



SPECTRA OF THE MONTH

**MEASURING ORGANIC MATTER IN SOIL
WITH NIR ABSORBANCE SPECTROSCOPY**

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INTRO

BACKGROUND OF APPLICATION

Earth Day is an annual event that is celebrated globally on April 22 in support of environmental protection. Earth Day was proposed in 1969 at a UNESCO Conference as “a day to honor the Earth and the concept of peace”, and the first event was observed just a year later, which included a nationwide environmental teach-in in the United States. While the event was focused on the U.S. in 1970, it has grown to a global event that includes 1 billion people in over 193 countries today. Indeed, in 2016, the Paris Agreement that included a historic climate protection treaty was signed by over 120 countries, including the U.S., the U.K., and China, on Earth Day. To commemorate this day of environmental awareness, we decided to measure the Earth itself in the form of soil measurements. Soil is composed of many molecules, minerals, and organic matter, with some strongly associated with spectroscopic absorbance peaks in the near infrared (NIR) spectrum. These include free water OH features at 1400 nm and 1900 nm and clay lattice OH features at 1400 nm and 2200 nm. Peaks at 1400 nm and 1900 nm can also be indicators of moisture. Additionally, spectra from the visible range have also been correlated to organic matter measurements as well as pH value.

This experiment aims to reproduce these measurements of organic matter in soil samples using absorbance spectroscopy in both the visible and NIR spectral ranges. Multiple soil samples were collected from the front and back yard of our office and measurements from each sample were compared to determine if common peaks were present (Figure 1).



FIGURE #1: Soil samples used for this experiment (from left to right: soil sample 1, soil sample 2, and soil sample 3).

DESCRIPTION OF SPECTROSCOPY SETUP

In this experiment (Figure 2), we employed two spectrometers, starting with the [NIR256-2.5-HSC-EVO](#) model. Tailored for NIR measurements up to 2.5 μm , this model boasts a high-sensitivity optical bench paired with cutting-edge electronics, delivering outstanding performance with a scan sample speed of 0.54 ms and integration times as rapid as 10 μs . Equipped with our reliable InGaAs array detector and ultra low-noise electronics board, the [AvaSpec-NIR256-2.5-HSC-EVO](#) features USB3.0 and Giga-Ethernet connection ports. Its versatility includes multiple grating and slit options, as well as digital and analog I/O ports for shutter control, pulse modulation of light sources, and spectrometer gain adjustment between High Sensitivity and Low Noise modes. This instrument covered a wavelength range of 1000-2500 nm with a 50-micron slit configuration.



FIGURE #2 Experimental setup: Soil samples are placed in small petri dishes and measured within an integrating sphere with a built-in halogen light source.

The second unit employed was our compact spectrometer [AvaSpec-NXS2048CL](#), also known as [NEXOS™](#). Utilizing our innovative semi-automated manufacturing process, it guarantees enhanced consistency and reproducibility across units. It offers USB2.0 communication and RS232 and SPI communication protocols, a CMOS linear array detector, ultra-low stray light as low as 0.1%, and a signal/noise ratio of 375:1. Furthermore, this spectrometer can be customized with a wide range of gratings (13 total available) and the replaceable slit option is now standard for non-OEM units. This instrument was optimized for the visible range as well as the start of the NIR with a 350-1100 nm wavelength range and had a 50-micron slit installed.

The light source used for the absorbance measurements was a built-in halogen light in our [AvaSphere-50-LS-HAL-12V integrating sphere](#). While specifically designed for reflection applications, this integrating sphere is also useful for low reflecting materials and NIR measurements, where signal strength can be limited. The built-in halogen light source provides diffused halogen light on the sample without the losses associated with fiber-optic coupling, with up to 160 times more light on the sample compared to our standard reflectance integrating sphere. The [AvaSphere-50-LS-HAL-12V](#) has an internal diameter of 50 mm, a 10 mm sample port, an SMA-terminated reference port, and a direct collimated SMA-port for collection of the signal with any of our [AvaSpec spectrometers](#).

Other accessories used for this experiment included our TR-Stage to mount the integrating sphere, a [WS-2 white reference tile](#) for the reference measurement, a 400-micron core bifurcated fiber optic cable ([FCB-400UVIR-1-ME](#)) to connect the two spectrometers to the integrating sphere, and multiple petri dishes to hold the reference tile and soil samples over the integrating sphere port.

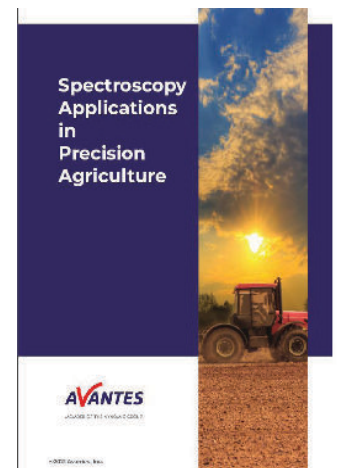
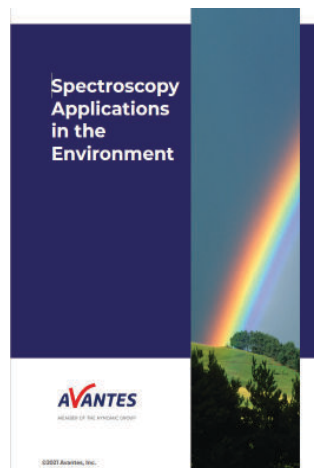
DESCRIPTION OF METHODOLOGY

The soil samples used for this experiment were two soil samples taken from the front yard of our office and one sample taken from the backyard of our office. The soil samples all underwent a sifting process to remove larger chunks from the samples and achieve a relatively fine texture. All the samples were left to air dry over a day to let some water evaporate from the samples but were not heated to a higher temperature that would remove all moisture. The samples were individually poured into petri dishes for measurement.

For data analysis, we used the Absorbance mode in AvaSoft, our exclusive custom software package. This mode is specifically designed for absorbance applications, where the reference measurement will report 0 A.U. (absorbance units) and 5 A.U. when the light source is turned off. In this experiment, our WS-2 white reference tile was used as the reference. To match the measurement distance and medium of the soil samples, the reference tile was also placed in a petri dish for the reference measurement. For the Nexos, we used an integration time of 1 ms and set averaging to 1000. For the NIR256-2.5-HSC, we used an integration time of 10 ms and set averaging to 100. Integration time can be adjusted to increase or decrease the amount of light being measured at one time and affects the overall magnitude of the reported spectrum, and higher averaging provides more consistency between scans. The averages were set to their respective values to achieve this high level of consistency as well as to match the total measurement times for the two instruments. For a more uniform presentation of the spectra, an additional setting was used to merge the two spectrometer measurements.

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TEST DATA AND RESULTS

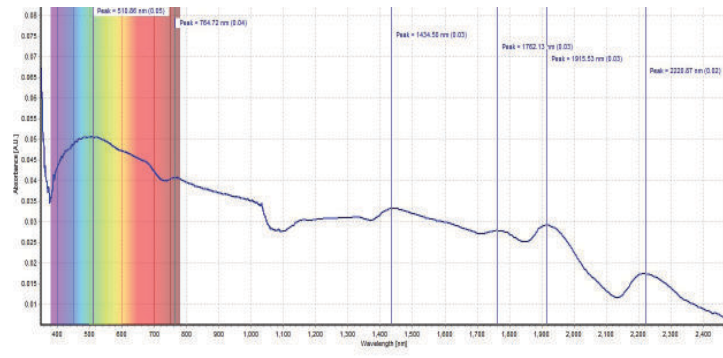


FIGURE #3: Absorbance spectrum of soil sample 1 (absorbance peaks at 510.86 nm, 764.72 nm, 1434.50 nm, 1762.13 nm, 1915.53 nm, and 2220.87 nm).

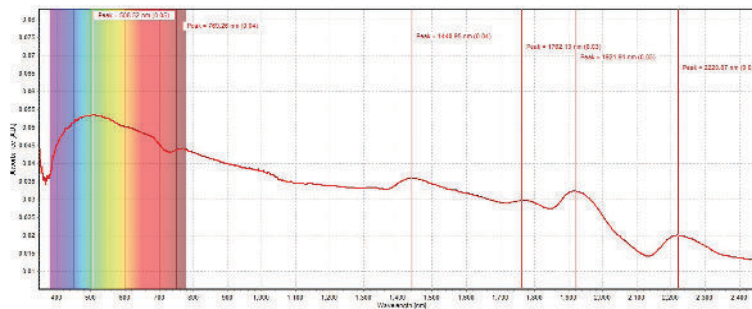


FIGURE #4: Absorbance spectrum of soil sample 2 (absorbance peaks at 508.52 nm, 769.26 nm, 1440.95 nm, 1762.13 nm, 1921.91 nm, and 2220.87 nm).

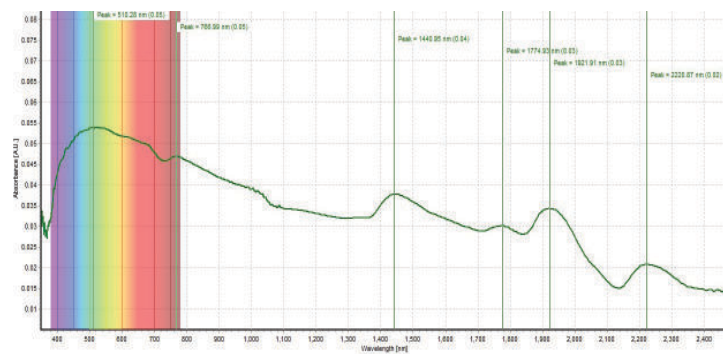


FIGURE #5: Absorbance spectrum of soil sample 3 (absorbance peaks at 510.28 nm, 766.99 nm, 1440.93 nm, 1774.93 nm, 1921.91 nm, and 2220.87 nm).

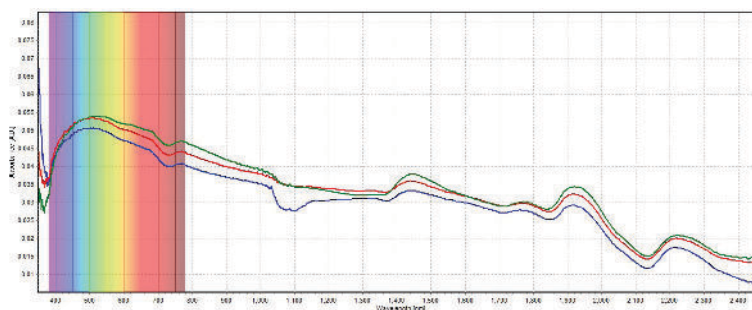


FIGURE #6: Spectra of soil sample 1 (blue), soil sample 2 (red), and soil sample 3 (green), shown together for comparison.

ANALYSIS

All three soil samples show absorbance peaks in similar areas of the spectrum. Soil sample 1 showed absorbance peaks at 510.86 nm, 764.72 nm, 1434.50 nm, 1762.13 nm, 1915.53 nm, and 2220.87 nm (Figure 3). Soil sample 2 had similar peaks at 508.52 nm, 769.26 nm, 1440.95 nm, 1762.13 nm, 1921.91 nm, and 2220.87 nm (Figure 4). Soil sample 3 also showed similar peaks at 510.28 nm, 766.99 nm, 1440.93 nm, 1774.93 nm, 1921.91 nm, and 2220.87 nm (Figure 5). A graph of all the spectra together is included for comparison of all samples (Figure 6).

The NIR peaks around 1400, 1900, and 2200 nm have previously been correlated to organic matter in soil, so these peaks are promising to observe. While it would be ideal to see the peak locations be exact matches between the samples, this variation could be attributed to minor differences in the soil composition or the measurement surface, as the powdery texture of the soil can cause light scattering that may lead to less consistent results. That being said, the absorbance peak near 2200 nm was consistently 2220.87 nm for all three samples, illustrating high repeatability for this peak measurement. The NIR absorbance peak around 1765 nm could not be directly attributed to any quantity in the soil but was also rather consistent in each measurement. The same was determined for the two absorbance peaks seen in the visible range around 510 nm and 765 nm. While no direct correlation could be found, further post-processing could be used to link these peaks to a property of soil such as total carbon or pH value.

CONCLUSION

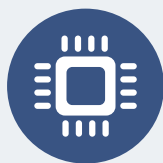
This experiment highlights the use of spectroscopic analysis in detecting organic matter in soil. Unfortunately, other quantities such as pH value could not be clearly deduced from the absorbance measurements. The peaks at 1400 nm, 1900 nm, and 2200 nm have been well documented as absorbance peaks for organic matter present in soil, while the other peaks observed in our spectral data were not as obviously correlated. Further quantification could be utilized to link these absorbance peak to another property associated with soil such as pH.

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