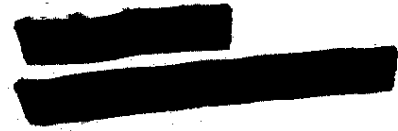


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# INVESTIGATION OF COOLING COIL CORROSION IN RADIOACTIVE-WASTE STORAGE TANKS

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PREPARED FOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION UNDER CONTRACT AT(07-2) 1

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# **INVESTIGATION OF COOLING COIL CORROSION IN RADIOACTIVE-WASTE STORAGE TANKS**

by

**R. S. ONDREJCIN**

Approved by

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## **ABSTRACT**

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The high frequency of cooling coil leaks observed in high-heat waste storage tanks soon after sludge removal operations is attributed to pitting, according to laboratory corrosion studies. Experimental evidence showed that the most likely series of events leading to coil leakage was 1) excessive dilution of basic nitrite in the waste, 2) initiation of attack in crevices due to oxygen depletion cells, and 3) acceleration of the attack by sulfate dissolved from the sludge.

When sludge was slurried with water, the interstitial liquid was diluted. Nitrite, the anodic inhibitor that prevented attack on coils and tanks in normal operation when its concentration was 0.5 to 3.0M, could accelerate attack if it was diluted to  $10^{-4}$  to  $10^{-3}$ M. Attack was presumably initiated at oxygen depletion cells. The presence of sulfate, which was leached from the sludge, produced a conductive solution that could produce high current densities at the corroding steel surface.

The proposed series of events leading to coil leakage agrees with the observations previously made on one leaking coil that was removed from Tank 2F after sludge removal in 1967. Examination revealed pitting that had originated on the outside of the coils. This pitting was attributed to oxygen depletion cells in coil crevices. This type of attack is not likely to affect the tank walls because of the absence of crevices in the walls. Leaks produced by pitting attack have not been observed in tank walls.

To prevent recurrence of the pitting attack on cooling coils during future sludge removal operations, the sludge should be slurried 1) with waste diluted by no more than one hundredfold with water, or 2) with a 500-ppm nitrite- $H_2O$  solution at pH 12. Either solution should preclude pitting damage to the coils.

## **CONTENTS**

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Introduction	5
Background	5
Waste Storage Operations	5
Description of Cooling Coils	7
History of Cooling Coil Leakage	7
Experimental Results	12
Miniature Cooling Coil Tests	12
Polarization Studies	14
Additional Pitting Experiments	16
Pitting Mechanism and Prevention	18
Acknowledgment	20
References	21

## LIST OF FIGURES

- 1 HHW Storage Tank [Type I, 750,000-gal  
( $2.84 \times 10^6$ -liter) Capacity] 6
- 2 Incidence of Cooling Coil Leaks in  
Tanks 1F-8F, 9H, and 11H-16H 9
- 3 Perforated Upper Horizontal Cooling Coil,  
Tank 2F 10
- 4 Pits Beneath Loose Porous Material on Cooling Coil 11
- 5 Microstructure of Cooling Coil Under Porous  
Material 12
- 6 Polarization Curves of Steel in Dilute  
(ten-thousandfold) Synthetic Tank 11H Waste  
With and Without  $\text{CaSO}_4$  15
- 7 Pitting and Prevention of Pitting in A 106  
Mild Steel 17
- 8 Typical Crevices Formed by Cooling Coil Supports  
in Tanks 1F-8F and 9H-12H 19

## LIST OF TABLES

- 1 HHW Tank Cooling Coil Failure History 8

## **INVESTIGATION OF COOLING COIL CORROSION IN RADIOACTIVE-WASTE STORAGE TANKS**

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### **INTRODUCTION**

This report summarizes results of laboratory studies on pitting corrosion of mild steel associated with leaks of cooling coils in tanks used for storage of liquid high-heat waste (HHW) at the Savannah River Plant (SRP). The objective of this work was to establish the cause of frequent cooling coil leaks in HHW tanks following sludge removal operations and to develop methods to prevent recurrence of leaks. The sludge was removed by slurrying with water after the supernate had been removed.

During 1966-1969, supernate was pumped from five HHW tanks and the remaining sludge was slurried with water and pumped to other tanks. An average residual sludge depth of about two inches remained. During and shortly after sludge removal, cooling coil leaks were much more frequent than during normal operating conditions. Coil leaks cannot allow escape of radioactivity from the tanks, and since spare cooling coils were installed when the tanks were constructed the coil failures have not had a significant effect on waste management operation.

Sludge removal will continue to be necessary for efficient waste management. For example, tentative plans call for removing the sludge from Tank 16H to prepare the tank for retirement. Later, sludge may be removed from other tanks for incorporation into concrete or glass as part of the long term waste management program to solidify defense liquid waste for eventual shipment to a federal repository.<sup>1</sup>

### **BACKGROUND**

#### **Waste Storage Operations**

HHW's from SRP's nuclear fuel reprocessing areas are stored in large underground carbon-steel tanks, Figure 1. Radioactive decay heat must be extracted from the wastes during the first several years of storage until short-lived radioisotopes have decayed. Decay heat is removed by corrosion-inhibited cooling water flowing through carbon steel pipes (cooling coils) to exterior heat exchangers.

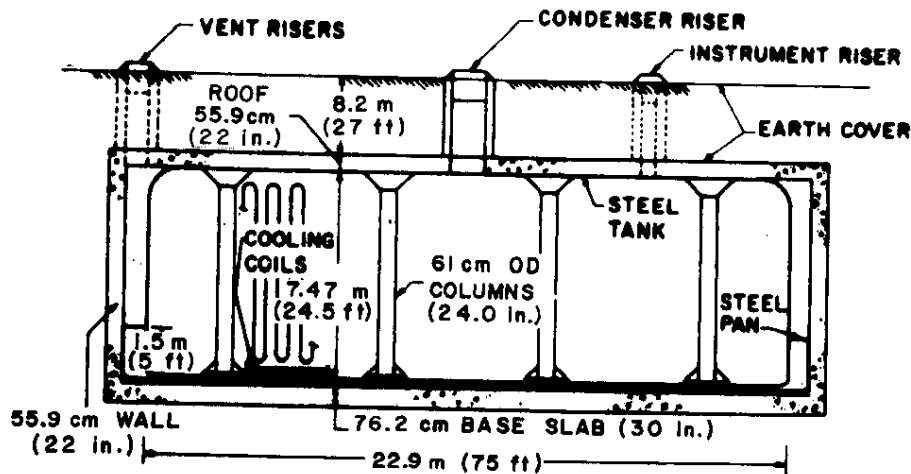


FIGURE 1. HHW Storage Tank  
[Type I, 750,000-gal ( $2.84 \times 10^6$ -liter) Capacity]

Most of the wastes in the tanks are byproducts of separations processes that produce acidic wastes containing suspended manganese dioxide. The wastes are made basic with sodium hydroxide before storage in tanks. The major constituents of the basic wastes are  $\text{Na}^+$ ,  $\text{NO}_3^-$ ,  $\text{Al}(\text{OH})_4^-$ , and  $\text{OH}^-$ . Some of the nitrate is converted to nitrite by radiolysis and nitrite then becomes a major constituent of the waste. The hydroxide addition causes insoluble hydrated oxides, mainly of iron, aluminum, and mercury, to precipitate. The insoluble constituents settle to the bottom of the waste tanks as a sludge.<sup>2</sup>

After the waste is stored for about one year to allow short-lived isotopes to decay and the sludge to separate, the supernate is removed and evaporated to reduce waste volume. The condensed evaporator overheads are passed through a bed of zeolite ion exchanger for removal of traces of  $^{137}\text{Cs}$ , and after being monitored for radioactivity are discarded to a seepage basin. The hot, concentrated evaporator bottoms are returned to other waste tanks in the tank farm where the solutions cool and salts crystallize out. The remaining supernate is subsequently recycled through the evaporator for additional volume reduction.

The waste tank farm is managed so that certain tanks are used to store fresh waste and to accumulate sludge, and other tanks are used as salt receivers. HHW tanks can therefore be classified on this basis. However, in the early stages of separations plant operation, most tanks received fresh waste and contained some sludge; later the concept of crystallizing the salt was adopted to minimize the waste volume and to decrease waste mobility. Sludge was therefore removed to a residual depth of about two inches from five tanks during 1966-1969. These five tanks are now filled to practical limits with salt and are quiescent.



In the twenty years of waste tank operation, stress-corrosion cracks have developed in the primary steel walls of several of the tanks, and several cooling coils have leaked. The frequency with which new leaks developed in the cooling coils was unusually high during and immediately after sludge was removed from the tanks.

As described below, the cooling coils failed from pitting corrosion. However, neither at this time nor since, has there been any evidence that corrosion damage by pitting has occurred in the primary tank walls; no leakage of waste from tanks has been attributed to pitting. Ultrasonic testing has shown that neither general corrosion nor pitting severe enough to cause wall thinning has occurred. This has been confirmed by visual inspection of accessible areas of the tanks. Pitting corrosion probably has not occurred in tank walls because of the absence of crevices, which are required to initiate the oxygen-depletion cells (see below) that are required for pitting attack.

### Description of Cooling Coils

The HHW tank cooling coils are fabricated from ASTM Grades A 53 and A 106 mild steels, nominal 2-in. schedule 40 pipe [0.154-in. (3.91 mm)-thick wall]. The Type I tanks contain 34 vertical and 2 horizontal coils; Type II tanks have 40 vertical and 4 horizontal coils. The total length of the vertical coils is about seven times that of the horizontal coils.

Approximately ten percent of the cooling coils now leak, as shown in Table 1. When coil leakage is discovered, flow of cooling water through the leaking coil is discontinued; the result is decreased cooling capacity of the coil system. In Tank 11H, for example, cooling capacity is reduced by the eighteen coils that no longer function, although sufficient cooling capacity remains for its continued use.

### History of Cooling Coil Leakage

Tank 10H was the only tank to experience any cooling coil leaks during the first ten years of operation. After seven leaks in coils had been found in Tank 10H, a caustic deficiency in the supernate was discovered, and the deficiency was corrected by the addition of 42,500 gallons (161,000 liters) of 50% NaOH over a period of several months beginning in April 1957. The NaOH was

TABLE 1

## HHW Tank Cooling Coil Failure History

Tank <sup>a</sup>	<u>Vertical</u>		<u>Horizontal</u>	
	Total	Leaking	Total	Leaking
1F <sup>b</sup>	34	2	2	0
2F <sup>b</sup>	34	6	2	1
3F <sup>b</sup>	34	1	2	0
4F	34	0	2	0
5F	34	0	2	2
6F	34	0	2	0
7F	34	0	2	0
8F	34	1	2	0
9H <sup>b</sup>	34	6	2	2
10H <sup>b</sup>	34	19	2	2
11H <sup>c</sup>	34	17	2	1
12H	34	0	2	0
13H	40	0	4	0
14H <sup>c</sup>	40	4	4	2
15H	40	0	4	0
16H	<u>40</u>	<u>0</u>	<u>4</u>	<u>0</u>
Total	568	56	40	10

a. Tanks 1F-8F, 9H-12H; Type I,  $0.75 \times 10^6$  gal. ( $2.84 \times 10^6$  liters), 12 internal support columns. Tanks 13H-16H; Type II,  $1.03 \times 10^6$  gal. ( $3.90 \times 10^6$  liters), 1 internal support column.

b. Sludge successfully removed; since used as salt receiver.

c. Sludge partially removed; since used as fresh or aged waste receivers.

added to combat potential corrosion conditions; no other coils leaked in Tank 10H until sometime after sludge removal was completed. Only four other cooling coils in the remaining fifteen Types I and II waste tanks leaked prior to sludge removal operations. Thus, if the Tank 10H experience is discounted, the incidence of cooling coil leakage prior to sludge removal was approximately 0.02 leaks per tank year of operation. The causes of the other leaks that were observed before sludge removal are unknown, but if they were corrosion induced, they did not proceed according to the mechanism described in this report.

Sludge removal operations were attempted on seven tanks beginning in 1967. From 2 weeks to 7 months after sludge removal, the incidence of cooling coil leakage increased, and four tanks had 33 leaks in cooling coils during the short periods that the tanks contained a residual of sludge-slurry water. Coil leaks in Tanks 11H and 14H began about two weeks after sludge removal; addition of evaporator bottoms to tanks that contained the residual sludge sharply reduced the incidence of coil failures.

A summary of the rate of cooling coil leaks is shown in Figure 2. Coils leaked 500 times more frequently shortly after sludge removal than during normal storage. Furthermore, leaks were also six times more frequent after the tanks were returned to service than before sludge removal, probably because many of the coils that did not leak had been damaged during sludge removal.

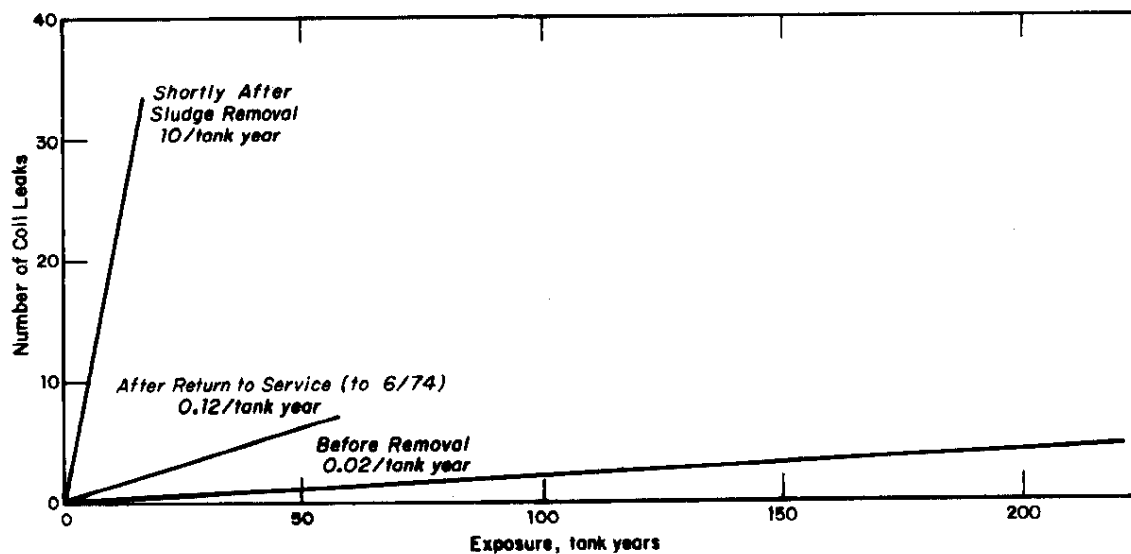


FIGURE 2. Incidence of Cooling Coil Leaks in Tanks 1F-8F, 9H, and 11H-16H

Tank 10H is omitted from Figure 2 because it appears to be a special case in which the caustic deficiency may have caused corrosion of coils before sludge removal. Although after sludge removal and salt addition, the coils in Tank 10H were exposed to sludge-water slurry for eight months with no additional coil leaks, eleven coils leaked during the following four years. These later leaks also appear to be a special case. During the four-year period, flow through the cooling coils was stopped to maintain tank temperature in the 85 to 100°C range. During the stagnant period, the chromate corrosion inhibitor normally in the cooling water probably became depleted to a concentration at which it acted as a pitting agent.<sup>3</sup> The resulting pitting would progress from the inside out and eventually cause the pitted coil to leak. Corrosion of coils in Tank 10H was probably associated with chromate-depleted cooling water. Corrosion by chromate-depleted water was prevented either by periodic flushing of coils or by a continuous, but low coolant flow (restricted by an orifice) to avoid stagnation.

Actual leak sites have been examined in only one cooling coil. In 1967, leaks were located in both horizontal and vertical coils of Tank 2F. A 16-in. (0.40-m) section was removed from the upper horizontal coil; mechanical interferences prevented removal of a vertical coil section. The leaks in the horizontal section of coil were caused by pitting from the outside (Figures 3 and 4) and apparently occurred during sludge removal. Pitting initiated under loose porous material and was attributed to an oxygen depletion cell. Intergranular attack was also observed under some of the scale (Figure 5).

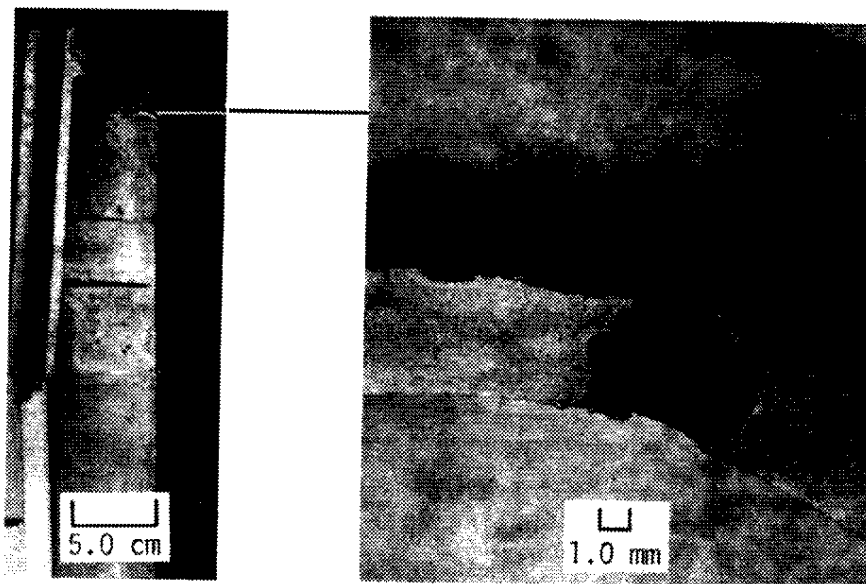
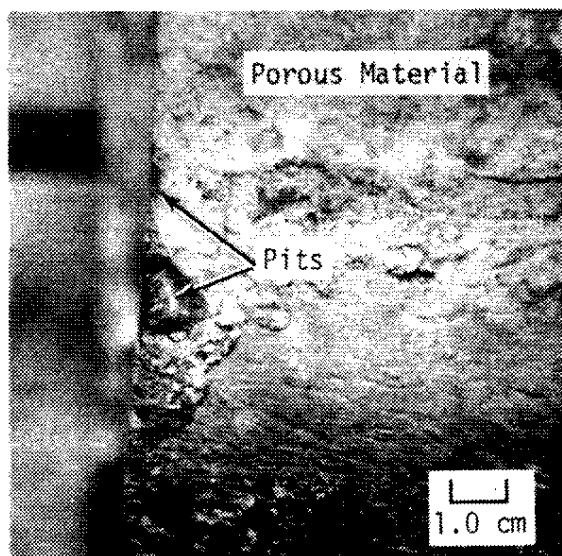
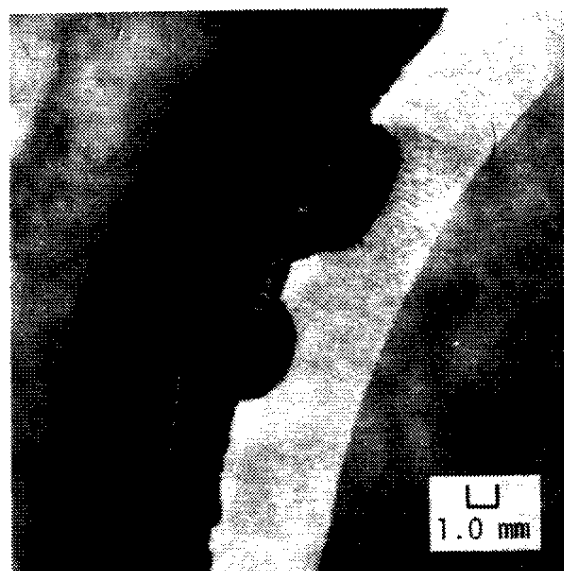


FIGURE 3. Perforated Upper Horizontal Cooling Coil, Tank 2F



a. Porous Material and Pits



b. Transverse Section of Pits Shown Above

FIGURE 4. Pits Beneath Loose Porous Material on Cooling Coil

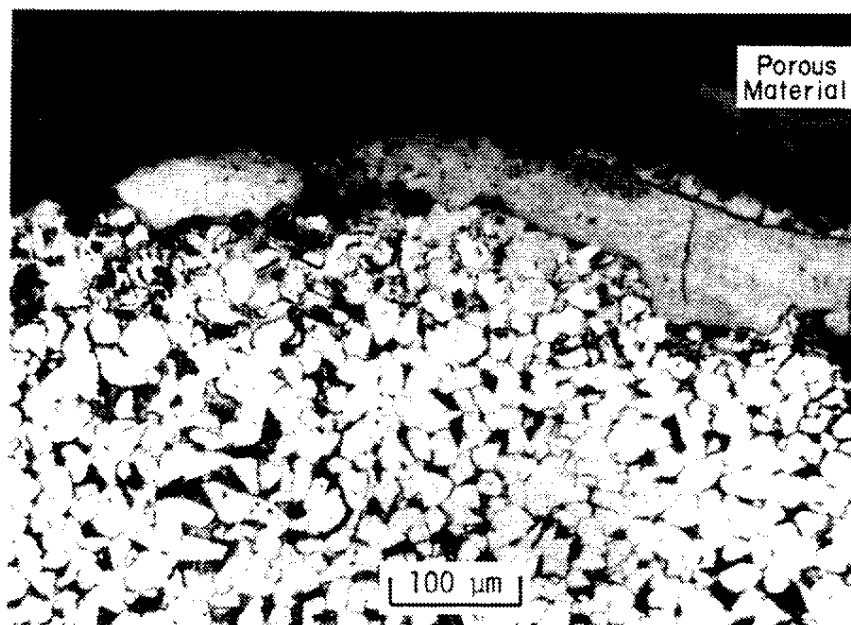


FIGURE 5. Microstructure of Cooling Coil Under Porous Material

Further attempts have been made to locate leaks in cooling coils by several methods, but none have been located; therefore, no additional leaking sections of coils were removed for examination.

## EXPERIMENTAL RESULTS

### Miniature Cooling Coil Tests

Preliminary tests were performed with miniature cooling coils of A 106 steel that were cooled with circulating tap water while they were immersed in various test solutions that could be heated if desired.

Pitting from slurring operations alone was apparently greatest in Tank 11H. Consequently, supernate and sludge compositions in the pitting studies discussed below were based, as much as possible, on compositions in this tank. Observed pitting of cooling coils may have been caused by supernate. However, this could not be verified with actual supernate because Tank 11H supernate had lost its identity when it was mixed with other supernates and the mixture evaporated. Consequently, pitting tests were made by immersing miniature cooling coils in a synthetic Tank 11H supernate. The miniature coils were not attacked at 92°C, but became coated with a thin layer of insoluble salts. The metal under the salts remained shiny.

An equal volume of synthetic sludge was then added to the supernate at 40°C. The sludge contained 14 constituents and was formulated according to Savannah River Laboratory studies of solid waste forms for analyses made in high-level wastes.<sup>4</sup> Only mild pitting, a rate of <0.001 in./mo (0.025 mm/mo), was observed. Pitting rate was not increased following a one-thousandfold dilution, and the metal stayed bright and shiny without becoming coated. The only inhibitor in HHW that could keep steel bright and shiny was basic nitrite; other components were too dilute when diluted by one thousand.

A ten-thousandfold dilution of Tank 11H synthetic supernate with sludge addition increased the pitting rate at 40°C by about a factor of three, to 0.003 in./mo (0.076 mm/mo). Although slurrying sludge with very dilute supernate did increase the pitting rate, the rate was far too low to account for the 0.150-in./mo (3.81 mm/mo) penetration that was observed in Tank 11H on 18 coils during sludge removal.

Analyses of HHW supernates account for only 10 to 30% of the sulfate predicted from process flowsheets.<sup>2</sup> Recently, analyses showed the sludge in Tank 16H was 0.09 molal in  $\text{SO}_4^{2-}$ , and 50% of this dissolved in water washes.<sup>5</sup> Thus, the original sludge removal operations that involved pumping out the supernate, then slurrying the sludge with water, could increase the concentration of sulfate relative to the other components in the liquid phase of the slurry.

The combination of increased sulfate and dilution of basic nitrite inhibitor in the waste could lead to rapid perforation of the steel coils. Sulfate that is present in the sludge and then dissolved in the slurry solution should promote pitting.<sup>6</sup> Dilution of an anodic inhibitor, such as nitrite, below the level of effective inhibition is considered an improper practice that can cause pitting and sometimes accelerate corrosion.<sup>7</sup> The possible extent of such acceleration was estimated by several experiments designed to establish the electrochemistry of pitting in sludge-supernate-water solutions.

## Polarization Studies

In polarization studies of steel specimens, surface current densities were measured continuously as a function of applied potential. The anodic polarization curve corresponds to oxidation at the test electrode; conversely, the cathodic polarization curve corresponds to reduction at the test electrode. Mild steel specimens were immersed in diluted (ten-thousandfold) Tank 11H supernate containing  $0.03M SO_4^{2-}$  to simulate slurries developed in waste tanks. The polarization curves for such specimens are shown in Figure 6. The  $0.03M SO_4^{2-}$  concentration was selected to represent that which is produced by slurring  $CaSO_4$ , a possible sludge sulfate, which is relatively insoluble.

The maximum current density possible under the test conditions can be defined from the intersection of the anodic and cathodic Tafel<sup>7</sup> slopes (Figure 6). The steep anodic Tafel slope shows that the cell is under anodic control<sup>8</sup> and that the basic nitrite, an anodic inhibitor, is not effective at this concentration when sulfate is present. The uniform corrosion rate,  $R_m$ , calculated by Faraday's Law for this specimen is 0.05 in./mo (1.27 mm/mo). If the corrosion were localized as pitting, the rate could easily be as high as that observed in coil penetrations, viz., 0.15 in./mo (3.81 mm/mo).

### *Interpretation of Curves Related to Pitting*

The hyperbolic shape of the anodic polarization curve for the test solution demonstrates a freely corroding system; the curve for steel in waste at the same dilution but without sulfate (see inset in Figure 6) shows a limited current density until  $O_2$  evolution at high anodic potentials. The possible corrosion rate without sulfate is about 0.002 of the rate calculated for the freely corroding system with sulfate.

The specimen used in the determination of the anodic polarization curve in solutions that contained sulfate was pitted and corroded. Pitting can be initiated either by an anodic discontinuity in the metal or by a differential aeration cell. One common mode of pit formation is by an oxygen depletion cell which may develop in a crevice that restricts solution circulation. In an idealized reversible depletion cell when the Nernst equation would apply, for example, with platinized platinum electrodes, an emf of a few hundredths of a volt is developed in a dilute electrolyte saturated with oxygen at 0.2 atm pressure at the anode and a similar medium saturated at 1 atm pressure at the cathode.



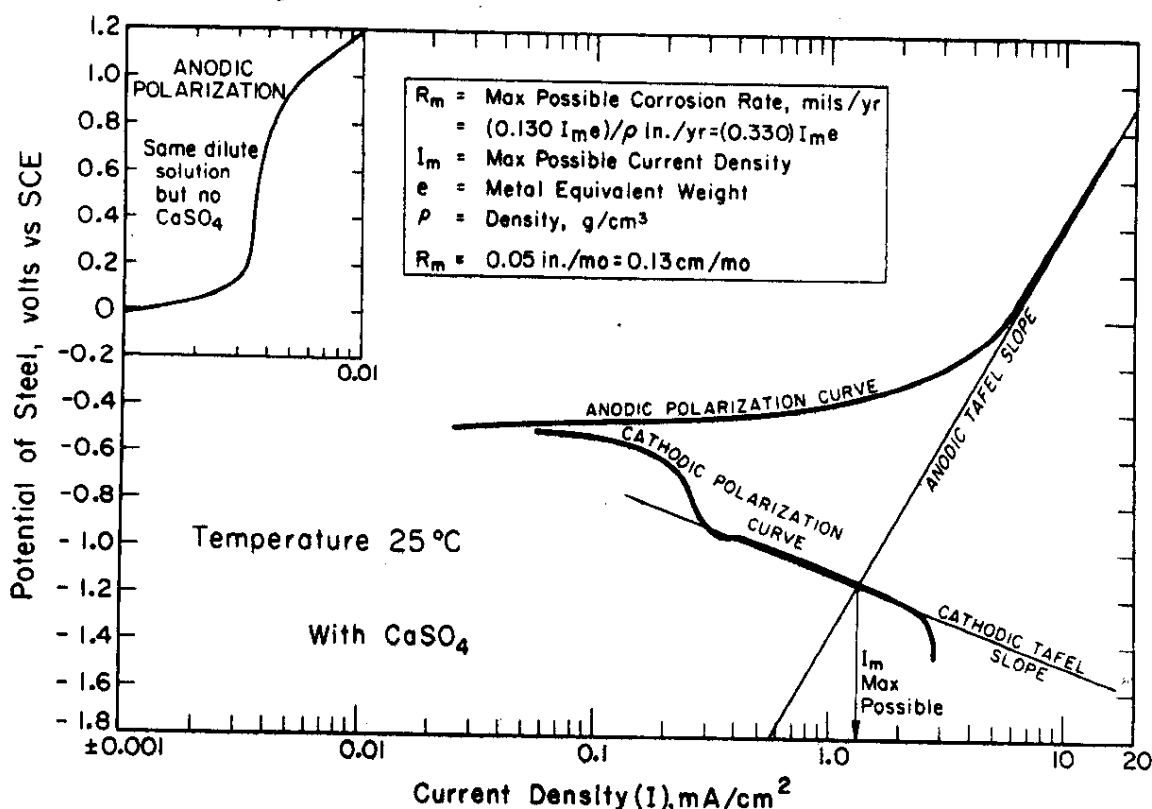


FIGURE 6. Polarization Curves of Steel in Dilute (ten-thousandfold) Synthetic Tank 11H Waste With and Without  $\text{CaSO}_4$

Under actual cell conditions, if iron electrodes are used, an electrically conductive iron oxide forms at the cathode and acts as an oxygen electrode; iron dissolves at the anode to form  $\text{Fe}^{2+}$ . The Nernst equation predicts a potential of 1.26 V for an  $\text{Fe}^{2+}$  concentration of 0.1M at the anode, a pH of 7, and an  $\text{O}_2$  pressure of 0.2 atm (air-saturated) at the cathode. In practice, the actual potential is lower because the Nernst equation requires reversible reactions, and the reduction reaction at the iron oxide cathode is not reversible. However, actual potential differences of 0.5 to 0.6 V between an active pit (anode) and a conductive steel oxide have been measured.<sup>9,10</sup>

### *Pitting Demonstration and Pitting Prevention*

In laboratory demonstrations, classical active-passive pitting occurred when a potential of 0.5 V was applied to steel samples in the same pH 9 synthetic solution that was used to determine polarization curves (with  $\text{CaSO}_4$ ) shown in Figure 6. The active portion of the cell is the bottom of the pit (anode), where iron is oxidized,  $\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$ . The passive portion of the cell is the iron oxide ring (cathode) around the pit, where oxygen is reduced,  $\frac{1}{2}\text{O}_2 + \text{H}_2\text{O} + 2\text{e}^- \rightarrow 2\text{OH}^-$  (Figure 7). The 0.5-V potential produced a pitting rate sufficient to perforate a cooling coil pipe with a 0.15-in. (3.81 mm) wall thickness in six days, and thus could explain the coil perforations produced during sludge removal. Less severe pitting but more severe general corrosion occurred at lower potentials, and at 0.4 V, the pitting was still sufficient to produce cooling coil perforation in 10 to 15 days.

Pitting of the steel did not occur when the waste was not excessively dilute or when adequate inhibitor was used. Figure 7b shows the same steel subjected to the same conditions of potential, viz., 0.5 V anodic to open circuit, time 1/2 hr, and room temperature. The steel was not attacked when exposed to Tank 11H solution diluted one hundredfold or to water at pH 12 containing 500 ppm  $\text{NO}_2^-$ . The small spots seen in Figure 7b are artifacts on the steel surface.

### **Additional Pitting Experiments**

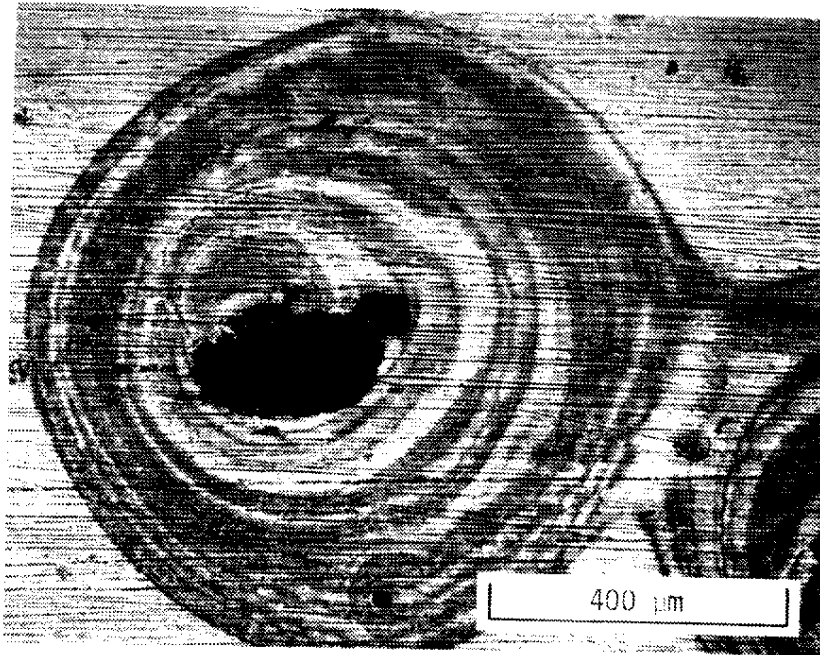
Two attempts were made to reproduce, without electrochemical control, the pitting mechanism described above. In both cases, a ten-thousandfold dilution of Tank 11H supernate containing 0.03M  $\text{SO}_4^{2-}$  was used.

In the first attempt, two 3/4-in. x 4-in. (19 x 102-mm) strips of steel were bolted together to form a tapered crevice. Opposite the bolted end the strips were separated with 0.010-in. (0.25 mm)-thick *Teflon*.<sup>\*</sup> Immersion in solution for one month caused slight corrosion and pitting, but the penetration rate was inconsequential relative to that observed in cooling coils.

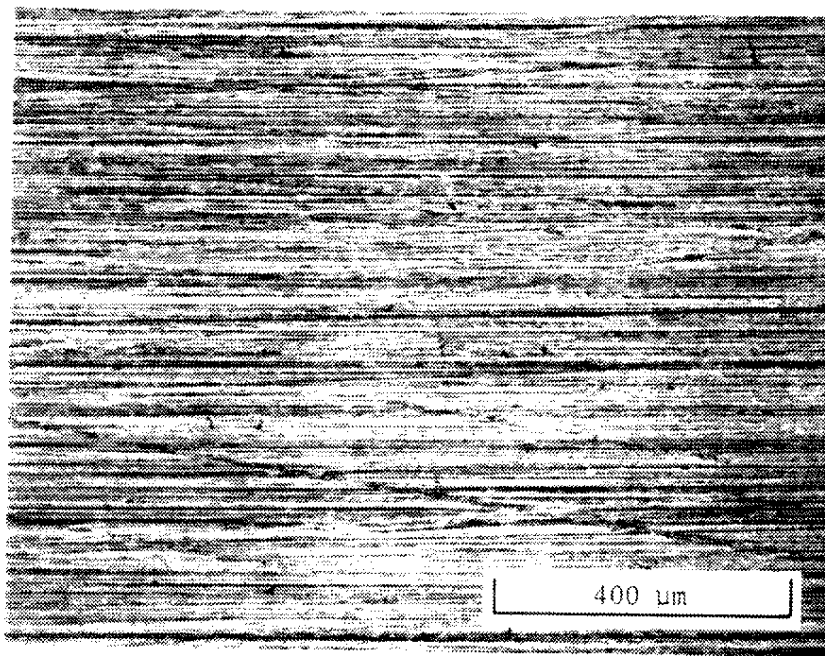
In the second attempt, pits of 0.001-0.002-in. (0.025-0.050 mm) depth were initiated in mild steel coupons by an oxygen depletion cell. Single coupons suspended in each of two containers were electrically connected. One solution was deaerated with helium;

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<sup>\*</sup> Du Pont trademark for fluorocarbon resin.



a. Active-Passive Pitting at Ten-Thousandfold Dilution of Tank 11H Supernate



b. No Attack at One-Hundredfold Dilution of Tank 11H Supernate

FIGURE 7. Pitting and Prevention of Pitting in A 106 Mild Steel

the other was purged with air; the solutions were ionically connected by a salt bridge. Pitting formed on the sample in the deaerated half cell in about three days. The coupons were then disconnected, and either iron or mercury sludge was added (to supply  $\text{Fe}^{3+}$  or  $\text{Hg}^{2+}$ ) to accelerate pitting. One pit penetrated about 0.005 in. (0.127 mm) in one month, but most pits initiated by  $\text{O}_2$  depletion did not propagate. Failure to produce rapid pitting in this experiment probably was due to drainage of acidic solution from the pits when the sample was transferred. The pH of 9 limited the concentration of accelerating ions. For example, the  $\text{Fe}^{3+}$  concentration at pH 9 theoretically would be only  $10^{-6}$  ppb.

## PITTING MECHANISM AND PREVENTION

The experimental studies indicate that cooling coils can be perforated by pitting corrosion after sludge removal if residual sludge and water remain in the tank. This conclusion is based on tests with synthetic wastes. The proposed mechanism for pitting is initiation at oxygen depletion cells which may develop in crevices. Initiation is followed by rapid corrosion accelerated by dilution of the anodic inhibitor, basic nitrite, and by sulfate that dissolved from the sludge during removal operations. This mechanism indicates that pitting attack of coils should not occur under normal operating conditions, because the solution compositions are not conducive to it.

Crevices are abundant in the waste tanks, especially around vertical cooling coil supports as shown in Figure 8. Corrosion, started as oxygen depletion cells in the crevices, can eventually produce potentials of the order of 0.5 V between pits and matrix metal in a sludge-water slurry. Sulfate and dilute nitrite can accelerate this corrosion. Any crevice would be suitable for pit initiation, even if the crevice were formed by insoluble salts precipitated on the cooling coil pipes.

Coil perforation did not occur in all tanks because the basic nitrite inhibitor was not sufficiently diluted during slurring of sludge. Basic nitrite is an effective inhibitor in SRP waste diluted one-thousandfold or less, although crevices reduce its effectiveness to some extent. Cooling coils in Tank 10H survived eight months in a slurry of sludge without cooling coil damage, but 18 coils in Tank 11H leaked after only one month in a similar slurry. Apparently, the wastes in Tank 10H were not diluted as much as were those in Tank 11H, and the basic nitrite remained effective even though sulfate was present. The conclusion on waste dilution in Tanks 10H and 11H was later verified through plant records.

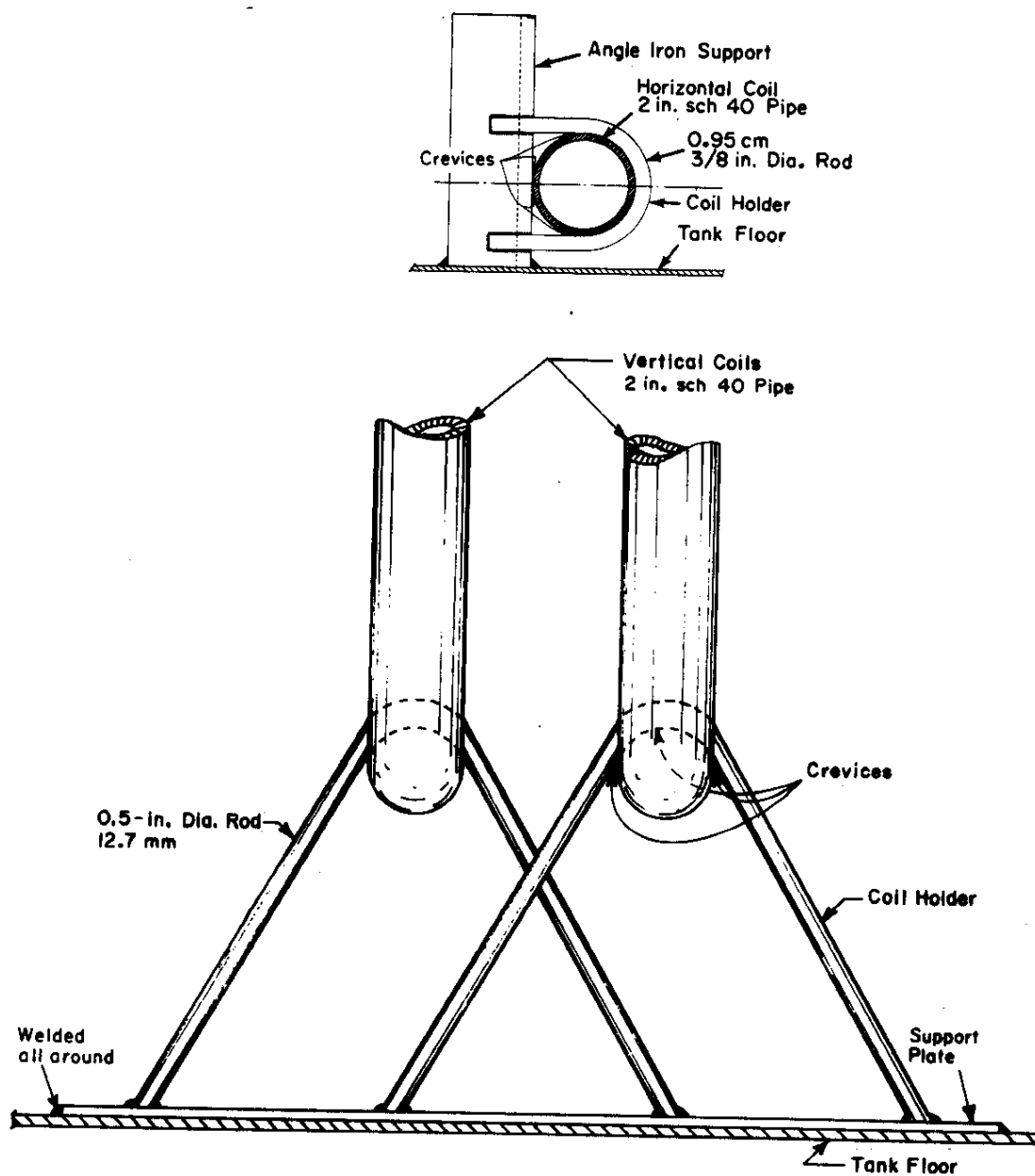


FIGURE 8. Typical Crevices Formed by Cooling Coil Supports in Tanks 1F-8F and 9H-12H

In future sludge removal operations, the waste supernate should not be diluted by more than a factor of one hundred. Corrosion of the cooling coils should be precluded by this limited dilution. If supernate diluted by one hundredfold is not a satisfactory medium for slurring sludge, an aqueous solution containing 500 ppm nitrite at pH 12 could be used. This would be a minimal increase in the total waste burden, requiring about 7500 lb (3401 kg) of sodium nitrite per million gallons ( $3.78 \times 10^6$  liters) of slurry water.

#### ACKNOWLEDGMENT

The cooling coil examinations shown in Figures 3-5 were performed by the Equipment Engineering Section of the Savannah River Plant.

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