Food and Agriculture Organization of the United Nations

Global Soil Laboratory Network:

Basic guidelines on how to produce a soil sample for proficiency testing

GLOBAL SOIL PARTNERSHIP

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Basic guidelines on how to produce a sample for proficiency testing

This document aims to provide laboratories with basic guidelines on how to prepare a sample for proficiency testing. It specifies the adequacy of resource requirements, the importance for documentation, the sample preparation requirements, the need for homogeneity and stability testing and the need for defined methods of labelling, packaging and dispatch.

Personnel requirements

Staff employed at each stage should be suitably qualified and trained to carry out the task in hand.

Facility requirements

Items of equipment used at each stage must be suitable for the purpose. Checks should be made on their suitability for use if this will impact on the quality of the final product.

Controls on the environmental conditions required for specific stages should be put in place, particularly for elimination of potential contamination and prevention of loss of sample integrity.

Sample identification

The use of specific sample should be authorized by the local manager or by a person with authority to ensure that all resource (staff and facilities) are suitable and available. The sample identifier should be unique and unambiguous.

Any potential health and safety considerations need to be established and recorded.

The quantity of material (a) to be collected and (b) to be made available to the proficiency testing (PT) scheme should be defined.

The sample should be described in such a way as to provide pertinent information to the analyst.

Documentation

The processes undertaken at each stage should be recorded and stored in a retrievable manner.

The records of preparation should be submitted to the central scheme coordinator with the prepared samples.

Physical preparation process

All stages in the process should be planned.

Sample collection

The method of sample collection should be recorded. Information on the sampling site (location, vegetation cover etc.) should be included as well as considerations on its proximity to urban and industrial centers for the purpose of identifying the potential for soil contamination. This information will be used to assess the suitability of the soil for inclusion and for ensuring provision of a contrasting range of soil types and for sourcing replicated samples if need in the future.

Pre-preparation of material

Steps should be undertaken as necessary to preliminarily screen a sample prior to processing, e.g. removal of foreign objects from the sample before packaging for transport to preparation facility.

The field sample should be free from surface organic matter (i.e. the litter layer of organic residues that are not incorporated into the mineral soil) and contaminating material.

Specific pre-preparation steps should be recorded.

Initial drying

The sample should be dried before processing.

The method of drying should be recorded.

Methods of drying may include but are not limited to air drying, if the ambient temperature is sufficiently warm and the humidity allows, oven drying to a maximum of 40˚C, or freeze drying to a maximum of 40˚C, in a well ventilated area.

Disaggregation

After the initial drying, dried samples that have formed hard clumps should be broken down using a method designed to prevent the crushing of individual grains.

The method of sample disaggregation should be recorded together with the target endpoint, and the apparatus used, including its material, e.g. wooden pestle and mortar, ceramic pestle and mortar, hardened steel mallet, fly press.

Screening/sieving

Samples may contain foreign objects that do not form part of the actual soil sample. Such samples should have such objects removed. The removal of foreign objects and otherwise unnecessary components, if undertaken, should be recorded.

The soil sample should be processed in such a way as to be representative of samples on which analytical tests are carried out (i.e. milled or unmilled as required). The sample condition does not necessarily need to be the same as would be used for a specific test in routine analysis, rather it is important that all laboratories carry out analysis using the same type of sample.

Secondary drying of screened fraction

Optional use of a secondary drying stage should be recorded, e.g. if the screened material is discernably moist.

Milling

Where abrasive milling is carried out to reduce particle size, the method used should provide a consistent method for milling all portions. Material should be milled to pass through a 0.5 mm sieve.

Processes used should be recorded, e.g. the type of mill used, milling vessel material (e.g. agate, stainless steel, wood), maximum final particle size, procedures adopted for controlling dust and potential contamination.

Homogenization

Homogenization of the material should be carried out on the whole bulk volume of material at one time.

The process for how the bulk material is homogenized and its duration should be recorded, e.g. V-blender/rotating barrel/roller blender for 2/8/24 hours.

Subdividing

Dividing the bulk into individual portions should be conducted in a way that is representative and minimizes between-sample variation.

The process for how the material is subdivided in a representative manner should be recorded, e.g. riffle splitter, rotary divider.

Packaging

Prepared materials should be stored in inert leak-proof containers that are sufficiently robust so as to withstand dispatch to global destinations. The number of portions to be made available to the scheme organizer will be agreed each Round depending upon factors like nature of the PT scheme (international/national), customs procedures/requirements, number of parameters to be determined.

Samples should be packaged into portion sizes to be agreed each round to allow for sufficient material for requested tests. The portion sizes used and how the material is packaged should be recorded, e.g. plastic sachet, plastic jar, glass jar.

Labelling

Sample labels should be clear and unambiguous and should include details of material type (e.g. silty soil).

The method of sample labelling to indicate how samples are identified should be recorded, e.g. method of labelling, information incorporated.

Sterilization

Soil samples need to conform to national importation regulations of the receiving countries. If this involves a sterilization stage, any such sterilization should not affect the parameters in the soils for which the soil will ultimately be tested.

If adopted, the process of sterilization undertaken, the dose and time applied and the stage in the preparation process should be recorded.

> *e.g. the soil samples must be irradiated or otherwise rendered biologically benign to comply with international and/or national bio-security regulations or requirements. Sterilization by irradiation should be used to provide a dose of 50 kGy, for example using a ^{60}Co source.*

Responsibility for being permitted to handle soil samples (possibly also to sterilize after use) should rest with the receiving the laboratory, which itself intends to participate in the PT scheme. Nevertheless, any steps taken for sterilization of the bulk may be helpful.

Storage

The materials should all be stored under the same conditions to preserve uniformity. Storage conditions used at the preparation facility and the duration of storage should be recorded.

Participating laboratories should be notified of any specific required storage conditions for the material.

Assessment of homogeneity and stability

The analytical and statistical methods used to assess sample homogeneity must be fit for purpose and should be recorded. Records should specify which elements/parameters have been monitored, which statistical procedures used and what the acceptance criteria are.

Sample homogeneity should be assessed using the following procedure:

- At least 10 containers of each sample are selected at random and batched according to the principles described by Thompson *et al*. (2006). Samples for homogeneity testing must be taken from prepared samples in their final form – i.e. after irradiation or disinfection.
- All homogeneity testing shall be conducted in duplicate and in random order using methods of sufficient precision for one or a selection of tests (e.g. usually Total N (LECO or NIR/MIR), pH, extractable K or extractable P). Other tests may be added or substituted as necessary.
- Results from the homogeneity testing are used to estimate the variation between samples. The homogeneity of the sample batch is initially assessed by ANOVA and calculating the variation between samples. If the standard deviation of the sample batch is less than 5 percent, samples are released for use in proficiency rounds.
- If the homogeneity of the batch does not meet acceptance criteria, homogeneity of samples may have affected assessments of laboratory performance. If this occurs, the Provider will consult with the Technical Working Group regarding actions most appropriate to each circumstance.

Typically, once dried and prepared, soil samples can be regarded as stable for a period of at least one year. However, if sample stability is an issue for certain parameters (e.g. available ammonium, available nitrate), the specific tests undertaken to demonstrate that samples are sufficiently stable for the duration of the PT analysis window (i.e. from dispatch to close of data reporting) should be recorded.

Distribution

Material should be dispatched to the central scheme coordinator in secure robust external packaging.

Packaging materials that themselves may have importation restrictions, e.g. wood, some recycled materials, should be avoided.

The method of dispatch to the central scheme coordinator for subsequent distribution should be recorded.

All external labeling should meet the required national/international requirements.

The method of shipping to the central scheme coordinator should meet the required national/international requirements.

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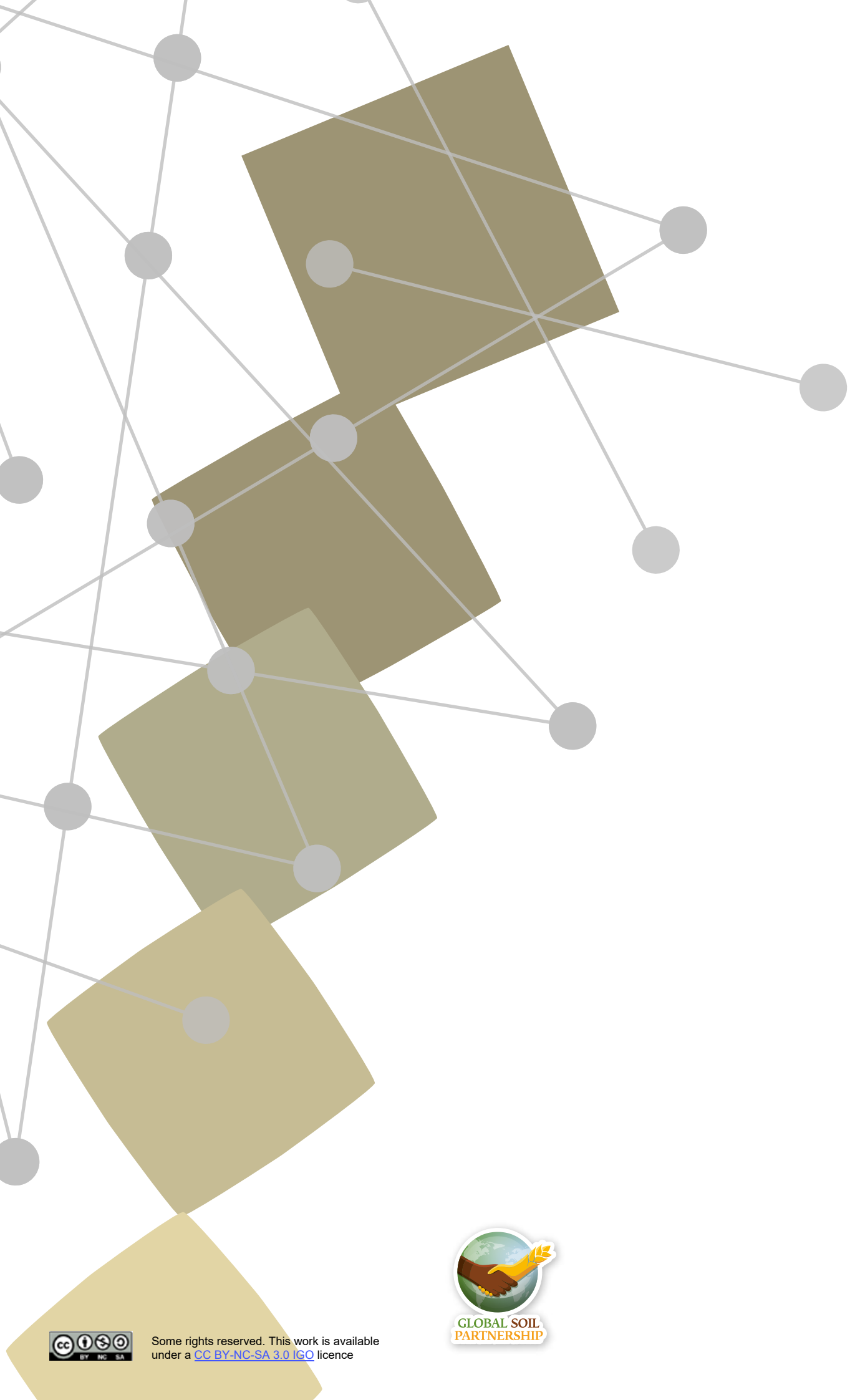

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