Rapid Characterization of Mixed Waste by FTIR-Fiber Optic Method

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RAPID CHARACTERIZATION OF MIXED WASTE BY FTIR-FIBER OPTIC METHOD

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ABSTRACT

Tank waste characterization requires various analytical systems to identify and quantify the chemical composition and water content of Hanford Site high-level waste. Safe long-term storage of the waste depends on its chemical and physical data. An analytical database is also the key to the design and implementation of pre-treatment and disposal processes.

To provide a faster, cheaper, and safer technique to monitor the moisture content of tank waste, two types of near-infrared (NIR) diffuse reflectance fiber optic probes interfaced to a Fourier transform infrared (FTIR) spectrometry system were studied. Lower absorptivities in the NIR region enable longer pathlengths to be used leading to easier nondestructive sampling. Both overtone and combination bands of water can be used for moisture measurements. While a previous report[1] provides evidence for the feasibility of using fiber optic probes, the results were strictly qualitative. In this study, the fiber optic probe is installed in a hot cell making it possible to characterize highly radioactive mixed waste rapidly and quantitatively.

In seeking a strategy to identify individual species in the waste with minimal sample preparation, a modular transfer optic system equipped with a mid-infrared diffuse reflectance sampler was assessed. Light pipes were used to present the sample to the FTIR spectrometer. Its performance for obtaining rapid, high quality mid-infrared (MIR) spectra of mixed waste is compared with FTIR-photoacoustic spectroscopy.

I. INTRODUCTION

Hanford Site waste tanks contain high-level mixed waste in the form of sludge, saltcake, and supernates. Moisture has been identified as one of the critical parameters that impacts the safety of these tanks, particularly those containing organics and cyanoferrates. It has been postulated that significant concentrations of fuel (organics and/or cyanoferrates) in contact with oxidizers (nitrates and nitrites) under dry conditions and elevated temperatures could undergo propagating exothermic reactions. Experiments have shown that a tank waste with moisture content greater than 17 wt% and organics less than 5 wt% regardless of its cyanoferrate content is considered safe. [2,3] Chemical analyses and moisture measurements are, therefore, necessary to ensure compliance with safety and environmental requirements, provide stability and maintain the integrity of these tanks.

II. EXPERIMENTAL

A. Instrumentation

A dual-bench FTIR spectrometry system (FTS 60A, Bio-Rad Analytical Laboratory, Cambridge, MA) equipped with a quartz beamsplitter and mercury-cadmium-telluride (HgCdTe) and indium antimonide (InSb) detectors was used to record all NIR spectra. To provide a faster, cheaper, and safer technique to monitor the moisture content of tank waste, two types of NIR diffuse reflectance fiber optic probes were interfaced to the FTIR spectrometry system. These were (a) a Westinghouse Savannah River Company (WSRC, Aiken, SC) fiber optic probe and (b) an AXIOM™ probe (AXIOM Analytical Inc., Irvine, CA). The fiber optic probes served as light conduits to detect analytes via optical changes of properties such as intensity or wavelength.

The WSRC probe is a six-around-one bifurcated probe. Its configuration and operation are described in detail elsewhere[1]. Briefly, the probe has a common leg and is bifurcated into two legs joined together in a zip cord configuration. The common leg has a center fiber (detector fiber) surrounded by six other fibers (light source fibers). Each fiber has a 400-μm diameter, very low-OH silica core, a 440-μm diameter doped silica cladding, a 470-μm polyamide buffer, and a 700-μm polymer jacket. The polymer jacket was removed by immersion in acetone and stripped off using a wire stripper. The buffers of the common leg were molded together using an epoxy resin to form a bundle. The terminal at the common end was beveled toward the center fiber and then bonded to a 20-mil sapphire window.

The schematic design of the AXIOM probe is depicted in Figure 1. The probe consists of a collimator, metallic light guide, focusing optics, and a sapphire window. A customized standbar/mount is installed at the terminal of the probe to allow reproducible positioning and to avoid altering the probe's field of view. A separate bifurcated fiber optic cable (Fiberguide Industries, Stirling, NJ) is attached to the probe terminal. The common leg of the fiber optic cable contains 80 very low-OH fused silica fibers that were then divided randomly to form two legs, each leg consisting of 40 fibers (i.e., 40 fibers to the infrared source and 40 fibers to the detector assembly). Each fiber has a very low-OH pure silica core (OD = 200 μm), fluorine-doped silica clad (OD = 220 μm), and a polyamide jacket (OD = 245 μm). The numerical aperture of each fiber is 0.26. The optical diameter of the complete bundle is 2.3 mm and that of each of the bifurcated legs is 1.6 mm. The fiber cables use standard SMA connectors. These connectors provide a "click-fit" for sampling reproducibility and for fast and easy connection between the fiber bundle and the AXIOM probe.

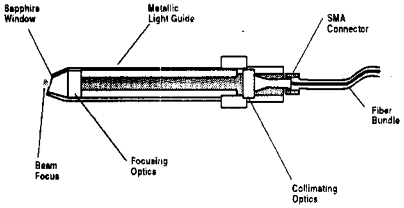


Figure 1. Fiber Optic Diffuse Reflectance Probe

The complete FTIR-fiber optic system consists of the FTIR spectrometer and associated peripherals (Figure 2), a fiber optic accessory for mounting and positioning the fiber optic cable and the NIR detector and the fiber optic probe.

Figure 2. FTIR Spectrometry System

B. Calibration of Fiber Optic System

The calibration of the fiber optic probes was performed using simulated nonradioactive waste mimicking the actual composition of waste tank waste.[4] Simulants with water contents ranging from 5 wt% to 50 wt% were prepared by adding known amounts of water to weighed portions of the dried simulants. Grinding the mixture with an agate mortar and pestle produced slurries/pastes that appeared homogeneous. Water contents of the prepared simulants were verified from the weight loss of the simulants after oven drying overnight at 110 °C.

Near-infrared spectra of simulated waste (T-Plant, U-Plant, BY-104, SY-101, and In-Farm), real mixed waste, and known components of these wastes were collected from 10,000 cm^{-1} (1000 nm) to 4000 cm^{-1} (2500 nm) with 256 scans at a resolution of 8 cm^{-1}. Mixed waste spectra were gathered using a laboratory hood and a hot cell. A single-beam spectrum of KBr was used as background.

Calibration involved the use of regression mathematics to fit the water values of the simulant training sets to their NIR spectra. Spectra from 7500 cm^{-1} to 4500 cm^{-1} containing the first overtone (6900 cm^{-1}) and combination (5150 cm^{-1}) bands of water were used for the spectral analysis. A commercial partial least squares (PLS) package fully integrated with the FTIR system was utilized to calculate water values.

C. Modular Transfer Optic System

Fiber optic probes are potentially the most attractive means to communicate between the FTIR spectrometer and the sample. However, available MIR fibers exhibit fundamental absorption in the fingerprint region and are not resistant to high gamma radiation fields. Nevertheless, MIR spectra offer attractive benefits in the form of distinct and meaningful bands, robust and straightforward calibration, proven diagnostic capability, and insensitivity to spectral artifacts.

In seeking a strategy to identify individual species in mixed waste with minimal sample preparation, a modular transfer optic system equipped with mid-infrared (MIR) diffuse reflectance accessory was assessed. Communication between the FTIR spectrometer (Figure 3a) and the diffuse reflectance accessory (Figure 3b) is accomplished by using a series of hollow metallic light pipes (AXIOM Analytical, Irvine, CA). The light guide approach is easier to implement and more economical. It is also completely adjustment-free. The detector's optical geometry uses a simple lens to focus the infrared radiation emerging from the light guide onto the infrared detector (liquid nitrogen-cooled MCT). The focusing element is a 90° off-axis reflecting paraboloid.

Figure 3a.

Figure 3b.

The diffuse reflectance accessory (DR) shown in Figure 3b consists of a pair of off-axis parabolic reflectors (paraboloids). Placing the paraboloids at right angles with each other compensates for any possible aberration that occurs for off-axis radiation.

Samples were placed in small stainless pans and the infrared beam was then focused at the center of the sample with a spot size of 1 mm and a half-aperture angle of 7.5°. In all cases, the samples were examined neat with no particle size reduction. Spectral measurements were made from 5000 cm^{-1} to 600 cm^{-1} at a resolution of 8 cm^{-1} using an MCT detector. The sample spectra were ratioed against a KBr spectrum (diffuse reflection) or aluminum mirror (specular reflection) collected under the same condition as those of the samples and transformed into diffuse absorbance format. It should be emphasized that this was just an initial exploratory evaluation to determine the modular transfer system's usefulness to collect the MIR spectra of high-level mixed waste. Diffuse reflectance spectra are sensitive to particle size distribution, water content, and sample packing.

III. RESULTS AND DISCUSSION

A. Spectra of Individual Components of Waste

The NIR spectra of some inorganic components of the waste (simulated or real) collected by the AXIOM fiber optic probe assembly are displayed in Figures 4a and 4b. Except for disodiummononickel ferrocyanide [$Na_2NiFe(CN)_6$], all chemicals were analytical or spectroscopy grade and used "as received". The $Na_2NiFe(CN)_6$ was prepared in the laboratory and dried prior to use. All sample spectra were ratioed to the spectrum of KBr and transformed to diffuse absorbance plots.

The FTIR spectrometer interfaced to sensitive detectors such as MCT and InSb made possible the use of low-energy throughput diffuse reflectance spectroscopy to record the NIR spectra of the individual components of the waste. Slight absorption is discerned in the region from 5500 cm^{-1} to 4500 cm^{-1} for sodium salts. The spectra indicate that $NaNO_3$, $CsNO_3$, $NaNO_2$, Na_2CO_3 (Figure 4a), NaCl, and NaF (Figure 4b) are poor absorbers (absorbance <0.05) in the NIR region. Sodium chloride (NaCl), for example, is an infrared-transparent material having no known absorption band in the NIR and MIR regions. However, in the solid state, the infrared radiation is transmitted from one particle to the other by total reflectance at contact between particles.

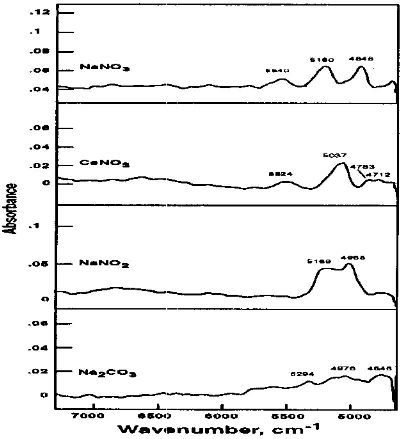


Figure 4a. NIR Reference Spectra

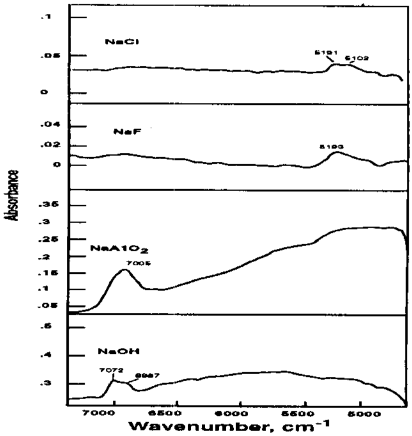


Figure 4b. NIR Reference Spectra

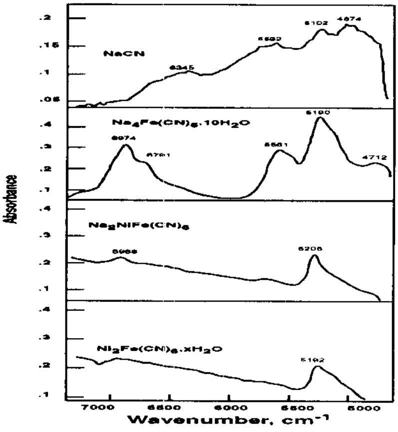


Figure 5a. NIR Reference Spectra

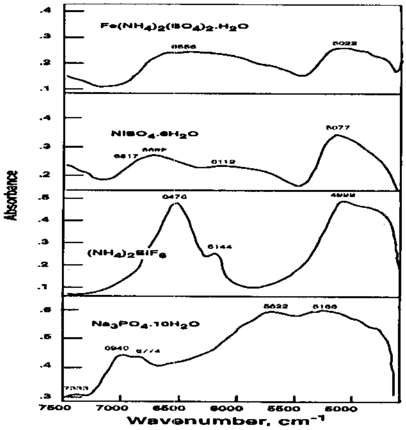


Figure 5b. NIR Reference Spectra

It is believed that the lateral diffusion may be responsible for some energy loss which appears as an absorption in the NaCl spectrum as well as those of the other sodium salts. When these salts are dissolved in water, they are capable of modifying the NIR spectrum of water in a characteristic and measurable way.

Cursory examination of these spectra indicates that the most visually accessible information region for the other components of the waste occurs from 7500 cm^{-1} to 4000 cm^{-1}. A substantial part of the signal in this region is not a result of absorption but of scattering. The tilting of baselines at either higher or lower frequencies is caused by scattering.

Sodium aluminate (NaAlO$_2$) and NaOH (Figure 4b) exhibit weak absorption bands in the region where the first overtone of the OH stretching vibration occurs. On the other hand, NaCN has multiple weak bands covering the entire water absorption range (6345 cm^{-1}, 5532 cm^{-1}, 5102 cm^{-1}, and 4874 cm^{-1}).

Two broad bands from the first overtone of the OH stretching vibration at 6900 cm^{-1} and the combination of the OH stretching and OH bending vibrations at 5150 cm^{-1} are the dominant features of hydrates [Figure 5a: $Na_4Fe(CN)_6 \cdot 10H_2O$, $Ni_2Fe(CN)_6 \cdot xH_2O$ and Figure 5b: $Fe(NH_4)_2(SO_4)_2 \cdot H_2O$, $NiSO_4 \cdot 6H_2O$, and $Na_3PO_4 \cdot 10H_2O$]. These bands tend to be weak and broadly overlapping making it impossible to single out other bands in the sample spectra. This necessitates the use of fairly sophisticated software to correlate the observed spectra with the concentration of the other species in the sample.

Absorption in the NIR region can be used for the identification of organic compounds through the CH, OH, and NH overtone vibrations. Sensitivity can greatly be improved by separating the organic compounds from water which absorbs very intensely in this region. Figure 6 displays the NIR spectra of tributyl phosphate (TBP), n-paraffin hydrocarbon (NPH) and ethylenediaminetetraacetic acid tetrasodium salt trihydrate ($Na_4EDTA \cdot 3H_2O$). The first overtones of the CH_3- and CH_2- stretching vibrations are strongest at 5900 cm^{-1} and 5700 cm^{-1} with weaker combination bands at 7200 cm^{-1}.

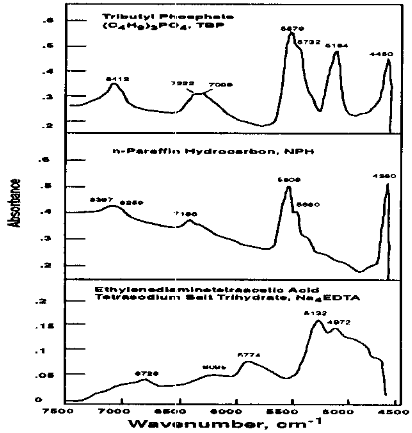


Figure 6. NIR Reference Spectra for Three Organic Compounds

Their second overtones appear at 8400 cm^{-1}. The band at 6726 cm^{-1} may be caused by the first overtone of the NH stretching vibration.

B. Quantitative Determination of Moisture

The most pressing concern in performing quantitative spectral measurements using the WSRC probe is the difficulty of obtaining reproducible data because of the small field of view (about 1 mm compared to 2.3 mm for the AXIOM probe) and inhomogeneous sample surfaces. Reflectance geometries proved to be prone to noise from surface inhomogeneities. Subtle spectral variations occurred on sets of four (4) replicate measurements taken from different locations in the same sample for each of the simulant examined. These variations included the appearance of weak interference fringes superimposed on the sample spectra. From quantitation standpoint, these fringes increase noise of the spectral data, thus degrading the performance prediction of moisture using the PLS software. For this reason, the AXIOM fiber optic probe, which is more insensitive to spectral variations, was the fiber probe of choice for the quantitative measurement of water in mixed waste. All moisture determinations involving high-level mixed waste were conducted in a hot cell.

Lower absorptivities in the NIR region enabled longer pathlengths to be used leading to easier nondestructive sampling. The full sample spectrum from 7500 cm^{-1} to 4500 cm^{-1} was utilized for the calibration and prediction of moisture contents. Both overtone and combination bands of water were used to monitor water contents of the samples.

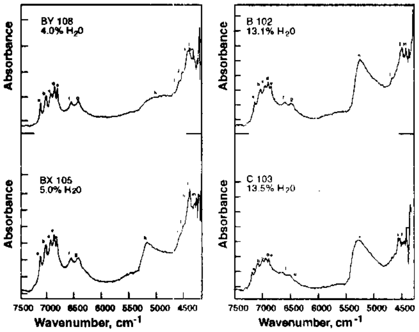
The results of the PLS validation of the AXIOM fiber optic probe using real high-level radioactive mixed waste are summarized in Table 1. The calibration model was

Table I. Standard Error of Prediction Results

Smoothed, Mean-Centered 0-35 wt% Simulant Data Set Using 10-Factor PLS Model			Raw, Mean-Centered 0-35 wt% Simulant Data Set With 7500cm^{-1} Shifted to 0.0 Using a 9-Factor PLS Model		
PREDICTED	ACTUAL	ERROR	PREDICTED	ACTUAL	ERROR
18.09	16.3	1.79	30.37	16.3	14.07
15.78	16.3	-0.52	26.98	16.3	10.68
16.21	16.3	-0.09	24.85	16.3	8.55
32.24	26.3	5.94	41.12	26.3	14.82
30.66	26.3	4.36	42.22	26.3	15.92
31.69	26.3	5.39	42.87	26.3	16.57
17.19	18	-0.81	27.67	18	9.67
17.74	18	-0.26	27.91	18	9.91
14.85	18	-3.15	25.34	18	7.34
26.7	13.3	13.4	41.34	13.3	28.04
30.66	13.3	17.36	39.6	13.3	26.3
30.44	13.3	17.14	36.49	13.3	23.19
11.47	25.8	-14.33	23.73	25.8	-2.07
21.98	25.8	-3.82	29.19	25.8	3.39
25.35	25.8	-0.45	32.14	25.8	6.34
Standard Prediction Error		8.35	Standard Prediction Error		8.34

tested using raw and smoothed mixed waste spectra. The standard error of prediction (SEP) is 8.35. Table 1 indicates that no advantage is gained by smoothing the spectra. Perhaps the addition of more water standards to the calibration set will improve prediction of water making the PLS model applicable to all Hanford mixed waste. Work is continuing to improve the performance of the method.

Typical spectra of mixed waste with low moisture contents are displayed in Figure 7a. Semi-dried waste are characterized by the presence of small bands in the 7000 cm^{-1} region (first overtone of the OH stretch). Partial dissolution of the waste results in changes in peak shapes, notably the disappearance of these small bands in the 7000 cm^{-1} region as depicted in the spectra of wet samples (Figure 7b). It is evident that the small bands are caused by combined water, possibly, hydrates. Broadening of the band is indicative of more free water in the sample. However, the dominant features of all of these spectra are the overtone and combination bands of water.



a. 7149	e. 6843	j. 4520
b. 7051	f. 6586	k. 4415
c. 6957	g. 6482	l. 4404
d. 6895	h. 5250	m. 4341
	i. 4570	

Figure 7a. Spectra of Tank Waste with Low Moisture Content

C. MIR Characterization

This phase of the study deals primarily with qualitative analysis and species identification. Diffuse reflectance spectroscopy monitors the characteristics of the bulk sample and is affected by thickness variation and particle size. In general, diffuse reflectance is only usable for quantitative measurement if the sample is ground up and mixed with a transparent scattering matrix (KBr).

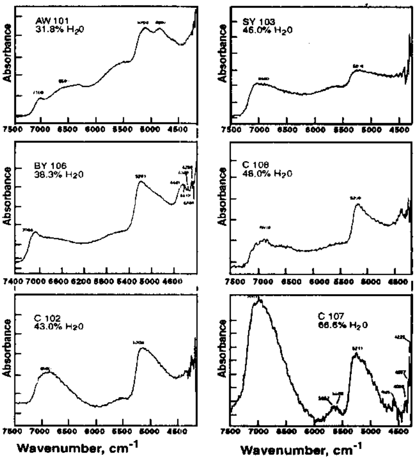


Figure 7b. Spectra of Tank Waste with High Moisture Content

Additionally, interference fringes (optical interference between light reflected from the front and back surfaces of the sample) resulting from the use of neat samples cannot simply be subtracted from the sample spectrum because they do not depend monotonically on thickness.

Figure 8a shows the diffuse absorbance spectra of two oven-dried simulants, BY-104 and SY-101 obtained by modular transfer optics. These spectra are those of neat samples and are compared with the photoacoustic spectra of aliquots (also neat) of the same simulants but were freeze-dried at -50 °C for 6 hours (Figure 8b). The diffuse reflectance spectra have more distinct bands for chemical structure elucidation than the photoacoustic spectra. However, neat diffuse reflectance spectra are susceptible to distortion near strong absorption bands because of the large changes in the reflectivity (refractive index). These reflectivity changes result in a large specular component that acts in opposition to the diffuse component. With the AXIOM diffuse reflectance unit of the modular transfer optic system, the specular component may be eliminated as described in the experimental section. This capability of the AXIOM diffuse reflectance unit needs to be addressed in future work on characterization of organics in mixed waste. The photoacoustic spectra in Figure 8b demonstrate the difficulty of interpreting and quantifying saturated photoacoustic bands. For example, the nitrate content cannot be monitored quantitatively via the nitrate band at 1363 cm^{-1}. Like diffuse reflectance spectroscopy, photoacoustic spectroscopy depends on weak bands for quantitation of species of interest. The photoacoustic technique may be an alternative approach to quantitation but this technique cannot be utilized without prior removal of water.

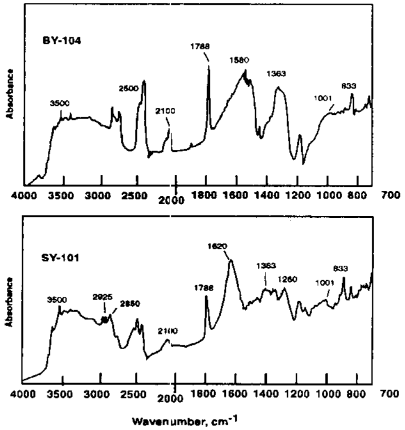


Figure 8a. Simulants (Modular Transfer Optics Method)

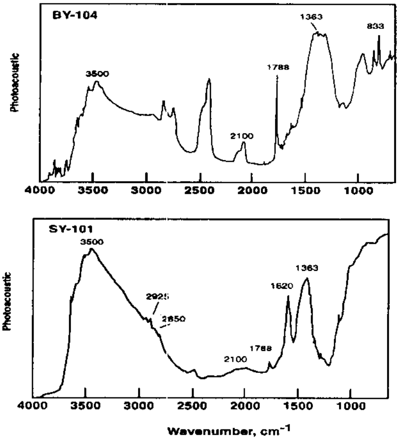


Figure 8b. MIR Spectra of BY-104 and SY-101 Waste Simulants (Photoacoustic Method)

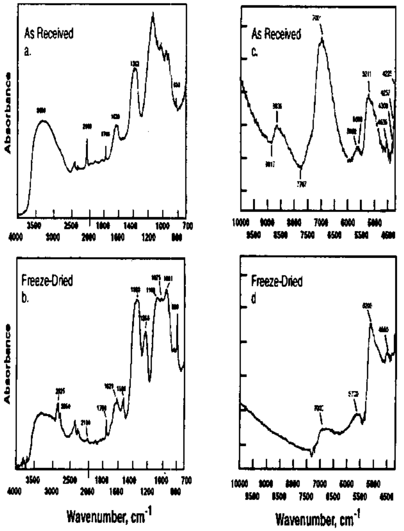


Figure 9. Spectra of Tank C-107 Waste
a. MIR Spectrum (As Received)
b. MIR Spectrum (Freeze Dried)
c. NIR Spectrum (As Received)
d. NIR Spectrum (Freeze Dried)

Application of the MIR modular transfer optic technique to characterize high-level radioactive mixed waste (tank C-107) is demonstrated in Figure 9. It appears that this technique can be used to measure moisture content in mixed waste remotely. The spectral region from 3700 cm^{-1} to 1500 cm^{-1} may be utilized for PLS spectral treatment. This region contains the OH stretching (3400 cm^{-1}) and bending (1620 cm^{-1}) vibration bands of water. The reliability of this technique to predict water contents can be compared with its PLS NIR counterpart described earlier.

A comparison of the MIR spectrum of the freeze-dried C-107 sample with its NIR spectrum reveals that only water can be determined in the NIR region. In contrast, organics, nitrates, and nitrites can be quantified in the MIR region making the MIR modular transfer optic system more suitable for measuring the concentration of fuels and oxidizers in the waste.

IV. CONCLUSIONS

The results of the tests confirm the usefulness of simulants to predict and model moisture concentrations of high-level mixed waste using partial least squares multivariate analysis. The complexity of the calibration is considerable but the extreme simplicity of its subsequent use for testing unknown moisture concentrations in mixed waste fully justifies the time spent for calibrating the fiber optic probes. The observed standard error of predicting moisture values is high and needs improvement. Use of mixed waste from additional waste tanks, to reflect the uniqueness of each tank waste in establishing quantitative changes in sample spectra, is recommended.

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