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7. Abstract This report discusses work done to investigate the feasibility of using non-contact optical absorption to remotely sense the surface moisture content of salt cake materials. Optical measurements were made in a dimensionally scaled setup to investigate this technique for in-situ waste tank applications. Moisture measurements were obtained from BY-104 simulant samples with 0 wt%, 10 wt%, and 20 wt% moisture content using the back-scattered light from a pulsed infrared optical parametric converter (OPC) laser source operating from 1.51 to 2.12 micron. An InGaAs detector, with 0.038 steradian solid angle (hemisphere = 6.28 steradians) collection angle was used to detect the back-scattered light. This work indicated that there was sufficient back-scatter from the BY-104 material to provide an indication of the surface moisture content.		
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Task 2B Report

**Investigation of Remote Sensing
Scale Up for Hot Cell and Waste Tank Applications**

**CPAC Optical Moisture Monitoring of Hanford Waste Tanks
Project**

Westinghouse Hanford Company

Contract MRL-SVV-562951

Prepared by

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Table Of Contents

Summary	1
Introduction	2
Experimental Layout	3
Figure 1	3
Light Source and Optical Path	3
Table I	4
Sample Handling, Geometry, and Scattered Light Detection	5
Figure 2	5
Figure 3	7
Data and Analysis	8
Table II	8
Table III	9
Atmospheric Water Vapor Interference	10
Table IV	11
Conclusions	12

SUMMARY

We report the results of measurements that provide preliminary insight into the feasibility of using non contact remote sensing methods for salt cake moisture content tracking. A pulsed infrared Optical Parametric Oscillator (OPO) source operating from 1.51 μm to 2.12 μm served as the light source for the Task 2B measurements during a three day trial period at the OPO manufacturer's facility in Santa Clara, CA. The data show that (1) at least the order of 10% of the incident radiation scatters, (2) the moisture content of the salt cake affects the scattering, (3) moisture in the atmosphere affects the measurements as conducted, and (4) additional work is needed to establish the precision of the moisture sensing concept. The experiments create the foundation for definitive measurements to be made in the follow-on Task 3 D that is to be conducted before August 1994, and help to define what their layout might entail. This work was supported by the U. S. Department of Energy's Tank Waste Remediation System Program (EM-30).

Introduction:

Task 2B: *Investigation of Remote Sensing Scale Up for Hot Cell and Waste Tank Applications*, provided a format for preliminary investigations on questions concerning whether the basic methodology for moisture content measurement can be scaled to tank dimensions and conditions. Of particular interest to the research team were the use of pulsed sources, such as lasers or optical parametric converters, as the scattering light sources, and the influence of head space moisture on the observed light scattering. Since the dimensions over which the measurements would be made in a tank setting are hundreds of times larger than those made using infrared, or near infrared, spectrometers, higher brightness (power per unit area and frequency) from the source may be required to obtain good signals. The possibility of the head space moisture influencing the results of the measurements represents a potential source of systematic error which could influence the feasibility of remotely monitoring the moisture in tank salt cake.

In this task we investigated the basic parameters of the problem on a scale larger than that for spectrometers but reduced from the actual tank dimensions. Our experiments investigated the feasibility of using pulsed, high brightness sources for making moisture measurements, the signal levels obtainable, and the effect of moisture along the path on the results. Comparison of the data with data from NIR reference spectra taken with a spectrometer provide a basis for assessing the results.

Experimental Layout:

We designed this initial set of experiments on the basis of simplicity and utility in defining future experimental layouts. Figure 1 provides the schematic layout of the Task 2B experiments reported here.

Figure 1: Experimental Schematic

light source and optical path:

The light source consisted of a Optical Parametric Converter (OPC) and a Nd^{3+} :YAG pump laser. The parametric converter takes a photon from the pump laser and using a nonlinear crystalline medium converts the photon into two photons, each of less energy but summing to the pump photon energy. In this way tunable near and mid infrared light may be generated. In operation, the system performs like a dye laser but with a much greater tuning range.

Table I summarizes the typical performance specifications of the light source. The output of the converter contained two orthogonally-polarized components, a signal and an idler, whose photon energies summed to that of one $1.06 \mu\text{m}$ pump photon. The signal wavelength stepped scanned from $1.51 \mu\text{m}$ to $2.12 \mu\text{m}$ while the simultaneous idler wavelength scanned from $4.0 \mu\text{m}$ to $2.12 \mu\text{m}$. Based upon the results of Task 2A, the region of interest for the highest precision measurements is

from 1.1 μm to 2.5 μm , with special interest at the edges of strong water absorption such as at 1.59 μm . We employed a sapphire prism in separating the two wavelengths and directed the signal, 1.51 μm to 2.12 μm , through two alignment apertures and onto the sample: the separation scheme required realignment of the converter output to the sample with each change in wavelength. Typically one to five millijoules per pulse reached the sample. At the most sensitive detector wavelengths, neutral density filters reduced the laser pulse energy into the microjoule range.

Table I: OPC Performance Summary

SPECIFICATION	VALUE	COMMENT
Tuning Range	1.45 - 4.0 μm	signal and idler
Pulse Energy	8 mJ 10 mJ 8 mJ 4 mJ 2 mJ 0.5 mJ	1.5 μm 2.0 μm 2.5 μm 3.0 μm 3.5 μm 4.0 μm
Pulse Repetition Rate	10 Hz	set by pump
Pulse Length	2-5 nsec	nominal and dependent upon pump pulse length
Bandwidth	- 6 cm^{-1}	measured in near-IR
Beam parameters	<5 x D.L. producing a 6.5 mm diameter spot at the sample	starting with a 4mm spot diameter and a divergence of $1.55 \times 10^{-3} \text{ m/m}$
Nd:YAG Pump Requirement	>150 mJ at 532 nm and >350 mJ at 1.064 μm	separable, linear polarizations

Mirrors created a path length of 4.25 meters between the converter output and the sample. The spot size produced at the sample was roughly 6.5 mm. To maintain alignment with changes in the wavelength, we carefully centered the beam through two apertures placed two meters apart immediately before the sample after each step in wavelength. Prior to each measurement we also inserted a calibrated power meter in the

optical path before the sample, and after the second aperture, and measured the power being delivered at ten pulses per second.

sample handling, geometry, and scattered light detection:

For these tasks three samples of BY104 salt cake simulant were prepared, each with a differing amount of moisture by weight; 0 wt%, 10 wt%, and 20 wt%. Figure 2 shows the reference spectra for the samples taken with a conventional near infrared spectrometer with fiber attachment. The spectra show the variation in absorbance with wavelength and moisture content. The spectra illustrate the basic trends to be expected in the scattering data reported here.

The samples remained sealed until used in the experiments. A 45-degree inclined plane mounted on a stand supported a pile of each sample for the experiment; Figure 3 shows the arrangement. This arrangement allowed a simulation of striking a salt cake surface while maintaining the sample integrity and an open geometry for scattered light detection. The highest moisture content sample required mixing before use since some separation had occurred.

***Figure 2: Salt Cake Simulant Reference Spectra
Absorbance as a Function of Wavelength***

An InGaAs detector with built in preamplifier provided scattered light detection for these first experiments. The detector is most sensitive in the range from less than 0.7 μm to 1.5 μm , outside of which its response decreases strongly. The detector received the light incident on an all metal off-axis parabolic reflector located 15 cm from the sample and capable of cleanly focusing onto the small InGaAs detector element. The collection optic was located at the same height as the incident light beam and at about 45 degrees off the plane of incidence. The distance between the sample and the collection optic was set by space considerations and the need to isolate it inside a light shield.

Detectors located at larger distances or with different optics may be compared to these results by accounting for differences in the solid angle the two systems intercept. The collection optic in this experiment intercepted a solid angle fraction, defined as the fraction of the 2π radian half sphere intersected by it, of roughly $\Delta\Omega_1/\Omega = 6 \times 10^{-3}$. For a similar collection optic at 100 times the distance, we have $\Delta\Omega_2/\Omega = 6 \times 10^{-7}$. The expected signal levels in the second case would be 10^{-4} of those observed here.

The detector showed a high sensitivity out to 1.6 μm but it dropped rapidly at longer wavelengths as expected. To account for the varying detectivity, the signals for the 10 wt% H_2O and 20 wt% H_2O samples are normalized to those of the 0 wt% sample. The detection system and sample were surrounded by a light shield to reduce scattered 1.064 μm light from the OPC from reaching the detector.

Figure 3: Sample Arrangement

Each measurement consisted of stepping the laser to a given wavelength, checking the alignment through the apertures, measuring the energy per pulse being delivered to the sample, measuring the background noise, and the signal given by the peak voltage measured on the oscilloscope monitoring the detector output.

Data and Analysis:

Table II provides the raw data taken using the experimental set-up described above.

Table II: Raw Data

Wavelength (microns)	Energy per pulse (mJ)	Baseline (mV)	[Signal + Baseline] (mV)	Net Signal (mV)	Net Signal /Energy per pulse (mV/mJ)
0% H ₂ O					
1.51	4.0E-04	0.0	2130.0	2130.0	5325000.0
1.59	3.8E-03	0.0	8900.0	8900.0	2342105.3
1.67	2.0	82.0	3820.0	3738.0	1869.0
1.77	4.2	15.0	181.0	166.0	39.5
1.83	6.4	15.0	245.0	230.0	35.9
1.89	5.8	23.0	204.0	181.0	31.2
1.96	5.4	20.0	125.0	105.0	19.4
2.04	6.5	20.0	151.0	131.0	20.2
2.12	4.5	26.0	76.0	50.0	11.1
9.97% H ₂ O					
1.51	4.0E-04	150.0	1630.0	1480.0	3700000.0
1.59	2.5E-03	100.0	4420.0	4320.0	1728000.0
1.67	2.6	80.0	5100.0	5020.0	1930.8
1.77	10.0	50.0	1540.0	1490.0	149.0
1.89	9.6	0.0	860.0	860.0	89.6
2.04	7.7	13.0	244.0	231.0	30.0
2.12	5.0	15.0	208.0	193.0	38.6
19.97 % H ₂ O					
1.59	3.8E-03	70.0	2420.0	2350.0	618421.1
1.67	2.6	15.0	9950.0	9935.0	3821.2
1.77	8.4	-17.0	240.0	257.0	30.6
1.89	6.3	-13.0	52.0	65.0	10.3
2.04	6.7	-15.0	46.0	61.0	9.1
2.12	4.6	-14.0	53.0	67.0	14.6

The sixth column of Table II provides the net peak voltage signal normalized to the incident pulse energy. Because the detector shows a strong roll-off in response with increasing wavelength, the data for the 10 wt% sample and the 20 wt% moisture sample are ratioed to the 0 wt% moisture sample in order to account for the wavelength dependence of the detector. Columns 2 and 3 of Table III presents the results of ratioing the data to that for 0 wt%. A similar comparison for the reference spectra of Figure 2 can be made if the data of Figure 2 is converted from absorbance units to reflectance. We converted portions of the reference spectra absorbance data in Figure 2 to reflectance data using the relation given by the manufacturer, that is,

$$R = \frac{1}{10^{\text{Absorbance}}}$$

The comparison values for the reference spectra made using the conversion are presented in columns 4 and 5 of Table III.

Table III: Ratioed Data for Scattering Using Pulsed Light Source and NIR Spectrometer

wavelength in microns	10% relative to 0%	20 % relative to 0%	NIR Reference 10% relative to 0%	NIR Reference 20% relative to 0%
1.51	0.7			
1.59	0.7	0.3	0.45	0.21
1.67	1.0	2.0	0.44	0.20
1.77	3.8	0.8	0.41	0.16
1.89	2.9	0.3	0.23	0.04
2.04	1.5	0.5	0.21	0.04
2.12	3.5	1.3	0.26	0.07

From the tabulated data we observe that the measured absolute magnitudes between the ratios measured in this experimental arrangement and those from the reference spectra differ, though each data set generally show similar trending. For example, the data at 1.59 μm shows a decrease in the reflected light between 10 wt% and 20 wt% moisture samples of 2.3 and 2.1 times for the measured and reference data respectively. The fact that the measured pulsed data follow the

trends expected on the basis of the NIR spectral data supports the idea that remote sensing using pulsed sources is possible. The difference in the magnitudes may reflect the differing collection geometries in the two experiments. We note that an exception to the correlation between the NIR reference data and the Task 2B data occurs at $1.67\text{ }\mu\text{m}$ and no explanation is readily available. We point out however that each data point taken in the present experiment is taken by hand and is not the result of averaging. It is possible that an error in the measurement was made. However, both sets of data show the same trends across the wavelength range.

We have made no attempt to derive a calibration set from this data. The main thrust of the present experiments was to demonstrate that reasonable signals could be obtained in a control experiment using LIDAR-like remote sensing. An important parameter in determining the feasibility of using remote sensing is the extent to which the solid scatters in the mid infrared. An estimate of the fraction of incident light scattered from the sample may be made by estimating the energy incident on the detector and knowing the approximate collection on efficiency of the optical system. The estimated detector factor at $1.59\text{ }\mu\text{m}$ is $2 \times 10^6\text{ V/W}$ based upon a comparison of detectors in this general class. With an observed voltage of 8.9 V arising from $4 \times 10^{-6}\text{ J}$ incident on the sample, a signal duration of roughly $400\text{ }\mu\text{sec}$, and a solid angle collection factor of 6×10^{-3} , we estimate that between 10 % and 20 % of the incident light is scattered. This rough order of magnitude comparison is consistent with expectations on the scattering efficiency of a solid and that no pathological problems in the surface scattering properties of the salt cake exist.

Atmospheric Water Vapor Interference

We conducted a simple measurement to verify that atmospheric water vapor would affect the observed measurements. Using an ultrasonic vaporizer we directed water vapor into the optical path to the sample. The path length and actual humidity content were not determined but we did measure the pulse energy after the major region influenced by

the vapor source in order to correct for simple absorption of the incident radiation. Table IV gives the results of the measurements.

Table IV: Measured Net Signals Corrected for OPO Output Energy with and without Water Vapor in the Optical Path

wavelength	Net signal without water vapor in path (mV)	Net signal with water vapor in path (mV)
1.59 μm	2350	1040
1.89 μm	5.8	5.0
740 nm	4750	3000

One sees that the water vapor causes an attenuation of the signal. Since most of the incident radiation loss due to absorption or scattering of the light to the sample is accounted for, the observed decrease may be due to increased absorption between the sample and the detector. The strongest change in signal strength occurs near the OH first overtone, appearing at 1.45 μm for the solids under study.

One simple method of avoiding the atmospheric water lines in future experiments would be to use a narrow bandwidth source (less than 3 GHz linewidth) for the scattering source. This would allow tuning between absorption atmospheric H₂O absorption features. Because of the broadening of the absorption lines of the water in the solids, this should have little or no effect on the desired signal while greatly reducing the atmospheric interference.

Conclusions

The simplicity of the experimental set-up and the lack of a detector optimized for the measurements limits the present results by limiting their precision and the ability to take large amounts of data for signal averaging. However the data do show that in general we can observe reasonable single shot signals at very modest energies, that the observed trends parallel those observed in data from a conventional NIR spectrometer, and that the magnitude of the scattering is consistent with scattering from solids (typically 0.1 to 0.9 of the incident radiation). A simple test illustrates that the presence of atmospheric water could strongly influence the measurements at wavelengths where the water absorption is strong and that the next round of experiments should be prepared to deal with this issue more fully.

The experiments done to date provide direction for the next phase of work. A major concern to be addressed in the next rounds of experiments must be whether or not remote sensing measurements have the inherent statistical precision required for moisture monitoring. The next experiments should be conducted in a manner to allow signal averaging, use a better optimized detector, and allow purging of the light path and detector area for detailed studies on the sources of systematic and random errors, such as water vapor and pulse energy fluctuations.

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