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Zrak na delovnem mestu - Določanje organskih dušikovih spojin v zraku s tekočinsko kromatografijo in masno spektrometrijo - 1. del: Določanje izocianatov preko derivatov dibutilamina

Workplace air - Determination of organonitrogen compounds in air using liquid chromatography and mass spectrometry - Part 1: Isocyanates using dibutylamine derivatives

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Workplace air — Determination of organonitrogen compounds in air using liquid chromatography and mass spectrometry —

Part 1: Isocyanates using dibutylamine derivatives

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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).

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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 2, *Workplace atmospheres*.

This third edition cancels and replaces the second edition (ISO 17734-1:2013), which has been technically revised.

The main changes are as follows:

— this document has been extensively revised to bring it into line with the current ISO/IEC Directives, Part 2, including updates to structure, terminology, and drafting style;

— the references have been reviewed and updated, and obsolete references have been removed or replaced where appropriate;

— the Scope has been revised to comply with current ISO requirements and to improve clarity on the applicability of the method;

— an error regarding the presentation of the analytical performance of the method has been corrected.

A list of all parts in the ISO 17734 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

Isocyanates are organic compounds containing the isocyanate functional group (–N=C=O) and have been used in industry for about 50 years. They are commercially important chemicals mainly used for the production of polyurethane (PUR). In spite of controls to limit exposures, there are adverse health effects such as asthma, contact dermatitis and hypersensitivity pneumonitis as consequences of exposure to isocyanates in some industrial sectors.

The analytical method for the determination of isocyanates in workplace air must be sensitive due to the high irritation and sensitization properties of isocyanates. Extremely low occupational exposure limits (OELs) exist in many countries, and concentrations well below the OEL, often less than one hundredth of the limit, are often to be determined. Isocyanates are very reactive and therefore cannot be analysed directly. Derivatization during sampling is required in order to prevent interfering reactions. Hundreds of different isocyanates are used in industry, and many more are formed during thermal degradation of PUR. Therefore, high selectivity of the analytical method is required for accurate results.

The determination of isocyanates in the work environment using di-*n*-butylamine (DBA) as a reagent and liquid chromatography-mass spectrometric detection (LC-MS) has been demonstrated to be a robust method. The development of the method was initiated when difficulties using the "older" methods during sampling of isocyanates in complex atmospheres were encountered (e.g. thermal decomposition of PUR).[1][2][3] The reaction rate between DBA and isocyanates was found to be fast, and high concentrations can be used to secure instantaneous reactions and eliminate problems with interfering compounds.[4][5] Using impinger flasks containing a reagent solution and a filter in series efficiently collects and derivatizes isocyanates in both the gas and the particle phase.[6] LC-MS/MS of the isocyanate-DBA derivatives enables highly selective and precise determinations down to levels below 10^{-6} of the OEL[7].

Solvent-free sampling can also be performed by using a tube coated with a DBA-impregnated glass fibre filter followed by an impregnated filter. An impregnation solution containing DBA together with an acid is used, and the formed ion pair reduces volatility. DBA remains on the filter even after 8 h of sampling[8].

Monomeric isocyanates that are formed during thermal decomposition of polymers [typically PUR and phenol-formaldehyde urea (PFU) resins], such as isocyanic acid and methyl isocyanate, can also be determined.[6][7][8][9][10] Volatile isocyanate-DBA derivatives can be determined using gas chromatography-mass spectrometric detection (GC-MS).[9] Using the DBA method and derivatization with ethyl chloroformate makes simultaneous determinations of amine, aminoisocyanates and isocyanates possible, as described in the companion method in ISO 17734-2[11].

For quantification, reference compounds are necessary but are only available for a few monomeric isocyanates. Most of the isocyanates that are used in industry for the production of PUR can only be obtained in technical grade mixtures. Many isocyanates that are formed during thermal degradation are not available and are not easily synthesized. In this method, a nitrogen-sensitive detector has been used for quantifying isocyanates in reference solutions. This technique has been demonstrated to be a useful tool, together with MS characterization, in greatly facilitating the production of reference solutions[10][12][13].

For quantifying isocyanates in complex mixtures, MS detection appears to be the current best available detection technique and provides a unique possibility of identifying unknown compounds. This method has enabled assessment of new areas for which exposure to isocyanates was not known previously and has identified new kinds of isocyanates in the work environment[6][7][8][9][10][12][13].

Workplace air — Determination of organonitrogen compounds in air using liquid chromatography and mass spectrometry —

Part 1: Isocyanates using dibutylamine derivatives

1 Scope

This document specifies a method for the determination of organic isocyanates in workplace air using derivatization with di-n-butylamine (DBA) and chromatographic analysis.

This document is applicable to the determination of a wide range of organic isocyanates present in both the gas phase and the particle phase in workplace atmospheres, including monofunctional isocyanates such as isocyanic acid (ICA), methyl isocyanate (MIC), ethyl isocyanate (EIC), propyl isocyanate (PIC), butyl isocyanate (BIC) and phenyl isocyanate (PhI); monomeric diisocyanates, including 1,6-hexamethylene diisocyanate (HDI), 2,4- and 2,6-toluene diisocyanate (TDI), 4,4′-methylenediphenyl diisocyanate (MDI), 1,5-naphthyl diisocyanate (NDI), isophorone diisocyanate (IPDI) and 4,4′-dicyclohexylmethane diisocyanate (H12MDI); and multifunctional isocyanates, including oligomeric, prepolymeric and polymeric forms such as biuret-, isocyanurate- and allophanate-adducts.

The method covers air sampling using impingers, filters or combinations thereof, and analysis by liquid chromatography with mass spectrometric detection.

The useful analytical range is approximately 2,5 ng to 500 ng of isocyanate per sample. For a nominal air sample volume of 15 l, this corresponds to approximately 0,2 µg/m^3 to 33 µg/m^3. These values can vary depending on the isocyanate analysed.

This document does not apply to the determination of amines or aminoisocyanates. For the determination of amines and aminoisocyanates, the method specified in ISO 17734-2[11].

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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 13137, *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

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

3.1
isocyanate
chemical compound containing one or more –N=C=O functional groups

3.2
monomer
chemical compound that joins with other identical compounds to form dimers, trimers, oligomers or polymers

Note 1 to entry: Classes of isocyanate monomers (include: monoisocyanates, containing one isocyanate functional group (e.g. methyl isocyanate), diisocyanates [e.g. di(4-isocyanatophenyl)methane (MDI)] and triisocyanates [e.g. tri(4-isocyanatophenyl)methane].

3.3
diisocyanate
chemical compound with two isocyanate functional groups

3.4
oligomer
compound of low relative molecular mass with multiple isocyanate functional groups, formed by the combination of isocyanate monomers

3.5
prepolymer
isocyanato-terminated reaction product of a di- or poly-isocyanate that has a stochiometric deficiency, with a hydroxyl-terminated polyol

Note 1 to entry: These compounds are further reacted to form polyurethanes or similar compounds.

3.6
technical isocyanate
isocyanates produced by isocyanate manufacturers and used as is or with other components according to its use in the industry

4 Principle

Samples are collected by drawing a known volume of air through a midget impinger flask followed by a filter. The impinger contains 10 ml of 0,01 mol·l^{-1} of DBA in toluene and the filter is a glass fibre filter with no binder.

Solvent-free sampling can also be performed by drawing air through a tube coated with a DBA-impregnated glass fibre filter followed by an impregnated filter. An impregnation solution containing DBA together with acetic acid is used, the ion pair so formed reduces the volatility and enables long-time sampling.

After sampling, deuterium-labelled isocyanate-DBA derivatives (used as internal standard) are added to the sample solutions. The excess reagent and solvent are evaporated, and the samples are dissolved in acetonitrile. The samples are analysed using reversed-phase LC and electrospray (ESP)-MS detection, monitoring positive ions. Quantification is made by monitoring selected ions. See Figure 1.

Quantification and qualitative determinations can be performed using different LC-MS or LC-MS/MS techniques. Liquid chromatography-chemiluminescent nitrogen detection (LC-CLND) or liquid chromatography-ultraviolet detection (LC-UV) for aromatic isocyanates, can be used for the determination of higher concentrations of isocyanates.

Reference materials can be characterized using LC-MS/CLND. For characterization of volatile compounds, gas chromatography-thermionic specific detector (GC-TSD) can also be used.

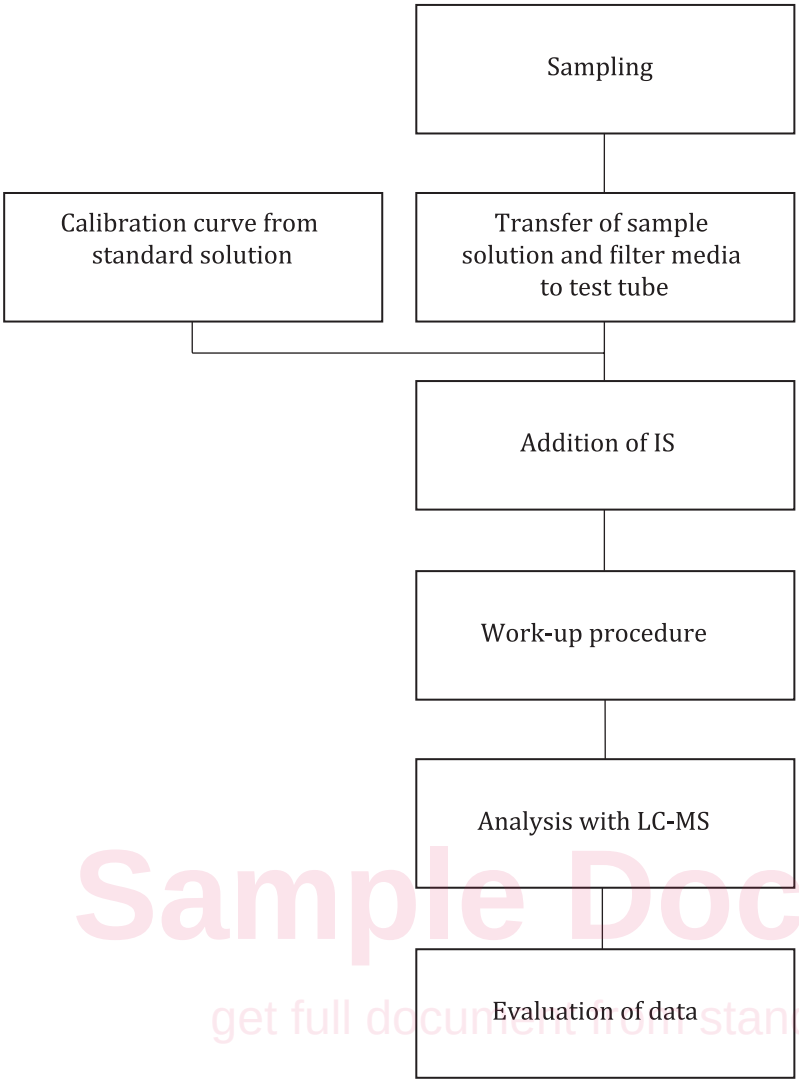

Figure 1 — Example of a flow diagram of the whole procedure for sampling, sample preparation, analysis and reporting

5 Reagents and materials

5.1 DBA reagent.

Analytical grade di-*n*-butylamine is commercially available.

5.2 Solvents.

The reagent solvent, typically toluene and other solvents, such as acetonitrile, isooctane and methanol, should be of liquid chromatographic quality.

5.3 Formic acid, concentrated formic acid, analytical grade.

5.4 Acetic acid, concentrated acetic acid, analytical grade.

5.5 Pyridine, analytical grade.

5.6 Sodium hydroxide, reagent grade.

5.7 Ethyl chloroformate, analytical grade.

5.8 Urea, analytical grade.

5.9 1,3 dimethyl urea, analytical grade.

5.10 Water, in accordance with ISO 3696, grade 2 water (electrical conductivity less than 0,1 mS/m and resistivity greater than 0,01 MΩ·m at 25 °C).

5.11 Sulfuric acid 5 mM, 0,27 ml of concentrated sulfuric acid (98 %) analytical grade is added to 1 000 ml of water.

5.12 Reagent solution.

In a 1 l volumetric flask, dilute 1,69 ml of DBA in toluene and make up to the mark. The solution is stable and no special care during storage is necessary.

5.13 Reagent solution, for solvent-free sampler.

5.13.1 Solution 1: 0,74 mol·l^{-1} DBA.

Mix 80 ml methanol and 12,5 ml DBA in a 100 ml volumetric flask. Then while stirring, slowly add 4,16 ml of acetic acid to the flask. Finally, add methanol to the flask, and make up to the mark.

5.13.2 Solution 2: 1,5 mol·l^{-1} DBA.

Mix 60 ml methanol and 25 ml DBA in a 100 ml volumetric flask. Then while stirring, slowly add 8,32 ml of acetic acid to the flask. Finally, add methanol to the flask, and make up to the mark.

5.14 HPLC mobile phases.

5.14.1 LC-MS.

The weak mobile phase (mobile phase A) consists of water and acetonitrile at a volume fraction of 95 % water and 5 % acetonitrile, with 0,05 % formic acid. The strong mobile phase (mobile phase B) consists of water and acetonitrile at a volume fraction of 5 % water and 95 % acetonitrile, with 0,05 % formic acid. The mobile phases are degassed prior to use. The mobile phases are degassed prior to use.

5.14.2 LC-CLND.

The weak mobile phase (mobile phase C) consists of water and methanol at a volume fraction of 95 % water and 5 % methanol, with 0,05 % formic acid. The strong mobile phase (mobile phase D) consists of water and methanol at a volume fraction of 5 % water and 95 % methanol, with 0,05 % formic acid. The mobile phases are degassed prior to use.

6 Preparation of standard solutions

6.1 Reference compounds

Reference compounds are necessary for LC-MS determination of isocyanate derivatives. For the commercially available isocyanates, the DBA derivatives are easily prepared by direct derivatization with DBA. DBA derivatives for the isocyanates not commercially available can be made from the bulk material or