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Hanford Tank Safety Project

Minutes of the Tank Waste Science Panel Meeting July 20, 1990

February 1991

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Pacific Northwest Laboratory
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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.

**Minutes of the
Tank Waste Science Panel Meeting
July 20, 1990**

Compiled by
D. M. Strachan
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February 1991

Prepared for
the U.S. Department of Energy
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Summary

The second meeting of the Tank Waste Science Panel was held July 20, 1990. Science Panel members discussed the prioritization of various analyses to be performed on core samples from tank 101-SY, and were asked to review and comment on the draft Westinghouse Hanford Company document "Analytical Chemistry Plan." They also reviewed and discussed the initial contributions to the report titled *Chemical and Physical Processes in Tank 101-SY: A Preliminary Report*.

Science Panel members agreed that a fundamental understanding of the physical and chemical processes in the tank is essential, and strongly recommended that no remediation measures be taken until there is a better understanding of the chemical and physical phenomena that result in the episodic gas release from tank 101-SY.

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Minutes of the Tank Waste Science Panel Meeting July 20, 1990

Introduction

The second meeting of the Tank Waste Science Panel (Science Panel) was conducted July 20, 1990, at the Registry Hotel, Denver, Colorado. The agenda is provided in Attachment A, and a list of attendees is given in Attachment B.

The principal objectives of this meeting were to: 1) review and discuss any new information related to the physical and chemical processes occurring in tank 101-SY; 2) discuss individual members' preliminary contributions to the report titled *Chemical and Physical Processes in Tank 101-SY: A Preliminary Report*; 3) determine additional contributions needed to complete the report; and 4) determine the schedule for completion of the report. In addition, the Science Panel conducted general business activities, reviewed and commented on the draft report titled *Tank Waste Science Panel, Summary of Activities, June 26-27, 1990*, and reviewed and commented on the chemical and physical analyses proposed to be conducted on core samples obtained from tank 101-SY.

Science Panel activities conducted in each of the agenda items shown in Attachment A are discussed in the following text.

General Panel Business

Science Panel members have received inquiries and suggestions related to the processes occurring in tank 101-SY from other technical personnel. The Science Panel members agreed that such inquiries will be conveyed to T. H. Dunning, Jr., Science Panel Co-Chairman, for subsequent referral to the appropriate subcommittee chairs for review and consideration.

Several individuals have been suggested as additional members of the Science Panel or as consultants to the Science Panel. Copies of brief vitae on several of these individuals were distributed for information and review. In the general discussion, the Science Panel members agreed that there is a strong need for a very good inorganic solution chemist, a chemist with expertise in the thermodynamics of high ionic strength solutions, a radiation chemist with expertise in the radiation chemistry of nitrate/nitrite-containing solutions, an engineer/physicist with expertise in fluid dynamics, and others. It was concluded that T. H. Dunning, Jr. would contact some of the individuals who have been suggested with the intent of determining their potential interest and availability; no commitments would be made.

Comments regarding the draft report of the June 26-27, 1990, meeting of the Science Panel were solicited. Review comments received either at the present meeting or transmitted to either D. M. Strachan, Science Panel Executive Secretary, or L. G. Morgan, Project Manager, will be incorporated in the report and the final report will be transmitted to the Westinghouse Hanford Company (WHC).

The Science Panel members also discussed the public availability of the minutes of the Science Panel meetings. The members agreed that the minutes, or reports of Science Panel activities, should be

published as publicly available reports through normal Pacific Northwest Laboratory (PNL)^(a) and Department of Energy (DOE) clearance procedures.

Proposed Chemical and Physical Analyses of Tank 101-SY

WHC formally requested that the Science Panel review the draft "Analytical Chemistry Plan" for core samples obtained from tank 101-SY. The emphasis of the Science Panel review was to insure that the data needed for development of models or descriptions of the chemical and physical processes occurring in tank 101-SY would be obtained, and that the priorities assigned to various analyses (a concern only if sample size is limited such as may occur in sampling the crust region of tank 101-SY) are defensible. See Attachments C and D for copies of the draft "Analytical Chemistry Plan" and the proposed prioritization of analyses, respectively.

The members of the Science Panel did not have time during this meeting to review and comment on the draft "Analytical Chemistry Plan" itself. Members interested in the plan took copies with them; any review comments will be provided directly to L. G. Morgan, Project Manager, for transmittal to WHC. The review of the analyses proposed and the prioritization of these analyses led to several recommendations by the Science Panel.

In general, the Science Panel members felt that there should be two priority rankings - one concerning the importance of performing the analysis and one that takes into account the feasibility of performing the analysis. Several valuable analyses had received low priority rankings because the Science Panel members recognized that the analyses cannot be performed because the instrument or procedure is not in place, especially for radioactive samples, or there would be insufficient sample for the full suite of analyses. A two-column ranking system would better illustrate the problems associated with a sample that is radioactive or too small while still indicating the importance of the information that would be obtained from the proposed analyses.

The Science Panel strongly recommended that any excess sample from the core sampling operation be properly archived. It is expected that not all of the core samples will be analyzed completely. However, there may be a need to analyze these samples, as well as the samples for which analytical data are obtained, at some future date. In addition, these analyses must meet all technical and quality assurance requirements and the results should be subjected to external peer review. To insure that this is accomplished, the individuals handling the samples, both in the actual tank sampling operations and the analytical laboratories, need to be made aware of the importance of these samples. In the analytical laboratories, this importance could be communicated by assigning a chemist to be actively involved in each of the analyses to be performed. This indicates to those performing the analyses that the samples being analyzed are not routine, but are special and the results of the analyses are very important.

The Science Panel decided that the following analyses should be added to those already designated to be performed (see Attachment D for those analyses designated to be performed):

- analyses for hydrogen peroxide, organic hydroperoxides, and organic diperoxides

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- analyses for dissolved gases, particularly hydrogen, nitrous oxide, ammonia, and oxygen, nitrogen and carbon dioxide
- determination of shear stress versus shear rate down to zero shear rate for rheology
- particle size analyses
- thermogravimetric analysis/differential scanning calorimetry (TGA/DSC) to determine potential for ignition/explosion
- total organic carbon analyses at both WHC and PNL

In general, the panel had indicated that the analyses listed above are needed during in the first meeting of the Science Panel. This is documented in the report *Tank Waste Science Panel, Summary of Activities, June 26-27, 1990*. These analyses are discussed further in the following text.

The analyses for hydrogen peroxide, organic hydroperoxides, and organic diperoxides are needed to determine if these species exist in the waste. These species are potential products of radiolysis and are "high energy" species whose presence would indicate the need for additional precautions in the management of these wastes and consideration of potential mitigation processes. Conversely, the absence of these species eliminates this concern.

Gas analyses are needed for gases that are either trapped or dissolved in the waste, and for those evolved during the TGA/DSC analyses. It was recommended that Science Panel member E. P. Horwitz, Argonne National Laboratory (ANL), be contacted by WHC and/or PNL to obtain additional information on analytical techniques developed or used at ANL that relate to these gas analyses.

Expert interpretation of the results of the TGA/DSC analyses is needed to determine the potential explosivity or ignitability of the materials analyzed. Not all exotherms noted in such DSC analyses indicate the potential for explosion or sustained exothermic reactions. Further, the differential thermal analysis (DTA)/TGA/DSC tests need to be conducted in both air and in an atmosphere of inert gas. Interpreting the results of such tests would provide information important to determining whether the tank plenum should be filled with an inert gas and also whether additional moisture should be added to the crust.

Determination of the particle size of solids, particularly in the region near the bottom of the tank, is necessary for the development of a model for the physical processes occurring in tank 101-SY. In addition, the Science Panel members indicated that they would still like to have a sample obtained and analyzed from the immediate surface of the crust. Those analyses would give information on the condition of the crust, i.e., moisture content, presence or absence of ammonium nitrate, content of sodium carbonate, etc.

As the laboratory research program to determine the chemical and physical mechanisms progresses, there will be a need to compare the information from the actual samples with the results from the laboratory research program. Results from the laboratory studies may also suggest analyses that should be performed on the actual samples.

The results of the Science Panel members' review of the chemical and physical analyses to be obtained on core samples from tank 101-SY will be formally transmitted to WHC by L. G. Morgan, Project Manager.

Physical Processes

The initial contributions to the Science Panel's report titled *Chemical and Physical Processes in Tank 101-SY: A Preliminary Report* were reviewed and discussed. The discussion focused on the potential mechanisms of gas generation and release in tank 101-SY. The potential mechanisms of gas generation are discussed in the chemical processes section of this report.

The members of the Science Panel did not draw more detailed conclusions regarding an understanding of the physical processes occurring in tank 101-SY, but the following text summarizes the discussions related to these physical processes.

The Science Panel again emphasized the need to know the rheological properties of the fluid portion of the waste contained in tank 101-SY; this is particularly important in the lower region of the tank. The viscosity of the fluid regions needs to be measured at very low shear rates and the data extrapolated to zero shear rate. This information is required to determine the degree to which the fluids behave as Newtonian fluids. Determination of particle size of solids in these fluid regions is also required to calculate settling rates.

Recent and additional data on the vertical temperature profile of the waste in tank 101-SY were also reviewed but no additional insights were obtained. These data indicate a vertical temperature profile similar to that observed previously.

The members of the Science Panel indicated that a more mechanistic understanding of the physical processes occurring in tank 101-SY required both a modeling effort and experiments conducted at an appropriate scale. The modeling effort can be initiated now using available information and existing calculational techniques as an aid to determine the plausibility of the proposed hypothesis on the physical process of heat buildup in the lower region of waste in tank 101-SY and gas release from this region at more-or-less regular intervals.

Experimental studies to simulate the waste behavior should be conducted at an appropriate scale. These experiments must be carefully planned and coordinated with the modeling effort. The exact scale of the experiments needed may require using a waste volume of the order of 100 L. It is likely that these studies should be conducted initially using nonradioactive materials; and if effects of radiation are included, these studies will require an external radiation source or the use of actual waste.

Total gas flow rate measurements are needed in tank 101-SY so that during a gas release event the total volume of gas (and its composition) can be determined accurately.

The Science Panel requested information on the status of their recommendations to obtain pressure data at various levels within tank 101-SY and to measure the permeability of the crust using helium injection. The Science Panel was informed that these recommendations had been transmitted to WHC, but to date, no action had been taken or planned. The Science Panel reiterated the value of the information that would be obtained from these measurements and requested that these recommendations again be transmitted to WHC.

The Science Panel was also informed that an individual at Sandia National Laboratory had been identified who indicated that he had an existing computer code (model) that can be used to understand the

physical processes occurring in tank 101-SY. A member of the Science Panel was identified to contact this individual and determine if the model is applicable.

Chemical Processes

Discussion on chemical processes occurring in tank 101-SY focused on potential generation mechanisms for the production of hydrogen and nitrous oxide. Initially, i.e., during the first meeting of the Science Panel, the Science Panel members thought that radiolysis was not a major cause for the production of hydrogen gas. However, from a review of available data and some preliminary calculations, it appears that radiolysis may have a more dominant role in the production of hydrogen gas than originally estimated. Calculations previously performed by a WHC staff member^(a) suggest that almost any quantity of hydrogen gas can be generated depending on the "G-value"^(b) that is selected from the literature for the radiolysis of water in an alkaline solution. The quantities of hydrogen that could be produced include the quantity observed in the gases released from tank 101-SY. Further support for the need to determine the role of radiolysis in the production of hydrogen is given when the purely chemical mechanisms are considered.

Potential chemical mechanisms for the production of hydrogen remain undetermined. Delegard's (1980) studies suggest that N-(2-Hydroxyethyl)ethylenediaminetriacetic acid (HEDTA) and glycolic acid (hydroxyacetic acid, HOAcOH) participate in the chemical reactions to generate hydrogen and nitrous oxide. The implication that these complexants are necessary to produce hydrogen and nitrous oxide, whereas other complexants such as ethylenediaminetetraacetic acid (EDTA) do not, suggests that an alcohol group is needed on the complexant to facilitate the reaction. However, calculations based on Delegard's (1980) work and the estimated quantities of HEDTA and glycolic acid in tank 101-SY indicate that only about 20% of the gases could be coming from the chemical reactions proposed by Delegard (1980). Unfortunately, Delegard's (1980) proposed reactions remain unverified because he did not determine what organic species were being generated during the reactions.

The Science Panel recommended that the flow sheets and chemical inventories for B-Plant be reviewed to determine if the quantity of glycolic acid estimated to have been added to tank 101-SY is reasonable. If glycolic acid was used as a buffering agent in the B-Plant flow sheet, there may be significantly greater quantities in tank 101-SY (and other tanks) than originally estimated.

A chemical reaction mechanism that results in the generation of nitrous oxide and other constituent gases was proposed and presented by one of the Science Panel members. The proposed mechanism offers a plausible explanation for the chemical production of nitrous oxide that is consistent with the previous results that indicate only complexants containing an alcohol group participate in the reactions. The proposed mechanism is under review by the members of the Science Panel. Of particular interest, the proposed chemical mechanism can be tested in a series of critical experiments to determine its validity.

In addition to the mechanism proposed by a member of the Science Panel, the potential role of transition metal ion reactions with the organics was briefly addressed. The Science Panel was provided a

(a) Gough, G. 1990. Internal letter to G. L. Dunford, "Hydrogen from Radiolysis in Tank 101-SY," 82314-90-035. Westinghouse Hanford Company, Richland, WA.

(b) G-value. G = number of molecules produced or destroyed by 100 electron-volts of energy absorbed.

brief write-up by a PNL staff member regarding the potential reaction chemistry of polyoxomolybdates in the waste tanks (see Attachment E) for further information and consideration.

One member of the Science Panel indicated that chemical concentration cells (i.e., an electrochemical reaction mechanism) in the waste could also be a potential source of the gases generated. This potential reaction mechanism could, in principle, be determined experimentally in the tank.

Finally, the issue of providing an inert gas cover in the tank plenum was discussed. Some members of the Science Panel strongly support the addition of an inert gas to the tank plenum to preclude the reaction of oxygen with waste constituents that could result in additional safety concerns.

Preparation of Preliminary Report

The Science Panel is actively working to meet its objective of preparing an initial draft of the report titled *Chemical and Physical Processes in Tank 101-SY: A Preliminary Report*. The report will contain all the information currently known about the physical and chemical processes occurring in tank 101-SY. In addition, the report will contain recommendations for studies and measurements needed to answer the unresolved questions and issues related to the behavior of tank 101-SY. The individual subcommittee chairs will again receive and collate individual contributions to the report and transmit this material to T. H. Dunning, Jr., Science Panel Co-Chair, for incorporation into an initial draft report; these contributions are to be received by T. H. Dunning, Jr. by August 3, 1990.

The initial draft of the report is expected to be ready for review by the Science Panel members by mid-August 1990. Review comments will be received and addressed and the Science Panel will meet again either in late August or early September 1990 (dependent on individual Science Panel members schedules) to finalize the report.

Action Items

The following actions were agreed to by the members of the Science Panel:

- Contributions by individual members to the initial draft of the Science Panel's report will be submitted to the respective sub-committee chairs.
- Sub-committee chairs will transmit the collated report material to T. H. Dunning, Jr. not later than August 3, 1990.
- T. H. Dunning, Jr. will integrate the various contributions into an initial draft of the report and submit the report to the members of the Science Panel for review and comment by about mid-August 1990.
- Individual Science Panel members will review and comment on the initial draft report by late August 1990.
- The Science Panel will meet either in late August or early in September 1990 to finalize the report.
- L. G. Morgan offered to have PNL conduct a comprehensive review and evaluation of the literature regarding radiolysis of solutions relevant to the tank waste. This offer was accepted by the Science Panel and will be initiated.

- A member of the Science Panel was delegated to contact the individual at Sandia National Laboratory to determine the applicability of a computer code (model) to model the physical processes occurring in tank 101-SY.
- A member of the Science Panel was delegated to search for an expert analytical organic chemist who would be able to advise the Science Panel on appropriate analytical techniques to determine and quantify the various organic species in solutions such as those found in tank 101-SY.
- A member of the Science Panel recommended that an individual identified at W. R. Grace Co. be contacted for advice regarding analyses of various complexants. It was agreed that PNL would make this contact.
- The summary of the first Science Panel meeting will be revised per comments received from members of the Science Panel and the final report will be transmitted by PNL to WHC.
- The Science Panel's recommendations regarding the analyses to be conducted on core samples from tank 101-SY and their prioritization will be transmitted by PNL to WHC.
- PNL will transmit to WHC the necessity of conducting modeling, laboratory studies, and obtaining pertinent information from tank 101-SY and the various recommendations of the Science Panel.

Recommendations

The following recommendations were provided by the members of the Science Panel:

- A fundamental understanding of the physical and chemical processes is essential and is not just for "academic interest."
- Work related to developing an understanding of the physical and chemical processes occurring in tank 101-SY should not be limited to WHC or PNL. The studies should be conducted based on available expertise, interest, and technical credibility.
- An understanding of the physical and chemical processes will require adequate resources and time; it is not a short-term effort.
- Some Science Panel members strongly recommend providing an inert gas in the tank plenum to preclude additional safety concerns related to the reaction of oxygen with waste constituents.
- The Science Panel strongly recommends that no remediation measures be taken until there is a better understanding of the chemical and physical phenomena resulting in the episodic gas release from tank 101-SY.

Reference

Delegard, C. 1980. Laboratory Studies of Complexed Waste Slurry Volume Growth in Tank 241-SY-101, RHO-LD-124, Rockwell Hanford Operations, Richland, WA.

Attachment A - Agenda

Agenda

*Tank Waste Science Panel
20 July 1990
Registry Hotel, Denver, Colorado*

- 8:00 Breakfast, Room 427
- 8:30 General Panel Business
TH Dunning, Jr.
- 9:00 Proposed Chemical & Physical Analysis of Tank 101-SY
LG Morgan
- 9:30 Physical Processes Involved in Gas Generation
WJ Thomson
- 11:00 Chemical Processes Involved in Gas Production
WW Schulz
- 12:30 Lunch, Room 427
- 1:30 Discussion of Preparation of Preliminary Report
TH Dunning, Jr.
- 3:00 End of Meeting

Attachment B - Attendees

Tank Waste Science Panel Members

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ATTACHMENT C - ANALYTICAL CHEMISTRY PLAN

Note: The following document has been provided for review and comment by the Tank Waste Science Panel. It has not completed internal Westinghouse Hanford Company reviews and is subject to change. Its contents should not be released without permission of the Westinghouse Hanford Company.

**ANALYTICAL CHEMISTRY PLAN
FOR TANK 101-SY**

B. W. Hall
G. D. Johnson

July 17, 1990

ANALYTICAL CHEMISTRY PLAN

I. INTRODUCTION

The purpose of this document is to provide a description of the procedures and analyses to be used for the core samples obtained from the 101-SY double shell waste tank. The basis for this work is given in Section 4 of the document "Safety Improvement Plan - Hydrogen In Waste Tanks," Rev. 1 (Ref. 1), which was issued on June 6, 1990. To assure that the core drilling of tank 101-SY can be conducted with minimal risk to employees, the public and the environment a readiness review will be conducted to assure that these operations can be accomplished within the limits imposed by the Operational Safety Requirements.

II. OBJECTIVE

The objective of the analytical chemistry work is to provide the requisite data for the tasks working on safety, modeling and engineered remedies.

High priority has been placed on early characterization of the crust for both safety and remedial work. Since the current core drilling equipment does not assure that a total crust sample will be obtained, efforts are underway to conduct the drilling such that two core sampling operations will be conducted on the crust. This is needed to provide sufficient material for the physical and chemical tests.

The data requirements for the safety, modeling and engineered remedies tasks were recently discussed at a meeting (Ref. 2) and a summary of these needs are listed below.

1) Safety Analysis Needs

- * Physical Properties of the Crust
- * Mechanical Properties of the Crust
- * Presence of Heat Sensitive Materials
- * Presence of Shock Sensitive Materials
- * Toxicological Components and Aerosols Subject to Release
- * Flammability of Materials

2) Engineered Remedial Actions

- * Requirements for the Crust
 - * Specific Gravity
 - * Thickness
 - * Compressive Strength
 - * Penetration Resistance
 - * Dissolution Properties

* Requirements for the Slurry

- * Specific Gravity
- * % Settled Solids
- * Solids Settling Rate
- * Shear Stress vs. Shear Rate Rheogram
- * Particle Size Distribution
- * pH

* Requirements of the Sludge

- * Specific Gravity
- * Solids Settling Rate
- * % Settled Solids
- * pH
- * Wt. % and Vol. % Centrifuged Solids
- * Shear Stress vs. Shear Rate Rheogram
- * Particle Size Distribution
- * Shear Strength
- * Penetration Resistance
- * Abrasive Behavior (Miller No. for Worst Case)

3) Mechanistic Modeling

* Requirements for the Crust

- * Composition (anions, cations, organics, radionuclides)
- * Density
- * Wt. % Water
- * Differential Scanning Calorimetry/Specific Heat
- * Thermal Conductivity

* Requirements for the Slurry

- * Composition
- * Solids Settling Rate
- * Density of Both Phases
- * Polarized Microscopy (bubbles)
- * Specific Heat
- * Thermal Conductivity
- * Viscosity
- * Surface Tension
- * Gas Generation Rate and Composition

* Requirements for the Sludge

- * Composition
- * Solids Settling Rate/% Settled Solids
- * Density
- * Thermal Conductivity
- * Specific Heat
- * Polarized Microscopy

- * Particle Size Distribution
- * Shear Strength
- * Shear Stress vs. Shear Rate Rheogram
- * Gas Generation Rate and Composition

III. CORE EXTRUSION AND SAMPLE HANDLING

Extruded segments of waste, about 19 inches in length, will be supplied by the core drilling operation. The waste in tank 101-SY has crust that lies on top of a slurry/sludge. It is anticipated that there will be several distinct layers in the slurry and sludge and at least two layers in the crust. In order to ensure that representative samples are obtained from the various layers a detailed plan (Ref. 3) has been prepared for the handling of the extruded segments. This plan provides flow sheets and instructions for core extrusion, crust processing, layer transitions, and segment homogenization.

IV. CHEMICAL AND PHYSICAL ANALYSES

A description of the chemical and physical tests to be performed on the core samples was provided in the Safety Improvement Plan (Ref. 1). Since there have been a few minor modifications, a portion of Ref. 1 is provided in this document.

A flow chart for the analyses is shown in Figure 1. The preparation of the segments is given in Ref. 2, as discussed in the previous section. The analyses shown at the bottom of Figure 1 will be conducted first. These tests provide an early indication of the reaction potential (DSC/TGA) and physical properties of the crust. Chemical analyses and other physical property tests conducted on representative segments are shown in the top portion of the figure. Table 1 (reproduced from Ref. 1) provides the rationale for the various analyses and indicates the end use of the results. A functional grouping of the various analyses is shown in Table 2. It should be noted that some of the tests will require development before they can be implemented on radioactive samples. Of those tests requiring development, only the thermal conductivity and explosivity require long term (> 3 months) development.

One test not discussed in detail in Ref. 1 is that of measuring the amount and composition of gas evolved from heated samples of the crust and slurry. A detailed plan has been prepared for these special tests (Ref. 4).

Near term development activities will be centered on analyses for complexants and other organic products. Some of the materials used in the core drilling can interfere with the detection of certain organic compounds. Water will be used as the hydrostatic fluid during the core sampling. In order to account for the water introduced into the sample a tracer is added to the water. The proposed tracer is fluorescein and tests are underway to determine its effectiveness as a tracer. Discussions are being held to determine how this substance might interfere with the analytical techniques

for organics. Another problem area is the lubricant used in the drill rig. Efforts are underway to find a high molecular weight silicone that would not mask the expected organic products.

Developmental work on the procedures for organic analyses is required. Methods being developed include liquid chromatography/mass spectroscopy, liquid chromatography and gas chromatography coupled with Fourier Transform Infrared Detection and gas chromatography/atomic emission detection. These methods will both supplement and complement each other. Procedures for handling and transporting the samples used for volatile and semi-volatile analysis are being revised to include minimization of head space and reduction of sample temperature.

V. PERFORMING LABORATORIES

Chemical analyses for complexants and other organic products will be conducted at PNL. In addition, PNL will also conduct a portion of the radiometric analyses and will perform the rheological evaluations. All other tests will be conducted by the Analytical Chemistry and Process Chemistry Laboratories at WHC.

VI. LABORATORY PROCEDURES

Westinghouse Hanford Co.

The methods used to submit samples for analysis and the procedures use for the various analyses are given in the Analytical Chemistry Services Laboratories Quality Assurance Plan (Ref. 4). This document describes the quality assurance plan, training and qualification of personnel, laboratory procedures, laboratory records, and the control of records, equipment, materials and measurements.

Pacific Northwest Laboratories

The analytical chemistry and physical testing procedures used at PNL to support the 101-SY analyses are listed below.

1) Organic

- * GC screen for volatiles: PNL-MA-599 method PNL-ALO-330
- * GC screen for semivolatiles: PNL-MA-599 method PNL-ALO-340
- * GC/MS analysis for volatiles: PNL-MA-599 method PNL-ALO-335
- * GC/MS analysis for semivolatiles: PNL-MA-599 method PNL-ALO-345
- * TOC analysis
 - * Solids: PNL-MA-597 method 7-40.37
 - * Solutions: PNL-MA-597 method 7-40.7

2) Radionuclide

- * 241Am: HTA-4-35
- * 237Np: PNL-597 method 7-40.17
- * 79Se: PNL-597 method 7-40.15
- * 63Ni and 94Nb: PNL QA level III only, only a working procedure in place, 3-6 weeks and funding would be needed to obtain approval. Expected completion 9/15/90.

3) Gass Mass Spec

- * PNL-MA-597 method 7-40.18

4) Physical Testing

- * All requested tests: PNL-MA-599 method WHC-053-1

VII. SCHEDULE AND PRIORITIES

The schedule for the core sampling and analyses is shown in Figure 2. This schedule assumes that the next gas venting of tank 101-SY will occur about July 25, 1990. Initial data on the physical properties of the crust and reaction potential (DSC) are needed by October 26, 1990, for updated safety analyses and for input to the engineered remedies plan.

As indicated in Ref. 1, the sampling and analytical activities directed at the hydrogen issue in 101-SY will be supportive of the work required by the Tri-Party Agreement (TPA). However, any analyses conducted for the TPA activities on 101-SY material will be done as lower priority work with respect to the analyses required for 101-SY safety issues.

Because of the uncertainties in obtaining a full length sample of the crust and high probability of finding various distinct layers in the crust and the slurry, it will be necessary to establish a priority for the various physical and chemical tests. It is quite probable that insufficient material will be available for all of the tests on some of the core segments. Meetings will be held during the week of July 16 to July 20 to establish the priority list. Results will be transmitted to the laboratories by an internal memorandum.

VIII. RESPONSIBILITIES

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Cost Account Manager	G. D. Johnson	6-8295
Coordinator for PNL Work	H. R. Brager	6-3637
Core Drilling	S. E. Kelly	3-3266
Core Sampling & Analytical Requirements	B. W. Hall	3-5915
Core Extrusion	R. S. Edrington	3-1972
Sample Preparation	"	"
Chemical Analyses	D. A. Dodd	3-2154
Gas Generation Tests	D. L. Herting	3-2532
Data Analysis & Reporting	TBD	
Quality Assurance	TBD	

Pacific Northwest Laboratories

<u>Task</u>	<u>Person</u>	<u>Phone #</u>
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Organic Analysis	E. W. Hoppe	6-2126/3125
Organic Analysis Development	J. A. Campbell	6-0899
Radionuclide Analysis	L. R. Greenwood	6-6918
Gas Mass Spectroscopy	M. W. Goheen	6-3358
Physical Testing	J. M. Tingey	6-2580

IX. REPORTING

Weekly status reports will be issued to provide a brief summary of all activities associated with core sampling and analysis. Preliminary results of physical and chemical tests will be reported by memo or letter. Supporting document reports will be issued for the October 26, 1990 and January 18, 1991, milestones shown in Figure 2.

X. REFERENCES

1. Safety Improvement Plan - Hydrogen In Waste Tanks, Letter Report, H. F. Daugherty to R. E. Gerton, Corr. # 9001478B R3, June 7, 1990.
2. Internal Memo, G. D. Johnson to H. Babad, et al., "Meeting Minutes - Analytical Chemistry Plan," July 13, 1990.
3. Test Plan: 101-SY Core Extrusion and Sample Handling.
4. Laboratory Test Plan for Gas Production from Double Shell Slurry Waste in Tank 241-SY-101, D. L. Herting and E. L. Washington, WHC-SD-WM-TP-084.
5. Analytical Chemistry Services Laboratories Quality Assurance Plan, G. B. Svancara and S. S. Moss, SD-CP-QAPP-001, July 20, 1989.

FIGURE 1.

ANALYSIS FLOW FOR 101-SY CORE SAMPLES

TABLE 1

WHC

Rev. 1

Figure 4-8. Basis for Specified 241-SY-101
Core Sample Analysis. (sheet 1 of 2)

<u>Analysis</u>	<u>Justification</u>
- On individual segments or portions of segments.	[1] These parameters are needed to assess gas-bubble migration mechanisms in the slurry.
<u>Rheological</u> [1],[2] Penetration Resistance Shear Stress/Shear Rate Rheogram Shear Strength	[2] Mechanical data are essential to the development of engineering remedies (e.g., crust busting).
<u>Physical</u> Compressive Strength (crust) [2] Specific Gravity [1],[3],[15] Particle Size Distribution [4], [15]	[3] These parameters essential to the calculation of mass balances. [4] This analysis can identify inhalable particles (<10 microns) subject to release in the event of an accident.
<u>Other</u> X-Ray Diffraction [6] Polarized Microscopy [5],[6] VOA (Volatile Organic Analysis) [7],[11] DSC/TGA (Differential Scanning Calorimetry/Thermal Gravimetric Analysis) [8]	[5] This analysis may provide insight into the mechanism of gas formation in the slurry. Microscopy can reveal the extent of gas adsorption on slurry particles.
- On composites of contiguous segments representative of distinguishable waste layers	[6] These analyses can identify reactive (shock or heat sensitive) crystalline compounds such as NO_3^- .
<u>Physical</u> Specific Gravity [3],[15] Solids Settling Rate [15] Settled Solids (vol%) [15] Centrifuged Solids (wt% and vol%) [3]	[7] These analyses are essential to the identification and quantification of the gas producing (slurry growth causing) compounds. [8] Thermal analysis is essential to the assessment of waste reactivity (exothermic reaction potential, and organic degradation rate); thermal analysis may also be useful in determining kinetic parameters.
<u>Chemical</u> pH/OH- [9] ICP (Inductive Coupled Plasma Spectroscopy)* [10],[11] CN [11] Hg [11] Se [11] As [11] Anions (NO_2 , NO_3 , SO_4 , PO_4 , CO_3 , Cl, F) [6],[9],[11] $\text{NH}_3/\text{NH}_4^+$ [7],[11] Wt% water [14]	[9] These analyses can quantify components that are known to affect the organic degradation rate. [10] ICP is used to characterize the cations (metals) in the waste, some of which may act as catalysts in organic reactions.

Figure 4-8. Basis for Specified 241-SY-101
Core Sample Analysis. (sheet 2 of 2)

<u>Analysis</u>	<u>Justification</u>
<u>Organic</u> [7],[11],[13] TOC (Total Organic Carbon) Semi-Volatile Organics Chelator/Complexants, chelator fragments, carboxylic acids***	[11] These analyses quantitatively identify toxological components (listed wastes, heavy metals, etc.) susceptible to release in an accident.
<u>Radiochemical</u> [11],[12] GEA (Gamma Energy Analysis)** Am-141 H-3 I-129 Nb-94 Ni-63 Np-237 Pu isotopic Se-79 Tc-99 Total Alpha Total Beta Sr-90 C-14	[12] These analyses quantitatively identify alpha, beta, and gamma emitters -- all of which contribute to radiolysis (and ultimately, hydrogen gas generation). Radio-nuclide characterization is also necessary to evaluate the feasibility of future lab studies on actual tank waste. [13] These analyses can quantitatively identify the organic "fuel" in the waste that may be susceptible to reaction with NO ₃ -. [14] Water content affects heat capacity and thermoconductivity, two important parameters in assessing the safety of future core sampling efforts. Water and other fluids also act as lubricants during core sampling. [15] These parameters are related to porosity and permeability.

*ICP analytes: Ag, Al, B, Ba, Bi, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Pd, Si, Sn, Sr, Ta, Ti, Zn, Zr.

**GEA analytes: Ba-140, Ce-141, Ce-144, CePr-144, Co-58, Co-60, Cr-51, Cs-134, Cs-136, Cs-137, Eu-152, Eu-154, Eu-155, Fe-59, Hf-181, Hg-203, I-131, Kr-85, La-140, Mn-54, U-235, Y-91, Zr-95. (These isotopes are normally obtained in the routine GEA analysis.)

***These analyses require further methods development.

FIGURE 2. CORE SAMPLE SCHEDULE

TABLE 2
CHEMICAL/PHYSICAL ANALYSES

Types of Compounds

Anions
Cations
Total Organic Carbon
Complexants/Chelators *
Organic Groups *
X-Ray Diffraction
Volatiles/Semi-Volatiles

Reaction Potential and Stability

Differential Scanning Calorimetry
Thermo-Gravimetric Analysis
X-Ray Diffraction
Explosivity *

Presence of Gas

Microscopy
Off-Gas Studies (Herting)

Physical Property

Density
Particle Size
Crust Strength *
Shear Strength
Wt. % Water
Vol. % Solids
Viscosity
Porosity *
Specific Heat
Surface Tension *
Thermal Conductivity *

Radionuclides

Total Alpha
Total Beta
Gamma Energy Analysis
Cesium and Many Others
Pu Isotopic
Specific Tests for Selected Elements

Toxological

Hg, Se, As, CN, etc.

Attachment D - Proposed Prioritization of Analyses

PRIORITY FOR 101-SY ANALYSES

THE FOUR LEVELS OF PRIORITY ARE:

1. Top priority; data needed in near term for input to update safety analyses and for engineering remedies.
2. Data needed now, but conduct test only if enough material is available.
3. Informative data; but not needed for near term activities.
4. Lowest priority because development required and test most likely will not be ready in time for use in the hot cells.

TABLE 1
CHEMICAL/PHYSICAL ANALYSES

<u>Types of Compounds</u>		<u>Priority</u>
Anions	(need hydroxide)	1
Cations		1
Total Organic Carbon	(need good statistics)	1
Complexants/Chelators *		4
Organic Groups *		4
X-Ray Diffraction		1
Volatiles/Semi-Volatiles,		1
<u>Reaction Potential and Stability</u>		
Differential Scanning Calorimetry		1
Thermo-Gravimetric Analysis		1
X-Ray Diffraction		1
Explosivity *		4
<u>Presence of Gas</u>		
Microscopy		3
Off-Gas Studies (Herting)(should do TOC/CO ₃ on residual material)		3
<u>Physical Property</u>		
Density		1
Particle Size		3
Crust Strength *		2
Shear Strength		2
Wt. % Water	(need drainable liquid)	1
Vol. % Solids		NA
Viscosity		2
Porosity *		1
Specific Heat	(get from TGA)	1
Surface Tension *		4
Thermal Conductivity *		4
<u>Radionuclides</u>		
Total Alpha		2
Total Beta		1
Gamma Energy Analysis		1
Cesium and Many Others	(need Sr 90)	
Pu Isotopic		3
Specific Tests for Selected Elements		TBD
<u>Toxological</u>		
Hg, Se, As, CN, etc.		3

* Requires Development

**Attachment E - Potential Reaction Chemistry
of Polyoxomolybdates in the Waste Tanks**

Molybdate Chemistry Relevant to Waste Tanks. Polyoxomolybdates

At least one general class of molybdenum species warrant some consideration as to their potential reactions with the organic species stored in Hanford waste tanks. This class of compounds is made up of the isopoly and heteropolyoxoanions of molybdenum. These isopolyoxoanions contain Mo and oxygen only, where the heteropolyoxoanions additionally contain a heteroatom such as P, Si, or other ions and metals.

The aqueous acid/base and structural chemistry of the group V-A and VI-A oxometallates (syn. oxoanion) have been widely studied; some of the earliest work dates to original citations by Berzelius in the 1820's. A number of the great names of inorganic chemistry published extensively in this area in the 1920-1950 era, including Werner, Pauling, Hoard, and Keggin, among others. This body of work generally involved the complex ionic equilibria and structural chemistry of V, Mo and W oxoanions in aqueous solution, and the relationship between nucleation, structure and pH. The idealized structures (shown schematically as the linked polyhedral MO_6 units) of several of the more ubiquitous examples are illustrated below.

Heptamolybdate $[\text{Mo}_7\text{O}_{24}]^{6-}$

Decavanadate $[\text{V}_{10}\text{O}_{28}]^{6-}$

Dodecatungstophosphate $[\text{PW}_{12}\text{O}_{40}]^{3-}$

While much of the chemistry associated with this early work is educational, it may have little meaning in the context of the chemistry of waste tanks at Hanford due to the extremely high pH of the solutions stored here. Most of the chemistry covered by those studying the purely aqueous chemistry within this class occurs within a pH range more neutral to acidic. At higher pH values in purely aqueous solution, nucleation is low, yielding only species such as $[\text{MoO}_4]^{2-}$ at pH 9 or higher.

However, in the mid-1970's, a resurgence of interest in the chemistry of the molybdates and tungstates occurred for several related reasons: (1) potential was seen for these systems to act as good models for heterogeneous metal-oxide/surface adsorbate interactions with reference to catalytic hydrocarbon oxidation, and (2) they were inadvertently found to produced some very interesting stoichiometric and homogeneous catalytic reactions in their own right. Several of these more modern studies (W.G. Klemperer, et al.(Univ. Illinois); W. Knoth, et al.(DuPont); J.W. Johnson et al (Exxon)) focused on the isolation of organic and organotransition metal adducts of some of the more common oxoanions from less reactive media than water (in order to simplify the equilibria). Common organic solvents were used with controlled amounts of water in order to individually generate the various $\text{M}_x\text{O}_y^{n-}$ species. The first attempts resulted in the formation of the tetra-nuclear cluster shown below: $[\text{RCHMo}_4\text{O}_{14}(\text{OH})]^{3-}$

This trivalent anion resulted from the oxidation of the dichloromethane solvent by the molybdate species. These reactions are much more pertinent to the basic pH region in mixed aqueous/organic solvent systems. The reactivity of dimolybdate toward chlorocarbon and functionalized organic molecules (including common chelates) has been shown to be quite general. Carbomolybdate clusters of similar character have been isolated from the reaction of a series of aldehydes and organic acids (and their conjugate bases) with various salts of dimolybdate. These reactions result in the partial oxidation of the hydrocarbon. One of the driving forces for the formation of the carbomolybdate clusters is the transition of the tetrahedral coordination of the dimolybdate species to the octahedral structures of the higher nucleated species. Any available, mildly nucleophilic oxygen can be taken-up during the nucleation phase of cluster growth. It is a virtual certainty that the chemistry of the various chelates present in solution in the waste tanks would produce similar chemistry. What is uncertain relates to what reactions and nucleation would occur under specific pH conditions. The structural and reaction chemistry of these systems has been studied by multinuclear NMR (^{17}O , ^{13}C , ^1H and W), X-ray crystallography and other common laboratory methods.

Much similar chemistry to that noted for the tetramolybdate species above has been noted in very recent work, some under patent as catalysts for oxidation of hydrocarbons in homogeneous solution, and some esoteric structural chemistry. However, for the oxometallates themselves, the primary (if not the only) hydrogen sink in the system should be water. It is unlikely that these species, or any like them involving the numerous chelates available, could produce H_2 . These species are quite likely to aid in the degradation of functionalized organic species in solution. Also noted in a quick review of the literature over the past four years are several references to Mo-Fe oxometallates which have produced some related reactions in alkaline solution, involving red/ox of hydrocarbon and nitrogen species.

A more thorough literature search is needed, and possibly several detailed discussions researchers who have studied the reaction chemistry of oxo-molybdates in alkaline solutions. Professor Walter Klemperer (Univ. Illinois), and Dr. James Wroblewski (Catalytica Company) would be knowledgeable contacts. Several research groups at duPont Central Research were very active in molybdate/tungstate chemistry in the early 1980's, most notably Walter Knoth (retired). The tetranuclear molybdate research mentioned above is that of the Klemperer; Catalytica has recently filed for a broad patent for several molybdate systems.

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