BRITISH STANDARD

BS ISO 16702:2007

Workplace air quality — Determination of total organic isocyanate groups in air using 1-(2-methoxyphenyl) piperazine and liquid chromatography

ICS 13.040.30

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British Standards

Full document details: BS ISO 16702:2007

National foreword

This British Standard is the UK implementation of ISO 16702:2007. It supersedes BS ISO 16702:2001 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee EH/2, Air quality, to Subcommittee EH/2/2, Workplace atmospheres.

A list of organizations represented on this committee can be obtained on request to its secretary.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2008

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ISBN 978 0 580 56230 3

Amendments/corrigenda issued since publication

Date	Comments

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INTERNATIONAL STANDARD

ISO 16702

Second edition
2007-12-15

Workplace air quality — Determination of total organic isocyanate groups in air using 1-(2-methoxyphenyl)piperazine and liquid chromatography

Qualité de l'air des lieux de travail — Dosage des groupements isocyanates organiques totaux dans l'air par dérivatisation avec la 1-(2-méthoxyphényl)pipérazine et par chromatographie en phase liquide

Reference number
ISO 16702:2007(E)

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Foreword

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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 16702 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This second edition cancels and replaces the first edition (ISO 16702:2001), which has been technically revised.

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Introduction

Isocyanates (molecules containing the NCO functional group) are highly reactive molecules widely used in industry in paints, polyurethane foams, plastics, and adhesives. They are known respiratory sensitisers and are the major cause of chemically induced occupational asthma. Exposure to isocyanates can occur by inhalation and possibly by contact. Australia, Ireland, and the United Kingdom have set long-term occupational exposure limits (8 h time-weighted average) of 20 µg/m^3 [total isocyanate (NCO) group] and short-term limits (15 min) of 70 µg/m^3 for workplace air. In addition, Finland has set a short term limit (15 min) of 35 µg/m^3 and Sweden has set long-term occupational exposure limits (8 h time-weighted average) of 5 ppb [1)] [total isocyanate (NCO) group] and short-term limits (15 min) of 10 ppb for workplace air. These limits are for total isocyanate, i.e. monomeric and all polymeric (also called oligomeric, polyisocyanates, oligo-isocyanates or prepolymeric) isocyanates.

Sampling and analysis of airborne isocyanates is not easy. Isocyanates occur in a variety of chemical forms, such as monomers, oligomers, larger and more structurally complex polymers, and mixtures of all these forms. Isocyanate oligomers and polymers are commonly used in industry as they are less volatile than the monomers and so pose less of a vapour hazard. Isocyanates occur in a variety of physical forms, e.g. vapours, aerosols, and liquids. A sampling method that is suitable for one physical form of isocyanates is not automatically suitable for another. In the workplace, other substances are also present in the air, such as water vapour, dust, amines and alcohols, depending on the product and process that is being used, and these can interfere with the liquid chromatography (LC) analysis. Polymeric isocyanate standards are not available, yet these species must be quantified to give a total isocyanate result.

Due to the reactive nature of the isocyanate group, analysis in the workplace is commonly carried out by trapping isocyanates with a derivatisation reagent to produce a stable derivative. This International Standard method is based upon the UK method for isocyanate determination, MDHS25/3[1].

The method traps the isocyanate with 1-(2-methoxyphenyl)piperazine (MP) to form a stable urea derivative. The urea derivative is analysed by LC with electrochemical (EC) and ultraviolet/visible (UV/vis) detection. Isocyanates for which a standard exists or can be prepared can be quantified using a UV/vis detector. This has the advantage that a UV/vis detector is more stable than an EC detector. However, for the majority of industrially used polymeric isocyanates, no standards exist and these compounds are quantified using the EC detector, which oxidises the methoxy group on the MP derivatisation reagent. As this group is common to all MP derivatised isocyanates, the polymeric species can be calibrated using the corresponding isocyanate monomer.

The procedure used for sampling of workplace isocyanates depends upon their physical form. Filters have been found to sample vapour effectively. An impinger/filter combination is recommended for aerosol sampling. This method has been found to be suitable for the commonly occurring mono- and diisocyanates i.e. methylenebis(phenylisocyanate) (MDI), phenylisocyanate (PI), toluene-2,6-diisocyanate and toluene-2,4-diisocyanate (TDI), 1,6-(diisocyanato)hexane (HDI), isophoronediisocyanate (IPDI), naphthyldiisocyanate (NDI), methylenebis(cyclohexylisocyanate) (hydrogenated MDI) and butylisocyanate as well as polymeric isocyanates based on these monomers.

1) Parts per billion (thousand million).

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Workplace air quality — Determination of total organic isocyanate groups in air using 1-(2-methoxyphenyl)piperazine and liquid chromatography

1 Scope

This International Standard gives general guidance for the sampling and analysis of airborne organic isocyanate (NCO) compounds in workplace air.

This International Standard is appropriate for a wide range of organic compounds containing isocyanate functional groups, including isocyanate monomers and prepolymers. Examples of aromatic monomers include toluene diisocyanate (TDI) (both 2,4- and 2,6-diisocyanatotoluene), naphthyl diisocyanate (NDI) (1,5-diisocyanatonaphthalene) and methylenebis(4-phenylisocyanate) [MDI, systematically named as di-(4-isocyanatophenyl)methane]. Examples of aliphatic monomers include isophorone diisocyanate (IPDI, systematically named as 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethylcyclohexane), methylenebis(cyclohexylisocyanate) (hydrogenated MDI, HMDI) and 1,6-diisocyanatohexane (HDI) (also known as 1,6-hexamethylenediisocyanate). Monomers containing a single isocyanate moiety (e.g. methyl isocyanate, ethyl isocyanate, phenyl isocyanate, hexyl isocyanate) are produced during thermal degradation of polyurethanes, i.e. flame bonding and laser cutting. Isocyanate polymers, also called polyisocyanates, homopolymers, oligomers or prepolymers, are derived from the diisocyanate monomers by self-condensation or reaction with polyols. Polymeric diisocyanates are widely used in the polyurethanes, paints and coatings, and adhesives industries.

This International Standard is appropriate for measuring any product containing free isocyanate groups. It was developed primarily for the commonly used MDI, HDI, and TDI, and their oligomers and polymers[1]. It has also been used for IPDI, HMDI, and NDI, and their oligomers and polymers. The exposure limit for isocyanates in the UK requires measurement of total isocyanate groups, i.e. monomeric diisocyanates, oligomeric, prepolymeric and polymeric diisocyanates and monoisocyanates. Because there are a wide range of isocyanate structures and molecular masses, the chromatographic conditions used will need to be varied according to the isocyanate formulation being determined. If both isocyanates and amines are believed to be present, and both need to be determined, a standard which enables the simultaneous determination of both amines and isocyanates may be more appropriate[2]. This method has also been modified to allow determination of mono-isocyanates produced during thermal degradation[3], the use of mass spectrometric detection[4] and other sampling equipment, e.g. 37 mm filters and other filter cassettes, but these modifications are not covered in this International Standard. If a modified version of this method is being used, it is the responsibility of the user to demonstrate that the modifications are valid.

The method is used to determine time-weighted average concentrations of organic isocyanates in workplace atmospheres, and is suitable for sampling over periods in the range 0,5 min to 8 h. The method is designed for personal monitoring, but can also be used for fixed location monitoring by suitable modification.

NOTE The objective of air monitoring is usually to determine worker exposure and, therefore, the procedures described in this method are for personal sampling in the breathing zone. The method can be used for background or fixed location sampling. However, it should be recognised that, due to aerodynamic effects, samplers designed for personal sampling do not necessarily exhibit the same collection characteristics when used for other purposes.

The method is suitable for the measurement of airborne organic isocyanates in the concentration range from approximately 0,1 µg/m^3 to 140 µg/m^3 for a 15 l sample volume. The qualitative and quantitative detection limits for isocyanate, defined as three times and 10 times the standard deviation of six blank determinations, have been found to be typically between 0,001 µg and 0,004 µg of isocyanate per sample, respectively (EC detection). For a 15 l air sample, these values correspond to qualitative and quantitative detection limits of 0,07 µg/m^3 and 0,3 µg/m^3, respectively.

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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 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*

EN 1232, *Workplace atmospheres — Pumps for personal sampling of chemical agents — Requirements and test methods*

3 Terms and definitions

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

3.1 Isocyanate chemical species

3.1.1
isocyanate
a chemical compound with one or more isocyanate (nitrogen carbon oxygen) functional groups

3.1.2
monomer
a chemical compound that joins with other identical compounds to form dimers, trimers, oligomers or polymers

EXAMPLE 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.

3.1.3
diisocyanate
a chemical compound with two isocyanate functional groups

3.1.4
oligomer
a compound of low relative molecular mass with multiple isocyanate functional groups, formed by the combination of isocyanate monomers

3.1.5
polyisocyanate
oligo-isocyanate
an isocyanate compound with multiple isocyanate functional groups

3.1.6
prepolymer
the isocyanato-terminated reaction product of a di- or poly-isocyanate with a stochiometric deficiency for a hydroxyl-terminated polyol; these compounds are then further reacted to form polyurethanes or similar compounds

3.2 Analytical definitions

3.2.1
time-weighted average concentration
concentration of a chemical agent in the atmosphere, averaged over the reference period

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3.2.2
field blank
sampler that is taken through the same handling procedure as a sample, except that it is not used for sampling, i.e. sampling media is loaded into a sampler, transported to the sampling site, derivatised when field samples are derivatised, and analysed with field samples

3.3 Statisitical definition

uncertainty
⟨of measurement⟩ parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand

[ISO/IEC Guide 98:1995[5], 2.2.3]

NOTE 1 The parameter can be, for example, a standard deviation (or a given multiple of it), or the width of a confidence interval.

NOTE 2 Uncertainty of measurement comprises, in general, many components. Some of these components can be evaluated from the statistical distribution of the results of series of measurements and can be characterised by standard deviations. The other components, which can also be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information. This is often referred to as type A and type B evaluations of uncertainty, respectively.

4 Principle

The choice of sampling device used in this method depends upon the physical form of the isocyanate being sampled. For an isocyanate aerosol, a glass impinger containing 1-(2-methoxyphenyl)piperazine (MP) solution backed by a filter impregnated with the MP reagent is used. For an isocyanate vapour, then an MP impregnated filter may be used on its own.

A measured volume of air is drawn through a glass impinger containing 1-(2-methoxyphenyl)piperazine (MP) solution backed by a filter impregnated with the MP reagent (isocyanate aerosol) or a filter impregnated with the MP reagent (isocyanate vapour). Any organic isocyanates present will react to form non-volatile urea derivatives. The resultant solution is concentrated and analysed by high-performance liquid chromatography (HPLC) with ultraviolet/visible (UV) and electrochemical (EC) detection. Isocyanate-derived peaks are identified on the basis of their EC and UV responses and also by diode array detection (DAD) spectral library matching, mass spectrometry (where available), and comparison with derivatising bulk[6]. For isocyanates for which a standard MP derivative is available, e.g. HDI, MDI, TDI isomers, UV can be used for quantification. If no suitable standard is available, i.e. for isocyanate oligomers, prepolymers and polymers, quantification is by EC, using the relevant isocyanate monomer standard for calibration. The total isocyanate-in-air concentration is calculated from the sum of all the isocyanate-derived peaks.

5 Reagents and materials

Use only reagents of recognised analytical grade and only distilled water or water of equivalent purity.

5.1 MP reagent [1-(2-methoxyphenyl)piperazine]

This reagent is commercially available at appropriate (> 98 % by mass) purity.

5.2 Reagent solvent

The reagent solvent, commonly toluene, should be of chromatographic quality. It must be free from compounds co-eluting with the substances of interest. Before use for the preparation of impregnated filters or for preparation of monomer standards, it is advisable to dry the solvent with anhydrous calcium chloride or

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magnesium sulfate. This step may be omitted for preparation of the absorbing solution as it will pick up atmospheric moisture during sampling.

5.3 Reagent solutions

5.3.1 Absorbing solution

Accurately weigh approximately 50 mg of MP and transfer to a dry 100 ml volumetric flask. Dissolve and make up to the mark with reagent solvent, and mix thoroughly. Dilute 10 ml of this stock solution to 100 ml with reagent solvent in a second volumetric flask to give a 260 µM absorbing solution.

5.3.2 Preparation of solution for impregnating filters (solution A)

Accurately weigh out approximately 0,25 g of MP and transfer to a 25 ml volumetric flask. Make up to the mark with anhydrous reagent solvent and shake to mix.

5.3.3 Stability of reagent solutions

Prepare fresh solutions weekly.

5.4 Calibration standards

5.4.1. Preparation of monomer derivatives

Add 0,1 g of the appropriate isocyanate (~1 mmol for the common diisocyanates such as HDI, TDI and MDI) to 0,6 g (~3 mmol) of MP dissolved in dry toluene (10 ml) and leave to stand for 1 h. A white crystalline urea is precipitated. Collect this on a filter paper (e.g. Whatman No 1[2)]) and wash several times with dry toluene to remove excess reagent. Recrystallise the urea from toluene, by warming to about 60 °C and slowly adding methanol to dissolve the urea. Allow to cool and then filter the resulting crystals, washing with cold, dry toluene. Dry the solid in air. The urea derivatives of the mono- and most diisocyanates are only slightly soluble in toluene but readily soluble in methanol or acetonitrile.

5.4.2 Alternative procedure for the less soluble isocyanate derivatives

MDI and HMDI are rather insoluble in toluene and the alternative method of preparation given below may be more suitable for these compounds. Slowly add a solution of the appropriate isocyanate (0,25 g, ~2 mmol NCO for MDI and HMDI) in dichloromethane (25 ml) to a solution of 1-(2-methoxyphenyl)piperazine (1 g, ~5 mmol) in dichloromethane (50 ml). A white suspension will form. Add this dropwise to a beaker of hexane (500 ml) while stirring. Filter the resultant precipitate and redissolve it in a minimum volume of dichloromethane. Add hexane to reprecipitate the solid, filter this and wash with hexane. Dry the urea derivative in air.

NOTE This second method may also be used for isocyanate oligomers, polymers and prepolymers.

5.4.3 Preparation of standard solutions of recrystallised isocyanate monomer derivatives

5.4.3.1 Weigh out a known mass of the urea derivative, place in a 100 ml volumetric flask and make up to the mark with acetonitrile or methanol. Take aliquots of this solution and dilute volumetrically in acetonitrile or HPLC mobile phase to create a series of standard solutions over the NCO concentration range 0,01 µg/ml to 1,0 µg/ml.

5.4.3.2 Prepare further standard solutions if the concentration range of the samples exceeds that of the standards.

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2) Example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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5.4.3.3 The concentration of isocyanate in the standard, ρ_{NCO}, in micrograms per millilitre, is given by Equation (1):

$$\rho_{NCO} = \frac{\rho_U M_{NCO} n}{M_U} \quad (1)$$

where

ρ_U is the concentration, in micrograms per millilitre, of the urea derivative in the standard;

M_{NCO} is the relative molecular mass of NCO;

n is the number of isocyanate groups per molecule;

M_U is the relative molecular mass of the urea derivative.

5.5 Stability of isocyanate ureas and their solutions

Stock solutions of isocyanate monomer derivatives have been found to be stable for ~ 6 months if kept in a freezer[7]. A mixture of 2,4-TDI and 2,6-TDI on filters and in toluene solution has been found to be stable for up to 90 days (73 %, filter, and 81 %, toluene solution, recoveries, respectively)[8]. MDI on filters has been found to be stable for at least 6 months [HSE Workplace Analysis Scheme for Proficiency (WASP)[1] data]. An isocyanate prepolymer [Desmodur N 3390[3)]] spiked onto MP filters was found to be stable for 27 days (average recovery 91 ± 11 %, spiked at three levels, 0,1, 1 and 2 µg/filter)[9].

5.6 HPLC mobile phase

The exact composition of the mobile phase used depends on the isocyanate formulation being determined. The more acetonitrile in the mobile phase, the faster the peaks will elute. A “slow” mobile phase can be used for monomeric diisocyanates and monoisocyanate MP derivatives. For the polymeric isocyanate MP derivatives, a “fast” mobile phase is more suitable. Care must be taken to elute all the polymeric MP derivatives and not to lose any monomeric species under the acetylated MP reagent peak at the start of the chromatogram.

5.6.1 Preparation of “slow” mobile phase

A “slow” mobile phase, suitable for the determination of monomeric diisocyanates and mono-isocyanates, is prepared as follows. Dissolve 5 g of anhydrous sodium acetate in 1 l water. Adjust the pH of this solution to 6,0 with glacial acetic acid. Add 550 ml of this solution to acetonitrile (450 ml) and degas this solution by filtering under vacuum or by bubbling a stream of helium through it to give a volume mixture of 45 % acetonitrile and 55 % sodium acetate buffer.

5.6.2 Preparation of “fast” mobile phase

A “fast” mobile phase, suitable for the determination of polymeric diisocyanates, is prepared as follows. Dissolve 5 g of anhydrous sodium acetate in 1 l water. Adjust the pH of this solution to 6,0 with glacial acetic acid. Add 400 ml of this solution to acetonitrile (600 ml) and degas this solution by filtering under vacuum or by bubbling a stream of helium through it to give a volume mixture of 60 % acetonitrile and 40 % sodium acetate buffer.

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5.7 Calibration blend atmosphere

Prepare an atmosphere of a known concentration of the substance or substances of interest in air by a recognised method. Methods described in ISO 6145 (all parts) are suitable. Confirm the delivered atmosphere concentration using an independent method.

6 Apparatus

Before sampling and analysis, clean all glassware, including impingers (8.2).

Usual laboratory apparatus and, in particular, the following.

6.1 Sampler

The choice of sampler used depends on the form in which the isocyanate is present. For vapour phase isocyanates, sampling can be carried out using an impregnated filter only. For mixtures of airborne particles and vapour, the use of an impinger backed by an impregnated filter is recommended. Details of alternative sampling procedures are given below.

6.2 Filter

Filters of diameter 25 mm are suitable for use in the selected sampler. The chosen filter type should have a capture efficiency of not less than 95 % and be suitable for collection of stable samples of isocyanate. MP-impregnated glass fibre [GF/A[4)]] filters have been found to be suitable.

6.3 Filter holder

Details of suitable sampling heads are given in MDHS14/3[10]. A 25 mm Institute of Occupational Medicine head fitted with a stainless steel cassette is recommended for filter samples. For aerosol sampling using the impinger/filter combination, it has been found to be more convenient to use the 25 mm Swinnex[5)] filter holder.

6.4 Midget impinger

A number of designs of bubblers and impingers are available[11],[12]. A midget impinger consists of a graduated receiver and a tapered inlet tube.

NOTE “Non-spill” impingers are commercially available.

6.5 Sampling pump

The pump shall fulfil the requirements of EN 1232 or equivalent. The sampling pump should be in accordance with prevailing safety regulations.

6.6 Tubing

Plastic, rubber or other suitable tubing about 900 mm long of appropriate diameter to ensure a leak-proof fit to both pump and sample tube or tube holder, if used. Fluoroelastomer or similar tubing has been found to have fewer problems due to extraction of contaminants associated with it. It is not recommended to use any tubing upstream of the first collection element (filter or impinger) as sample losses may occur.

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