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**Kakovost tal - Določevanje policikličnih aromatskih ogljikovodikov (PAH) - Plinska kromatografija z masno selektivnim detektorjem (GC-MS)**

Soil quality - Determination of polycyclic aromatic hydrocarbons (PAH) - Gas chromatographic method with mass spectrometric detection (GC-MS)

Qualité du sol - Dosage des hydrocarbures aromatiques polycycliques (HAP) - Méthode par chromatographie en phase gazeuse avec détection par spectrométrie de masse (GC-SM)

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# INTERNATIONAL STANDARD

# ISO 18287

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## Soil quality — Determination of polycyclic aromatic hydrocarbons (PAH) — Gas chromatographic method with mass spectrometric detection (GC-MS)

*Qualité du sol — Dosage des hydrocarbures aromatiques polycycliques (HAP) — Méthode par chromatographie en phase gazeuse avec détection par spectrométrie de masse (CG-SM)*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18287 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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## Introduction

This International Standard is principally based on the extraction method described in ISO 13877. It is modified for the use of gas-chromatography with mass spectrometric detection and is applicable for different PAH pollution levels of soils.

Two alternative extraction methods, A and B, are described in this International Standard.

Method A (two-step method): Extraction of the field-moist soil sample with acetone and petroleum ether, followed by the removal of acetone by washing the extract with water as prescribed in ISO 13877.

Method B (one-step method or on-line method): Extraction of the field-moist soil sample with a mixture of acetone, petroleum ether and water in the presence of sodium chloride. This method is preferred for soil samples with a high content of organic matrix.

Experience has shown that these two methods are applicable with comparable results to less as well as highly polluted soils

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# Soil quality — Determination of polycyclic aromatic hydrocarbons (PAH) — Gas chromatographic method with mass spectrometric detection (GC-MS)

**SAFETY PRECAUTIONS** — Certain PAH are highly carcinogenic and must be handled with extreme care. Contact of solid materials, solvent extracts and solutions of standard PAH with the body must not be allowed to occur. PAH may co-distil with solvent and become deposited outside of stoppered bottles; all containers containing solutions of PAH in solvent must therefore always be handled using gloves which are solvent resistant and preferably disposable. PAH contamination of vessels may be detected by irradiation with 366 nm UV light. Vessels containing PAH solutions should be stored standing in beakers to contain any spillage in the case of breakage.

Solid PAH are most dangerous and give rise to a dust hazard due to their crystals becoming electrostatically charged. These materials must only be handled where proper facilities are available (e.g. adequate fume hoods, protective clothing, dust masks). It is strongly advised that standard solutions be prepared centrally in suitably equipped laboratories or are purchased from suppliers specialized in their preparation.

Solvent solutions containing PAH must be disposed of in a manner approved for the disposal of toxic wastes.

## 1 Scope

This International Standard specifies the quantitative determination of 16 polycyclic aromatic hydrocarbons (PAH) according to the priority list of the Environmental Protection Agency, USA (EPA, 1982). This International Standard is applicable to all types of soil (field-moist or chemically dried samples), covering a wide range of PAH contamination levels.

Under the conditions specified in this International Standard, a lower limit of application of 0,01 mg/kg (expressed as dry matter) can be ensured for each individual PAH.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 14507:2003, *Soil quality — Pretreatment of samples for determination of organic contaminants*

ISO 10381-8, *Soil quality — Sampling — Part 8: Guidance on sampling of stockpiles*

ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis*

## ISO 18287:2006(E)

### 3 Principle

The extraction is carried out using acetone and petroleum ether. Acetone is an efficient extractant, in particular because it is able to break down soil aggregates. Petroleum ether increases the efficiency of the extraction and is necessary as solvent in the subsequent concentration procedure. An extraction method for PAH should at least use 50 ml of acetone and 50 ml of petroleum ether (for a chemically dried sample). If a wet sample is used, the amount of acetone should be increased to at least 100 ml. Different ways of extraction can lead to the same results provided that the above recommendations are observed. If the sample contains a large amount of water, or if water has been added, sodium chloride (NaCl) should be added to improve the efficiency of the extraction.

Two alternative extraction methods, A and B, are described in this International Standard.

**Method A (two-step method):** A field moist soil sample is extracted two times with acetone, then petroleum ether is added to the acetone extract. The extract is washed two times with water. The organic layer is dried with anhydrous sodium sulfate.

**Method B (one-step method or on-line method):** A field-moist soil sample is extracted with a fixed ratio of a mixture of acetone, petroleum ether, water and sodium chloride. An aliquot of the organic layer is dried with anhydrous sodium sulfate.

If necessary, a clean-up step using adsorption chromatography on silica gel, as well as a concentration step, may be included.

The extract is then analysed by capillary gas-chromatography. The identification and the quantification of the PAH is made with mass spectrometric detection, using appropriate deuterated PAH as internal standards.

### 4 Reagents

All reagents used shall be of recognized analytical grade and free from PAH. A blank determination shall be carried out to ensure that the reagents do not contain PAH in detectable concentrations.

- 4.1 **Water**, use only distilled water or water of equivalent purity.
- 4.2 **Acetone**, for residue analysis.
- 4.3 **Petroleum ether**, for residue analysis (boiling range 40 °C to 60 °C).
- 4.4 **Cyclohexane**, for residue analysis.
- 4.5 **Isooctane**, for residue analysis.
- 4.6 **Sodium chloride**, anhydrous.
- 4.7 **Sodium sulfate**, anhydrous, for residue analysis.
- 4.8 **Magnesium perchlorate** or **suitable drying agent**.
- 4.9 **Silica gel 60**, for column chromatography, particle size 63 µm to 200 µm.
- 4.10 **Silica gel 60**, water content:  $w(\text{H}_2\text{O}) = 10 \%$  (mass fraction).

Use silica gel 60 (4.9), heated for at least 3 h at 450 °C, cooled down in a desiccator (5.13) and stored with magnesium perchlorate or a suitable drying agent (4.8). Before use, heat for at least 5 h at 130 °C in a drying oven (5.14). Then allow to cool in a desiccator and add 10 % water (mass fraction) in a flask. Shake intensively by hand for 5 min until all lumps have disappeared and then for 2 h in a shaking machine (5.3).