

 	Code: METH11V01	Date: July 30, 2014
		Author: Robin Chacha

STANDARD OPERATING PROCEDURES

METHOD FOR ANALYSING SOIL SAMPLES FOR MINERAL COMPOSITION USING XRD

Contact Details: Telephone: +254 (20) 7224000/4235/4279/4163 Email: icraf-speclab@cgiar.org : Laboratory Manager, Soilspec_Lab_Manager@cgiar.org Website: www. http://worldagroforestry.org/research/land-health	Address Details: World Agroforestry Centre, Mailing: P.O Box 30677-00100 Nairobi, Kenya Physical address: United Nations Avenue Off Limuru Road, Gigiri, Nairobi, Kenya
--	--

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

METHOD DOCUMENT CONTROL LOG

	Name and position	Signature
Author(s)	Beatrice Oware – Laboratory Technician	<i>[signature for completeness and correctness of document]</i>
Verifiers	Robin Chacha – Senior Laboratory Technician	<i>[signature for completeness and correctness of document]</i>
		<i>[signature for completeness and correctness of document]</i>
Authorizer	Mercy Nyambura – Laboratory Manager	<i>[signature for completeness and correctness of document]</i>



Distribution of valid copies of this document:

G143D	X-Ray Diffraction Lab
-------	-----------------------

Revise before:	<i>[date]</i>
Historical data	<i>[dates of previous issues]</i>
Date retrieval:	<i>[to be filled in once the document has become obsolete and is archived]</i>

Changes in this version compared to previous version:

[Describe what has changed in the text of this version compared to the previous version]



 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

SCOPE AND APPLICATION

The determination of the types and relative amounts of the minerals present in soil (soil mineralogy) is determined routinely because of its strong influence on soil behavior, its use in soil classification, and its relevance to soil genetic processes. ICRAF Soil-Plant Spectral Diagnostic Laboratory uses X-ray diffraction technique for qualitative and quantitative assessment of minerals in soils. X-ray diffraction is the most powerful technique used for analysis of minerals and offers mineral phase's identification and quantification. The standard operation describes the Whole rock approach where the sample is prepared into a random powder which helps in analysis of total amounts and identification of non-clay minerals present. The analysis provides information about the minerals present in a sample and also the abundance. It can be applied to all types of soil samples. X-ray diffraction has a wide range of applications in geology, material science, environmental science, chemistry, forensic science, and the pharmaceutical industry, among others. In Agriculture it has application in qualitative and quantitative analysis of actual minerals in top soils and sub soils, in classification of soils in terms of weather able and nutrient rich minerals to measure soil fertility potential and use of mineralogical data in pedo-transfer functions to directly predict soil functional properties.

PRINCIPLE

Powder diffraction is routinely used as a fingerprint identification technique of various solid materials in the laboratory. It is a high-tech, rapid, cheap and non-destructive technique for qualitative and quantitative analysis of crystalline compounds; about 95% of all solid materials in the soil are crystalline. When X-rays interact with a crystalline substance or powder, a diffraction pattern called a diffractogram is produced and can be quantified. Information obtained from this patterns include phase composition of a sample, types and nature of crystalline phases (minerals) present, crystal structure, amount of amorphous (OM) content, micro strain, size and orientation of crystallites. XRD has become an indispensable method for materials investigation, characterization and quality control. The angle and intensity of the diffracted beam recorded by a detector forms a diffraction pattern, which provides information about a sample. The diffraction pattern for every phase is as unique as a fingerprint. Phases

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

with the same chemical composition can have drastically different diffraction patterns. Use of the position and relative intensity of a series of peaks is used to match experimental data to the reference patterns in the database such as the Powder Diffraction File (PDF). The PDF contains over 200,000 diffraction patterns of crystalline phases. Modern computers have automated search/match programs that determine what phases are present in a sample by quickly comparing the d spacing of the unknown to those of known materials in the International Centre for Diffraction Data (ICDD) Powder Diffraction File (PDF).

ABBREVIATIONS AND DEFINITIONS



XRD - X-Ray diffraction

RELATED DOCUMENT

- Laboratory manuals (quality, service, safety, training)
- Policy documents
- EQUIP 06 XRD
- EQUIP 09 Balances
- EQUIP 11 Centrifuges
- EQUIP 12 Ovens
- EQUIP 17 McCrone Micronizing Mill
- EQUIP 23 All software
- QC 03 Quality assessment
- Laboratory Safety Manual
- XRD Logbook

RELATED FORMS

- XRD sample list
- WF 10 -Soil mineralogy analysis using Bruker D2 PHASER Diffractometer System workflow

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha



SAFETY AND ENVIRONMENT (Refer to HS 01 XRD Safety manual)

Regulation of the manufacture and use of analytical X-ray instruments is adopted from the United States system. When used properly, X-ray instruments pose few risks to careful users. Care should be taken when using any analytical X-ray instrument. Never put any part of your body in the expected path of the main beam. Strictly only trained personnel should be allowed to operate the XRD spectrometer and should have studied the operator's manual thoroughly. Manufacturer's overall radiation safety instructions should be adhered to. Eating, drink, or smokes are prohibited in laboratory. For other safety information refer to Laboratory Safety manual.

REQUIREMENTS

- **Equipment**
 - a. A D2 PHASER Diffractometer with appropriate software
 - b. McCone micronizing mill
 - c. Weighing balance
 - d. Centrifuge
 - e. Oven
 - f. Vortex mixer

- **Material**
 - a. Corundum and quartz standard reference material
 - b. Standard 55mm sample discs made from PMMA
 - a. C0klmkoolant, Innovatek cooler fluid
 - b. Soft brush
 - c. PH-indicator strips (non- bleeding)
 - d. Weighing paper (4"x4")
 - e. Mortar and pestle

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

- f. Test Sieve, 250 μm mesh
- g. Spatulas
- h. Squeeze bottles
- i. Non fluffy lab tissues
- j. Hygrothermometer

- **Chemical**

- a. Hexane 99 %
- b. Ethanol 99%
- c. Deionized water



PROCEDURE

- **Sample preparation (Refer to METH 11)**

- a. Milling - Samples are milled using a McCrone Grinding Mill with agate grinding elements in a jar. The unique grinding action of the mill rapidly reduces particles to sub micrometer sizes and mixes for homogenization required for quantitative and qualitative analytical methods (Approx. 10 μm). Make sure to retrieve as much sample as possible after milling. Centrifuge to remove ethanol, decant, dry and crush sample

- **Sample mounting (randomly oriented mounts)**

- a. Load the randomly prepared samples into the well of a low background sample holder and tap gently on the bench to help fill and pack to avoid sample displacement which causes peak shifts.
- b. Using a sharp razor, tap the sample surface slowly in all directions to distort orientation a few times, and then continue to level while gently removing the loose

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

excess sample powder by scrapping off from the edges of the well of the sample holder.



- c. It is very important to have the correct sample level to the well surface since any error in the height of the sample will cause peak displacement.
- d. After measurement the disc can be off loaded and washed with tap water and then re-used.

▪ **Sample measurement**

- a. Switch on the instrument and let it warm for thirty minutes. (Refer to EQUIP 06)
- b. Pull down the spherical handle of the stage and place onto sample holder into the sample position of the stage(Goniometer)
- c. Lift the sample back into the sample measurement position by pulling up the spherical handle of the stage and slide down the instrument door. Press down door handle with force in order to close it correctly.
- d. Activate the High Voltage
- e. Set measurement parameters for a typical Lynx eye (Refer to EQUIP 06).
- f. Select start button to initiate acquisition.

▪ **Data acquisition**

The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles. A peak in intensity occurs when the mineral contains lattice planes with d-spacings appropriate to diffract X-rays at that value of θ . Results are presented as peak positions at 2θ and X-ray counts (intensity) in the form of a table or an x-y plot (shown above). Intensity (I) is either reported as peak height intensity, that intensity above background, or as integrated intensity, the area under the peak. When sample material is analyzed by XRD a range of diffracted peaks will occur and a diffractogram is obtained. The angle of each peak is used to identify the mineral

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

phase while the intensity of the peak will indicate relative amount present. The result of an XRD measurement is a Diffractogram showing:-

- a. Phases present (peak positions),
- b. Phase concentrations (peak heights),
- c. Amorphous content (background hump)
- d. Crystallite size/strain (peak widths).

A search of the ICDD standard database of x-ray diffraction patterns enables quick phase identification for a large variety of crystalline samples.

QUALITY CONTROL

Sample preparation

During sample loading or packing of the sample on the disc holders the key point to consider is the preferred orientation and its effect on the diffractogram.

The density of the packing has to be moderate to allow X-ray beam to go through the particles to achieve maximum good particle statistics.



Sample particles must be randomly oriented and have a flat, smooth and homogeneous surface to achieve a good resolution grains less than 10µm is needed.

Calibration Standards

Use mounted corundum to check verification of the instrument working conditions at least once a month. Use of quartz standards to check to check the intensity of the quartz peak in the sample. Maximum counts high peak 9000 but with Soil Plant Spectral Diagnostic lab method, we get more than 3000 counts for the quartz peak and this is a good confirmation of our sample preparation procedure

Personnel

Personnel working at the sample reception laboratory performing sample processing and preparations should meet background and training specifications outlined qualifications and should follow the guidance provided concerning safety and protective outerwear.

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

Workflow

A workflow chart showing work procedure is displayed as guidance in order used to reduce the potential for contamination. Laboratory manuals and notebooks also should remain in designated rooms. The laboratory uses electronic data reporting to avoid this potential source of contamination. Standard bench sheets are used in the various processes and ensures that all necessary data is recorded in a consistent manner.

Equipment

The equipment used to perform sample preparations should function properly to generate reliable data. Here should be a scheduled maintenance service including setup, calibration, repair, record keeping to verify that equipment is functioning properly. Every equipment should be dedicated to a specific laboratory room and its instrument manuals from the manufacturer should be kept in the room

Data Recording and Record Keeping



The generation of all environmental data should be completely and consistently documented, so the results can be assessed by an independent party. If data are maintained electronically, data should be backed up on a regular basis and stored in a separate location from the original data, if possible. All data recording should be checked by the laboratory supervisor for correctness and completeness, and each entry should be checked for accuracy of transcription.

Diffraction evaluation

1. The intensity of the quartz peak should be more than 10000 counts, at a d -spacing 3.34, 4.26, 1.82 .other minerals appearing at the exact position of the d-spacing. e.g. calcite at d- spacing of 3.03
2. Narrow peak represent non clay minerals(silicates, carbonates, oxides) while the broader peaks represent clay mineral

Phase identification (Refer to QC 03)

1. Use a quartz pattern as a quality control peak to check if it has a peak at the right position(at d-spacing of 3.34,4.25,1.80)
2. Check if one is able to search matches the other minerals phases by matching the database and the pattern standards to identify.

 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

3. Checking on the reproducibility of the patterns when scanned again .No shifting of peaks at their position, same peak with height and intensity

REFERENCES

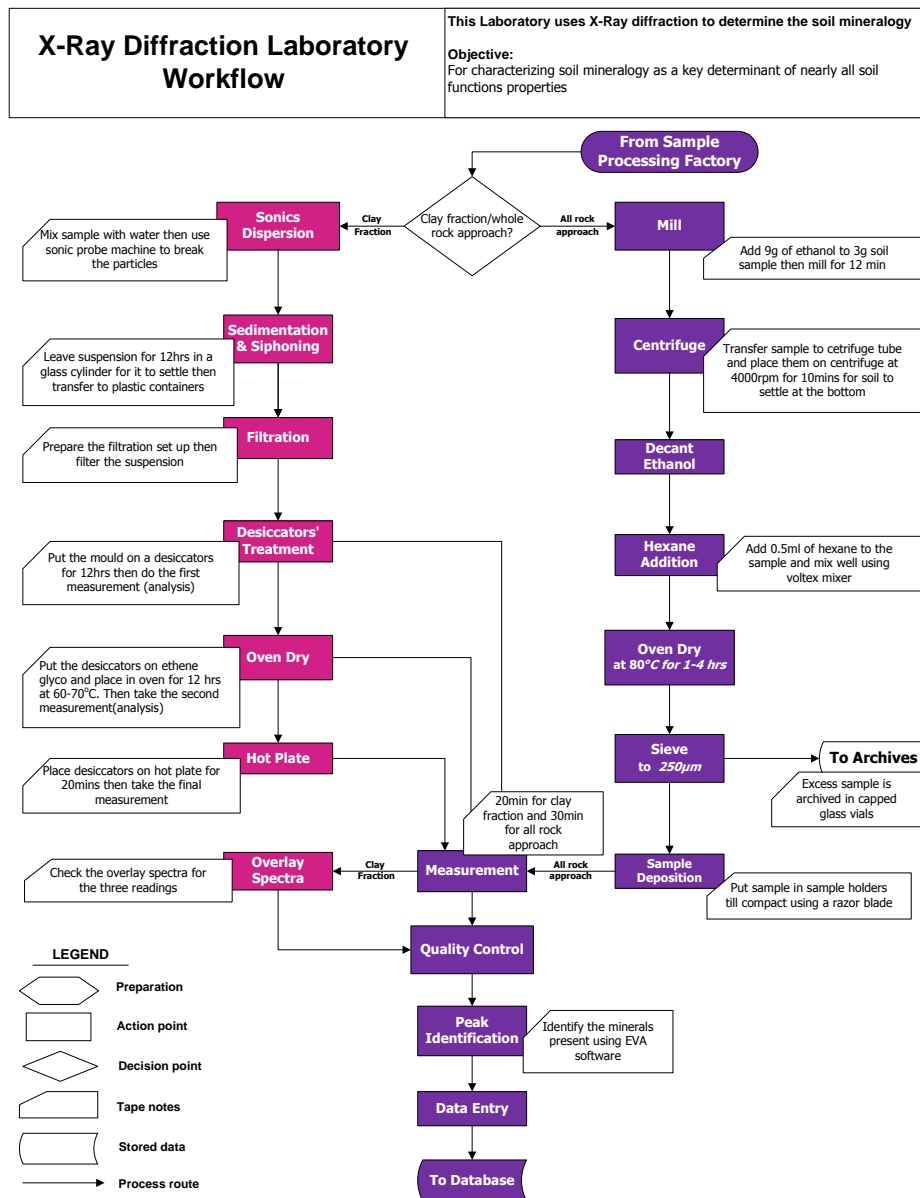
- PHASER user’s manual Doc M88-EXX141
- D2PHASER from unpacking to first measurement Doc M88-EXX155 VI
- D2 PHASER service manual Doc M88-EXX143 VI



ATTACHMENTS

- Knime software
- File maker software

ANNEX1

Work Flow



 	STANDARD OPERATING PROCEDURE	Date: July 30, 2014
	Code: METH 11V01	
	Title: Method for analyzing soil samples for mineral composition using XRD	Author: Robin Chacha

ANNEX 2

Reference	Interval	
Corundum mount Al ₂ O ₃ (commercial standard)		Checking performance and verification of the instrument. With peaks occupying same position(no shifting) Run Monthly.
Quartz Mineral SiO ₂ (Commercial standard)		Checking peak position, intensity and shape Run Monthly.