

STANDARD OPERATING PROCEDURES

NEAR-INFRARED DIFFUSE REFLECTANCE SPECTROSCOPY METHOD FOR ANALYSING SOILS AND PLANTS USING BRUKER MULTI PURPOSE ANALYSER SPECTROMETER

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characteristics in Mid IR range using MPA	

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 near IR spectra recorded with MPA module. The technique involves shinning of near infrared light on the samples and capturing of the diffuse 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 MPA spectrometer Module.

PRINCIPLE

The MPA Module is designed for measurements in diffuse reflection and transmission.

The optics of a diffuse reflection accessory is designed in such a way that detection of diffuse reflected light is optimized and the detection of specular reflected light is minimized. A DRIFT 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

- DRIFT Diffuse Reflectance Fourier Transform spectroscopy.
- OPUS –Bruker's Optical User Software
- STD standard soil sample
- PVC Poly Vinyl Chloride
- SOP standard operating procedures
- NIR- near infra-red
- MPA Multi-Purpose Analyze



RELATED DOCUMENT

- Multi-Purpose Analyzer Technical Manual
- Sample Reception and Preparation SOP
- NIR 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.

REQUIREMENTS

- Equipment
 - a. Rolling pin
 - b. PVC
 - c. 2mm sieve
 - d. Sample holders
 - Small Petri dish 7cm diameter (Duroplan)
 - Wide Petri dish 9 cm diameter (Duroplan)
 - Vials
 - Mug 9cm diameter
 - Cuvette (for liquids –transmission)
 - e. Spatula
 - f. Non-fluffy cloth



NOTE: No reagents or chemicals used

PROCEDURE

- Sample Analysis
 - a. 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 MPA Module)
 - b. Select the advance data collection option in OPUS and load the ICRAF soil and plant diagnostics laboratory adopted method by selecting and opening:
 - ICRAF MPA_SPHEREMACROSAMPLEICRAF.XPM. (for solid samples diffuse reflectance) Refer page 23 of the MPA manual. Or
 - MPA_SAMPLECOMPARTMENTICRAF30.XPM (for liquid samples -transmission) (page 16).
 - c. Create a folder that identifies the batch of soils being analyzed on the computer connected to the instrument
 - d. Sample scanning process in carried out in OPUS LAB software (Refer page 33 of the MPA technical manual).
 - As a part of quality control, the lab records spectra of selected standard soils, Grey standard and Kaolinite every day. These spectra are saved in a separate folder C:\ICRAF \RAW\DAILY STANDARDS.
 - f. 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 page 33 of the MPA SOP manual.
 - g. Place the sample holder (petri dish) on the instrument against the Infra-red light and start scanning. Refer page 58 (MPA manual).

QUALITY CONTROL

- Reference Material
 - a. **Reference standards:** Infrared ICRAF laboratory uses Katumani, High Black Cracking Clay Soil (HBCCS) and Mua soils, Grey Standard and Kaolinite as reference standards. Everyday new spectra are 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 internal gold reference is used to correct the background
 - c. Calibration: No calibration is required
 - d. Duplicates: Not used
 - e. **Quick Compare:** Perform opus quick compare of the standards to see how the instrument is performing (refer to the MPA Technical manual).

1. Data Validation

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

b. Supervisor Review

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

REFERENCES

2. Multi-Purpose Analyzer User Manual, BRUKER OPTIC, 2003, page1 – page 48



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ANNEX

1. Work Flow



*Method not frequently used



2. Infrared MPA spectrometer standards fact sheets

MPA Instrument calibration standards

These are standards used for all analysis in the ICRAF Soil-Plant diagnostic laboratory.

- Heavy black cracking clay soils (HBCCS), Katumani, and MUA standards are run every day in the morning before you start any analysis and every time you start on a new site.
- Grey card, Kaolinite (clay) and White sand are commercial Standards which are run every day in the morning before you start any analysis and every time you start on a new site.

3. Quality Control fact sheet

PROCESS	DESCRIPTION
Background Measurement	The instrument is set in a way that it forces the
	user to perform Background measurement after
	every 1 hour. The measurement normally
	increases the signal to noise ratio of the spectrum
	produced (improves the quality of the spectra)
Measurement of the selected soil standards.	The standards used in ICRAF infrared laboratory
	are HBCCS, Katumani, MUA, Kaolinite, Grey card
	and White Sand. The measured spectra are used
	to monitor the performance of the instrument.
Check on the quality of the spectrum measured	Always monitor the instrument moisture. Ensure
	that the spectrum measured does not have any
	noise due to moisture and carbon dioxide. The
	moisture noise peaks are expected at around
	3600cm and 1600cm wave numbers while the
	carbon dioxide peak is expected at around
	2400cm wave numbers.