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## Review of Corrosion Inhibition in High Level Radioactive Waste Tanks in the DOE Complex

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### ABSTRACT

Radioactive waste is stored in underground storage tanks at the Department of Energy (DOE) Savannah River Site (SRS). The waste tanks store supernatant liquid salts, consisting primarily of sodium nitrate, sodium nitrite, sodium hydroxide, and sludge. An assessment of the potential degradation mechanisms of the high level waste (HLW) tanks determined that nitrate-induced pitting corrosion and stress corrosion cracking were the two most significant degradation mechanisms. Controls on the solution chemistry (minimum nitrite and hydroxide concentrations) are in place to prevent the initiation and propagation of pitting and stress corrosion cracking in the tanks. These controls are based upon a series of experiments performed using simulated solutions on materials used for construction of the tanks. The technical bases and evolution of these controls is presented.

**Keywords** : high level waste tank, corrosion control

### INTRODUCTION

Radioactive waste is stored in underground storage tanks at the Department of Energy (DOE) Savannah River Site (SRS). The high level wastes will be processed in several of the tanks and then transferred by piping to other site facilities for further processing before they are stabilized for ultimate disposal. Based on waste removal and processing schedules, many of the tanks, including those with acceptable defects, will be required to be in service for times exceeding the initial intended life. Until the waste is removed from storage, transferred, and processed, the materials and structures of the tanks must maintain a confinement function by providing a barrier to the environment and by maintaining acceptable structural stability during design basis events (DBEs), which include loadings from both normal service and abnormal (e.g., earthquake) conditions. A corrosion control program is in place to maintain the structural integrity of these waste tanks throughout their intended service. The technical bases and the details of the corrosion control program are presented. A brief introduction to the waste tank design and fabrication is followed by a description of the solution chemistry controls, with the current issues and focus presented at the end.

## Waste Tank Design/Fabrication

The SRS has three types of double-shell tanks that are currently in service, identified as Types I, II, and III; they were not stress-relieved after construction. The Types I and II tanks were made of ASTM A285 steel during the 1950's and 1960's. The Type III tanks were made of ASTM A516/A537 steel during the 1960s and 1970s and have been stress relieved.

### Type I/II Tanks

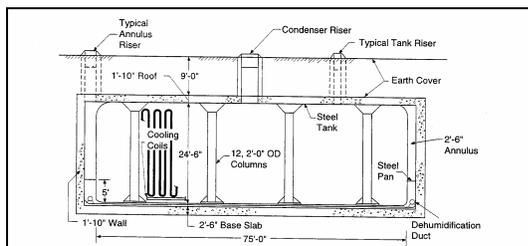
Type I and Type II waste tanks were made of ASTM Type A285-50T, Grade B steel, with the nominal composition shown in **Table 1**. The tanks were fabricated from semi-killed, hot-rolled plate material.

**Table 1:** ASTM Requirements for Chemical Composition for A285-50T, Grade B Firebox Quality

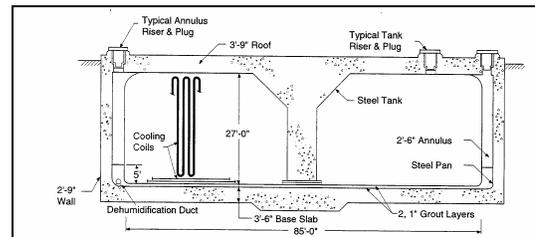
For plates $\leq 0.75$ " thickness	<u>Composition, %</u>			
	<b>C<sub>max</sub></b>	<b>Mn<sub>max</sub></b>	<b>P<sub>max</sub></b>	<b>S<sub>max</sub></b>
	0.2*	0.8	0.035	0.04

\*C = 0.22 wt.% for plate of  $0.75 < \text{thickness} \leq 2$ "

Type I tanks (shown in **Figure 1**) have a nominal capacity of 750,000 gallons, are 75 feet in diameter, and 24 ½ feet high. The primary tanks are a closed cylindrical tank with flat top and bottom constructed from ½ in. thick steel plate. The top and bottom are joined to the cylindrical sidewall by curved knuckle plates. Type II tanks (shown in **Figure 2**) have a capacity of 1,030,000 gallons, are 85 feet in diameter, and 27 feet high. The primary container for Type II tanks consists of two concentric steel cylinders assembled with a flat bottom and flat top forming a doughnut. The top and bottom are joined to the outer cylinder by rings of curved knuckle plates. Single-butt girth welds join each of the plates in both, Type I and Type II waste tanks. The tanks are constructed with a top weld to the top of the tank, middle welds between plates, and bottom welds to the bottom of the plate. A 5-foot high steel pan provides partial secondary containment for the tanks and a concrete vault encompassing the primary tank and the steel pan provides another barrier before waste can reach the ground. Neither Type I nor Type II waste tanks are stress relieved.



**Figure 1:** Type 1 High Level Waste Tank Schematic.



**Figure 2:** Type II High Level Waste Tank Schematic.

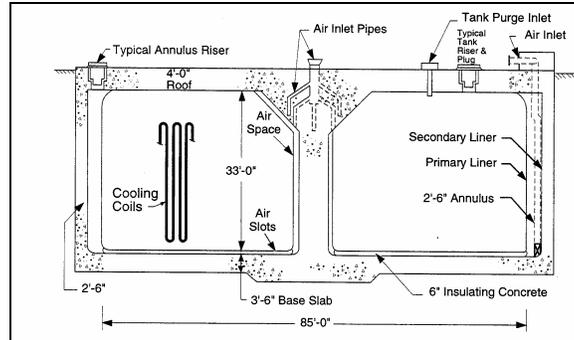
**Type III Tanks**

The most recently constructed tanks, designated Type III, were built from hot-rolled ASTM A516-Grade 70 or hot-rolled ASTM537-CI.1 normalized steel. The normalizing heat treatment (analogous to annealing) optimizes notch toughness and hence increases resistance to brittle fracture. The nominal compositions according to ASTM Standards are shown in **Table 2**.

**Table 1:** Nominal Compositions of A516-70 and A537-CI.1

Steel Specification	C <sub>max</sub> (wt%)		Mn <sub>max</sub> (wt%)	P <sub>max</sub> (wt%)	S <sub>max</sub> (wt%)
<b>A516 – Grade 70</b>	t = 0.5in.	0.27	0.6 – 0.9	0.035	0.035
	0.5 < t =0.2 in.	0.28	0.6 – 1.2	0.035	0.035
<b>A537 – Class 1</b>	0.24		t = 1.5in. 0.7 – 1.35	0.035	0.035

Each tank (as shown in **Figure 3**) is 85 feet in diameter and 33 feet high with a capacity of 1,300,000 gallons. Type III tanks have a toroidal shape similar to the Type II design. Each primary vessel is made of two concentric cylinders joined to washer-shaped top and bottom plates by curved knuckle plates. The secondary vessel is 90 feet in diameter and 33 feet high (i.e., the full height of the primary tank) and is made of 3/8 inch thick steel.



**Figure 3:** Type III High Level Waste Tank Schematic

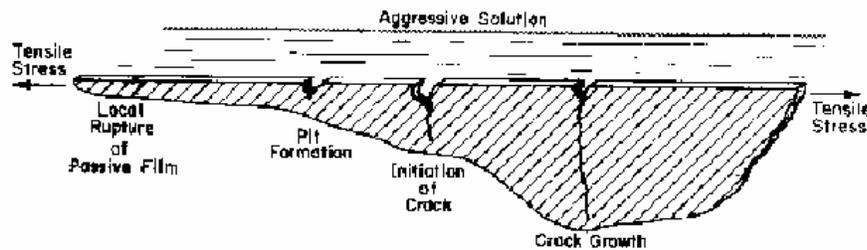
The primary tank sits on a 6-inch bed of insulating concrete within the secondary containment vessel. The concrete bed is grooved radially so that ventilating air can flow from the inner annulus to the outer annulus. Any liquid leaking from the tank bottom or center annulus wall would move through the slots and would be detected at the outer annulus.

The secondary vessel is 5 feet larger in diameter than the primary vessel, with an outer annulus 2-1/2 feet wide. The secondary vessel is made of 3/8-inch steel throughout. Its sidewalls rise to the full height of the primary tank. The nested two-vessel assembly is surrounded by a cylindrical reinforced concrete enclosure with a 30-inch wall. The enclosure has a 48-inch-flat-reinforced concrete roof, which is supported by the concrete wall, and a central column that fits within the inner cylinder of the secondary vessel.

Each primary tank was stress-relieved in place after all burning, cutting, welding, and other high-temperature work below the liquid fill line had been completed. Full stress relief at 1100°F was accomplished in accordance with the general requirements of the ASME Boiler and Pressure Vessel code.

### PRIMARY CORROSION MECHANISMS

The waste tanks store supernatant liquid salts, consisting primarily of sodium nitrate, sodium nitrite, and sodium hydroxide, and sludge. Nitrate-induced stress corrosion cracking was determined to be the principal degradation mechanism for the primary liner in Type I and II high level waste tanks through electrochemical studies and metallurgical evaluation of in-tank material. [1] Metallurgical evaluations were performed on a sample trephined from a tank with a known leak site. The analysis revealed that the intergranular stress corrosion cracking was typical of nitrate induced cracking on low carbon steel. A schematic of the mechanism for pitting or stress corrosion cracking is shown in **Figure 4**.

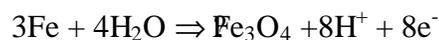


**Figure 4:** Schematic of Stress Corrosion Cracking Mechanism

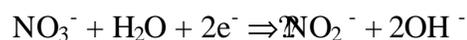
### Stress Corrosion Cracking

The waste solutions contain anions which can both cause or inhibit stress corrosion cracking. Nitrate or hydroxide may initiate SCC, however, the presence of either will inhibit cracking by the other. Nitrite which is present in the waste will also inhibit cracking. [2] Electrochemical polarization studies were performed to determine the root cause of the stress corrosion cracking. The studies showed that carbon steels are susceptible to nitrate SCC in potential ranges between -0.3 to 1.1 V (SCE), while hydroxide SCC occurs at potentials between -0.8 to -1.0V (SCE). [3] In addition hydroxide SCC only occurs at temperatures in excess of 100°C, whereas nitrate SCC may occur at lower temperatures. [4] Given that the open-circuit potential measured in several waste tanks is between -0.44 to -0.064 V and the temperature of the waste is generally less than 100°C, the observed cracking was determined to be caused by the nitrate. [5]

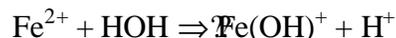
During corrosion in the nitrate solutions, carbon steel reacts anodically by:



and the cathodic reactions sum to:



Since the cathodic reaction takes place on the walls of the crack on the metal surface, and the anodic reaction takes place at the crack tip, the  $H^+$  and the  $OH^-$  do not react. However, the crack tip may become acidic due to the hydrolysis of the ferrous ion by the following reaction:



The acidic crack tip leads to a more anodic open circuit potential and stimulation of the cathodic reactions. These lead to more aggressive crack growth.

Cracks are initiated at carbon present in solid solution or as iron carbides ( $Fe_3C$ ) at the grain boundaries. The carbon acts as an efficient cathode with the adjacent metal surface as the anode. Cracks propagate along the grain boundaries of a material as the tensile stress maintains a crevice in which the solution remains aggressive towards the metal. The corrosion rate is influenced by the nitrate reduction kinetics and diffusion of reactants to cathodic sites. [6] Nitrate is more easily reduced as the solution becomes more acidic. Work at the Naval Research Laboratory has shown that during cracking the solution at the crack tip has approximately a pH of 3. [7]

The propagation of nitrate stress corrosion cracking in mild steel can be controlled by the anodic dissolution rate at the crack tip or the time necessary for the oxide film to rupture. The controlling mechanism was determined by measuring the activation energies for continuous crack growth (typical of anodic dissolution) and discontinuous crack growth (typical of oxide film rupture or crack growth by fracture). The activation energy for nitrate SCC is reported to be 27 kJ/mol for continuous crack growth, as measured in wedge opening loaded (WOL) specimens, and 56 kJ/mol for discontinuous crack growth as measured with compact tension specimens (CT). [8] Typically, the initiation time for continuous crack growth can be controlled by the time it takes for formation of oxides along the grain boundaries, or the crack tip chemistry to become aggressive. The activation energy for discontinuous crack growth is typical of oxygen diffusion in grain boundaries or the oxide-metal interface. Ultimately, the activation energies indicate that nitrate SCC is controlled by anodic dissolution in specimens with decreasing stress intensity, and by fracture of grain boundary oxide in specimens with increasing stress intensity.

### **Pitting**

The electrochemical reactions are much the same as for stress corrosion cracking, in the absence of the associated tensile stress. However, pitting is considered to be autocatalytic in nature, and may propagate through-wall if uninhibited. [9] Once a pit starts to grow, the conditions developed are such that further pit growth is promoted. The anodic and cathodic reactions are separated spatially. The local pit environment becomes enriched in metal cations when the cathodic reactant is depleted. These metal cations attract negative anions (e.g.  $Cl^-$ ,  $NO_3^-$ ) to maintain charge neutrality. The pH within the pit is lower due to hydrolysis and the absence of the local cathodic reaction:



This acidic environment creates a more aggressive situation that is self-propagating.

### **SOLUTION CHEMISTRY CONTROLS**

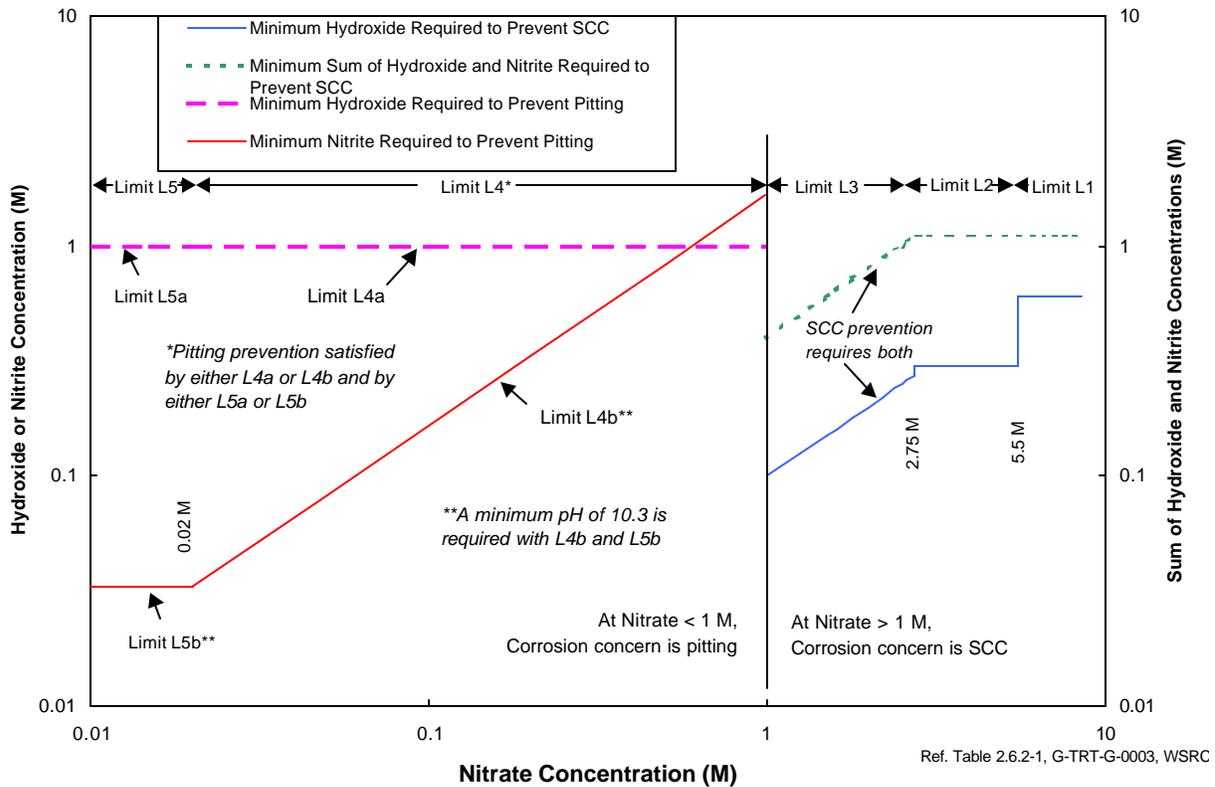
Sodium hydroxide and nitrite are used to inhibit nitrate-induced stress corrosion cracking and nitrate-induced pitting corrosion in the liquid phase of the waste tanks. General corrosion is also prevented by the inhibitor levels specified for these two localized corrosion modes. The addition of hydroxide and nitrite keep the pH above the vulnerable regime and also inhibit the cathodic reaction. The corrosion control program is based upon empirical data used to determine vulnerability to stress corrosion cracking and pitting at typical compositions of waste. A summary of the corrosion controls and a technical basis for each limit is presented in the following sections.

The inhibitor concentration limits are organized and listed in **Table 3** by nitrate concentration range. The hydroxide and nitrite concentration limits address nitrate-induced corrosion in the concentration range 0.02 M to 8.5 M nitrate in five steps, labeled L1 to L5, as shown in **Figure 5**. Limits L1, L2, and L3 cover the range 1 M to 8.5 M and specify the minimum hydroxide concentration and the minimum sum of the hydroxide and nitrite concentrations that are required to prevent stress corrosion cracking. Limits L4 and L5 cover nitrate concentrations below 1 M.

**Table 2: Corrosion Control Limits for the HLW Tanks**

**Minimum Corrosion-Inhibitor Concentration Limits**

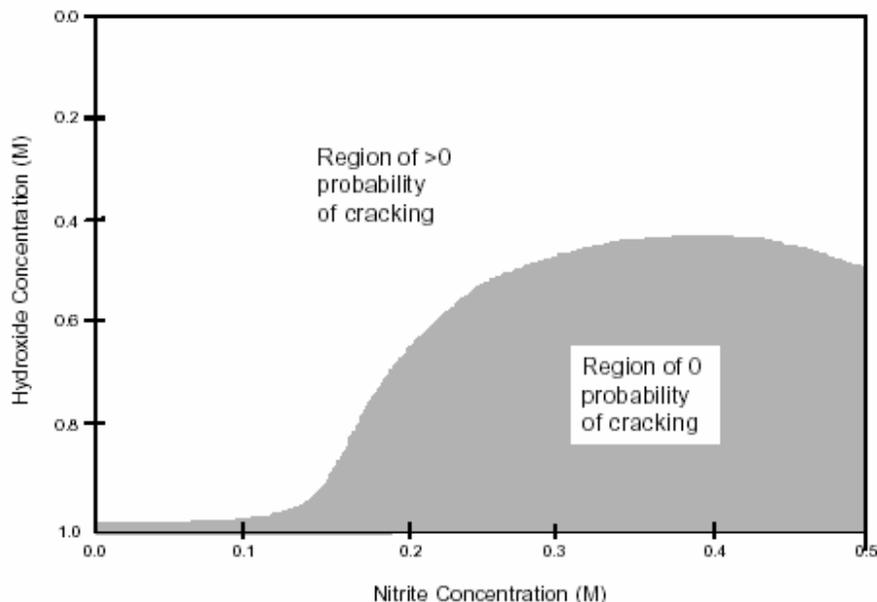
<u>Applicability</u>	<u>Parameter</u>	<u>Minimum Needed</u>	<u>Units</u>
L1. $5.5 < [\text{NO}_3^-] \leq 8.5$ Molar	$[\text{OH}^-]$	0.6	Molar
	$[\text{OH}^-] + [\text{NO}_2^-]$	1.1	Molar
L2. $2.75 < [\text{NO}_3^-] \leq 5.5$ Molar	$[\text{OH}^-]$	0.3	Molar
	$[\text{OH}^-] + [\text{NO}_2^-]$	1.1	Molar
L3. $1.0 \leq [\text{NO}_3^-] \leq 2.75$ Molar	$[\text{OH}^-]$	$0.1[\text{NO}_3^-]$	Molar
	$[\text{OH}^-] + [\text{NO}_2^-]$	$0.4[\text{NO}_3^-]$	Molar
L4. $0.02 < [\text{NO}_3^-] < 1.0$ Molar AND $[\text{OH}^-] < 1.0$ Molar			
L4a. – Either Inhibit with $[\text{OH}^-]$ OR Inhibit with $[\text{NO}_2^-]$	$[\text{OH}^-]$	1.0	Molar
L4b. For $[\text{NO}_3^-]$	$[\text{NO}_2^-]$	$0.038x[\text{NO}_3^-]x10^{1.64}$	Molar
L4c. For $[\text{Cl}^-]$	$[\text{NO}_2^-]$	$6.11x10^{1.64+1.34x\log[\text{Cl}^-]}$	Molar
L4d. For $[\text{SO}_4^{=}]$	$[\text{NO}_2^-]$	$0.04x10^{1.64+0.84 \log[\text{SO}_4^{=}]}$	Molar
	pH	10.3	pH Units
L5. $[\text{NO}_3^-] \leq 0.02$ Molar AND $[\text{OH}^-] < 1.0$ Molar			
L5a. – Either Inhibit with $[\text{OH}^-]$ OR Inhibit with $[\text{NO}_2^-]$	$[\text{OH}^-]$	1.0	Molar
L5b. For $[\text{NO}_3^-]$	$[\text{NO}_2^-]$	$0.00076x10^{1.64}$	Molar
L5c. For $[\text{Cl}^-]$	$[\text{NO}_2^-]$	$6.11x10^{1.64+1.34x\log[\text{Cl}^-]}$	Molar
L5d. For $[\text{SO}_4^{=}]$	$[\text{NO}_2^-]$	$0.04x10^{1.64+0.84 \log[\text{SO}_4^{=}]}$	Molar
	pH	10.3	pH Units
L6. Influent to waste tanks from other areas on site. *	pH	9.5	pH Units



**Figure 5: Corrosion Control Limits as a Function of Nitrate Concentration**

**Limit L1: 5.5 M < [NO<sub>3</sub><sup>-</sup>] ≤ 8.5 M**

Limit L1 defines inhibitor requirements to prevent stress corrosion cracking and is based upon experimental results of crack propagation tests with wedge-opening-loaded (WOL) specimens of A285 Grade B carbon steel in the temperature range of 35°C to 75°C and the nitrate concentration range 5.5 to 8.5 M. [10] The dependent variable in these tests was the presence or absence of growth of a pre-formed crack after the immersion of the specimen in the test environment. The results were statistically analyzed to produce a plot of the probability of crack growth as a function of the hydroxide and nitrite concentrations. The probability of crack growth was found to be independent of the temperature and the nitrate concentration over the range studied. **Figure 6** shows the graph of the probability of crack growth, from 0 to 1, at the given nitrite and hydroxide concentrations. The region of zero-probability of crack growth in the lower right portion of the graph is the desired operating region, and it is conservatively bounded by a minimum of 0.6 M hydroxide and a minimum of 0.5 M nitrite. Increasing the hydroxide concentration above 0.6 M allows the decreasing of the nitrite concentration while maintaining zero-probability, such that a minimum hydroxide and nitrite concentration sum of 1.1 M is specified, as shown in **Figure 1**. A maximum temperature of 70°C is applied to Limit L1.



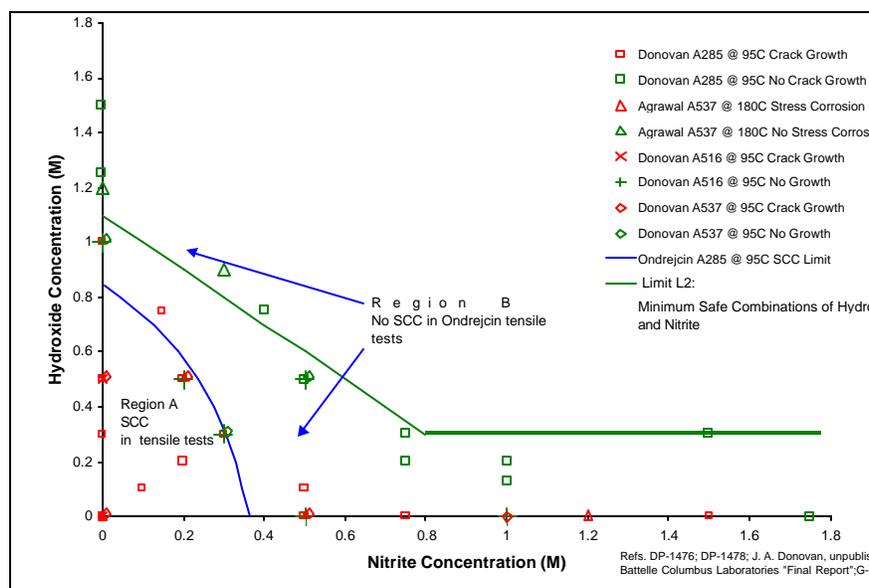
**Figure 6:** Probability of crack growth in WOL tests of A285 Grade B steel [10]

### Limit L2: 2.75 M $\leq$ $[\text{NO}_3^-] \leq$ 5.5M

Limit L2 also specifies the nitrate concentration ranges encountered in on-going waste receipt, storage, and evaporation operations. Wastes subject to L2 are typically those that are aged or partially evaporated. The primary corrosion mechanism identified in this chemistry range is nitrate-induced stress corrosion cracking (SCC). Pitting has not been observed in wastes containing  $> 1$  M nitrate, because these wastes contain high hydroxide concentrations, which prevent this form of localized corrosion. Limit L2 identifies the inhibitor requirements to prevent SCC and specifies a minimum hydroxide concentration of 0.3 M along with a minimum sum of the hydroxide and nitrite concentrations of 1.1 M for nitrate in the range 2.75 to 5.5 M.

Limit L2 is based on the results of slow-strain-rate tensile tests in the range 1.5 to 5.5 M nitrate and on WOL tests at 5 M nitrate, both on A285 Grade B carbon steel. [11] **Figure 7** shows WOL data points, representing the presence or absence of crack growth from A285 tests, along with supporting data from newer waste tank steels A516 and A537 used in the SRS Type III and IIIA tanks and data from a Battelle Columbus Laboratories study. [12] **Figure 7** also shows a curve representing the hydroxide and nitrite concentrations that satisfy the criterion for the onset of stress corrosion cracking, as determined by the slow-strain-rate tensile (SSRT) tests.

The SSRT tests were concluded to reveal the presence of stress corrosion cracking when the specimen's total elongation to failure fell below 13%. The elongation to failure was modeled as a second-order polynomial in the nitrate, nitrite, and hydroxide concentrations and the temperature. **Figure 7** shows the combinations of hydroxide and nitrite concentrations that solve this polynomial at 95°C at 13% elongation. Limit L2 carries a maximum temperature of 105°C, or 112°C if the sum of hydroxide and nitrite concentrations exceeds twice the nitrate concentration.



**Figure 6:** Corrosion Control Limit L2 with Supporting SCC Data for ASTM A285, A516, A537 Steels in 5M Sodium Nitrate.

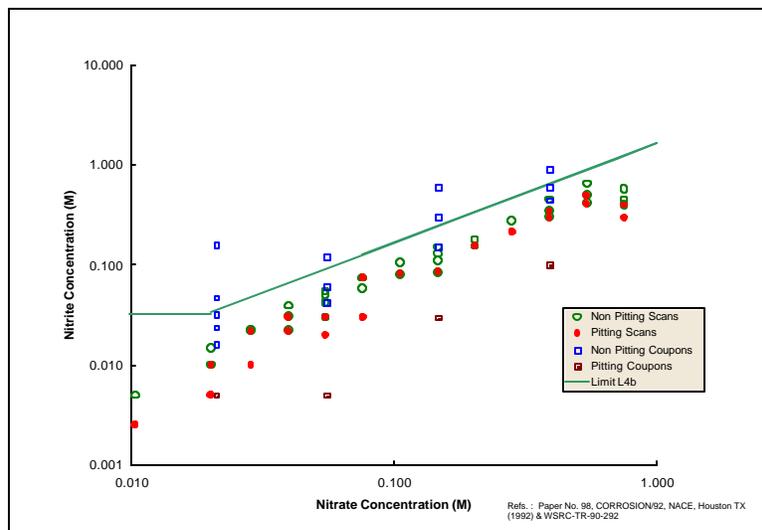
### Limit L3: $1 \text{ M} \leq [\text{NO}_3^-] \leq 2.75 \text{ M}$

Limit L3 addresses the nitrate range that is typical of fresh waste, historically received from nuclear reprocessing operations at SRS. The limit is specified to prevent nitrate-induced stress corrosion cracking. The Limit L3 is based on an engineering judgment, historical experience of waste tank corrosion prevention, and the previously mentioned SSRT test data. A minimum hydroxide concentration of 0.1 M was conservatively selected to maintain inhibiting conditions in 1M nitrate waste solutions. The minimum hydroxide concentration and the minimum sum of hydroxide and nitrite in L3 over the range 1 M nitrate to 2.75 M nitrate were selected to transition smoothly to the L2 limit at 2.75 M nitrate (see **Figure 5**). Limit L3 carries a maximum temperature of 70°C, or 105°C if the sum of hydroxide and nitrite concentrations exceeds twice the nitrate concentration.

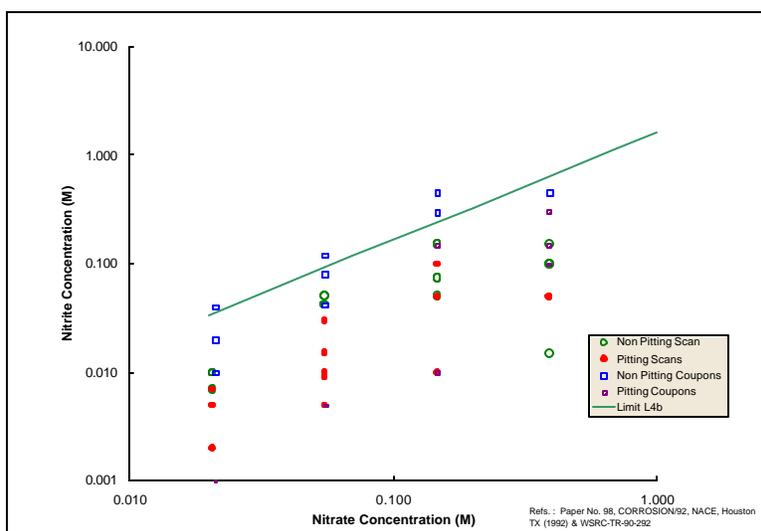
### Limit L4: $0.02 \text{ M} < [\text{NO}_3^-] < 1.0 \text{ M}$ and Limit L5: $[\text{NO}_3^-] < 0.02 \text{ M}$

Limits L4 and L5 apply to dilute waste solutions, i.e. solutions with a nitrate concentration  $< 1\text{M}$ , where nitrate-induced pitting, chloride-induced pitting, and sulfate-induced pitting are the corrosion mechanisms of concern. Pitting has not been observed in wastes containing  $> 1 \text{ M}$  nitrate, because these wastes contain high hydroxide concentrations, which prevent this form of localized corrosion. Stress corrosion cracking is not a concern in waste solutions with  $< 1 \text{ M}$  nitrate, based on the historical experience of SRS waste tanks. Tanks subject to the L4 and L5 limits are those that prepare waste for transfer to the waste vitrification facility and those that store dilute low-heat wastes. Pitting may be prevented by either a minimum hydroxide concentration or by a minimum nitrite concentration along with a minimum pH. Limit L4a specifies the minimum hydroxide concentration of 1 M and is based on the historical experience of the absence of pitting at  $\geq 1 \text{ M}$  hydroxide concentration. Limit L4b specifies the minimum nitrite concentration required to prevent pitting over the 0.02 to 1 M nitrate concentration range at 40°C.

Limit L4 is based on the results of electrochemical polarization scans and coupon immersion tests. [13,14] A least squares fit of the relevant nitrate concentration and test temperature to the minimum nitrite concentration established by the polarization scans at temperatures of 23, 30, 40, 50, and 60°C forms the basis. **Figure 8 and Figure 9** show the data at 40°C and 30°C with the L4b limit. L4b incorporates a safety factor of 1.5 on the required nitrite concentration that was selected based on engineering judgment. Nitrite inhibition is allowed by Limit L4b (and L5b) only up to 40°C; higher temperatures require the application of L4a (and L5a), to which a maximum temperature of 100°C applies. The addition of the L5 limits sets a lower bound on the required nitrite concentration independent of the nitrate concentration. Limits L4 and L5 also contain experimentally determined minimum nitrite limits for chloride and sulfate, which are also known to be able to induce pitting in carbon steel. [15] In the event that the nitrate anion is in low concentration and is not the principal corrosive anion, minimum nitrite levels can be calculated based upon the chloride or sulfate concentrations. The greatest nitrite concentration based on nitrate, chloride, or sulfate becomes the controlling limit.



**Figure 7:** Corrosion Control Limits L4b and L5b with Supporting Data at 40°C.



**Figure 8:** Corrosion Control Limits L4b and L5b with Supporting Data at 30°C.

## CONCLUSIONS

The Savannah River Site and Hanford sites within the DOE complex store high level alkaline radioactive waste in large underground storage tanks, made of low carbon steel. A corrosion control program is in place at each of the sites to prevent nitrate induced stress corrosion cracking and pitting, known to be the primary degradation mechanisms of these tanks. Hydroxide and nitrite are used to inhibit stress corrosion cracking and pitting corrosion in the liquid phase of the waste. General corrosion is also prevented by the inhibitor levels specified for these two localized corrosion modes. The chemistry control program continues to evolve as corrosion issues continue to arise, particularly during the waste removal and clean-up operations.

## CURRENT ISSUES

Although, these solution chemistry controls are in place, recent experience has shown that steel not in contact with the bulk waste solution or slurry, but exposed to the “vapor space” above the bulk waste, may be vulnerable to the initiation and propagation of corrosion, including pitting and stress corrosion cracking. Previous research has also determined that degradation is possible at the liquid-vapor interface due to hydroxide depletion.

Scoping experimentation has been performed to determine the potential for vapor space corrosion and liquid/air interface corrosion in ASTM A285 and A537 steels. Corrosion studies were performed with flat, unstressed coupons in nitrate, nitrite, and hydroxide solutions. The results suggested that the potential for vapor space and liquid/air interface general corrosion and pitting exists even with inhibited solution chemistry. [16] Further experimentation is planned to quantify the magnitude of the corrosion and also to determine the potential for stress corrosion cracking under similar conditions. Once these parameters are established, the corrosion control program will be modified to inhibit further degradation.

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