

Standard operating procedure for handling and preparation of soil samples for chemical and physical analyses



Global Soil Laboratory Network GLOSOLAN	GLOSOLAN-SOP-01	
HANDLING AND PREPARATION OF SOIL SAMPLES FOR CHEMICAL AND PHYSICAL ANALYSES	Version number : 2 Effective date : Octob	Page 1 of 11 er 28, 2019

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VERSION HISTORY

N°	Date	Description of the modification	Type of modification
01	30 July 2019	Finalisation of the draft version	Compilation of all inputs received by RESOLANs
02	28 October 2019	Final review of the SOP at the	Revision of steps in the SOP, final discussion and agreement
03	2019	3rd GLOSOLAN meeting	ililai discussion and agreement
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1. Brief introduction

Soils in the landscape can be, by nature, quite heterogeneous both across a paddock and down the profile and all due care and attention needs to be employed to ensure that the sample taken in the field truly represents what was intended. While the laboratory does not always have control over the sampling process, every attempt needs to be taken to ensure that all subsamples and aliquots for analysis are fully representative of the sample presented to the laboratory. To do this, samples are required to be homogenised as much as possible, usually by reducing and conforming the aggregate size and thorough mixing.

2. Scope and field of application

This SOP aims to provide guidance for sample handling and preparation prior to chemical and physical analysis to ensure sample material is prepared in a reproducible manner to achieve a representative portion for analysis.

3. Definitions

PSA - Particle Size Analysis;

H&S – Health and Safety;

CEC - Cation Exchange Capacity;

XRF - X-Ray Fluorescence;

ICP-MS - Inductively Coupled Plasma Mass Spectrometry

4. Responsibilities

The supervision of sample preparation procedures and the control of sample preparation administration, including consideration for the safe handling and disposal of samples by personnel and import/export restrictions on samples are the responsibility of the laboratory manager. The correct application of this procedure is the responsibility of sample handling and preparation laboratory staff identified as having authorisation in their training records.

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5. Apparatus

Equipment used for the preparation of soil samples for analysis should conform to the requirements of the analytical methodologies e.g. avoid metal contamination.

All sample preparation equipment to be operated in accordance with relevant technical procedures, ensuring traceability of samples, records and maintenance and to ensure the safety of operation.

6. Health and safety

A risk assessment should be carried and if required the necessary safety protocols, sample handling and preparation facilities and waste disposal procedures should be in place if considering receipt of samples that may present a risk to health.

All procedures for sample handling and waste management should conform to the country's relevant workplace health and safety legislation and demonstrate a commitment to creating a working environment and culture that prevents harm to all staff, contractors, volunteers and visitors. Key to this commitment is the requirement to manage risks to health and safety as far as reasonably practicable.

Where required, samples should be screened for radioactivity using a calibrated radiation monitor and results recorded. A local assessment should be undertaken to define an appropriate limit, taking into consideration the laboratory environment (e.g. ventilation, extraction). For example, at the British Geological Survey, this limit was defined for specific facilities as 10 µSvh⁻¹ at 5cm, above which there are additional safety requirements for sample handling or samples are not accepted into the laboratories.

Prior to receipt of samples, as much information about the sample origin should be gained as possible. For example, biohazards, application of chemicals to soil or contaminated land, organic, metal, asbestos pollution.

7. Procedures

7.1. Outline

Soil samples are rendered into a suitable form for chemical and physical analysis using one of the standard preparation routes given in Figure 1 of this procedure where appropriate.

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7.2. Records

Sample receipt should be carried out by the primary laboratory contact who records, using an appropriate Sample Registration Form (SRF) in paper or electronic format, the customer sample identification, preparation and analytical processes required, number and type of sub-samples needed. An electronic sample list with customer identification, should be submitted to the laboratory along with any relevant field metadata. Overall, work should be signed off by a senior member of staff with laboratory management responsibility and a unique laboratory identifier applied for traceability.

Samples received and labelled identifiers should be checked against the sample list submitted to check for anomalies, and if they occur, the customer should be informed immediately and the issue resolved before proceeding. Throughout the analytical pathway, any deviations from the required preparation and the unique identification number of equipment used should be recorded in case of later anomalies and to provide traceability.

7.3. Sample description

Soils registered for analysis are described on an 'as received' basis, prior to any sample preparation, including drying.

If required, sample colour is assessed using a Munsell Colour chart and a generic colour assigned. Soil texture is assessed using the major textural component by comparison with a set of soils of known texture designated for this purpose. Descriptions of moisture and size of sample are recorded for the benefit of the Laboratory only and are not reported to the customer.

7.4. Determination of sample preparation procedure

It is the responsibility of the Laboratory Manager to determine the preferred sample preparation procedure based on the information provided on the SRF. The choice of the appropriate procedure is dependent upon the nature of the soil sample received, and the analytical requirements of the prepared sample or sub-samples. Where appropriate, the proposed procedure is agreed with the customer before preparation commences.

The appropriate points at which to enter the flow diagram in Figure 1 will be dependent on the nominal particle size and condition of the samples received and the analytical requirements. Deviations from the following preparation procedures may be appropriate for non-routine samples.

7.5. Sub-sampling

Riffle splitting, cone quartering or random sub-sampling may be used at any point in Figure 1. The sub-sampling method used is at the request of the customer or the laboratory manager. Unique sample identifiers should follow with each sub-sample created to ensure traceability.

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7.6. Drying

Samples are processed on an 'as received' basis, unless preparation can only be carried out on dried material, or when a particular drying regime is specifically requested by the customer or the laboratory manager. In general, for volatile elements (e.g. mercury, iodine) air drying at slightly above ambient temperature is preferable to enable evaporation of moisture, with sample material determining the appropriate temperature (e.g. soil type; Cragin and Foley, 1985). At the GLOSOLAN 2019 meeting in Rome, the consensus was 35 ± 5 °C (covers a range of climates) in a ventilated area or drying oven, providing the risk of external contamination or sample cross contamination can be avoided. The drying process can be assisted by breaking up/disaggregating any large soil aggregates, and to spread the soil onto sheets or shallow trays. In some cases, such as for organic contaminants to be measured in soil samples, freeze drying will be required to ensure sample preservation.

7.7. Disaggregation

Disaggregation of the sample should be undertaken in a well ventilated space to reduce dust transmission. The soil sample will require pulverising, either mechanically or with a mortar and pestle, ideally using non-metallic equipment to minimise sample contamination. The disaggregated soil should be passed through a 2 mm stainless steel or nylon sieve and debris removed, such as plant material or stones.

Sub-sampling of the soil can be achieved using a riffle splitter or cone and quartering. A reference/archive sample may be retained and remaining portions made available for appropriate chemical analyses e.g. <2 mm for soil pH, extractable nutrients/metals, CEC, particle size analyses etc. or onward grinding for chemical methods requiring a smaller particle size e.g. dissolution for total elemental analyses.

7.8. Sieving/Grinding/Milling

Sub-sampling from <2 mm to smaller particle size can be achieved by sieving or grinding depending on the analytical test required as determined by the method SOP . For example, sieve or grind to 0.5 mm, <63 μ m by hand (agate mortar and pestle) or mechanically (ball mill) to <3 μ m for total elemental analyses (e.g. XRF, ICP-MS) or organic material content by loss-on-ignition.

7.9. Packaging

Use of suitable sufficiently strong inert packaging material, whether plastic bag/bottle or paper bag, for storage or transport of samples should maintain sample integrity e.g. protection from moisture, pests, spillage or contamination.

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7.10. Storage and disposal

Samples should be stored in a designated storage facility, with good ventilation and protection from the weather either until the analyst requires the sample or for short/long-term storage. Sample tracking should be undertaken for all samples and sub-samples through maintenance of records regarding sample transfer, preparation/analytical processes and location within the laboratories/stores and subsequent return to the client or disposal.

Prior to disposal of unused portions following analysis, the laboratory should check with the client as to whether they wish to have soil samples returned or, if for disposal, consider possible biohazard/chemical hazards for the required disposal route.

7.11. Cleaning

All equipment need to be thoroughly cleaned and inspected between samples. All workstations should be kept clean and inspected between samples, in particular between batches of samples to minimise sample cross contamination.

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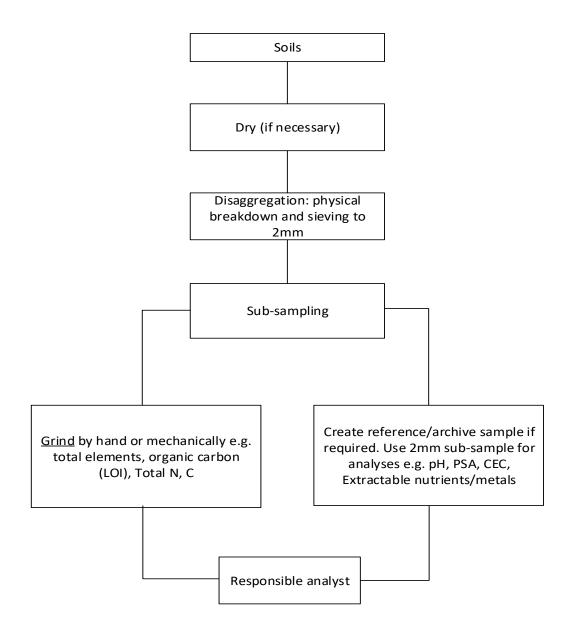


Figure 1. Flow diagram for soils requiring sample preparation for chemical and physical analysis

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Appendix II. List of authors

Main authors:

- Dr Michael Watts, British Geological Survey, United Kingdom
- Dr Charles Gowing, British Geological Survey, United Kingdom
- Ms Nopmanee Suvannang, GLOSOLAN Chair, Thailand
- Mr Rob De Hayr, Department of Environment and Science, Science Division, Chemistry Centre, Australia

Appendix III. Contributing laboratories

GLOSOLAN thanks the following laboratories for completing the GLOSOLAN form on the method and providing information on their Standard Operating Procedure for handling and preparation of soil samples for chemical and physical analyses, which were used as baseline for doing the global harmonization:

From the Asian region:

- Soil Resource Development Institute, Bangladesh
- SPAL, National Soil Services Centre, Bhutan
- ICAR-Indian Institute of Soil Science, Bhopal, India
- National Agriculture and Food Research Organization, Japan
- Quality Determination Analytical Section Department of Agriculture, Malaysia
- Department of Agricultural Research (DAR), Myanmar
- Fauji Fertilizer Company Limited, Pakistan

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- Department of Soil and Environmental Sciences, The University of Agriculture, Peshawar,
 Pakistan
- Department of Agriculture, Region 3, Phillippine
- Horticultural Crops Research and Development Institute, Department of Agriculture, Sri Lanka
- Office of Science for Land Development, Thailand

From the Pacific region:

- SAFT, USP, Samoa
- Scientific Research Organisation of Samoa, Samoa

From the Near East and North African region:

- Soil and Fertilizers, Bahrain
- Ministry of Science and Technology, Directorate of Agricultural Research, Soil and water resources Centre, **Iraq**
- Central Lab, Tunisia
- Agricultural Research & extension Authority, Renewable Natural Resources Research Center, Yemen

From the African region:

- LASEP/ITRAD, Chad
- National Soil Testing Center (NSTC), Ethiopia
- Kenya Agriculture and Livestock Research Organization (KALRO), Kenya
- University of Eldoret, Kenya
- Laboratoire des Radioisotopes, Madagascar
- Department of Agricultural Research Services, Chitedze Agricultural Research Station,
 Malawi
- National Soil And Fertilizer Laboratory, Kaduna, Nigeria
- Analytical Laboratory for Soil and Plant, Rwanda
- Institut de recherche pour le développement (IRD), Senegal
- Zambia Agriculture Research Institute, Zambia
- Soil Science University of Zimbabwe, Zimbabwe

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From the European region:

- Bundesamt für Wasserwirtschaft Institut für Kulturtechnik und Bodenwasserhaushalt,

 Austria
- University of Zagreb, Faculty of Agriculture, Soil Science Dpt., Croatia
- University of Zagreb, Faculty of Agriculture, Department of General Agronomy, Croatia
- UKZUZ, Czech Republic
- Aarhus University, AGRO University laboratory, Denmark
- Institut de recherche pour le développement (IRD), France
- Food Chain Safety Centre Non-profit Ltd., Hungary
- Latvian State Forest Research Institute "Silava", Latvia
- Laboratório de Solos e Fertilidada/Escola Superior Agrária Instituto Politécnico de Castelo Branco/Escola Superior Agrária, Portugal
- National Institute for Agricultural and Veterinary Research (INIAV), Portugal
- Central Research Institute for Soil Fertilizers and Water Resources, Turkey
- Forest Research, United Kingdom

From the Eurasian region:

 Institute of Biology of Komi Science Centre of the Ural Branch of the Russian Academy of Sciences (IB Komi SC UB RAS), Russian Federation

From Latin America:

- INTA Inst. de Suelos. Laboratory, Argentina
- Agencia de Regulación y Control Fito y Zoosanitario, AGROCALIDAD, Ecuador
- Laboratorio de Suelos DGRN-MGAP, Uruguay

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