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**Ambient air — Determination of total (gas and particle-phase) polycyclic aromatic hydrocarbons — Collection on sorbent-backed filters with gas chromatographic/mass spectrometric analyses**

iTeh STANDARD PREVIEW

*Air ambiant — Détermination des hydrocarbures aromatiques polycycliques totales (phase gazeuse et particulaire) — Prélèvement sur filtres à sorption et analyses par chromatographie en phase gazeuse/spectrométrie en masse*

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## Contents

Page

Foreword.....	iv
Introduction.....	v
1 Scope .....	1
2 Normative references .....	1
3 Terms and definitions .....	1
4 Principle.....	2
5 Limits and interferences .....	2
6 Safety measures .....	3
7 Apparatus .....	4
8 Reagents and materials .....	7
9 Preparation of sampling media .....	7
10 Sampling.....	8
11 Sample preparation.....	11
12 Sample analysis.....	13
13 Calculations.....	15
14 Quality assurance.....	16
15 Method detection limit, uncertainty and precision.....	17
Annex A (normative) Performance characteristics.....	18
Annex B (informative) Physical properties of selected PAH .....	19
Annex C (informative) Example of field operations data sheet .....	20
Annex D (informative) Example of a typical PAH chromatogram .....	21
Annex E (informative) Characteristic ions for GC/MS detection of selected PAH .....	23
Bibliography .....	24

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12884 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient air*.

Annex A forms a normative part of this International Standard. Annexes B, C, D and E are for information only.

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## Introduction

This International Standard is applicable to polycyclic aromatic hydrocarbons (PAH) composed of two or more fused aromatic rings. It does not apply to polyphenyls or other compounds composed of aromatic rings linked by single bonds. Several PAH are considered to be potential human carcinogens. PAH are emitted into the atmosphere primarily through combustion of fossil fuel and wood. Two-ring and three-ring PAH are typically present in urban air at concentrations ranging from ten to several hundred nanograms per cubic metre ( $\text{ng}/\text{m}^3$ ); those with four or more rings are usually found at concentrations of a few  $\text{ng}/\text{m}^3$  or lower. PAH possess saturation vapour pressures at 25 °C that range from  $10^{-2}$  kPa to less than  $10^{-13}$  kPa. Those with vapour pressures above  $10^{-8}$  kPa may be substantially distributed between phases depending on ambient temperature, humidity, types and concentrations of PAH and particulate matter, and residence time in the air. PAH, especially those having vapour pressures above  $10^{-8}$  kPa, will tend to vaporize from particle filters during sampling. Consequently, a back-up vapour trap is included for efficient sampling. Except for PAH with vapour pressures below  $10^{-9}$  kPa, separate analyses of the filter and vapour trap will not reflect the original atmospheric phase distributions at normal ambient temperature because of volatilization of compounds from the filter.

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# Ambient air — Determination of total (gas and particle-phase) polycyclic aromatic hydrocarbons — Collection on sorbent-backed filters with gas chromatographic/mass spectrometric analyses

## 1 Scope

This International Standard specifies sampling, cleanup and analysis procedures for the determination of polycyclic aromatic hydrocarbons (PAH) in ambient air. It is designed to collect both gas-phase and particulate-phase PAH and to determine them collectively. It is a high-volume (100 l/min to 250 l/min) method capable of detecting 0,05 ng/m<sup>3</sup> or lower concentrations of PAH with sampling volumes up to 350 m<sup>3</sup>. The method has been validated for sampling periods up to 24 h.

Precision under normal conditions can be expected to be  $\pm 25\%$  or better and uncertainty  $\pm 50\%$  or better (see annex A, Table A.1).

This International Standard describes a procedure for sampling and analysis for PAH that involves collection from air on a combination fine-particle filter and sorbent trap, and subsequent analysis by gas chromatography/mass spectrometry (GC/MS).

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## 2 Normative references

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The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6879:1995, *Air quality — Performance characteristics and related concepts for air quality measuring methods*.

ISO 9169:1994, *Air quality — Determination of performance characteristics of measurement methods*.

ISO/TR 4227:1989, *Planning of ambient air quality monitoring*.

## 3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

### 3.1 sampling efficiency

$E_s$   
ability of the sampler to trap and retain PAH

NOTE The %  $E_s$  is the percentage of the analyte of interest collected and retained by the sampling medium when a known amount of analyte is introduced into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use.

### 3.2 dynamic retention efficiency

$E_r$   
ability of the sampling medium to retain a given PAH that has been added to the sorbent trap in a spiking solution when air is drawn through the sampler under normal conditions for a period of time equal to or greater than that required for the intended use

## 4 Principle

### 4.1 Sampling

An air sample is collected directly from the ambient atmosphere by pulling air at a maximum flowrate of 225 l/min (16 m<sup>3</sup>/h) through first a fine-particle filter followed by a vapour trap containing polyurethane foam (PUF) or styrene/divinylbenzene polymer resin (XAD-2). Sampling times may be varied depending on monitoring needs and the detection limits required. The total volume of air sampled shall not exceed 350 m<sup>3</sup> unless the appropriate deuterated PAH or other suitable standards are added as internal standards to the PUF or XAD-2 sorbent before sampling to validate retention efficiency.

### 4.2 Analysis

After sampling a fixed volume of air, the particle filter and sorbent cartridge are extracted together in a Soxhlet extractor. The sample extract is concentrated by means of a Kuderna-Danish concentrator (or other validated method), followed by a further concentration under a nitrogen stream if necessary, and an aliquot is analysed by gas chromatography/mass spectrometry. The results derived represent the combined gas-phase and particulate-phase air concentrations of each PAH analysed.

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## 5 Limits and interferences

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### 5.1 Limits

PAH span a broad spectrum of vapour pressures (e.g. from  $1,1 \times 10^{-2}$  kPa for naphthalene to  $2 \times 10^{-13}$  kPa for coronene at 25 °C). Table B.1 in annex B lists some PAH that are frequently found in ambient air. Those with vapour pressures above about  $10^{-8}$  kPa will be present in the ambient air, distributed between the gas and particulate phases. This method permits the collection of both phases. However, particulate-phase PAH may be lost from the particle filter during sampling due to desorption and volatilization [1] to [8]. During summer months, especially in warmer climates, volatilization from the filter may be as great as 90 % for PAH with vapour pressures above  $10^{-6}$  kPa [3] and [8]. At ambient temperatures of 30 °C and above, as much as 20 % of benzo[a]pyrene and perylene (vapour pressure =  $7 \times 10^{-10}$  kPa) have been found in the vapour trap [1]. Therefore, separate analysis of the filter will not reflect the concentrations of the PAH originally associated with particles, nor will analysis of the sorbent provide an accurate measure of the gas phase. Consequently, this method requires coextraction of the filter and sorbent to permit accurate measure of total PAH air concentrations.

NOTE This method collects all airborne particulate matter up to at least 40 µm. Particulate-phase PAH are concentrated on fine particles in the ambient atmosphere. Therefore, the use of a particle size-limiting inlet (e.g. PM<sub>10</sub> or PM<sub>2,5</sub>), if required, should have little effect on total PAH measurements.

This method has been evaluated for the PAH shown in annex B. Other PAH may be determined by this method, but the user shall demonstrate acceptable sampling and analysis efficiencies. Naphthalene and acenaphthene possess relatively high vapour pressures and may not be efficiently trapped by this method. The sampling efficiency for naphthalene has been determined to be about 35 % for PUF and about 60 % for XAD-2 [9]. The user may estimate the sampling efficiencies for PAH of interest by determining dynamic retention efficiency of the sorbent. The %  $E_r$  generally approximates the %  $E_s$ .



## 5.2 Interferences

Method interferences can be caused by contaminants in solvents, reagents, on glassware and other sample processing hardware that result in discrete artifacts and/or elevated baselines in the detector profiles. Glassware shall be scrupulously cleaned (e.g. by acid washing, followed by heating to 450 °C in a muffle furnace, and solvent-rinsed immediately prior to use). All solvents and other materials shall be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences can be caused by contaminants that are coextracted from the sample. Additional clean-up by column chromatography shall be required.

The extent of interferences that can be encountered using gas chromatographic techniques has not been fully assessed. Although the GC/MS conditions described allow for resolution of most PAH, some PAH isomers may not be chromatographically resolvable and therefore cannot be distinguished from each other by MS. Interferences from some non-PAH compounds, especially oils and polar organic species, may be reduced or eliminated by the use of column chromatography for sample clean-up prior to GC/MS analysis. The analytical system shall be routinely demonstrated to be free of internal contaminants such as contaminated solvents, glassware, or other reagents that may lead to method interferences. A laboratory reagent blank shall be analysed for each batch of reagents used to determine if reagents are contaminant-free.

Alkyl PAH, if present, may coelute with analytes of interest, but should rarely present problems. Methylacenaphthalene coelution with fluorene is the most likely potential problem, but the identity of fluorene can be confirmed by monitoring secondary ions.

Heteroatomic PAH (e.g. quinoline) should not cause interferences, even if co-elution occurs when the primary and secondary mass ions are used for identification.

Exposure to heat, ozone, nitrogen dioxide (NO<sub>2</sub>) and ultraviolet (UV) light may cause PAH degradation during sampling, sample storage and processing. These problems shall be addressed as part of a standard operating procedure (SOP) prepared by the user. Where possible, incandescent or UV-filtered (excluding wavelengths below 365 nm) fluorescent lighting shall be used in the laboratory to avoid photodegradation during analysis.

**NOTE** Reactive gases, such as ozone and nitrogen oxides, should not significantly affect sample integrity when sampling typical ambient atmospheres. Whereas losses of up to 50 % of benzo[a]pyrene spiked onto air filters (with or without the presence of particulate matter) are likely to occur when ambient air is passed through such filters, especially in atmospheres with high ozone concentrations, studies have shown that reactive losses are insignificant during normal sampling or for benzo[a]pyrene spiked onto filters at near-ambient levels [3], [8], [10] and [11].

Smoking of tobacco products in the sample preparation or analytical laboratory or in adjoining areas may result in contamination of samples with PAH.

## 6 Safety measures

**WARNING — Benzo[a]pyrene and several other PAH have been classified as carcinogens. Care shall be exercised when working with these substances.**

This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. The user shall be thoroughly familiar with the chemical and physical properties of targeted substances.

All PAH shall be treated as carcinogens. Pure compounds shall be weighed in a glove box. Unused samples and standards are considered to be toxic waste and shall be properly disposed of according to regulations. Laboratory benchtops and equipment shall be regularly checked with a hand-held long wavelength UV lamp (366 nm) for fluorescence indicative of contamination.

Some solvents specified in this International Standard may present health hazards if breathed or absorbed through the skin. Hexane is of particular concern. Special care should be exercised when using this solvent. All operations that require working with this solvent should be performed in a fume hood.

## 7 Apparatus

### 7.1 Sampling

#### 7.1.1 Sampling module

A typical collection system consisting of a particle filter backed up by a sorbent trap is shown as an example in Figure 1 [12]. It consists of a metal filter holder (Part 2) capable of holding a 102 mm circular particle filter supported by a 1,2 mm stainless steel screen with 50 % open area and attaching to a metal cylinder (Part 1) capable of holding a 64 mm o.d. (58 mm i.d.) × 125 mm borosilicate glass sorbent cartridge. The filter holder is equipped with inert sealing gaskets [e.g. polytetrafluoroethylene (PTFE)] placed on either side of the filter. Likewise, inert, pliable gaskets (e.g. silicone rubber) are used to provide an air-tight seal at each end of the sorbent cartridge. The glass sorbent cartridge is indented 20 mm from the lower end to provide a support for a 1,2 mm stainless steel screen that holds the sorbent. The glass sorbent cartridge fits into Part 1, which is screwed onto Part 2 until the sorbent cartridge is sealed between the gaskets. The sampling module is described in [12]. Similar sampling modules are commercially available.

#### 7.1.2 High-volume pumping system

Any air sampler pumping system capable of providing a constant air flow of up to 250 l/min (15 m<sup>3</sup>/h) through the sampling module may be used. It shall be equipped with an appropriate flow-control device, a vacuum gauge to measure pressure drop across the sampling module or other suitable flow monitoring device, an interval timer, and an exhaust hose to carry exhausted air at least 3 m away from the sampler. The inlet of the sampler may be oriented upward or downward. If oriented upward, a rain and dust shelter shall be provided.

NOTE The choice of inlets is at the discretion of the user. Particulate-associated PAH are principally concentrated on fine particles; therefore, the particle-size cut-point of the inlet will have little, if any, effect on total PAH measurements.

#### 7.1.3 Flow calibrator

A calibrated manometer or other suitable flow-measuring device capable of being attached to the inlet of the sampling module.

#### 7.1.4 Particle filters

Micro-quartz-fibre, 102 mm diameter binderless, acid-washed, with a filtration efficiency of 99,99 % mass fraction or better for particles below 0,5 µm in diameter, or other appropriate size filter depending on the specific sampling module used.

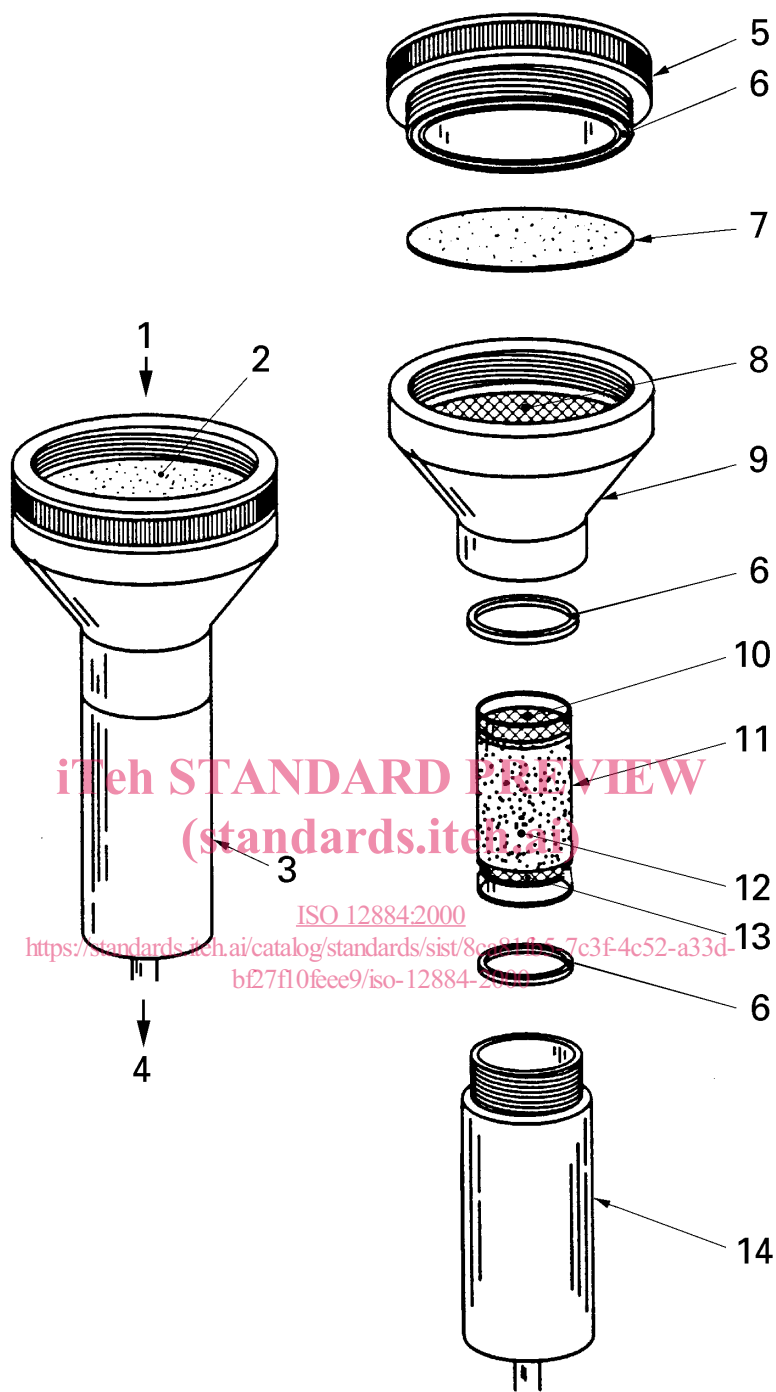
NOTE Glass-fibre or quartz-fibre filters coated or impregnated with PTFE have been used for collection of particulate-associated PAH [13]. Use of these filters in lieu of those specified requires validation of performance by the user.

**7.1.5 Polyurethane foam**, polyether type, density 22 mg/cm<sup>3</sup>, cut into cylinders 76 mm long × 62 mm diameter or other appropriate size depending on the specific sampling module used.

**7.1.6 Adsorbent resin**, of styrene/divinylbenzene polymer (XAD-2), spherical beads, 500 µm diameter, precleaned.

NOTE The sampling system described in 7.1.1 to 7.1.6 has been shown to efficiently trap PAH with three or more rings at a sampling rate of 225 l/min and sample volumes of 350 m<sup>3</sup> and lower [4], [6], [9], [14] to [20]. Other samplers utilizing larger filters (e.g. 200 mm × 250 mm) and higher capacity sorbent traps (e.g. tandem 77 mm × 62 mm PUF plugs) have been used to collect PAH from larger air volumes (e.g. 700 m<sup>3</sup>) [1], [2], [5], [7], [21] to [28].

**7.1.7 Gloves**, polyester or latex rubber, for handling cartridges and filters.



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**Key**

- |                             |                                      |                              |
|-----------------------------|--------------------------------------|------------------------------|
| 1 Air flow inlet            | 6 Sealing gasket                     | 11 Glass cartridge           |
| 2 Particle filter           | 7 102-mm quartz-fibre filter         | 12 Sorbent (XAD-2 or PUF)    |
| 3 Assembled sampling module | 8 Filter support screen              | 13 Retaining screen          |
| 4 Air flow exhaust          | 9 Filter holder (Part 2)             | 14 Cartridge holder (Part 1) |
| 5 Filter retaining ring     | 10 Retaining screen (if using XAD-2) |                              |

**Figure 1 — Example of sampling module**

**7.1.8 Sample containers**, airtight, labelled and screw-capped (wide-mouth, preferably glass jars with PTFE-lined lids), to hold filters and sorbent cartridges during transport to the analytical laboratory.

**7.1.9 Ice chest**, to hold samples at a temperature of 0 °C or below during transport to the laboratory after collection.

**7.1.10 Data sheets**, for each sample, for recording the location and sampling time, duration of sampling, starting time, and volume of air sampled.

## 7.2 Sample preparation

**7.2.1 Soxhlet extractor system**, of volume 200 ml, with 500 ml flask and appropriate condenser. If glass sorbent cartridge is extracted without unloading, a 500 ml extractor and 1 000 ml flask are required.

**7.2.2 Kuderna-Danish (KD) concentrators**, of volume 500 ml, 10 ml graduated tubes with ground-glass stoppers, and 3-ball macro-Snyder column.

**7.2.3 Evaporative concentrators**, including microevaporator tubes of 1 ml capacity, micro-Snyder columns (optional), water bath with  $\pm 5$  °C temperature control, nitrogen blow-down apparatus with adjustable flow control.

### 7.2.4 Cleanup columns

Chromatography columns of e.g. length 60 mm, inner diameter 11,5 mm.

### 7.2.5 Vacuum oven

Drying oven system capable of maintaining a vacuum at 30 kPa to 35 kPa (flushed with nitrogen) overnight.

**7.2.6 Laboratory refrigerator/freezer**, capable of cooling from 4 °C to -20 °C.

**7.2.7 Glove box or high-efficiency hood**, for handling highly toxic standards, with UV-filtered light source.

**7.2.8 Vials**, of volume 40 ml, borosilicate glass.

**7.2.9 Minivials**, of volume 2 ml, borosilicate glass, with conical reservoir and screw caps lined with PTFE-faced silicone disks, and a vial holder.

**7.2.10 Erlenmeyer flasks**, of volume 50 ml, borosilicate glass.

**7.2.11 Boiling chips**, solvent-extracted, silicon carbide or equivalent, grain diameter 0,3 mm to 0,9 mm.

**7.2.12 Spatulas**, PTFE-coated or of stainless steel.

**7.2.13 Tweezers and forceps**, PTFE-coated or of stainless steel.

## 7.3 Sample analysis

### 7.3.1 Gas chromatograph/mass spectrometer

Analytical system complete with gas chromatograph coupled with a mass spectrometer and data processor, suitable for splitless injection, and all required accessories, including temperature programmer, column supplies, recorders, gases and syringes.

**7.3.2 GC columns**, fused silica capillary column of length 30 m to 50 m, inner diameter 0,25 mm, coated with crosslinked 5 % phenyl methylsilicone of 0,25  $\mu$ m film thickness, or other suitable columns.

Ferrules made up of no more than 40 % mass fraction of graphite (e.g. 60 % polyimide and 40 % graphite by mass fraction) shall be used at the GC column injection inlet to avoid possible absorption of PAH.