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*.

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

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 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*

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: $w(H_2O)$ = 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 desactivated 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.[1)]

Table 1 — Native PAH and deuterated PAH

PAH reference substances	CAS No.	Deuterated PAH internal standard substances
Naphthalene	91-20-3	Naphthalene-d_8
Acenaphthene	83-32-9	Acenaphthene-d_{10}
Acenaphthylene	208-96-8	
Fluorene	86-73-7	
Anthracene	120-12-7	
Phenanthrene	85-01-8	Phenanthrene-d_8
Fluoranthene	206-44-0	
Pyrene	129-00-0	
Benz[*a*]anthracene	56-55-3	Benz[*a*]anthracene-d_{12}
Chrysene	218-01-9	
Benzo[*b*]fluoranthene	205-99-2	Benzo[*e*]pyrene-d_{12}
Benzo[*k*]fluoranthene	207-08-9	
Benzo[*a*]pyrene	50-32-8	
Indeno[1,2,3-*cd*]pyrene	193-39-5	Perylene-d_{12}
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 a 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

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 seperate 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.

Other extraction techniques, such as ultrasonic extraction, microwave-assisted extraction or pressurized fluid extraction (PFE), may be suitable. However if using other extraction techniques, the comparability to the method described in this International Standard shall be proven.

7.1.2 Method B (one-step method or on-line method): Weigh 10 g to 25 g of the field-moist sample and place it in a 500 ml conical flask (5.7). After adding 1 ml of the mixed internal standard solution (4.12.3) (10 µg of each deuterated PAH) and subsequently 50 ml of water (4.1), 40 g of sodium chloride (4.6), 100 ml of acetone (4.2) and 50 ml of petroleum ether (4.3) to the test sample, close the flask with the screw cap provided with an inlay of polytetrafluoroethylene (PTFE) and extract by shaking thoroughly on a shaking machine (5.2) for at least 6 h. After allowing for settling, decant the supernatant organic layer into a conical flask and dry the extract over anhydrous sulfate (4.7) for 1 h. A centrifuge (5.12) may be used for the separation of the organic layer. Transfer an aliquot (e.g. 30 ml) of 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 2 ml with a concentration apparatus (5.4) under reduced pressure. The last step of concentration may 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.

7.2 Clean-up procedure

Clean-up shall be used if compounds are present that can interfere with the PAH of interest. If no or negligible interfering substances are present, clean-up is not necessary and the clean-up procedure is optional. Depending on the substances to be removed, different clean-up procedures may be used. Before application of the clean-up to real samples, the laboratory shall ensure that recoveries after clean-up are at least 80 % for all relevant PAH (including internal standards).

As an example, clean-up of the extract with silica gel adsorption chromatography may be carried out as follows.

Prepare an adsorption column by placing a small plug of glass wool (5.15) at the bottom of the chromatographic tube and packing it dry with 4 g of silica gel (4.10). Add about 1 cm of anhydrous sodium sulfate (4.7) to the top of the column. To condition the column, eluate it with 10 ml of petroleum ether (4.3). As soon as the elution solvent reaches the top of the column packing, transfer the concentrated extract (2 ml) to the top of the column with a Pasteur pipette (5.10). Rinse the concentrator twice with 1 ml of the elution solvent and transfer also to the top of the chromatographic column. Eluate with approximately 70 ml of petroleum ether (4.3) and collect the eluate in a point-shaped, calibrated test tube. Add 100 µl of isooctane (4.5) and concentrate the solution at room temperature with a gentle stream of nitrogen until the volume is reduced to about 0,5 ml. Add an appropriate amount of cyclohexane (4.4) to obtain a defined volume (e.g. 2 ml) of the final solution. This final solution, which is suitable for GC-MS analysis, contains nominally 1 µg/ml of each of the deuterated PAH as the internal standard.

NOTE 1 Commercially available cartridges can also be used.

NOTE 2 Clean-up of the extract using aluminium oxide is effective if more polar disturbing compounds need to be removed.

7.3 Gas-chromatographic analysis with mass spectrometric detection

7.3.1 Setting up the gas chromatograph

Set up the gas chromatograph (5.17) in such a way that optimum separation of the PAH is achieved. For example, optimize the gas chromatograph starting from the following conditions:

Separation column: Capillary column, non-polar to medium-polar stationary phase, film thickness 0,25 µm, length 30 m, internal diameter 0,25 mm

Oven temperature programme: 60 °C for 2 min
30 °C/min up to 120 °C
5 °C/min up to 300 °C
300 °C for 15 min

Injector temperature: 260 °C

Splitless injection: 1 µl, keep the split closed 1,8 min

Carrier gas: helium, at 0,8 ml/min to 1 ml/min

7.3.2 Mass spectrometric (MS) conditions

MS interface temperature: 295 °C

Filament on: 6 min

Selection of mass numbers: The following mass numbers (see Table 2) may be used for the quantitative analysis in selected ion monitoring mode.

Table 2 — Mass numbers of polycyclic aromatic hydrocarbons

Compound	Mass number Unit of atomic mass (amu)
Naphthalene-d_8 (internal standard)	136
Naphthalene	128 (129)
Acenaphthylene	152 (151)
Acenaphthene-d_{10} (internal standard)	164
Acenaphthene	154 (153)
Fluorene	166 (165)
Phenanthrene-d_{10} (internal standard)	188
Phenanthrene	178 (179)
Anthracene-d_{10} (internal standard)	188
Anthracene	178 (89)
Fluoranthene-d_{10} (internal standard)	212
Fluoranthene	202 (101)
Pyrene	202 (101)
Benz[*a*]anthracene-d_{12} (internal standard)	240
Benz[*a*]anthracene	228 (114)
Chrysene	228 (114)
Benzo[*b*]fluoranthene-d_{12} (internal standard)	264
Benzo[*b*]fluoranthene	252 (253)
Benzo[*k*]fluoranthene	252 (253)
Benzo[*a*]pyrene	252 (253)
Benzo[*e*]pyrene-d_{12} (internal standard)	264
Perylene-d_{12} (internal standard)	264
Indeno[1,2,3-*c,d*]pyrene	276 (138)
Dibenz[*ah*]anthracene	278 (139)
Benzo[*ghi*]perylene	276 (138)

7.3.3 Calibration of the method using an internal standard

This is an independent method for the determination of the mass concentrations and is not influenced by injection errors, the volume of water present in the sample or matrix effects in the sample, provided that recovery of the compounds to be analysed is about equal to that of the standard.

Add a specific mass of the internal standard (10 µg) to the soil test sample as to the calibration solutions. The mass concentration of the standard shall be the same for calibration and analysis. Run the GC-MS analysis with the calibration solutions, prepared as described in 4.12.4. Calculate the relative response ratio for the native PAH and the deuterated PAH, after obtaining a calibration curve by plotting the ratio of the mass concentrations against the ratio of the peak areas (or peak heights) using Equation (1):

$$\frac{A_{\mathrm{n}}}{A_{\mathrm{d}}} = s \cdot \frac{\rho_{\mathrm{n}}}{\rho_{\mathrm{d}}} + b \qquad (1)$$

where

A_{n} is the measured response of the native PAH, represented by peak area);

A_{d} is the measured response of the deuterated PAH, represented by peak area;

s is the slope of the calibration function;

ρ_{n} is the mass concentration of the native PAH in the calibration solution, in micrograms per millilitre (µg/ml);

ρ_{d} is the mass concentration of the deuterated PAH in the calibration solution, in micrograms per millilitre (µg/ml);

b is the intercept of the calibration curve with the ordinate.

8 Evaluation

Calculate the mass fraction of the individual PAH from the multipoint calibration of the total method by using Equation (2):

$$w_{\mathrm{n}} = \frac{(A_{\mathrm{n}} / A_{\mathrm{d}}) - b}{s \cdot m \cdot w_{\mathrm{s}}} \cdot \rho_{\mathrm{d}} \cdot f \cdot V \qquad (2)$$

where

w_{n} is the content of the individual PAH found in the sample, in milligrams per kilogram (mg/kg), on the basis of the dry substance;

A_{d} is the measured response of the deuterated PAH in the sample extract;

A_{n} is the measured response of the native PAH in the sample extract;

ρ_{d} is the mass concentration of the deuterated PAH in the sample extract, in micrograms per millilitre (µg/ml);

m is the mass of the soil test sample used for extraction, in grams (g);

w_{s} is the content of the dry substance in the field-moist sample, determined according to ISO 11465, in percent mass fraction (%);

f is the ratio of the total organic solvent volume used for extraction (e.g. 150 ml) to that of the aliquot (e.g. 30 ml) used for the analysis, according to method B (f = 5 in this case);

V is the volume of the final solution, in millilitres (ml);

s is the slope of the calibration function;

b is the intercept of the calibration curve with the ordinate.

The result shall be expressed in milligrams per kilogram (mg/kg) of dry soil, rounded to one significant figure after the decimal point.

9 Performance characteristics

The performance characteristics are given in Annex B.

10 Quality assurance

With each series of soil samples to be analysed, a blank measurement and the recovery measurement of the total method shall be carried out with a sandy soil or other suitable soil which is spiked with PAH.

11 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) complete identification of the sample;

c) a reference to the extraction procedure used (method A or method B);

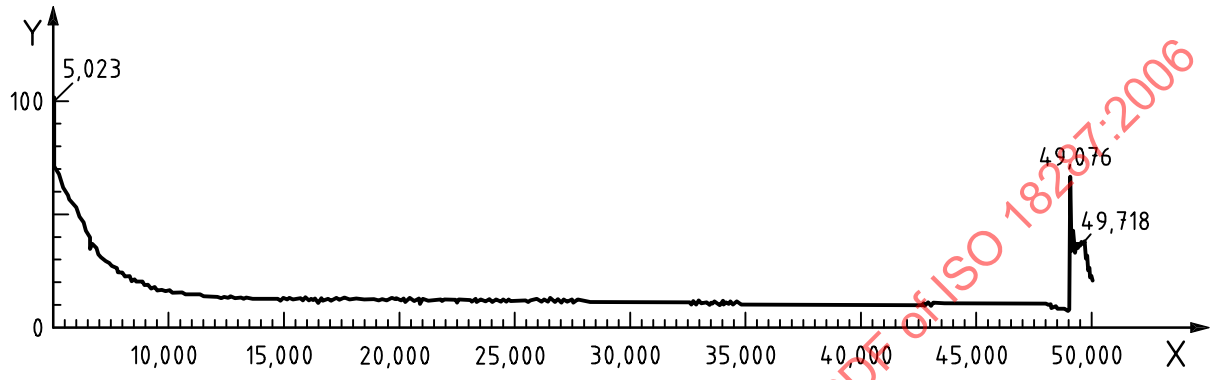
d) the results of the determination;

e) any details not specified this International Standard or which are optional, as well as any factor which may have affected the results.

Annex A
(informative)

Examples of typical GC-MS chromatograms and instrument conditions

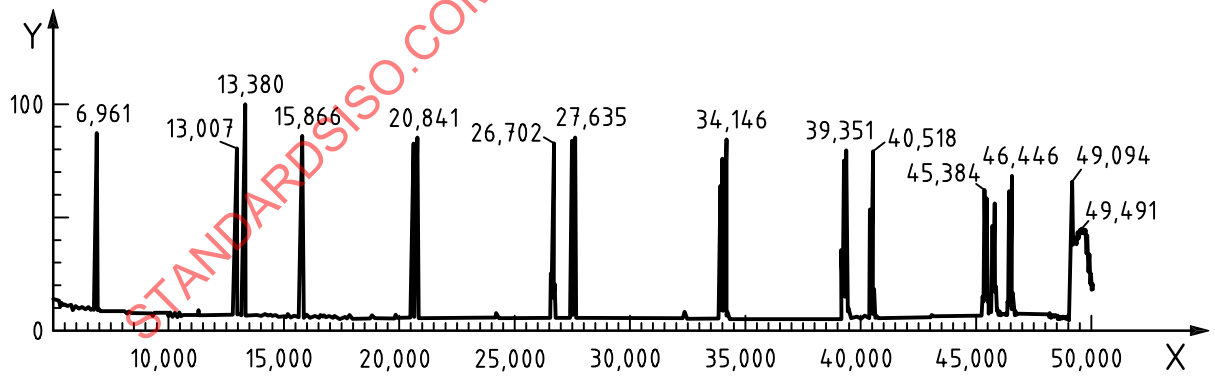
The data for Figures A.1 to A.3 are taken from Reference [5].



Key

X is the retention time, min
Y is the peak height, %

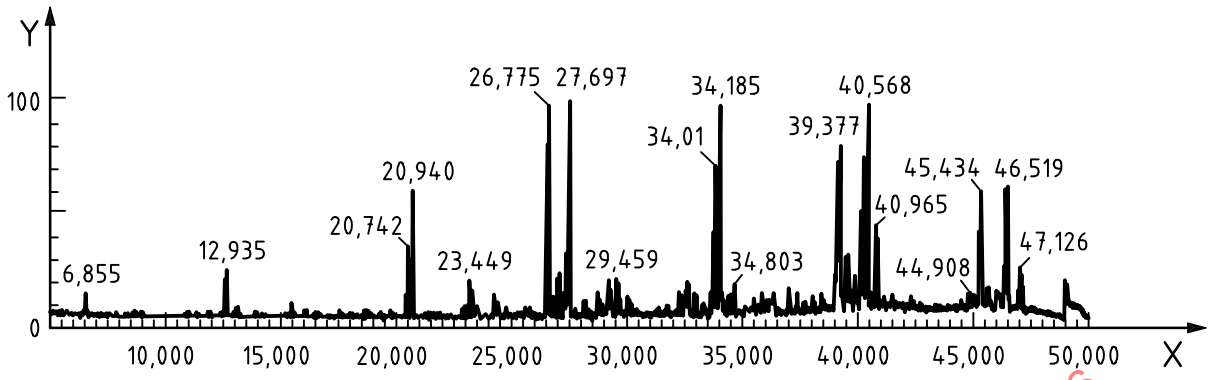
Figure A.1 — Total ion chromatogram of the blank of the total method
(GC and MS conditions are stated below)



Key

X is the retention time, min
Y is the peak height, %

Figure A.2 — Total ion chromatogram of a multi-component calibration standard in cyclohexane, approximately 10 ng/µl (20 ng/injection)
(GC and MS conditions are stated below)



Key

X is the retention time, min

Y is the peak height, %

Figure A.3 — Total ion chromatogram of a real-life soil sample (Method B: 10 ml aliquot from 150 ml extraction solvent, concentrated to 1 ml)

Table A.1 — PAH with the indicative retention times (according to Figure A.3)

Compound	Acronym	Formula	Molar mass	Retention time min
Naphthalene	NP	$C_{10}H_8$	128,18	6,96
Acenaphthylene	ACY	$C_{12}H_8$	152,20	13,00
Acenaphthene	ACE	$C_{12}H_{10}$	154,20	13,38
Fluorene	FLN	$C_{13}H_{10}$	166,23	15,87
Phenanthrene	PHE	$C_{14}H_{10}$	178,24	20,65
Anthracene	ANT	$C_{14}H_{10}$	178,24	20,84
Fluoranthene	FLU	$C_{16}H_{10}$	202,26	26,70
Pyrene	PYR	$C_{16}H_{10}$	202,26	27,63
Benz[*a*]anthracene	BaA	$C_{18}H_{12}$	228,30	33,99
Chrysene	CHR	$C_{18}H_{12}$	228,30	34,15
Benzo[*b*]fluoranthene	BbF	$C_{20}H_{12}$	252,32	39,22
Benzo[*k*]fluoranthene	BkF	$C_{20}H_{12}$	252,32	39,35
Benzo[*a*]pyrene	BaP	$C_{20}H_{12}$	252,32	40,52
Indeno[1,2,3-*cd*]pyrene	IcdP	$C_{22}H_{12}$	276,34	46,45
Dibenz[*a,h*]anthracene	DBahA	$C_{22}H_{14}$	278,35	45,38
Benzo[*ghi*]perylene	BghiP	$C_{22}H_{12}$	276,34	45,72

GC conditions:	CARLO ERBA MEGA 5300
Separation column:	SPB-1701, SUPELCO; 30 m; 0,32 mm; inner diameter; film thickness 0,25 µm
Carrier gas:	Helium, 5,0 70 kPa column pressure constant flow
Temperature programme:	50 °C for 1 min 20 °C/min to 90 °C 5 °C/min to 290 °C 5 min isotherm
Injection conditions:	PTV(Programmable Temperature Vaporization) 2 µl injection volume 60 s split less
	Temperature programme of PTV: Start: 90 °C Final temperature: 320 °C Heating rate approximately 30 °C/min

MS conditions:

	Full spectra 0,60 s/scan

FISONS QMD 1000

	Mass range: 60 amu to 500 amu

Annex B
(informative)

Results of interlaboratory comparisons

An interlaboratory trial was carried out in Germany in 1998 by the Environmental Agency of Hesse *(Hessische Landesanstalt für Umwelt)* with 25 laboratories participating, using Method B. The results are shown in Tables B.1 and B.2.

Another interlaboratory trial was carried out in Germany in 1997 by the Federal Office for Research on test materials (BAM) *(Bundesanstalt für Materialforschung und -prüfung)* with two soil samples, using Method A. The results are shown in Tables B.3 and B.4.

Table B.1 — Soil 1 — Summary of the results of the interlaboratory comparison on Method B

Compound	$\bar{x}$ mg/kg	s_r mg/kg	CV_r %	s_R mg/kg	CV_R %
Naphthalene	12,9	1,15	9,0	6,0	47
Acenaphthylene	3,51	0,27	7,7	1,4	41
Acenaphthene	0,94	0,06	6,4	0,4	40
Fluorene	3,66	0,26	7,2	1,2	33
Phenanthrene	17,2	0,99	5,7	3,6	21
Anthracene	6,91	0,50	7,2	2,1	31
Fluoranthene	21,2	1,21	5,7	4,8	23
Pyrene	17,2	1,15	6,7	5,2	30
Benz[*a*]anthracene	5,95	0,36	6,1	1,5	26
Chrysene	5,63	0,32	5,6	1,9	34
Benzo[*b*+*k*]fluoranthene	7,78	0,39	5,0	2,3	30
Benzo[*a*]pyrene	5,78	0,35	6,0	1,9	34
Dibenz[*a*,*h*]anthracene	0,70	0,06	9,1	0,3	46
Benzo[*ghi*]perylene	4,56	0,29	6,4	1,5	33
Indeno[1,2,3-*cd*]pyrene	4,25	0,26	6,2	1,3	30
Sum of 16 PAH	120,6	5,40	4,5	32,4	27

$\bar{x}$ is the mean of the results.

s_r is the repeatability standard deviation.

CV_r is the relative repeatability standard deviation.

s_R is the reproducibility standard deviation.

CV_R is the relative reproducibility standard deviation.