BS ISO 17734-2:2013

BSI Standards Publication

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

Part 2: Amines and aminoisocyanates using dibutylamine and ethyl chloroformate derivatives

National foreword

This British Standard is the UK implementation of ISO 17734-2:2013. It supersedes BS ISO 17734-2:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/2/2, Work place atmospheres.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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INTERNATIONAL STANDARD

ISO 17734-2

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

Part 2: Amines and aminoisocyanates using dibutylamine and ethyl chloroformate derivatives

Détermination des composés organiques azotés dans l'air par chromatographie liquide et spectrométrie de masse —

Partie 2: Amines et aminoisocyanates par les dérivés de la dibutylamine et du chloroformate d'éthyle

Reference number
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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This second edition of ISO 17734-2 cancels and replaces ISO 17734-2:2006, which has been technically revised.

ISO 17734 consists of the following parts, under the general title *Determination of organonitrogen compounds in air using liquid chromatography and mass spectrometry*:

— *Part 1: Isocyanates using dibutylamine derivatives*

— *Part 2: Amines and aminoisocyanates using dibutylamine and ethyl chloroformate derivatives*

Introduction

In many applications, when considering isocyanates as a workplace contaminant, there is also a need to investigate the presence of aminoisocyanates and amines. During thermal decomposition of polyurethane (PUR), not only isocyanates, but also amines and aminoisocyanates, are formed.[1][2][3][4][5][6]

The determination of isocyanates in the work environment using DBA as a reagent has been demonstrated to be a robust method (see ISO 17734-1). Using the DBA method and derivatization with ethyl chloroformate in the following work-up procedure makes simultaneous determination of amines, aminoisocyanates, and isocyanates possible.[6][7]

For quantification of amine and aminoisocyanate derivatives, reference compounds are necessary, but are only available for a few diamines. Aminoisocyanates cannot be analysed directly because they react with themselves. In this method, a nitrogen-specific detector has been used for quantification of amine and aminoisocyanate derivatives 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.[6]

INTERNATIONAL STANDARD ISO 17734-2:2013(E)

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

Part 2: Amines and aminoisocyanates using dibutylamine and ethyl chloroformate derivatives

1 Scope

This part of ISO 17734 gives general guidance for the sampling and analysis of airborne amines and aminoisocyanates in workplace air. It is strongly recommended that the determination of amines and aminoisocyanates is made together with the determination of isocyanates in air, using DBA as a reagent (see ISO 17734-1).

The method can be used for simultaneous determinations of amines, such as 4,4'-methylenediphenyldiamine (4,4'-MDA), 2,4- and 2,6-toluenediamine (2,4- and 2,6-TDA), and 1,6-hexamethylenediamine (1,6-HDA), and compounds containing both isocyanate and amine groups, such as 4,4'-methylenediphenyl aminoisocyanate (4,4'-MAI), 2,4-, 4,2-, and 2,6-toluene aminoisocyanate (2,4-, 4,2-, and 2,6-TAI), and 1,6-hexamethylene aminoisocyanate (1,6-HAI). The method is suitable for collecting amines and aminoisocyanates in both the gas and particle phases. The instrumental detection limit for the amines is about 5 nmol/sample and for the aminoisocyanate, it is about 0,3 nmol/sample. For a 15 l air sample, this corresponds to 0,4 $ng \cdot m^{-3}$ for TDA and 0,03 $ng \cdot m^{-3}$ for TAI.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16200-1, *Workplace air quality — Sampling and analysis of volatile organic compounds by solvent desorption/gas chromatography — Part 1: Pumped sampling method*

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*

3 Principle

The method permits the simultaneous sampling and analysis of amines, aminoisocyanates, and isocyanates. Only amines and aminoisocyanates are discussed in this part of ISO 17734, because isocyanates are considered in ISO 17734-1.

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 $mol \cdot l^{-1}$ of di-*n*-butylamine (DBA) in toluene, and the filter is a glass fibre filter with no binder. After sampling, deuterium-labelled amine-ethyl chloroformate (ET) and 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 liquid chromatography (LC) and electrospray (ESP) mass spectrometric (MS) detection, monitoring positive ions. Quantification is made by quantifying selected ions.

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

