

 	Code: METH07V01	Date: July 31, 2014 Author: Dickens Ateku
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STANDARD OPERATING PROCEDURES

METHOD FOR ANALYSING SAMPLES FOR SPECTRAL CHARACTERISTICS IN MID IR RANGE USING ALPHA

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METHOD DOCUMENT CONTROL LOG

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SCOPE AND APPLICATION

This is a World Agroforestry Centre's Soil-Plant Spectral Diagnostic Laboratory SOP for analysis of various chemical properties in both soils and plants using mid IR spectra recorded with Alpha R module. The technique involves shining of mid infrared light on the samples and capturing of the diffused and reflected light in to raw file known as the interferogram. The spectrum thus recorded bears the qualitative and quantitative chemical aspect of the sample for further use with various data processing systems. This SOP is applicable to those doing analysis on infrared laboratory using ALPHA spectrometer Module.

PRINCIPLE



The ALPHA R Module is designed for measurements in diffuse reflection. When Mid IR light is incident on a rough or a matte surface, two types of reflection occur: Specular reflection (i.e. the light reflects directly off the surface) and the diffuse reflection (the IR light penetrates the sample surface and is partly diffusely reflected, i.e. reflected over all angles, due to the rough surface). The optics of a diffuse reflection accessory is designed in such a way that detection of diffusely reflected light is optimized and the detection of specular reflected light is minimized. DRIFTS (diffuse reflectance infrared fourier transform spectroscopy) is an analyzing technique in FT-IR spectroscopy that makes use of the phenomenon of diffuse reflection. The key advantage of these techniques is that it enables analysis of strongly scattering and absorbing samples unlike transmission measurements and equally has high signal intensity.

ABBREVIATIONS AND DEFINITIONS

SOP –Standard Operating Procedures

MIR – Mid Infra-Red

STD – standard soil sample

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RELATED DOCUMENT

- ALPHA spectrometer sop
- sample reception and preparation sop
- MIR workflow
- Infrared health and safety

RELATED FORMS



- Login form
- Recording sheet

SAFETY AND ENVIRONMENT

- Electrical hazards: Electrical systems must conform to the ICRAF standards. No shock hazards exist inside the instruments. Do not try to repair the faulty electrical system but call for assistance from an authorized service representative or an individual with training in electronic repair. The instruments require a third-wire protective grounding conductor. Three-to-two wire adapters are unsafe for these instruments.
- Refer to the safety instructions in the equipment manual available in the lab.

REQUIREMENTS

- **Supplies**
 - a. 70% Ethanol (for cleaning the Aluminium sample holding cups)
 - b. Ear buds (for cleaning the Aluminium sample holding cups)
 - c. Tissues
- **Equipment**
 - a. Retsch motor grinder RM 200
 - b. Aluminium sample holding cups
 - c. Micro spatula

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PROCEDURE

▪ Sample preparation and loading

- Collect about 10 g subsample from air-dried 2-mm sieved soils and minerals that are processed through SOP. Use coning and quartering method in case of large samples. (Refer to the Alpha Spectrometer SOP pages 10 and 11)
- Grind the sample using RM 200 Retsch motor grinder to a fine powder to attain a particle size between 20- 53 μm (0.5mm).
- Load the sample into aluminium sample cup by filling it with the fine ground soil. It takes about 1-2 g depending on the type of soil. (Refer to the Alpha Spectrometer SOP page 12)
- Remove the excess soil and level surface with a straight-edged tool such as a straight spatula, glass slide or small knife.
- Place the loaded samples in the sample holder tray which accommodates 20 samples.
- Prepare a data sheet indicating sample IDs and sample position in the loading tray. Ensure that you enter the sample ID immediately after filling the cup

▪ Recording spectra measurement

- Power up the instrument and log into the OPUS software (Technician must get himself/herself familiarized with operating instructions of the instrument by going through the ICRAF soil and plant spectral diagnostic laboratory adopted version of user manual for Alpha R Module)
- Select the advance data collection option in OPUS and load the ICRAF soil and plant diagnostics laboratory adopted method (**ICRAF Drift.xpm**.) refer to SOP.
- Create a folder that identifies the batch of soils being analyzed on the computer connected to the instrument. All spectra recorded on test samples should be saved in a separate folder. For example C:\ICRAF\RAW\SOTUBA SITE SOILS.....for all Sotuba soils. Refer to SOP.

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- d. Place the standard gold cap in the scanning position, close the sample compartment, and make background corrections by clicking on “background single channel” under “basic menu” tab of “measurement” window. Refer to SOP.
- e. Replace the gold sample cup with STD Mua sample and position it in the scanning position and record the spectra by clicking on the “check signal menu” Refer to SOP.
- f. Check the ADC count (signal position and amplitude count). If necessary optimize the signal reading for the standard sample (ICR_Mua STD signal) by adjusting the screw knob on the drift sample compartment and save peak position
- g. Finally click on the “basic menu” icon to save the highest amplitude recorded. A beeping sound confirms the saving of the amplitude and position settings
- h. Replace the standard Mua sample cup with that of test sample and place it in the scanning position.
- i. Enter the sample serial number (SSN) on sample description menu and click on the “sample single channel” tab to scan the sample
- j. Open the sample compartment and replace the scanned sample with the next sample on the sample holder and scan.

QUALITY CONTROL

▪ Reference Material

- a. Reference standards: Mau soil and White sand are used a reference standard.
Everyday a new spectrum is recorded on these samples before analyzing test samples and checked against the previous records. Appropriate correction actions where necessary are implemented. Normally no adjustments are required.
- b. Blanks: A standard gold cap is sued to correct the background
- c. Calibration: No calibration is required.

▪ Data Validation

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- a. Check the sample data and QC data to verify that the data to be reported is based on acceptable analyses and meet acceptable limits.
- b. Verify that Mua standard spectra have been entered into the appropriate logs.
- c. Verify that the results are saved in appropriate folder and format.
- d. Place data in the laboratory database.

▪ **Supervisor Review**

- a. Check the sample data and QC data to verify that the data to be reported is based on acceptable analyses and meet acceptable limits
- b. If any errors are found, return data for repeat analysis.

REFERENCES

- ALPHA – R Module for diffuse Reflection Measurement, 1st Edition 1009974, BRUKER OPTICS, May 2008, page 1–page 9
- ALPHA Installation Instructions, 1st Edition 1005543, BRUKER OPTICS, April 2007, page 1–page 35

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ANNEX

Work Flow

