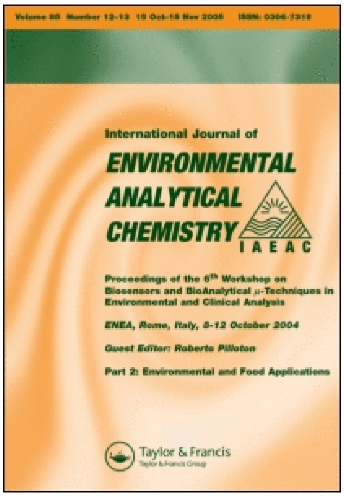

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

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To cite this Article Brorson, T. , Sangö, C. , Skarping, G. and Renman, L.(1990) 'Evaluation of Chromatographic Methods for the Determination of Isocyanates in Air', International Journal of Environmental Analytical Chemistry, 38: 3, 399 — 413

To link to this Article: DOI: 10.1080/03067319008026944

URL: http://dx.doi.org/10.1080/03067319008026944

Intern. J. Environ. Anal. Chem., Vol. 38, pp. 399–413
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EVALUATION OF CHROMATOGRAPHIC METHODS FOR THE DETERMINATION OF ISOCYANATES IN AIR

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(Received 13 June 1989)

Chromatographic methods for the determination of toluene diisocyanates (TDI) in air, at concentrations of 20–40 $\mu g/m^3$, were evaluated. Test atmospheres were generated by a gas-phase permeation principle. The GC method was based on sampling in impingers containing an acidic aqueous solution (0.4 M HCl), where the isocyanates are hydrolysed into the corresponding amines. The pentafluoropropionic acid derivatives of the amines were analysed using glass capillary GC with thermionic detection. The HPLC methods were based on reaction with the amine reagents 9-(N-methylaminomethyl)-anthracene (1×10^{-4} M in toluene) and 1-(2-methoxyphenyl)-piperazine (2×10^{-4} M in toluene). The urea derivatives were determined using UV detection. The sampling efficiency for gaseous TDI with toluene as absorbing solution was 97%. Sampling efficiencies in various acidic aqueous solutions were 83–88%. The relative standard deviation for the various methods was ca. 6%. No sampling losses for TDI, due to influences of interfering substances (diethylamine, dimethylethylamine, N-methylmorpholine, DABCO, aniline, ethanol, phenol) using acidic aqueous 0.4 M HCl were obtained. Diethylamine, N-methylmorpholine and DABCO affected the analytical results when toluene solutions of the amine reagents MAMA and MPP were used.

KEY WORDS: Isocyanates, air, reagents, sampling efficiency, interfering substances.

INTRODUCTION

Diisocyanates are used for the preparation of polyurethane polymers (PUR) which have important industrial applications, e.g., flexible and rigid polyurethane foam coatings, adhesives and elastomers. Diisocyanates react with water, alcohols or phenols and amines to form amines, urethanes and ureas. Tertiary aliphatic amines

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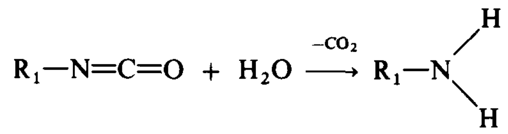
are commonly used as catalysts in the polyurethane industry. Diisocyanates show an irritating effect on the respiratory tract, are well known as asthma inducers, and are strongly suspected for hypersensitivity pneumonitis.[1,2] The use of prepolymerized isocyanates also constitutes a potential health hazard, e.g., when used for spraying applications where diisocyanate aerosols are formed.[13,24,65]

The toxic effects make it necessary to monitor these substances in working atmospheres, and to develop appropriate methods for their determination at very low concentrations. The Swedish threshold limit value (TLV) for diisocyanates is 70 μg/m^3 (5 min ceiling value), which corresponds to 0.35 μg of TDI per sample, for a 5 min sampling period at an air flow of 1 l/min. The use of high-sensitive analytical methods is therefore essential.

A number of methods have been developed for the analysis of isocyanates in air. Up to the late seventies colorimetric methods were mostly used.[49,50,55,56] However, these methods lack sensitivity and selectivity, and have now to a great extent been replaced by chromatographic methods, liquid chromatography (HPLC) being most frequently used. All chromatographic methods are, however, discontinuous and time-consuming, and at present the only continuous monitoring devices are paper-tape monitors..[3-5]

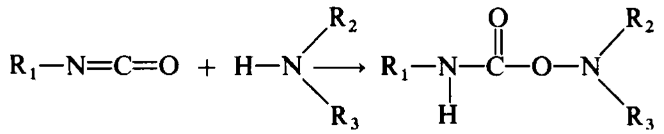
A survey of chromatographic methods for the determination of isocyanates in air is shown in Table 1. For the chromatographic analysis four main approaches have been used:

Hydrolysis to the corresponding amine



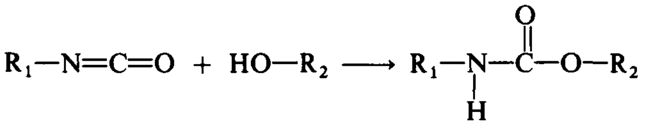
$$R_1{-}N{=}C{=}O + H_2O \xrightarrow{-CO_2} R_1{-}NH_2$$

Reaction with an amine to an urea derivative



$$R_1{-}N{=}C{=}O + H{-}NR_2R_3 \longrightarrow R_1{-}NH{-}C(=O){-}O{-}NR_2R_3$$

Reaction with an alcohol to an urethane derivative



$$R_1{-}N{=}C{=}O + HO{-}R_2 \longrightarrow R_1{-}NH{-}C(=O){-}O{-}R_2$$

Direct determination without further derivatization

Table 1 Chromatographic methods for the determination of isocyanates in air

Reagent	*Chemical abstract (CA) no.*	*Chromatographic method*	*Sampling method*	*Detection method*	*Reference*
Acidic solution	—	GC	Impinger	ECD, TSD, MS	32–39, 51
		HPLC	Impinger	UV, EC	40,52–54
N-(4-nitrobenzyl)-N-*n*-propylamine, "nitro reagent"	62869-74-3	HPLC	Impinger	UV	6–10, 14
			Sorbent		11
			Filter		12, 13
1-(2-pyridyl)-piperazine	34803-66-2	HPLC	Impinger	UV	15, 63
			Sorbent		16–18
1-(2-methoxyphenyl)-piperazine (MPP)	35386-24-4	HPLC	Impinger	UV/EC	19, 20
1-Naphthalemethylamine	118-31-0	HPLC	Impinger	F	21
9-(N-methylaminomethyl)-anthracene (MAMA)	73356-19-1	HPLC	Impinger	F/UV	22
			Sorbent		23, 24
			Filter		25
N-methyl-1-naphthalenemethylamine	14489-75-9	HPLC	Impinger	F/UV	26
Diethylamine	109-89-7	HPLC	Sorbent	UV	27
Aniline	62-53-3	HPLC	Impinger	UV	28
p-Aminophenol	123-30-8	HPLC	Impinger	EC	29
Ethanol	64-17-5	HPLC	Impinger	UV, EC	9, 30, 44
		GC	Impinger	TSD, MS	31, 62
Direct analysis (no reagent)	—	GC	Impinger	ECD, TSD	41–43

HPLC, high performance liquid chromatography; GC, gas chromatography; UV, ultraviolet detection; EC, electrochemical detection; F, Fluorescence detection; ECD, electron capture detection; TSD, thermionic specific detection; MS, mass spectrometer.

Only a few comparative studies[61,64] and reviews[1,4,57,59,60] of analytical methods for the determination of isocyanates in air, have been made. The knowledge about how to choose a suitable method, e.g., when sampling in industrial environments with the coexistence of interfering chemicals, is also limited. The aim of this investigation therefore was to evaluate three standard chromatographic methods, based on the use of different reagents, for the determination of toluene diisocyanate (TDI) in air, to evaluate sampling efficiencies using different absorption liquids, and to determinate sampling losses for TDI due to interfering substances.

MATERIALS AND METHODS

Chemicals

Chemicals were obtained from the following suppliers. 2,4-Toluenediamine (2,4-TDA), 2,6-toluenediamine (2,6-TDA), 2,4-toluene diisocyanate (2,4-TDI), HCl, min. 37% (w/w) and dimethylamine were from Merck (Darmstadt, FRG); 2,6-toluene diisocyanate (2,6-TDI) was from Janssen (Beerse, Belgium). N-Methylmorpholine, 1,4-diazobicyclo(2,2,2)-octane (DABCO), di-*n*-butylamine (DBA) and 1,6-hexamethylene diisocyanate (HDI) were from Fluka (Buchs, Switzerland). Aniline was from Mallinckrodt Chemical Works (St Louis, MI, USA). Diethylamine and phenol were from BDH (Poole, UK). Ethanol, spectroscopic grade, was from Kemetyl AB (Sweden). Pentafluoropropionic anhydride (PFPA) was from Pierce (Rockford, IL, USA). Acetonitrile, HPLC grade, toluene, HPLC grade and methanol were from Lab-Scan (Dublin, Ireland) and 1-(2-methoxyphenyl)-piperazine hydrochloride from Aldrich Chemie (Steinheim, FRG). Acetic acid anhydride was from Riedel-de Haen AG (Hannnover, FRG) and 9-(N-methylaminomethyl)-anthracene hydrochloride from Synthelec (Lund, Sweden).

Instrumental set-up

The HPLC equipment consisted of a ConstaMetric III high-pressure pump, a Spectro-Monitor III variable-wavelength UV detector (Laboratory Data Control, Riviera Beach, FL, USA) and a Rheodyne 7125 loop injector (Rheodyne, Berkeley, CA, USA). A Varian Model 3500 gas chromatograph (Varian, Walnut Creek, USA) equipped with a Varian thermionic detector (TSD) and Varian 8035 automatic on-column injector was used for the capillary GC measurements. Chromatograms were recorded on a Shimadzu C-R3A integrator (Shimadzu Corp., Kyoto, Japan). For the air sampling model HFS 113 SKC pumps (SKC Inc., Eighty Four, USA) were used.

Columns for HPLC were of stainless steel (200 mm × 5 mm I.D.), packed with 5 μm Nucleosil C_{18} (Macherey-Nagel, Düren, FRG). The GC capillary column was of fused silica, with a Chrompack CP-Sil-8CB (Chrompack, Middelburg, The

Netherlands) chemically bonded stationary phase (12 m × 0.32 mm I.D., film thickness 1.1 μm).

Generation of Test Atmospheres of TDI

Test atmospheres of TDI were generated in a ca. 8 m^3 stainless-steel test chamber described elsewhere.[58] A gas-phase permeation method was used and the permeation tubes, made of silicon rubber, contained a mixture of 2,4-TDI and 2,6-TDI (65:35). The system was capable to generate low and steady concentrations of TDI.

For the method comparison, the TDI test atmospheres ranged from 20 to 40 $\mu g/m^3$, which corresponds to approx. 50 and 100%, respectively, of the current Swedish Control Limit for TDI.

Sampling

For the sampling methods used for the collection of TDI, 30 ml midget impingers containing 10 ml of the absorption reagent were used. A volume of 10 l of air was drawn through the absorption reagents at a speed of 1 l/min. All sampling was made at the inlet of the test chamber, and all connections were glass to glass. During sampling the air temperature was 22 °C, and the relative humidity (RH) was 35–42%.

Procedure

In the method based on hydrolysis to the corresponding amine, sampling was achieved in 0.4 M HCl. After sampling 4 ml of the absorbing solution were transferred to a test tube. Three ml of toluene and 4 ml of saturated NaOH were added, and the mixture was shaken for 5 min at room temperature. Two ml of the organic layer were transferred to a new test tube where 20 μl of pentafluoropropionic anhydride (PFPA) were added, and the mixture was immediately shaken for 3 min. The excess of anhydride reagents and formed acid was removed by shaking with 2 ml of 1 M phosphate buffer (pH 7.5). An 1.5 ml aliquot of the toluene layer was evaporated at 30 °C. A specially constructed vacuum desiccator connected to an aspirating pump containing up to 25 test tubes, was used. The dry residue was solved in 150 μl of a toluene solution containing the internal standard (isobutyl chloroformate derivative of di-*n*-butylamine, 1 ng/μl) and then analysed by GC–TSD.[33] Standards of the TDA–PFPA derivative were synthesized and diluted to appropriate concentrations. Comparison was made with spiked absorbing solutions; the recovery excluding the sampling step was better than 95% ($n = 10$, RSD ca. 4%).

The amine reagents 9-(N-methylaminomethyl)-anthracene (MAMA) and 1-(2-methoxyphenyl)-piperazine (MPP) were reacted with TDI and urea derivatives were formed. In the MAMA method, the diisocyanate was reacted with a 1×10^{-4} M solution of MAMA in toluene.[22] After sampling the toluene was evaporated to dryness (temp., 50 °C) using a flow of nitrogen. By the use of a flow of nitrogen and a specially constructed heat-bed 16 impingers could be evaporated simultaneously. The urea derivatives were solved in 1 ml of the mobile phase and analysed by HPLC using UV detection at 254 nm. Standard MAMA derivatives were synthesized and diluted to appropriate concentrations. A comparison was made with spiked absorbing solutions; the recovery excluding the sampling step was better than 95% ($n = 10$, RSD ca. 4%).

The MPP method used a 2×10^{-4} M solution of the reagent in toluene.[19] After sampling, the toluene was evaporated to dryness (temp., 40 °C) using a flow of nitrogen. Fifteen μl of acetic acid anhydride in acetonitrile (50:50, v/v) was added to acetylate the excess of reagent. The solution was evaporated to dryness (temp., 40 °C) using a flow of nitrogen. The urea derivatives were solved in 1 ml methanol and analysed by HPLC using UV detection at 242 nm.

The quantitative analysis of the urea derivatives was based on peak height measurements; the PFPA derivatives were quantified by their peak heights relative to that of the internal standard. Full details of the procedures are given in the published methods.[19,22,23] All methods were applied to a mixture of 2,4-TDI and 2,6-TDI.

RESULTS AND DISCUSSION

Chromatography

The methods evaluated included sampling in acidic aqueous solution and conversion of the isocyanate to the corresponding amine and PFPA derivatization, and reaction with the amine reagents MAMA and MPP in toluene to ureas.

Chromatograms recorded for TDI at a concentration of ca. 40 μg/m^3 are shown in Figures 1–3. Acceptable separation was achieved between the TDI isomers for all three methods. However, some problems were encountered with the methods based on the amine reagents, as spurious peaks occurred. Impurities in the reagents and, for the MAMA reagent, decomposition by light may cause these artefacts. In the MPP method an excess of acetic acid anhydride caused the occurrence of extra peaks, too little caused decomposition of the column by unreacted reagent. No problems were observed in the case of PFPA derivatization with subsequent GC–TSD analysis.

For the amine derivatives UV detection was used. Higher sensitivity and better selectivity can be achieved by fluorescence (MAMA reagent) or electrochemical (MPP reagent) detection. When analysing complex workroom air double detection, e.g., a combination of UV and fluorescence detectors or UV and electrochemical detectors, allow us to establish the identity of a peak. For the method utilizing sampling in acidic aqueous solution, TSD detection was performed. Higher sensitivity and better selectivity can be achieved by the use of ECD or MS detection. However, the purpose of this study was to determine TDI at the

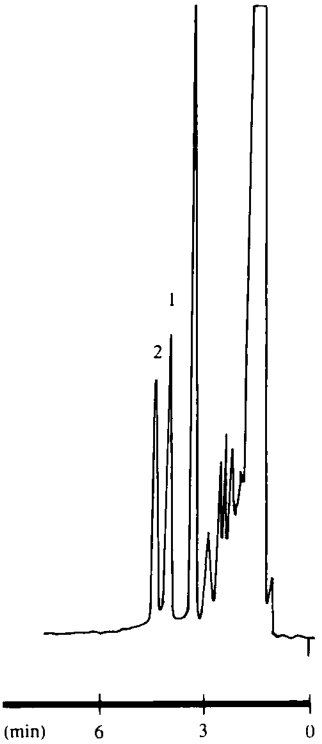


Figure 1 Chromatogram of 9-(N-methylaminomethyl)-anthracene reagent (MAMA) and urea derivatives of (1) 2,6-TDI and (2) 2,4-TDI. Eluent: acetonitrile–water (80:20 v/v), the water phase containing 3% v/v triethylamine phosphate (pH 3.0). Flow rate: 2.0 ml/min. Sample volume: 20 μl. Detection: UV at 254 nm. Isocyanates: ca. 20 μg 2,4-TDI/m^3, ca. 20 μg 2,6-TDI/m^3. 10 l air sample. 200 mm × 5 mm I.D. 5 μm Nucleosil C_{18} column.

threshold limit value; therefore, the sensitivity using UV or TSD detection was adequate.

Quantitative Analysis

As expected only minor differences between the results for the three different standard methods were found (Table 2), and as a conclusion one may point out that the methods are adequate for the determination of diisocyanates at low concentrations in air. The relative standard deviation (RSD) for the total amount of TDI determined by the different methods was 6–8%.

Five air samples were taken at each TDI concentration with duplicate determination, and the precision in the measurements was for all methods 3–8%, which was within the expected range. The differences probably were caused by minor changes in the experimental conditions, e.g., variations in pump flow-rates.

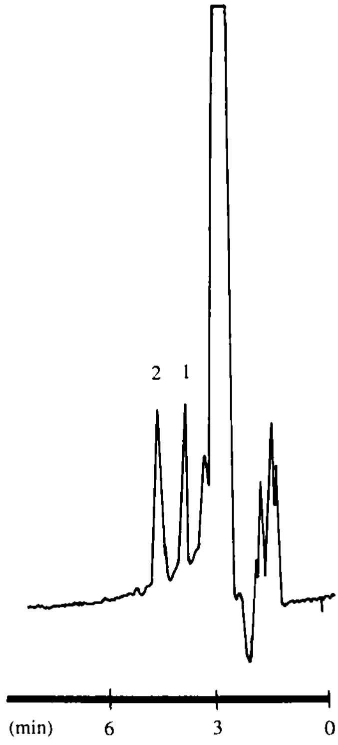


Figure 2 Chromatogram of 1-(2-methoxyphenyl)-piperazine reagent (MPP) and urea derivatives of (1) 2,6-TDI and (2) 2,4-TDI. Eluent: acetonitrile–water (60:40 v/v), the water phase containing 0.1 M sodium acetate buffer (pH 6.0). Flow rate: 1.5 ml/min. Sample volume: 10 μl. Detection: UV at 242 nm. Isocyanates: ca. 20 μg 2,4-TDI/m^3, ca. 20 μg 2,6-TDI/m^3. 10 l air sample. 200 mm × 5 mm I.D. 5 μm Nucleosil C_{18} column.

Absorption Solutions

Sampling efficiency for gaseous TDI in different absorption solutions was determined by coupling three impingers in series. Yields were obtained with dilute acids as absorbing solutions and with toluene as absorbing solution using the MAMA reagent. Sampling efficiency was calculated as the ratio of the isocyanate content in the first impinger and the sum of the contents in all three impingers (Table 3). The highest sampling efficiency was obtained for toluene (97 %). Literature values for other sampling methods utilizing toluene[11] are of the same magnitude as those in Table 3. The higher values for toluene compared with acidic absorbing solution may be ascribed to the higher solubility rate of TDI in toluene. The derivatization reactions taking place in the absorbing solution are of minor importance for the sampling efficiency, since these reactions do not occur momentary.

Pure water gives about the same sampling efficiency as dilute acids. However, at higher isocyanate contents in the air, secondary reactions may take place between free amino groups and non-hydrolyzed isocyanates in distilled water. Furthermore,

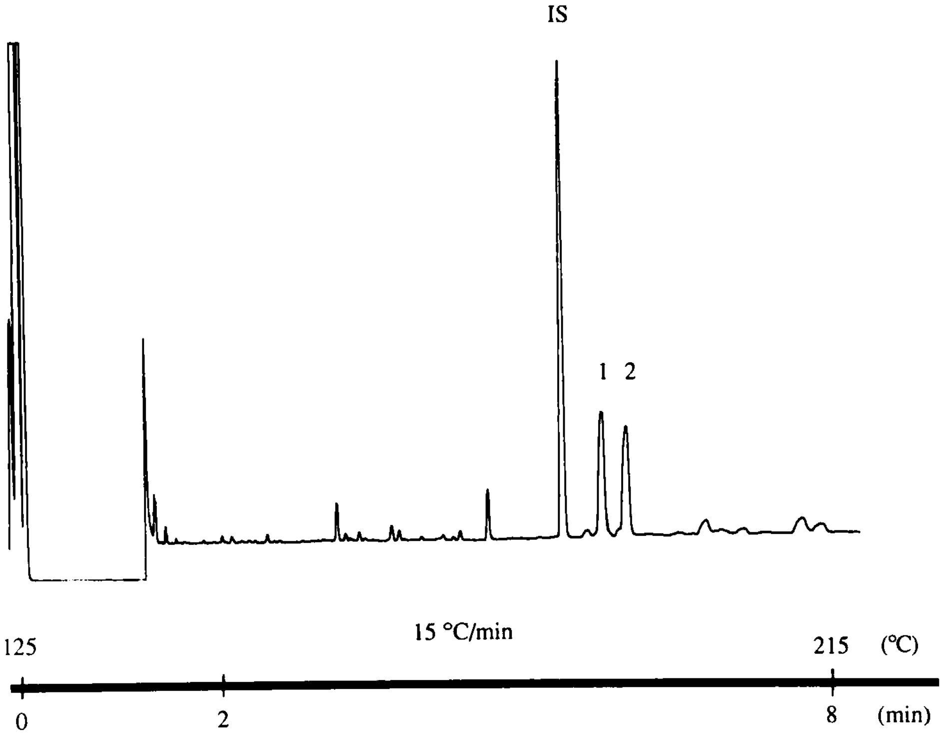


Figure 3 Chromatogram of toluene diisocyanate (TDI), after hydrolysis to the corresponding amine toluenediamine (TDA), by sampling in 0.4 M HCl and PFPA derivatization. (1) PFPA derivative of 2,6-TDA, (2) PFPA derivative of 2,4-TDA, (IS) internal standard (isobutyl chloroformate derivative of di-*n*-butylamine, 1 ng/μl). Temperature programming as shown; final temperature, 280 °C. Injector: initial temperature, 100 °C; final temperature, 250 °C (100 °C/min). TSD bead heating current, 2.2 A; bias, 4 V; temperature, 280 °C. Carrier gas: helium at 4 ml/min. Isocyanates: ca. 20 μg 2,4-TDI/m^3, ca. 20 μg 2,6-TDI/m^3. 10 l air sample. A fused silica capillary column (30 m × 0.29 mm) coated with DB-5 bonded stationary phase (J & W Scientific, Folsom, LA, USA) with a film thickness of 0.25 μm was used.

the stability of free amines is inferior to that of amine salts. Therefore, pure water is not recommended for the sampling of isocyanates. It was also investigated if the sampling efficiency for gaseous TDI could be increased by using fritted glass bubblers instead of impinger flasks. No significant improvement was registered.

Interfering Substances

When determining isocyanates in different industrial environments, other reactive compounds present in the atmosphere may interfere with the analysis. Such interfering substances are tertiary amines of technical quality used as catalysts in the production of PUR. Phenol and aniline may be present in the atmosphere when moulding using the "cold-box" method.[57]

Table 2 Comparison of three chromatographic methods for the determination of TDI in air[a]

Analytical method	*Air concentration I*				*Air concentration II*			
	2,4-TDI ($\mu g/m^3$)	*RSD* (%)	*2,6-TDI* ($\mu g/m^3$)	*RSD* (%)	*2,4-TDI* ($\mu g/m^3$)	*RSD* (%)	*2,6-TDI* ($\mu g/m^3$)	*RSD* (%)
Acidic solution (0.4 M HCl)	10.2	7	9.6	3	20.9	3	17.9	6
1×10^{-4} M MAMA in toluene	9.3	7	10.9	6	18.3	7	21.1	7
2×10^{4} M MPP in toluene	11.1	6	9.8	8	19.6	6	23.6	6

[a] Sampling in 30 ml midget impingers containing 10 ml of the used sampling solution. Air flow rate, 1.0 l/min, sampling time 10 min. Five samples at each air concentration. Temperature, 22 °C; relative humidity, 40–42 %. All values are corrected with respect to sampling efficiencies.

Table 3 Sampling efficiency for gaseous TDI in different absorbing liquids[a]

Absorption liquid	*Sampling efficiency* (%)	
	2,4-TDI	*2,6-TDI*
Toluene (0.0001 M MAMA)	97	97
3.2 M HCl	83	77
0.4 M HCl	85	80
0.04 M HCl	87	83
0.004 M HCl	88	85
0.4 M HCl/HAc	87	83
0.05 M H_2SO_4	88	83
Water	87	83

[a] Three impingers containing the reagent coupled in series by glass to glass connections. Sampling efficiency calculated as the ratio of TDI content in impinger no. 1 and sum of contents in all impingers. Air flow rate, 1.0 l/min; sampling time, 10 min; no. of samples, 5; RSD 2%. Diisocyanates: 50 μg 2,6-TDI/m^3 and 50 μg 2,4-TDI/m^3.

The influence of various substances (Table 4) was investigated using the different chromatographic methods. Amounts of interfering compounds corresponding to an air content of 1–100 ppm were dissolved in the absorbing solutions, 0.4 M HCl, 1×10^{-4} M MAMA and 2×10^{-4} M MPP, respectively. A volume of 10 l TDI-containing air was then drawn through the impinger and the yield of TDI determined. Comparison was made with tests performed with absorbing solutions free of interfering substances.

No significant losses (i.e. <5%) due to interfering substances were observed when utilizing 0.4 M HCl as absorbing solution. For MAMA and MPP, the losses were considerable and varied with the kind of interfering substance. The losses are due to the fact that the interfering substances compete with the isocyanate for MPP or MAMA in the derivatization reaction. The reactivity of the tested substances is in the order:[45] aliphatic amine > aromatic amine > alcohol > phenol. Accordingly, losses are especially expected in the presence of primary and secondary amines. The losses obtained for the three tertiary amines shown in Table 4, may be due to the fact that these are of technical quality, and are contaminated by primary and/or secondary amines. For example, the N-methylmorpholine used contained 1% morpholine, giving losses of ca. 90% at 100 ppm which may be compared with similar losses at 1 ppm for diethylamine. Several authors[12,46,47] have previously reported low yields of TDI in the presence of 1,4-diazabicyclo(2,2,2)-octane (DABCO) in the gas phase. Whether the reaction in these cases took place in the gas phase in the sampling device, due to amine catalysed surface reactions with silanol groups present, or with impurities in the amine used has not been investigated.

During sampling, water vapour can be dissolved in the toluene and cause hydrolysis of TDI. However, tests with water-saturated toluene (MAMA reagent) gave no significant reduction in yield compared to that obtained with sodium-dried toluene in the absorbing solution. Adding DABCO, a catalyst for the hydrolysis of TDI, to water-saturated and sodium-dried toluene, respectively, was

Table 4 Sample losses for TDI due to interfering substances[a]

Interfering compound	*Concentration*[b] *(ppm)*	*Losses due to interfering substances*					
		Method I		*Method II*		*Method III*	
		2,4-TDI	*2,6-TDI*	*2,4-TDI*	*2,6-TDI*	*2,4-TDI*	*2,6-TDI*
Diethylamine	1	*	*	30	34	90	90
	10	*	*	98	99	99	99
	100	*	*	99	99	100	100
Dimethylethylamine	1	*	*	*	*	*	*
	10	*	*	*	*	*	*
	100	*	*	*	*	27	26
N-methylmorpholine	1	*	*	*	*	10	5
	10	*	*	6	6	26	26
	100	*	*	16	18	93	93
1,4-Diazabicyklo(2,2,2)-octane (DABCO)	1	*	*	*	*	*	*
	10	*	*	31	28	12	9
	100	*	*	40	32	53	52
1,4-Diazabicyklo(2,2,2)-octane (DABCO) from a solution of 33% in polyol	1	*	*	12	12	*	*
	10	*	*	14	17	11	11
	100	*	*	48	44	45	47
Aniline	1	*	*	*	*	*	*
	10	*	*	*	*	*	*
	100	*	*	*	*	*	*
Phenol	1	*	*	*	*	*	*
	10	*	*	*	*	*	*
	100	*	*	*	*	*	*
Ethanol	1	*	*	*	*	*	*
	10	*	*	10	9	*	*
	100	*	*	22	19	*	*

[a] *Method I*, sampling in 0.4 M HCl, analysis by GC–TSD; *Method II*, sampling in toluene with 2×10^{-4} M MPP reagent; *Method III*, sampling in toluene with 1×10^{-4} M MAMA reagent. All sampling in 30 ml midget impingers containing 10 ml of each sampling solution. Air flow rate, 1.0 l/min; sampling time, 10 min. Diisocyanate concentration: 70 μg 2,4-TDI/m^3 and 70 μg 2,6-TDI/m^3.

[b] Corresponding air concentrations.

*No significant losses (i.e. <5%).

also tested with MAMA as reagent. No significantly diminished yield of TDI (at a level of 10 ppm DABCO) due to hydrolysis was found. Further experiments are needed before the possibility of amine-catalysed hydrolysis can be ruled out as a source of losses. The results in Table 4 are based on experiments with single interfering substances, but combined effects may be expected in the presence of several substances. Thus, the effect of ethanol was found to be insignificant. However, if a tertiary amine is also present, losses of TDI can occur, since amines act as catalysts for the reaction between alcohols and isocyanates.

It can be concluded that, when using the amine reagents (MPP in toluene and MAMA in toluene), low values for TDI are obtained, if the atmosphere contains primary and secondary amines. Such amines are, e.g., present in the catalyst added during the manufacture of PUR. However, it is felt that the risk of atmospheric pollution with reactive amines is small in this case, since the main part of these is consumed by reaction with isocyanates in the liquid phase. Because of the risk of low isocyanate values when using these amine reagents, it is recommended also to apply sampling in acidic solution, if the presence of primary and secondary amines is suspected. A further advantage is that when using sampling in acidic solutions, the nature of polluting amines can be determined, which is of interest from a hygienic point of view.

CONCLUSIONS

Sampling and analysis of TDI in industrial environments is a difficult task, but methodological problems can be diminished by skilled operators and adequate analytical routines. Although the standard methods using amine reagents are sensitive and selective, the presence of interfering chemicals, e.g., primary and secondary amines, may substantially disturb the analysis.

To obtain correct informtion about the isocyanate content in such complex workroom air, it is therefore recommended to simultaneously use two methods based on a different principle. One method may be sampling in acidic solution, derivatization of the formed amine with a reagent such as PFPA, and analysis by means of GC–TSD, the other method may be sampling in toluene with an amine reagent such as MAMA or MPP, and analysis by HPLC.

Acknowledgements

We thank Åsa Amilon and Kristina Fureby at the Department of Occupational and Environmental Medicine, University Hospital, Lund. This investigation was supported by a grant from the Swedish Work Environment Fund (grant 88-0161).

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