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Effect of Atmospheric Humidity on CPAC Optical Moisture Prediction Models - Interim Report

Near-Infrared; Optical Absorption; Moisture Sensing; Hanford Waste Tank Material; Tank Head-Space Interference; Atmospheric Absorption.

The tank head space water vapor will be a strong interference in the implementation of the NIR system as a non-contact camera sensor. The impact of water vapor can be compensated for by explicitly including the water vapor spectral response in the NIR spectra used to generate the PLS (partial least squares) calibration model. PLS models constructed with NIR spectra where water vapor absorption was included as an interferant (from a LOWTRAN computer atmospheric absorption model) predicted moisture from NIR spectra with RMSE (root mean square error) fit errors in the range of 0.729-0.743 wt% moisture. This error correlates well with tank needs and earlier prediction test data where the water vapor was ignored. The models worked equally well with or without the water vapor interferant present in the sensed NIR spectra.

Water vapor can also be compensated for if the humidity of the tank head space is measured and the path length between NIR system and waste surface is measured. A simulation using water vapor data from a LOWTRAN atmospheric model, showed wt% moisture values.

The conclusion is that water vapor is a strong interferant in waste applications of NIR moisture sensing, however, it can be compensated for with only a small impact on the sensing models and their prediction accuracy.
EFFECT OF ATMOSPHERIC HUMIDITY
ON CPAC OPTICAL MOISTURE
PREDICTION MODELS
INTERIM REPORT

October 1994
ABSTRACT

The tank head-space water vapor will be a strong interference in the implementation of the near-infrared system as a noncontact camera sensor. The impact of water vapor can be compensated for by explicitly including the water-vapor spectral response in the spectra used to generate the partial least squares calibration model. Partial least squares models constructed with near-infrared spectra in which the interfering water-vapor absorption had been included (from a LOWTRAN atmospheric-absorption model) predicted moisture from near-infrared spectra with root-mean-square error fit errors in the range of 0.729-0.743-wt% moisture. This correlates well with tank needs and earlier prediction test data in which the water vapor was not included. The partial least squares calibration models worked equally well with or without the water-vapor interference present in the sensed near-infrared spectra. The atmospheric water vapor can also be compensated for by measuring the humidity of the tank head space and the path length between the near-infrared system and the waste surface. A simulation using water-vapor data from a LOWTRAN atmospheric model showed that adding water-vapor absorption changed only the offset of the predicted weight-percent moisture values. The conclusion is that water vapor is a strong interference in waste applications of near-infrared moisture sensing. However, it can be compensated for with only a small impact on the sensing models and their prediction accuracy.
EXECUTIVE SUMMARY

The interim results of modeling and experimental testing to show the impact of atmospheric moisture on the measurement of water content from near-infrared absorption of waste-simulant material are presented in this report. In previous 2A and 2B Tasks, the University of Washington's Center For Process Analytical Chemistry has shown that the near-infrared backscatter spectra from waste simulants could be used to determine moisture by remote sensing of tank waste materials. This earlier work had indicated that atmospheric moisture in tank head space might present a major interference to the spectroscopic moisture determination.

This report contains the results of work to determine the impact of tank head-space atmospheric moisture on the measurement of waste moisture content from near-infrared backscatter spectra. Atmospheric-absorption simulation tests were completed using a computer-based atmospheric model called LOWTRAN and experimental measurements made with a chamber containing known amounts of water vapor. The results show strong interference from head-space moisture. However, the simulation studies also show that correcting for the water-vapor absorption should be possible with no loss in moisture-detection sensitivity.

Two specific approaches were identified and investigated:

1. The impact of water vapor can be compensated for by measuring the humidity of the tank head space and the path length between the near-infrared system and the waste surface. It was shown, using

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simulated water-vapor data from the LOWTRAN computer model, that adding the water-vapor absorption to the reflectance spectra changed only the offset of the predicted weight-percent moisture values. This offset error is related to both the humidity and path length of the water-vapor spectral interference. Therefore, for this compensation method, both relative humidity and path length must be known in order to estimate the correction factor.

2. The impact of water vapor can be compensated for by explicitly including the water-vapor spectral response in the near-infrared spectra used to generate the partial-least-squares calibration model. The advantage of this approach, which appears to be more robust, is that the actual humidity and path length within the tank at the time of measurement need not be known. However, calibration spectra with the expected spans of humidity and path-length variations likely to be encountered during use in a tank are needed in order to build the partial-least-squares calibration model.

The partial-least-squares models generated from near-infrared backscatter spectra worked as well with or without the interfering water-vapor spectral impacts. Root-mean-square error fit errors in the range of 0.729-0.743-wt% moisture were obtained from the modeling tests. Including the water-vapor spectral interference in the model does not increase the prediction error when the interference is not present. The model appeared to handle path-length changes as well as moisture-content changes and produce similar root-mean-square error fit errors. This response correlates very well with earlier test data in which water vapor was ignored. The earlier work by the Center For Process Analytical Chemistry produced root-mean-square error fit errors in the 0.5-wt% range with spectra that had no compensation for atmospheric water absorption.
Inherent in both simulation approaches is the assumption that the change in the water-vapor spectra will be linearly additive with the measured sample spectra (i.e., there are no interactions between changes in the water-vapor spectra because of humidity and path-length differences and the sample spectra because of bulk-water concentration). This will be verified with additional experimental testing.
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1.0 INTRODUCTION

This interim report describes the current status of the revised Task 3D contract work being completed for the Westinghouse Hanford Company by the University of Washington's Center for Process Analytical Chemistry (CPAC). In June 1994 the scope of this work was modified at Westinghouse Hanford Company's request to focus on determining the effect of atmospheric moisture/humidity on the predictions of bulk waste-tank moisture using spectroscopic measurements and CPAC calibration models. Previously the Task 3D work scope was to investigate only the issues related to scaleup of the CPAC moisture-monitoring methods for in situ remote sensing in the waste tanks. Earlier work indicated that atmospheric moisture might present major interference to the spectroscopic moisture determination. It was determined that this should be investigated before further development of full-scale remote sensing. This report describes the interim status of efforts currently underway at CPAC to address these issues.

There are two components to this work scope.

1. The first, and major, task is to simulate the effect of atmospheric moisture by combining near-infrared (NIR) spectra obtained from previous CPAC work with atmospheric-absorption spectra for water vapor from a computer-simulated atmospheric-absorption model called LOWTRAN (ONTAR 1990). The main goal of this task is to assess the impact of atmospheric moisture absorption on the performance of moisture-prediction models and to suggest possible methodologies whereby these effects could be minimized.

2. The second task is to generate actual spectral data from an experimental setup that has both waste simulants and an atmospheric path length. The main goal of this effort is to assess the viability of combining the atmospheric simulation model spectra with the simulated waste spectra and to see how well these convolved simulated spectra match the experimentally measured spectra.

2.0 EXPERIMENTAL SETUP

In this section we first describe the approach used to generate the simulated spectra containing both the measured spectral response to bulk-moisture variation in the waste simulants and the water-vapor spectra obtained from the LOWTRAN atmospheric-absorption computer simulation. The experimental efforts underway to generate the actual spectral responses needed to validate the simulated results are then discussed.
2.1 SIMULATIONS

This work is based on the data sets described in the Task 2A report previously transmitted to the Westinghouse Hanford Company (WHC 1993). These spectral data sets contain BY-104 simulant NIR spectra measured from 1100 nm to 2500 nm using a fiber-optic sampling probe and a commercially available NIR spectrometer. A number of spectra were obtained at different moisture levels and simulant compositions. In this current work, the water-vapor NIR spectra for two different atmospheric humidity levels (80% and 99% relative humidity [RH]) and twelve different optical path lengths (from zero to 60 in) were used to test the sensitivity to atmospheric absorption. The LOWTRAN spectra are presented as transmission spectra and extrapolation between wavelength points (from 248 to 700 wavelengths) was required in order to number the wavelength points for the CPAC-measured spectra. This was done by first converting the LOWTRAN spectra to absorbance by taking the negative base-10 logarithm of the transmission spectra. The LOWTRAN absorbance spectra were then added to the reflectance spectra (reported in absorbance units) that were measured by CPAC in the prior Task 2A work (WHC 1993).

Inherent in this approach are several assumptions. First, we need to assume that changes in the atmospheric humidity level will not affect the bulk-moisture spectra. If this assumption holds, then it should be valid to simply add the absorption spectrum from the atmosphere to the absorption spectrum from the waste material for which we wish to obtain moisture content. We should be able to test this assumption experimentally by measuring the bulk spectra under different humidity conditions using very short atmospheric path lengths. Second, we will assume that the relative intensities of the absorbance bands in the two spectral sources are close to those that would be measured. This allows us to avoid the complication of additional weighting, or scaling, of the two spectral sources. We should be able to verify this from the planned experimental program. Finally, this approach should provide relevant information, provided that the in-tank relative humidity is known or can be measured and that the effective atmospheric path length is known or can be accurately determined during the spectral measurement.

2.2 EXPERIMENTAL MEASUREMENT SETUP

In order to collect the experimental data needed to verify the spectral addition simulations, we have to be able to measure the spectral response to both the bulk-moisture variation and the atmospheric water-vapor variation at the same time. The first experimental limitation is the relatively weak NIR absorption of water in the vapor phase compared with NIR absorption from the condensed phase. In order to model the dimensions of a Hanford waste-tank head-space region, relatively long spectral path lengths are needed. In our initial experiments we simply positioned the fiber-optic probe in our existing spectrometer setup a short distance (zero to 4.5 in.) from the sample surface and measured the spectra under different humidity conditions. Because of the diffuse pattern of light exiting from the fiber-optic probe and the diffuse reflectance from the tank simulants, the optical signals were very small. We found that we could not achieve long atmospheric path lengths and still obtain reasonable signal-to-noise in the bulk spectral response even with high signal averaging with this setup. This is a limitation of the fiber-probe transmission/collection efficiency and of the intensity of the spectrometer.
light source. With short path lengths, the spectral sensitivity to humidity changes was very low.

We then completed the construction of an optical chamber, shown in Figure 1, to increase the effective atmospheric path length and improve the light-collection efficiency. The chamber uses a large (approximately 7-in.-diameter) convex mirror to prevent the diffusion of light and provides an effective open path length of about 40 in. The fiber-optic probe is inserted at one end of the chamber, approximately 2 in. behind the focal point of the mirror. Humidity within the chamber is controlled by placing either a saturated NaCl solution (75% RH) or a humidity source (hot water) in a container in the bottom of the chamber and then bleeding-in dry air to obtain the desired humidity. The humidity within the chamber was monitored using a commercial relative-humidity probe.

Using this setup, we were able to obtain water-vapor spectra that closely matched the spectra estimated by the LOWTRAN atmospheric-absorption computer-simulation program (ONTAR 1990). Future work will include coating the mirror with a thin film of BY-104 simulant to obtain the bulk-moisture response and also to verify that air humidity does not affect the simulated waste spectral features.

3.0 RESULTS AND DISCUSSION

3.1 CONTROL MODEL TESTED WITH CONTROL+LOWTRAN SPECTRA

To assess the effect of atmospheric-humidity absorption on the CPAC moisture prediction models, we used the model previously derived from the CONTROL data set (described fully in the Task 2A report [WHC 1993]). This data set contained the NIR spectra from three replicate experiments in which the moisture content of the BY-104 simulant was varied from 0 to 25 wt% in 5-wt% increments. Two spectra were recorded per moisture level and the resulting data set consisted of 36 spectra. The partial-least-squares (PLS) two-factor model for this data produced a root-mean-square error (RMSE) of 0.76 wt% in the prediction of the sample's moisture. The LOWTRAN absorbance spectra corresponding to an atmosphere of 99% RH with 60-m and 3-m path lengths were then combined with each spectrum in the BY-104 CONTROL data set. The PLS model that was derived from the CONTROL data without atmospheric absorption was then used to predict moisture for these combined CONTROL+LOWTRAN spectra. The results are shown in Figure 2.

Figure 2 shows that the addition of the water-vapor absorption produces a constant offset in the predicted moisture values. The magnitude of the offset is related to the intensity of the atmospheric-moisture absorption (i.e., the simulated path length/relative humidity). The good news is that the water-vapor absorption does not seem to affect the slope of the prediction regression line. This implies that the PLS model is able to use the spectral information related to the bulk moisture even in the presence of the water-vapor spectral features. The reason for the increasingly negative bias in the prediction errors with increasing water-vapor interference is the second-derivative treatment of the spectra prior to developing the calibration model.
Figure 1. Experimental Apparatus for Open Path Humidity Experiments.
Figure 2. Bulk-sample Moisture Predicted from the CONTROL+LOWTRAN Spectra (99% Relative Humidity, 3-m and 60-m Path Lengths) Using the CONTROL Model.
or applying the model to predict moisture from the spectra. Figure 3 shows the measured spectrum of one of the 25-wt% moisture CONTROL samples along with the sample spectrum plus the LOWTRAN spectrum for 99% RH and a 60-m path length. As one can see from the plot, the water-vapor spectral features overlap with the leading edge of the bulk-water absorption bands at 1425 nm (the OH first overtone) and at 1925 nm (the OH combination band).

Figure 4 shows the second derivative of the Figure 3 spectra. Because of the partially overlapped nature of the water-vapor and bulk-water peak positions, the rising edge inflection point for the first water-vapor band (at approximately 1350 nm) occurs very close to the start of the bulk-water band at 1425 nm. The result is that, in the derivative spectra, the CONTROL spectra (i.e., spectra with no atmospheric impacts) has a positive peak between 1350 and 1400 nm while the CONTROL+LOWTRAN spectra (i.e., spectra with atmospheric impacts) has a negative peak in the same region. The same situation appears for the leading edge of the 1925 nm spectral band. The PLS model built from the CONTROL data is based on the presence of this positive peak in the derivative spectra at approximately 1375 nm and 1850 nm, but for spectra with the water-vapor absorption (CONTROL+LOWTRAN spectra) these regions have negative peaks.

If we develop a model on the unprocessed CONTROL data (i.e., no derivative) and use it to predict moisture from the raw CONTROL+LOWTRAN spectra, a positive bias results as shown in Figure 5.

3.1.1 Relative Humidity and Path-length Compensation

In order to quantify the effect of the water-vapor interference on the PLS model performance, we repeated the procedure with LOWTRAN spectra derived with a different atmospheric path length. Changing the path length is essentially equivalent to changing the water-vapor concentration of a fixed path length. Optical absorption is a function of density (or concentration) and the path length traversed. We varied the path length from zero (no water-vapor absorption) to 60 m (the maximum path length expected in a waste tank) and used the CONTROL PLS to predict weight-percent moisture from these CONTROL+LOWTRAN spectra. The results are shown in Table 1. Again, one can see that increasing the effective water-vapor spectral interference by increasing the open path length results in increased prediction errors and increasing offset (y-axis intercept) but does not affect the slope (sensitivity) of the regression-model predictions.

Therefore, if one can correct for the offset (the y-intercept), it should be possible to obtain the bulk weight-percent moisture in the presence of the water-vapor spectral interference. Figure 6 shows the y-axis intercept plotted against the LOWTRAN spectra path length (open circles in plot). Results are shown for both the actual values and the intercepts estimated from the second-order polynomial fit. The relationship between the prediction offset and the vapor-phase water-spectrum path length is mostly linear at the longer path lengths with slightly nonlinear deviation at the shorter path lengths. When a second-order polynomial is fit to the y-intercept versus path-length curve, the y-intercept values estimated from the polynomial fit (shown as the "x"s in Figure 3) agree very well with the actual y-intercept values.
CONTROL Spectrum and CONTROL+LOWTRAN Spectrum

- 25 Wt% CONTROL
- Plus LOWTRAN

Figure 3. Measured CONTROL Spectra for 25-wt% Moisture Sample and Sample with LOWTRAN (99% Relative Humidity, 60°m Path) Spectra Added.
If we develop a model on the raw CONTROL data (i.e., no derivative) and use it to predict moisture from the raw CONTROL+LOWTRAN spectra, a positive bias results, as shown in Figure 5.
Figure 5. Actual Versus Predicted Bulk Weight-percent Moisture from the Raw (i.e., No Derivative) CONTROL+LOWTRAN Spectra Using a Model Derived from the Raw CONTROL Spectra.
Table 1. Prediction Statistics for the Prediction of Bulk Moisture from the CONTROL Spectra Plus LOWTRAN Spectra at 99% Relative Humidity and Different Path Lengths Using the BY-104 CONTROL Model.

<table>
<thead>
<tr>
<th>Path length*</th>
<th>RMSE</th>
<th>Intercept</th>
<th>Slope</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.74</td>
<td>0.089</td>
<td>0.993</td>
</tr>
<tr>
<td>1</td>
<td>2.30</td>
<td>-2.09</td>
<td>0.993</td>
</tr>
<tr>
<td>3</td>
<td>4.12</td>
<td>-3.96</td>
<td>0.993</td>
</tr>
<tr>
<td>7</td>
<td>6.63</td>
<td>-6.50</td>
<td>0.993</td>
</tr>
<tr>
<td>12</td>
<td>9.05</td>
<td>-8.93</td>
<td>0.993</td>
</tr>
<tr>
<td>20</td>
<td>12.22</td>
<td>-12.11</td>
<td>0.993</td>
</tr>
<tr>
<td>27</td>
<td>14.63</td>
<td>-14.52</td>
<td>0.993</td>
</tr>
<tr>
<td>33</td>
<td>16.51</td>
<td>-16.41</td>
<td>0.993</td>
</tr>
<tr>
<td>40</td>
<td>18.57</td>
<td>-18.47</td>
<td>0.993</td>
</tr>
<tr>
<td>47</td>
<td>20.52</td>
<td>-20.42</td>
<td>0.993</td>
</tr>
<tr>
<td>53</td>
<td>22.11</td>
<td>-22.01</td>
<td>0.993</td>
</tr>
<tr>
<td>60</td>
<td>23.90</td>
<td>-23.80</td>
<td>0.993</td>
</tr>
</tbody>
</table>

* Path length in meters.
Figure 6: Plot of Bulk Weight-percent Moisture Regression Y-axis Intercept Versus the LOWTRAN Open Path Length.
The second-order polynomial fit equation is as follows:

\[ y\text{-intercept} = 0.0037 \text{(path length}^2) - 0.5847 \text{(path length)} - 1.6124. \quad (1) \]

The preceding analysis was conducted with the 99% RH LOWTRAN spectra. If we repeat the analysis with the 80% RH LOWTRAN spectra (over the same range of path length), we obtain a different set of y-intercept versus path-length curves, as shown in Figure 7. This shows the results for both the actual values (0 and x) and the second-order polynomial fit (solid and dotted lines) for the 80% RH and 99% RH, respectively. At 80% RH, the second-order polynomial fit between the path length and the y-intercept is given by the following equation:

\[ y\text{-intercept} = 0.0033 \text{(path length}^2) - 0.5099 \text{(path length)} - 1.4169. \quad (2) \]

From this analysis, it appears that if one could measure the tank head-space humidity (using a simple humidity sensor and assuming uniform humidity in the head space) and the open path length between the optical sensor and the sample (using some sort of range finder), it should be possible to correct the bulk-moisture predictions for the water-vapor interference by apply the appropriate correction factors. Again, this analysis is based on the assumption that changes in the humidity will not affect the bulk-moisture spectra (i.e., that the water-vapor absorbance spectra can simply be added to the bulk-sample absorbance spectra, which needs to be verified by additional experiments currently under way).

3.2 CONTROL+LOWTRAN MODEL TESTED WITH CONTROL AND CONTROL+LOWTRAN SPECTRA

An alternative approach to correcting for the vapor-phase moisture spectral interference is to explicitly include the atmospheric absorption as an interference when the PLS calibration model is constructed. In order to test this approach, the LOWTRAN spectra corresponding to 99% RH and path lengths of 3 m and 60 m were added to the CONTROL spectra (BY-104 simulant reflectance with no atmospheric absorption). Cross-validation studies indicated that this new data set (CONTROL+LOWTRAN) required three factors for the best predictive model. This is one more factor than the previous CONTROL model (without atmospheric absorption), indicating that the CONTROL+LOWTRAN model requires an additional factor to describe the atmospheric absorption. The percent variance described by each factor of the CONTROL+LOWTRAN PLS model is shown in Table 2.

In order to aid understanding of how the incorporation of the LOWTRAN water-vapor interference is propagated through the PLS model, the CONTROL+LOWTRAN scores and loadings are shown in Figures 5 through 8. Figure 5 shows the sample score values for the first factor of the CONTROL+LOWTRAN model while Figure 6 shows the corresponding wavelength loading factors. Figures 7 and 8 address the second factor. (To help with the interpretation, the scores are plotted versus the sample weight-percent moisture rather than the sample number.) It is clear that the first factor of this model is mainly describing the change in the spectra caused by the two different LOWTRAN path lengths. The group of samples with positive scores
Figure 7. Plot of Bulk Weight-percent Moisture y-axis Intercept Versus the LOWTRAN Spectra at Different Humidities and Path Lengths.
Table 2. Percent Variance Described by Each Factor of the CONTROL+LOWTRAN (99% RH, 3-m and 60-m Path Lengths) PLS Model.

<table>
<thead>
<tr>
<th>Factor number</th>
<th>X-block (spectra)</th>
<th>Y-block (moisture)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>This factor</td>
<td>Total</td>
</tr>
<tr>
<td>1</td>
<td>64.16</td>
<td>64.16</td>
</tr>
<tr>
<td>2</td>
<td>35.35</td>
<td>99.51</td>
</tr>
<tr>
<td>3</td>
<td>0.37</td>
<td>99.88</td>
</tr>
</tbody>
</table>
Figure 8. First-factor Sample Scores of the CONTROL+LOWTRAN Model Plotted Versus the Sample Bulk Weight-percent Moisture.
corresponds to the 60-m path-length LOWTRAN spectra added to the CONTROL spectra, and the group of samples with negative scores corresponds to the 3-m path-length LOWTRAN spectra added to CONTROL spectra. The small positive trend in the scores within each group indicates that while this factor is mainly describing the water-vapor interference, there is also some information related to the bulk-moisture level spectral response being described.

The loadings for the first factor of the CONTROL+LOWTRAN model are shown as the solid line in Figure 9. For comparison, the first-factor loadings of the CONTROL model are also plotted, shown as the dotted line. It is fairly clear that this first factor is mainly describing information related to the LOWTRAN vapor-phase water spectra and not the bulk-moisture peaks at 1425 nm and 1950 nm described by the first factor of the BY-104 simulant CONTROL model.

The corresponding plots for the second factor of the CONTROL+LOWTRAN model are shown in Figures 10 and 11 (scores and loadings, respectively). In Figure 10, the score values clearly reflect the linear spectral change related to the increased bulk-moisture content of the BY-104 samples, with only a small contribution (seen as the duplicate groups of samples with a small vertical displacement between groups) caused by the two different LOWTRAN spectral path lengths. Note that the main trend is decreasing, rather than increasing, with increasing bulk weight-percent moisture. This is purely a mathematical artifact of the arbitrary orientation (positive or negative) of the PLS model vectors. The loading vector for the second factor is plotted in Figure 11, along with the negative of the first-factor loadings of the CONTROL model. Again, it is entirely arbitrary whether a loading vector is described as going in the positive or negative direction. The only important aspect is that the scores and loading vectors remain consistent with each other. If we reverse the sign (i.e., orientation) of the loading vector, we must also reverse the sign of the score vector. From this plot it is clear that the second factor of the CONTROL+LOWTRAN model is nearly identical to the first factor of the CONTROL model. From this we can conclude that after corrections for the atmospheric water-vapor absorption are accounted for, the remaining information is essentially the same as the CONTROL model, which did not include atmospheric absorption.

Although not shown here, the third factor of the CONTROL+LOWTRAN model is also very similar to the second factor for the CONTROL model, although there is a small component of the water-vapor spectra that is described by this third factor.

3.3 MODEL COMPARISON

In comparing moisture prediction with the two models, using each only with the spectral data with which it was generated, the CONTROL+LOWTRAN model (a model made and tested with data containing atmospheric absorption) exhibited a RMSE fit error of 0.729 wt% moisture, which compares very well with the RMSE fit error of 0.743 wt% for the CONTROL model (no atmospheric absorption in either the model or test data). We then used the PLS model that was generated with atmospheric absorption to predict moisture for spectral data that had no atmospheric absorption. Using the CONTROL+LOWTRAN model to predict the bulk weight-percent moisture from the CONTROL data-set spectra
Figure 9. Wavelength Loading Vectors for the First Factor of the CONTROL+LOWTRAN and CONTROL Models.
Figure 10. Sample Scores for Second Factor of CONTROL+LOWTRAN Model Versus Sample Weight-percent Moisture.
Figure 11. Second-factor Loading of the CONTROL+LOWTRAN Model and First-factor Loading of the CONTROL Model.
resulted in a prediction RMSE of 0.729 wt%. This indicates that including the water-vapor spectral interference in the model does not increase the prediction error when the interference is not present. In addition, when we attempt to predict the bulk weight-percent moisture for spectra modified by a 33-m path length with 99% RH, we obtain a moisture prediction RMSE of 0.730 wt%.

Plots of the actual versus predicted weight-percent moisture for the CONTROL (no atmospheric moisture) spectral data set using the CONTROL+LOWTRAN model (built with data containing atmospheric absorption) are shown in Figure 12. If we add moisture to the spectral data, 33-m path-length LOWTRAN spectra at 99% RH, the actual versus predicted bulk weight-percent moisture for the model made with atmospheric absorption (CONTROL+LOWTRAN model), we obtain the results shown in Figure 13. In both cases, including the atmospheric absorption for different path lengths with a 99% RH in the calibration model allowed the PLS models to correct for the water-vapor interference without an appreciable increase in the overall prediction error. The key is to use spectral data that have moisture content (path length or concentration) as an interference so that the PLS model avoids high dependency on those spectral areas where there is atmospheric absorption.

Looking at the difference in the regression-coefficient vector between the CONTROL model and the CONTROL+LOWTRAN model, as shown in Figure 14, one can see how the addition of the water-vapor interference in the calibration model changes the relative importance of the different spectral regions. Because the water-vapor-phase spectra overlap slightly with the leading edge of the bulk-water absorption bands at 1425 nm (the OH first overtone) and 1950 nm (OH combination bands), the CONTROL+LOWTRAN regression-coefficient vector de-emphasizes these regions relative to the CONTROL model. The overlap is stronger for the 1425 nm OH band than for the 1950 nm band. At 1425 nm the CONTROL+LOWTRAN regression coefficient uses only the portion of the overtone band immediately after the peak maximum and the trailing edge. At 1950 nm the overlap is not so strong between the vapor phase and bulk moisture, and the CONTROL+LOWTRAN regression coefficient uses the area just before and after the peak maximum as well as the trailing edge of the peak. Because the 1950 nm bulk-moisture band is further displaced from the water-vapor absorption bands, this spectral band is emphasized more in the CONTROL+LOWTRAN model than in the CONTROL model.

4.0 EXPERIMENTAL MEASUREMENTS

Using the experimental apparatus shown in Figure 1 we have succeeded in measuring the vapor-phase water spectra on our NIR instrument. Figure 15 shows the spectrum we measured at 92% RH and the spectrum from LOWTRAN corresponding to a path length of 3 m and 99% RH. For this figure, the LOWTRAN spectrum was offset by 0.6 absorbance units and the experimentally measured spectrum was multiplied by 30 to facilitate the visual comparison. If we ignore the high-frequency noise (lower signal-to-noise ratio), in the experimentally measured spectrum, there is excellent agreement with the LOWTRAN model's spectrum. The correlation between the measured spectrum and the actual humidity levels within the test chamber was also fairly good.
Figure 12. Bulk weight-percent Moisture Predicted from the CONTROL Spectra Versus the Actual Weight-percent Moisture Value.
Figure 13. Moisture predicted from Control plus LOMTRAN using the Control+LOMTRAN Model (99% Relative Humidity, 33-m Path Length).
Figure 14. Regression Coefficient Vectors for the CONTROL Model and the CONTROL+LOWTRAN Model.
Figure 15. Experimentally Measured Water-vapor Spectrum and the LOWTRAN-estimated Spectrum.
Using a single-factor PLS model we are able to predict the humidity from the measured spectra with a RMSE error of 3% RH. This is very close to the specified accuracy of the reference humidity sensor/probe we used to obtain the moisture level inside the test chamber. We are currently working to obtain the BY-104 simulant spectra at different humidity levels.

One of the major difficulties in this effort is to obtain the bulk spectra with sufficient signal levels while maintaining the ability to observe the water-vapor spectral features. This is really a limitation of our light-source intensity and the efficiency in the collection of the reflected light. We have had some success with coating the mirror with a very thin film, or dusting, of the BY-104 simulant material. This allows us to use the focusing power of the mirror to help collect the light reflected from the BY-104 material while still giving us sufficient open path length to observe the water-vapor absorption using the light transmitted through the sample film. Quantitation of the amount of moisture in the thin sample film on the mirror's surface remains a problem to be addressed. At this point we are mainly interested in verifying that the surface absorption of water caused by humidity variations does not alter the features of the bulk-water spectra.

5.0 SUMMARY AND CONCLUSIONS

In this interim report, we have described work completed to date on the study of water-vapor interference on the spectroscopic moisture prediction method under development at CPAC. The absorption from atmospheric water vapor is an issue in the remote sensing of moisture with the NIR spectra in a waste tank. For any significant open path, head-space moisture will adversely affect the NIR spectra as an interference. This study had two parts. The first, and main, effort was to investigate the effect of atmospheric water-vapor absorption on the predictive ability of the models developed at CPAC using water-vapor spectra generated from a computerized atmospheric model (LOWTRAN computer program [ONTAR 1990]). The second effort was to verify the assumptions used in combining the measured spectral data with the simulated atmospheric-absorption (LOWTRAN) spectra by making actual spectra measurements in air with different relative humidities.

The results from the simulation study with the LOWTRAN spectra indicate that correcting for the water-vapor absorbance should be possible. Two approaches were investigated. First, if the head-space humidity of the tanks is known, or can be measured, and the open path length between the optical components and the sample can be measured, it should be possible to estimate a simple offset correction to the moisture predictions to account for the influence of water vapor on the model predictions. The simulations indicate that adding the water-vapor absorbance to the measured sample absorbance does not change the sensitivity of the model to bulk moisture but does cause a simple offset, or error, in the magnitude of the predicted weight-percent moisture values. This offset error is related to both the humidity and path length of the water-vapor spectral interference. Therefore, both relative humidity and path length must be known in order to estimate the correction factor.
A second, and perhaps more robust, approach is to explicitly include the water-vapor spectral response as an interference in spectra used to generate a PLS calibration model. The advantage of this approach is that the actual humidity and path length within the tank at the time of measurement need not be known. However, it does require more effort to collect spectra for the calibration that span the expected range of humidity and path-length variation likely to be encountered during tank analyses. Our simulation studies show that with only two different path lengths at a single humidity level we can develop a calibration model that is insensitive to both humidity and path-length effects without increasing the bulk weight-percent moisture prediction errors.

Inherent in both simulation approaches is the assumption that the change in the water-vapor spectra will be linearly additive with the measured sample spectra (i.e., there are no interactions between changes in the water-vapor spectra because of humidity and path-length differences and the sample spectra because of bulk-water concentration). This will have to be verified experimentally. Early experimental results indicate that the LOWTRAN water-vapor spectra closely match the actual water-vapor spectra measured on our instrument. We are still working to measure the sample spectra at different humidity levels to test the assumption described above.

6.0 REFERENCES
