
Indoor air —

Part 39:

Determination of amines —

**Analysis of amines by (ultra-) high-
performance liquid chromatography
coupled to high resolution or tandem
mass spectrometry**

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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.

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Indoor air —

Part 39:

Determination of amines — Analysis of amines by (ultra-) high-performance liquid chromatography coupled to high resolution or tandem mass spectrometry

1 Scope

This document, along with ISO 16000-38, specifies the measurement method for determining the mass concentration 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). The analytical procedure is covered by this document. The sampling procedure and the manufacturing of the samplers are covered by ISO 16000-38.

This document describes specifications for the chromatography and the mass spectroscopy for the amines. Measurement results are expressed in $\mu\text{g}/\text{m}^3$.

Although primarily intended for the measurement of amines listed in [Tables A.1](#) and [A.2](#), it can also be used for the measurement of other amines in indoor air. This document gives instructions and describes procedures for the inclusion of other amines.

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 μg up to 2 ng amine). The range of application can be expected to be from approximately 0,002 $\mu\text{g}/\text{m}^3$ (100 l sample) up to 2 000 $\mu\text{g}/\text{m}^3$ (5 l sample) for a common analytical equipment (e.g. Waters „TQD“) for the majority of the amines listed in [Tables A.1](#) and [A.2](#). 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.

The performance data of the analytical method is given in Annex B, particularly in [Tables B.1](#) and [B.2](#).

This document can be used also for the determination of amines in water if the detection limit is sufficient.

This document does not cover the determination of isocyanates in indoor air (nor in water samples) as corresponding amines (covered by ISO 17734-1 and ISO 17734-2).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 16000-38, *Indoor air — Part 38: Determination of amines in indoor and test chamber air — Active sampling on samplers containing phosphoric acid impregnated filters*

3 Terms and definitions

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

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

- ISO online browsing platform: available at <http://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.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

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” refers only to primary, secondary, and tertiary amines.

Not protonated amines are oxidation sensitive.

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

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4.2 Origin and occurrence of amines

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, e.g. 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, e.g. fish (aliphatic amines), and cigarette smoke (aromatic amines).

5 Analytical procedure

5.1 Preparation of sample solutions

ISO 16000-38 describes the procedure to get the aqueous eluate of the ammonium salts of the sampled amines. The sample solution (which contains already approximately 0,02 $\mu\text{mol}/\mu\text{l}$ up to 0,1 $\mu\text{mol}/\mu\text{l}$ phosphoric acid) can be used for the injection into the HPLC system without further treatment.

5.2 High-performance liquid chromatography

- stationary phase of separating column: pentafluorophenyl;
- common column dimensions: length 150 mm, inner diameter: 2,1 mm;

- common particle size: 3 µm (HPLC), 1,7 µm [Ultra Performance Liquid Chromatography (UHPLC)];
- column temperature: 40 °C;
- injection volume: 1 µl to 10 µl;
- mobile phase: acetonitrile and water with each 0,02 % formic acid (500 µl formic acid in 2,5 l acetonitrile or 2,5 l water, respectively);
- mobile phase composition: 28 % acetonitrile/72 % water, isocratic.

5.3 Tandem mass spectrometry

- ionisation: electrospray ionisation, positive mode (ESI+);
- source temperature: 120 °C;
- desolvation gas: nitrogen, 900 l/h;
- desolvation temperature: 420 °C;
- cone gas: nitrogen, 50 l/h;
- precursor ion: M+1;
- collision gas: argon;
- calibration: external and additionally with Pyridine-d5 as internal standard.

5.4 High resolution mass spectrometry

- ionisation: electrospray ionisation, positive mode (ESI+).

5.5 Sample sequence and external calibration

- external calibration: run calibration standards at least at the beginning and at the end of a sample sequence;
- every 25 samples or less and at the beginning and end, run a calibration blank and a calibration check solution of an independent source;
- every 50 samples (for example) and at the end of a run, analyse an interference check solution (ICS).

6 Testing of the suitability of the equipment and the instrumental analysis

6.1 General

In [Table A.3](#), several amines are listed which can interfere with another amine in the instrumental analysis. In order to check and confirm the suitability of the selected system and procedure following issues have to be tested.

6.2 High-performance or ultra-performance liquid chromatography (HPLC or UHPLC)

The chromatographic separation of following compounds has to be tested and confirmed:

- a) Diethylamine (11) and N,N-Dimethylethylamine (24);
- b) Isobutylamine (2) and n-Butylamine (10);
- c) 2-(Dimethylamino)ethanol (8) and 2-(Ethylamino)ethanol (25);

d) Diisobutylamine (14) and Di-n-butylamin (16).

Usually 4-Ethylmorpholine (4) and 2-(Diethylamino)ethanol (3) as well as Morpholine (13) and 2-(Dimethylamino)ethanol (8) cannot be separated sufficiently on pentafluorophenyl terminated stationary phases. These compounds have to be distinguished by high resolution or tandem mass spectrometry.

The chromatographic separation of Diethylamine (11) and N,N-Dimethylethylamine (24) is not always possible (depends mainly on the column). If tandem mass spectrometry is used for detection the ratio of the product ions (46 and 29 u/e; 6.3) can be used for discrimination.

6.3 Tandem mass spectrometry (MS-MS)

The selectivity of the tandem mass spectrometry has to be tested and confirmed for following potentially interfering amines or product ions, respectively:

	Relevant product ions
a) Diethylamine (11) and N,N-Dimethylethylamine (24):	ratio of 46 and 29 u/e
b) 2-(Dimethylamino)ethanol (8) and Morpholine (13):	ratio of 72, 70, 45 and 44 u/e
c) 2-(Ethylamino)ethanol (25) and Morpholine (13):	ratio of 72, 70, 45 and 44 u/e
d) 4-Ethylmorpholine (4) and 2-(Diethylamino)ethanol (3):	ratio of 100, 72, 45 and 44 u/e

For additional information see [Annex A \(Tables A.3 and A.4\)](#).

6.4 High resolution mass spectrometry (HRMS)

A possible interference due to a loss of H₂ during ionization has to be tested and excluded for following compounds:

- Morpholine (13) by 2-(Dimethylamino)ethanol (8);
- Morpholine (13) by 2-(Ethylamino)ethanol (25);
- 4-Ethylmorpholine (4) by 2-(Diethylamino)ethanol (3).

7 Reporting requirements

- reference to this document (ISO 16000-39);
- reason of the measurement (e.g. peculiar odour, product quality testing, process control);
- identification of the sample (distinct sample number);
- sample description (origin, e.g. active sampling of air referred to ISO 16000-38 in an office, a test chamber, car or manufacturing plant and subsequent elution with water);
- if appropriate, reference to ISO 16000-38, otherwise detailed description of the sampler and the sampling procedure;
- date and time of sampling;
- short description of the location of sampling;
- sampling conditions: relative humidity, temperature and pressure;
- volume of sampled air (in l);

- if appropriate: description of transportation and storage of the loaded sampler;
- applied eluent volume (elution of the amines and the phosphoric acid from the sampler referred to ISO 16000-38);
- if appropriate: description of (further) sample preparation (e.g. subsequent dilution);
- short description of the instrumental analysis (e.g. MS/MS or HRMS);
- target analytes with CAS number;
- limit of detection and limit of quantification in the analysed solution in $\mu\text{g}/\text{l}$ and in the air sample in $\mu\text{g}/\text{m}^3$ for all target analytes;
- mass concentration of all target analytes in the analysed solution in $\mu\text{g}/\text{l}$ with 3 significant digits, where necessary specify “below limit of quantification”;
- mass concentration of all target analytes in the air sample in $\mu\text{g}/\text{m}^3$ with 2 significant digits;
- measurement uncertainty;
- if appropriate, further necessary specifications and details.

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