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7. Abstract

This test plan presents the guidelines and requirements for acceptance of the S-type fiberglass material for use as the liquid observation well casing material. The plan for evaluating the physical properties of the candidate fiberglass materials when subjected to radiation, corrosive chemicals, and high temperatures typically found in the waste tanks are outlined. The tests also include tube connection evaluations. Finally, the test plan identifies the participants, their responsibilities, and the schedule for completion of the work.

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TEST PLAN FOR QUALIFICATION OF THE S-TYPE FIBERGLASS MATERIALS FOR USE AS THE LIQUID OBSERVATION WELL CASING

1.0 INTRODUCTION

In-tank liquid observation wells (LOWs) were installed in the early 1980's to provide a reliable leak detection system for the Hanford 200 East and 200 West Area Tank Farms. Most of the current LOWs are E-type fiberglass tubes which are closed at one end, inserted vertically into the tank waste, and suspended from tank risers. Neutron and gamma ray surveillance probes are deployed inside the LOWs for vertical tank scans that detect the liquid height in the surrounding waste. This system has proved to be a reliable method of monitoring tank waste liquid levels. Currently, 57 of the 149 single-shell tanks have an installed, functional LOW. The other single-shell tanks are scheduled to have LOWs installed in them. This opportunity is being taken to change the LOW casing material and installation method because the present LOW material contains neutron absorbing borosilicate glass and because of disadvantages in the present LOW insertion method.

Several single-shell tanks containing potentially reactive wastes, such as ferrocyanide or organics, do not have any LOWs. The potential for unwanted exothermic reactions become more favorable as the moisture content of the waste decreases. Maintaining an adequate waste moisture concentration in these tanks will ensure the waste remains intrinsically safe. A second generation of probes is being developed to facilitate waste moisture measurements. New materials for the LOWs have been suggested to enhance the performance of these probes.

This test plan focuses on testing S-type fiberglass materials for use as the LOW casing material and testing of the interface between the fiberglass-resin composite of the tube and a stainless steel adaptor ring which will be inserted in the tube end. This interface must be secure and leak resistant in order for the LOW to function properly. The stainless steel adaptor ring is necessary for attachment of an ultra high-pressure water borehead to the tube end to achieve a decrease in the disturbance of the surrounding waste upon installation (Hertelendy 1993). Therefore, the tube joint and adaptor ring connection must be adequately verified to ensure that the system functions as intended. The verification method appropriate for the interface relies on qualification testing as described in WHC-CM-6-1, *Standard Engineering Practices*.

2.0 OBJECTIVE

The objective of this test is to select a suitable S-type fiberglass material for the LOW casing which will facilitate tank waste moisture measurement using the neutron probe (Watson 1993) and electromagnetic induction probe (Appendix A). This objective will be met by utilizing a material for the LOW that contains minimal amounts of thermal neutron absorbers, is nonmagnetic, is nonconductive, and has sufficient structural strength to survive the tank environment for 20 years. To qualify, such a LOW material requires measuring the material properties of the selected fiberglass

materials under worst case expected operating conditions and also testing the LOW interfacial connections for leakage under extreme conditions.

Two different kinds of S-type fiberglass for use as a casing material will be evaluated. The two types of fiberglass are a filament wound S-glass fiber with Union Carbide epoxy and a bi-directional S-glass cloth with Derakane 411 vinyl ester resin. These two types of fiberglass were picked because they satisfy the requirements imposed by the moisture measurement probes. To satisfy the primary objective, both types of fiberglass will be exposed to the expected radiation and corrosive chemical environments that will exist in the waste tanks for a 20-year life span. The fiberglass showing the least amount of radiation and chemical damage after extensive testing, and having the minimum required structural properties will be chosen as the next generation LOW casing material.

After the LOW casing material has been selected, accepting testing of the manufactured LOWs will be performed to verify the bond strength and leak resistance of the LOW adaptor ring connection, and tube to tube connection under heat and pressure conditions. Because the fiberglass tube and adaptor ring are composed of different materials, their adhesion and response to temperature changes must be checked to ensure LOW integrity. The fiberglass tube can only be fabricated in 914 cm (30 ft) sections. Two sections of tubes will have to be joined together to meet the length requirements and the integrity of this joint will have to be tested. To be complete, the testing should satisfy several objectives. First, the pull test should verify that the interfacial joints can withstand an axial force equal to three times the weight of a 1676 cm (55 ft) length of the subject pipe with the ultra high-pressure water borehead attachment. If this test is successful, the pull test should continue until the force required to separate the interface connection is met, the composite tube fails, or the test equipment reaches its force limit. Second, the test should supply a visual verification that neither the tube-adaptor ring interface nor the tube to tube joint leaks when the external surface of the tube is exposed to a pressure of 276 kPa gauge (40 psig) and a temperature of 49 °C during the experiment. Finally, the force required to bend the tube at the joint connection shall be greater than the necessary force to bend the same joint connection made out of E-type fiberglass.

Because fiberglass is non-metallic, the electrical conductivity is expected to be negligible and not interfere with the electromagnetic induction probe. No electrical conductivity tests are planned on the fiberglass material samples because of its negligible electrical conductivity. The stainless steel adaptor ring is conductive and magnetic; however, it will be located at the bottom of the LOW, below the waste region in the tank. The electromagnetic probe scan, because of the stainless steel adaptor ring's finite size and location, will not be affected by the presence of this localized quantity of magnetic material.

3.0 SCOPE

This test plan establishes guidelines and direction for qualification testing of the LOW casing material and acceptance testing of the LOW interfacial connections. Most of the testing will be contracted out, except for the leakage testing. The material properties testing will be performed by

Pacific Testing Laboratory (PTL) in Seattle, Washington. The gamma irradiation testing will be performed by Battelle Pacific Northwest laboratories (PNL). No radioactive materials will be produced in these tests. Hazardous chemicals used in the tests will be disposed of in accordance with WHC-CM-7-5, *Environmental Compliance*.

The fiberglass tube samples for testing will be supplied by two vendors, C-K Composites and Composites USA. The corrosive chemicals will be supplied by the Westinghouse Hanford Company (WHC) Chemical Engineering Laboratory organization. They will be responsible for the assembly and disassembly of the LOW material test container. The irradiation tests will be performed at the Gamma Irradiation Facility operated by PNL. The LOW material test container will be transported to and from the Chemical Engineering Laboratory and the Gamma Irradiation Facility. The waste tank simulant chemicals will be disposed by the Chemical Engineering Laboratory waste management supervisor.

The LOW adaptor rings will be supplied by Quest Integrated, Inc. (Quest). Quest has also been contracted to supply a testing apparatus for the leakage experiments planned under the LOW adaptor ring connection acceptance testing. Any manufacturer of S-type fiberglass wanting to compete for the LOW casing contract will supply samples of the LOW material tubes with an attached adaptor ring provided by WHC. The interfacial bond strength test, or pull test, as well as the leakage test and the bending test will be contracted out. These test will be performed in accordance with the American Society for Testing and Materials standards.

4.0 DESCRIPTION OF TESTS

The following sections describe the tests for qualifying the S-type fiberglass for use as a casing material for the LOW (RHO 1983). The two S-type fiberglass materials that will be tested under this plan are a hand lay-up bi-directional S-glass cloth with Derakane 411 resin and a filament wound S-glass with epoxy and aromatic amine hardener.

4.1 MEASUREMENT OF MATERIAL PROPERTIES

The material properties testing will be performed by an outside contractor. The contracting company, PTL, is certified as having the necessary equipment and meeting or exceeding the requirements set forth for engineering laboratories by ASTM E329-93 and ASTM E548-91 standards. PTL is also fully accredited by the American Association of Laboratory Accreditation in the area of destructive testing. PTL has an active Quality Assurance Inspection and Monitoring Program in place that can meet WHC Quality Assurance requirements specified in WHC-CM-4-2, *Quality Assurance Manual*.

4.1.1 Material Properties

The material samples are S-type fiberglass tubes with dimensions of 61 cm (2 ft) in length, 7.937 cm (3.125 in.) inside diameter, and 0.317 cm (0.125 in.) wall thickness. The composition of S-type fiberglass is approximately 65% SiO₂, 25% Al₂O₃, and 10% MgO. The following material properties of the tubes will be tested under room (21 °C) and elevated temperatures (122 °C) (Deichelbohrer 1982):

Tensile Strength including Tensile Modulus
 Flexural Modulus, 3 point bend
 Coefficient of Thermal Expansion
 Surface Roughness
 Inside Diameter Roundness
 Resin/Glass Ratio

These tests will be performed twice on unirradiated material at each temperature to verify their accuracy. If the test results vary by more than 20%, additional testing may be required. The tests will also be replicated after the material samples have been exposed to gamma radiation, corrosive chemicals, and high temperature conditions. The later conditions are predicted to be the worst case operating environment for the LOWs over their 20-year life span.

Microscopy testing will be performed on the material samples before and after irradiation to measure the amount of cracking that has taken place. This microscopic analyses will measure the depth and width of the cracks formed by radiation. PNL will perform the microscopy tests.

4.1.2 Irradiation Environment

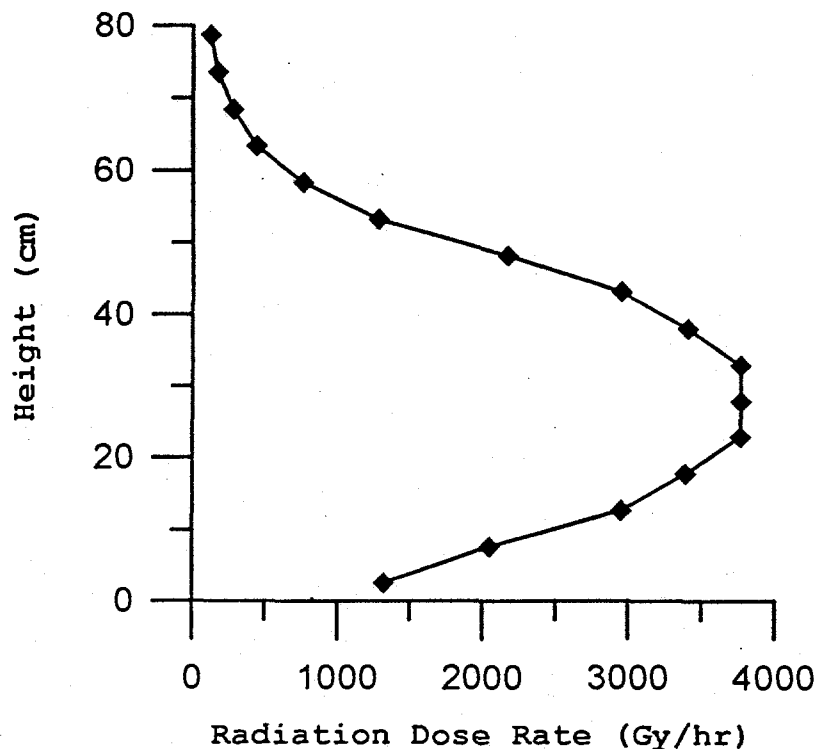
Because of geometric configuration of the ⁶⁰Co source and irradiation tubes, the dose rate varies significantly throughout the length of the irradiation tubes. The irradiation environment shall be at an average exposure of 2.46 10³ Gy/h. Figure 4.1 gives the dose rate as a function of height in Tube 16. The material samples will be irradiated at the bottom 61 cm (24 in.) of Tube 16. Tube 16 has the maximum exposure field and therefore shall be utilized (Hinman 1993).

The irradiation of the LOW material sample shall last until an average gamma radiation dose of 10⁶ Gy has been achieved. This expected maximum total gamma radiation dose simulates a radiation field of 5 Gy/h for 20 years. This dose amount corresponds to the highest radiation dosage that the LOWs are expected to experience in a 20 year lifetime of operation. The LOW material sample shall also be in contact with a corrosive chemical environment suggested by Waste Tank Safety Program corresponding to a mixture of the following chemicals:

3.0 M NaOH
 2.0 M NaNO₂
 5.0 M NaNO₃
 2.5 M NaAlO₂
 0.5 M Na₂CO₃
 0.1 M Na₂Cr₂O₇

These salts will be added to hot distilled water to obtain the maximum solubility (Deichelbohrer 1982, Deichelbohrer et al. 1982).

The pressure and temperature in the LOW material test container will be controlled. The pressure build up from radiolysis of the chemicals shall be less than 103 kPa gauge (15 psig). Calculations show that the pressure build up will be approximately 36.5 kPa gauge (5.3 psig) for the total gamma exposure (Jonah et al. 1994, Powell 1994, Roblyer et al. 1994). The temperature of the corrosive mixture will be maintained constant at 102 ± 1 °C during irradiation conditions by measuring the corrosive chemical temperature using thermocouples and surrounding the LOW material test container with heating tape. This value is higher than the maximum temperature expected in the waste tanks, corresponding to 94 °C in tank SX-108 (Hanlon 1994).



December 1994

Figure 4.1 Gamma Irradiation Facility's Tube 16 Radiation Field.

4.1.3 Equipment and Facilities

The irradiation and waste tank simulant chemical exposure shall be performed at the Gamma Irradiation Facility operated by PNL. WHC personnel will assemble the LOW material test container containing the LOW material sample and corrosive chemicals. This assembled LOW material test container will be delivered sealed to PNL for irradiation in their Gamma Irradiation Facility. The duration of the irradiation shall be for a total gamma radiation dose of 10^6 Gy. After achieving the desired radiation exposure, the LOW material test container will be given an external survey to the

requirements of DOE Order 5400.5 and issued an unconditional release. The LOW material test container shall then be delivered to the Chemical Engineering Laboratory for disassembly and removal of the LOW material sample. The LOW material test container shall be cleaned and reused for the next test. The LOW material sample shall also be cleaned and sent to PTL. The LOW material test container shall be designed in accordance with WHC-CM-6-3, *Drafting Standards Manual*. Figure 4.2 shows a cross sectional view of the LOW material test container (Skians 1982).

Gamma irradiation will be performed on the material samples at the bottom of a 213 cm (7 ft) diameter by 417 cm (164 in.) deep irradiation tank located in the Gamma Irradiation Facility. There are 37 stainless steel tubes in the tank, each sealed at the bottom, to allow material samples to be irradiated in a dry environment. The tubes range in size from 5.08 cm to 15.24 cm (2 to 6 in.) diameter. Tube 16 offering the maximum exposure will be utilized. For radiation shielding purposes the tank is filled with water and maintained at a temperature of 19 °C. Cobalt-60 sources are used in the bottom of the pool to provide the gamma exposure rates. Dose rates in the gamma irradiation tubes were calibrated by PNL during September 1985 and the dose rate in Tube 16 was recalibrated in April 1993. The irradiation of the LOW material sample shall last until an average gamma exposure of 10^6 Gy has been achieved. This total gamma radiation dose simulates a radiation field of 5 Gy/h for 20 years. This total dose amount also corresponds to the highest radiation exposure that the LOWs are expected to experience in their 20-year lifetime of operation.

Each set of material properties tests given in Section 4.1.1 will require one tube having the dimensions 61 cm (2 ft) in height, 7.937 cm (3.125 in.) inside diameter, and 0.317 cm (0.125 in.) wall thickness to complete all of the tests. Therefore, four tubes will be necessary for each type of fiberglass tested. Two tubes for each fiberglass tested will be irradiated and exposed to corrosive chemicals. Each tube will be tested at a different temperature corresponding to room or elevated temperatures.

4.1.4 Waste Disposal

The waste tank simulant chemicals shall be stored in a satellite accumulation area in accordance with the waste disposal management written inspection plan. The chemical waste containers will be stored near the assembly/disassembly of the LOW material test container. The chemical container will be secured and closed in the satellite accumulation area when not in use. The same type of container that the chemicals were shipped in will be used to store the waste chemicals after each experiment. All containers will be clearly labeled with a list of compound chemicals stored. The Material Safety Data Sheets will be maintained with the chemical containers and easily accessible. Finally, waste disposal management will dispose of the waste chemicals from the satellite accumulation when all four irradiation experiments have been completed by complying with environmental regulations.

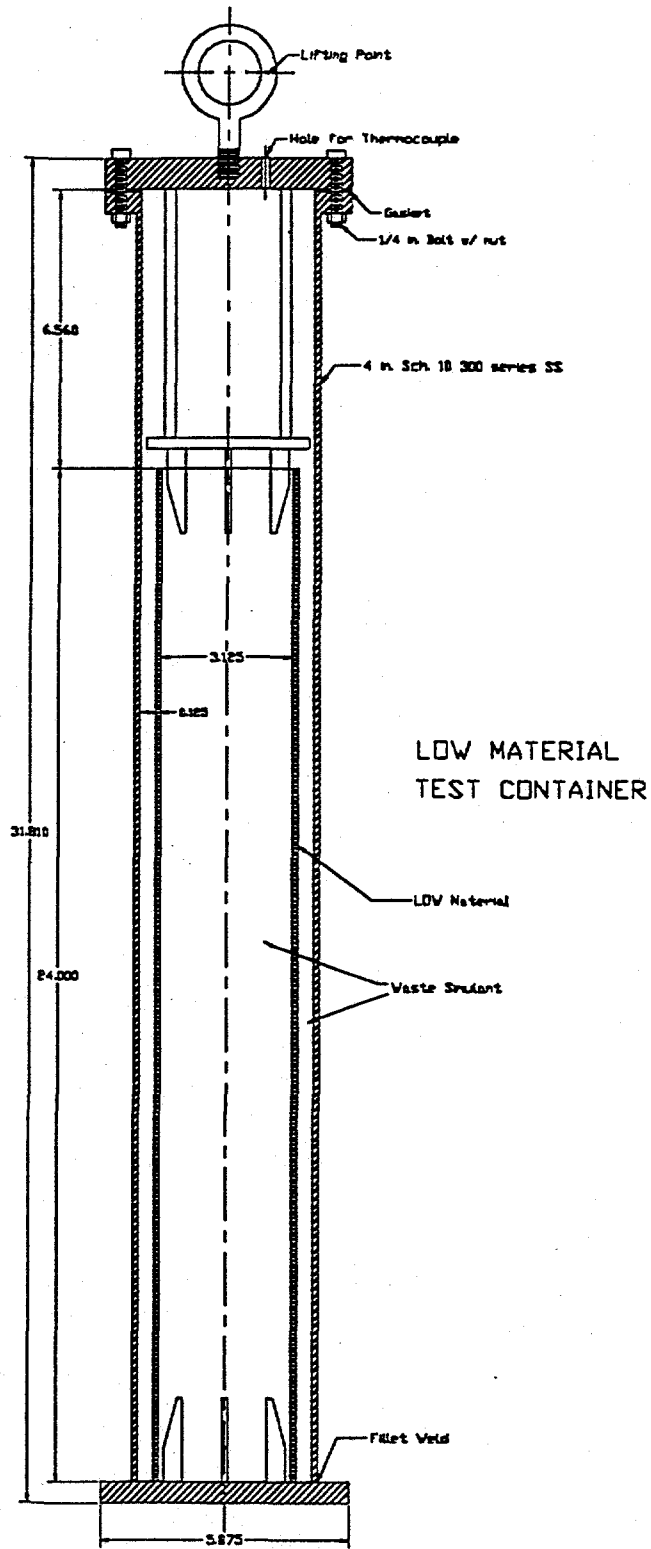


Figure 4.2. Cross Sectional View of the Liquid Observation Well Material Test Container.

4.1.5 Data

The radiation field and temperature of the LOW material test container shall be monitored by PNL personnel. The temperature of the experiment will be maintained at 102 ± 1 °C by wrapping the exterior of the stainless LOW material test container with a heating tape which will be covered with insulation. The temperature will be maintained through a K-type thermocouple immersed in the liquid/salt corrosive chemicals and connected to an Athena Temperature Controller. A second K-type thermocouple immersed in the chemicals will be connected to a digital temperature read-out instrument to verify controller setting. Both thermocouples will be calibrated to be accurate to $\pm 1\%$ of the measured temperature in °C. The thermocouples will be replaced after every test. PTL shall provide the necessary material properties testing results in accordance with the corresponding American Society for Testing and Materials standards. The measured material properties will be recorded in data sheets, Section 13.0.

4.1.6 Criteria and Constraints

The results of the material properties testing shall determine which LOW material is best suited for use as the LOW casing material. The tensile strength, tensile modulus, and flexural modulus shall not vary by more than 20% of the unirradiated values. The minimum inner diameter shall be 7.62 cm (3 in.) or greater. The material showing the least amount of radiation and chemical damage, while maintaining its integrity and strength, shall be the better of the two materials tested. This information together with the results of the LOW interfacial connections shall determine which material will be chosen for the LOW casing.

4.2 ACCEPTANCE TESTING OF LIQUID OBSERVATION WELL CONNECTIONS

The LOW will have two connections which need to be leak tested. The first connection occurs at the bottom of the LOW tube. This connection binds the fiberglass tube to a stainless steel adaptor ring, which is in turn connected to an ultra high-pressure water borehead for slicing through solid waste during installation of the LOW into the tank (Hertelendy 1993). The second connection is due to the length requirement for the LOW. The fiberglass tubes can only be manufactured in 914 cm (30 ft) sections. Therefore, two fiberglass tubes will have to be joined to meet the 1676 cm (55 ft) length requirement. The connection between the tubes will also be tested.

Part or all of the testing performed in this section will be contracted to an off-site vendor. WHC does not have the necessary equipment to perform all of the testing. The two main contractors are PNL and PTL. The contractor that can perform the work in the least amount of time, having an approved Quality Assurance program, and within budget allocation will be chosen.

These interfacial connections will not be exposed to gamma radiation nor corrosive chemical testing. No new material will be introduced other than stainless steel. Both connections will use the same epoxy or resin that the fiberglass materials are made up for the joint connections.

4.2.1 Interface Connections

The tube samples are a vendor supplied composite of S-type fiberglass. The first sample is 61 cm (2 ft) long, 0.317 cm (0.125 in.) thick, and has a 7.937 cm (3.125 in.) inner diameter. This tube sample has a stainless steel adaptor ring fastened to one end of the tube. The second sample consists of two tube samples, identical to the previous tube sample, joined together.

4.2.2 Test Conditions

The pull test will be performed at room temperature and pressure on a suitable strain gauge instrument. The leak test will be performed with an external leak test fixture fitting over one end of the fiberglass tube sample. The external leak test fixture is constructed with a suitable seal such that temperature and pressure may be controlled inside the fixture. This fixture will be designed and supplied by Quest. The bending test on the joints will be performed to measure the force necessary to bend the tube at its weakest point.

4.2.3 Equipment and Facilities

A strain gauge instrument will be needed to perform the pull test. A special external leak test fixture, designed by Quest, will be used for the leak test. This fixture is capable of providing one seal on the periphery of the adaptor ring and another on the periphery of the LOW tube. It can maintain 276 kPa gauge (40 psig) gas pressure to the area between the two seals in order to leak test the interface connections. Finally, the bend test will be performed with a three-point loading system utilizing center loading on a simply supported beam.

4.2.4 Data

The pull test shall measure the force necessary to separate the components or cause tube wall damage. The maximum design load for the LOW shall be equal to the force required to support three times the weight of a 1676 cm (55 ft) length of the LOW pipe with an ultra high-pressure water borehead attached to it. If breakdown does not occur within the maximum design load, the measurement need not be more precise than $\pm 5\%$.

The leak test will be confirmed through visual inspection only, therefore no range or precision analysis is required. The leak test shall be performed using water or a gas at an external pressure of 276 kPa gauge (40 psig) for 15 minutes per cycle for five cycles at the expected temperature (49 °C) of the waste. The inside of the LOW tube shall be monitored for water/gas during the pressure tests. The temperature and pressure at which the external leak test fixture holds the test tube shall be in the range of $\pm 5\%$ of the measured value.

The bend test shall measure the deflection of the tube to tube joint connection. The bend test will be performed by using a three-point loading system utilizing center loading on a supported beam. The maximum deflection shall be equal or less than the deflection of the E-type fiberglass LOW joint

connection. A calibrated testing machine with an error in the loading measuring system of $\pm 1\%$ shall be employed. It should be equipped with a deflection measuring device. The testing shall follow the standards given by ASTM D790-92.

4.2.5 Criteria and Constraints

This test shall be considered complete when the following three conditions have been met. First, the pull test has verified that the interfacial joints can withstand the maximum design load. Secondly, visual verification has shown that neither the LOW adaptor ring interface nor the tube to tube joints leak when the external surface of the tube is exposed to a pressure of 276 kPa gauge (40 psig) and a temperature of 49 °C. Finally, the tube joint connection has proven it can maintain its stiffness against deflection caused by the movement of solid waste in the tanks. This condition shall be met by comparing the force necessary to bend the S-type fiberglass at the joint connection to the present E-type fiberglass. The force necessary to bend the S-type fiberglass joint connection should be equal or greater than the force to bend the E-type fiberglass joint connection.

The main constraints are that the LOW casing material shall be flexible enough to withstand some impact from waste movement without buoyancy deformation, and the LOW inner diameter shall allow passage of a cylindrical instrument having an outside diameter of 7.303 cm (2.875 in.) and length of 165 cm (65 in.).

5.0 EXPECTED RESULTS

Both of the S-type fiberglass materials are expected to qualify for use as the LOW casing material (Reichmanis 1987). The fiberglass materials are inorganic, strong, nonflammable, and heat resistant, as well as resistant to chemicals, radiation, and moisture. These tests are being performed to provide documentation and justification for their usage.

5.1 MATERIAL PROPERTIES TEST

Both of the LOW casing materials are expected to be able to withstand the radiation and waste tank simulant chemical environment without any significant loss of strength or deformation (McManamy et al. 1992). The material shall have a minimum Young's modulus (elastic modulus in tension and compression) of 4.13 GPa after irradiation and exposure to corrosive waste tank simulant chemicals. The decrease in the coefficient of thermal expansion value due to irradiation is also expected not to affect the LOW adaptor ring interface (Coltman 1981, McManamy 1991). Finally, the tubes are expected to maintain their geometric shapes and allow passage of a cylindrical instrument having an outer diameter of 7.303 cm (2.875 in.) and a height of 165 cm (65 in.).

If both materials degrade under the gamma radiation and corrosive chemical environment, there will be two options. The first option is to continue using the present E-type fiberglass for the LOW casing material.

Even though E-type fiberglass contains borosilicate glass, both of the moisture measuring probes will function with this material. The other option is to begin testing other types of materials for use as the LOW casing. This option will delay the installation of the LOWs.

5.2 TUBE CONNECTIONS TEST

The LOW adaptor ring and tube to tube connections are expected to withstand an external pressure force greater than 276 kPa gauge (40 psig). A successful test would give a force value well above the minimum value described previously. The connections and the LOW tube material are also expected to be able to support three times its weight including the weight of the ultra high-pressure water borehead.

Neither the tube to tube connection nor the LOW adaptor ring interface are expected to fail the leak detection test. A successful test would constitute the LOW system withstanding a pressure and temperature without any moisture developing on the inside of the LOW (Schmunk 1984). The pressure shall be applied in cycles, five cycles of 15 minutes per cycle.

In case of failure, the wall thickness of the LOW material samples will be increased. The presently installed E-type fiberglass LOWs have a wall thickness of 0.51 cm (0.20 in.) and the S-type fiberglass sample tubes are being tested with a wall thickness of 0.317 cm (0.125 in.). If the sample tubes fails the pull, impact, leakage, or bend test, the wall thickness will be increased to 0.51 cm (0.20 in.). This increase in wall thickness will increase the structural strength and integrity of the LOW material tube. It will also provide an increase in strength to make the LOW adaptor ring and tube to tube connections stronger.

6.0 TEST PROCEDURE

Manufacturers of fiberglass wishing to compete for the WHC LOW contract to provide the LOWs will submit samples of the fiberglass material in the form of a pipe having an inner dimension of 7.937 cm (3.125 in.), wall thickness of 0.317 cm (0.125 in.), and height of 61 cm (2 ft). Two of the samples will include LOW adaptor rings and tube to tube joint connections. The testing will proceed in the following sequence.

1. Measure initial material properties at room temperature (21 °C).
Two 61 cm (2 ft) sections of tube samples will be shipped to PTL.
Each section corresponding to a different fiberglass material.
2. Measure initial material properties at elevated temperature (122 °C).
Two 61 cm (2 ft) sections of tube samples will be shipped to PTL. Each section corresponding to a different fiberglass material.
3. Install tube in LOW material test container.
Four samples consisting of 61 cm (2 ft) sections of tube per sample will be tested. The four samples will be made up of two tubes of filament wound S-glass fiber with Union Carbide epoxy and

two tubes of bi-directional S-glass cloth with Derakane 411 vinyl ester resin. The assembly and disassembly of the LOW material test container will be performed by the Chemical Engineering Laboratory.

4. Subject tube to radiation, corrosive chemical, and temperature simultaneously.
Each sample will be inserted into the LOW material test container and irradiated to an average exposure of 10^6 Gy. Four irradiation test will be performed, two tube samples from each type of fiberglass material tested. The testing will take place at the Gamma Irradiation Facility operated by PNL.
5. Measure final material properties at room temperature (21 °C).
Two 61 cm (2 ft) sections of tube samples will be shipped to PTL. Each section corresponding to a different fiberglass material.
6. Measure final material properties at elevated temperature (122 °C).
Two 61 cm (2 ft) sections of tube samples will be shipped to PTL. Each section corresponding to a different fiberglass material.
7. Perform leakage and pressure test at high temperature (49 °C).
Testing will be performed by WHC.
8. Perform pull test.
Testing will be performed by PNL or PTL. Two samples of LOW adapter ring connections and one sample of LOW tube to tube connection will be supplied to the contracted company for each type of fiberglass tested.
9. Perform bending test.
Testing will be performed by PNL or PTL. Two samples of LOW tube to tube connection will be supplied to the contracted company for each type of fiberglass tested.

Steps 1, 2, 5, 6, 8, and 9 will be contracted out to an outside laboratory. PTL will perform all material properties testing necessary, steps 1, 2, 5, and 6. Step 4 will be performed by PNL. However, test procedures will have to be developed and implemented for the assembly and disassembly of the LOW material test container, step 3. Step 7 will be performed in the Chemical Engineering Laboratory. The test procedure for testing the LOW interfacial connections will be developed upon delivery of the external test fixture for leak detection from Quest.

7.0 SAFETY

The work of setting up and conducting these tests involves the common industrial hazards of assembling, lifting, moving, and disassembling the LOW material test container. The other hazards include handling corrosive chemicals and disposing of them properly. The corrosive chemicals have a pH of less than 14 and one of the compounds, sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7$), in the

stimulant is classified as a carcinogen. Standard laboratory safety practices will be followed while testing. Applicable Material Safety Data Sheets will be maintained in the laboratory. Safety glasses and rubber gloves are required when handling the corrosive chemicals.

The LOW material test container will be irradiated in the Gamma Irradiation Facility. The pressure build up from radiolysis of the chemicals in the LOW material test container shall be less than 103 kPa gauge (15 psig). Since there are no neutron sources in the Gamma Irradiation Facility, no radioactive activation of test materials will occur. The Gamma Irradiation Facility is also free of surface contamination and therefore no surface contamination is expected on the surface of the LOW material test container. The corrosive chemicals will not be classified as mixed waste. Nevertheless, the LOW material test container shall be externally surveyed before it is unconditionally released from the Gamma Irradiation Facility to meet DOE Order 5400.5 requirements. PNL technicians shall be responsible for the irradiation of the LOW material test container. It is not expected that any person involved in this testing will receive any radiation dose as a consequence of these tests. The test procedures shall be performed in accordance with WHC-CM-4-3, *Industrial Safety Manual*.

The pull test and bending test shall be completed within established test procedures. Gloves will be worn when performing the pull test because the fiberglass tubes will heat up when stretched. The leak test experiment shall comply with the WHC pressure testing safety requirements given in WHC-CM-4-3, *Industrial Safety Manual*. Gloves will be worn because the outer surface of the external leak test fixture will be above 49 °C. These tests will be performed with all necessary safety precaution taken. No radiological or hazardous chemical safety are associated with this testing.

The performance of these tests is considered a Safety Class 3 activity. Failure of equipment associated with the irradiation or LOW tube connections testing could result in harm to facility workers caused by industrial safety hazards (e.g., lifting, moving, operating testing equipment). A job hazardous analysis will be performed on all tests conducted by WHC.

8.0 QUALITY ASSURANCE

All testing will be conducted in accordance with laboratory procedures approved by Nuclear Analysis and Characterization or their designee and as documented by this test plan and test procedures. All quality assurance related requirements for these tests will conform to the document requirements given in the WHC-CM-4-2, *Quality Assurance Manual*, for quality practices. The test plan has been categorized as Approval Designator ESQ. The design and fabrication of the LOW material test container shall not have Approval Designator status because the LOW material test container is not critical to the material properties testing. The test procedures for these experiments will require a Approval Designator SQ.

Quality assurance activities for all contractors involved in the design, construction, and testing of the LOW casing materials shall be executed in accordance with DOE 5700.6C, *Quality Assurance*, to provide the following assurances:

1. Design data and decisions are documented and traceable.
2. The design and design criteria are adequately supported by the proposed plans, specifications, and analysis.
3. The design meets the baseline design criteria.
4. Construction is performed in accordance with the definitive design documents.
5. Tests confirm the adequacy of the design, the quality of the manufactured components, and the operability and reliability of the design. The quality assurance activities are formulated and executed through the use of the project specific quality assurance plan in accordance with WHC-CM-4-2, *Quality Assurance Manual*.

9.0 ORGANIZATION RESPONSIBILITIES

The Nuclear Analysis and Characterization group of WHC has overall responsible for organizing and overseeing all LOW testing. The group will assign responsibilities related to the development of the self-installing LOW program. They will also prepare and review the test plan, test procedures, and final test reports. All engineers will comply with the responsibilities of a cognizant engineer given in WHC-IP-1026, *Engineering Practice Guidelines*. The following document describes the responsibilities of individuals and organizations involved in all phases of the LOW casing material testing, WHC-SD-WM-ETP-136, *Engineering Task Plan for the Development of the Self-Installing Liquid Observation Well*.

Safety management shall review the test plan and accept the test procedures for safety conformance. Safety management shall also provide inspection and support as required to conduct testing within the safety standards of WHC. Quality Assurance management shall approve the test plan and review the final test report. The final acceptance testing will be observed by Quality Assurance personnel.

Waste disposal facility management shall ensure that all dangerous waste generated, stored, and disposed activities are managed in compliance with WHC-CM-7-5, *Environmental Compliance*. Management will obtain proper designation of, and packaging for, all solid waste generated or to be generated at the facility.

10.0 SCHEDULE

The necessary documentation are currently being written to prepare for the testing described in this test plan report. These documents include the Functional Design Criteria, Test Plan, Testing Procedure, Engineering Task Plan, etc. The LOW material samples have been ordered from the fabricators and will be arriving by December 29, 1994. Mechanical drawings of the LOW material test container are currently being completed. These drawings will be issued as Development Control mechanical drawings to allow the machine shop to

begin fabricating the LOW material test containers before the drawings are released. The test procedure document and the LOW material test container shall be completed by December 29, 1994.

The irradiation test will begin on January 3, 1995. There are four tests and each one will require 17 days in a gamma dose rate field of 2.46×10^5 Gy/h field to achieve the desired total gamma exposure of 10^6 Gy. These tests can only be performed one at a time. Therefore, it is estimated that the irradiation experiments will be completed by March 17, 1995. The pre-irradiation testing of the material properties will begin on January 3, 1995 and last until February 17, 1995. An emergency requisition is being written to authorize WHC procurement to contract the material property testing to PTL in Seattle, Washington. The post-irradiation testing of the material properties will begin on January 23, 1995 and last until April 21, 1995.

Quest will deliver 10 adaptor rings by January 30, 1995. These adaptor rings will be delivered to any manufacturer willing to compete for the LOW contract meeting the technical specifications for the LOW. A request for bids will be issued in the beginning of January for the LOW contract. At the same time, conceptual design drawings will be drawn for the two different types of LOW casing materials being tested. The testing of the LOW adaptor ring connection and tube to tube connection will commence on February 17, and last until April 21, 1995. WHC does not have the necessary equipment to perform these tests. These test will be contracted out to either PNL or PTL. Two months are planned for the leakage tests to account for modification of the LOW material thickness, resin, epoxy, or adaptor ring that may be deemed necessary. Finally, the final test report describing all of the tests performed and recommending the best material for use as the LOW casing will be completed by May 30, 1995.

11.0 REPORTS

Results of the tests will be documented in a supporting document. The preparation, format, control, retention, and traceability of all supporting documents will conform to the requirements of appropriate sections of WHC-CM-6-1, *Standard Engineering Practices*. Drawings of the LOW material test container will be included in the report along with any repairs, adjustments, maintenance, or other unplanned events.

A final report will be issued by the Nuclear Analysis and Characterization group of WHC. It will describe the test results and the LOW material selection as well as describing the design and development of the ultra high-pressure water borehead on or before May 30, 1995. This aggressive delivery schedule assumes that the development and testing programs will proceed without any delays or setbacks. The final report will contain the complete mechanical drawings of the LOW material test container, and irradiation and corrosive chemical material test results. The report will also cover how well the S-type fiberglass material can withstand the tank environment and permit through-wall interrogation by neutron and electromagnetic induction probes. The final report will also document all tests performed on the LOW casing materials as well as the LOW adaptor ring connection and tube to tube connection.

12.0 REFERENCES

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- ASTM D790-92, *Standard Test Methods of Unreinforced and Reinforced Plastics and Electrical Insulating Materials*, American National Standard, Washington, D.C.
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- WHC-CM-7-5, *Environmental Compliance*, Westinghouse Hanford Company, Richland, Washington.
- WHC-IP-1026, *Engineering Practice Guidelines*, Westinghouse Hanford Company, Richland, Washington.

13.0 DATA SHEETS

Data will be recorded on data sheets with the following format for the material properties testing.

LOW MATERIALS PROPERTIES EVALUATION

Material:

No. Test	Temperature	Thickness	Width	Length	Stress	Strain
1. Initial Tensile						
2. Modulus						
3. Initial Tensile						
4. Strength						
5. Initial Three Point						
6. Bend						
7. Initial Coefficient of Thermal						
8. Expansion						
9. Initial Surface		Microcentimeters Rms:			Glass/Resin Ratio:	
10. Initial ID		Maximum:			Minimum:	
11. Stress Exposure	Gamma Field					
	Time:					
	Dose:					
	Temperature:					
12. Final Tensile						
13. Modulus						
14. Final Tensile						
15. Strength						
16. Final Three Point						
17. Bend						
18. Final Coefficient of Thermal						
19. Expansion						
20. Initial Surface		Microcentimeters Rms:			Glass/Resin Ratio:	
21. Initial ID		Maximum:			Minimum:	
22. Equipment Serial No.				Calibration Date:		
23. ASTM/other procedures used in testing:						
24. Comments:						

All testing will be performed under ASTM guidelines.

Data will be recorded on data sheets with the following format for the pull, bend, and leakage tests.

LOW TUBE CONNECTIONS EVALUATION

TEST NO.	TEST		EXPERIMENTER SIGNATURE	DATE
1 Mat 1	Pull Test		Force: Failed:	
2 Mat 2	Pull Test		Force: Failed:	
3 Mat 1	Bend Test		Force: Failed:	
4 Mat 2	Bend Test		Force: Failed:	
5 Mat 1	Leak Test	Temp: Pres.: Time:	Visual:	
6 Mat 2	Leak Test	Temp.: Pres.: Time:	Visual:	

Pull Test Equipment Identification: _____

Impact Test Equipment Identification: _____

Bend Test Equipment Identification: _____

Leak Test Equipment Identification: _____

Quality Assurance Verification: _____

Comments: _____

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APPENDIX A

BATTELLE-PACIFIC NORTHWEST LABORATORY FERROCYANIDE SAFETY PROJECT

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**ELECTROMAGNETIC MOISTURE MEASUREMENT
IN WASTE TANK SIMULANTS AND IONIC SOLUTIONS**

R.L. Hockey

October 29, 1993

Letter Report of Work Performed for
Westinghouse Hanford Company

Pacific Northwest Laboratory
Richland, Washington

Electromagnetic Moisture Measurement In Waste Tank Simulants And Ionic Solutions

Introduction:

The objective of this task was to assemble a test apparatus and conduct preliminary experiments to evaluate the capabilities of electromagnetic induction technology for moisture measurement in materials that simulate the wastes found in the single-shell tanks (SST's) at Hanford. Support provided to this task was limited to the fourth quarter of FY 1993 only which limited the investigation to a preliminary evaluation. During this period efforts were focused on; conducting a literature search to identify and review prior work relevant to this task, development and characterization of electromagnetic induction sensor coils, identification and procurement of waste tank simulants to represent the various wastes found in typical SST's, developing procedures for conducting moisture measurement experiments, and conducting preliminary experiments to determine the suitability of electromagnetic induction for determining various moisture levels in the waste simulants.

Experimental Results:

In the fourth quarter of FY 1993 experiments were performed to investigate how the electrical resistivity of ionic salt solutions and waste tank simulants behave when their moisture content varies. The effects related to sample resistivity were measured using a noncontact method. Ionic solutions containing various amounts of water were placed into sealed test-tubes and surrounded by an induction coil. The impedance of the coil depends on the resistivity of the material placed inside the coil. A Hewlett Packard impedance analyzer, controlled by a computer, recorded both the real (resistance) and imaginary (reactance) components of the coil's impedance.

To determine the effect different ions may have on the impedance measurements, several different ionic compounds were tested using the above technique. These included:

- Westinghouse Hanford prepared T-Plant ferrocyanide flowsheet simulant containing 25% ferrocyanide by weight.
- PNL prepared 101-SY simulant (NaOH-based)¹ for double-shelled waste tanks.
- NaCl.

¹ S.A. Bryan, L.R. Pederson, J.L. Ryan, R.D. Scheele, J.M. Tingey, 1992 "Slurry Growth, Gas Retention, and Flammable Gas Generation By Hanford Radioactive Waste Tanks: Synthetic Waste Studies, FY 1991" PNL-8169, Pacific Northwest Laboratory, Richland, WA.

- Na_2SO_4 .
- BiPO_4 , NaCl , Na_2SO_4 and 101-SY (NaOH based) simulant all mixed in equal weight amounts.

The reader is referred to Appendix A for more information on the simulants used in this work.

With the exception of the T-Plant simulant containing $\text{Na}_2\text{NiFe}(\text{CN})_6$, the dry salts were weighed and mixed with known amounts of deionized water to determine moisture content by weight. The target moisture percentages were 0%, 10%, 20% and 40%. It was very difficult to achieve anything less than 10% moisture by adding water directly to the salt. The water tended to dissolve only the surface layer leaving a very nonuniform mixture. This was confirmed by measuring the coil impedance as it was moved with respect to the test-tube. In relatively homogeneous solutions/slurries, the coil impedance varied less than 1% when moved from top to bottom of the tube. Only with the NaOH based 101-SY simulant was it possible to achieve a uniform distribution of 5% water by weight. However the moisture range of most interest, to assure waste tank safety, is between 15% and 30%. Therefore, minimal effort was devoted to making solutions/slurries with less than 10% moisture.

In the case of the T-Plant ferrocyanide flowsheet simulant containing $\text{Na}_2\text{NiFe}(\text{CN})_6$, testing began with a sludge containing 70% moisture which was then oven-dried down to 40% and 0%. The error in determining 40% moisture in the T-Plant simulant sludge was $\pm 15\%$ because of voids and the difficulty in getting a uniform mixture. Future experiments should start with the wettest sludge and then use a more controlled dry-down procedure to obtain the desired moisture content.

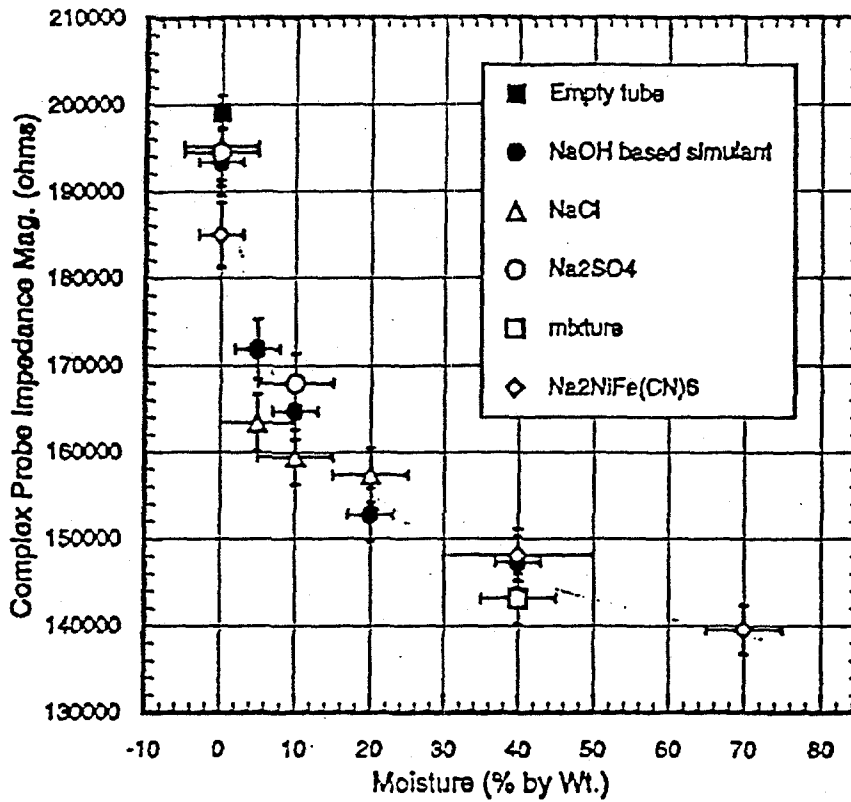
Figure 1 shows the complex impedance measurement as a function of moisture content for the two simulants, sodium chloride, sodium sulfate, and the mixture containing BiPO_4 , NaCl , Na_2SO_4 and 101-SY simulant all mixed in equal weight amounts. The error bars along the moisture axis are estimated uncertainties in the actual moisture content of the samples. Error bars along the impedance axis are estimated from calibration tests performed on the experimental apparatus.

Principal Observations:

This preliminary investigation shows that a strong correlation exists between the electrical resistivity of ionic solutions and their moisture content. In conducting this feasibility study several other observations were made:

- The impedance measurements for the two tank waste simulants with 40% moisture are very close. This gives a preliminary indication that the impedance measurement is not a strong function of composition for these two different simulants. The substantial difference observed at 0% moisture is unexplained. It can be speculated that the difference is due to the presence of the nickel ions, and perhaps this difference is less significant as water is added. Unfortunately, the T-Plant ferrocyanide flowsheet simulant was received only three days before the end of funding for this phase of the project, so

Figure 1. Magnitude of Complex Impedance Plotted Versus Percent Moisture.



Key to Figure 1.

- NaOH based simulant; PNL prepared 101-SY simulant.
- Mixture; equal weights of BiPO₄, NaCl, Na₂SO₄ and 101-SY simulant.
- ◇ T-Plant ferrocyanide flowsheet simulant.

only three moisture content measurements were made. One objective of further work will be to make more comparisons between these two simulants over the 10-40% moisture range.

- There is a small but discernible difference between the sodium chloride measurement and the 101-SY (NaOH-based) simulant measurement. These two simulants have quite different compositions, but a calibration based on sodium chloride would yield only a 5% error in moisture content for the simulant in the 5-20% moisture content range. Westinghouse Hanford tank safety experts believe that a 5% error in the moisture measurement is acceptable.
- From the limited data available, sodium sulfate is indistinguishable from the 101-SY (NaOH-based) simulant in the neighborhood of 10% moisture.
- The mixture containing BiPO₄, NaCl, Na₂SO₄ and 101-SY simulant is just barely distinguishable from the simulants at 40% moisture content.

From the limited data obtained to date, the solution/slurry composition appears to have a relatively small effect on the impedance measurement. Further work is needed to confirm this, but the results to date indicate that the electromagnetic method is a promising one for measuring moisture content of underground storage tank waste.

Recommendations:

Future efforts should be focused on performing similar experiments in an effort to refine the procedure in order to reduce the size of the error bars shown in Figure 1. If test results continue to appear encouraging, a probe should be constructed to measure moisture in simulants contained in 55-gallon drums, preferably the same ones being used by Westinghouse Hanford to calibrate an active neutron probe. This same electromagnetic probe could be tested in a quarter-scale tank containing waste tank simulants prior to final testing in actual single-shell tanks.

APPENDIX A

CHEMICAL COMPOSITION OF TANK WASTE SIMULANTS

APPENDIX A

Table 1. CHEMICAL COMPOSITION OF PNL PREPARED 101-SY SYNTHETIC WASTE.

Component	Concentration	
	Moles/liter	Wt%
NaAlO ₂	2.1	10.7
Na ₄ EDTA	0.17	4.0
Na ₃ HEDTA	0.35	8.3
NaCl	0.35	1.3
Na ₃ PO ₄	0.20	3.3
NaNO ₂	3.1	13.3
NaNO ₃	3.1	16.4
Na ₂ CO ₃	0.4	2.6
NaF	0.1	0.26
NaOH	2.9	7.2
Cr(NO ₃) ₃	1.5 x 10 ⁻³	0.04
CU(NO ₃) ₂	2.1 x 10 ⁻⁴	0.00
Fe(NO ₃) ₃	2.0 x 10 ⁻³	0.05
Ni(NO ₃) ₂	3.1 x 10 ⁻³	0.06

Table 2. COMPOSITIONS OF FERROCYANIDE FLOWSHEET SIMULANT SLUDGE FRACTIONS.

Constituent	Dried U Plant 2 Bottom Fraction (Wt%)	Dried In Farm 1 Bottom Fraction (Wt%)	Dried T Plant Top Fraction (Wt%)
Bound Water	2.5	5.8	≈ 4
Sodium Nitrate	45.2	39.8	27.1
Sodium Nitrite	10.9	11.4	6.1
$\text{Na}_2\text{NiFe}(\text{CN})_6$	8.3	25.6	11.6
Nickel Sulfide	0.0	2.7	0.0
Inert Solids	≈ 33	≈ 15	≈ 55
Free Water*	64	48	69

* This value applies to the as-prepared sludge prior to vacuum drying at 60°C for 18 hr.