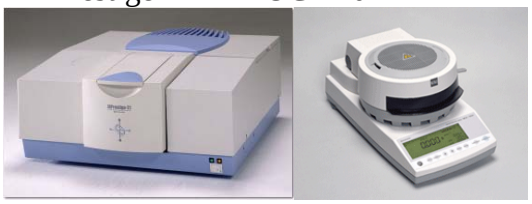
Quantitative Moisture Analysis of Tobacco by FT-NIR Spectroscopy

Introduction:
The quantitative analysis of unknown moisture content in solid samples is one of countless analytical methodologies that have evolved as a direct result of advances in analytical instrumentation. Traditionally, the moisture content of a material was determined by monitoring the samples weight as it undergoes a drying process on a moisture balance. While this method is both simple to perform and cost efficient, the time of analysis is inherently limited by the drying cycle. In addition, some pharmaceutical and biological materials can be adulterated and/or destroyed by the heating process. FT-NIR spectroscopy, however, has the intrinsic ability to nondestructively determine the moisture content of thermally sensitive samples in 'real-time'. This application note will validate the capacity of FT-NIR spectroscopy for quantitative moisture analysis in shredded tobacco leaves.

Sample Preparation:
Virginia Gentleman loose tobacco was selected for this method comparison. Seven samples (6g each) were removed from an envelope of tobacco purchased from a local convenience store. The tobacco portions were then transferred into Ziploc bags and sprayed with zero, one, two, or three sprays of de-ionized water using an industrial spray bottle. These samples were set aside as the calibration set. The remaining three samples were used to validate the calibration curve that was created through a partial least squares (PLS) analysis. All tobacco samples were then sealed and left overnight to ensure the complete absorption of the water by the tobacco. The following day, the tobacco sample was placed uniformly in a beaker and measurements were conduced.

Instrumentation:
IRPrestige MOC-120H



The FT-NIR spectra from the artificially wetted tobacco were collected on an IRPrestige-21 spectrophotometer fitted with an optional NIR kit and controlled by a PC operating IRSolution software (Ver. 1.20). The NIR kit for the IRPrestige extends the instruments measurement range to 12,500-340 cm^{-1}, includes a secondary light source (tungsten lamp), a secondary detector (InGaAs), and a CaF_2 beam splitter. The IRPrestige was also fitted with the IntegratIR integrating sphere accessory and a rotating sample plate from Pike Technologies. These accessories provided the ability to collect a

representative spectrum from the entire shredded tobacco sample. This in turn eliminated concerns regarding possible inhomogeneity of the moisture in a sample. A gold diffuse reflectance standard was used to collect the background spectra.

The Shimadzu MOC-120H moisture balance was programmed to automatically determine the moisture content using a temperature of 110 ^{o}C with a stop point < 0.05 % change in moisture over a 30 second measurement interval. The experimental analysis also utilized the integrated Microsoft direct connect feature for data transfer.

Results and Discussion:

One of the essential components for a successful quantitative spectroscopic analysis is a representative background scan. The top panel in Figure 1 presents the excellent background spectrum that is obtained using the gold diffuse reflectance standard. The bottom panel illustrates the efficiency of the linear baseline correction function available in the Shimadzu IRSolutions software package.

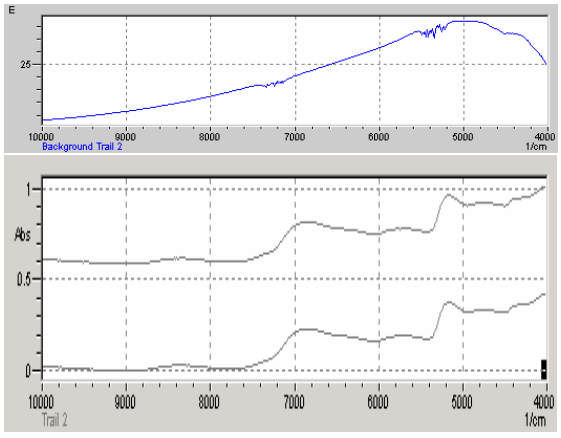


Figure 1: (Top) Typical background scan collected using a gold reflectance standard. **(Bottom)** The upper spectrum is the raw data and the lower spectrum has undergone a successful linear baseline correction.

Each tobacco sample within the calibration set was analyzed and underwent a linear baseline correction. The overlaid spectra are presented in Figure 2. In general, the absorbance of the tobacco sample increased as the moisture increased (i.e., number of sprays).

Figure 2: The FT-NIR spectra of the calibration set illustrate the general trend between absorbance and the number of sprays.

Next, a simple calibration curve was constructed from these spectra (collected in triplicate) using the PLS capabilities of the IRSolution software. The reference values for the analysis were the values obtained with the moisture balance and the direct connect feature (Table 1).

In order to test the robustness of our FT-NIR method, the sample set was run using the calibration curve. When the FT-NIR values were compared to values obtained with the moisture balance we

calculated a correlation coefficient of 0.96 for the two methods (Table 2).

	Temp (C)	Weight (g)	% loss
0	35	0.9989	0
0.5	58	0.9843	1.46
1	107	0.9416	5.74
1.5	110	0.9042	9.48
2	111	0.8495	14.96
2.5	110	0.7986	20.05
3	110	0.7747	22.44
3.5	110	0.7656	23.36
4	110	0.7581	24.11
4.5	109	0.7524	24.68
5	109	0.7474	25.18
5.5	110	0.7427	25.65
6	110	0.7388	26.04
6.5	110	0.7353	26.39
7	110	0.7324	26.68
7.5	109	0.7304	26.88
8	110	0.7283	27.09
8.5	110	0.7265	27.27
9	109	0.7242	27.5
9.5	110	0.7217	27.75
10	110	0.7199	27.93
10.5	111	0.7186	28.06
11	110	0.7173	28.19
11.5	110	0.7163	28.29
12	110	0.7151	28.41
12.5	110	0.7135	28.57

Table 1: Raw data of the amount of water loss over time obtained from the Shimadzu MOC-120H moisture balance and its built in Microsoft direct connect feature.

% Moisture using IR-Prestige	% Moisture using Moisture Balance
29.253	30.65
28.973	30.73
28.306	29
28.685	29.32
28.666	28.88
28.525	29.55
28.944	28.07
28.333	29.31
28.299	28.72
28.175	28.97
27.994	28.55

Table 2: Comparison of data obtained using the IR-Prestige and the MOC-120H moisture balance

Conclusions:

The presented data clearly demonstrates that there is a strong correlation between the percent moisture obtained between the FT-NIR and the moisture balance methods.

In addition, it should be recognized that the analysis of a single sample in the validation set was analyzed in less than a minute by the FT-NIR method versus a drying time of ~16 minutes on the moisture balance. This application note confirms that the Shimadzu IR-Prestige and IR Solution software can be used for real-time quantitative moisture analysis.