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**CONCEPTUAL DESIGN ANALYSES
for HANFORD SITE
DEPLOYABLE REMOTE SPECTROSCOPY SYSTEMS**

B. L. Philipp
F. R. Reich

September 1994

Westinghouse Hanford Company
Richland, Washington

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**CONCEPTUAL DESIGN ANALYSES
for HANFORD SITE DEPLOYABLE REMOTE
SPECTROSCOPY SYSTEMS**

B. L. Philipp
F. R. Reich

EXECUTIVE SUMMARY

In a remote, non-contact camera concept, moisture monitoring system, the near-infrared back-scatter spectra from the waste surface can be strongly impacted by the absorption of the water in the waste tank head-space atmosphere. To meet the required accuracies, this interferent must be factored into the design of the sensing system and calibration models. A PLS model, constructed with atmospheric moisture as an interference, can predict moisture from NIR spectral data independent of atmospheric moisture levels and path-length variations.

Experimental work is being completed to verify model performance with the atmospheric interferences compensated by the PLS models. The next step with the non-contact camera concept is to complete a field-scale demonstration. A path-length/relative humidity compensation model will need to know path-length within 1-2 meters and relative humidity (RH) to +/-5 percent to keep error impacts on a PLS model below an estimated 1-2 wt%.

Water content and particle size will affect the strength of optical back-scatter from a waste surface. WHC's experiments with tank simulants has indicated that the depth of penetration of a sample is proportional to water content. Optical penetration of very dry 241-BY-104 was small, less than 0.25 cm (0.1 inch) while sample penetration exceeded 0.625 cm (0.25 inch when there was 15-20 wt% water content.

More experimental work is needed to verify the magnitudes of these errors, and to quantify the impact of angle and water content on signal strength for the camera concept. Wetter surfaces will exhibit more specular reflection than dryer surfaces. Longer penetration will enhance the signal strength of the moisture readings because the optical back-scatter which is being used to determine moisture content comes from a much larger volume of the wet material. The limiting case is free standing water which is expected to act like a mirror with nearly zero back-scatter. This will create optically black regions in a camera scan. These regions could be assumed to be areas that are extremely wet with standing water where moisture content is not critical. Additional testing should be done to determine the range of optical back-scattering with angle and moisture content as independent parameters.

The University of Washington Center for Process Analytical Chemistry (CPAC), under contract to WHC, has completed several infrared spectroscopy moisture monitoring feasibility studies. Initial feasibility testing using 241-BY-104 simulant was completed by CPAC in 1993. This demonstrated that moisture content data could be extracted from spectra ranging from visible (400 nm) to mid-infrared (10 microns). The most promising region found was the NIR region (1.1 - 2.5 microns) where optical components are currently readily available. With 241-BY-104 simulant tests, moisture prediction accuracies in the 0.5-1.5 wt% were measured over a 0-24 wt% moisture range (Reich 1993). In 1994, CPAC demonstrated that reasonable and detectable signals could be obtained for a remote camera system with a remote path-length of 4.25 meters (14 ft). The affects of head-space gas were ignored in this field demonstration (Reich 1994).

Issues related to particle sizes are being studied by the University of Idaho under contract to WHC and were not addressed in this report.

Two concepts are identified in this report for implementing NIR spectra based moisture sensing system; remote fiber optic probe and remote NIR camera. The remote fiber optic probe would be a scaled up extension of the fiber optic probe concepts that CPAC and WHC have been using to study the feasibility of NIR moisture sensing. This requires the use of radiation resistant fiber optics, but is not sensitive to head-space issues that impact the NIR camera concept. No analysis was completed for this concept in this report. The remote NIR camera system is similar to the non-contact, infrared camera devices now being used to measure temperature remotely.

The non-contact NIR camera system concept can be implemented in two basic configurations. Both configurations are sensitive to the path-length, the concentration of NIR absorbing gasses in the tank head-space region, and the optical properties of the waste surface:

1. Source scan: A tunable laser scanned over the waste surface with back-scattered light being detected by a broadband detector
2. Detector scan: A broadband source, such as a tungsten filament source, focussed at a point on the waste surface with the back-scattered light being collected by a spectrometer with a wavelength scanning detector (CCD detector).

The head-space moisture will impact the moisture detection regions of the NIR back-scatter from a waste surface. To estimate the impact of the head-space relative humidity, a LOWTRAN computer model of atmospheric absorption was mathematically combined with the CPAC 241-BY-104 sample data. This shows the following:

1. If a principle-least-squares (PLS) model is built without including the water in the head-space region, there is a large error in the predicted moisture level from the spectral response. The prediction error is dependent on the pre-model signal processing with over-prediction when raw data is used and under-prediction when differentiated spectral data (to remove base-line effects) is used. Both errors were well beyond a ± 10 wt% prediction error envelop.
2. If a PLS model is built with spectra that includes the anticipated atmospheric absorption, the predicted results are well within a ± 5 wt% error prediction window. This indicates that the use of NIR spectra with PLS can successfully be used to indicate waste surface moisture, provided that a modified PLS model is constructed with the appropriate spectra that have atmospheric absorption as an interference. The modified PLS model is not sensitive to either path-length or to the concentration of moisture.
3. If a path-length/relative humidity compensating model is used, path-length must be known within 1-2 meter (3-7 ft) at high relative humidities (80-100 percent RH) to keep moisture prediction errors below 1-2 wt%. Relative humidity measurements are less stringent with ± 5 -10 percent RH errors needed to keep error impacts below 1-2 wt%. Humidity errors impact the longer path-lengths more than shorter path-lengths.

To demonstrate surface moisture mapping, simulant materials with varying moisture content need to be used with either the source scan or the detector scan concept in a mock-up test with tank-scale dimensions.

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LIST OF TERMS

ALARA	As low as reasonably achievable
CPAC	Center for Process and Analytical Chemistry (University of Washington)
DOE	U.S. Department of Energy
NIR	Near Infrared
OPC	Optical parametric converter
PLS	Principle-Least-Squares
UST-ID	Underground Storage Tank Integrated Demonstration
WHC	Westinghouse Hanford Company
WSRC	Westinghouse Savannah River Company

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1.0 INTRODUCTION

Waste Tank Safety is providing resources for the development and testing of remote Near Infrared (NIR) spectroscopy moisture monitoring systems for use in the characterization of Hanford's waste tank materials. Similar efforts are receiving assistance provided by the U.S. Department of Energy (DOE) Underground Storage Tank Integrated Demonstration (UST-ID). Under waste Tank Safety and UST-ID guidance for FY-95, this is to be a joint effort involving Westinghouse Hanford Company (WHC), conceptual testing at University of Washington's Center for Process and Analytical Chemistry (CPAC), and work at several DOE laboratories, including Westinghouse Savannah River Company (WSRC) Laboratory.

1.1 PURPOSE

There are two basic concepts for using optical absorption to measure tank waste moisture:

1. Non-contact camera systems similar to an infrared temperature sensing camera.
2. Cone Penetrometers or robotic arms that transport a sensor head to the waste surface.

This document provides the conceptual design and system requirements for a NIR system deployable using remote cameras for use in the Hanford Site's in-situ waste tank applications. Only non-contact camera system concepts are considered in this analysis.

The intent is to provide conceptual design options based on the operating restrictions and environmental characterizations for a NIR Spectroscopy Moisture Monitoring system that can be deployed into a Hanford Site waste tank through a tank riser. An in-situ NIR camera system based on these principles could have a positive impact on all aspects of the Hanford Site characterization mission, including ALARA, schedule, and costs.

1.2 BACKGROUND

Nuclear waste by-products from the Hanford Site weapon production missions are stored in a number of large single and double shell underground tanks. Access to the tank interiors is provided by a limited number of risers which range in size from 10.2 to 106.7 cm (4 to 42 in.) in diameter and (8 to 30 ft) in total length. Tank wastes generally consist of mixtures of hazardous chemicals and radionuclides typically found as layered salt cake, sludge, and supernatant. Some wastes generate toxic or potentially flammable gases and thus, equipment in the tank vapor space must meet the safety requirements for operation in a Class 1, Div 1, Group B (Hydrogen) environment. (NFPA 493 - National Fire Protection Assoc).

1.2.1 Problem Description

Moisture has been identified as one of the critical tank waste parameters that impacts the safety status of the wastes, particularly tanks containing ferro/ferricyanide materials. A graded safety assessment was developed as part of the answers to a prior Unreviewed Safety Question (USQ) which existed for these tanks. The assessment separated tanks into 3 categories, each with varying levels of moisture and total cyanide content (Meacham 1994):

1. Intrinsically Safe: The tank waste contains more than 24 wt% moisture content, unlimited total cyanide content, temperature < 90 °C.
2. Conditionally Safe: The tank waste contains less than 24 wt% moisture content, but no more than 8 wt% total cyanide, and temperature < 90 °C.
3. Unsafe: Tank waste contains less than 24 wt% moisture, more than 8 wt% total cyanide, and temperature > 90 °C.

Since water content is affected by a number of factors, including gravity, one hypothesis, currently being tested by Waste Tank Safety, is that the surface of the waste contains a minimum of water compared to the material deeper in the tank. Assuming this hypothesis is correct, a minimum internal waste water content will be obtained by measuring the surface water content.

At present, tank wastes are typically characterized by physically removing and analyzing core samples. The chemical analyses are completed in laboratories that are capable of safely handling radioactive materials. Alternative interrogation methods (such as remote NIR Spectroscopy) are urgently needed to provide faster, safer, cheaper, and better ways to assess tank wastes. Near infrared analysis is a nondestructive technique that take advantage of the tendency of water molecules to absorb specific wavelengths of NIR energy. When a sample containing water is exposed to those wavelengths, a certain portion of the energy will be absorbed by the water, and the remainder will be reflected. By measuring the reflected energy, the concentration of water in the sample can be determined (Reich 1993).

1.3 ASSUMPTIONS

1. The surface of the waste contains a minimum of water compared to the material deeper in the tank.
2. Optical losses will occur because of scattering from the waste surface.
3. Because of the high relative moisture in the tank head space, moisture will be the dominant atmospheric transmission absorber.
4. If the tank atmosphere normally contains an aerosol or fog, a "clear" condition occurs at some time in each tank when the NIR measurements may be obtained.

1.4 PRINCIPLES OF INFRARED OPTICAL ABSORBANCE

The molecular bonding of water (vibrational and rotational modes) produce strong optical absorbance zones that range from the visible through the infrared region. This absorbance is normally a problem when attempting to use optical methods for speciation and characterization in materials that contain any significant amounts of free water. However, these absorbance zones (which peak in several near-infrared, mid-infrared, and visible optical bands) can be used to indicate the concentration of water in a material.

The tank waste materials are largely found in slurries, wet salt cake, and some supernates. All of these forms are generally optically opaque which prohibits the use of optical transmission principles. However, optical reflectance methods potentially can be used as indicated by the strong absorbance spectrum from a 241-BY-104 simulant with different weight percent water shown in Figure 1-1. The spectral curves are from samples with increasing water content (0 to 24 wt%). The strong absorbance bands at 1.45nm and 1.9nm are clearly visible.

There are several potential methods using infrared absorption to measure waste moisture content. An optical remote sensor could be set up to transmit a beam of radiation across the tank vapor space (initial intensity I_0) that would be reflected off the surface of the waste to be monitored and returned to a detector (measured intensity I). However, gas molecules in the beam's path (water vapor, CO_2 , N_2O , CH_4 , etc.) will absorb a portion of the radiation intensity at specific wavelengths which are unique for each molecule. These absorption losses generate interference issues for a non-contact type camera concept.

Optical signal transmission is related to absorbance and scattering in the following manner:

$$\text{Transmission } (I/I_0) = \exp(-\{\text{Absorbance} + \text{scattering losses}\})$$

where absorbance is a function of the gas concentration, signal pathlength, and molecular structure.

The optical signal losses in the near infrared spectral region are mainly caused by water vapor and carbon dioxide. There will also be aerosol and molecular scattering losses, as well as waste surface scattering losses governed by the surface and the signal beam's incident angle.

In this analysis, optical losses from the aerosol scattering will be ignored. The assumption is that tank atmosphere aerosol existence is not continuous, but is dependent on external conditions, and that clear periods can be found at sometime during which the measurement campaign may be completed. If necessary, visual monitoring cameras could be deployed with the NIR camera systems to determine tank conditions.

1.5 MOISTURE MONITORING FEASIBILITY STUDY

A moisture sensing study completed by the University of Washington Center for Process and Analytical Chemistry (CPAC) in FY-93, indicated that the waste moisture could be sensed remotely using the optical scattering and absorption from the surface of a 241-BY-104 salt cake simulant (CPAC Moisture Study WHC-SD-WM-ER-288). Tank 241-BY-104 salt cake simulant is the most chemically representative of all the simulants. This hazardous simulant also has the highest average value for all mechanical and physical parameters measured.

CPAC compared the moisture measurement performance of three optical regions;

visible (0.400-1.1 micron),
near infrared (1.1-2.5 micron),
and mid-infrared (500-4000 cm^{-1}).

Calibration models were developed and tested, then were used to compare the relative sensitivity to moisture for each wavelength band. The best prediction results for the sample weight percents were for the near infrared bands (1.1 - 2.5 micron) using the second derivative of the sample data.

Figure 1-1 shows an example of the raw NIR data while Figure 1-2 show the resultant curves after the second derivative has been taken of the raw data in Figure 1-1.

Figure 1-1. CPAC Task #1 NIR Data Set

Figure 1-2. 2nd Derivative of CPAC Task #1 NIR Data Set.

For the NIR bands, a 0.5 to 1.5 wt% prediction error was measured for moisture concentrations from 0 to 20 wt% moisture content (Figure 1-3). This data was obtained with a non-contacting fiber optic probe with a small (0.25 to 1.0 in.) open atmospheric gap that can be ignored.

Figure 1-3. CPAC "Reported" 2-Factor, Centered, 2nd-Derivative Model.

The NIR optical region also appears feasible for the Hanford applications because optical components such as lenses, optical fibers, and filters are readily available with the ruggedness to handle the waste tank environment.

An initial investigation into the feasibility of remote sensing for hot cell and waste tank applications was also performed at CPAC (Reich 1993). In this test, a pulsed infrared test system operating from 1.41 to 2.12 micron was used to measure samples of 241-BY-104 salt cake simulant with differing amounts of moisture by weight (0 wt%, 10 wt%, 20 wt%) using a remote pathlength of 4.25 meters (14 ft). This experiment was designed to demonstrate the basic feasibility for a non-contact sensing concept. The major concern was the existence of a measurable optical signal reflecting off of a waste surface.

The test data showed that for these samples, 10 percent of the incident radiation is scattered. If collected, this signal would be available for determining moisture content. CPAC determined that the moisture content of the waste affects the scattering, but also noted that atmospheric relative humidity causes a signal attenuation that will impact any measurements being obtained.

2.0 CONCEPTUAL DESIGN DESCRIPTIONS

The studies by CPAC have shown a basic feasibility for measuring moisture from the NIR surface reflectance. There are several concepts for implementing this technology for the Hanford Site waste tanks: contact methods such as a Cone Penetrometer deployed sensor, and non-contact methods such as the remote NIR camera concept. Only the non-contact camera moisture monitoring system concept is addressed in this document.

2.1 REMOTE SYSTEM ALTERNATIVES

The fundamental concept for a non-contact, camera type moisture monitoring system is shown in Figure 3-1. The objective is to obtain moisture data as a function of the position on the surface of the waste. This requires an optical system with a relatively small field of view (considered a point source) and a system to systematically scan this over the waste surface. At each surface point, a segment of the NIR spectra will be recorded. Surface moisture data will then be extracted from this spectral response. NIR moisture monitoring systems are able to measure the surface moisture content.

Due to the size of the tank, it is assumed that focussing optics and scanning systems will be needed for both the light source and detector to provide a reasonable optical signal level. The focussing optics increase power density for a broad illumination source (other than a laser source) and provide a degree of signal discrimination for the detector.

There are two basic concepts for implementing surface scanning while maintaining a high optical signal, described as follows:

1. Scan source: A tuned laser source and a broadband detector.
2. Scan detector: A broadband source (focused) and a scanned detector.

2.1.1 Scanned Laser Source with Broad Band Detector

In this concept, the NIR spectra at a surface point would be obtained with a tunable laser source and a broad spectral band detector. One potential laser candidate is a NIR laser makeup of a NdYAG laser coupled to an optical parametric converter (OPC). The parametric converter takes a photon from the pump laser (NdYAG) and using a nonlinear crystalline medium converts the photon into two photons, individually of less energy but summing to the pump photon energy. The output wavelength is varied by changing the tuning of the OPC. At least one commercial source of these lasers is currently available over the near infrared optical range. The tunable laser output would be directed through optics and moveable mirrors (which keep the laser beam small) and systematically scan this beam across the waste surface.

Another possible "tunable" source would be an ensemble of single wavelength emitting lasers. Each laser would have a slightly different wavelength such that the ensemble of the outputs covers the NIR spectral

regions of interest. This would require a much more sophisticated optical scanning system to focus the output of each laser spot at the same tank surface position.

Several types of NIR detectors may be considered for this application, including an InGaAs detector with a 1.1-1.7 micron spectral response. Other detectors include some of the more exotic HgCdTe detectors which may require cooling to reduce noise. The detector would include collecting optics (probably reflecting elements) that provide the detector with a large optical collecting efficiency (small optical f/number). The optical axis would be made coincident with the laser and potentially scanned with the laser beam to enhance the system optical efficiency and background discrimination.

2.1.2 Scanned Detector with Broad-Band Laser Source

In this concept, the illumination source would be a broad-band source, such as a tungsten filament lamp, with optics that focuses the lamp output at or near the waste surface (a design trade-off would provide the focusing parameters for this optical system). The reflective light from the waste surface (broad-band light) would be collected by a high-efficiency optical collecting system coupled to a spectrometer and detector module. The detector would be a high-sensitivity broad-band detector, similar to those described above.

Note: The laser power density (watts/cm²) and the detector optical sensitivity amps/received power density (watts/cm²), as well as potential radiation damage to the detector, cable, optics, etc. is a design issue for both cases.

2.1.3 Radiation Impacts

If either the detector or the laser are sensitive to the adverse tank environment, particularly radiation, a periscope system using reflective components, or a fiber optic bundle could be used to couple the laser source light into the tank to the sensor head.

For fiber optic bundles, radiation resistant pure silica optical fibers have good optical properties in the NIR, 1.5 micron range, and are commercially available from several sources. Radiation testing has shown minimal darkening with pure silica fiber optics at dose rates up to 300 R/h. This option would change the interfacing optics within the tank and incur some optical losses by the fiber optic cabling. All other optical components could be made from radiation resistant components.

2.2 WASTE TANK INTERFACING ALTERNATIVES

The available tank access include 30.48 cm (12 inch) observation ports and 10.16 cm (4 inch) risers. Depending on the realization chosen and other considerations, the source and receiver can be inside or outside of the tank. If they are outside of the tank, either non-contact fiber optics or a fixed optic periscope will be required for the signal transfer.

Because of the tank environment, decontamination and decommissioning need to be planned for in the actual design. Optical windows or 10mil plastic bags can be used to protect the equipment from contamination.

2.2.1 Safety Classification and Required Approvals

In-tank monitoring instruments used for the Westinghouse Hanford Waste Tank applications for all waste tanks, other than 106-C and 101-SY, must satisfy a Safety Class 3 Equipment Classification level, before they may be installed, (Payne 1993). Safety Class 3 Equipment Classification level is for Systems and components that address the health and safety of facility workers and radioactive, chemical, or thermal releases to the environment.

At a minimum, documentation at WHC must satisfy a minimum Required Approval of (SQ) Documentation Approval Level for Safety Class 3 systems with Environmental Assurance (E) as deemed appropriate. An ESQ Required Approval is a designator for documentation that impacts occupational safety [including "as low as reasonably achievable" (ALARA) principles], environmental monitoring, and requires quality assurance verification of conformance to requirements.

Westinghouse Hanford Company is operated using the policy of ALARA for risks and radiation exposure. All interactions with hazardous and radioactive materials must be conducted in a manner to minimize risks and exposures. Good practices also requires the minimization of additional waste generation, specifically including the avoidance of needs for decontamination. Prevention of contamination is one of the preferred modes of operation.

Note: Systems used for monitoring waste tanks 106-C and 101-SY may need to satisfy a Safety Class SC-1* Equipment Classification level, contingent upon DOE granting a deviation against DOE 6430.1A. Documentation would need to satisfy an Impact Level SQ Documentation Approval Level for Safety Class 1* systems with Environmental Assurance (E) as deemed appropriate.

2.2.2 Quality Assurance and Quality Control

All work will be conducted in accordance with the relevant quality requirements of WHC-CM-6-1, EP-1.0, "Engineering Practices", i.e. Design Control, Design Verification, Change Control, Interface Control, Procedures, and Drawings. Hardware that is deployed in Hanford's waste tank applications must be in compliance with National Environmental Protection Act requirements and have appropriate documentation to address safety and regulatory issues associated with the deployment activity.

3.0 OPTICAL DESIGN ANALYSIS

The optical design considerations inside the Hanford Site waste tanks will be identical for the design alternatives discussed above. The optical considerations within the tank include:

- Absorption from the tank head-space atmosphere
- Waste material reflectance scattering variations
- Optical scattering from air-borne particles (fog).

Figure 3-1. Conceptual Implementation Geometry.

The analyses below are based on the conceptual implementation geometry shown in Figure 3-1.

3.1 HEAD SPACE ABSORPTION

Atmospheric absorption losses are a function of the transmission distance or signal pathlength. Figure 3-2 shows the NIR spectral range transmission losses for an 80 percent RH atmosphere for pathlengths of 0 to 60 meters. This data was computer generated using LOWTRAN, an atmospheric modelling program.

Figure 3-2. Atmospheric Signal Losses (80 Percent Relative Humidity)

NIR signal transmission through a tank vapor space may be affected by molecular absorption, molecular scattering, aerosol scattering, aerosol absorption, and turbulence (particularly for actively ventilated tanks). Vapor space molecular absorption in the NIR region will primarily be caused by water vapor (H_2O) and to a lesser extent by carbon dioxide (CO_2). Depending on the tank, methane (CH_4) and nitrous oxide (N_2O) may also be perceptible absorbers. Aerosols may contribute significantly to scattering when aerosol density is high, especially in those tanks that contain fogs. If aerosols and fogs are neglected, water and water vapor will likely be the dominant NIR absorbers because of the high moisture content in the tanks and the tank head space. Therefore, the impacts of other gases and atmospheric scattering will be neglected in this analysis.

To test the feasibility of calculating tank waste water content while using a remote system with atmospheric humidity effects, an apparent absorbance spectra was created, as follows.

For simulation, the CPAC 241-BY-104 simulant NIR reflectance spectra was used along with the atmospheric transmission data above, to generate an atmospheric modified sample set. Figure 3-3 shows the water absorption modified reflectance from the 241-BY-104 simulant saltcake.

The mathematical details for combining the spectral data are described in Appendix A.

Figure 3-3. Apparent Absorbance.

**Figure 3-4. 2nd Derivative of CPAC Data
Including Relative Humidity Transmission Effects.**

The second derivative of the generated spectral data (Figure 3-4) above compared with the sample derivative data previously presented (Figure 1-2 {see inset}) visibly shows the NIR region atmospheric water and carbon dioxide absorption band impacts on the 241-BY-104 NIR spectra.

3.1.1 Surface Contact Data Model

The data sets, Figures 3-3 and 3-4, can now be evaluated using the Principle-Least-Squares (PLS) Model that was developed previously. The Predicted versus Actual Weight Percent performance curve for the PLS model using spectra modified by atmospheric absorption is shown in Figures 3-5 and 3-6. The corresponding performance curve for the PLS model using the unmodified data was shown is Figure 1-3. This PLS model was generated for spectra unaffected by atmospheric moisture. As can be seen in Figure 3-5, this PLS model now "over" predicts the moisture content of the 241-BY-104 sample surface. The combined effects of the humidity and the pathlength are shown in Figure 3-8. This figure shows that a variable pathlength or a variable moisture content have the same impact on the NIR spectra. Each point on the plot is the integration of optical energy over the 1.3 to 1.45 micron spectral increment with a variable pathlength (0 to 33 meters). A 3 meter (9.8 ft) pathlength in a 50 percent RH atmosphere has the same spectral energy losses as a 7 meter (23 ft) pathlength in a 20 percent RH environment.

Figure 3-5. 2-Factor PLS Model (raw data).

Figure 3-6. 3-Factor PLS Model (derivative data).

Figure 3-7. Prediction Error for 6.5 wt% Sample Due to Signal Pathlength.

Figure 3-8. Atmospheric Transmission vs RH (percent) and Distance.

As can be seen in Figures 3-6 and 3-7 parameters such as relative humidity and atmospheric transmission distance affect the measurement of the significant wavelengths. Clearly, if additional parameters affect the data, then either the error needs to be acceptable or the calibration model should include these parameters.

3.1.2 Atmospheric Transmission Data Model

The following figures display the predicted weight percent values using the total CPAC data set modified to account for atmospheric transmission to generate the Principle Least Squares model.

Because of an offset in the CPAC raw data that appears to be related to weight percent, the generated raw data models performed better than the derivative models. It is possible that this offset will not exist in valid remote waste tank data.

The maximum water that 241-BY-104 simulant can assimilate is approximately 25 wt%. The CPAC 25 wt% data is sufficiently different that a 0-25 wt% model may be too broad in scope. The PLS model predictions imply that there might be an improvement in the estimate error if multiple models are used for smaller weight percent ranges. After an initial range scoping prediction to determine an appropriate model range, the data could be reanalyzed using a 0-15 wt% or 15-25 wt% model, for example.

Figure 3-9. 2-Factor PLS Raw Data Model
CPAC Task 1 Data With 80 and 100 Percent RH Data

Figure 3-10. 3-Factor PLS Raw Data Model
CPAC Task 1 Data With 80 and 100 Percent RH Data.

Figure 3-11. 2-Factor PLS Derivative Data Model
2nd Derivative CPAC Task 1 Data With 80 and 100 Percent RH Data.

Figure 3-12. 3-Factor PLS Derivative Data Model
2nd Derivative CPAC Task 1 Data With 80 and 100 Percent RH Data.

3.2 OPTICAL SCATTERING

The tank wastes are made up of both solids and liquids, with the bulk of the material being a slurry. A non-contact camera system will only see the surface of the waste which is hypothesized to contain less moisture than wastes deeper in the tanks. This will be an advantage, as indicated below, in that the dryer wastes are expected to exhibit broader optical scattering than wastes with high water content. The optical reflectance/scattering of the waste surface is expected to be dependent on a number of factors, including:

- size of the particulates in the wastes
- optical geometry between the waste surface, illumination source, and detector
- ratio of solid to liquid

3.2.1 Optical Geometry:

The optical geometry between the illumination source, the waste surface, and the detector will have an impact on the back scattered light that is available to the detector. In a scale-demonstration completed by CPAC under contract to WHC, CPAC estimated that 10 to 20 percent of an incident laser beam was scattered by a 241-BY-104 simulant with moisture contents of 0, 10 and 20 wt%. The optical setup for these tests was based on an optical geometry where the an illuminating laser beam was incident at a 45 degree angle to the waste surface with a detector aligned at a 45 degree angle to the plane of incidence formed by the laser and waste surface. This arrangement assured that the detector would not be seeing any specular reflectance from the 241-BY-104 material. In addition, there is no free water in the simulant for these moisture concentrations. These tests did not show the results of potential geometries in the tank where angles larger than 45 degrees between waste surface, laser and detector can be produced.

Some insight into optical scattering can be obtained by examining the optical scattering from ideal surfaces. Figure 3-13 shows examples of optical scattering from different idealized surfaces for an illumination beam that is normal to the plane of the surface (90 degrees to the surface plane which is the horizontal plane). This is a polar plot where the radius from the axis apex is plotted as a function of angle from the vertical axis which is coincident with the axis of the illuminating beam. As a reference, two 45 degree lines are shown on the plot, one at a +45 degree angle from the reference vertical axis and one at a -45 degree angle.

The larger, outer curve shows the expected optical scattering from a Lambertian or ideal surface scatterer. This probably is close to what we would expect from a dry salt cake made up of small particles. Other scattering patterns have a decreasing width and show what happens to the optical scattering as a surface becomes more mirror-like or has increasing specular reflectivity. In this case, the limit for specular reflectance is a perfect mirror which will reflect the incident beam back on itself. Wastes with high moisture content are expected to exhibit more specular reflection than wastes with less moisture. This has been observed with simulants. This is not a particularly devastating disadvantage because wastes with high water content will probably be beyond the safety limits established for the waste

materials. The low moisture areas where moisture content is an issue are expected to exhibit good optical scattering, supporting a non-contact camera type of measurement concept. The scattering plots also show that a decreasing level of light as the incidence angle increases. This means that in a waste tank application, we can expect the optical signal level to vary as the waste surface is scanned.

Figure 3-13. Optical Geometry.

CPAC's test with 241-BY-104 simulant, conducted at a 45 degree angle of incidence for the illumination and detector, demonstrated the feasibility for the initial non-contact camera concept. Further testing is necessary to determine the magnitude of the sample signal scattering levels, the angular sensitivity, and any optical limitations that prevent the acquisition of NIR spectra and the extraction of moisture content from it.

3.2.2 Ratio of Solid to Liquid:

Based on photographic surveys and core sample analyses, tanks with high water content generally have exposed salt cake as well as areas of standing water. The liquids are made up of water that is usually saturated with ions and has a high pH (12 to 14). The refractive index of these liquids will be

higher than that of pure water which raises the optical reflectance. To near infrared light, those surface areas with standing liquids are expected to behave like mirrored surfaces which have strong specular reflectance and very little scattered light. The issue is that the low backscattering from the liquids will make these standing liquid areas appear black, as evidenced by photographs taken using backscattered light in the tanks. This is not a moisture issue, but rather a signal processing issue where these optically black areas must be dealt with by the signal processing system.

Testing with simulant materials by WHC indicated that the optical depth of penetration in a sample is dependent on the water content and the opacity of the sample. Optical tests with dry samples of sodium nitrate, which is translucent, had a very small depth of penetration for an incident light beam. The dry surface of the sodium nitrate was a very good optical scatterer. When water was added to these same samples, the optical depth of penetration increased significantly as indicated by the increased light escaping from a sample vial and by changes in the optical spectra due to interferences from the sides of the vials. The conclusion from this is that the sample volume for an optical backscatter measurement with waste materials may not be limited to a thin, micron thick, surface. The water content will couple light deeper into the sample, increasing the interrogation volume of an optical beam.

Currently a contract is in place with the University of Idaho to study the impact of sample morphology (particle size, moisture content, absorbance) on the "sampling volume" of an optical beam.

3.2.3 Size of Waste Particulates

At this time we can say very little about the impact of particulate size, except that it will affect optical scattering. Detailed hot cell analysis of tank wastes have indicated particle sizes for most of the wastes are in the 1-50 micron range. This should produce good optical scattering for low moisture content wastes. More on this will be learned when the University of Idaho completes their study on the impacts of particle sizes with optical probes.

4.0 FUNCTION AND DESIGN REQUIREMENTS - WASTE TANK ENVIRONMENT DESCRIPTION

Fiber optic probes and cameras must be capable of handling the environment created by the radioactive, caustic waste, as well as the physical environment from the deployment activities. The laser and the spectrometer would be outside the harsh tank environment in a protective shelter, but would still be exposed to the blowing dust and other factors of an outdoor operating environment.

4.1 HANFORD SITE ENVIRONMENT

Components external to the tank, such as cable sections and connectors waiting to be used, will be located in what is classified as an outdoor environment. Environmentally sensitive monitoring components shall be

protected by appropriate enclosures. However, although the equipment will be protected by enclosed shelters, access to the shelter will expose the equipment to the environment. This shelter could be located either outside or inside the tank farm perimeter fence, or next to a tank riser.

Hanford Tank Farm Surface Environment

ambient temperatures: ranging from -20°F to 120°F
relative humidity: from 5 percent to 100 percent
wind speeds: up to 80 miles per hour with blowing dust
radiation dose rate:

- a.) In the immediate vicinity of the tank risers, dose rates range from less than 0.5 mR/hr to 70 mR/hr.
- b.) background (outside of a. above) less than 0.5 mR/hr.

4.2 IN-SITU WASTE TANK ENVIRONMENT

4.2.1 In-Tank Physical Data

Temperature:	65° - 190°F
Pressure:	-2.5" to +1" H ₂ O (measured in head space)
Radiation flux:	75 - 900 R/hr (in head space)
• Radiation:	Up to 10.5E+03 R/hr (gamma and beta)
gamma:	within waste less than 2E+03 R/hr
Relative Humidity:	10 percent - 100 percent (head space)
Moisture Content:	>20 percent for solid waste
Specific Gravity:	1.2 to 2.0 for solid or liquid waste
Corrosivity:	Condensed vapors - pH from 8 to 14 liquids - pH from 8 to 14, up to 3.56M NaOH solids - pH from 8 to 14 and up to 3.56M NaOH
Hazardous location:	Class 1, Division 1, Group B per NEC Article 500.

4.2.2 In-Tank Chemical Data

A brief summary of the chemical data for the contents of Tank 101-SY is presented below. This data provides some typical information of the environment in which a fiber optic probe would need to function. All values and ranges are approximate.

In general, the physical environment and the list of waste chemical composition below can be applied to most of the waste tanks on the Hanford Site. Depending on the specific tank, other important constituents such as ferrocyanide may be added to the list. The mass of ferrocyanide included in the waste in the single-shell tanks can vary from 0 - 1 Kg-moles up to 100 - 200 Kg-moles.

SOLID SLUDGE SUMMARY

<u>Chemicals</u>	<u>Concentrations</u> molarity (kg/liter)	<u>Probable major mineral phases:</u>
sodium	7 - 16	
nitrate	1.5 - 3	sodium nitrate
nitrite	1.3 - 3.4	sodium nitrite
hydroxide	1.2 - 2.7	sodium aluminum hydroxide
aluminum	0.7 - 2.5	
carbonate	0.2 - 0.5	sodium carbonate
chloride	0.2 - 0.4	
phosphate	0.06 - 0.2	
sulfate	0.03	
TOC ^a	9 - 25 g/L	
Sr-89/90	$6 \times 10^3 - 7 \times 10^5$ $\mu\text{Ci/L}$	
Tc-99	$9 \times 10^1 - 2 \times 10^2$ $\mu\text{Ci/L}$	
Cs-137	$3 \times 10^5 - 7 \times 10^5$ $\mu\text{Ci/L}$	
Pu-239/240	$8 \times 10^0 - 8 \times 10^1$ $\mu\text{Ci/L}$	

^a Total Organic Carbon

Note: Concentrations of constituents tend to increase with depth.

4.3 SIMULANT PHYSICAL PARAMETERS

Currently, little data exists for physical characterization of single shell tank saltcakes. However, several simulants were developed for temperature envelope testing of a universal core sample drill bit (Wanner 1993). Although the simulants were designed to emulate the chemical nature of the Single Shell Tanks, only major chemical components were used in the synthetic formulation. The effects of tank aging with crystal growth, as well as additional minor component chemical impurities are not accounted for. Additionally, all organics, heavy metals, and radionuclides have been omitted to simplify waste disposal requirements and to minimize any potential safety hazards.

More substantial data is needed to verify the following synthetic saltcake properties. Each simulant was developed to represent waste salt cake and does not address the sludge or the supernate properties.

Engineering Testing and the Geotechnical Engineering Laboratory at Westinghouse Hanford Company physically tested Sulfur K-mag and three synthetic saltcake samples for the temperature envelope testing of a universal core sample drill bit, and to support activities related to testing and verification of a large cone penetrometer.

Tank 241-BY-104 salt cake simulant was the most chemically representative of all the simulants. This hazardous simulant also had the highest average value for all mechanical and physical parameters measured.

4.3.1 Tank 241-BY-104 Simulant

The simulant composition below was initially based upon analytical data obtained in 1976. The analysis contained limited chemical and physical data for both sludge and supernate, however potential flaws in the composition have been identified. To produce a uniform saltcake in the 241-BY-104 simulant, soluble sodium metasilicate, calcium nitrate, magnesium nitrate, and manganese nitrate were used instead of insoluble silicon dioxide, calcium carbonate, magnesium oxide, and manganese oxide. The additional sodium hydroxide used to precipitate ferric hydroxide from ferric nitrate was omitted. This modified 241-BY-104 simulant data provides an estimate of some of the chemical and physical properties expected in the salt cake.

Tank 241-BY-104 Modified Simulant Recipe

<u>COMPOUND</u>	<u>FORMULA</u>	<u>WEIGHT %</u>
sodium nitrate	NaNO_3	81.8%
sodium aluminate	NaAlO_2	7.5%
sodium hydroxide	NaOH	1.7%
sodium metasilicate	Na_2SiO_3	1.5%
ferric nitrate	$\text{Fe}(\text{NO}_3)_3$	1.0%
sodium phosphate	Na_3PO_4	0.7%
calcium nitrate	$\text{Ca}(\text{NO}_3)_2$	0.4%
magnesium nitrate	$\text{Mg}(\text{NO}_3)_2$	0.2%
manganese (II) nitrate	$\text{Mn}(\text{NO}_3)_2$	0.2%
Water	H_2O	5-25%

4.3.2 Simulant Physical Parameters

241-BY-104 SIMULANT TESTS (averages)					
BULK DENSITY	UNCONFINED COMPRESSION	DIRECT SHEAR STRENGTH	PENETROMETER RESISTANCE	THERMAL CONDUCTIVITY	
(gm/cm ³)	Stress (lb/in ²)	Stress (lb/in ²)	Resistance (lb/in ²)	Temp. (K)	Thermal Conductivity (W/m*K)
1.41	3264	459	34507	334	1.66

5.0 CONCLUSIONS AND RECOMMENDATIONS

1. The absorption of the water in the waste tank head-space atmosphere strongly impacts the NIR back-scatter spectra from the waste surface and must be factored into the design of the sensing system and calibration models.
2. A PLS model, constructed with atmospheric moisture as an interference can predict moisture from NIR spectral data independent of atmospheric moisture levels and path-length variations. Experimental work should be completed to verify the model performance with atmospheric interferences compensated by the PLS models
3. A path-length/relative humidity compensation model will need to know path-length within 1-2 meters and RH to +/-5 percent to keep error impacts on a PLS model below an estimated 1-2 wt%. Experimental work is needed to verify the magnitudes of these errors.
4. Water content, particle size, and geometry will affect the strength of optical back-scatter from a waste surface. More experimental work is needed to quantify the impact of angle and water content on signal strength for the camera concept.

The next step with the non-contact camera concept is to complete a field-scale demonstration. Simulant materials with varying moisture content need to be used in a mock-up with tank-scale dimensions to demonstrate surface moisture mapping. The mockup camera system for this test would be either the source scan or the detector scan concept.

6.0 REFERENCES

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NATIONAL CODES AND STANDARDS

American National Standards Institute

ANSI Y14.5m - Dimensioning and Tolerancing for Engineering Drawings

National Fire Protection Association

NFPA 70 - National Electrical Code

NFPA 493 - Standard for Intrinsically Safe Apparatus and Associated Apparatus for Use in Class I, II, and III, Division 1, Hazardous Locations

NFPA 497A - Recommended Practice for Classification of Class I Hazardous (Classified) Locations for Electrical Installation in Chemical Process Areas

NFPA 497M - Manual for Classification of Gasses, Vapors, and Dusts for Electrical Equipment in Hazardous (Classified) Locations

Underwriter's Laboratory

UL 698 - Standard for Industrial Control Equipment for Use in Hazardous Locations

UL 913 - Standard for Intrinsically Safe Apparatus and Associated Apparatus for Use in Class I, II, and III, Division 1, Hazardous (Classified) Locations.

APPENDIX A

OPTICAL RELATIONSHIPS

The purpose of this discussion is to identify the optical relationships involved in using optical reflectance for spectroscopy applications. The Hanford Site tank waste materials are made up largely of salt cake and slurries forms with some supernate. For the waste tank materials, we will use optical reflectance measurements to obtain spectroscopy related information. Although the preferred method for optical spectroscopy is a transmission measurement where pathlength can be carefully measured and controlled, use with high scatterers or absorbers is not practical.

Figure A-1. Open-Path System.

In this analysis, we are assuming an optical open-path system architecture as shown in Figure A-1. Optical energy, $I_s(\lambda)$, emitted from a broad band light source, travels through an atmospheric path of d before it intercepts the surface of the waste. The (λ) shows that the optical energy is a function of optical wavelength. The optical energy at the surface of the waste will be spectrally modified by the absorption of the atmosphere, particularly the atmospheric moisture. Therefore the energy at the waste surface will be:

$$\begin{aligned} I_i(\lambda, x) &= T_a(\lambda, x) * I_s(\lambda) \\ &= 10^{-a(\lambda, x)} * I_s(\lambda) \end{aligned}$$

A-1

where $I_s(\lambda)$ is the energy at the output of the light source. $T(\lambda, x)$ is the spectral transmission of the atmosphere as a function of wavelength, λ , and pathlength x , which is expressed as atmospheric absorbance $a(\lambda, x)$. $I_i(\lambda, x)$ is the optical energy at the waste surface modified by the absorption from traversing a path x in the tank head-space.

After intercepting the waste surface, some of the optical energy will penetrate the waste and be absorbed by water and other tank waste constituents. The refractive index differences between the tank materials, water and trapped gases will also produce a strong optical scattering. Some of the scattered light will exit the surface of the waste in the direction of the optical detector. The reflected light, since it carries spectral information about waste content, will be roughly proportional to light that has transmitted through an unknown thickness of waste. Therefore we can express this reflected light as:

$$\begin{aligned} I_R(\lambda, x) &= T_w(\lambda, x) \\ &= 10^{-b(\lambda, x)} * I_i(\lambda, x) \end{aligned} \quad A-2$$

where $T_w(\lambda, x)$ is the transmittance of the waste which is then expressed as $b(\lambda, x)$, the optical absorbance that will be dominated by the water in the waste material.

This reflected light will then reach the detector after being modified by the atmospheric absorption of the head-space. Similar to A-1 above, the optical energy at the detector can be expressed as:

$$I_d(\lambda, x) = 10^{-a(\lambda, x)} * I_R(\lambda) \quad A-3$$

If we combine A-1 through A-3, the optical energy at the detector will be:

$$I_d(\lambda, x) = 10^{-a(\lambda, x)} * 10^{-b(\lambda, x)} * 10^{-a(\lambda, x)} * I_i(\lambda) \quad A-4$$

Using the optical definitions from Reference 1, which are as follows:

$$\begin{aligned} \text{optical opacity} &= 1/(\text{optical transmittance}) \\ \text{optical density} &= \text{optical absorbance} \\ &= \log(\text{opacity}) \\ &= \log(1/\text{optical transmittance}) \end{aligned}$$

or,

$$\text{optical transmittance} = 10^{-(\text{optical absorbance})}$$

We then can express equation A-4 for our open-path optical system in optical terms as follows:

$$\begin{bmatrix} \text{Optical} \\ \text{energy at} \\ \text{the detector} \end{bmatrix} = \begin{bmatrix} \text{Atmospheric} \\ \text{Optical} \\ \text{Transmittance} \end{bmatrix} * \begin{bmatrix} \text{Waste Water} \\ \text{Optical} \\ \text{Transmittance} \end{bmatrix} * \begin{bmatrix} \text{Optical} \\ \text{Source} \\ \text{Energy} \end{bmatrix}$$

Reference:

Leo Levi, Applied Optics: A Guide to Optical System Design, Volume 1; John Wiley and Sons, New York, NY., 1968; pp 4-5.