

 	Code: METH09V01	Date: July 30, 2014
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STANDARD OPERATING PROCEDURES



METHOD FOR ANALYSING SAMPLES FOR SPECTRAL CHARACTERISTICS IN NEAR IR RANGE USING MPA

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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 shining of 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 MPA spectrometer Module.



PRINCIPLE

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

- PVC - Poly Vinyl Chloride
- SOP – standard operating procedures
- MIR- mid infra-red

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- MPA - Multi-Purpose Analyzer

RELATED DOCUMENT

- Multi-Purpose Analyzer (MPA) 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.

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



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- 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 manual.
 - e. As a part of quality control, the lab records spectra of Mua standard soil, Grey standard and Kaolinite every day. These spectra are saved in a separate folder C:\ICRAF \RAW\DAILY STANDARDS. Identify the spectra recorded on the standard soil by naming it as “mua_standard” and the machine adds the date to this name.

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- 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:** 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 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 refer to the MPA SOP.

▪ 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

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- 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.

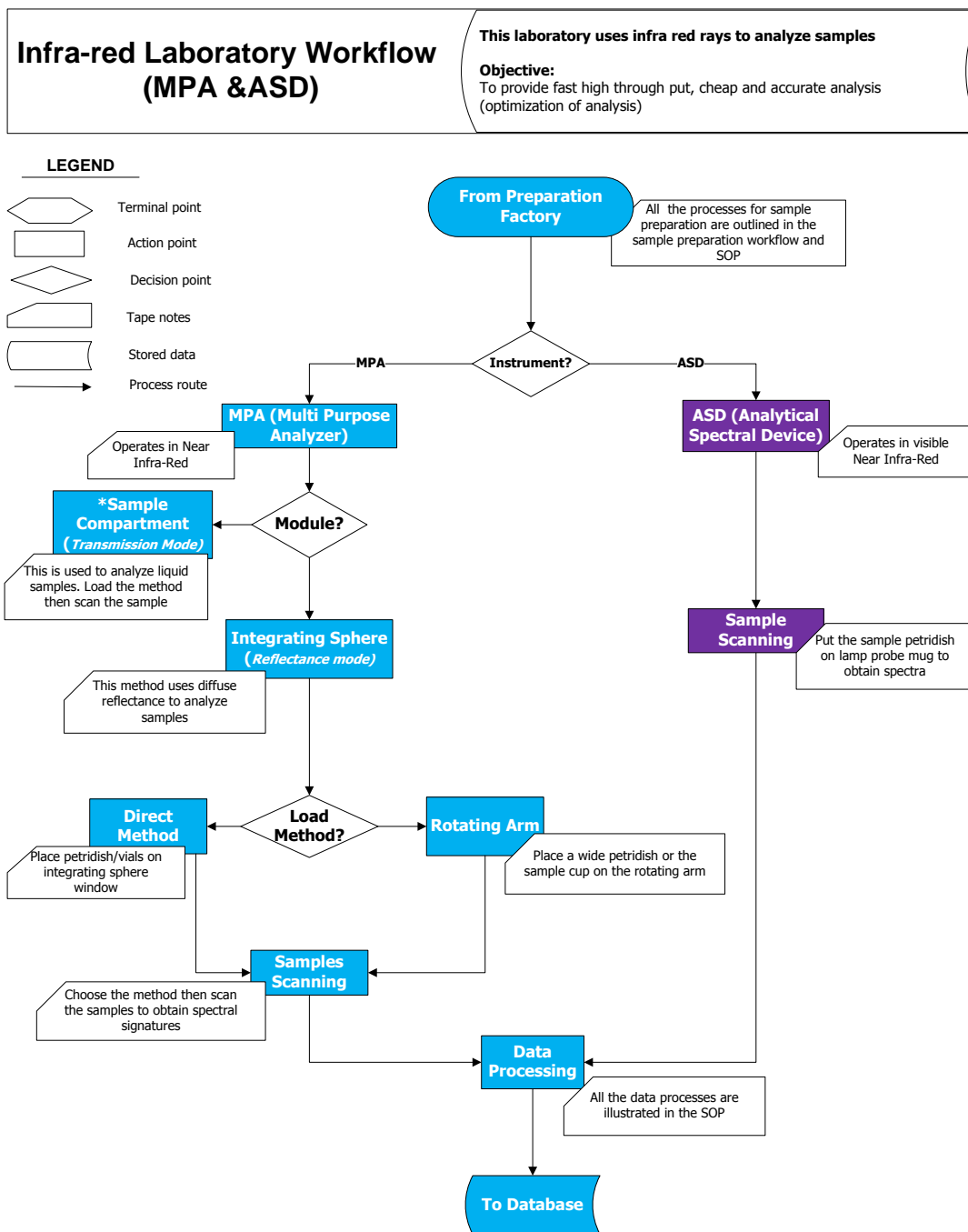
REFERENCES

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



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ANNEX

Work Flow



*Method not frequently used

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