



# Standard Test Method for Determination of Gaseous Hexamethylene Diisocyanate (HDI) in Air with 9-(N-methylaminomethyl) Anthracene Method (MAMA) in the Workplace<sup>1</sup>

This standard is issued under the fixed designation D6562; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of gaseous hexamethylene diisocyanate (HDI) in air samples collected from workplace and ambient atmospheres. The method described in this test method collects separate fractions. One fraction will be dominated by vapor, and the other fraction will be dominated by aerosol. It is not known at the present time whether this represents a perfect separation of vapor and aerosol, and in any case, there are not separate exposure standards for vapor and aerosol. Therefore, in comparing the results for isocyanate against a standard, results from the two fractions should be combined to give a single total value. The reason for splitting the sample into two fractions is to increase analytic sensitivity for the vapor fraction and also to give the hygienist or ventilation engineer some information concerning the likely state of the isocyanate species. The analyses of the two fractions are different, and are provided in separate, linked, standards to avoid confusion. This test method is principally used to determine short term exposure (15 min) of HDI in workplace environments for personal monitoring or in ambient air. The analysis of the aerosol fraction is performed separately, as described in Test Method [D6561](#).

1.2 Differential air sampling is performed with a segregating device.<sup>2</sup> The vapor fraction is collected on a glass fiber filter (GFF) impregnated with 9-(N-methylaminomethyl) anthracene (MAMA).

1.3 The analysis of the gaseous fraction is performed with a high performance liquid chromatograph (HPLC) equipped with ultraviolet (UV) and fluorescence detectors.

1.4 The range of application of this test method, using UV and fluorescence detectors both connected in serial, has been validated from 0.006 to 1.12  $\mu\text{g}$  of monomeric HDI/2.0 mL of desorption solution, which corresponds to concentrations equivalent to 0.0004 to 0.075  $\text{mg}/\text{m}^3$  of HDI based on a 15-L air sample. Those concentrations correspond to a range of vapor phase concentrations from 0.06 ppb(V) to 11 ppb(V) and cover the established threshold limit value (TLV) value of 5 ppb(V).

1.5 The quantification limit for the monomeric HDI, using the UV detection, has been established as 0.012  $\mu\text{g}/2$  mL of desorption solution and as 0.008  $\mu\text{g}/2$  mL, using the fluorescence detector. These limits correspond to 0.0008  $\text{mg}/\text{m}^3$  and 0.0005  $\text{mg}/\text{m}^3$  respectively for an air sampled volume of 15 L. These values are equal to ten times the standard deviation (SD) obtained from ten measurements carried out on a standard solution in contact with the GFF, whose concentration of 0.02  $\mu\text{g}/2$  mL is close to the expected detection limit.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. See Section 9 for additional hazards.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>3</sup>

[D1193 Specification for Reagent Water](#)

[D1356 Terminology Relating to Sampling and Analysis of Atmospheres](#)

[D1357 Practice for Planning the Sampling of the Ambient Atmosphere](#)

[D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps](#)

[D6561 Test Method for Determination of Aerosol Monomeric and Oligomeric Hexamethylene Diisocyanate \(HDI\)](#)

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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<sup>2</sup> The sampling device for isocyanates is covered by a patent held by Jacques Lesage et al, IRSST, 505 De Maisonneuve Blvd. West, Montreal, Quebec, Canada. If you are aware of an alternative to this patented item, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

in Air with (Methoxy-2-phenyl-1) Piperazine (MOPIP) in the Workplace

## 2.2 Other Standard:

Sampling Guide for Air Contaminants in the Workplace<sup>4</sup>

## 3. Terminology

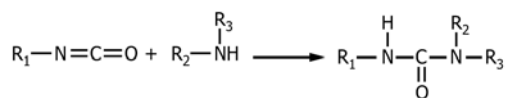
3.1 For definitions of terms used in this test method, refer to Terminology **D1356**.

## 4. Summary of Test Method

4.1 Vapor and aerosol fractions are sampled simultaneously by using a segregating sampling device. The aerosols are collected on a polytetrafluoroethylene (PTFE) filter while the gaseous fraction is being adsorbed on the second filter made of glass fiber impregnated with MAMA.

4.2 The analysis of the oligomer in the aerosol fraction is performed separately in accordance with the procedure described in Test Method **D6561**.

4.3 Diisocyanates present as vapors react with the secondary amine function of the MAMA, impregnated on the GFF to form a urea derivative (**1,2**) as shown in **Fig. 1**.<sup>5</sup>



**FIG. 1**

Desorption of the GFF is done by using a solution mixture of 67 % N,N-dimethylformamide and 33 % of a 30:70 buffer-acetonitrile mixture. Monomeric and oligomeric diisocyanates are separated by using a reversed phase HPLC column, followed by UV (254 nm) and fluorescence detectors (254-nm excitation and 412-nm emission) in series (**3**).

4.4 Concentration of urea derivative contained in the samples is calculated by using an external standard of the appropriate urea derivative.

## 5. Significance and Use

5.1 HDI is mostly used in the preparation of paints. For the last ten years, the use of isocyanates and their industrial needs have been in constant growth.

5.2 Diisocyanates and polyisocyanates are irritants to skin, eyes, and mucous membranes. They are recognized to cause respiratory allergic sensitization, asthmatic bronchitis, and acute respiratory intoxication (**4-7**).

5.3 The American Conference of Governmental Industrial Hygienists (ACGIH) has adopted a threshold limit value - time weighted average (TLV - TWA) of 0.005 ppm (V) or 0.034 mg/m<sup>3</sup> (**8**). The Occupational Safety & Health Administration of the U.S. Department of Labor (OSHA) has not listed a permissible exposure limit (PEL) for HDI (**9**).

<sup>4</sup> Available from Institut de recherche en sante et en securite du travail du Quebec, Laboratory Division, Montreal, IRSST.

<sup>5</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

5.4 Due to its low LOD and low required volume (15 L), this test method is well suited for monitoring of respiratory and other problems related to diisocyanates and polyisocyanates. Its short sampling times are compatible with the duration of many industrial processes, and its low detection limit with the concentrations often found in the working area.

## 6. Interferences

6.1 Any substances, including strong oxidizing agents, that can react with the MAMA reagent impregnated on the GFF can affect the sampling efficiency.

6.2 Any compound that has the same retention time as the hexamethylene diisocyanate 9-(N-methylaminomethyl) anthracene (HDIU) derivative and contributes to the UV signal is an interference. Chromatographic conditions can sometimes be changed to eliminate an interference. The response factor (RF) ratio from the UV and fluorescence detectors gives a good indication to the analyst about the possibility of an interference.

## 7. Apparatus

### 7.1 Sampling Equipment:

7.1.1 *Personal Sampling Pump*—Equipped with a flow-monitoring device (rotameter, critical orifice) or a constant-flow device capable of drawing 1.0 L/min through the sampling device for a period of at least 4 h.

7.1.2 *Double Filter Sampling Device*, 37 mm in diameter, three-piece personal monitor, plastic holder loaded with a PTFE filter close to the mouth, followed by a GFF impregnated with MAMA and by a plastic back-up pad.<sup>6</sup> The GFF is impregnated with an amount of MAMA in the range from 0.07 to 0.25 mg.

7.1.3 *Flow Measuring Device*, used in accordance with Practice **D5337**.

### 7.2 Analytical Equipment:

7.2.1 *Liquid Chromatograph*, an HPLC, equipped with a UV (254-nm wavelength) and fluorescence detectors (412-nm emission and 254-nm excitation) and equipped with an automatic or manual sampling port injection.

7.2.2 *Liquid Chromatographic Column*, an HPLC stainless steel column, capable of separating the urea derivatives. This test method recommends a 150 by 3.2-mm internal diameter stainless steel column packed with 3 μm C-18, or an equivalent column.

7.2.3 *Electronic Integrator*, or any other effective method for determining peak area counts.

7.2.4 *Analytical Balance*, with a precision of ± 0.0001 g.

7.2.5 *Microsyringes and Pipets*—Microsyringes are used in the preparation of urea derivatives and standards. An automatic pipet, or any equivalent equipment, is required for sample preparation.

<sup>6</sup> The sole source of supply of the apparatus known to the committee at this time is Omega Specialty Instrument, Chelmsford, MA and is prepared in accordance with Patent No. 4 961 916 (**10**). If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

7.2.6 *pH Meter*, or any equivalent device capable of assaying a pH range between 2.5 and 7.

7.2.7 *Three-neck Flask*, for the synthesis of the HDIU standard (see 8.13).

7.2.8 *Magnetic Stirrer*, or any other equivalent device.

7.2.9 *Glass Jars*, 30 mL, and lids, capable of receiving 37 mm filters, used for sample desorption.

7.2.10 *Reciprocating Shaker*, or any other equivalent device.

7.2.11 *Vacuum Filtration System*, filter with 0.22- $\mu$ m pore size polyamide filters, or any equivalent method.

7.2.12 *Syringe Operated Filter Unit*, syringes with polyvinylidene fluoride 0.22- $\mu$ m pore size filter unit, or any equivalent method.

7.2.13 *Injection Vials*, 1.5 mL vials with PTFE-coated septums.

7.2.14 *Bottle*, amber bottle with cap and PTFE coated septum for conservation of stock and standard solutions of HDIU, or any equivalent equipment.

## 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>7</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, water shall be reagent water as defined by Type 2 of Specification D1193, HPLC grade.

8.3 *Acetonitrile (CH<sub>3</sub>CN)*, HPLC grade.

8.4 *Buffer*—Transfer 30 mL of triethylamine (see 8.14) into a 1-L volumetric flask, and dilute to volume with HPLC grade water. Acidify the solution to pH = 3 with phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) (see 8.11). Filter the buffer under vacuum with a 0.22- $\mu$ m pore size filter.

8.5 *Desorption Solution*, a solvent mixture of 67 % (v/v) of dimethylformamide (see 8.7) and 33 % (v/v) mobile phase (see 8.10).

8.6 *Dichloromethane*, reagent grade.

8.7 *N,N-Dimethylformamide*, reagent grade.

8.8 *Helium (He)*, high purity.

8.9 *9-(N-Methylaminomethyl) Anthracene (MAMA) (F.W. 221.31)*, 99 % purity.

8.10 *Mobile Phase*, a solvent mixture of 75 % (v/v) of acetonitrile (CH<sub>3</sub>CN) (see 8.3) and 25 % (v/v) of buffer (see 8.4).

8.11 *Phosphoric Acid (H<sub>3</sub>PO<sub>4</sub>)*, reagent grade

8.12 *Hexamethylene Diisocyanate (HDI)*, (F.W. 168), 98 % purity.

8.13 *Hexamethylene Diisocyanate 9-(N-methylaminomethyl) Anthracene Derivative (HDIU)* (see 11.2.1).

8.14 *Triethylamine*, purity 98 % min.

## 9. Hazards

9.1 **Warning**—Diisocyanates are potentially hazardous chemicals and are extremely reactive. Refer to material safety data sheets for reagents.

9.2 **Warning**—Avoid exposure to diisocyanate and solvents. Sample and standard preparations should be done in an efficient operating hood. For remedial statement, see Ref (11).

9.3 **Warning**—Avoid skin contact with isocyanates and all solvents. N,N-Dimethylformamide is highly toxic. Chronic effects include damage to liver and kidneys. See Ref (12).

9.4 **Warning**—Wear safety glasses at all times and other laboratory protective equipment if necessary.

## 10. Sampling

10.1 Refer to Practices D1357 and D5337 for general information on sampling.

10.2 This test method recommends sampling in accordance with the method described in the Ref (10, 11, 13).

10.3 Equip the worker, whose exposure is to be evaluated, with a filter holder connected to a belt-supported sampling pump. Place the filter holder pointing downward, if possible, at an optimum angle of 45° from horizontal in the breathing zone of the worker. Draw air through the sampling device and collect 15 L at a rate of approximately 1.0 L/min.

10.4 For stationary monitoring, use a tripod or any other support to locate the sampler in a general room area at a height equivalent to the breathing zone.

10.5 A field blank is used to monitor contamination during the combined sampling, transportation, and storage process. Open the field blanks in the environment to be sampled, and immediately close them. Process field blanks in the same manner as samples. Submit at least one field blank for every ten samples.

10.6 Immediately after sampling, open the cassette, withdraw the PTFE filter, place it in a glass jar containing 5 mL of MOPIP derivatization solution (see Test Method D6561), and close the jar. This filter is used to analyze the aerosol fraction of diisocyanates (see Test Method D6561).

10.7 Close the cassette, leaving the GFF and the plastic pad support. The GFF is used to analyze the vapor fraction of diisocyanates.

10.8 Send the jars and the cassettes to be analyzed to the laboratory. Keep away from light.

## 11. Calibration and Standardization

11.1 *Sample Pump Calibration*—Calibrate the sampling pump (see 7.1.1) with a cassette (see 7.1.2) between the pump

<sup>7</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

and the flow measuring device (see 7.1.3), in accordance with Practice D5337. Calibrate the pump before and after sampling. If the flow rate after the sampling differs by more than 5 % from the flow rate before sampling, invalidate the sample.

### 11.2 Reference Standards:

#### 11.2.1 HDIU Derivative Synthesis:

11.2.1.1 In a 25-mL volumetric flask, transfer 325  $\mu\text{L}$  of HDI (see 8.12) (2 mmoles) and dilute to volume with dichloromethane (see 8.6).

11.2.1.2 In a 50-mL volumetric flask, dissolve approximately 1.3 g (6 mmoles) of MAMA (see 8.9). Transfer the solution into the three-neck flask.

11.2.1.3 Slowly add the HDI solution (see 11.2.1.1) to the MAMA solution contained in the pre-heated (25°C) three-neck flask. Using a magnetic bar, stir the solution for a period of 60 to 90 min.

11.2.1.4 Cool down the resulting solution on crushed ice.

11.2.1.5 Filter on a medium-speed filter paper or any equivalent filter.

11.2.1.6 In a beaker or flask, dissolve the precipitate in a warm solvent such as dichloromethane (see 8.6). Place the container into an ice bath for recrystallization, and filter, using a medium-speed filter paper or any equivalent filter.

11.2.1.7 Confirm the urea derivative with a mass spectrum: the HDI-MAMA has a molecular weight of 610, and check its purity by comparison of the melting point (200°C).

11.2.1.8 The conversion factor from HDIU to HDI is 0.2754

11.2.2 *Stock Standard Solution of HDIU*—Weigh precisely approximately 12.5 mg of HDIU, transfer into a 100-mL volumetric flask, and dilute to volume with N,N-dimethylformamide (see 8.7). Store in an amber bottle. Calculate the HDI concentration by using the conversion factor from HDIU to HDI.

### 11.3 Blanks:

11.3.1 The field blank described in 10.5 is prepared and analyzed as a sample (see 12.1).

11.3.2 A blank laboratory is used to check contamination that may occur during laboratory manipulations. Use blank laboratory, and process as a sample (see 12.1).

11.3.3 Use desorption solution as a solution blank, and process as a sample (see 12.1).

### 11.4 Quality Controls:

11.4.1 Using a 25- $\mu\text{L}$  microsyringe, draw 15  $\mu\text{L}$  of the HDIU stock solution and spike onto an impregnated GFF. Transfer the GFF into a glass jar, and let it dry with an open lid. Process as a sample (see 12.1). Dilute the HDIU stock solution in a proportion of 1/10 with the desorption solution, and then proceed in the same manner as the previous preparation.

11.4.2 In an analysis sequence, analyze both quality controls at least once.

### 11.5 Calibration Curve:

11.5.1 To prepare working standards, prepare at least three dilutions from the standard stock solutions (see 11.2.2) within the concentration range from 0.006 to 1.12  $\mu\text{g}$  of monomeric HDI/2mL of desorption solution. Those concentrations cover the range from 0.0004 to 0.075  $\text{mg}/\text{m}^3$  for an air sampled volume of 15 L.

11.5.2 In a glass jar containing a GFF, transfer 2 mL of each standard solution. Process standards as samples (see 12.1)

11.5.3 Analyze by high performance liquid chromatography in accordance with the method described in 12.2.

11.5.4 Prepare the calibration curve by plotting peak area values against micrograms of HDI per 2 mL of desorption solution. A coefficient of correlation equal or greater than 0.995 must be achieved.

11.5.5 In daily routine procedures, inject one working standard every ten samples to check the stability of the instrument response.

## 12. Procedure

### 12.1 Sample Preparation:

12.1.1 Using tweezers, remove the GFF from the cassette and transfer into a glass jar. Process field and laboratory blanks in the same manner as samples.

12.1.2 Using a pipet or any equivalent device, add 2.0 mL of desorption solution (see 8.5) to the glass jar. Close the jar tightly.

12.1.3 Shake the samples for 30 min on a reciprocating shaker (see 7.2.10), or any equivalent device. Keep away from light.

12.1.4 Filter the resulting solution with a 0.22- $\mu\text{m}$  polyvinylidene fluoride filter (see 7.2.12) mounted on a disposable syringe. Transfer a fraction of the sample to an injection vial (see 7.1.13).

12.1.5 Analyze sample, blank, and quality control solutions in accordance with the conditions described in 12.2. Use the same injection technique and injection volume for samples, blanks, quality controls, and external standards.

12.1.6 Calculate the monomeric HDI concentration in the sample, as specified in Section 13.

### 12.2 HPLC Conditions:

12.2.1 Analyze by high performance liquid chromatography, using a suitable column (see 7.2.2) and the mobile phase, as described in 8.10. Typical conditions are as follow:

Column Temperature:	Room Temperature (20 to 25°C)
Flow rate:	0.6 mL/min.
Ultraviolet:	254 nm
Fluorescence:	254 nm excitation 412 nm emission
Injection volume:	15 $\mu\text{L}$

Analytical conditions serve as a guideline and may need to be modified, depending on instrumentation, column condition, detectors, and so forth.

## 13. Calculation

13.1 Calculate the concentration of the monomeric HDI, using the following equation:

$$M_{\text{mono}} = (A - B)/m \quad (1)$$

$$C_{\text{mono}} = M_{\text{mono}}/V \quad (2)$$

where:

- $M_{\text{mono}}$  = mass of the HDI monomer in sample ( $\mu\text{g}$ ),
- b and m = Y intercept and slope, respectively, obtained from the linear regression,
- A = area count of the peak.

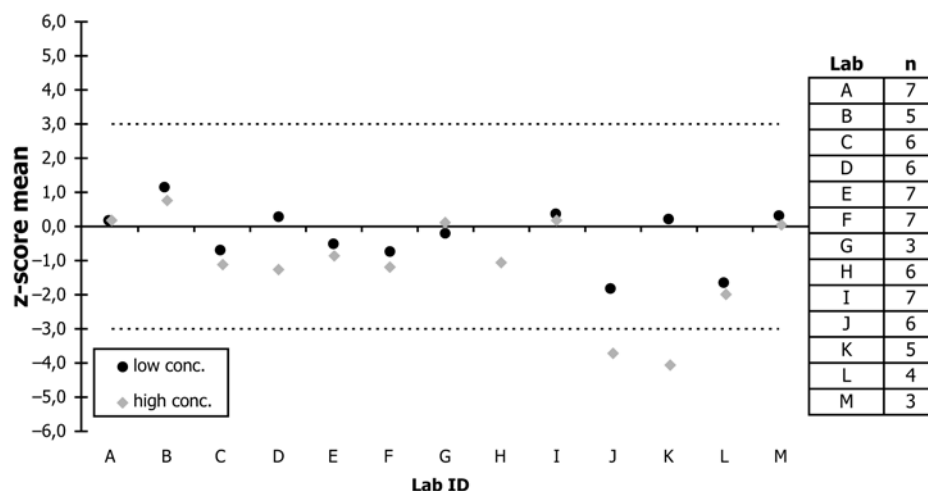


FIG. 2 Means of the Z-Scores Obtained by 13 Laboratories after  $n \geq 3$  Participations to an Interlaboratory Evaluation

$C_{mono}$  = concentration of HDI monomer in air ( $mg^3/m^3$ ),  
and  
 $V$  = volume of air sampled (L).

#### 14. Report

14.1 Report the following information:

14.1.1 Concentration of monomeric HDI in  $mg/m^3$  obtained from Eq 2, which is added to the result of monomeric HDI obtained in the same sample by Test Method D6561.

#### 15. Precision and Bias

15.1 Precision:

15.1.1 Precision on a Complete Calibration Curve (same lab, same operator)—To measure the relative standard deviation (RSD) and the recovery percentage, six concentration levels were tested six times. A GFF was placed in the standard solution to evaluate the possibility of potentially interfering compounds being extracted from the filter, or isocyanate becoming irreversibly bound to the filter. The working standards were prepared in accordance with the procedure in 11.5 and covers the following range: 0.006, 0.056, 0.140, 0.280, 0.561, and 1.12  $\mu g/2$  mL of desorption solution. Using both UV and fluorescence detectors, the RSD for concentrations within the range from 0.0004 and 0.075  $mg/m^3$  was equal to 0.022 and 0.007 respectively.

15.1.2 Recovery Percentage—To evaluate the recovery percentage, the standard solutions and equivalent standard solutions, which were in contact with the GFF, were analyzed. The average recovery percentage ( $n = 42$ ) for all seven HDI concentrations were  $101.2 \pm 0.079$  for the UV detector and  $102.4 \pm 0.027$  for the fluorescence detector.

15.1.3 Precision of the Apparatus—The precision of the apparatus was calculated from ten measurements carried out on a concentration equivalent to 0.0034  $mg/m^3$ . The RSDs for the UV and the fluorescence detection were respectively 0.02 and 0.004.

15.1.4 Repeatability of the Daily Quality Controls (same lab, different operators, same lab procedure, two different concentrations)—Compilation of daily quality controls, prepared as described in 11.4, was done on two different concentrations, over a period of 24 months, including three different operators. For the HDI working standard corresponding to 0.034  $mg/m^3$ , the RSD was 0.1034 for the UV detector, and for a concentration of 0.003  $mg/m^3$ , the RSD was 0.1015, using the fluorescence detector.

15.1.5 Results of an Interlaboratory Evaluation—The RSD calculated from an average of ten participating laboratories over seven rounds is 0.27 for low-range concentrations ( $n = 71$ ) and is 0.17 for high-range concentration ( $n = 75$ ).

15.2 Accuracy—Fig. 2 contains the average of the z-scores of 13 different laboratories that participate to an on-going interlaboratory evaluation using this test method. The evaluation is performed once a year.

#### 16. Keywords

16.1 air monitoring; dual filter sampling system; hexamethylene diisocyanate; high-performance liquid chromatography; 9-(N-methylaminomethyl) anthracene; sampling and analysis; workplace atmospheres

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