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Indoor air —

Part 38: Determination of amines in indoor and test chamber air — Active sampling on samplers containing phosphoric acid impregnated filters

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

A list of all parts in the ISO 16000 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

ISO 16000 (all parts) describe general requirements relating to the measurement of indoor air pollutants and the important conditions to be observed before or during the sampling of individual pollutants or groups of pollutants, as well as the measurements procedures themselves.

The definition of indoor environment is given by ISO 16000-1. Dwellings [living rooms, bedrooms, do-it-yourself (DIY) rooms, sport rooms and cellars, kitchens and bathrooms], workrooms or workplaces in buildings which are not subject to health and safety inspections with respect to air pollutants (e.g. offices, salesrooms), public buildings (e.g. restaurants, theatres, cinemas and other meeting rooms) and passenger cabins of motor vehicles and public transportation are among the most important types of indoor environment.

Indoor air —

Part 38:
Determination of amines in indoor and test chamber air — Active sampling on samplers containing phosphoric acid impregnated filters

1 Scope

This document specifies a method for the determination of primary, secondary and tertiary aliphatic and aromatic amines in indoor air using accumulated sampling and high-performance liquid-chromatography (HPLC) coupled with tandem mass spectrometry (MS-MS) or high resolution mass spectrometry (HRMS). It specifies the sampling procedure for determining the mass concentration of amines as mean values by sampling the amines on phosphoric acid impregnated filters. The analytical procedure of the measurement method is covered by ISO 16000-39.

Measurements, performed with samplers containing phosphoric acid-impregnated inert supporting material and operating at specified flow rates for specified sampling periods are described in this document. Requirements regarding sample volume are also defined.

The range of application of this document concerning the concentrations of amines in indoor air depends on the linear range of the calibration line and hence on the gas sample volume (here: from 5 l up to 100 l), the eluate volume (from 1 ml up to 5 ml), the injection volume (from 1 µl up to 10 µl) and the sensitivity of the analytical equipment (e.g. linear range from 2 pg up to 2 ng amine). The range of application can be expected to be from approximately 0,002 µg/m^3 (100 l sample) up to 2 000 µg/m^3 (5 l sample) for a common analytical equipment[1)] for the majority of the amines listed in Annex A. The analysis of derivatives of ethanolamine is usually about 10 times more sensitive and the analysis of short-chained aliphatic amines is usually about 10 times less sensitive than the analysis of an average amine.

Although primarily intended for the measurement of amines listed in Annex A, this document can also be used for the measurement of other amines in indoor air.

This document describes procedures for the fabrication and gives requirements for the use of glass tubes containing impregnated filters out of phosphoric acid-impregnated glass wool as samplers, but does not exclude other samplers with proven equal or improved properties. This document also gives procedures for the demonstration of equivalence of other sampler types or methods.

This document does not cover the determination of amines in other media like water or soil. Furthermore, it does not cover the determination of isocyanates in indoor air as corresponding amines (covered by ISO 17734-1 and ISO 17734-2). Quaternary amines are also not included in this document.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

1) Waters "TQ-D" is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO online browsing platform: available at https://www.iso.org/odp

— IEC Electropedia: available at https://www.elctropedia.org/

3.1
amines
nitrogen containing compounds with a sufficient vapor pressure (>10^{-3} Pa) and a free electron pair at the nitrogen atom which can be protonated by phosphoric acid

4 Amines in indoor air

4.1 Properties of amines

Amines are basic and polar substances.

There are primary, secondary, tertiary, and quaternary amines.

Quaternary amines are not included in this document as they have no free electron pair and therefore have very different properties. In this document, the term “amines” includes primary, secondary, and tertiary amines only.

Not protonated amines are oxidation sensitive.

The reaction of amines with acids results in ammonium salts of the amines. The ammonium salts are not oxidation sensitive.

4.2 Origin and occurrence of amines in indoor air

Amines are produced by technical chemical processes and processing and in addition by biotic or abiotic decomposition of nitrogen compounds. Besides sources of biological origin, indoor air sources of amines could be, for instance, products containing polyurethane, especially foams, such as in vehicle seats, mattresses, pillows, and upholstered furniture or as thermal insulation or sound absorbing material. Several amines, in particular aromatic amines, are known as harmful compounds. Furthermore, most amines have an unacceptable odour in combination with a low odour threshold.

Further sources are for example food, such as fish (aliphatic amines) and cigarette smoke (aromatic amines).

5 Sampling strategy — Measurement procedure

5.1 Structure and properties of the samplers

The basic structure of an amine sampler for active sampling consists of a container, preferably a glass or plastic tube, and a filter. The filter is impregnated with an acid with low vapour pressure and without an oxidizing effect. In this regard, phosphoric acid is most suitable. The filter material itself shall be inert, such as untreated glass wool, glass spheres or glass frits. Basically, the inert filter material could be a porous, inert polymer.

The suggested sampler consists of a glass tube with 6,25 mm outside diameter on a length of 60 mm, and a tapering on a length of 20 mm with an outside diameter of the tip of 2,5 mm. The suggested filter consists of 50 mg of untreated glass wool impregnated with approximately 100 µmol or 9,8 mg H_3PO_4, respectively.

The sampler that is described as an example in this document (see 5.2) is handmade.

Until now, no verified commercially available amine samplers for active sampling are known.

Alternative samplers shall be tested by the procedure described in 5.9.

5.2 Manufacturing of the samplers

5.2.1 General

The manufacturing of active samplers with phosphoric acid impregnated glass wool is described in the following:

Chemical agents:	470 µl phosphoric acid 85 % (density = 1,685 g/cm^3)
	200 ml acetonitrile
	2,5 g untreated glass wool
Laboratory equipment:	500 ml round bottom flask
	Glass tubes (6,25 mm outside diameter on a length of 60 mm, tapering on a length of 20 mm with an outside diameter of the tip of 2,5 mm)
	Rotary evaporator
	1 000 µl automatic pipette

5.2.2 Implementation

Approximately 100 ml acetonitrile are filled into a 200 ml volumetric flask. Then 470 µl phosphoric acid 85 % are filled into the flask and mixed with the acetonitrile. After that, the flask is filled up to the 200 ml mark with acetonitrile and the solution is mixed again to get a 34,3 mmol/l H_3PO_4 solution in acetonitrile.

2,5 g glass wool is transferred into a 500 ml round bottom flask together with 200 ml 34,3 mmol/l H_3PO_4 solution in acetonitrile. (2,74 nmol H_3PO_4/g glass wool).

The acetonitrile is distilled off in a rotary evaporator at 100 r/min and at a temperature of 90 °C to 100 °C.

Each glass tube is packed with (50,0 ± 0,5) mg of the resulting impregnated glass wool (corresponds to 137 µmol H_3PO_4/sampler if all H_3PO_4 remains on the glass wool and if the acid is equally distributed). The impregnated glass wool is compressed thoroughly in the glass tube. Both opening of the glass tube are sealed by suitable plastic caps.

Until application, the samplers are stored in the freezer at −36 °C.

5.2.3 Verification

The mass of phosphoric acid on each sampler (approximately 9,8 mg or 100 µmol, respectively) is verified by titrating 5 samplers for each batch (10 % of the batch) with a 0,01 molar aqueous solution of sodium hydroxide and phenolphthalein as an indicator. The change of colour corresponds to the second equivalence point (complies with approximately 20 ml 0,01 M NaOH solution).

$H_3PO_4 + NaOH \rightarrow NaH_2PO_4 + H_2O$

$NaH_2PO_4 + NaOH \rightarrow Na_2HPO_4 + H_2O$ (transition of the indicator from colourless to purple)

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$H_3PO_4 + 2NaOH \rightarrow Na_2HPO_4 + 2H_2O$