

	Code: METH10V01	Date: 31 July, 2014 Author: Dickens Ateku
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STANDARD OPERATING PROCEDURES

METHOD FOR ANALYSING SAMPLES FOR SPECTRAL CHARACTERISTICS IN VISIBLE NEAR IR RANGE USING ASD

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 World Agroforestry Centre	 Soil-Plant Spectral Diagnostics Lab	STANDARD OPERATING PROCEDURE		Date: 31 st July,2014
		Code: METH10V01		Author: Dickens Ateku
		Title Method for analysing samples for spectral characteristics in Mid IR range using ASD		

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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 visible and near IR spectra recorded with ASD module (The spectrum attained covers the range between 350nm to 2500nm, the indicated Visible Near Infrared region of the electromagnetic spectrum). The technique involves shining of visible and near 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 ASD spectrometer Module

PRINCIPLE



The ASD Module is designed for measurements in diffuse reflection. When visible and near 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. ASD is a Dispersive spectrometer and thus uses holographic gratings to split the light.

ABBREVIATIONS AND DEFINITIONS

ASD - Analytical Spectral Device

NIR – near infra-red

SOP – standard operating procedures

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RELATED DOCUMENT

- Analytical Spectral Device (ASD) 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. Three-to-two wire adapters are unsafe for these instruments.
- Refer to the safety instructions in the equipment manual available in the lab.

REQUIREMENTS

- **Equipment**
 - a. Analytical Spectral Device (ASD)
 - b. Mug probe lamp
 - c. White reference spectralone
 - d. Rolling pin
 - e. PVC paper
 - f. 2mm sieve
 - g. Small Petri dish 7cm diameter (Duroplan)
 - h. Spatula
 - i. Non –fluffy cloth

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PROCEDURE

▪ Sample preparation and loading



Ensure the petri dish is clean and load each sample against the designated laboratory id.

Refer to Analytical Spectral Device (ASD) SOP page 2

▪ Recording spectra

Once the spectrometer has warmed up, start the spectrometer software by either double-clicking on the Indico Pro icon on the desktop or by selecting the software from the “Start” menu at the bottom. Refer to Analytical Spectral Device (ASD) SOP page 2

- From the File pull-down menu select New Project or click on the first icon on the opened Indico Pro start up window. A new project window pops up. To start a new project and enter the project name. Refer to Analytical Spectral Device (ASD) SOP page 3
- Click on change directory to make a directory path where you want to save your spectra. A new directory path window pops up. Once you have selected the folder where you want to store your spectra, click on ok. Refer to Analytical Spectral Device (ASD) SOP pages 3 to 5
- Now optimize the instrument for the lighting conditions you will be using. Make sure the light on the mug probe lamp is on, place the white reference on top of the mug probe window and click on the baseline icon for the dialog window to pop up. Make sure the “optimize first” message is checked before clicking on yes. Refer to Analytical Spectral Device (ASD) SOP page 6
- Put the Petri dish with the sample on the mug probe window and Click on the scan icon to scan the sample. The Indco pro dialog window pops up once the instrument has finished scanning the sample for the first time. Refer to Analytical Spectral Device (ASD) SOP page 8
- Turn the Petri dish at an angle of 90 degrees after the first scan to capture any variation due to sample distribution within the Petri dish. Click on yes and **scan again** the sample for the second time .The same Indco pro dialog window pops up. Refer to Analytical Spectral Device (ASD) SOP page 8
- Save the averaged spectrum by Clicking on the save icon and enter the name of the spectrum (SSN) on the file name column of the save spectrum file window that pops up and click on ok to save it. This save spectrum file window closes once you have clicked on ok to save the spectrum. Spectrum names will be the SSN for each sample. Refer to Analytical Spectral Device (ASD) SOP page 9
- When closing the Indico Pro soft ware a dialog window pops up asking if you want to save the change you have made, click on yes to save the work you have done. Refer to Analytical Spectral Device (ASD) SOP page 14

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QUALITY CONTROL

▪ Reference

- a. Reference material: Mua soil, Grey Standard and Kaolinite are used 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 White spectral on reference is used to correct the background
- c. Calibration: This is a dispersive kind of spectrometer and is subjective to instrumental drifts, instrument calibration are therefore done at Analytical Spectral Devices plant in California USA but we do have own internal reference materials that we have been using over time and can use in monitoring instrument wavelength shift (drift), specific absorbance/reflectance reading are taken at specific wavelength and this information is entered in to our ICRAF Soil and Plant Spectral Diagnostic Laboratory database. The main reference materials that we are currently using are Kaolinite powder, Grey Satd1, Mylar and Card on the ASD spectrometer. Refer to Analytical Spectral Device (ASD) SOP page 15



▪ 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.

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REFERENCES

- Field Spectro-radiometer Analytical Spectral Device Manual

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ANNEX

Work Flow

