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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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Published in Switzerland

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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.

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## 1 Scope

ISO 18287:2006  
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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*

### 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.

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### 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).



Store the deactivated silica gel in the absence of air and use it for a maximum of one week.

#### 4.11 Reference substances, internal standards

Choose as internal standards substances whose physical and chemical properties (such as extraction behaviour, retention time) are similar to those of the compounds to be analysed. Deuterated PAH should be used as internal standards for the GC-MS method for the evaluation of results. Verify the stability of the internal standards regularly. Table 1 lists native and deuterated PAH.<sup>1)</sup>

**Table 1 — Native PAH and deuterated PAH**

PAH reference substances	CAS No.	Deuterated PAH internal standard substances
Naphthalene	91-20-3	Naphthalene-d <sub>8</sub>
Acenaphthene	83-32-9	Acenaphthene-d <sub>10</sub>
Acenaphthylene	208-96-8	
Fluorene	86-73-7	
Anthracene	120-12-7	
Phenanthrene	85-01-8	Phenanthrene-d <sub>8</sub>
Fluoranthene	206-44-0	
Pyrene	129-00-0	
Benz[a]anthracene	56-55-3	Benz[a]anthracene-d <sub>12</sub>
Chrysene	218-01-9	
Benzo[b]fluoranthene	205-99-2	Benzo[e]pyrene-d <sub>12</sub>
Benzo[k]fluoranthene	207-08-9	
Benzo[a]pyrene	50-32-8	
Indeno[1,2,3-cd]pyrene	193-39-5	Perylene-d <sub>12</sub>
Dibenz[ah]anthracene	53-70-3	
Benzo[ghi]perylene	191-24-2	

#### 4.12 Standard solutions for GC-MS

If a commercially available certified standard stock solution of the relevant PAH is used, the calibration solutions with different levels of PAH concentrations are prepared by diluting an appropriate volume of stock solution with cyclohexane (4.4) in volumetric flasks (5.8).

##### 4.12.1 Single-substance stock solution

If no certified standard solutions are used, prepare single-substance stock solutions by weighing approximately 10 mg of each of the standards “native PAH” and “deuterated PAH” (see Table 1) to the

1) Certified solutions of PAH and single solid PAH substances with certified purity are available from a limited number of suppliers, e.g. Institute for Reference Materials and Measurements (IRMM), B-2440 Geel, Belgium. National Institute of Science and Technology, Office of Standard Ref. Data, Washington D.C. 20234 USA, or from other commercial providers. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these suppliers.

nearest 0,1 mg in a 50 ml volumetric flask, dissolving them in cyclohexane (4.4) and diluting to the mark with cyclohexane (200 µg/ml).

Store the single-substance stock solutions in a dark place at a temperature of about –15 °C to –18 °C.

#### 4.12.2 Stock solution containing mixed PAH standards

Transfer 1 ml to 5 ml (adjust the volume for individual components according to their occurrence in soil) of the single-substance stock solution (4.12.1) to a 100 ml volumetric flask and dilute to the mark with cyclohexane (2 µg/ml to 10 µg/ml).

#### 4.12.3 Stock solution containing mixed internal standards

Transfer 5 ml each of the single-substance stock solutions (4.12.1) of the deuterated PAH into a 100 ml volumetric flask and dilute to the mark with cyclohexane (10 µg/ml).

Store the diluted standard solutions 4.12.2 and 4.12.3 at about 4 °C, protected from light and evaporation.

NOTE Stock solutions are stable for about 1 year.

#### 4.12.4 Calibration standard solutions

Prepare a series of calibration standards over a suitable range (e.g. 0,20 µg/ml to 5,0 µg/ml) by transferring 1 ml to 5 ml of the stock solution containing mixed PAH standards (4.12.2) and 1 ml of the mixed internal standard solution (4.12.3) to a 10 ml volumetric flask and dilute to the mark with cyclohexane. Each of the calibration standards contains nominally 1,0 µg/ml of each of the deuterated PAH.

NOTE Calibration standard solutions are stable for about 1 year.

## 5 Apparatus

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Usual laboratory glassware.

All glassware shall be thoroughly cleaned, preferably in a dishwasher using a customary cleaning procedure, followed by rinsing with acetone and subsequently with petroleum ether. Heating of glassware at 450 °C for 2 h is also allowed.

**5.1 Brown glass sample containers**, of nominal capacity 1 l, with screw caps provided with an inlay of polytetrafluoroethylene (PTFE).

**5.2 Sample divider**.

**5.3 Shaking machine**, with horizontal movement (200 to 300 movements per minute).

**5.4 Concentration apparatus**, Kuderna Danish, or **rotary evaporator**.

**5.5 Water bath**, adjustable up to 100 °C.

**5.6 Stainless steel dish**.

**5.7 Conical flasks**, of 100 ml, 500 ml and 1 000 ml capacity, with screw caps provided with an inlay of polytetrafluoroethylene (PTFE).

**5.8 Volumetric flasks**, of 10 ml, 50 ml and 100 ml nominal capacity.

**5.9 Separating funnels**, with a capacity of 250 ml, 500 ml and 1 000 ml.

**5.10 Pipettes**, of 1 ml, 2 ml, 5 ml and 10 ml, and **Pasteur pipettes**.

- 5.11 Syringe**, of 100 µl, graduated in microlitres.
- 5.12 Centrifuge**, with **tubes** of 100 ml capacity, with screw caps.
- 5.13 Desiccator**.
- 5.14 Drying oven**, adjustable up to 150 °C.
- 5.15 Glass wool**, silanized.
- 5.16 Chromatography column**, with storage tank at the top and PTFE stopcock at the bottom, with length 250 mm and internal diameter 10 mm.
- 5.17 Gas chromatograph**, equipped with a suitable injection system, capillary column and a mass spectrometric detector (GC-MS).
- 5.18 Capillary columns**: Use a fused silica capillary column with a length of 30 m and internal diameter of 0,22 mm, coated with a film of cross-linked non-polar polysiloxane or slightly polar modified polysiloxane with an efficient separation. The column shall be suitable to separate benzo[a]pyrene and benzo[e]pyrene.

## 6 Sampling, preservation and pretreatment

Carry out sampling in accordance with ISO 10381-1 and ISO 10381-8. Store field-moist samples for no longer than 7 days in suitable containers in a dark place at a temperature below 10 °C (refrigerator). If necessary, use the procedure for the treatment of large samples described in ISO 10381-8 to reduce the size of the sample to 1 kg. Pretreat the obtained sample in accordance with ISO 14507:2003 (use procedure 8.4 to obtain a pretreated field-moist sample and procedure 8.3 to obtain a chemically dried sample). If freeze-drying of the sample is prescribed, follow ISO 16720.

Determine and record mass fractions less than 2 mm and greater than 2 mm.

Determine the content of the dry matter in the field-moist soil in accordance with ISO 11465.

## 7 Procedure

### 7.1 Extraction procedure

**7.1.1 Method A (two-step method)**: Weigh 10 g to 25 g of the field-moist sample and place it in a conical flask (5.7) or a screw cap centrifuge tube of 100 ml capacity (5.12). After adding 1 ml of mixed internal standard solution (4.12.3) (10 µg of each deuterated PAH) and subsequently 50 ml of acetone (4.2) to the test sample, close the conical flask with a screw cap provided with an inlay of polytetrafluoroethylene (PTFE) and extract by shaking thoroughly for 1 h on a shaking machine (5.3). Add 50 ml of petroleum ether (4.3), shake again, decant then shake with another portion of 50 ml of petroleum ether. After allowing settling, decant the supernatant. Combine the extracts and remove the acetone and other polar components by shaking twice with 400 ml portions of water (4.1). Discard the water.

Dry the organic layer over anhydrous sodium sulfate (4.7), transfer the dried extract to the concentration apparatus (5.4) and add 100 µl of isooctane (4.5) as a keeper.

Concentrate, on a water bath (5.5) set at 40 °C, to about 10 ml with a concentration apparatus (5.4) under reduced pressure. The last step of concentration can be done by using a gentle stream of nitrogen at room temperature. The solution, which is now suitable for GC-MS analysis, contains nominally 1 µg/ml of each of the deuterated PAH.

**NOTE** For highly polluted soil samples, clean-up steps and concentration steps may not be necessary.