General instructions

How to produce a soil sample for the proficiency testing of the Global Soil Laboratory Network - GLOSOLAN -
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How to produce a soil sample for the proficiency testing of the Global Soil Laboratory Network (GLOSOLAN)

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Acknowledgements

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Instructions on how to produce a soil sample for GLOSOLAN proficiency testing

This document aims to provide sample providers with instructions on how to prepare a sample for GLOSOLAN proficiency testing. It specifies the sample preparation requirements, the methods of labelling, packaging and dispatch including the preparation of travel documents.

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 GLOSOLAN coordinator will provide sample providers with information on the type of samples to prepare depending on the parameters to analyse in the GLOSOLAN PT. The use of a specific sample should be authorized by the local manager or by a person with authority to ensure that all resources (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 GLOSOLAN coordinator will provide sample providers with information on the quantity of material (a) to be collected and (b) to be made available to the PT scheme.

The sample providers should provide the GLOSOLAN coordinator with the information contained in Annex I to provide pertinent information on the samples 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.

The central scheme coordinator is responsible for preparing all the documentation needed by the GLOSOLAN coordinator to ship the samples:

- Sample information form (see Annex I)
- Phytosanitary certificate or other types of certificate (radiation certification etc.)
  (if applicable – translated into English)

Please consult the check list in Annex II before sending the samples to FAO HQ.

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 needed 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 discernibly 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. 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.

For the GLOSOLAN PT 20xx, samples should be prepared as per Table 1.
<table>
<thead>
<tr>
<th>Soil ID</th>
<th>Sample provider</th>
<th>Contact person</th>
<th>Soil type</th>
<th>Number of labs to serve</th>
<th>Replicates (Y/N), specify number</th>
<th>Grams of soil per bag</th>
<th>Analysis to perform on soil</th>
</tr>
</thead>
<tbody>
<tr>
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</tbody>
</table>
Labelling

Soil samples should be labelled as per the following example:

<table>
<thead>
<tr>
<th>Soil ID</th>
<th>Replicate 1</th>
<th>Replicate 2</th>
<th>Replicate 3</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>GLO-A</td>
<td>GLO-A</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>GLO-B</td>
<td>GLO-B1</td>
<td>GLO-B2</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>GLO-C</td>
<td>GLO-C1</td>
<td>GLO-C2</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>GLO-D</td>
<td>GLO-D1</td>
<td>GLO-D2</td>
<td>GLO-D3</td>
<td></td>
</tr>
</tbody>
</table>

*Extra notes:* Example, for the GLOSOLAN PT 20xx all samples belonging to a same soil type (soil ID) will have the same code. Sample providers should send the samples to FAO HQ in leak- and water-proof boxes. Please use one box per each soil type.

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 minimum 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 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 in the sample information form in Annex I.

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%, 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 GLOSOLAN coordinator at FAO Headquarters in Rome should meet the required national/international requirements.

References


Annex I – Sample information form

These information should be submitted to the GLOSOLAN coordinator by the sample providers at the time samples are sent to the FAO Headquarters in Rome.

**Ring Test Sample Information**

<table>
<thead>
<tr>
<th><strong>Type of sample</strong></th>
<th>[Example: Soil]</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Origin of the samples</strong></td>
<td>[Example: Mexico]</td>
</tr>
<tr>
<td><strong>Soil sample preparation</strong></td>
<td>[Example: Soil had been prepared and tested for homogeneity (homogeneity of spectra, obtained with the NIR technique) and stability, air dry and sieve. Soil mesh size &lt;0.5 mm.]</td>
</tr>
<tr>
<td><strong>Additional treatments</strong></td>
<td>[Example: In order to ensure total sterilization, all samples were treated using Co-60 gamma radiation. Radiation certificate attached.]</td>
</tr>
<tr>
<td><strong>Weight of soil samples</strong></td>
<td>[Example: 3 samples of 100 g of soil each. Total weight: 300 g]</td>
</tr>
<tr>
<td><strong>Number of participating laboratories</strong></td>
<td>[Example: 100]</td>
</tr>
<tr>
<td><strong>Expiration date of the samples</strong></td>
<td>[Example: Not applicable as long as the plastic bag containing the sample is not opened and kept at a temperature below 40°C.]</td>
</tr>
</tbody>
</table>
Annex II – Check list

Sample providers are kindly asked to complete and send the attached check list to the GLOSOLAN coordinator at the time of shipping the samples to FAO Headquarters in Rome.

<table>
<thead>
<tr>
<th>Object</th>
<th>Tick if applicable</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Personnel requirements qualified and trained to carry out the task</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Facility suitable to carry out the task</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Samples prepared as per the instructions in this manual and those provided by the GLOSOLAN coordinator</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample information form completed and sent to the GLOSOLAN coordinator</td>
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<td></td>
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<tr>
<td>Phytosanitary certificate (if applicable - translated in English) available and sent to the GLOSOLAN coordinator</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample collection and preparation recorded – documents available upon request</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Homogenization of the samples executed as per the instruction</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Samples subdivided, coded, labeled, sterilized and packed as per the instructions.</td>
<td></td>
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</table>
The Global Soil Partnership (GSP) is a globally recognized mechanism established in 2012. Our mission is to position soils in the Global Agenda through collective action. Our key objectives are to promote Sustainable Soil Management (SSM) and improve soil governance to guarantee healthy and productive soils, and support the provision of essential ecosystem services towards food security and improved nutrition, climate change adaptation and mitigation, and sustainable development.

GLOSOLAN is a Global Soil Laboratory Network which aims to harmonize soil analysis methods and data so that soil information is comparable and interpretable across laboratories, countries and regions. Established in 2017, it facilitates networking and capacity development through cooperation and information sharing between soil laboratories with different levels of experience. Joining GLOSOLAN is a unique opportunity to invest in quality soil laboratory data for a sustainable and food secure world.

Thanks to the financial support of