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Hanford Tank Safety Project

A Survey of Available Information on Gas Generation in Tank 241-SY-101

March 1991

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On the cover:

An aerial photograph of the SY waste tank farm under construction (circa 1975) on the Hanford site in southeastern Washington State. Tank 241-SY-101 is the tank in the foreground to the right.

A Survey of Available Information on Gas Generation in Tank 241-SY-101

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Preface

This document, as the title implies, is a survey of the available information on gas generation in tank 241-SY-101. It was written for the expressed purpose of introducing the members of the newly formed Tank Waste Science Panel to the subject of episodic gas releases from tank 241-SY-101 and to the pertinent available information. Since this document was written, the understanding of the phenomenon has increased. Therefore, those familiar with the meetings of the Tank Waste Science Panel may note departures from the way the members of the Panel currently think about the subject. Subsequent documents will chronicle the evolution in the understanding by the members of the Panel on the subject of the chemical and physical mechanisms taking place in tank 241-SY-101 that give rise to the episodic release of gases.

Abstract

As a result of a concerted effort to determine the chemical and physical mechanisms underlying the generation and episodic release of gases from tank 241-SY-101, more commonly known as tank 101-SY, the Tank Waste Science Panel has been established at the Pacific Northwest Laboratory.^(a) Four of the members of this panel met to screen the available information on tank 101-SY and provide to the remaining members a shortened list of references that could be used to assess the mechanisms underlying the generation and episodic release of gases from tank 101-SY. This document is the result of this preliminary screening of information for the Tank Waste Science Panel and was provided to the Panel members at their first meeting.

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Introduction

In April 1990, Bond, Pruett, and Schulz (1990) wrote a letter to Mr. Michael Lawrence, then manager of the Richland Office of the Department of Energy, summarizing their findings concerning the safety of the Nuclear Waste Tanks at the Hanford Site. These findings prompted a concerted effort on the part of the Department of Energy (DOE) and Westinghouse Hanford Company to determine the mechanisms associated with the episodic release of potentially explosive gas mixtures from one of these tanks - tank 241-SY-101, more commonly known as tank 101-SY. As part of this effort a panel of expert chemists and engineers was assembled with the charter to develop a mechanistic understanding of the gas build up and release from tank 101-SY. This panel of experts is called the Tank Waste Science Panel. The members of this panel were selected by the staff at the Pacific Northwest Laboratory for Westinghouse Hanford Company. This report is the first in a series of reports that will document the progress by the members of the Tank Waste Science Panel to understand the mechanisms by which gases are generated and released from tank 101-SY.

Before the full panel met, four of the panel members met to screen the available information on tank 101-SY and provide to the rest of the panel members a shortened list of references as well as a discussion of the available information and a discussion of the preliminary results from this survey. This document is the result of this initial investigation and was given to the remaining Panel members prior to the first meeting of the Tank Waste Science Panel.

Structure of Report

This report is structured to achieve the stated goal. Background information and data concerning the history of the episodic release of gases from tank 101-SY are given in Sections 2.0 and 3.0. Laboratory experiments have been performed by several scientists at Westinghouse Hanford Company, formerly Rockwell Hanford Company. Results from the pertinent experiments are summarized in Section 4.0. Based on this information and data base, the Panel members formed a preliminary understanding of the mechanisms involved in the generation and release of gases from tank 101-SY (Section 5.0). The unresolved questions surrounding this information and data are summarized in Section 6.0. These open questions serve as a starting point to define a laboratory program in which the chemical and physical mechanisms in tank 101-SY will be determined.

General Background

Over the years since 1942, liquid and solid wastes have been accumulated from the various processes used at the Hanford Site to separate plutonium from uranium and the fission products in spent metallic uranium fuel. Significant amounts of these wastes were sent to single-shell tanks for storage. In the early 1970s, a decision was made to transfer all liquid waste from single-shell tanks to double-shell tanks. Before sending the waste to double-shell tanks, these wastes were to be concentrated using crystallizer-evaporators. The product of this process was a thick slurry, dubbed "double-shell slurry", consisting of a solution high in NaOH, NaNO₃, NaNO₂, NaAlO₂, dissolved organic complexants (EDTA, HEDTA, NTA, HOAcOH, etc.), and other salts (sulfates and phosphates). Other wastes resulting from current processes were similarly concentrated, and the aqueous portions were stored in double-shell tanks.

A variety of waste types have been generated over the years, but the key wastes of concern for tank 241-SY-101, commonly known as tank 101-SY, are the high-saline wastes indicated above and those wastes containing high organics. One such organic-containing waste (Table 1) was dubbed "complex concentrate." This waste originally contained about 6 M organic carbon after leaving the evaporator. Processing problems with this waste prevented it from being fully concentrated. That is, although the organic content was high, the salt concentrations were not as high as in the "double-shell slurry."

In the mid 1970s, the first crystallizer-evaporator was constructed and cold tested. In 1977, the evaporator was tested with radioactive waste and the product was pumped to tank 101-SY for storage. Shortly after transferring approximately 250,000 gallons of double-shell slurry to tank 101-SY (Simpson 1984), the waste volume began to increase. The institutional controls, however, were set to alarm for a decrease in volume, which usually signifies a leak. Initially, it was administratively difficult to deal with a tank in which the waste volume was increasing. Later that year, complex concentrate was added to the tank (Simpson 1984, Table 1^(a)) in an effort to dilute the waste and quench the slurry growth. Three more waste additions were made between 1977 and 1980 (see Table 2). During this time period, a crust developed on the top of the liquid waste and has grown in thickness, further complicating an already difficult-to-interpret situation. In March 1981, the first growth and collapse cycle was observed; these cycles have continued more or less regularly since that time. Again, institutional alarms are set for a decrease in volume, but there is no evidence for a leak in the annulus between the double shells.

Table 1. Composition of Tank 102-AZ

<u>Constituent</u>	<u>Concentration (mol/L)</u>	<u>Constituent</u>	<u>Concentration (mol/L)</u>
Al	0.018	Mn	0.036
Ca	0.016	Na	7.62
Cr	0.0028	Ni	0.010
Fe	0.069	Zn	0.0017
K	0.017	Zr	0.0011
Mg	0.0014	TOC	6.0

(a) References are given with the Table/Figure/Page of interest noted.

Table 2. Contents of Tank 101-SY

Action/ Comp.	Waste 1	Waste 2	Waste 3	Waste 4	Waste 5
Date	APR 1977	NOV 1977	JUN 1978	AUG 1978	OCT 1980
Volume ^(a)	275.5	365.6	133.3	59.5	231.3
NaOH ^(b)	3.02	2.10	2.11	4.50	3.76
NaAlO ₂	1.46	0.994	0.409	3.28	3.59
NaNO ₂	2.72	1.86	1.10	5.23	5.47
NaNO ₃	2.72	3.44	3.92	4.88	4.95
Na ₂ CO ₃	0.8	0.474	0.932	0.002	0.14
Na ₂ SO ₄	0.07	0.065	0.07	0.805	0.07
Na ₃ PO ₄	0.152	0.148	0.066	0.123	0.30
TOC ^(c)	14.52	26.32	34.27	26.6	18.01
⁹⁰ Sr ^(d)	0.013	0.16	0.074	0.45	0.005
¹³⁷ Cs	0.74	1.76	0.28	0.28	1.11

(a) Volumes are in 1000's of gallons.

(b) Concentrations are in moles per litre.

(c) Total Organic Carbon values are in grams of carbon per litre.

(d) Radionuclide concentrations are in curies per litre.

Observations from Tank 101-SY

As indicated above, slurry growth and collapse cycles have occurred since March 1981, and slurry growth has been taking place since the first waste was placed in the tank in April 1977. There are two measurement techniques for obtaining waste levels in these tanks: conductivity probe and manual tape measurement. Since the wastes in these tanks are normally highly saline liquid wastes, a conductivity probe is a reliable method of finding the liquid height. However, this waste is completely crusted over with a solid of unknown composition and an estimated thickness between 4 in. and 4 ft. Thus, for tank 101-SY, both the manual tape and the conductivity probe have been used as mechanical means of measuring the waste level. The results of these measurements are documented by Reynolds (1990, Page 16).

In an attempt to quench the cyclic growth and collapse of the waste in the tank, pipes were placed into the tank through risers; these pipes extended from the top of a tank riser to the bottom of the tank. Water was added periodically through these pipes over the time period from February 1985 to mid 1988 (Leggett 1990, Page 13). Air injection was attempted as a method to sparge the gases from the tank and ameliorate the gas release (Leggett 1990, Page 13). Both techniques failed to stop gas release. Water injection tended to keep the amplitude of the gas release low and the gas release frequency higher than air injection. Air injection appeared to raise the waste height baseline on which the cycles were occurring and in January 1989 air injection was halted. Since that time, the waste height baseline has been decreasing toward the value observed before the start of air injection (Leggett 1990, Page 13). Interestingly, water injection at various depths during August and September 1984 seemed to promote an uneven decrease in the waste level (Sasaki 1984, Page 2 and Table 1).

During a gas release, the gas composition has been measured. The principal components are, in addition to air, H_2 and N_2O (Leggett 1990) with minor amounts of NH_3 (about 2000 ppm measured during air lancing [Campbell 1987]). The major portion of the gas release occurs in about 3 h (Leggett 1990) and has been estimated to involve about 7000 cubic feet of gas at ambient pressure. Although the tank is exhausted and the nominal steady state pressure is about -2.5 in. of water, the pressure has been noted to rise to a positive 0.1 in. of water during a release.

The contents of the tank have been sampled by punching a hole in the crust and inserting a pole with a bottle attached. When the bottle was at the desired sampling level, a rubber stopper was pulled from the bottle. In this manner, samples from three liquid layers (top, middle, and bottom) were obtained and are reported by Mauss (1986). It is interesting to note that the viscosity of the solution in each layer exhibits different behavior. The solution from middle layer exhibits Newtonian behavior, i.e., the viscosity is independent of shear rate, whereas the solutions from the upper and lower levels exhibit non-Newtonian rheological properties (Mauss 1986). The total organic content is consistent with an analysis by Strachan et al. (1986, Figure 3.2 Page 8 and Table 3.1 Page 9) of a complex concentrate waste from tank 107-AN, which contains a waste similar to the complex concentrate waste in tank 101-SY. Strachan et al. (1986) also report the organic content of the waste by chemical species. These species represent some of the organics originally used in the chemical processes and some of the decomposition products of those organics (Table 3).

Table 3. Organic Content^(a) for Tank 107-AN Waste

	Concentration ^(b)	
	mM	µC/L
Chelating/Complexing Agents		
Citric Acid	64.39	4.61
N-12-Hydroxyethylenediaminetetraacetic Acid (HEDTA) ^(c)	37.53	4.53
Ethylenediaminetetraacetic Acid (EDTA)	31.41	3.77
Methane Tricarboxylic Acid	17.32	1.45
Nitriotriacetic Acid (NTA)	7.33	0.53
Chelator Fragments		
Ethylenediaminetetraacetic Acid (ED3A) ^(d)	17.91	1.72
N-12-Hydroxyethyl)ethylenediamine-N ^o N ^o -diacetic Acid (HEDDA) ^(e)	2.39	0.26
N-(ethylene)ethylenediaminetetraacetic Acid (H ₂ DTA)	2.28	0.23
N-12-Hydroxyethyl)iminodiacetic Acid (HEIDA) ^(f)	2.14	0.18
N-12-Hydroxyethyl)-N ^o -imethyl)ethylenediamine-N ^o N ^o -diacetic Acid (MeHEDD'A) ^(g)	1.83	0.20
N-Imethyl)ethylenediamine-N ^o N ^o -diacetic Acid (MeEDD'A) ^(h)	1.02	0.08
Iminodiacetic Acid (IDA)	187.90	10.92
Molecular Weight (MW) Species⁽ⁱ⁾		
A: MW 122	0.90	0.04
F: MW 173	0.23	0.02
J: MW 247	0.90	0.08
Carboxylic Acids		
Docos-13en-oic Acid	2.50	0.67
Hexanedioic Acid	2.04	0.15
Hexaoecanoic Acid	2.04	0.39
Phthalic Acid	1.10	0.10
Nonanedioic Acid	0.83	0.07
Tetraoecanoic Acid	0.68	0.12
Pentanedioic Acid	0.60	0.04
Octaoecanoic Acid	0.54	0.11
Hydroxybutanedioic Acid	0.33	0.01
Butanedioic Acid	0.10	0.01
Alkanes		
nC ₂₃ - nC ₃₅	7.77	2.50
Phthalate Esters		
Dibutylphthalate	1.24	0.23
Diethylphthalate	0.05	0.01
Total Organic Carbon (TOC)	3670	44.00

% TOC Identified - 75.1%

- (a) Identified as methyl esters (BF₃/methanol)
- (b) Quantitation based on six GC analyses with FID detection: the standard deviations are: citric acid, ±5.7%; EDTA, ±7.0%; HEDTA, ±12.2%; NTA, ±3.9%; ED3A, ±2.6%; MeEDD'A, ±9.1% Methane Tricarboxylic Acid, HEDDA and MeHEDD'A, ±7.0% each; and MW species, ±7.0% each.
- (c-h) Identified by GC-MS and FT-IR as: (c) MW 288 lactone; (d) MW 244 lactam; (e) MW 230 dehydrated dimethylester; (f) MW 173 lactone; (g) MW 230 lactone; (h) and MW 186 lactam.
- (i) MWs assigned to unknown chelator fragments on the basis of electron impact (70 eV) GC-MS.

In 1990, a temperature profile was taken of the waste in tank 101-SY. These temperature data were collected before and after a gas release (Leggett 1990, Page 28). From these data there appear to be three distinct regions in the tank. At the top is the crust, which appears to have good thermal insulator properties. At the bottom of the tank is a layer in which heat appears to be generated and lost through the bottom of the tank and to the middle layer. The content in the middle part of the tank appears either to have relatively high thermal conductivity or to be a well-stirred system. After the gas is released, the thermal profile is relatively uniform.

Laboratory Experiments

Since 1977, work has been carried out in the laboratory in an attempt to understand the phenomena of slurry growth and gas generation. However, these studies are not complete and several open questions remain. The studies that have been completed are summarized in this section.

Laboratory studies indicate that hydroxyethylenediaminetetraacetic acid (HEDTA) and hydroxyacetic acid (HOAcOH) are needed for the waste to generate gases, e.g., H_2 , N_2O , and NH_3 , in quantities sufficient to measure in the head space above the reactants (Jansky and Meissner 1983 and 1984). These gases are generated using nonradioactive chemicals, thereby implying that chemical reactions constitute one of the major sources of these gases. Gases are not generated when HEDTA and HOAcOH are absent and other organics are present. Slurry growth, i.e., expansion of the liquid phase without gas generation, may occur in the absence of organics, but the evidence is weak. In the case of no organics, it is uncertain as to what gases, if any, cause slurry growth.

In one of the most complete pieces of work, Delegard (1980) found the gas generation to be first order in the $NaAlO_2$ concentration. The dependence of the reaction rate on the other components of the solution was more complex. In particular, the dependence on the NaOH concentration increased linearly to about 1 M, was a maximum at about 1.5 M, and decreased above 1.5 M. Delegard suggested an activated complex involving AlO_2^- and HEDTA as a mechanism for gas generation. The temperature dependence of the gas generation rate was also investigated and found to exhibit Arrhenius behavior. The temperature dependence of the slurry growth was not investigated.

The composition of the gases released from the slurry has been determined and found to vary as a function of time (Jansky and Meissner 1983). The gas mixture is H_2 -rich at the start. The relative concentration of the H_2 gas decreases with increasing time. The analyses, however, show the same gases are generated in the laboratory studies as in the tank, e.g., H_2 , N_2O , and NH_3 . Jansky and Meissner (1983) reported 900 ppm NH_3 gas using a Dräger tube. How these gases are generated under the oxidizing conditions prevalent in the tank is uncertain. However, Barney^(a) indicates that the high ionic strengths of these solutions causes the negative ΔG 's for reactions between HEDTA and nitrate/nitrite that yield H_2 gas.

A complication in the above studies should be noted here. In early studies carried out at 120°C, Teflon beakers were used as the containment vessels. Teflon is very permeable to ambient gases as well as to the gases generated during the experiment. Diffusion of these gases through the vessel walls could have affected the results.

Although it is not strictly a laboratory study involving slurry growth or gas generation, the phase diagram of the slurry system is important to understand. Barney (1976) carried out a series of experiments to help determine the best operating parameters for the crystallizer-evaporators. It is this study that enables one to best understand the genesis of the double-shell slurry that has been stored in the double-shell tanks at the Hanford Site. Figure 3 from Barney (1976) indicates that to obtain maximum waste reduction the evaporators should operate with a solution that is about 2 M NaOH or greater. Below 2 M

(a) Barney, G. S. 1990. Personal communication.

NaOH various phases of hydrated alumina precipitate. One of these phases is very gelatinous and can cause severe processing problems. Above about 3 M NaOH and 3 M NaAlO₂, NaAlO₂ precipitates but does not cause processing problems. Other salts also precipitate during the processing; these include NaNO₃, NaNO₂, and other minor sodium salts of constituent ions such as sulfate and phosphate.

Preliminary Findings

The following preliminary findings have been made by the authors:

- 1) Slurry growth and gas release require the presence of organics. In particular, laboratory studies indicate that gas release requires that HEDTA, HOAcOH, or similar organic be present. Inconsistent with this conclusion, however, is the apparent 100-h half-life of HEDTA measured in laboratory studies (Delegard 1980). A 100-h half-life is inconsistent with the almost 10-yr history of gas releases from Tank 101-SY.
- 2) The latest analyses from tank 101-SY indicate that the total organic content of the solution is greater at the bottom of the tank than at the other two sampling points. However, the organic speciation has not been performed for this tank, although the speciation has been done for a similar waste in Tank 107-AN.
- 3) The thermal profile of the waste as a gas release approaches suggests that the gas is generated in the bottom layer (~200 in.) of the tank. Gas bubbles would be expected to lower the thermal conductivity of the solution and the radionuclide decay heat generation would cause the temperature to rise in an area of low thermal conductivity. Furthermore, the profile suggests that the heat is lost from the bottom layer in both the upward and downward directions, and that the solution from a depth of 200 in. to 360 in. from the bottom is either well stirred or has very high thermal conductivity. The sharp thermal gradient at the crust suggests that it is a good thermal insulator or at least much better than the solutions under it. The temperature profile after a gas release indicates either that the system becomes well mixed as a result of that gas release or that the gas dissipates a substantial amount of the heat. These observations do not preclude gas generation and steady-state release above the bottom layer.
- 4) Gas volumes and calculations suggest a concentrated source of gas near the bottom of the tank where the overburden pressure is likely to approach 2 atmospheres. This interpretation of the data is at least consistent with the thermal profiles, but the accumulation of gases under the crust cannot be ruled out at this time as a contributing part of the gas release mechanism.
- 5) The gas volumes and compositions are not known precisely. The composition appears to vary with time, but is dominated by H_2 and N_2O with minor amounts of NH_3 .

Open Questions

Below are listed some of the open questions that need to be addressed by the Tank Waste Science Panel:

- 1) What is the gas composition exiting the tank before, during, and after a gas release? Complementary instrumentation, e.g., one instrument that can be used to detect N_2O and NH_3 , for instance, and one for H_2 , is needed to reliably quantify the gases that are released. Sufficient samples need to be collected so that statistics can be used to determine uncertainties. Instruments/techniques with lower detection limits are needed especially before and after a gas release.
- 2) What are the composition and physical properties of the crust, e.g., chemical composition, phases present, porosity, permeability, etc?
- 3) Where is the gas being generated in the tank? Samples from the bottom, middle, and top need to be collected as a function of time.
- 4) What gases cause slurry growth? Are they different from those released during a gas release? If the gases are the same for both phenomena, what conditions cause growth and what conditions cause gas release?
- 5) What physical mechanisms can give rise to the observed releases? Which of these are credible under the conditions of tank 101-SY?
- 6) What happens to the solution concentrations as a function of time? Are particular species being consumed by the reaction? How long will the reactions be sustained?
- 7) What role do trace elements play in the slurry growth and gas release mechanisms?
- 8) Is corrosion of the tank walls/bottom a major contributor to the gas generation? What is needed to rule out corrosion? If corrosion cannot be ruled out, is the corrosion substantial enough to compromise the tank integrity?
- 9) What are the roles of radiolysis and radiolytic decomposition of organics? To what extent are these mechanisms contributors to the problems in tank 101-SY or any other tank?
- 10) Are the decomposition products of HEDTA and HOAcOH major actors in the gas release or are they inert? What about the decomposition products of the other organics?
- 11) What research is needed to adequately define the problem and define a safe and reliable remediation?
- 12) Comparing the results from recent analyses of tank 101-SY contents with earlier analyses suggests that there are confounding analytical errors for nitrate, nitrite, and total organic carbon. The difference between the early and the recent analyses suggests that about 1.4 M nitrate, 0.6 M nitrite, and 0.5 M organic carbon are unaccounted. These values cannot be attributed to gas release because the values would suggest much higher gas releases than observed. Are these new values for the tank

contents indicative of reactions taking place in the tank or are they indicative of the type of analytical error that is expected for these solutions or possible inhomogeneities in the tank?

- 13) The nitrogen chemistry is of interest. The following are some of the nitrogen compounds present in the waste:

<u>Oxidation State</u>	<u>Species Present</u>	
	<u>Vapor Phase</u>	<u>Aqueous Phase</u>
-1	NH ₃	Organic Amines
0	N ₂	--
+1	N ₂ O	--
+2	NO	--
+3	--	NO ₂ ⁻
+4	NO ₂	--
+5	--	NO ₃ ⁻

What chemistry can give rise to this suite of oxidation states?

- 14) Is it possible that the reactions indicated by the events in Tank 101-SY are taking place in other tanks?

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