and looking for any corrosion under the washer area.

Pitting corrosion was addressed by visually examining the coupon surfaces with a 6X (some with a 30X) magnifier to look for pits and by performing cyclic polarization electrochemical tests.

Preferential corrosion in the welded area was addressed by visually examining the coupons in the welded area for a knife-like cut into the surface of the coupon next to a weld.

Interface corrosion was addressed by examining those coupons partially immersed in the liquid.

Heat-tint corrosion was addressed by examining the welded coupons for corrosion in the heat-tint areas next to the welds.

Table 6 summarizes the types of corrosion tests and evaluation methods.

Table 6. Corrosion Types and Test Methods Summary.

Types of Corrosion	Evaluation Methods	Remarks
STRESS-CORROSION CRACKING (SCC) Vapor Liquid	U-bend, constant rate extension test (CERT) tension, and wedge-opening-load (WOL) tests U-bend test suspended above simulated waste Tests immersed in simulated waste	Corrosion literature indicates that 304L SS is susceptible to SCC. The tank interior has both vapor and liquid zones; both were addressed.
PITTING CORROSION Vapor Liquid	Base metal suspended in simulated waste vapor Cyclic polarization test in simulated waste	Corrosion literature indicates that 304L SS is susceptible to pitting in waste containing chloride. Both vapor and liquid zones were addressed.
CREVICE CORROSION (CC) Base metal Weld metal	Crevice-corrosion test with a crevice simulated on the base and weld metals	Because of the expected presence either of sludge layers or objects lying on the bottom of the tank, CC was addressed. The tanks will be of welded construction.
GENERAL CORROSION Base metal in vapor Base metal in liquid Welded base metal in liquid	Base metal suspended in simulated waste vapor Base metal immersed in simulated wastes Welded base metal immersed in simulated wastes	General corrosion occurs on the tank walls. Tests were performed to determine at what rate it would occur. All areas of the weld were examined.
PREFERENTIAL CORROSION IN WELDED AREAS Vapor zone Liquid zone	Welded base metal suspended above solution Welded base metal immersed in simulated wastes	Because welds are typically anodic to the base metal and welds are located in both the liquid and vapor zones, both cases were addressed.
INTERFACE VAPOR/LIQUID (IV/LC) CORROSION	Welded metal immersed half-way into simulated wastes	Because there is a vapor/liquid transition zone in the tank, IV/LC was addressed.
HEAT-TINT CORROSION Vapor zone Liquid zone	Welded base metal suspended above simulated vapor Welded base metal immersed in simulated wastes	The formation of the tint modified the surface composition leaving it possibly susceptible to corrosion.
INTERGRANULAR CORROSION	Addressed by test at mill (supplier tested).	Evaluated by ASTM A 262, Practice A and B.
CONDENSATE CORROSION		Condensate corrosion was addressed by collecting condensate and determining, by chemical analysis, whether it were more or less corrosive than its original solution. If the condensate was less corrosive than the original solution, then the corrosiveness of the original solution would be the determining value.

7.0 TEST MATERIALS AND FABRICATION CONDITIONS

The MWTf tanks are to be fabricated by welding without post-weld heat treatment. Thus, the welding effects on the materials selected for construction formed a major part of the corrosion tests because welding affects the corrosion resistance of the selected materials.

Table 7 shows the welding processes used on the specimens. Specimens without weldments were heat treated as shown below.

Table 7. Materials Selected for Corrosion Testing.

Alloy/Specification	Filler Metal, Weld Metal	Weld Process for Test Coupons
ASME SA-240 - 304L SS	SFA-5.9, ER308L	GTAW, GMAW
ASME SB-463 - Alloy 20	SFA-5.9, ER320LR	GTAW
ASME SB-443 - Alloy 625*	SFA-5.14, ERNiCrMo-3	GTAW

ASME = American Society of Mechanical Engineers

AWS = American Welding Society

GTAW = gas-tungsten arc welding

GMAW = gas-metal arc welding

SFA = ASME designation for the AWS filler metal specifications

Sensitizing Temperature in Test Alloys

304L SS 500 °C ± 5 °C (932 °F ± 9 °F) for 110 ± 10 hours

Alloy 20 680 °C ± 10 °C (1,256 °F ± 18 °F) for 1 hour

Alloy 625* 800 °C ± 10 °C (1,472 °F ± 18 °F) for 1 hour

*Specimens of Alloy 625 were procured but not tested.

8.0 TEST COUPON IDENTIFICATION

Each test coupon in the corrosion test was uniquely identified (stamped). Table 8 summarizes the coupon identification numbers: quantity, corrosion specimen type, alloy, and fabrication conditions.

8.1 CORROSION COUPON (SPECIMEN) DESCRIPTIONS

8.1.1 Combined U-Bend and Immersion Coupons

The combined immersion and U-bend test pieces had dimensions of 5 by $\frac{1}{2}$ by $\frac{1}{8} \pm .015$ in. as described in ASTM G 30. The test pieces included the bolt and nut of Alloy 20 alloy and insulated ceramic washers as described in ASTM G 58. The U-bend test pieces were mill-annealed and included both longitudinal and transverse weld specimens. The non-welded specimens were sensitized by heat treatment. The surfaces were ground with 200- $\pm$ 20-grit alumina abrasive. Figure 2 shows the U-bend specimen in detail A.

8.1.2 Constant Rate Extension Rate Specimens

The CERT test pieces were $\frac{1}{2}$ -in. wide by $\frac{1}{8} \pm .005$ -in. thick and had a gauge length of 1 in. There were welded and unwelded test pieces. In the case of welded specimens, the weld was located in the center of the gauge length. The welded-tension test specimens were made in accordance with ASTM G 49-85. Figure 2 shows the CERT specimen in detail B.

8.1.3 Wedge-Opening-Load Specimens

The WOL specimens were 1-in. thick. Although initial high-stress intensity was used, it lay within the limits of plane-strain loading conditions. Figure 2 shows the WOL specimen in detail C.

8.1.4 Crevice-corrosion Coupons

The crevice-corrosion test pieces were welded longitudinally and were 2 by 1 by $\frac{1}{8} \pm .015$ in. They included a Teflon crevice washer and a nut and bolt of the same materials as the base material to fasten the crevice washer to the test piece. The crevice-corrosion test pieces were welded, and the weld on the test piece was ground flat. The crevice-corrosion washer was located over the weld. Figure 2 shows the crevice specimen in detail D.

8.1.5 Electrochemical Specimens

The cyclic polarization tests was performed on $\frac{1}{2}$ -in. diameter by $\frac{1}{2}$ -in. long unsensitized cylindrical specimens in accordance with ASTM G 5-87. Figure 2 shows the electrochemical specimen in detail E.

8.2 CORROSION TEST MATRIX

Table 9 is a matrix listing the corrosion coupon type, the number of corrosion coupons, the weld condition and the environment in which it was located. Corrosion coupons of only one type of metal were immersed in a given solution.

Table 8. Corrosion Specimen Identification.

Quantity	Specimen Type	Alloy	Identification	Fabrication Conditions
36	U-bend	304L SS	18 stamped 1T, 2T, to 18T 18 stamped 1P, 2P, to 18P	Perpendicular (transverse) to rolling direction Parallel to rolling direction Mill-annealed and sensitizing heat treatment at 500 °C for 110 hr
108	U-bend	304L SS AWS 308L	54 stamped 19T, 20T, to 72T 54 stamped 19P, 20P, to 72P	Perpendicular to rolling direction Parallel to rolling direction Mill-annealed with a transverse weld in center
36	U-bend	304L SS AWS 308L	18 stamped 73T, 74T, to 90T 18 stamped 73P, 74P, to 90P	Perpendicular to rolling direction Parallel to rolling direction Mill-annealed with a longitudinal weld along center line of specimen
36	U-bend	Alloy 20	18 stamped 1TC, 2TC, to 18TC 18 stamped 1PC, 2PC, to 18PC	Perpendicular to rolling direction Parallel to rolling direction Mill-annealed with a sensitizing heat treatment at 680 °C for 1 hr
108	U-bend	Alloy 20 AWS ER320LR	54 stamped 19TC, 20TC, to 72TC 54 stamped 19PC, 20PC, to 72PC	Perpendicular to rolling direction Parallel to rolling direction
36	U-bend	Alloy 20 AWS ER320LR	18 stamped 73TC, 74TC, to 90TC 18 stamped 73PC, 74PC, to 90PC	Perpendicular to rolling direction Parallel to rolling direction
100	Crevice	304L SS AWS 308L	stamped 1 to 100	Mill-annealed, longitudinally centered weld by GTAW
100	Crevice	Alloy 20 AWS ER320LR	stamped 101 to 200	Longitudinally centered weld by GTAW
15	CERT	304L SS AWS 308L	stamped 1 to 15	Mill-annealed, welded by GTAW, supplied in "as-machined" condition
10	CERT	304L SS	stamped 1 to 10	No weld, sensitized at 500 °C for 100 hours, cut perpendicular to rolling direction
12	WOL	304L SS	stamped 1 to 12	No weld, sensitized at 500 °C for 100 hours, crack direction parallel to rolling direction
10	WOL	Alloy 20	stamped 13 to 22	No weld, sensitized 680 °C for 1 hour, crack parallel to rolling direction
5	Electro-chemical	304L SS	stamped 1 to 5	No weld, no sensitization heat treatment

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Table 9. Corrosion Test Matrix.

The following numbers and types of corrosion test specimens represent the estimated minimum requirements for corrosion testing of each material for each test solution.

Test Types	Unwelded	Transverse Weld	Longitudinal Weld	Remarks
Combined Immersion and U-Bend Test				U-Bend restrained with insulated bolt
Liquid	3	3	3	
Liquid/Vapor		3		
Vapor		3		
Crevice				Serrated crevice washer of Teflon
Liquid			3	
Liquid/Vapor			3	
Vapor			3	
Cyclic Polarization				
Liquid	1			
Constant Rate Extension Tension (CERT)				Welded specimens machined from center of butt-welded $\frac{1}{4}$ -in. plate
Liquid	1	1		
Wedge-Opening-Load (WOL)				Sensitized material
Liquid	1			

Total number of specimens of each material to be tested in each simulated waste solution is 25, excluding cyclic polarization and CERT tests.

Figure 2. Sketches of the Corrosion Test Coupons.

9.0 TEST MATERIALS AND CONDITIONS CERTIFICATIONS

9.1 MATERIALS CERTIFICATIONS

The corrosion specimen supplier was Metal Analysis Inc., Huntington Park, California, for Kaiser Engineers Hanford Purchase Order K68574.

9.1.1 Alloy Certifications

The metal suppliers of the 304L stainless steel coupons were Avesta Stainless Steel Inc., British Steel, and Washington Steel Corporation.

304L SS Avesta Stainless Steel Inc., Schaumburg, Illinois, supplied Heat #55901, dated 9/30/92, for ASME Code Section VIII material, alloy specification ASTM A 240-91A, 304L, 1½- by 96- by 184-in., solution annealed at 1,950 °F, water cooled or rapidly cooled by air, rolled, annealed and pickled (HRAP).

British Steel Stainless, P.O. Box 161, Shepcote Lane, Sheffield S9 1TR, UK, supplied ASTM A 240-90, 304L, hot-rolled softened and descaled, ¾- by 240- by 96-in. plate, cast (Heat) #Y3465, dated 10/12/91.

Washington Steel Corporation, Washington, Pennsylvania, supplied Heat #212094, dated 7/10/91, for ASME Code VIII material, ASTM A 240-90, 304L, ¾-in. sheet stock 48 by 48 in.

Alloy 20 Carpenter Technology Corporation, Reading, Pennsylvania supplied Heat #500394, dated 2/18/93, alloy specification ASTM B 463-91, Alloy 20, ¾- by 0.120- by 48-in. flat sheet, stainless, cold-finish, annealed, and Heat #500456-2A, dated 3/17/93, 1½-in. flat plate, 144-in. long, stainless, hot finish, annealed, and pickled.

9.1.2 Weld-Metal Certifications

308L SS The weld metal for the CERT specimens is identified as HT #IC 6A10067W, AWS SFA-5.9 Class ER308L, ¾-in. plate weld wire, supplied by Harris Soldering & Brazing, Cincinnati, Ohio.

ER320LR The weld metal for the welding was supplied by Metals Analysis Inc. It is identified as Heat #300079, specification AWS A5.9-81 Grade ER320, 3/32-in. weld wire.

9.1.3 Heat-Treating Certifications

304L SS Heat treating of the specimens was performed by Thermo Electron Corporation, Los Angeles, California, heat-treated Heat #212094, ¾- by 24- by 24-in. sheet stock; Heat #Y3465, ¾- by 20- by 7-in. plate; and Heat #55901, 1½- by 4- by 20-in. plate, in nitrogen medium at 932 °F ± 18 °F (500 °C ± 5 °C) for 100 ± 10 hours.

Per the review performed by the welding engineer (Appendix G), the heat treating is acceptable.

Alloy 20 Heat treating of the specimens was performed by Thermo Electron Corporation, Los Angeles, California, heat-treated Heat #500394, 0.125- by 18 by 24-in. plate; Heat #500456-2A, 1½- by 4- by 18-in. plate; and Heat #500456-2A, 1½- by 4- by 11-in. plate in argon medium, 1,256 °F ± 18 °F (680 °C ± 10 °C) for 1 hour. Per the review by the welding engineer (Appendix G), the heat treatment is acceptable.

9.1.4 Welding Certifications

304L SS Metal Analysis Inc. subcontracted the gas-metal arc welding (GMAW) of the ¾-in. thick CERT specimens to La Habra Welding. Accurate Weld Testing Laboratory did the welding procedure qualification tests, per the requirements of ASME Section IX, for La Habra Welding.

The welding of the U-bend and the corrosion coupons was performed by EBTEC/Radcliffe (EBTEC) with materials supplied by Metals Analysis Inc.

Alloy 20 The welding of the U-bend and the corrosion coupons was performed by EBTEC with materials supplied by Metal Analysis Inc.

The welding data submitted by Metal Analysis Inc. was reviewed and found to be adequate for the purpose of providing corrosion specimens (Appendix G).

9.1.5 Chemical Certifications

All solutions were mixed from reagent grades.

9.1.6 Solution Analyses

All solutions were analyzed to confirm conformance with the specified composition.

10.0 TEST EQUIPMENT

10.1 U-BEND, CREVICE, AND WEDGE-OPENING-LOAD TESTS

The U-bend, crevice-corrosion, and WOL test vessels were heated in hot oil baths. The three silicon oil baths were used to heat the 36 Teflon test vessels for the corrosion tests at PNL Building 314, Laboratory 43. A recirculating water chiller was used for the condensers. Teflon specimen holders were used in the Teflon test vessels. The temperature profile of one silicon oil bath was mapped in detail, and the temperature was shown to be constrainable to the required ± 3 °C by appropriate positioning of aluminum foil thermal barriers.

Level indicators were made up and installed in each reaction vessel. Plastic gutters were installed in the laboratory to carry the 36 air sparge/sampling lines, thermocouple leads, level-sensor leads, and level-indicator diodes leads.

10.2 CONSTANT RATE EXTENSION TEST

10.2.1 Slow-Strain-Rate Tests

The slow-strain-rate tests conducted used the gear-driven loading device and autoclave system shown in Figure 3. After the tensile specimen was placed in an autoclave and electrically isolated with alumina washers, the autoclave was filled with the appropriate solution, and the autoclave head was tightened to minimize evaporation.

After the test temperature of 97 °C was reached, a load was applied to the specimen by means of the gear-driven loading device shown at the top of Figure 3. The displacement rate was controlled by a constant-velocity motor and a series of gear reductions to turn a precision jack. This displacement was transmitted to the specimen through a sliding seal in the autoclave head.

The load applied to the specimen was measured with a load cell, and displacement was measured with a linear-variable differential transformer (LVDT). Both instruments were located between the autoclave and the jack assembly. The output of the load, displacement, and temperature transducers was recorded digitally with a calibrated data-acquisition system.

Slow-strain-rate tests were continued until the specimen fractured. Oxidizing conditions were maintained during the test by flowing 2 to 3 L/day of air through the plenum in the autoclave. After the test was completed, the specimen was measured to determine the reduction of area (RA) and elongation. Yield strength (YS) and ultimate tensile strength (UTS) were calculated from the test records and the dimensions of the pre-test specimen.

10.3 POTENTIODYNAMIC SCANNING EVALUATION

The electrodes were supplied to PNL with a 600-grit surface finish. Figure 4 shows the electrochemical cell, an off-the-shelf Princeton Applied Research unit. The 1-L cell was placed in a heating mantle and heated to the test temperature (97 °C). Test solutions were allowed to equilibrate with air.

A double-junction silver, silver chloride reference electrode was placed directly into solution so that it was at cell temperature. The outer junction contained test solution to minimize chloride leakage into the cell solution.

The electrodes were cleaned (in high-purity acetone followed by high-purity ethanol) and attached to the electrode holder, and the cell assembled. Test solution (750 mL) was added and the system heated to the test temperature. After one hour at temperature, the potential scan was started at a scan rate of 0.2 mV/s. The scan started at the open circuit potential and advanced anodically to a potential of approximately 1.0 V relative to the reference electrode; then the scan was reversed until the open-circuit potential was reached again. Current-potential data were recorded either on an X-Y recorder or by a data-acquisition system.

E_b and E_{prot} were determined and reported, as these indicate the propensity of a material to pit. If E_b is within 200 mV of the open circuit potential, the material is almost certain to nucleate pits.

Figure 4 shows a photograph of the electrochemical equipment.

Figure 3. Slow-Strain-Rate Test.

LVDT = Linear-variable differential transformer

Figure 4. Photograph of the Electrochemical Testing Device.

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11.0 TEST PREPARATIONS

Each sample and coupon was identified uniquely, with the identification documented. All coupons were cleaned ultrasonically in chloride-free detergent, degreased with reagent-grade acetone and ethanol, and stored in a desiccator until tested.

All samples were inspected visually for visual defects. All defects were photographed.

All test solutions were analyzed before the test. The pH of the test solutions was adjusted during the test.

The purity of the water used in the test solution was controlled and documented.

The heat tints (oxide films associated with the welding operation) were left on the welded coupons.

The U-bend test specimens were surface ground with 200- $\pm$ 20-grit alumina abrasive. After they were bent, the legs were restrained by an insulated bolt.

Crevice-corrosion test coupons were welded, and the weld was ground flat. A crevice washer was placed over the weld and bolted to the coupon. A hole was drilled through the test coupon weld to allow the attachment of the crevice washer to the coupon. The bolt and nut were torqued to 50 in./lb.

Cyclic polarization test samples were polished to a 600-grit finish. Test details are outlined in ASTM G 61-86.

The CERT test pieces were gauge marked and measured accurately with calibrated calipers for overall specimen length and distance between gauge marks. These measurements were recorded.

The WOL specimens were polished in the pre-crack area to allow crack length to be determined accurately.

The WOL specimens were [re-cracked with a servohydraulic machine in load control. The load was adjusted as the fatigue crack grew so that plane-strain conditions were maintained. The general guidelines of ASTM E 399 were followed for pre-cracking. After they were pre-cracked, the specimens were loaded to the desired stress-intensity (K_I) level (20 kip/in² or 22 MPa/in²) by the application of torque to the loading bolt, while the stress intensity was monitored by means of a clip gauge affixed to the mouth of the specimen.

The same solutions were used for the duration of the test. Distilled water was added to make up for evaporation losses during the test.

12.0 TEST QUALITY CONTROL

The impact level of the materials selection program was 3Q. The corrosion-test program at PNL was conducted to PNL Quality Assurance Impact Level 2. Westinghouse Hanford Company-approved test procedures were supplied for all testing. Material certifications were provided for all test materials. Chemical analysis (purity) and a list of the manufacturers was required for all chemicals used to make the waste test solutions. Calibration records were maintained for all test equipment.

13.0 SPECIMEN EXAMINATION

The following types of corrosion were evaluated and the results documented: stress-corrosion cracking, pitting corrosion, crevice-corrosion, uniform or general corrosion, liquid/air-interface corrosion, vapor-zone corrosion, intergranular corrosion, heat-tint corrosion and preferential corrosion in the welded area.

- All corrosion-test specimens and tests were evaluated by a corrosion scientist.
- Uniform corrosion in the liquid was determined by weight-loss tests of all the U-bend coupons that were totally immersed in the solution, as outlined in ASTM G 31-72. Any preferential corrosion or pitting in the base metal, heat-affected zone, or weld was recorded.
- Evaluation for the liquid/vapor interface corrosion was accomplished by inspection of the specimen surface in the interface region. The extent, appearance, and uniformity of the interface corrosion were recorded. The amount of significant interface corrosion was determined by weight-loss and direct measurements.
- Vapor-zone corrosion was determined by weight-loss tests of the U-bend coupons that were totally suspended in the vapor zone, as outlined in ASTM G 31-72. Any preferential corrosion in the base metal, heat-affected zone, or weld was recorded.
- Pitting corrosion was evaluated primarily by observation of the U-bend and crevice specimens. Evaluation was by inspection of the specimens under ordinary light with a low-power 20X binocular microscope or with a magnifying glass to see whether any pits were present on the surface of the test coupons. The standard for evaluation of the pitting corrosion was determined by ASTM G 46-76. The depth, number, size, shape, and location of pits were recorded. Cyclic polarization studies were unable to confirm the test observations.
- Examination for crevice corrosion was similar to that for pitting corrosion except that the test specimen was evaluated after the crevice washer was removed. Crevice corrosion on the welded and non-welded areas were documented. The depth, number, size, shape, and location of any crevice-corrosion regions were recorded.
- Stress-corrosion cracking was evaluated by U-bend, WOL, and CERT tests. Stress-corrosion cracking by U-bend was determined by inspection of the test coupons for cracks. The location, number, shape, and length of the cracks were evaluated. Type, number, and depth of cracks were to be determined by binocular microscope and metallography. If the specimen was fractured, the fracture was photographed and described in writing. All data were recorded.

Stress-corrosion cracking (SSC) by CERT testing was indicated by reduced tensile strength, reduced ductility, and fractographic evidence of brittle fracture of the specimen. The gauge length was inspected for cracks. The final diameter of the gauge section in the reduced neck was

measured and recorded. A scanning electron microscope (SEM) examination of the fracture surface was performed. A description and photograph of the fracture was recorded.

At the end of the exposure period, the WOL specimens were removed from the test solution and unloaded. The crack-opening displacement was recorded during the unloading to verify the actual stress intensity at the end of the exposure period. To determine the final load, each specimen was loaded in a servohydraulic test machine to the same displacement, then the specimens were heat treated to 600 °C to provide a heat-tint surface to define the existing crack. The specimen fracture surfaces then were exposed by cyclic loading, and the extent of SCC growth was measured. The nine-point method from ASTM E 813 (or an equivalent) was used to determine the final crack length. After the extent of crack growth was determined, the final crack length was used to estimate the threshold stress intensity for SCC growth, K_{ISCC} .

- Stress-corrosion cracking of a WOL specimen is indicated by advancement of the crack front during exposure of the specimen to a potentially SCC-producing environment.
- All test specimens were color photographed before and after they were cleaned. A description of any changes in the test specimen or test solution was documented.
- The color and description of any solids in the test solution were documented.
- The corrosion effect of leaving welding heat-tint on the specimens was determined and documented.

14.0 ARCHIVE

For future reference, the test corrosion coupons and representative samples of unused specimens will be archived for three years by PNL in desiccators in sealed bags, then turned over to WHC Materials and Welding Engineering.

15.0 TEST RESULTS

The amount of corrosion on each of the approximately 450 test specimens (coupons) was measured or determined and tabulated and documented by PNL. A summary of these results follows.

15.1 GENERAL CORROSION (Uniform)

The general (uniform) corrosion U-bend test results on 304L SS and Alloy 20 are presented in Table 10.

15.2 CREVICE/PITTING CORROSION

The crevice and pitting corrosion test results are presented in Table 11 (304L SS and Alloy 20).

15.3 CONSTANT RATE EXTENSION TEST

The test results from the CERT testing of 304L SS are presented in Table 12.

15.4 WEDGE-OPENING-LOAD

The crack lengths of the WOL-test specimens were measured before and after test with a travelling microscope accurate to ± 0.0001 in. (0.0025 mm). The results of these investigations could not be used to identify crack extension positively, primarily because the extent of crack progress was so small, in general, that distinguishing the exact end of the crack introduced an error of such magnitude that it made the measurement results inconclusive. An examination of the fracture surface of each specimen then was made with the aid of a scanning electron microscope (SEM) to determine whether a change of fracture mechanism could be distinguished for environmentally induced cracking. No fractographic evidence could be found for crack extension in any of the 304L SS WOL specimens. In the case of the Alloy 20 WOL specimens, micrographs 29-13 (solution 1) and 29-17 (solution 5) (Appendix A) appear to offer evidence of such environmentally induced cracking, as in each micrograph evidence of intergranular cracking appears at the crack "end." The intergranular attack lies between the transgranular pre-crack surface and the transgranular post-test fatigue surface, i.e., the surface generated during the separation of the two halves of the WOL specimen by fatigue loading. While the evidence is not dramatic, the observation of apparent caustic-induced crack extension are consistent with the evidence obtained from the cracked crevice specimen (Appendix A), which also cracked while being exposed to solution 1.

15.5 ELECTROCHEMICAL

Extensive electrochemical potentiodynamic scans were performed on 304L SS in a solution; the tests indicated that pitting and crevice-corrosion were not expected to occur. These results were not confirmed by the immersion tests. A detailed report and the potentiodynamic scans appear in Appendix E.

15.6 STRESS-CORROSION CRACKING

One U-bend corrosion coupon having a transverse weld was taken from each solution, cross-sectioned, and visually examined under a microscope at high magnification. No SCC was found. Photographs of the cross sections are shown in Appendix B.

15.7 HEAT TINT CORROSION

The corrosion coupons were examined visually for heat-tint corrosion, e.g., signs of unusual corrosion, such as pitting, that might be associated with the tint area. No such corrosion was found.

15.8 PREFERENTIAL CORROSION

The corrosion coupons were examined visually for preferential corrosion in the welded area. No such corrosion was found.

Table 10. Test Results - Uniform Corrosion, 304L SS and Alloy 20.

Solution	Liquid Zone	Liquid/Vapor Zone	Test Results
Uniform Corrosion Test Results - 304L SS (mils/y)			
1	0.3	0.3	0.02
2	0.02	0.01	0.01
3	0.03	0.03	0.02
4	0.4	0.47	0.01
5	0.2	0.3	--
6	0.1	0.3	0.01
7	0.02	0.03	0.01
8	0.02	0.02	0.02
9	0.01	0.01	0.01
Uniform Corrosion Test Results - Alloy 20 (mils/y)			
1	0.01	Not measured	Not measured
2	0.002	Not measured	Not measured
3	0.01	Not measured	Not measured
4	0.01	Not measured	Not measured
5	0.01	Not measured	Not measured
6	0.02	Not measured	Not measured
7	0.005	Not measured	Not measured
8	0.003	Not measured	Not measured
9	0.003	Not measured	Not measured

Table 11. Test Results - Crevice/Pitting Corrosion, 304L SS and Alloy 20.

Solution	Crevice-corrosion	Pitting Corrosion
Alloy 304L SS		
1	No	No
2	No	No
3	Yes	Yes
4	No	No
5	No	No
6	No	No
7	No	No
8	No	No
9	No	No
Alloy 20		
1	No	No
2	No	No
3	No	No
4	No	No
5	No	No
6	No	No
7	No	No
8	No	No
9	No	No

Table 12. Constant Rate Extension Test - 304L SS (Unwelded, Sensitized).

Strain Rate	YS (kip/in ²)	UTS	% Elong	% RA
Air Tests				
1 X 10 ⁻⁵ /s	36.7	76.1	61.1	70.5
1 X 10 ⁻⁶ /s	34.3	73.8	58.2	70.8
2 X 10 ⁻⁷ /s	--	--	--	--
Solution 3				
1 X 10 ⁻⁵ /s	27.2	72.3	52.9	72.7
1 X 10 ⁻⁶ /s	32.4	72.2	52.7	70.9
2 X 10 ⁻⁷ /s	33.1	71.4	48.5	68.6
Solution 4				
1 X 10 ⁻⁵ /s	35.7	72.3	74.4	53.4
1 X 10 ⁻⁶ /s	29.4	70.4	69.5	48.0
2 X 10 ⁻⁷ /s	33.3	70.9	49.4	70.0

CERT - 304L SS (Welded)

Strain Rate	YS (kip/in ²)	UTS	% Elong	% RA
Air Tests				
1 X 10 ⁻⁵ /s ^(a)	39.3	75.0	53.1	69.2
1 X 10 ⁻⁶ /s ^(b)	38.2	69.4	29.8	27.8
2 X 10 ⁻⁷ /s	--	--	--	--
Solution 3				
1 X 10 ⁻⁵ /s ^(a)	44.8	69.7	31.4	47.4
1 X 10 ⁻⁶ /s ^(b)	36.7	73.6	49.7	68.3
2 X 10 ⁻⁷ /s ^(b)	36.6	71.1	32.1	25.9
Solution 4				
1 X 10 ⁻⁵ /s ^(b)	48.7	67.7	44.5	28.4
1 X 10 ⁻⁶ /s ^(a)	46.5	71.6	45.7	41.5
2 X 10 ⁻⁷ /s	--	--	--	--

- (a) Specimen failed in gauge section, but not in weld.
(b) Specimen failed in weld.

YS = yield strength

UTS = ultimate tensile strength

RA = reduction of area

16.0 TEST CONCLUSIONS

Conclusions about whether the alloys are acceptable for use in the construction of the MWTF primary tank are based on each alloy's performance in the test in meeting the acceptance criteria stated in the MWTF corrosion test plan (Carlos 1993). The conclusions for 304L SS and Alloy 20 are stated below.

Corrosion Type

Met test plan acceptance criteria (Yes or No)

304L SS Alloy

General Corrosion (weight loss)

Vapor zone	Yes
Liquid/vapor zone	No (solution 4 only)
Liquid zone	Yes

Pitting Corrosion

Vapor zone	No (solution 3 only)
Liquid/vapor zone	No (solution 3 only)
Liquid zone	No (solution 3 only)

Crevice corrosion

Vapor zone	Yes
Liquid zone	No (solution 3 only)

Stress-corrosion cracking

U-bend	Yes
CERT	Yes (no significant reduction of YS/UTS)
WOL	Yes

Heat Tint

Yes

Cracks

Yes

Electrochemical

Yes

Alloy 20

General Corrosion (weight loss)

Vapor zone	Yes
Liquid/vapor zone	Yes
Liquid zone	Yes

Pitting Corrosion

Vapor zone	Yes
Liquid/vapor zone	Yes
Liquid zone	Yes

Crevice corrosion

Vapor zone	Yes
Liquid zone	Yes

Stress-corrosion cracking

U-bend	Yes
CERT	Not tested
WOL	No (slight crack extension, solution 1)

Heat Tint

Yes

Cracks

No

Electrochemical

Not tested

17.0 MATERIAL RECOMMENDATIONS

Based on the corrosion test results, the following recommendations are offered for each material.

Alloy 304L SS

Alloy 304L SS material cannot be recommended unconditionally for the environmental conditions defined in the FDC. Pitting was found in the vapor, liquid/vapor, and liquid zones in solution 3, a pH 5, high nitrate and chloride, low nitrite solution. High uniform corrosion was found in the liquid/vapor zone in solution 5, a high alkaline (pH of 14+), nitrite and chloride, low nitrate solution.

Overall, 304L SS is not recommended for use over the entire chemistry range listed in the FDC. For 304L to be recommended, the tank chemistry would have to be restricted to alkaline solutions above pH 10.5 and the tank design temperatures be lowered to less than 60 °C (140 °F) because of the pitting corrosion found in the 304L SS material. The 60 °C, pH 10.5 recommendation was formulated by the TSIP after review of our data (see Section 2.0). We have no basis at this time to recommend other limits. Part of our follow-on efforts will concentrate on feasibility of expanding the limits of 304L SS and possible test programs for doing so.

Alloy 20

Alloy 20 material is not recommended because in the crevice-corrosion tests, it cracked in solution 1, a highly alkaline (pH of 14+), high nitrate and chloride solution.

It is recommended that Alloy 20 should not be further considered and an alternative material should be investigated.

Other Materials

Alloy 316L, the 6% molybdenum series austenitic stainless steels, and a high-nickel alloy, should be considered for further investigation.

Secondary Tank Liner

Based on the MWTF FDC design (Kendall 1991), the normal benign desert atmospheric annulus environment and the Washington State Dangerous Waste Regulations (WAC-173-303) 90 day limit to retention of any waste in the annulus, 304L is recommended for the secondary tank material.

Secondary Tank Piping

Based on the MWTF FDC process piping design (Kendall 1991), short waste transfer durations, 20 years of 304L piping history without waste-related (compatibility) failures, and the test results reported herein, 304L is recommended for the process primary piping.

18.0 REFERENCES

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- Carlos, W. C., August 1993, *Multi-Function Waste Tank Facility Corrosion Test Plan*, WHC-SD-W236A-TP-001, Rev 1, Westinghouse Hanford Company, Richland, Washington.
- Kendall, B. A., November 1991, *Functional Design Criteria, Multi-Function Waste Tank Facility Project W-236*, WHC-SD-W236-FDC-001, Rev. 0, Westinghouse Hanford Company, Richland, Washington.
- Pawel, S. J., July 1992, *Preliminary Investigation of Corrosion of 1020 Mild Steel and Type 304L and 316L Stainless Steels in Liquid Low Level Wastes Simulating Melton Valley Storage Tank Requirements*, Y/DV-1149, Martin Marietta Energy Systems, Inc., Oak Ridge, Tennessee.
- Wiersma, B. J., and J. I. Mickalonis, March 1991, *Corrosion Study of Replacement Materials for Hazardous Low Level Waste Processing Tanks*, WSRC-TR-91-138, Westinghouse Savannah River Company, Savannah River Laboratory, Aiken, South Carolina.

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APPENDIX A

Photographs of Cracked Crevice-Corrosion Coupons

The crack was found between the support hole and the top of the coupon. No plastic deformation was observed. The crack appears to be environmentally assisted.

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Alloy 20 - Solution 1

Obverse Side

Edge Side

Reverse Side

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APPENDIX B

Photomicrographics of Corrosion Coupons

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Specimen 22T - Solution 1
Typical specimens, no cracking observed.

6.5X

31.5X

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APPENDIX C

Photographs of Typical Pitted Corrosion Coupons

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304L SS - Solution 3
pitting in edge of specimen, pitting under ZrO_2 washer.

36T

78T

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APPENDIX D

Photographs of Constant Rate Extension Rate Specimens

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304L SS - CERT Tests

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UTS = ultimate tensile strength

El. = elongation

sec = second

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APPENDIX E

Photographs of Wedge-Opening-Load Specimens

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304L SS - Solution 3
New Tank General Corrosion Test - 120 Days, 97 °C

WHC-SD-W236A-TRP-001
Rev. 0

WOL 3

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304L SS - Fracture Surface - Solution 3
Post-Cracked (Specimen 28-3)

SPECIMEN 28 - 3

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E-3

WHC-SD-W236A-TRP-001
Rev. 0

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304L SS - Solution 3
Post-Cracked (Specimen 28-3)

50X

250X

1500X

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Alloy 20 - Solution 1
Post-Cracked (Specimen 29-14)

SPECIMEN 29 - 14

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250X

Alloy 20 - Solution 1
Post-Cracked (Specimen 29-14)

250X

500X

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APPENDIX F

Pacific Northwest Laboratory Report on Electrochemical Scanning Test

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Final Report
September 1993

DETERMINATION OF THE ELECTROCHEMICAL/CORROSION CHARACTERISTICS
OF CANDIDATE WASTE TANK STRUCTURAL MATERIALS IN SIMULATED
WASTE SOLUTIONS USING THE POTENTIODYNAMIC
SCAN (CYCLIC POLARIZATION) TEST TECHNIQUE

1.0 INTRODUCTION

This report describes the results of a study on 304L SS to determine the pitting and crevice-corrosion behavior.

2.0 EXPERIMENTAL

ASTM Standards G-61¹ and G-5² along with Test Procedure 20735-3 were followed for the electrochemical tests. Electrode specimens were right circular cylinders of 304L SS with a surface area of 5.29 cm² (600 grit surface finish). The 304L SS electrodes were cleaned in high purity acetone followed by high purity ethanol. The counter electrode was a graphite rod. The test solutions (air saturated) were placed in a one-liter glass electrochemical cell which was heated to 97 ± 3 °C by a heating mantle.

A double junction silver, silver chloride reference electrode was placed directly into the test solution so that it was at cell temperature. The outer junction contained the test solution in order to minimize chloride leakage into the cell solution. Before each test, the potential of the silver, silver chloride reference electrode was checked against a calomel electrode (at room temperature) to insure that it was working correctly. The reported specimen potentials are relative to this 4.0 M KCl silver, silver chloride reference electrode at 97°C. This scale permits the potentials to be easily placed on the Standard Hydrogen Scale, if needed, in the future.

The test protocol called for the test solution (750 ml) to be added to the cell and the system heated to the test temperature. After one hour at temperature, the open circuit potential was recorded and the potential scan started at a scan rate of 0.2 mV/second. The scan started at the open circuit potential, advanced anodically until a potential approximately 1.0 V relative to the open circuit potential was reached, and then the scan was reversed until the open circuit potential was returned to. Current-potential data was recorded on a data acquisition system for later analysis. E_{pit} (pitting potential) and E_{prot} (protection potential) were determined as described in the above ASTM standards and test procedure.

The nine test solutions are shown in Table 1. A minimum of one test was run in each test solution. For a few solutions, additional pitting tests were carried out to assist in the assignment of E_{pit} and E_{prot} .

Table 1
Solution Composition and Test Data

Solution #	OH ⁻	Cl ⁻	F ⁻	NO ₂ ⁻	NO ₃ ⁻	E ₈₉₅ , V 97.5° C
1	5.80	.007	.003	.001	2.0	-.55
2	1E-9	.007	.0024	.0012	.0102	-.05
3	1E-9	.20	.10	.001	1.9	-.04
4	6.15	.20	.01	.001	.010	-.37
5	5.89	.007	.003	.93	.02	-.44
6	5.72	.20	.01	.91	1.9	-.59
7	1E-9	.20	.10	.95	.05	+.14
8	1E-9	.007	.003	1.00	2.0	+.21
9	1E-6	.0073	.0021	.0010	.0102	-.10

3.0 DISCUSSION

The potentiodynamic plots for the nine solution are shown on pages F-5 through F-16, and the open circuit potentials (corrosion potentials) are recorded in Table 1. Solution 7 (pages F-11 and F-12) showed evidence in two tests (the test was repeated) of pitting, both from a characteristic signature in the potentiodynamic scan and visual confirmation from the specimen at the tests end. This pitting is believed to be unlikely to occur in practice because the protection potential (+0.48 V) was too far away from the corrosion potential (+.14 V) for pitting to initiate. The protection potential is the interpreted as the minimum potential that pit initiation will occur. The probability for pitting increases as the potential becomes more positive, relative to the protection potential.

Solution 8 in its initial test (page F-13) also showed pitting by both the potentiodynamic signature and visual observation. When this test was repeated with the potentiodynamic scan going to lesser anodic potentials (1.3 V for the first scan versus .75 V for the repeat scan), pitting behavior was not observed. Clearly, the anodic portion of the scan more positive than +.75 V initiated the pitting process. This potential regime for pitting is so extremely far removed from the corrosion potential (+.21 V) that it would never be encountered in practical applications. Crevice-corrosion (a crevice was formed between the specimen and a Teflon washer of the specimen holder) was not a significant problem. In summary, pitting and crevice-corrosion is not expected to be a problem with 304L SS in the solutions studied in this test.

4.0 CONCLUSIONS

The tests in the nine solutions indicate that pitting and crevice-corrosion are not expected to be a problem.

5.0 REFERENCES

1. ASTM G-61, Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron, Nickel, or Cobalt-based Alloys.
2. ASTM G-5, Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements.

304L, Solution 1, 97 C

304L, Solution 93-2 97 C

304L, Solution 3 97 C

304L, Solution 4 97 C

304L, Solution 5 97 C

304L, Solution 6 97 C

304L, Solution 7, 97C

304L, 7 REPEAT TEST

304L, Solution 8. 97 C

304L, 8" REPEAT TEST

304L, 8 Repeat Test #2

304L, Solution 3 97 C

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APPENDIX G.

Miscellaneous Correspondence Related to the
Corrosion Test Program

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Westinghouse
Hanford Company

Internal
Memo

From: Waste Management Engineering 22170-93-012
Phone: 6-6631 H5-49
Date: June 23, 1993
Subject: RECOMMENDED WASTE COMPOSITION CHANGES TO THE MWTF FDC - REV 1

To: J. M. Light R3-49

cc: J. N. Appel	S4-58	P. R. Golberg	84-08
W. B. Barton	L4-75	B. D. Groth	84-08
A. L. Boldt	H5-49	G. L. Parsons	S4-57
K. D. Boomer	H5-49	B. C. Simpson	R2-12
W. F. Brehm	H5-67	G. E. Stegen	G6-06
W. C. Carlos	H5-52	K. C. Strong	R2-12
P. J. Certa	L4-75	J. A. Voogd	H5-49
D. W. Fritz	T1-30	E. D. Waters	S4-58
S. K. Farnworth	H5-55	L3/File	

- References:
1. Kendall, B. A., *Project W-236, "Multi-Function Waste Tank Facility," Functional Design Criteria*, (WHC-SO-W236-FDC-001, Rev. 0), August 31, 1992.
 2. Internal Memo, P. J. Certa, J. R. Divine, and M. J. Kupfer to J. M. Light, "Recommended Waste Composition Change to the MWTF FDC," dated February 25, 1993.
 3. Internal Memo, J. S. Garfield to J. N. Appel, et. al., "TWRS Systems Engineering Study Double shell Tank Waste Inventories, Rev. 1," Letter Number 25900-92-029, dated September 18, 1992.
 4. Boomer, K. D., 1993, *Tank Waste Technical Options Report* (WHC-EP-0616, Rev. 0), Westinghouse Hanford Company, Richland, Washington.

INTRODUCTION

Appendix A of the Functional Design Criteria (FDC) for the Multi-Function Waste Tank Facility (MWTF), Ref. 1, provides waste composition design criteria for the MWTF based only on the feed specifications for the Hanford Waste Vittrification Plant (HWVP). This letter provides chemical compositions for additional wastes that could be stored in the MWTF. These compositions should serve as a basis for revision of Appendix A of the FDC; and also a basis for corrosion testing of candidate materials for construction of the tanks.

J. M. Light
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June 23, 1993

22170-93-012

This revision to Reference 2 provides an updated table (Attachment 1) of estimated maximum chemical concentrations. This revision of the letter summarizes the SST and DST wastes as separate categories.

The MWTF compositions should support:

- Storage of retrieved double-shell tank (DST) waste
- Storage of retrieved single-shell tank (SST) waste
- Water or caustic washing of solids¹
- Storage of wastes resulting from processing waste at the Initial Pretreatment Module (IPM) to destroy organic complexants¹
- Storage of waste feed to the HWVP.

The table in Attachment 1, Estimates of the Maximum Concentrations of Chemicals Expected in the MWTF, compares the existing design criteria chemical compositions in the FDC with estimated chemical compositions for retrieved DST and SST wastes and IPM compositions. The table includes known drivers for the waste compositions and expected impact on the corrosion of the MWTF tanks.

DISCUSSION

The columns in the table labeled "FDC, Appendix A", are the data from that source converted from weight percent oxides to molarity.

The DST data are a composite of DST waste solids and supernatant (Ref. 3) and (if needed) retrieval water that could be sent to the MWTF. Minimum and maximum concentrations for each element for DST wastes are shown.

The columns labeled SST are the bulk composition ranges for SST waste in the 12 different SST farms (Ref. 4). Since much of the aqueous phase has been removed from the SSTs, the compositions listed include additional water (approximately two times the mass of the solids in the SST waste), assumed to be needed for retrieval of the waste. Because of the lack of characterization information for SST wastes, the Table does not distinguish between water insoluble solid phases and water soluble or supernatant phases. It should be noted that Reference 4 uses data from TRAC that has been adjusted so that the total inventory agrees with that in the EIS.

If the concentration of a particular element is not known, a blank space is shown in the table. This does not necessarily indicate the absence of that element. There may be cases where waste from individual tanks may not represent the average compositions for certain components shown. These isolated cases can, under certain conditions, result in an increase in the

¹Sludge washing operations and support of HWVP and IPM are planned to be disscoped from the MWTF project. They are included here since they are currently in the scope.

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corrosion to the tank material of construction. These maximum concentrations of key components in some waste types are noted in the "REMARKS" column of the table.

Water washing DST waste solids in the new tank farm would remove a significant percentage (up to 97%) of the Na^+ , NO_2^- , NO_3^- , and OH^- . Thus, after several washes with water the pH of the wash solutions should approach approximately 7. For conservatism, a minimum pH of 5 should be assumed. Also, pH 5 has been chosen as the minimum specification value for the cross-site transfer line Operating Specifications Document. Water washing SST sludge would also remove significant amounts of the sodium salts; however, washing efficiencies may not be as high as with DST waste since significant mineralization has occurred in SST waste to form sodium aluminosilicate and insoluble phosphate phases. The compositions for both DST and SST washed wastes are encompassed by the minimum and maximum concentrations shown in the table. If a more specific composition for a particular type of washed waste is required, e.g., for setting experimental conditions for corrosion testing, a more detailed analysis beyond that used for preparation of this letter will be needed.

Several different organic complexant destruction processes are under consideration for the IPM. For the wet air oxidation process, organic compounds are oxidized to $\text{CO}_2 + \text{H}_2\text{O}$; NO_2^- is oxidized to NO_3^- . For an ozone process, organic compounds are assumed to produce oxalate, formate, and carbonate by-products; NO_2^- is oxidized to NO_3^- . For a calcination process, all organic compounds are oxidized to $\text{CO}_2 + \text{H}_2\text{O}$; NO_2^- and NO_3^- are converted to NO_x which volatilizes. The residue from the calcination process (primarily Na_2O) will dissolve in water to produce OH^- . The degradation products from these processes result in significant changes in the concentrations of CO_3^{2-} , NO_2^- , NO_3^- , and OH^- in CC waste prior to transfer to the MWTF. Only these components are shown in the IPM column of the table.

For values that could directly or indirectly affect tank corrosion, the known driver responsible for these values are shown in the "DRIVER" column. That is, the approximate volume of the waste that drives the maximum/minimum values in the Table, along with the offending tank(s), farm(s) or waste type(s) is shown. Possible corrosion mechanisms for these components are listed in the "Impact" column.

J. M. Light
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CONCLUSION

The attached table provides for your consideration the chemical compositions of tank waste that could potentially be stored in the MWTF. Use of these compositions is recommended for providing the basis to revise Appendix A of the MWTF FDC. This information will in turn provide a better basis for developing the design criteria for the MWTF.

If you have any questions, please contact M. J. Kupfer on 376-6631.

J. R. Divine
Chief Engineer
ChemMet, Ltd., P.C.

M. J. Kupfer
Principal Scientist
Waste Management Engineering

mke

Attachment (1)

Estimates of the Maximum Concentrations of Chemicals Expected in the MWTF

Species	FDC, Appendix A (a)		Retrieved Waste (b)				PM Expected	DIMER (Mgal)	IMPACTS, if changed	REMARKS
	Anion/Cation		DST		SST					
	min mol/L	max mol/L	min mol/L	max mol/L	min mol/L	max mol/L				
Ag			0	0.0013						
Al	0.01	0.51	0.06	1.1	0.029	0.6		CC (4)	corrosion (c), (d)	
As			0	0.0088						
B			0	0.013					(c)	
Ba	0	0.13	0	0.0004	0	0.0014				
Be					0	0.078				
Ca	0	0.35	0.0014	0.10	0	0.17				
Cl	0	0.077	0	0.0074	0	0.0007				
Co	0	0.028	0.0087	0.28	0.0001	0.091		CC, PFP (5)		
Cu			0	0.02						
Fe	0.025	0.75	0.0004	0.28	0.0057	0.88				
Hg			0	2.8E-06	0	0.0001				
K			0.044	0.55	0.0002	0.0095				
La, Nd	0	0.049	0	0.0088	0	0.0010				
Mn			0.0004	0.048						
Mn	0	0.23	0.0003	0.18	0.0008	0.41		SST (2)		
Mn	0	0.055	0.0000	0.0028						
Na	0.037	0.71	1.5	10.7	1.5	7.1		DSSF (5)		dependent variable
N	0	0.11	0.0002	0.0080	0	0.042				B Tank Farm
Pb			0	0.0049	0	0.12				
Pb, Pb	0	0.005	0	0.0083						
Si (SiO2)	0	0.28	0.0024	0.0280	0.0004	0.48		SST (2)	Abrasion; (c)	Tanks 105-C and 106-C
Ti	0	0.05	0	0.0020						
U	0	0.11	0	0.0082						
Zr (ZrO2)	0	0.32	0	0.3	0	0.088		NCRW (1)	Deposits; (c), (d)	
Acetate					0	0.0055				
Chlorate			0	0.03	0.0042	0.08		SST	corrosion (c)	Tank 103-C
Oxalate							0.34	CC (4)	corrosion (c)	IPM organic destruction
Formate							0.34	CC (4)	corrosion (c)	IPM organic destruction
EDTA			0	0.018	0	0.011		SST (2)	corrosion (c)	
MBTA			0	0.021				CC (4)		Some C Farm tanks may be 0.3 M
Fe(Cr)3					0	0.025		SST (2)		
Q	0	0.008	0.003	0.17	0	0.022		CC (4)	SCC, Pitting	some CC tank supernate may go to 0.45 M
CO3	0.01	0.5	0.03	0.88	0.014	0.38	2.7	CC (4)	SXC	
F	0	0.38	0.014	1	0.0010	0.71		NCRW (1)	SXC	source: NCRW solids, 2 tanks; usually <0.5
Fluorin Prod	0	0.037	0	0.0001						
NO2			0.1	1.8	0.0088	0.83	1	DSSF (5)	Protection	103 SY - 2.8M; nitrite inhibits corrosion
NOx (NO3)	0	0.58	0.15	3.8	0.04	5.1	4.7	SST (5)		103 SY - 8.6M
Q1			0.24	4.4	0.25	8.8	4.8	SST (4)	SCC, Protection	Free Oil; total including bound Oil >>7M
P04	0	0.058	0	0.4	0.0007	3.8		SST (5)	Deposits	B & T Tank Farms
SO4	0	0.025	0.003	0.18	0.010	0.22		SST, DST (2)	Deposits	
TOC	0	0.812	0	2.0			2.1	CC (4)	corrosion (c)	
H2	5	14								many wastes are > 1 M Oil

(a) HWVP Feed Specification - expressed in mol/L

(b) Assumes waste is uniformly dispersed

(c) Can inhibit biotite corrosion at low pH

(d) Can form deposits which may lead to crevice corrosion

(e) Expected to increase the corrosion rate

Definitions:

CC - complexed concentrate; DST - Double Shell Tank; IPM - Initial Pretreatment Module;

DSSF - Double Shell Slurry Feed; NCRW - Neutralized Cladding Removal Waste;

PFP - Plutonium Finishing Plant;

SCC - Stress Corrosion Cracking; SST - Single Shell Tank

- (a) HWWP Feed Specification - expressed in mol/L
 (b) Assumes waste is uniformly dispersed
 (c) Can inhibit biotite corrosion at low pH
 (d) Can form deposits which may lead to crevice corrosion
 (e) Expected to increase the corrosion rate

Definitions:
 CC - complexed concentrate; DST - Double Shell Tank; IPM - Initial Pretreatment Module;
 DSSF - Double Shell Slurry Feed; NCRW - Neutralized Gladding Removal Waste;
 PFP - Plutonium Finishing Plant;
 SCC - Stress Corrosion Cracking; SST - Single Shell Tank

**Westinghouse
Hanford Company****Internal
Memo**

From: Materials and Welding Engineering 23440/JPH:kjs:93-5
Phone: 373-1817
Date: November 23, 1993
Subject: MULTI-FUNCTION WASTE TANK FACILITY CORROSION TEST DATA REVIEW

To: W. C. Carlos H5-52

cc: W. F. Brehm H5-67
E. M. Epperson B4-08
G. M. Foster E6-37
R. E. Westerman P8-44
JPH File/LB

The Multi-Function Waste Tank Facility corrosion test data (Phase 1) has been reviewed. Particular attention was given to the material certifications and welding records supplied by Metal Analysis Inc. The following comments are provided for your consideration.

1. Add the following to the list of material certifications for the corrosion tests performed by Metal Analysis Inc. With these additions all of the base metals are certified.

British Steel Stainless
PO Box 161, Shepcote Lane
Sheffield S9 1TR, UK

ASTM A240-90, 304L, Hot Rolled Softened and Descaled,
0.375 (3/8) x 240 x 96 in. plate, Cast (Heat) No. Y3465.
Dated 10/12/91.

Carpenter Technology Corporation
PO Box 14662
Reading, PA 19612-4662

ASTM B463-91, 20CB-3, Stainless Cold Finish Annealed,
0.120 X 48 X flat sheet metal, Heat No. 500394. Dated 02/18/93.

ASTM B463-92, 20CB-3, Stainless Hot Finish Annealed Pickled,
1.25 x 96 x flat plate, Heat No. 500456-2A (2A = 144" long).
Dated 03/17/93.

2. The sensitizing heat treatments performed by Thermo Electron Corp. on the 304L stainless and Alloy 20 base metals are acceptable.

W. C. Carlos
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23440/JPH:KJS:93-5

3. Metal Analysis Inc. subcontracted the gas metal arc welding (GMAW) of the 3/8" thick CERT specimens to La Habra Welding. Accurate Weld Testing Lab did the welding procedure qualification tests, per the requirements of ASME Section IX, for La Habra Welding. This is the only involvement either La Habra Welding or Accurate Weld Testing Lab had with Metal Analysis Inc. and the corrosion tests.
4. The U-BEND and CREVICE specimen welding and laser cutting was done by a subcontractor (EBTEC) with materials furnished by Metal Analysis Inc. Based on the documents provided for this review, there were no specific requirements for the welding procedure or welder qualification for the bead-on-plate welds. A telecom, 11/22/93, with Mr. Gregg Homolka of Metal Analysis Inc. verified that all base metals and weld filler metals had a chemical analysis overview performed prior to use by EBTEC.
5. The data reviewed does not support the supposition that all welding was accomplished by certified welders in accordance with procedures qualified to national standards, i.e., ASME or AWS. However, the welding was done under controlled conditions and is adequate for the purpose of providing corrosion specimens.

Original signed by

J. P. Hauptmann, Senior Engineer (Welding)
Materials and Welding Engineering

ggb

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