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# Materials Evaluation for a Transuranic Processing Facility

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Prepared for the U.S. Department of Energy  
Office of Environmental Restoration  
and Waste Management



**Westinghouse**  
**Hanford Company** Richland, Washington

Hanford Operations and Engineering Contractor for the  
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**MASTER**

**MATERIALS EVALUATION FOR A TRANSURANIC  
PROCESSING FACILITY**

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**ABSTRACT**

*The Westinghouse Hanford Company, with the assistance of the Pacific Northwest Laboratory, is developing a transuranium extraction process for pretreating double-shell tank wastes at the Hanford Site to reduce the volume of transuranic waste being sent to a repository. The bench-scale transuranium extraction process development is reaching a stage where a pilot plant design has begun for the construction of a facility in the existing B Plant. Because of the potential corrosivity of neutralized cladding removal waste process streams, existing embedded piping alloys in B Plant are being evaluated and "new" alloys are being selected for the full-scale plant screening corrosion tests. Once the waste is acidified with  $HNO_3$ , some of the process streams that are high in F<sup>-</sup> and low in Al and Zr can produce corrosion rates exceeding 30,000 mil/yr in austenitic alloys.*

*Initial results are reported concerning the applicability of existing plant materials to withstand expected process solutions and conditions to help determine the feasibility of locating the plant at the selected facility. In addition, process changes are presented that should make the process solutions less corrosive to the existing materials.*

*Experimental work confirms that Hastelloy<sup>1</sup> B is unsatisfactory for the expected process solutions; type 304L, 347 and 309 S stainless steels are satisfactory for service at room temperature and 60 °C, if process stream complexing is performed. Inconel<sup>2</sup> 625 was satisfactory for all solutions.*

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<sup>1</sup>Hastelloy is a trademark of Haynes International, Incorporated, Kokomo, Indiana.

<sup>2</sup>Inconel is a trademark of The International Nickel Company, New York, New York.

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LIST OF TERMS

AISI	American Iron and Steel Institute
CC	complexant concentrate
DST	double-shell tank
INEL	Idaho National Engineering Laboratory
NCRW	neutralized cladding removal waste
PFP	Plutonium Finishing Plant
TRU	transuranic
TRUEX	transuranium extraction
WINCO	Westinghouse Idaho Nuclear Company
WSRC	Westinghouse Savannah River Company

## MATERIALS EVALUATION FOR A TRANSURANIC PROCESSING FACILITY

## 1.0 INTRODUCTION

The Westinghouse Hanford Company is developing, with the assistance of the Pacific Northwest Laboratory, a chemical process for pretreating double-shell tank (DST) wastes at the Hanford Site. Typically, these wastes contain sufficient amounts of long-lived transuranic (TRU) materials that, after treatment, require storage in a repository within a borosilicate glass matrix.

Because of the expense for production and long-term storage of this glass waste form, a nitric acid-based processing method is being developed that will concentrate the TRU-containing portion of the waste. The residual low-level waste, which will constitute the bulk of the treated material, will be cast in grout.

Currently, the Hanford Site plans to pretreat the following three DST waste types:

- Complexant concentrate (CC) waste--16,000 m<sup>3</sup>
- Plutonium Finishing Plant (PFP) waste--1,500 m<sup>3</sup>
- Neutralized cladding removal waste (NCRW)--4,500 m<sup>3</sup>.

A pilot plant will be built in an existing facility to obtain samples of TRU material, each weighing 200 kg, from the above wastes for further testing and process development. While the pilot plant process evaluations and upgrades are being conducted, construction of a full-scale treatment facility is planned.

Because the waste contains fluorides and chlorides along with other chemical species, it became apparent that some acidified wastestreams would be particularly aggressive toward most of the in-plant embedded austenitic stainless steel piping. Corrosion rates as high 30,000+ mil/yr at 60 °C have been recorded for American Iron and Steel Institute (AISI) type 304L stainless steel in F-HNO<sub>3</sub> solutions. Because of this high corrosivity, corrosion evaluations are being performed on "old" metal specimens characteristic of existing embedded piping in the plant and are planned to be done on "new" alloys to be used in processing equipment. These evaluations will be done to define operating limits.

This paper will review waste composition variation and its influence on selection of corrosion test solutions; the self-complexing nature of most, but not all, of the NCRW streams; and corrosion test evaluation of the five in-plant "old" metals noted previously. "New" alloys selected for the full-scale plant and plans for their corrosion evaluation, along with a discussion of the expected process condition changes recommended to reduce the corrosivity of the streams, are included.

## 2.0 BACKGROUND

Understanding waste composition variation within DSTs is critical because of the impact on subsequent corrosion behavior of components during acid processing. In addition, the assessment of free HF by experimental means is important for assessing the corrosivity of simulated waste solutions. Before discussing waste composition effects and evaluating alloy corrosion, it is important to present some background on pretreatment processing and tank composition.

### 2.1 PRETREATMENT PROCESSING

The pretreatment processing that is common to all three wastes consists of the following four steps:

- Solids dissolution
- Solid-liquid separation
- Solvent extraction (transuranium extraction [TRUEX] process) to separate out the TRU components
- Product neutralization and concentration.

The undissolved solids and TRU-containing portion of the process effluent make up the feed to the Hanford Waste Vitrification Plant. The remaining portion will be fed to the Grout Treatment Facility.

### 2.2 WASTE TANK COMPOSITION VARIATIONS

The material in the DSTs has settled, leading to a nonuniform distribution of components. An example of the waste composition variation for the three types of waste is shown in Table 1; organic compounds in the CC waste are not shown in this table. The first column shows the various tank chemical species while the second and third columns show the highest and lowest concentration of species in the specific waste believed to be most corrosive, namely, NCRW (tank 103-AW). The fourth column shows the range of average concentrations of each waste type (NCRW, PFP, and CC) between different tanks.

Table 1 does not show that the maximum and minimum values for the various species don't always occur at the same level in a given tank nor do they all fluctuate with the same periodicity. Figure 1 shows the tank composition variation, top to bottom, for five particular species in NCRW tank 103-AW: F, Al, Zr, Cl, and Cr.

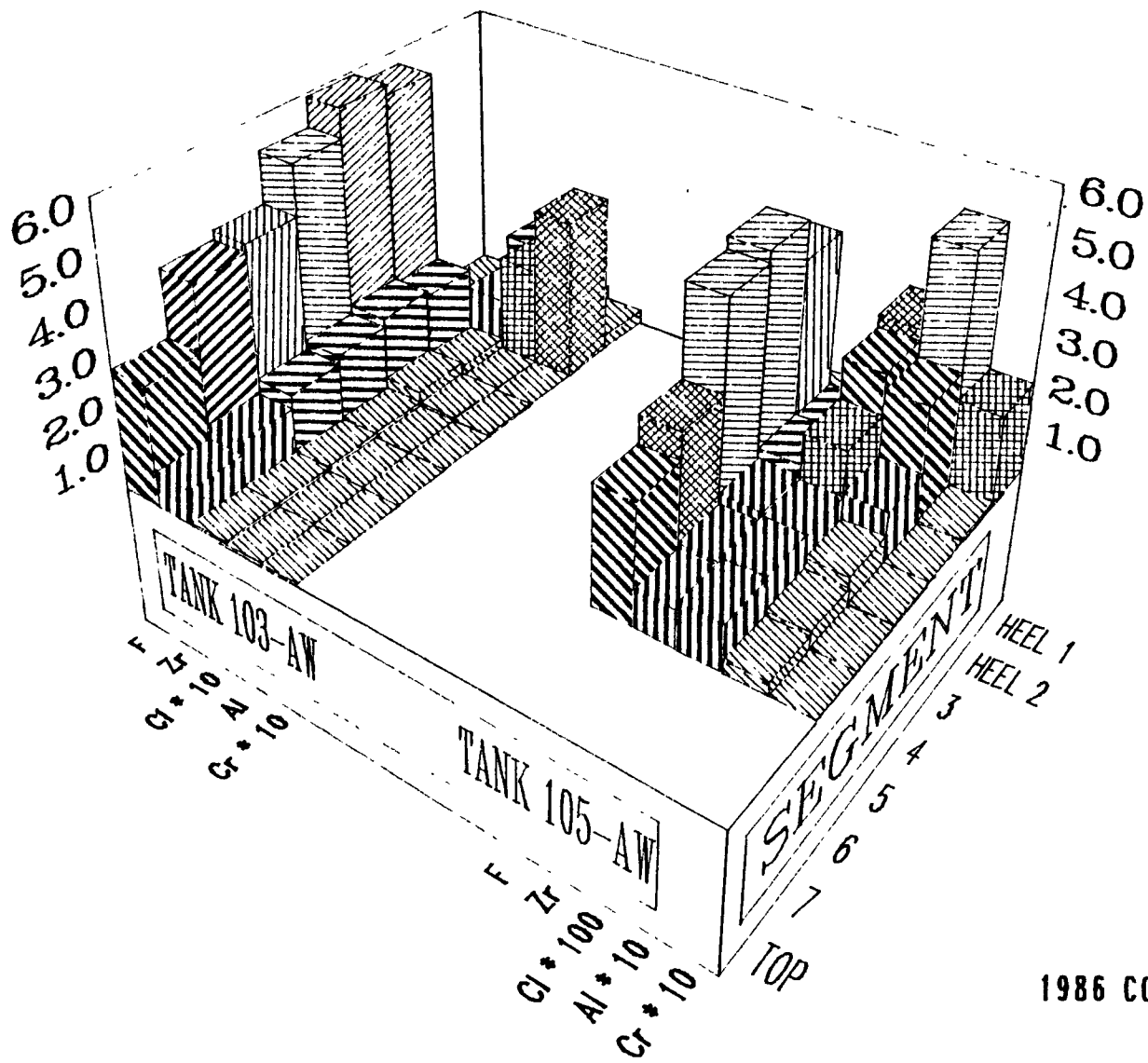
Table 1. Comparison of Undiluted Tank Waste Compositions.

Species	Concentration (M) NCRW, Tank 103-AW		Range of Average Concentration Values (M) for NCRW, PFP, & CC Wastes
	Maximum	Minimum	
Na	15.9	1.08	7.1 to 15.2
Al	2.35	0.0016	0.33 to 3.2
Zr	1.79	0.0013	1.1
Cr	0.38	1.8 E-05	0.047 to 0.46
Cr <sup>+6</sup>	--	--	0.044
K	0.59	0.056	0.049 to 0.11
B	0.12	2.8 E-04	0.021 to 0.041
Si	0.47	0.011	0.011 to 0.18
As	--	--	0.164
Ca	0.018	2.3 E-04	0.019
Fe	0.021	1.8 E-05	0.019 to 0.144
Mn	9.8 E-03	8.8 E-05	0.013 to 0.045
P	--	--	0.684
Se	--	--	0.162
F <sup>-</sup>	6.2	0.002	0.156 to 3.8
Cl <sup>-</sup>	0.18	0.004	0.034 to 0.36
CO <sub>3</sub> <sup>-2</sup>	0.26	0.02	0.094 to 1.05
NH <sub>3</sub>	0.021	0.005	0.057
NO <sub>2</sub> <sup>-</sup>	1.48	0.017	0.41 to 1.79
NO <sub>3</sub> <sup>-</sup>	0.14	0.043	0.65 to 5.85
OH <sup>-</sup>	1.95	0.57	0.18 to 1.8
PO <sub>4</sub> <sup>-3</sup>	--	--	0.035 to 0.864
SO <sub>4</sub> <sup>-2</sup>	0.014	0.000	0.074 to 0.144

Note: Radioactive species, not noted in the table, include <sup>95</sup>Nb, <sup>95</sup>Zr, <sup>106</sup>Ru, <sup>135</sup>Sb, <sup>134</sup>Cs, <sup>137</sup>Cs, <sup>144</sup>Ce, <sup>60</sup>Co, <sup>154</sup>Eu, <sup>155</sup>Eu, U and Pu.

CC = Complexant concentrate  
 NCRW = Neutralized cladding removal waste  
 PFP = Plutonium Finishing Plant

Figure 1. Variation of Composition as a Function of Tank Core Segment.



1986 CORE SAMPLES

The vertical segments of stratified waste in the NCRW waste tanks, from which the samples were removed for analysis, were generally around 19 in. long; several were as low as 4 to 12 in., depending on the location. In addition, the total depth assessed in tank 103-AW was 129 in. while tank 105-AW was 95 in., which caused some difference in the number of samples analyzed and plotted (see Figure 1).

That a corroding and a complexing species are out of desired registry can be seen for  $F^-$  and  $Al$ , in Figure 1. The  $F^-$  concentration in both tanks starts relatively high ( $-2.5M$ ), just below the top; progresses to 5 to  $6M$  around the middle; and virtually disappears (particularly in tank 103-AW) several increments above the tank bottom ( $-0.002M$ ). Meanwhile, the complexing species,  $Al$ , is relatively low ( $-0.002$  to  $0.0089M$ ) throughout much of the tank depth, becoming high only near the bottom ( $-2.35M$ ) where the need for complexing its corresponding ( $-0.002M$ )  $F^-$  is minimal.

The other, generally more abundant complexing species,  $Zr^{+4}$ , varies from about  $0.8M$  near the top to about 1 to  $1.5M$  in the middle and becomes very low ( $\sim 0.002M$ ) at the top and near the bottom. The  $Al/F$  ratio, which is important for reducing  $F^-$ /acid uniform corrosion rate in austenitic stainless steel varies from a low of about  $0.002M$  at the top and bottom of tank 103-AW [note that the  $(Zr+Al)/F$  ratio is also low  $-0.004M$  at the same point] to as high as  $0.33M$  near the middle. This relationship is shown in Figure 2 and tabulated in Tables 2 and 3. The average values are 0.26 and 0.29, respectively, for tanks 103-AW and 105-AW. Based on the composition fluctuation shown in Figure 1, the low  $Zr/F$  ratio as well as a low  $Al+Zr/F$  ratio, particularly at the top and near the bottom of tank 103-AW, will subsequently produce high corrosion rates in low Cr-Ni stainless steel embedded piping when the waste is acidified.

Based on experience at the Hanford Site and at the Idaho National Engineering Laboratory (INEL)-Westinghouse Idaho Nuclear Company (WINCO), for optimum uniform corrosion control of type 304L stainless steel, the  $(Al+Zr)/F$  or the  $Al/F$  ratio should exceed 0.5. For example, the uniform corrosion rate of type 304L stainless steel in a  $2M$   $HNO_3$ - $1M$  HF solution was reduced from about 55,200 mil/yr (boiling) to about 420 mil/yr by increasing the  $Al/F$  ratio from zero to 0.5. Correspondingly, an  $(Al+Zr)/F$  ratio of about 0.34 in an Al-Zr raffinate solution with  $1.2M$   $H^+$ - $3.8M$   $F^-$  reduced corrosion rates to about 110 mil/yr at 35 °C (Newby and Hoffman 1967). Figure 3 illustrates that to reduce rates to plant acceptable levels ( $<10$  mil/yr) a  $(Al+Zr)/F$  ratio  $>0.5$  is required (Newby and Hoffman 1967).

Figure 2. Variation of Zr+Al Concentration to F Concentration.

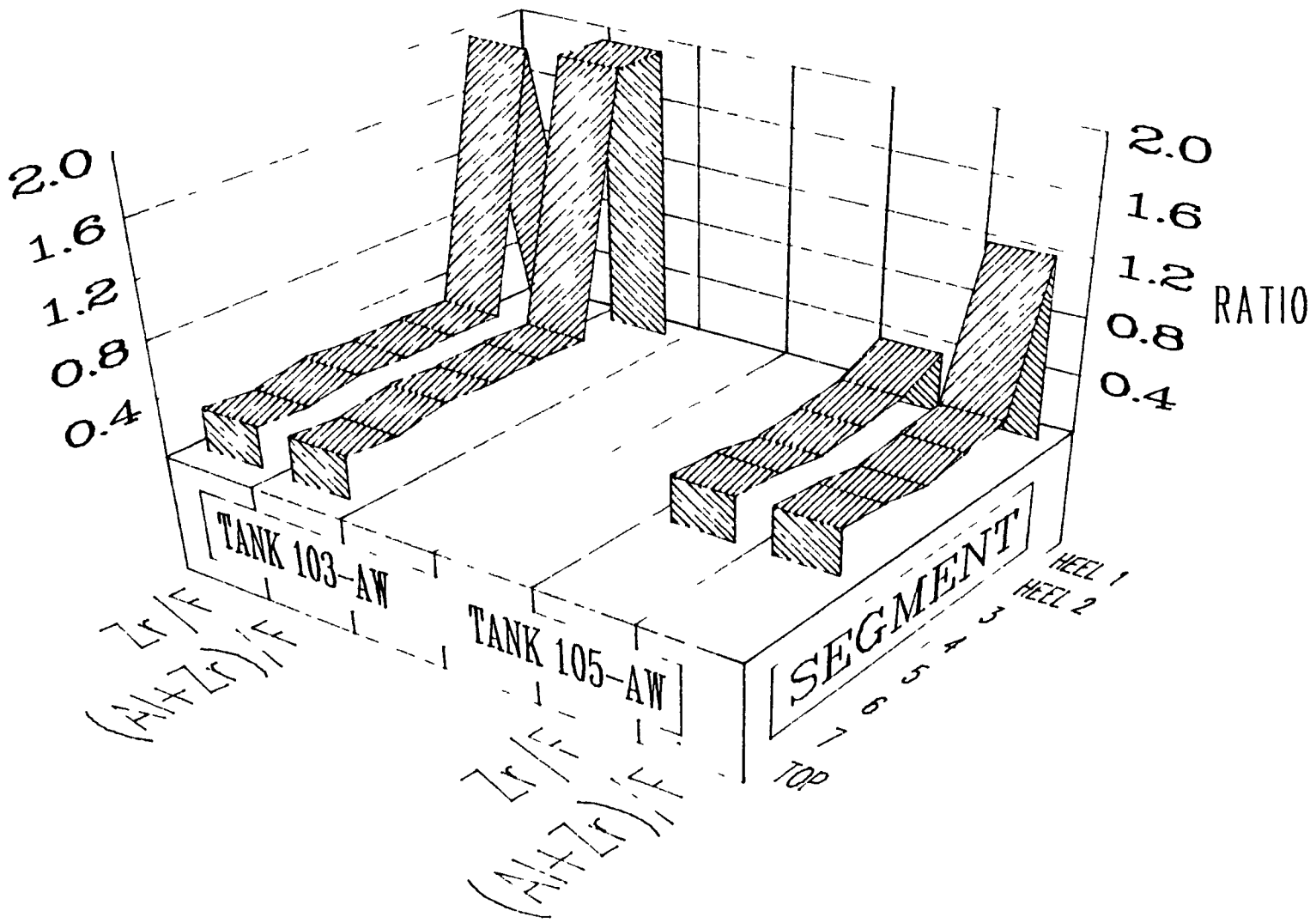


Figure 3. Corrosion Rate of Type 304L in HF-HNO<sub>3</sub> Solutions With and Without Al(NO<sub>3</sub>)<sub>3</sub>.

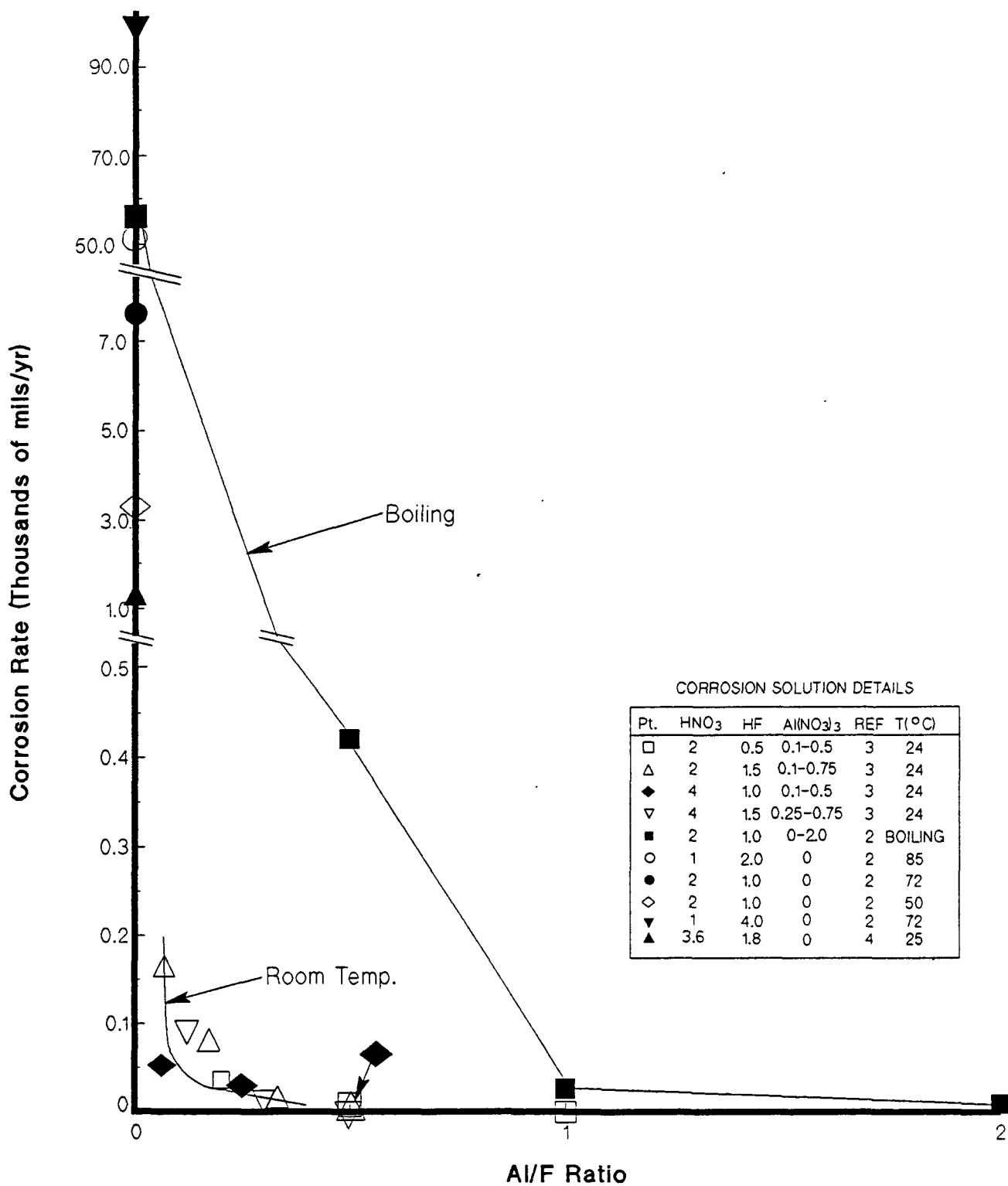




Table 2. Zr/F and (Al+Zr)/F Ratios in Tank 103-AW.

Species	Core Segment						
	7931 (supernate)	7927	7928	7929	7933	7930	7932
Al	0.0016	0.0167	0.0119	0.0179	0.0179	0.0259	0.0894
Zr	0.0013	0.749	1.02	1.29	1.36	1.49	1.55
F	0.737	2.568	4.042	4.358	4.067	5.305	5.648
Zr/F	0.0018	0.292	0.252	0.296	0.334	0.281	0.274
(Al+Zr)/F	0.004	0.298	0.255	0.300	0.339	0.286	0.290
Species	Core Segment						
	7934	7935	8022	7936	7948	7938 (bottom)	
Al	0.0861	0.377	0.0181	1.90	2.35	0.561	
Zr	1.77	1.42	1.55	0.0035	0.0182	0.0024	
F	6.189	4.863	5.686	0.002	0.002	0.123	
Zr/F	0.286	0.292	0.273	1.75	9.1	0.019	
(Al+Zr)/F	0.30	0.370	0.276	952	1,184	4.58	

Avg Zr/F = 0.236 (omitting 7936 and 7948)

Table 3. Zr/F and (Al+Zr)/F Ratios in Tank 105-AW.

Species	Core Segment					
	7937 (top)	7939	7940	7941	7942	7946 (bottom)
Al	0.0269	0.035	0.0567	0.109	0.250	0.498
Zr	0.782	0.880	1.08	1.28	1.24	0.24
F	2.525	3.105	5.043	5.289	4.982	0.587
Zr/F	0.310	0.283	0.214	0.242	0.277	0.409
(Al+Zr)/F	0.32	0.295	0.225	0.263	0.332	1.257

Avg Zr/F = 0.289

### 2.3 CORROSION EVALUATION OF "OLD" AND "NEW" ALLOYS

Current plans call for the pretreatment plant to use the approximately 40-yr-old Hanford Site B Plant. The selection of B Plant has caused inherent material problems in that the plant was not designed for a  $F^-/HNO_3$  process. Existing ("old") plant-embedded piping is composed of type 304L stainless steel, type 18-8 Cb stainless steel (now type 347 stainless steel), type 25-12 S Cb stainless steel (now 309S Cb), Hastelloy<sup>1</sup> B and Inconel<sup>2</sup> 625; these alloys, and one congener (e.g., 309 S for 309 S Cb) are being investigated for corrosion susceptibility.

Following evaluation of the "old" alloys, "new" alloys will be selected and corrosion-screened as candidate alloys for pilot plant component construction. A list of these alloys is discussed in the following text along with a planned method for corrosion-screening.

It is important to determine if plant-acceptable rates of corrosion can be attained through control of free HF to a presently undefined level by combining analytical chemical analyses and corrosion evaluations. An analytical evaluation of free HF will be done to assist in the selection of corrosion test solutions and, eventually, to use as a device for in-situ plant measurement of HF. A more detailed description of the development of a selective ion electrode (for HF assessment) along with an electrode for  $H^+$  evaluation will be discussed in another paper.

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<sup>1</sup>Hastelloy is a trademark of Haynes International, Incorporated, Kokomo, Indiana.

<sup>2</sup>Inconel is a trademark of The International Nickel Company, New York, New York.

### 3.0 PROCESS CHANGES THAT REDUCE CORROSION POTENTIAL

#### 3.1 PILOT PLANT OPERATING LIFE

The pilot plant is expected to be processing the corrosive NCRW solution for about 6 mo. If the average uniform corrosion rate is kept below 10 to 20 mil/yr using complexant additions, then corrosion product effects on downstream problems should be reduced also. The net effect is that corrosion rates in this level should not adversely effect the selection of B Plant for the pilot plant site.

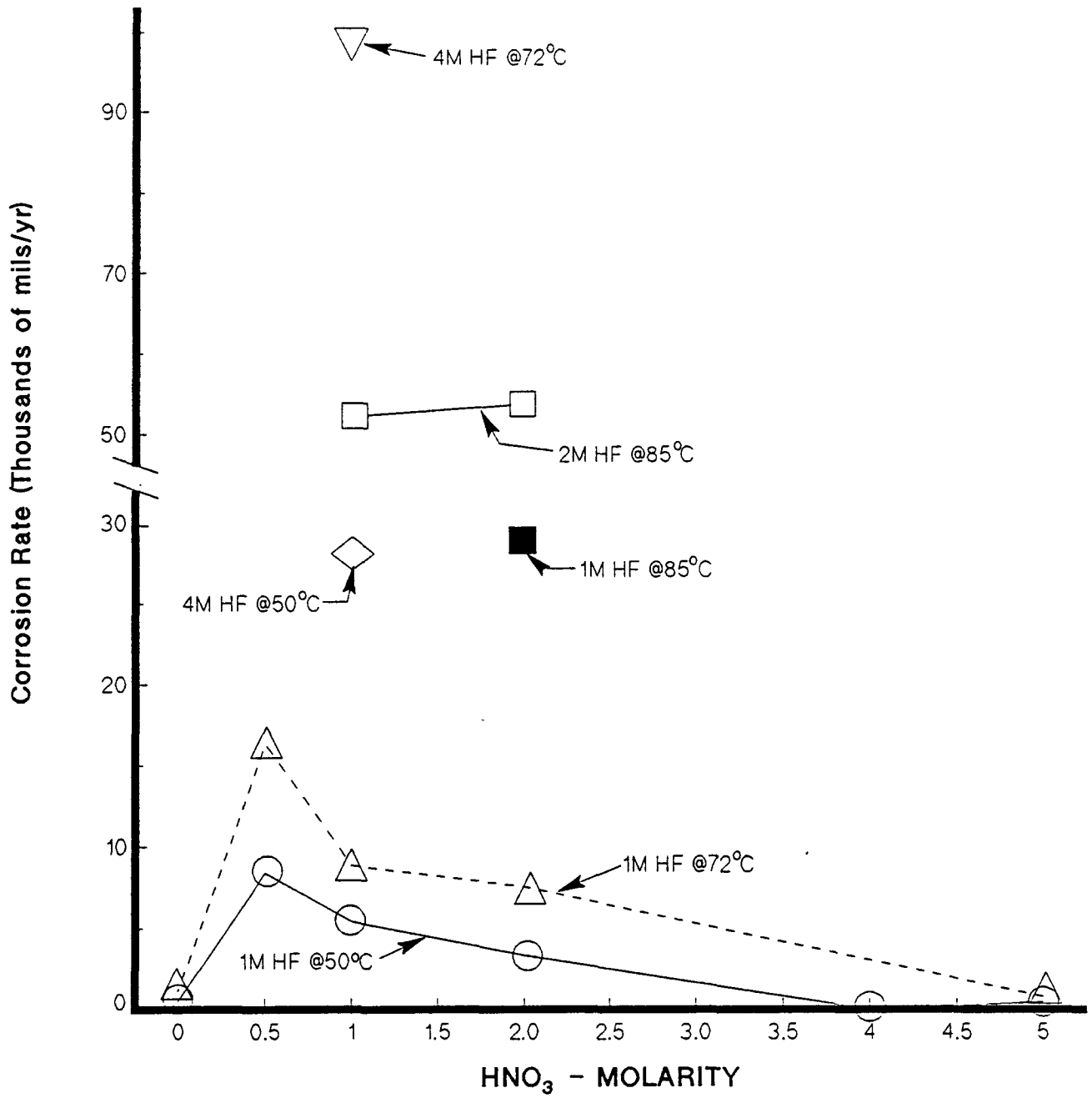
#### 3.2 RECOMMENDED PROCESS OPERATING CONDITIONS CHANGES

Literature indicated that several factors contribute to the corrosive behavior of  $\text{HNO}_3$ -HF solutions. These factors include exposure times, process temperatures, available  $\text{F}^-$  complexants, and  $\text{HNO}_3$  concentration. For example, as seen in Figure 4, the maximum corrosion rates in uncomplexed  $\text{HNO}_3$ -HF solutions occur at  $\text{HNO}_3$  concentrations of 0.5M. Based on a literature search of acidified corrosion test solutions that contained relatively few chemical components, the following recommendations for mitigation or reduction of corrosion in 304L stainless steel and other embedded piping are as follows:

- Total  $\text{F}^-$  concentration levels are maintained at 1M or less
- Process temperatures should be maintained less than 72 °C (preferably 25 °C); corrosion rates double for every 15 °C rise in temperature
- $\text{HNO}_3$  concentration is maintained at 1.5M or higher
- The Al/F ratio should be maintained between 0.25 and 0.5 to reduce free HF concentration
- Minimize solution residence time in embedded piping
- Quick response rinsing or flushing of these connectors and their contiguous components with water or an appropriately complexed solution after exposure to the process solutions.

From a corrosion viewpoint, the added effect of actual waste solutions containing other components can be either beneficial or detrimental.

Figure 4. Corrosion Rate of Type 304L Stainless Steel in HF-HNO<sub>3</sub> Solutions.



#### 4.0 EXPERIMENTAL PROGRAM

##### 4.1 SELECTION OF INITIAL CORROSION TEST SOLUTIONS

Corrosion testing of "old" and "new" alloys requires that solutions characteristic of the wastestreams be used. Because low levels of  $F^-$  complexing species Al and Zr will allow relatively high levels of HF to exist, it was decided that, at least in the beginning, that three NCRW-based solutions would be used for initial corrosion evaluation.

The three solutions selected were as follows:

- (A) 2M NaF-2M  $HNO_3$  -- Solution "A" was anticipated to be the worst-case condition. The NaF was used as the source of  $F^-$ , because the typical waste solutions generally contained relatively high amounts of Na.
- (B) "Near average" waste solution -- Solution "B" simulates NCRW waste solutions containing the two anticipated aggressive species,  $F^-$  and Cl, and three of the four complexing species, B, Si and Zr, and finally  $OH^-$ . The chemical makeup of this solution is presented in Table 4, except in Solution "B" there was no Al present. The solution was prepared with the NaOH added last, simulating the way in which the actual waste was prepared for storage in mild steel tanks at the Hanford Site.
- (C) "Average" waste solution + additional Al -- The same as Solution "B," except that the  $Al(NO_3)_3 \cdot 9H_2O$  concentration raised from 0.082 to 1.0M.

Other species as listed in Table 1 were omitted for the first tests. Reagent-grade chemicals were used to make all the solutions.

Table 4. Chemicals Used to Make a Simulated Average Neutralized Cladding Removal Waste [Diluted 3:1 w H<sub>2</sub>O].

Component	Molarity	Source Chemical
F <sup>-</sup>	0.950	NaF
Cl <sup>-</sup>	0.200	NaCl
Zr	0.275	ZrO(NO <sub>3</sub> ) <sub>2</sub> ·2H <sub>2</sub> O
Si	0.045	Na <sub>2</sub> SiO <sub>3</sub> ·9H <sub>2</sub> O
Al	0.082	Al(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O
B	0.010	H <sub>3</sub> BO <sub>3</sub>
OH <sup>-</sup>	0.263	NaOH

Both Solutions "B" and "C," after preparation, were acidified with HNO<sub>3</sub>, simulating the second step in the dilution and acidification to be done in the actual waste processing; acidification was halted after the H<sup>+</sup> level reached about 1.5M. Solution "B" was stored in Teflon<sup>1</sup> containers to minimize possible loss of free F<sup>-</sup> (or HF) from a reaction with a glass container. Solution "C" was stored in a glass container. A small portion of each solution was placed in a glass beaker with no apparent attack after about 60 h exposure. A fourth solution low in Zr but with an average Al content may be used later as a test solution.

After acidification, an aliquot sample will be returned to inert storage as a basis for comparing with later chemical analyses of post-test corrosion solutions. The H<sup>+</sup> levels are determined by titration. The HF levels will eventually be determined by a selective ion electrode process.

#### 4.2 CHEMICAL SPECIFICATIONS FOR "OLD" METAL CORROSION SPECIMENS

There are five different metal embedded piping systems in B Plant that could be used during the TRUEX pilot plant operation. They are as follows:

- Type 304L stainless steel
- Type 18-8 Cb (now denoted as 347) stainless steel
- Type 25-12S Cb (now denoted as 309S Cb) stainless steel
- Hastelloy B
- Inconel 625.

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<sup>1</sup>Teflon is a trademark of E. I. DuPont de Nemours, Company, Wilmington, Delaware.

Since B Plant was built in the 1940's, some of its alloys no longer exist with the same composition and exact replicates of some of these alloys are not readily available today. It is believed, however, that reasonable substitutes were obtained for the embedded piping alloys type 18-8 Cb, type 25-12S Cb, and Hastelloy B. Specifications to be used for purchase of "new" alloy specimens are detailed in Table 5.



Table 5. Chemical Analysis Specifications for B Plant Embedded Piping.

Type	Grade	C-O	Elemental Specifications												
			C	Cb	Ta	Co	Cr	Fe	Mn	Mo	Ni	P	Si	S	V
304L Stainless Steel	ASTM A240-88c	C	Standard Specification for Heat-Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet and Strip for Pressure Vessel, See Footnote 1												
347 Stainless Steel 18-8 Cb:	Grade 820A	O	0.10 max	10 x C content min to 1.0 max	--	--	18.0 min	--	1.25 to 2.5	--	10.5 min	0.035 max	0.75 max	0.03 max	--
	Grade 820c	O	0.10 max	10 x C content min to 1.0 max	--	--	17.0 min	--	2.5 max	--	9.5 min	0.04 max	0.75 max	0.04 max	--
	ASTM A240-88c	C	0.08	10 x C content min to 1.0 max	--	--	17.0 to 19.0	--	2.00 max	--	9.0 to 13.0	0.045 max	0.75 max	0.30 max	--
309 Cb Stainless Steel: 25-12 S Cb:	Grade 820B	O	0.07	8 x C content min to 1 max	--	--	22.0 min	--	2.5 max	--	12.0 min	0.04 max	0.75 max	0.04 max	--
	Grade 820D	O	0.10	8 x C content min to 1 max	--	--	22.0 min	--	2.5 max	--	12.0 min	0.04 max	0.04 max	0.04 max	--
	ASTM A240-88c	C	0.08 max	10 x C content min to 1.10 max	--	--	22.00 to 24.00	--	2.00 max	--	12.00 to 16.00	0.045 max	0.75 Max	0.03 max	--
Hastelloy <sup>2</sup> B	ASTM B333-84	C	Standard Specification for Nickel-Molybdenum Alloy Plate, Sheet, and Strip, See Footnote 3												
	Hastelloy B-2	C	0.02 max	--	--	1.00 max	1.00 max	2.0 max	1.00 max	26.0 to 30.0	remainder	0.04	0.10	0.03	--
	Hastelloy B	C	0.05 max	--	--	2.5 max	1.0 max	2.0 to 6.0	1.0 max	26.0 to 30.0	remainder	0.04 max	--	0.03 max	0.2 to 0.4
Inconel <sup>4</sup> 625	ASTM B443-84	C	Standard Specification for Nickel-Chromium-Molybdenum-Columbium Alloy (UNS No. 6625) Plate, Sheet, and Strip, See Footnote 5												

<sup>1</sup>ASTM, 1988, Standard Specification for Heat-Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet and Strip for Pressure Vessel, ASTM A240-88c, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>2</sup>Hastelloy is a trademark of Haynes International, Incorporated, Kokomo, Indiana.

<sup>3</sup>ASTM, 1984a, Standard Specification for Nickel-Molybdenum Alloy Plate, Sheet, and Strip, ASTM B333-84, American Society for Testing Materials, Philadelphia, Pennsylvania.

<sup>4</sup>Inconel is a trademark of The International Nickel Company, New York, New York.

<sup>5</sup>ASTM, 1984b, Standard Specification for Nickel-Chromium-Molybdenum-Columbium Alloy (UNS No. 6625) Plate, Sheet, and Strip, ASTM B443-84, American Society for Testing and Materials, Philadelphia, Pennsylvania.

C-O = Current Old

The 304L and Inconel 625 "old" alloys, specifications are believed to have not changed significantly since the various piping was purchased for B Plant. In this program, Hastelloy B-2 was substituted for the more difficult to obtain Hastelloy B; the compositions are similar but Hastelloy B-2 contains slightly less Fe and more Ni. Because they both contain low chromium (about 1%) neither alloy is expected to be particularly resistant to HNO<sub>3</sub> solutions.

Alloy 18-8 Cb, which was originally obtained through the old Hanford Site specifications, appears reasonably close to the present type 347 stainless steel specification. In contrast, 309 S appears to be the closest alloy to 25-12-S-Cb but contains no Cb. Type 309 Cb appears to be satisfactory but is classed as a nonstandard specification (AMS 1980), probably because its low production levels.

Specifications that were used for purchase for each of the above alloys are shown in Table 5, along with comparisons to "older" specifications, where available. Actual chemical specifications are shown only for the "old" alloys along with their more recent designations.

#### 4.3 CORROSION TEST SPECIMEN AND TEST PROCEDURE

Corrosion test specimens were all rectangular, 0.0625 x 0.5 x 1 in. with a 0.25 in. hole centered 0.25 in. from one end. One substitute "old" alloy, Hastelloy B-2, was obtained only in 5/64-in. thickness. The samples were laser cut with their edges cleaned by machining. Each sample was steel, stamped with the alloy type, and serially numbered for identification. They were also cleaned and prepared according to ASTM G1-88 (ASTM 1988b) and had a 120 grit equivalent surface finish. The samples were weighed to +/- 0.1 mg.

Specimens exposed to Solutions "A" and "B" utilized sealed Teflon containers. Solution volumes were maintained at, or more than, 125 mL/in<sup>2</sup> of exposed specimen. The specimens in Solution "A" were suspended with Teflon tape; those in Solution "B," by a glass rack. Three specimens of each material were maintained totally immersed (liquid phase specimens), three half immersed and half in the vapor phase (noted as interface specimens), and three suspended in the vapor (vapor phase specimens) above the respective solutions. Testing for crevice and pitting corrosion as well as condensate corrosion will be conducted later. Tests for sensitivity to stress-corrosion cracking is also being considered.

Testing procedure consisted of (a) weighing each coupon (to +/- 0.1 mg) before placing it in its environment and (b) exposing the coupons at test temperature +/- 2 °C for up to 120 d depending on their corrosion rate. Long-term tests will include monitoring of corrosion product metal ions, total F<sup>-</sup> and free F<sup>-</sup>, Cl<sup>-</sup>, and H<sup>+</sup> concentration. No solution aeration is planned at this time.

All corrosion testing procedures including specimen cleaning, chemicals, and fluids are also based on ASTM G1-88 (ASTM 1988b), and ASTM G31-72 (ASTM 1972) (reapproved in 1985).

#### 4.4 CHEMICAL SPECIFICATION FOR "NEW" ALLOY CORROSION SPECIMENS

The philosophy for selecting "new" alloys for plant component fabrication is two-fold: first, it is intended that the most corrosion-resistant, "most economic," and fabricable alloy(s) be selected. The term "most economic" could mean one of the most capital intensive but least expensive, when replacement or repair in a radioactive environment is considered overall. Second, if necessary, addition of more waste-borne complexants such as Al and/or Zr will be considered during plant operation, as a means of reducing plant corrosion rates.

Literature data indicate that alloys containing an increasing Ni+Cr content seem to trend toward lower corrosion rates in HF-HNO<sub>3</sub> solutions as shown in Figure 5. Other U.S. Department of Energy sites, WINCO and Westinghouse Savannah River Company (WSRC), recommend that midrange Ni+Cr alloys be considered if satisfactory uncomplexed solution corrosion rates could be obtained and if such alloys were resistant to localized modes of corrosion such as pitting and crevice corrosion, intergranular attack, and stress-corrosion cracking. With the previous information as a guide, a list of potential pilot plant alloys was made (Table 6) along with a systematic approach for preselecting candidate alloys for further screening tests. The screening corrosion tests are discussed later.

Figure 5. Metal Corrosion Rate Versus Ni and Cr Content in Alloy.

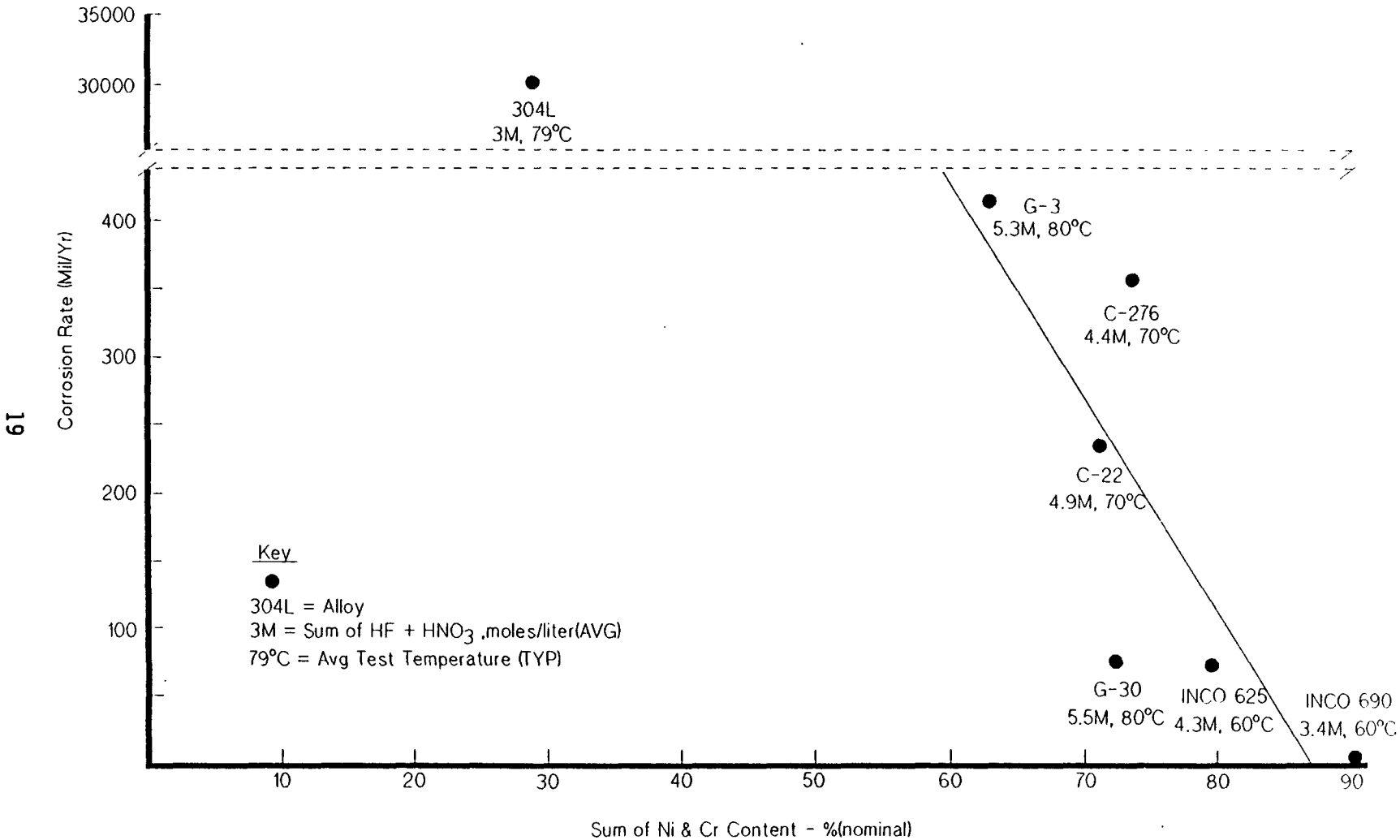


Table 6. List of Potential Transuranic Extraction Plant Alloys, Nominal Element Concentration(%).

No.	Alloy	Ni	Ni+Cr	Mo	Co	C
1	Ferrallium 255	5	31	2-4	0	0.04
2	Nitronic 50	12.5	34.5	1.5-3.0	0	0
3	Cabot 904L	26	47.5	4.5	0	0.02
4	Haynes 20 Mod.	26	48	4-6	0	0.05
5	Incoloy 801	32	52.5	0	0	0.10
6	330 SS	35.5	54	0	0	0.08
7	Carp. 20Cb <sub>3</sub>	35	55	2-3	0	0.07
8	Hastelloy <sup>a</sup> G-3	40	62.3	6-8	5.0	0.015
9	Incoloy 825	42	63.5	2.5-3.5	0	0.05
10	Hastelloy G	42	64.3	5.5-7.5	2.5	0.05
11	Hastelloy G-30	37.5	67	5.0	5.0	0.03
12	Hastelloy B-2	66.8	67.8	26-30	1.0	0.02
13	Hastelloy C-276	55	70.5	15-17	2.5	0.010
14	Hastelloy C	54	70	15-17	2.5	0.08
15	Hastelloy C-22	55	76.3	12.5-14.5	2.5	0.015
16	Hastelloy S	61	76.8	14-16.5	2.0	0.02
17	Inconel <sup>b</sup> 617	55	77	9	12.5	0.07
18	Hastelloy C-4	62	78	14-17	2.0	0.015
19	Cabot Alloy 625	58.8	79.5	8-10	1.0	0.10
20	Inconel 625	58	79.5	8-10	1.0	0.10
21	Inconel 690	60	90	0	0	0.05
22	Inconel 671	52	100	0	0	0.05

## Notes:

- (1) Ni and Ni+Cr are nominally the center of the allowable range.
- (2) Co or Mo are either shown as a range or a maximum.
- (3) C is the maximum allowable value.
- (4) Other alloying elements not listed here.

<sup>a</sup>Hastelloy is a trademark of Haynes International, Incorporated, Kokomo, Indiana.

<sup>b</sup>Inconel is a trademark of The International Nickel Company, New York, New York.

#### 4.5 CRITERIA FOR "NEW" ALLOY PRESELECTION

As noted previously, literature data and trends, cogent experience-based advice from other U.S. Department of Energy waste processing sites (WINCO and WSRC), and several conditions that generally reduce corrosion susceptibility are the primary bases for alloy preselection criteria. The preselection criteria are listed as follows.

- (1) Increasing Ni+Cr Content. Data in the literature show that increasing Ni+Cr content in a metal generally decreases uniform corrosion rate significantly (Figure 5) for HF-HNO<sub>3</sub> solutions. In addition, for optimum resistance to attack, Cr levels should be maintained at 14% or more; this level is generally the lower end of the Cr-range for some Hastelloy alloys.
- (2) WINCO and WSRC Experience. Based on their corrosion test and plant operation experience, both operations recommended that the selected alloys contain some Mo for pitting resistance. Concerning specific alloys, WINCO favored Hastelloy C-4 and C-22 for uncomplexed HF-HNO<sub>3</sub> solutions and Hastelloy G-30 (Norby 1988) for complexed solutions. The WSRC favored Hastelloy C-276; they also recommended medium-range Ni+Cr alloys such as type 330 stainless steel, if favorable plant complexing conditions could be assured and if satisfactory uncomplexed solution corrosion data were obtained.
- (3) Observations on Co. Many of the listed alloys (Table 6) contain Co in various amounts, and this should be considered as a criterion for selection. While the corrosion behavior of pure Co has not been as extensively documented as that of Ni, and generally possesses a lower overall corrosion resistance alone, with Cr, Co alloys become more noble. Thus, Co could have a beneficial effect in combination with both Ni and Cr.
- (4) C Level. Decreasing C level in some alloys is associated with a decrease in localized corrosion behavior. Thus, selection of candidate alloys with lowest C should be beneficial.
- (5) P and S Level. Decreasing P and S concentrations also lead to decreased localized corrosion because of the tendency for them to diffuse to and agglomerate in high concentrations at grain boundaries. Thus, alloys with lowest P and S, like those with the lowest C, could be beneficial.

Based on the previous statements the following preselection primary and secondary criteria were developed.

##### Primary Criteria:

- (1) Select alloys from a range of Ni+Cr contents (31% min to 100% max, Table 1).

- (2) Select Hastelloy C-4 and C-22 based on WINCO experience in uncomplexed solutions; G-30 in complexed solutions. Select Hastelloy C-276 based on WSRC experience.

Secondary Criteria:

- (3) Select alloys with a low and a relatively high Mo content.
- (4) Select alloys with a low and a relatively high Co content.
- (5) Eliminate alloys that are similar in alloy content (steps 1, 3 and 4) and also that are intermediate in Mo and Co levels.
- (6) The Cr level will equal or exceed 14% (lowest level noted for almost all alloys in Table 1) for optimum uniform corrosion resistance.
- (7) Select alloys with C levels less than or equal to 0.08 %.
- (8) Select alloys with lowest P and S content.

Based on the above criteria, 14 alloys were preselected for subsequent screening corrosion evaluation. They are listed in Table 7.

Table 7. Alloys Recommended for Purchase and Screening Corrosion Tests.

No.	Alloy	UNS No.	Applicable Specification
1	Ferralium <sup>a</sup> alloy 255	S32550	ASTM A240-88c <sup>b</sup>
2	Cabot <sup>c</sup> 904L	N08904	Cabot Brochure <sup>d</sup>
3	Haynes <sup>e</sup> 20 Mod	N08320	ASTM B620-84 <sup>f</sup>
4	Type 330 SS	N08330	ASTM B536-87 <sup>g</sup>
5	Carpenter <sup>h</sup> 20 Cb3	N08020	ASTM B463-84 <sup>i</sup>
6	Incoloy 825	N08825	ASTM B424-87 <sup>j</sup>
7	Hastelloy <sup>k</sup> G-30	N06030	ASTM B582-86a <sup>l</sup>
8	Hastelloy C-276	N10276	ASTM B575-86a <sup>m</sup>
9	Hastelloy C-22	N06022	ASTM B575-86a
10	Inconel <sup>n</sup> 617	N06617	None available; use Inconel Co. specification.
11	Hastelloy C-4	N06455	ASTM B575-86a
12	Inconel 625	N06625	ASTM B443-84 <sup>o</sup>
13	Inconel 690	N06690	ASTM B168-86 <sup>p</sup>
14	Inconel 671	None	None found; used ASM Vol III, p 155, for chem analysis; use Inconel Co. specification.

<sup>a</sup>Ferralium is trademarked by Langley Alloys Limited, Buckinghamshire, England.

<sup>b</sup>ASTM, 1988a, Standard Specification for Heat-Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels, ASTM A240-88c, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>c</sup>Cabot is trademarked by Cabot Corporation, Kokomo, Indiana.

<sup>d</sup>Cabot Wrought Products Division, Alloy 904L, Cabot Corporation, Kokomo, Indiana.

<sup>e</sup>Haynes is a trademark of Haynes International, Kokomo, Indiana.

<sup>f</sup>ASTM, 1984a, Standard Specification for Nickel-Iron-Chromium-Molybdenum Alloy (UNS N08320) Plate, Sheet, and Strip, ASTM B620-84, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>g</sup>ASTM, 1987a, Standard Specification for Nickel-Iron-Chromium-Silicon Alloys (UNS N08330 and N08332) Plate, Sheet, and Strip, ASTM B536-87, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>h</sup>Carpenter is trademarked by Carpenter Technology Corporation, Redding, Pennsylvania.

<sup>i</sup>ASTM, 1984b, Standard Specification for UNS N08020, UNS N08026, and UNS N08024 Alloy Plate, Sheet, and Strip, ASTM B463-84, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>j</sup>ASTM, 1987b, Standard Specification for Ni-Fe-Cr-Mo-Cu Alloy (UNS N08825 and UNS N08221) Plate, Sheet, and Strip, ASTM B424-87, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>k</sup>Hastelloy is a trademark of Haynes International, Kokomo, Indiana.

<sup>l</sup>ASTM, 1986a, Standard Specification for Nickel-Chromium-Iron-Molybdenum-Copper Alloy Plate, Sheet, and Strip, ASTM B582-86a, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>m</sup>ASTM, 1986b, Standard Specification for Low-Carbon Nickel-Molybdenum-Chromium Alloy Plate, Sheet, and Strip, ASTM B575-86a, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>n</sup>Inconel is a trademark of The International Nickel Company, New York, New York.

<sup>o</sup>ASTM, 1984c, Standard Specification for Nickel-Chromium-Molybdenum-Columbium Alloy (UNS N06625), Plate, Sheet, and Strip, ASTM B443-84, American Society for Testing and Materials, Philadelphia, Pennsylvania.

<sup>p</sup>ASTM, 1986c, Standard Specification for Nickel-Chromium-Iron Alloys (UNS N06600 and N06690) Sheet, Plate, and Strip, ASTM B168-86, American Society for Testing and Materials, Philadelphia, Pennsylvania.



#### 4.6 CORROSION SCREENING TESTS FOR "NEW" ALLOYS

The planned corrosion screening tests are centered around the use of uncomplexed and complexed HF-HNO<sub>3</sub> solutions containing selected chemical species. First screening tests are planned using at least two separate test solutions: a 2M NaF-2M HNO<sub>3</sub> solution and an "average" acidified (HNO<sub>3</sub>) NCRW solution low in one or both of the waste-borne complexing species, Al and Zr. The initial plan is to reject those alloys that exceed a uniform corrosion rate of >5 mil/d (1,765 mil/yr) for the first solution and >50 mil/yr for the "average," low complexant level acidified solution. For metals that pass the screening tests, testing will be continued using weldment specimens as the dominant specimen. Also, testing will be expanded to include assessment of crevice and pitting corrosion. Condensate corrosion tests are also planned but have not yet been defined. Intergranular attack and stress-corrosion sensitivity will be evaluated where susceptibility is suspected.

#### 5.0 EXPERIMENTAL RESULTS

Experimental results available thus far in the program cover a portion of the "old" alloy corrosion tests, mainly in Solution "A," and "B," and some in "C" (See Section 4.0 for description of solutions) and tested at room temperature. A tabulation of the corrosion rates for liquid, interface and vapor environments is shown in Table 8.

For Solution "A", the immersed specimens with the least Cr+Ni (type 304L and 347 stainless steel) corroded the most, about 1,800 and 1,200 mil/yr, respectively. Some increase was shown for the 304L interface specimens (2,200 mil/yr).

In Solution "B" the results differed significantly. Types 304L, 347 and 309 S stainless steel showed low corrosion rates for all three environments. However, 304L and 347 showed increased corrosion rates (-600 mil/yr) at 60 °C. Hastelloy B, an alloy with high Ni (-67%) and low Cr (-1%) showed the highest rate about, 2,200 mil/yr (liquid) and 1,700 mil/yr (interface). Inconel 625 showed no apparent corrosion or buildup.

In Solution "C" at 60 °C, Hastelloy B corroded excessively, about 13,700 mil/yr (average) while the four remaining alloys showed relatively little corrosion.

Table 8. Representative Corrosion Rates (mil/yr) of "Old" Alloys at Room Temperature in Liquid, Interface, and Vapor Phases.

Test Solutions	Test Environment	Metal Alloys				
		304L	347	309S	Hastelloy <sup>a</sup> B	Inconel <sup>b</sup> 625
Solution A - 25 °C	Liquid	1,840	1,200	176	6.4	0.5
	Interface	2,200	1,000	190	13.4	0.8
	Vapor	3.3 <sup>c</sup>	5.5	2.1	0.0	0.1 <sup>c</sup>
Solution A - 60 °C	Liquid	-	-	1,880	58.8	21.2
	Interface	-	-	2,463	77.5	21.1
	Vapor	-	-	10.4	77.3	3.3
Solution B - 25 °C	Liquid	0.63	1.4	0.2	2,200	0.0
	Interface	3.2	5.7	9.1	1,700	0.
	Vapor	0.0	0.0	0.0	35	0.0
Solution B - 60 °C	Liquid	551	681	-	-	-
	Interface	666	589	-	-	-
	Vapor	0.8	2.4	-	-	-
Solution C - 60 °C	Liquid	1.0	1.0	0.2	12,000	0.2
	Interface	1.6	1.9	0.1	15,300	0.2
	Vapor	0.2	0.3	0.2	1,000	0.1

<sup>a</sup>Hastelloy is a trademark of Haynes International, Incorporated, Kokomo, Indiana.

<sup>b</sup>Inconel is a trademark of The International Nickel Company, New York, New York.

<sup>c</sup>Calculated penetrations from weight gains assuming that the adherent corrosion product is Fe(NO<sub>3</sub>)<sub>2</sub>.

Additional testing at room and elevated temperatures (namely 60 °C) for extended testing times, up to 120 d and with other prototypic solutions are planned to more completely assess the corrosion behavior of "old" embedded piping alloys.

## 6.0 DISCUSSION

Because of the expected corrosivity of NCRW process streams, construction of the TRUEX pilot plant and subsequent full-scale plant requires that existing B Plant embedded piping alloys and "new" alloys for new components be made of highly corrosion resistant materials. In addition, assessment of the potential for monitoring free HF and H<sup>+</sup> concentrations as onstream monitoring techniques be examined.

Based on testing of the "old" or embedded piping alloys with three candidate solutions, Hastelloy B is unsatisfactory along with type 304L, 347 and 309 S stainless steels. In order for the latter three to be satisfactory at room temperature and 60 °C, complexing the process stream with Al and or Zr will be required to maintain low corrosion rates. Inconel 625 was the only alloy tested, which was satisfactory for both solutions at room temperature. Further work at proposed operating temperature levels, for longer times and in more prototypic test solutions, are necessary to more clearly grade the corrosion behavior of all the "old" alloys.

"New" alloys are planned to be selected that have a range of Ni+Cr contents with selected amounts of other critical elements such as Mo and Co and with limitations on P, S, and C; the latter should help minimize intergranular corrosion; Mo is believed to decrease pitting. The Co may be beneficial in a number of the alloys.

Specific alloys will be selected using screening corrosion tests to minimize the total number of "new" alloys that are further evaluated. After the screening corrosion tests are complete, further assessment of uniform and localized corrosion effects will be studied primarily using specimens that contain prototypical weldments.

## 7.0 SUMMARY

Westinghouse Hanford Company, with the assistance of Pacific Northwest Laboratory, is developing a TRUEX process for pretreating DST wastes at the Hanford Site. Because the wastes typically contain only small amounts radioactive transuranic elements, which require storage in the form of a borosilicate glass in a repository, the DST wastes will first be separated into nonTRU (low level) and TRU (high level) radioactive species. The nonTRU level wastes can then be directly cast in cementitious grout leaving only the much smaller volume TRU waste for more expensive final processing and storage in glass in the repository.

The bench-scale TRUEX process development is reaching a stage where a pilot plant design has begun for the construction of a facility in the existing B Plant. Because of the potential corrosivity of NCRW process streams, existing B Plant embedded piping alloys are being evaluated and "new" alloys are being selected for the full-scale plant screening corrosion tests. The high corrosivity conditions are brought about by stratification of the waste components in the tanks. In the NCRW tanks, this stratification or maldistribution occurs in the highly corrosive  $F^-$  species and the main  $F^-$  complexing species, Al and Zr, and other anions such as  $Cl^-$ . Once NCRW is acidified with  $HNO_3$ , some of the wastestreams that are simultaneously high in  $F^-$  and low in Al and Zr can produce corrosion rates exceeding 30,000 mil/yr in austenitic alloys. Thus, corrosion control through wastestream complexant additions and the use of more corrosion resistant alloys is imperative.

Work thus far, has been concentrated on evaluating the corrosion resistance of B Plant embedded piping alloys and specification and purchasing of "new" candidate alloys for initial screening corrosion tests.

Corrosion tests on "old" alloys have involved evaluation of uniform corrosion in liquid, interface, and vapor phases later to be followed by evaluations for crevice and pitting corrosion, condensate corrosion, and assessments of other localized corrosion modes such as intergranular attack and stress-corrosion cracking. Following the initial corrosion screening of "new" alloys, weldment specimens characteristic of pilot plant welds are expected to be the dominant specimen type. Erosion-corrosion may have to be considered depending on the abrasivity of the wastestreams.

Experimental work confirms that Hastelloy B is unsatisfactory for the first two waste solutions used; type 304L, 347 and 309 S stainless steels are satisfactory for service at room temperature and 60 °C, if wastestream complexing with Al or Zr is possible. Inconel 625 was satisfactory for both solutions. Further work at higher temperature levels (60 °C), additional test solutions, and for longer testing times are necessary to more clearly grade the corrosion service behavior of all the "old" alloys.

"New" candidate alloys were selected that display a range of Ni+Cr levels, several levels of Co and Mo and with limitations on C, P, and S. Screening corrosion tests are planned to further decrease the list of candidate alloys.

## 8.0 REFERENCES

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