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Stationary source emissions -- Determination of gas and particle-phase polycyclic aromatic hydrocarbons -- Part 1: Sampling

Émissions de sources fixes -- Détermination des hydrocarbures aromatiques polycycliques sous forme gazeuse et particulaire -- Partie 1: Échantillonnage

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Stationary source emissions — Determination of gas and particle-phase polycyclic aromatic hydrocarbons —

Part 1: Sampling

Émissions de sources fixes — Détermination sous forme gazeuse et particulaire des hydrocarbures aromatiques polycycliques —

Partie 1: Échantillonnage

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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 11338-1 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

ISO 11338 consists of the following parts, under the general title *Stationary source emissions — Determination of gas and particle-phase polycyclic aromatic hydrocarbons*:

- *Part 1: Sampling*
- *Part 2: Sample preparation, clean-up and determination*

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a group of aromatic hydrocarbons, some members of which are probable and others possible human carcinogens. Human exposure to PAHs can occur via food, soil, water, air and skin contact with materials containing PAHs. While PAH are formed in natural processes (e.g. forest fires), man-made atmospheric emissions of these compounds originate from the combustion of coal, gas, wood and oil, from a range of industrial processes such as coke production, aluminium smelting and from vehicles.

The quantification of atmospheric releases of PAH from stationary sources is an important part of the environmental impact assessment of certain industrial processes.

Stationary source emissions — Determination of gas and particle-phase polycyclic aromatic hydrocarbons —

Part 1:
Sampling

1 Scope

This part of ISO 11338 describes methods for the determination of the mass concentration of polycyclic aromatic hydrocarbons (PAHs) in flue gas emissions from stationary sources such as aluminium smelters, coke works, waste incinerators, power stations, and industrial and domestic combustion appliances.

This part of ISO 11338 describes three sampling methods, which are here regarded as of equivalent value, and specifies the minimum requirements for effective PAH sampling. The three sampling methods are the dilution method (A), the heated filter/condenser/adsorber method (B) and the cooled probe/adsorber method (C). All three methods are based on representative isokinetic sampling, as the PAHs are commonly associated with particles in flue gas.

Information is provided to assist in the choice of the appropriate sampling method for the measurement application under consideration.

This part of ISO 11338 is not applicable to the sampling of fugitive releases of PAHs.

NOTE Methods for sample preparation, clean-up and analysis are described in ISO 11338-2 and are intended to be combined with one of the sampling methods described in this part of ISO 11338 to complete the whole measurement procedure.

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 4225:1994, *Air quality — General aspects — Vocabulary*

ISO 9096:1992, *Stationary source emissions — Determination of concentration and mass flow rate of particulate material in gas-carrying ducts — Manual gravimetric method*

ISO 12141, *Stationary source emissions — Determination of mass concentration of particulate matter (dust) at low concentrations — Manual gravimetric method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4225 and the following apply.

3.1
chimney
stack or final exit duct on a stationary process used for the dispersion of residual process gases

3.2
mass concentration
concentration of a substance in an emitted flue gas, expressed in units of mass per cubic metre

3.3
polycyclic aromatic hydrocarbon
PAH
compound that contains two or more fused aromatic rings made up only of carbon and hydrogen atoms

3.4
stationary source emissions
gases emitted by a stationary plant or process and transported to a chimney for dispersion into the atmosphere

4 Principles and minimum requirements for the three sampling methods

4.1 Principles

Devices for the three sampling methods, illustrated in Figure B.1 (Method A), Figures B.2 and B.3 (Method B) and Figure B.7 (Method C), can be applied for the sampling of PAH from stationary sources. They are regarded as likely to produce equivalent results, however no comparative trials have been published to establish this.

In general, as PAHs are present in both the vapour phase and on particles, the PAHs are collected in several parts of the sampling train: the particle filter, condensate flask and solid or liquid adsorber. The choice of the sampling method depends on the measurement application (see Table A.1 and Table A.2).

The three sampling methods A, B and C are discussed in detail in Clauses 5, 6 and 7, respectively.

Annex A provides further information on the applicability of the sampling methods.

After extraction and clean-up, the PAH are quantified either by High Performance Liquid Chromatography (HPLC) using a fluorescence detector, diode array or UV detector, or by GC-FID/MS (low or high resolution MS). Applicable methods for preparation, clean-up and determination are described in ISO 11338-2.

4.2 General minimum requirements for all sampling methods

The following steps shall be carried out irrespective of the sampling method chosen.

a) Carry out isokinetic sampling at representative points in the duct cross-section, in accordance with ISO 9096.

b) Before sampling, rinse the inside of the sampling device with acetone, dichloromethane or methanol and then with toluene. Alternatively, immerse the parts in methanol and subject them to ultrasonic vibration for 2 h, and subsequently dry at 150 °C. Store these washings and analyse only if the results indicate that the sampler could have been contaminated before sampling, for example if the results unexpectedly exceed the relevant emission limit.

c) Carry out a leak check before every sampling procedure.

If the joints in the equipment are of ground glass, any slight leak can be dealt with by wetting the joints with a small amount of clean water. Greases should never be used for this purpose.

d) The minimum velocity in the sorbent bed shall be:

- for XAD-2 (thickness 50 mm, volume 35 cm^3): less than 34 cm/s;
- for PU foam (thickness 50 mm, volume 98 cm^3): less than 30 cm/s.

e) Check each batch of filters, solvents and reagents for preparation for background PAH levels.

f) The glass parts of the sampling devices shall be protected from light during and after sampling, cooled after sampling and cleaned after the extraction procedure.

g) Clean the probe thoroughly after each sample is taken. Add the probe rinse to the rinse from the rest of the sample.

h) If the probe is contaminated with particles which cannot be easily removed, wipe away the particles with a quartz wool swab steeped in acetone. Extract this quartz wool swab together with the filter.

i) If the sampling device is cleaned at the measurement site for reuse there, the probe, the nozzle, the filter casings and all other parts of the sampling apparatus which have been in contact with the sample gas and so could still be contaminated shall, after cleaning to remove the sample, be rinsed with acetone, dichloromethane or methanol and followed by a toluene rinse. As before, this sample shall be preserved in case of concern about cross-contamination between samples.

j) Extract samples within 1 week and preferably within 24 h. Store the samples in the dark at – 7 °C.

4.3 General preparation and sampling

The following steps shall be carried out irrespective of the sampling method chosen.

a) Choose the sampling location with regard to the safety of the personnel, the suitability of the measurement cross-section (in accordance with ISO 9096), accessibility and availability of electrical power.

b) Before the sampling starts, determine the flue gas density, pressure, temperature and if possible the gas composition. In addition, to ensure isokinetic conditions, determine the velocity and temperature profile across the cross-section of the flue gas channel. Choose the correct size of the sampling probe nozzle calculated from the flue gas velocity and the approximate maximum flowrate achievable through the sampler to ensure that the sampler will be capable of isokinetic sampling at all the measurement points in the duct cross-section.

c) The parts of the sampler which come into contact with the sample and which have been carefully cleaned in the laboratory shall be

- transported in clean boxes, all the components having been sealed carefully;
- assembled *in situ*, carefully avoiding contact with the operator's fingers on the parts of the sampling equipment which will later be in contact with the sample.

d) After sampling, store all materials containing sampled PAH under cooled conditions and protected from light.

The following data shall be recorded during sampling:

- sample volume (standard conditions);

- sample temperature (in the gas meter);
- mean flue gas velocity;
- diameter of the stack;
- moisture content of the flue gas;
- mean oxygen content of the flue gas during the sampling period;
- static pressure and temperature in the stack;
- sampling flowrate.

To provide an estimate of the contamination present in the sampler and sampling matrices before sampling and as a result of sampler assembly and transport, an additional sampling unit may be taken to the sampling location, rinsed and analysed. Unfortunately contamination incidents are random in their occurrence, and so this will only provide an estimate of the blank values. As a result, subtraction should not take place. However, results in which the contribution of this blank is substantial should be treated with caution.

NOTE In order to obtain information on the performance of the sampler, internal standards can be added to the sampling equipment (e.g. on the filters or the adsorbent) and their recovery measured. However, the internal standards are bound to the filter in a manner different to that by which the native PAHs are bound to the fly ash, so there may be differences in behaviour during sampling and extraction.

5 Method A — Dilution method

5.1 Principle

A proportion of the flue gas is collected isokinetically via a sampling probe heated to the temperature of the flue gas. The flue gas is cooled very rapidly to temperatures below 40 °C in a mixing chamber using dried, filtered and, if appropriate, cooled air. This dilution prevents condensation of water present in the gas sample. In addition, dilution seeks to minimize the reactions of the separated PAH with other flue gas components, e.g. NO, NO_2, SO_2, SO_3 and HCl. The sampling conditions are similar to the natural dilution and cooling processes of flue gases emitted into the atmosphere.

The diluted flue gas is then passed through a silicone-bonded glass fibre filter impregnated with paraffin oil. This retains PAH components with 4 to 7 rings. If sampling for more volatile PAHs (2- or 3-ring compounds) is required, a solid adsorbent1) can be incorporated downstream of the filter in the sampling train.

The sample gas flowrate through the sampling probe should be in the range of 2 m^3/h to 8 m^3/h. Normally within 1,5 h of sampling, sufficient PAH for analysis has been collected, usually in about 8 m^3 to 10 m^3. After sampling, the filter and, if used, the solid adsorbent are extracted and analysed.

A schematic representation of a tested sampling device is given in Figure B.1.

5.2 Minimum requirements

The following procedures shall be followed.

a) Keep the filter at < 40 °C. Avoid condensation on the filter.

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1) Porapak PS is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 11338 and does not constitute an endorsement by ISO of this product.