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LEAKS IN RADIOACTIVE-WASTE TANKS

by

R. M. Girdler

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Approved by

E. B. Sheldon, Superintendent
Separations Technology Section
Savannah River Plant

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E. I. DU PONT DE NEMOURS & COMPANY
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ABSTRACT

A number of leaks developed in four of sixteen underground storage tanks used for storage of alkaline radioactive waste from the separations area at the Savannah River Plant. The tanks are 75 to 85 feet in diameter and were field erected by welding; the welds were not stress relieved. The investigation of these leaks revealed no correlation with the history of the construction or usage of the tanks. Stress corrosion cracking is the most likely explanation of the leaks. Similar stress corrosion cracking in tanks of this type can be prevented by full stress relieving after welding has been completed.

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LEAKS IN RADIOACTIVE-WASTE TANKS

INTRODUCTION

A number of leaks have developed in four of the sixteen underground tanks used for storage of alkaline radioactive waste from the separations area of the Savannah River Plant. The carbon steel tanks, 75 and 85 feet in diameter, had been field erected by welding, but the welds were not stress relieved. Equipment for remote observation of leakage areas was designed and fabricated and most of the tanks in the area were viewed. The four tanks that are known to have leaked were examined in detail to determine the location of the leaks, and samples were taken from the wall of one of them to determine the nature and cause. Tank construction histories and usage were reviewed for possible correlation with the occurrence of leakage. Results of the investigation are given in this report.

SUMMARY

Numerous small leaks were seen in two tanks, most of them near, but usually not on, the welds; these leaks tended to seal themselves due to the accumulation of salts. Examination of several leaking sites, using remote equipment, and of samples taken from the tank wall showed many cracks. These cracks were presumed to have arisen from the chemical action of waste on steel under the influence of stress resulting from welding. The leaks in one tank almost certainly resulted from stress cracking, and this was very probably the cause of the leaks in the other.

No leaks were seen in those areas of the other two tanks that could be visually inspected, thus information was insufficient to establish the reason for the leakage.

The presence of leaks in some tanks, but not in others, could not be correlated with the history of the construction or usage of the tanks. All of the leaks occurred in one separations location (H Area); none occurred in the other location (F Area).

DISCUSSION

DESCRIPTION OF TANKS

At the Savannah River Plant, waste that is evolving appreciable fission product heat is stored in water-cooled carbon steel tanks.

Four of these tanks are known to have leaked. Figure 1 is a vertical section showing the main design features of tanks 14 and 16, two of the leaking tanks. As shown, this design embodied carbon steel plates (A-285 grade B, fire-box quality) welded together to form a free-standing tank 85' in diameter and 27' high. The tank roof is supported by the tank sides and by a single central column. All seam welds were X-rayed. These tanks were completed in February 1956.

The tanks stand in a concrete encasement, the bottom portion of which is carbon steel lined. The space between the tanks and the casement was provided for collection and control of leakage from the tanks, to prevent the escape of leaked waste to the environment. This annular space is normally ventilated with warmed air to decrease the rate of rusting of the steel. The existence of this space was most valuable in diagnosing the leakage problems discussed in this report.

The design of tanks 9 and 10 (which have also leaked) is similar to that of tanks 14 and 16 with the following exceptions:

- o Tanks 9 and 10 are 75' in diameter.
- o They have twelve (instead of a single) roof-supporting columns.
- o They are more deeply underground.
- o There are other minor constructional differences; e.g., the exterior surfaces of tanks 9 and 10 are painted.

Tanks 14 and 16 are discussed in more detail because their leakage problem has been more intensively studied.

TANK CONTENTS

In general, tanks 9 and 10 contained H-Area Purex waste, tank 14 contained H-Area Purex Waste at the time the first leak developed, and tank 16 contained HM waste. A chemical analysis of the three types of waste is given in the following table. Actual departures of the contents of the tanks from these compositions will be treated under the history of the individual tank.

As far as is known, none of the tanks containing F-Area Purex waste has developed leaks; some of the tanks containing H-Area Purex waste and others containing HM waste have not developed leaks either.

The composition of the radioactive waste in each of the waste tanks at significant times in their history is not completely known because:

- o Complete chemical analyses of the stored wastes had not been made in most cases; partial chemical analyses were made infrequently and irregularly.
- o The separations processes yielded waste subject to considerable variation in composition.
- o An imprecisely known fraction of some components tended to settle out as a sludge, thus causing the composition of the two phases to change.

However, the following table presents the approximate chemical composition of the three types of waste in question (including sludge). These compositions are based on a consideration of such factors as analyses, flowsheets, and consumption of chemicals.

<u>Chemical Composition of Waste Types, wt %</u>			
<u>Chemical</u>	<u>F-Area Purex</u>	<u>H-Area Purex</u>	<u>HM</u>
NaNO ₃	29.7	36.6	29.8
NaOH	1.0	1.1	3.8
NaAlO ₂	0.0	0.0	7.8
Na ₂ SO ₄	5.8	2.3	0.2
MnO ₂	0.3	0.6	0.0
Fe(OH) ₃	2.6	0.5	0.1
Mn(OH) ₃	0.1	0.1	0.0
KNO ₃	0.1	0.3	0.0
Hg(OH) ₂	0.0	0.0	0.1
H ₂ O	60.4	58.5	58.2

HISTORY OF TANK 16

The significant variables (level, temperature, etc) and events (detection of leakage, etc) in the history of tank 16 are displayed graphically in Figure 2. The first material added to this tank (on May 24, 1959) was 3400 gallons of 50% NaOH that formed a 1"-deep layer on the bottom of the tank. Between this date and the first positive evidence of a leak (November 1959), about 200 inches of HM waste was added to the tank. The only unusual operating incident recorded during this period was the probable addition of some acidic waste on

September 15, 1959. The time necessary to dissipate all acidic zones completely by casual mixing with the large amount of excess alkalinity present is not known, but it appears unlikely that this incident is related to the leaks. Accidental additions of acidic wastes have been made to other tanks in both areas without subsequent development of leaks. About 100 inches of waste was added between November 1959 and August 1960.

Between August 1 and September 1, 1960, the leakage rate increased to about 1-1/2 gallons per minute. Liquid in the annular space reached a depth of 4-1/2' on September 7, 1960. The material which had leaked from the tank was jetted to tank 14; however, it had overflowed the top of the encasement lining before the jet came into operation. On October 12, tank 16 was partially emptied into tank 15; when tank 16 level dropped below the middle horizontal seam weld, the leak seemed to stop. Since that time, no further transfers have been made into or out of this tank.

HISTORY OF TANK 14

As shown in Figure 3, tank 14 may have started leaking as early as April 1959 when a detecting instrument in the annular space alarmed. Radioactive (5 R/hr at 2") crystalline material was first seen in the annular space in May 1959, but no liquid was seen in this space until 8000 gallons of water was added to the tank in March 1960. A fluorescein dye test showed positively that liquid was leaking from the tank.

Starting in September 1960, waste additions were resumed; the tank level increased from 166" to 240-1/2" in December of that year. Further additions were not made because of increased leakage to the annular space; e.g., the depth of liquid in the annular space increased from 3-3/4" to 7" between November 1 and November 21. After additions to the tank were discontinued and the dehumidifier system was put into regular operation, the level in the annular space stopped increasing and actually decreased (this is attributed to the evaporation of water from the waste).

The first material added to this tank (on September 23, 1957) was 3400 gallons of 25% NaOH that formed a 1" deep layer on the bottom of the tank. Between this date and the appearance of leaks, it received only H-Area Purex waste of the nominal composition previously described. Analytical results from a sample of supernate taken on March 20, 1958, are shown in Figure 3. In August 1958, 7-1/2" of Purex high level waste from tank 9 was added to tank 14. As discussed in the next section, tank 9 material originally contained gluconic acid, used to prevent settlement of MnO_2 during transfer to the waste tank.

HISTORY OF TANK 9

As shown in Figure 4, tank 9 received liquid in July 1955, but it was nearly two years after the tank was filled before leakage was detected. All of the waste was from Purex processing of natural uranium fuel elements. However, the "head end process" waste contained considerable gluconic acid to solubilize the MnO_2 precipitate it contained. In the high radiation field this organic material decomposed and liberated CO_2 which consumed the excess caustic (see analysis of August 1957, Figure 4). To restore the excess alkalinity, 170,000 pounds of 50% NaOH was added to the tank in December 1957 (after leakage had started). After 19,000 pounds of water was added in January 1958, an additional 85,000 pounds of 50% NaOH was added. No further additions were made and an April 1958 analysis of the supernate (see Figure 4) showed that these caustic additions had restored an excess caustic concentration and eliminated the $NaHCO_3$.

Hydrogen content of the vapor space of the tank was 150% of the lower explosive limit in September 1956. Thereafter, it was controlled by forced ventilation of the tank.

The annular space was flushed with water several times and the flushes were added to the tank, but since March 1959 no further transfers have been made into or out of this tank or annular space.

HISTORY OF TANK 10

As shown in Figure 5, tank 10 became filled with Purex high activity waste in the latter part of 1956; a leak, evidenced by the observation of crystalline solids in the annular space, was detected July 16, 1959. When dissolved in water, the solids had a radioactivity of 3.1×10^7 counts/minute/gram, and the pH of the solution was greater than 11. Analyses (Figure 5) show that before the leak was found the excess caustic had become depleted, presumably due to reaction with CO_2 which was detected in the vapor space in March 1957.

TANK USAGE

Examination of the history of the four tanks that leaked reveals that none of them were subjected to temperatures more extreme than any encountered by the other tanks. (The highest sludge temperature recorded up to June 1964 was $215^\circ C$ in tank 5; the highest supernate temperature recorded was $55^\circ C$, also in tank 5.) Tanks that did not leak were filled as full as those that did leak.

It may be significant that tanks 9 and 10 were both subjected to depletion of excess alkalinity due to carbon dioxide generation from decomposing organic material. However, tank 3 (F Area) and tank 12 (H Area) had also been permitted to lose excess alkalinity at some time in their history, and they did not develop leaks.

Tanks 14 and 16 had never been exposed to conditions of depleted alkalinity, but all four leaking tanks had been exposed to solutions containing a considerable excess of NaOH before they leaked. All tanks contained an abundance of NaNO_3 .

As will be discussed in subsequent sections, it appears certain that tank 16 and probably tank 14 leaked because in weld-affected areas they developed stress corrosion cracks large enough to permit passage of liquid. In tanks 9 and 10 similar evidence was not noted; it may well be that the leaks developed from some other cause.

INSPECTION OF TANK WALLS

As shown in Figure 1, construction of the tanks is such that their outside walls can be inspected. The inspection must be made remotely due to the high gamma radiation field. The inspecting instrument could be inserted through the four large annular space access ports already provided; however, in this manner only about one-fourth of the tank would be visible. To increase the fraction of tank wall that could be inspected, additional holes, each 8 inches in diameter, were drilled into the annular spaces of tanks 14 and 16. (Figure 8 and 9 show, in developed elevations, the number and location of the additional holes and the wall area that may be seen from them.) The holes were drilled with a diamond core drill through the thick concrete pad that covers the tank top and annular space. Similar holes were not drilled in tanks 9 and 10, partly because there is about 11 feet of earth fill and 2 feet of concrete between the ground level and the annular space.

A television camera mounted on a mast with retractable booms (Figure 7) was built for wall searching and was useful in monitoring the removal of a sample of steel from the wall (described in the next section). A sectionalized periscope $2\frac{1}{2}$ inches in diameter (Figure 6), built by Lerma Engineering Company, was used for the bulk of the leak searching and for photographing detail with high magnification on color film.

Figure 8 is a developed elevation view of the outside wall of tank 16, the tank studied most extensively. The location of each significant leak is indicated by a spot and tail, roughly portraying the appearance of the leak. Figure 9 gives similar information for tank 14.

Figure 10 is a photograph showing a group of leaks near the bottom horizontal weld of tank 14. The white masses, tailing downward, are solids which have accumulated near the leak due to the evaporation of the liquid component of the waste under the influence of the air in the annular space.

About 25 individual leak sites were observed on the walls of tank 14; 134 were seen on tank 16. All of the leaks on tank 14 were near the bottom horizontal weld seam; leaks on tank 16 were seen near all 3 horizontal seams, 3 vertical seams, and at several sites not involved with seams between plates. The possibility that the latter leaks developed from welds for attachments to the inside surface of the wall was not excluded. It should be noted that tank 14 was never filled above the top horizontal seam.

The walls of tanks 9 and 10 were inspected with the periscope to the extent possible through 3 out of 4 of the standard large-diameter access holes to the annular space. About 20% of each tank wall could be examined. Neither tank showed any evidence of leakage through the side wall.

The large number, nature, and location of leaks in tanks 14 and 16 strongly suggest the occurrence of stress corrosion cracking under the influence of the high nitrate, alkaline solution, and stresses due to welding. A detailed examination of the fine structure of the leaks in tank 16 confirmed this.

FINE STRUCTURE OF THE LEAKS

Examination of the tank wall, as found, revealed the number and location of the leaks, but very little about the leaks themselves. More detailed information about the leaks in tank 16 was obtained, first by washing the accumulated salts from an individual leak and removing the rust by sandblasting while observing and photographing the leak area through the periscope after each operation. As shown in Figure 11, there were definite cracks in this location. The next step was the cutting and removal of a sample of the wall for laboratory inspection.

A plate 5-5/8 inches in diameter was removed from the wall of tank 16 to allow a detailed metallographic examination and chemical analysis of a portion of the tank wall in which the presence of leaks had been confirmed.

Removal was made from the annular space, using a cutter head which retained the cut-out sample with magnets (see Figure 12). The location

of the sample, selected to include two previously observed leaks, is shown in Figure 8. Prior to the removal this area was sandblasted, and cracks were observed where there had been deposits. The sample was centered approximately on the weld between the top knuckle and shell plates.

Because of the radioactive contamination of the sample, most of the examination was conducted in the high level caves at Savannah River Laboratory. A visual examination of the specimen was made prior to any cleaning operations. Two cracks could be seen on the exterior surface of the specimen, but a tightly adherent brown deposit obscured all minor details of the interior surface. Attempts to remove the surface layer by mechanical methods were not successful, primarily because of the magnetism in the sample that resulted from the use of magnets during its removal from the tank. Most of the coating on the inner surface was finally removed by briefly dipping the specimen in inhibited hydrochloric acid. The crack in the specimen is clearly shown in Figure 13.

A second sample 5-5/8 inches in diameter, centered approximately on the weld between the top knuckle and shell plate, was removed from the tank at a known leak point approximately 45 feet from the first sample (see Figure 8). Visual and metallographic inspections indicated this sample had the same characteristics with regard to cracking as the first sample, so it was concluded that representative samples of the failures in tank 16 had been obtained.

Chemical analysis of samples from the weld and from shell and knuckle plates is compared to the chemical analysis of the material as reported by the tank erector in the following tables.

Chemical Analysis of Knuckle Plate, Weld, and Shell Plate
From the Specimen Removed From Tank 16

	C	Mn	P	S	Si	Cu	Al
Knuckle plate	0.105	0.51	0.018	0.021	0.194	0.055	0.006
Weld	.088	.49	.016	.019	.192	.058	.005
Upper shell plate	0.080	0.41	0.011	0.018	0.105	0.082	0.007

Erector's Analyses of Material Used in Upper Shell Plates and
Upper Knuckle Plates of Tank 16, and A.S.T.M. Requirements

	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>
Heat No. 87G512	0.09	0.60	0.010	0.030
Heat No. 84G468	.09	.44	.009	.029
Heat No. 79G451	.11	.46	.010	.033
A.S.T.M. A-285				
grade B firebox	0.22	0.80	0.035	0.04
quality	max	max	max	max

Chemical analyses of the knuckle and shell plates from the sample agreed with mill-test reports submitted by the tank erector; they were within the limits specified for A.S.T.M. A-285 grade B firebox quality steel. Compositions of the weld material and plate material were essentially the same.

Good ductility was demonstrated by bend tests performed with strips cut from the decontaminated portion of the sample.

Very little general corrosion of the inner surface occurred during the seven months in which waste solution was in contact with the steel and the much longer periods in which the tank was empty or partially filled. Grinding marks which were made when the tank was erected are visible on the weld. Shallow pitting, about 0.020 inches deep, occurred at several locations.

All cracks were intergranular (Figure 14). One crack in the knuckle plate appeared to originate adjacent to the arc strike. Figure 15 shows a cross section through this arc strike. Neither of these two major cracks propagated into the weld.

Numerous small cracks were observed on the inner surface. These cracks, shown in Figure 16, were located in both the knuckle and shell plates in bands about one-half inch wide. The edge of these bands was located between 1/16 and 1/8 inch from the edge of the weld. The cracks ranged from 1/16 to 1/2 inch in length, and the deepest ones were removed by grinding about 1/32 inch from the surface.

CAUSE OF LEAKS

The observations that have been described lead to the conclusion that tank 16 leaked because of the development of numerous cracks, some of them all the way through the plate and large enough to pass liquid when under hydrostatic pressure.

Hydrogen charging of the steel had been suggested as a possible cause of cracking. The presence of atomic hydrogen is necessary for hydrogen charging of carbon steel which, depending upon the composition and mechanical properties of the steel, may result in cracking. The following test was conducted to investigate this possibility.

Five test "pillow" specimens were made by welding together two 3-inch-square flat pieces of representative 16-gage carbon steel around the periphery; these were immersed in waste tank 16. When inspected after 6 months of exposure, the specimens showed no evidence of the inflation which would have occurred if existing conditions were capable of charging steel with hydrogen. The specimens remained bright and showed no corrosion.

It was concluded that hydrogen charging was not a factor in causing cracks in tank 16.

Stress corrosion cracking, then, remains the most likely explanation of the leaks in tank 16 since the following conditions, characteristic of stress corrosion cracking of carbon steel, were present:

- o The cracking was intergranular.
- o The leaking occurred near welds where large residual stresses would be expected from thermal effects during welding.
- o The stressed steel was exposed to an aqueous solution containing considerable sodium nitrate and sodium hydroxide, agents known to promote stress corrosion cracking.

An extensive laboratory program showed that the stress corrosion cracks were possible in steel structures representative of tank 16 when exposed to waste solutions chemically similar to those in tank 16. The results of that program are the subject of a separate report.⁽¹⁾

It was positively concluded that tank 16 leaked because of cracks forming in highly stressed regions of the steel, under the chemical influence of the liquid contained in the tank.

Aside from the observation of leaking areas of tank 14, there is no direct evidence on the nature and cause of the leaks in this tank. However, the leaks in tank 14 were similar to those in tank 16, and were also located near the welds. Furthermore, the two tanks were constructed at the same time using similar techniques. The inference, then, is very strong that tank 14 also leaked because of stress corrosion cracks.

Tanks 9 and 10 also developed leaks, but there were none in those parts of the side walls that were inspected. These tanks were of a different design from that of tanks 14 and 16, and were constructed earlier. The cause of the leaks in tanks 9 and 10 is unknown; stress corrosion cracking is only one of several possible causes.

The complete absence of leaks in the rest of the cooled tanks at SRP does not necessarily require any significant difference in waste composition, storage conditions, steel composition, or construction methods. The possibility of such differences was the reason for considerable examination of tank histories and construction records. No correlation was found between these factors and the occurrence of leaks. It may be that many tanks which do not leak, actually do contain cracks in the weld-affected zone; this was not determined. As can be seen from Figures 14, 15, and 16, most of the cracks observed in the samples taken from tank 16 do not penetrate the steel or are too small to pass liquid. It is quite possible that the difference in the severity of cracking between a leaking and a nonleaking tank might be so small that examination of their histories would never reveal the difference in causative agents. The fact that the locations of leaks in a single tank were subject to a distribution other than random (see Figures 8 and 9) is good evidence that some very subtle or minor variation in a causative agent was enough to determine the presence or absence of leaks in a considerable area.

PREVENTION OF STRESS CORROSION CRACKING

Stress cracking in tanks of the type described in this report can be prevented by full stress relieving by heating the tank as a unit after welding has been completed. Such stress relieving has been applied commercially to tanks as large as these.

Laboratory experiments which verified the procedure and effectiveness of stress relieving tanks of this type are described in a separate report. ⁽¹⁾

ACKNOWLEDGMENT

The Engineering Assistance Section of the Savannah River Plant made a substantial contribution in the design and fabrication of the remote observation equipment. The Engineering Department in Wilmington published an interim report on waste leakage, and much of the work on stress corrosion cracking was done at the Savannah River Laboratory.

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FIG. 1 VERTICAL CROSS SECTION OF TANK 16

FIG. 2 GRAPHIC HISTORY OF TANK 16

Analysis			
Supernate		Sludge,	
Mar 20, 1958	Feb 26, 1960	Feb 26, 1960	
pH	>11*	>11	>11
Sp Gr	1.25	1.22	1.28
NaOH, g/l	16.6	27.6	23.3
Na ₂ CO ₃ , g/l	43.9	0	26.3
NaHCO ₃ , g/l	0	0	0
Cl ⁻ , g/l	0	-	-
Total solids, %	31.6	31.2	49.0 (calc)

* July 1959.

FIG. 3 GRAPHIC HISTORY OF TANK 14

FIG. 4 GRAPHIC HISTORY OF TANK 9

Supernate Analysis					
	Mar 1957	Apr 1957	Aug 1957	Apr 1958	Jul 1959
Sp Gr	1.23	-	-	1.271	-
CO ₃ ⁻⁻ , M	0.17	-	-	-	-
HCO ₃ ⁻ , M	0.2	-	-	-	-
CO ₂	*	-	-	-	-
NaOH, g/l	-	0	0	18.4	13
Na ₂ CO ₃ , g/l	-	18	56	57.8	68
NaHCO ₃ , g/l	-	16	8	0	0
Total solids, %	-	-	-	35.9	-

* Detected in Vapor.

FIG. 5 GRAPHIC HISTORY OF TANK 10

FIG. 6 PERISCOPE USED FOR TANK WALL INSPECTION

FIG. 7 TELEVISION CAMERA USED FOR TANK WALL INSPECTION

FIG. 8 FACE MAP OF TANK 16 WALL

FIG. 9 FACE MAP OF TANK 14 WALL

FIG. 10 WALL OF TANK 14. Note three leaks near lower horizontal weld and leaked material in annular space near dehumidifier duct. (Photographed through periscope.)

FIG. 11 LEAK POINT IN A VERTICAL SEAM OF TANK 16 PRIOR TO SANDBLASTING.
(Photographed through periscope, using 2 different magnifications.)

End View of Cutting Head

Cutting head Assembled in Mast

FIG. 12 DEVICE USED TO TREPAN SAMPLE FROM TANK WALL

FIG. 13 SAMPLE REMOVED FROM WALL OF TANK 16 (INTERIOR SURFACE AFTER SAMPLE WAS CLEANED). Approx 1X.

FIG. 14 PHOTOMICROGRAPH OF SAMPLE FROM WALL OF TANK 16.
Note pitting and cracking on exterior surface which resulted from
leakage of waste solution through main crack (at right side of
photograph). Approx 130X.

FIG. 15 PHOTOMICROGRAPH OF AREA NEAR ARC STRIKE ON TANK 16 WALL SAMPLE. Note major crack. Approx 23X.

FIG. 16 PHOTOGRAPH SHOWING NUMEROUS CRACKS ON INNER SURFACE OF TANK 16 WALL SAMPLE. Approx 2X.