



LEARNING TOXICOLOGY
THROUGH OPEN EDUCATIONAL
RESOURCES

ENVIRONMENTAL QUALITY SOILS MONITORING

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1. INTRODUCTION

The presentation below is part of the Module 6, Topic 4, as additional information related to Unit 3.

This unit / course will present:

- specific sampling and sample pre-treatment requirements for soil samples;
- standards for sampling and analytical methods available for the determination of the pollutants present in soils.

At the end of the course, students will be able to:

- describe how can be determined the quality parameters/ pollutants present in soils;
- use the information from the available European standards for sampling and analytical methods for the soil quality parameters determination.

2. SOILS SAMPLING

Samples of Interest and Representativeness of Soils Samples

The following samples are of interest for soil quality monitoring: soil collected from different depths and sediments (sometimes groundwater too). Of less interest for this unit are: litter, roots (mostly used for studies concerning pollutants up-take from soil, remediation studies), sewage sludge and solid waste materials deposited on soils.

The representativeness of soil samples should be taken into account. Soil composition differs with depth and meteorological variations.

Amount of Soils Sample

The amount of soil to be collected depends on the type of sample and on the determinations to be carried out. Thus, for full characterization of soil properties (particle size, texture, chemical composition) between 200-400 g of soil are needed. For the soil contaminants analysis, 5-100 g dry soil is required. For sediment analysis, the sampling quantity correlates with the waters one, being less than the amount of water.

Soils Sampling Tools



For soil sampling, different tools are used, such as: knives, spoons and trowels, augers (Figure 1.), tube samplers and dredges, used for sediments sampling.

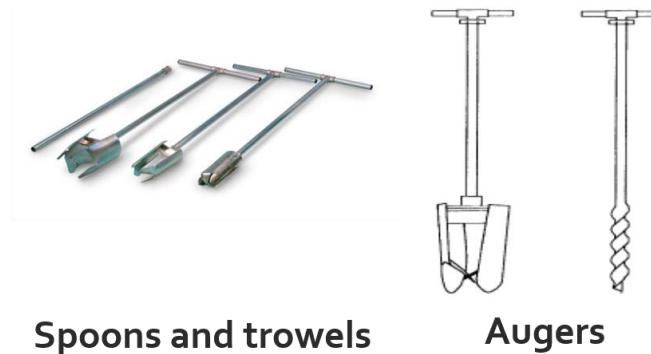


Figure 1. Tools used for soil sampling:

These tools are used for firstly removing the litter and other objects of no interest (where appropriate) than soil collection from different depths. Soil sampling must be kept under some cautions:

1. **no metallic instruments** (knives, spoons, trowels, augers) should be used when soil is collected for metals analysis;
2. **avoid introducing air** in soil sample – air may contribute to the oxidation of some compounds from soil.

European standards, from ISO series, for soil sampling are available for soil sampling, presented in Table 1.

Table 1. European standards for soil sampling (selection).

ISO standard	Standard topic
ISO 10381-2:2002	Sampling techniques
ISO/DIS 18400-104:2016	Sampling – Part 104: Strategies
ISO 18400-105/2017	Packaging, transport, storage and preservation of samples
ISO 18400-204/2017	Sampling of soil gas

Sample Preparation Techniques

Soil sample preparation techniques for pollutants analysis have already been detailed in Module 6, Topic 3, Unit 2.1.

For heavy metals analysis, the most commonly used pre-treatment techniques are mineralization and chemical dissolution (digestion). Table 2. presents several European standards, from ISO series, of sample preparation by dissolution / digestion, predominantly in acidic media.

Table 2. European standards for soil samples pre-treatment for heavy metals analysis (selection).

ISO standard	Standard topic
ISO 11466/1999	Extraction of microelements soluble in aqua regia
ISO 19730/2008	Extraction of trace elements from soil using ammonium nitrate solution
ISO 16729:2013	Digestion (microwave) of nitric acid soluble fractions of elements
ISO 14869-1/2017	Dissolution with HF and HClO ₄ (Al, Ba, Cd, Ca, Cr, Co, Cu, Fe, K, Li, Mg, Mn, Na, Ni, P, Pb, Sr, V, Zn)

For the semi-volatile (SVOC) and non-volatile (NVOC) organic compounds analysis, the pre-treatment techniques are based on extraction techniques:

1. Soxhlet (automated) extraction;
2. solvents extractions;
3. microwave assisted extraction.

Table 3. presents several European standards, from ISO series, of sample pre-treatment used for the semi-volatile (SVOC) and non-volatile (NVOC) organic compounds analysis.

Table 3. European standards for soil samples pre-treatment for organic compounds analysis (selection).

ISO standard	Standard topic
ISO 14507:2003	Pretreatment of samples for determination of organic contaminants
ISO 11464/2006	Pretreatment of soil samples for physico-chemical analysis

3. SOILS POLLUTANTS DETERMINATION

For the presentation of the soil quality parameters they were grouped into two categories, of inorganic origin or of organic one.

The inorganic indicators of soil quality are of interest:

- inorganic anions (sulfate, cyanides, sulfide, perchlorate);
- inorganic cations (Cd, Cr, Co, Cu, Pb, Mn, Ni, Zn);
- other trace elements soluble in aqua regia or nitric acid;
- compounds of the nutrient category: phosphorus, nitric-N, ammoniacal-N, and total soluble nitrogen.

Organic pollutants present in soils and of interest for soil monitoring are:

- volatile organic compounds (VOCs) such as ethers, halogenated hydrocarbons, aromatic hydrocarbons, phenols and chlorophenols;
- pesticides;
- linear alkylbenzene sulfonate (LASs);
- polycyclic aromatic hydrocarbons (PAHs);
- polychlorinated biphenyls (PCBs);
- dioxins and furans.

A selection of European standard methods, from ISO series, available for the determination of the above-mentioned inorganic compounds is given in Table 4. These standards are based on gravimetric, titrimetric, spectrometric, electrochemical and chromatographic methods.

Table 4. European standard methods for inorganic compounds analysis from soil (selection).

Soil quality indicator	Standard	Analytical method
phosphorus	ISO 11263/1994	spectrometry
carbonate	ISO 10693/1995	titrimetric
water-soluble and acid-soluble sulphate	ISO 11048/1995	gravimetric
total nitrogen	ISO 11261/1995	titrimetric
electrical conductivity	ISO 11265+A1/1998	electrochemically
dry matter and water content	ISO 11465/1998	gravimetric

Cd, Cr, Co, Cu, Pb, Mn, Ni, Zn (extract in aqua regia)	ISO 11047/1999	atomic absorption spectrometry
total sulphur	ISO 15178/2000	dry combustion
nitric-N, ammoniacal-N, total soluble nitrogen	ISO 14255/2005	segmented flow analysis
trace elements	ISO 22036/2008	inductively coupled plasma – atomic emission spectrometry
total cyanides	ISO 11262/2011	titrimetric
trace elements in aqua regia and nitric acid digests	ISO/TS 17073.2013	atomic absorption spectrometry
pH	ISO 10390/2015	electrochemically
perchlorate	ISO/DIS 20295/2016	ion chromatograph

For the determination of the previously presented organic compounds, as parameters for soil quality, a selection of the European methods, from ISO series is given in Table 5. Their determination is based in particular on spectrometric methods, gas chromatography and liquid chromatography.

Table 5. European standard methods for organic compounds analysis from soil (selection).

Soil quality indicator	Standard	Analytical method
total organic carbon (TOC)	ISO 14235/2000	spectrophotometry
organochlorine pesticides and polychlorinated biphenyls (PCBs)	ISO 10382/2002	gas-chromatography (GC)
herbicides	ISO 11264/2005	high-performance liquid chromatography (HPLC)
selected chlorophenols	ISO 14154/2005	gas-chromatography (GC)

polycyclic aromatic hydrocarbons (PAHs)	ISO 18287:2006	gas chromatographic-mass spectrometry (GC-MS)
organotin compounds	ISO/DIS 23161/2009	gas-chromatography (GC)
linear alkylbenzene sulfonate (LASs)	ISO/TS 13896:2012	high-performance liquid chromatography (HPLC)
polychlorinated biphenyls (PCBs)	ISO 13876:2013	gas chromatographic-mass spectrometry (GC-MS)
dioxins and furans and dioxin-like polychlorinated biphenyls	ISO 13914:2013	gas chromatographic-mass spectrometry (GC-MS)
polycyclic aromatic hydrocarbons (PAHs)	ISO 13859:2014	gas chromatography (GC) and high-performance liquid chromatography (HPLC)
selected phenols and chlorophenols	ISO/TS 17182/2014	gas chromatographic-mass spectrometry (GC-MS)
volatile aromatic hydrocarbons and halogenated hydrocarbons, naphthalene	ISO 15009:2016	gas-chromatography (GC)
volatile aromatic hydrocarbons and halogenated hydrocarbons, ethers	ISO 22155/2016	gas-chromatography (GC)

All these standard methods are subject of periodically reviewing, providing the analysts with the most recent editions.

Apart from the above-mentioned soil indicators, other pollutants might be of interest for soil quality, especially for research based monitoring. They are also from inorganic or organic origin, dissolved or insoluble substances in soil solution,

with low or up to high molecular weights, like the components of humic and fluvic acids are.

Similar to air and water analyses performed in laboratories, besides the standard analytical methods, used according to the regulations for certain soil quality parameters, other non-standard analytical methods for any pollutant of interest are also available and accepted in the laboratory practice. Both categories of analytical methods (standard and non-standard) are subject to method validation and laboratory accreditation, according to the ISO/IEC 17025:2005, with the recent revision, ISO/IEC 17025:2017.

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