



SLOVENSKI STANDARD

SIST ISO 16702:2012

01-april-2012

Nadomešča:
SIST ISO 16702:2002

Kakovost zraka na delovnem mestu - Določevanje celotnih organskih izocianatnih skupin v zraku z uporabo 1-(2-metoksifenil)piperazina in tekočinske kromatografije

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

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

<http://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892ff39/sist-iso-16702-2012>

Ta slovenski standard je istoveten z: ISO 16702:2007

ICS:

13.040.30 Kakovost zraka na delovnem mestu Workplace atmospheres
mestu

SIST ISO 16702:2012

en,fr

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ISO 16702:2012

<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892ff39/sist-iso-16702-2012>

**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*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ISO 16702:2012

<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892ff39/sist-iso-16702-2012>



ISO 16702:2007(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[SIST ISO 16702:2012](https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892f39/sist-iso-16702-2012)

<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892f39/sist-iso-16702-2012>

**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2007

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	v
Introduction	vi
1 Scope	1
2 Normative references	2
3 Terms and definitions.....	2
4 Principle.....	3
5 Reagents and materials	3
6 Apparatus	6
7 Sampling.....	7
7.1 Calibration of pump.....	7
7.2 General.....	7
7.3 Preparation of sampling equipment (general).....	8
7.4 Preparation of sampling equipment (filters).....	8
7.5 Preparation of sampling equipment (impingers).....	8
7.6 Collection of filter samples (vapour phase samples).....	8
7.7 Collection of impinger backed by filter samples (isocyanate aerosols).....	9
7.8 Measurements to be made at the end of the sampling period.....	9
7.9 Sample logging and field desorption of samples.....	9
7.10 Transportation.....	9
7.11 Field Blanks.....	10
8 Procedure	10
8.1 Safety precautions.....	10
8.2 Cleaning of glassware.....	10
8.3 Prereaction of impinger samples before HPLC analysis.....	10
8.4 Prereaction of filter samples before HPLC analysis	10
8.5 HPLC conditions.....	10
8.6 Determination of airborne isocyanate for monomeric isocyanates (UV detection)	11
8.7 Identification of polymeric isocyanates: EC/UV ratio approach.....	11
8.8 Confirmation of identification for polymeric isocyanates (prepolymers).....	12
8.9 Quantification of airborne isocyanate for polymeric isocyanates (EC detection).....	13
8.10 Sampling efficiency	13
9 Calculations.....	14
10 Interferences	14
11 Uncertainty of measurement	14
11.1 Introduction	14
11.2 Assessment of performance characteristics of the method — Sampling considerations (detailed ISO/IEC Guide 98:1995 approach).....	16
11.3 Assessment of performance characteristics of the method — Other considerations — (detailed ISO/IEC Guide 98:1995 approach).....	17
11.4 Mass of compound in field sample blank.....	21
11.5 Between-laboratory uncertainty contributions.....	22
11.6 Combined uncertainty	22
11.7 Expanded uncertainty	22
12 Stability.....	22
13 Test report	22

ISO 16702:2007(E)

14	Quality control	23
Annex A (informative)	Determination of sampling efficiency	24
Annex B (informative)	Data used for uncertainty estimates	25
Annex C (informative)	Combined uncertainties for isocyanate formulations	26
Annex D (informative)	Sample chromatograms	28
Bibliography		34

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST ISO 16702:2012](https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892ff39/sist-iso-16702-2012)

<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892ff39/sist-iso-16702-2012>

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.

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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ISO 16702:2012
<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892ff39/sist-iso-16702-2012>

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/m³ [total isocyanate (NCO) group] and short-term limits (15 min) of 70 µg/m³ for workplace air. In addition, Finland has set a short term limit (15 min) of 35 µg/m³ and Sweden has set long-term occupational exposure limits (8 h time-weighted average) of 5 ppb¹⁾ [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].

<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-45b4892970>

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), isophoronediiisocyanate (IPDI), naphthylidiiisocyanate (NDI), methylenebis(cyclohexylisocyanate) (hydrogenated MDI) and butylisocyanate as well as polymeric isocyanates based on these monomers.

1) Parts per billion (thousand million).

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.

<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e->

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/m³ to 140 µg/m³ 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/m³ and 0,3 µg/m³, respectively.

ISO 16702:2007(E)**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.

<https://standards.iteh.ai/catalog/standards/sist/4315e008-99b0-480b-9b8e-4504c892ff39/sist-iso-16702-2012>

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 stoichiometric 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

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 Statistical 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

ISO 16702:2007(E)

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 μ M 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 1²⁾) 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 μ g/ml to 1,0 μ g/ml.

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

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.

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

$$\rho_{\text{NCO}} = \frac{\rho_{\text{U}} M_{\text{NCO}}^n}{M_{\text{U}}} \quad (1)$$

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 3390³] 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 $\mu\text{g}/\text{filter}$)^[9].

5.6 HPLC mobile phase (standards.iteh.ai)

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.

3) 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.