Size selective isocyanate aerosols personal air sampling using porous plastic foams

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Abstract. As part of a European project (SMT4-CT96-2137), various European institutions specialized in occupational hygiene (BGIA, HSL, IOM, INRS, IST, Ambiente e Lavoro) have established a program of scientific collaboration to develop one or more prototypes of European personal samplers for the collection of simultaneous three dust fractions: inhalable, thoracic and respirable. These samplers based on existing sampling heads (IOM, GSP and cassettes) use Polyurethane Plastic Foam (PUF) according to their porosity to support sampling and separator size of the particles. In this study, the authors present an original application of size selective personal air sampling using chemical impregnated PUF to perform isocyanate aerosols capturing and derivatizing in industrial spray-painting shops.

1. Introduction
Polyurethane-based paints are widely used in the car industry owing to their outstanding technical features such as durability, color stability and resistance to abrasion, chemicals and weather extremes. Isocyanates (NCO) are important reactive chemicals, the so-called hardener of two-component polyurethane-based paints, and a leading cause of occupational asthma in industrialized countries [1-3]. Exposure to NCO can occur by inhalation and by dermal contact [4]. Sampling and analysis of airborne NCO is not easy. NCO occurs in a variety of chemical forms, such as, monomers, oligomers and mixtures of all these forms. NCO oligomers and prepolymers are commonly used in car spray-painting paints, as they are less volatile than the monomers. NCO occurs in a variety of physical form, for example, vapor, aerosol and liquids. A sampling method for NCO in the workplace should be suitable for all physical forms. Contrary to the American Conference of Governmental Industrial Hygienists (ACGIH) who recommended a threshold limit value (TLV) of 34 µg.m\(^{-3}\) (5 ppb) only on HDI monomer and nothing about prepolymer form [5], European countries like the UK Health and Safety Executive (HSE) has set a long-term workplace exposure limit (WEL, 8 h time weighted average reference period) of 20 µg.m\(^{-3}\) of total isocyanate –NCO group and a short-term limit (15 min) of 70 µg.m\(^{-3}\). Swiss regulation also adopts a short-term limit (15 min) of 20 µg.m\(^{-3}\) of total isocyanate –NCO group [6].

Because of the reactivity of NCO, sampling in the workplace is commonly carried out by chemical reaction of NCO with derivatization reagent to produce stable derivative that is easier to trap on the supports. In general, the sampling device recommended in the UK method MDHS 25/3 [7] depends upon the physical form of the isocyanate. Isocyanate vapor can be sampled by an impregnated filter and aerosols are most effectively sampled by a combination of impinger containing 1-(2-
methoxyphenyl)piperazine (MP) solution backed up by a MP impregnated filter. However, it is widely acknowledged that impingers are difficult to use for personal monitoring and several groups are currently researching the limitations and applicability of filter only sampling methods because they are easier to use.

The exposure of workers to aerosols and particles is a major concern for occupational health. Polyurethane Plastic Foam (PUF) provides a low-cost method for separating dust into health-related fractions in accordance with recognized sampling conventions (ISO/TR/7708 [8], EN481 [9]). As part of a European project (SMT4-CT96-2137), various European institutions specialized in occupational hygiene (BGIA, HSL, IOM, INRS, IST, Ambiente e Lavoro) have established a program of scientific collaboration to develop one or more prototypes of European personal samplers for the collection of particles according to the penetration curves. These samplers based on existing sampling heads (IOM, GSP and cassettes) use PUF according to their porosity to support sampling and size separation of the particles. Conventional applications of size selective PUF samplers were focused on general occupational dust monitoring [10-11], welding fumes [12], mining dust, metals, bio-aerosols [13-14], etc.

The health effect of isocyanate exposure in the gas phase and in different size fractions are not well understood and only a few papers report the results of field situation, depending spray-painting or thermal degradation operation, using denuder and cascade impactor sampler [15-18]. In addition, it is not known whether the selection characteristics of these methods comply with the CEN/ISO convention for inhalable, thoracic and respirable aerosol. Impinger, though offering an immediate solubilizing and derivatizing environment by the use of solvent, suffer from the inability to capture particles much smaller than 2 µm. In contrary, filters collect more effectively particulate, but because the lack of solvent, shown a less efficiency for the vapor phase reaction. In field situation of spray-painting shops and focus on the monitoring of commercial two-component paints based on isocyanates prepolymers, only less than 0.5% of monomer HDI remains in the conventional paint composition. In fact, impregnated filter method was sufficiently robust for field evaluation [17]. Concerning impregnated filter method, the only sensitive problem was the necessity to dissolve the derivatized isocyanates collected on the filters in acetonitrile (ACN) solution immediately after sampling and sampling time do not excess one hour.

In this study, an original application of size selective personal air sampling using chemical impregnated PUF to perform isocyanate aerosols capturing and derivatizing in paint shops, comply with the CEN/ISO convention, was presented.

2. Methodology

2.1. Reagents

1-(2-methoxyphenyl)piperazine (MP) was purchased from Aldrich (Chemie Brunschwig, Basel, Switzerland) and Hexamethylen diisocyanate (HDI) monomer from Eastman Kodak (Rochester, N.Y.). Desmodur N-100, which contains 99.3% of prepolymers, predominately in the form of biuret trimer of HDI, and Desmodur N-75, which contains also more than 99.2% of prepolymers, predominately in the form of isocyanurate trimer of HDI were obtained from Bayer Chemical (Bayer AG, Zürich, Switzerland). Both isocyanate reagents contain less than 0.5% of HDI. Local, commercial, two-component paints based on Desmodur N-75 (PolyJal, Jallut SA, Lausanne, Switzerland) were used for the generation of the aerosol. Glass fibre filters (GF/A) were obtained from Whatman and filter holders, Swinnex-type, 25-mm diameter were from Millipore.

2.2. Impregnation of filter, PUF and sample preparation

An Andersen impactor (Ambient Particle Sizing Sampler, model 2000, Andersen Inc., USA) equipped with glass fiber filters (Whatmann filter 934AH Ø 81mm) impregnated with MP solution (3 ml of solution of 1 mg.ml⁻¹ of MP in anhydrous toluene, which corresponds to about 3 mg of reagent on the filter) was used at 28.3 l.min⁻¹ (1 Cubic Feet per minute or CFM) for the collecting and the weighing.
of dust according to the size of particles. Conventional size-selective samplers such as cyclone (GS-3 Respirable Dust Cyclone, SKC), at flowrate 2.75 l.min\(^{-1}\) for 50% cut-point of 4.0 µm, and two-stage virtual impactor (Respicon Particle Sampler, Model 8522, TSI), inhalable fraction, cut point at 100 µm, thoracic fraction, cut point at 10 µm, respirable fraction, cut point at 4 µm, at flow rate of 3.11 l.min\(^{-1}\), were also used with the same derivatizing MP impregnation treatment. Porous polyurethane foam plugs of nominal porosity, 30 Pores Per Inch (PPI), black foam and 90 PPI white foam at different diameters (15 and 29.5 mm) were delivered by the coordinator to participants during the European project. The PUF plugs were ready to be adapted to conventional sampling heads such as IOM and BGIA GSP samplers. PUF plugs were first washed with detergent and water, then with distilled methanol by Sohxlet over night, dried in an oven at 100°C during 1 hour and finally impregnated with MP solution, similar to filter impregnation.

2.3. PUF sampling heads

The PUF sampling heads are schematically shown in Figure 1. It consists of a commercial glass fibre filter of 25 mm diameter (Gelman, Skan AG), which is mounted on a glass tube containing two PUF plugs at different porosity (35 and 90 PPI). These prototypes are designed based on the characteristics of conventional sampling heads cassette, IOM and GSP.

![Figure 1: PUF sampling heads prototype 1(top), flow rate 2 l/min, foam plugs (15 mm diameter): 35 PPI (black), thickness 9 mm and 90 PPI (white), thickness 12.5 mm, inlet cone orifice 6 mm diameter and PUF sampling heads prototype 2 (bottom), flow rate 3.5 l/min, foam plugs (29.5 mm diameter): 35 PPI (black), thickness 25 mm and 90 PPI (white), thickness 25 mm, inlet cone orifice 8 mm diameter. The conventional IOM and BGIA GSP samplers were placed at the right of the picture for the comparison.](image-url)
The diameters and thicknesses of foam plugs are set according to the sampling flow rate:

1. **Prototype 1**: flow rate 2 l/min, foam plugs (15 mm diameter): 35 PPI, thickness 9 mm and 90 PPI, thickness 12.5 mm, inlet cone orifice 6 mm diameter.
2. **Prototype 2**: flow rate 3.5 l/min, foam plugs (29.5 mm diameter): 35 PPI, thickness 25 mm and 90 PPI, thickness 25 mm, inlet cone orifice 8 mm diameter.

The pore size, dimensions of foam plugs, aerosol penetration characteristics and applications in particle size-selective sampling were already investigated in previous studies [10-14], and the entry characteristics of practical workplace aerosol samplers in relation to the health-related aerosol fraction was reported based on measurements of aerodynamic optical particle counter [19]. In this study, the authors prefer to adopt the pragmatic solutions [20] of inlet cone orifice to standard GSP sampler, with the inlet diameter was adapted to flow rate. Side-by-side comparisons of samplers were made using “inhalable mass fraction” of aerosols by weight.

### 2.4. Analysis of HDI and prepolymer

Analysis of derivatized HDI and prepolymer was based on MDHS 25/3 method [7, 17, 21, 22] by HPLC with UV and electrochemical detection (EC), the reference procedure for evaluation of the developed solid PUF samplers. Inter-method comparisons to appraise the performances and limits of the reference method were made within the context of previously published papers [21-23]. The deviations of analytical HPLC method used in this laboratory were 3%. Calculation of the total equivalent NCO group for the HDI prepolymer in terms of mass per unit volume of air was done by using the approach proposed by Silk et al. [24]. Essentially, the trimer structure of prepolymer contains three –N=C=O groups (major peak) and each prepolymer peak surface area, obtained from data system, was calculated as HDI-equivalent [17].

### 2.5. Generation of commercial aerosol HDI prepolymer

To study the size distribution of the isocyanate particles according to the spray-painting operations, we simulated these operations, first in a vertical aerosol test chamber [17], and second in our experimental chamber of 10 m$^3$ of volume. Briefly, isocyanate-based aerosols generated by a low airflow in an all-glass nebulizer were introduced downstream at the top of the vertical aerosol test chamber. Spatial distribution and temporal stability of the aerosol concentration in the test section of the chamber were measured and the coefficients of variation are between 5-8%. Performance of PUF samplers was also tested in real field situations in industrial spray-painting shops.

### 3. Results and discussions

#### 3.1. Penetration of particles through PUF plugs

The penetration of particles through foam plugs with selected geometry and porosity in prototype sampler was measured in laboratory tests and complied sufficiently well with the required EN 481 thoracic and respirable curve [25]. The experimental 50% penetration particle diameters (D$_{50}$) were 4.3 µm for respirable fraction (foam 90 PPI) and 10 µm for thoracic fraction (foam 35 PPI). This means the collected isocyanate aerosols on the first stage 35 PPI foam plug were assigned to extra-thoracic fraction (>10 µm). Thoracic fraction was the results the sum of the second 90 PPI foam plug and the backup filter and the respirable fraction <4.3 µm was the mass collected on the backup filter. Summing the three collected results (foam 35 PPI, foam 90 PPI and backup filter) provided the total inhalable fraction. Mass collections on different fractions were also tested using polydisperse dust (glass microspheres) in a wind tunnel and a calm air chamber [25, 26].

#### 3.2. Penetration of isocyanate-based prepolymer aerosols through PUF plugs

Table 1 summarized the experimental conditions of different samplers used during this comparative study. Two PUF prototypes, three cyclones, two-stage virtual Respicon impactor, Andersen impactor
and impingers were used at their respective sampling flow rate and different inlet geometries for comparison.

Table 1: Summary of experimental conditions

| Sampler               | Flow rate (l.min\(^{-1}\)) | Stage | Range (µm) | 50% Cut-point | Impregnation Volume (ml) 1 mg.ml\(^{-1}\) MP | Extraction Volume (ml ACN) |
|-----------------------|-----------------------------|-------|------------|----------------|---------------------------------------------|---------------------------|
| Impactor Andersen     | 2000                        | 0     | 8.3-17.0   | 11.0           | 3 ml                                        | 10                        |
|                       | Whatmann filter 934AH Ø 81mm| 1     | 5.2-10.5   | 7.0            | 10                                         |                           |
|                       |                             | 2     | 3.5-7.3    | 4.7            | 10                                         |                           |
|                       |                             | 3     | 2.7-5.0    | 3.3            | 10                                         |                           |
|                       |                             | 4     | 1.5-3.1    | 2.1            | 10                                         |                           |
|                       |                             | 5     | 0.75-1.6   | 1.1            | 10                                         |                           |
|                       |                             | 6     | 0.45-1.0   | 0.6            | 10                                         |                           |
|                       |                             | 7     | 0.3-0.68   | 0.4            | 10                                         |                           |
|                       |                             | F     | <0.4       | 0.2            | 10                                         |                           |
| Cyclone GS-3 GF/B Filter Ø 25mm | 2.75    | Filter | Respirable fraction | 4.0          | 0.3                                        | 3                         |
| Respicon 8522 GF/B Filter Ø 37mm with hole | 3.11    | Top     | <4.0       | 4.0            | 0.3                                        | 3                         |
|                       |                             | Middle | 4.0-10.0   | 10.0           | 3                                          |                           |
|                       |                             | Bottom | >10.0      | 100            | 3                                          |                           |
| Prototype 1 foam plugs Ø 15 mm, inlet cone orifice Ø 6 mm | 2.0    | 35 PPI PUF Black Foam | 7.0-11.0 | 10.0 | 2 | 3 |
|                       |                             | 90 PPI PUF White Foam | 3.3-11.0 | 4.3 | 3 | 3 |
|                       |                             | GF/B Ø 25mm | <3.3   | 0.2          | 0.3                                        | 3                         |
| Prototype 2 foam plugs Ø 29.5 mm, inlet cone orifice Ø 8 mm | 3.5    | 35 PPI PUF Black Foam | 7.0-11.0 | 10.0 | 3 | 10 |
|                       |                             | 90 PPI PUF White Foam | 3.3-11.0 | 4.3 | 9 | 10 |
|                       |                             | GF/B Ø 25mm | <3.3   | 0.2          | 0.3                                        | 3                         |

In practice, as reported Kenny [20], the only factor distinguishing the sample designs tested by Mark and Vincent [27, 28], is the orifice size and hence the inlet velocity. In contrast to the conventional IOM sampler (which has an orifice of 15 mm, a flow of 2 l.min\(^{-1}\) and an inlet velocity of 0.2 m.s\(^{-1}\)), the manikin's mouth has an elliptical orifice measuring ~6 x 30 mm, an average flow rate of 20 l.min\(^{-1}\) and an average inlet velocity of ~4.7 m.s\(^{-1}\). On the other hand, the GSP sampler has an orifice of 8 mm, a flow rate of 3.5 l.min\(^{-1}\) and an inlet velocity of 1.16 m.s\(^{-1}\), similar to Andersen impactor (inlet orifice 25.6 mm, flow rate 28 l.min\(^{-1}\), inlet velocity of 0.91 m.s\(^{-1}\), and PAS-6 personal sampler, University of Wageningen (inlet orifice 6 mm, flow 2 l.min\(^{-1}\), inlet velocity 1.18 m.s\(^{-1}\)). Finally, for inter-comparison facility, the authors prefer to adapt the inlet entry for two PUF prototypes with inlet velocity approaching the value of GSP, ranked by Kenny as the first most precise sampler at
0.5 - 1 m.s\(^{-1}\) velocity of wind tunnel [29], and Andersen values, about 1 m.s\(^{-1}\), this means for the IOM like PUF sampler, the inlet cone orifice of the 6 mm diameter gave 1.18 m.s\(^{-1}\) at an airflow of 2 l.min\(^{-1}\).

To illustrate the performance of size-selective PUF samplers, three rounds were performed in real situation, during spray-painting in a shop. Total NCO equivalent mass fraction expressed in mg.m\(^{-3}\) collected on different samplers: impactors, cyclone, PUF sampling heads and impingers were used and results reported in table 2.

**Table 2**: Summary of all results of different samplers in field situation, spray-painting shop with two-component HDI based paint, expressed in total NCO equivalent [mg.m\(^{-3}\)].

| Round | Sampler   | Respirable fraction | Thoracic fraction | Extra-Thoracic fraction | Total fraction | Inhalable |
|-------|-----------|---------------------|-------------------|-------------------------|----------------|-----------|
|       |           | < 4.3 µm            | < 10 µm           | > 10 µm                 |                |           |
| 1     | Prototype 1 | 0.412               | 0.748             | 2.322                   | 3.070          |           |
|       |           | 0.425               | 0.714             | 1.716                   | 2.430          |           |
|       |           | 0.394               | 0.740             | 1.572                   | 2.313          |           |
|       | Prototype 2 | 0.561               | 1.073             | 1.925                   | 2.998          |           |
|       |           | 0.482               | 1.013             | 1.782                   | 2.795          |           |
|       |           | 0.512               | 1.077             | 1.479                   | 2.556          |           |
|       | Cyclone   | 0.659               | -                 | -                       | -              | -         |
|       |           | 0.604               | -                 | -                       | -              | -         |
|       |           | 0.608               | -                 | -                       | -              | -         |
|       | Respicon  | 0.748               | 1.187             | 2.742                   | 3.015          |           |
|       | Andersen  | 0.757               | 0.967             | 1.262                   | 2.018          |           |
|       | Impinger  | -                   | -                 | -                       | 4.475          |           |
|       |           | -                   | -                 | 5.002                   | 4.839          |           |
| 2     | Prototype 1 | 0.519               | 0.852             | 2.162                   | 3.014          |           |
|       |           | 0.460               | 0.868             | 2.424                   | 3.291          |           |
|       |           | 0.462               | 0.828             | 2.211                   | 3.039          |           |
|       | Prototype 2 | 0.503               | 0.860             | 1.732                   | 2.592          |           |
|       |           | 0.511               | 0.896             | 1.856                   | 2.752          |           |
|       |           | 0.532               | 0.985             | 1.990                   | 2.974          |           |
|       | Cyclone   | 0.980               | -                 | -                       | -              | -         |
|       |           | 0.875               | -                 | -                       | -              | -         |
|       |           | 0.938               | -                 | -                       | -              | -         |
|       | Respicon  | 1.002               | 1.657             | 3.370                   | 3.904          |           |
|       | Andersen  | 0.789               | 1.018             | 1.112                   | 1.901          |           |
|       | Impinger  | -                   | -                 | -                       | 4.377          |           |
|       |           | -                   | -                 | 5.148                   | 4.187          |           |
| 3     | Prototype 1 | 0.090               | 0.206             | 0.397                   | 0.740          |           |
|       |           | 0.084               | 0.167             | 0.440                   | 0.607          |           |
|       |           | 0.110               | 0.204             | 0.489                   | 0.693          |           |
|       | Prototype 2 | 0.110               | 0.206             | 0.402                   | 0.607          |           |
|       |           | 0.095               | 0.200             | 0.418                   | 0.617          |           |
|       |           | 0.107               | 0.208             | 0.391                   | 0.599          |           |
|       | Cyclone   | 0.273               | -                 | -                       | -              | -         |
|       |           | 0.243               | -                 | -                       | -              | -         |
|       |           | 0.308               | -                 | -                       | -              | -         |
|       | Respicon  | 0.325               | 0.509             | 0.427                   | 0.794          |           |
|       | Andersen  | 0.133               | 0.185             | 0.168                   | 0.301          |           |
|       | Impinger  | -                   | -                 | -                       | 0.984          |           |
Remarks:

1. Prototype 1: PUF plugs Ø 15 mm, inlet cone orifice Ø 6 mm
2. Prototype 2: PUF plugs Ø 29.5 mm, inlet cone orifice Ø 8 mm
3. Respicon: extra-thoracic fraction = (inhalable – thoracic) x 1.5
4. Andersen:
   - Respirable fraction = sum of stages 2 to 7 and backup filter
   - Thoracic fraction = sum of stages 1 to 7 and backup filter
   - Inhalable fraction = sum of all stages and backup filter
5. Swiss Occupational Exposure Limits (OEL-VME): 0.02 mg.m\(^{-3}\) of total NCO equivalent

We observed very reproducible intra-PUF samplers results and acceptable comparative results of different samplers. For example at round 1, the total concentrations of NCO equivalent mass fraction of the respirable fraction (< 4.3 µm), were 0.394 to 0.561 mg.m\(^{-3}\) for PUF prototypes (calculated as NCO collected on the backup filter), 0.064 to 0.659 for cyclones, 0.748 for Respicon and 0.757 for Andersen impactor (calculated as the sum of stages 2 to 7 and backup filter). The thoracic fraction (<10 µm) was 0.714 to 1.170 mg.m\(^{-3}\) for PUF prototypes (calculated as the sum of 90 PPI foam plug and backup filter), 1.187 for Respicon and 0.967 for Andersen impactor (calculated as the sum of stages 1 to 7 and backup filter). No thoracic data were available with cyclone samplers, because loss inside the cyclone. Aerodynamic distribution of paint aerosols is about 10 µm, and the composition was in majority HDI isocyanurate prepolymer, with a small part of HDI monomer (<0.5%). In this case, size-selective PUF samplers were useful for personal monitoring, without the need of a heavy impactor or denuder devices. Compared to cyclones, PUF personal samplers give more details on different fractions, to comply with CEN/ISO convention: respirable, thoracic, extrathoracic and inhalable fractions.

4. Concluding remarks
Porous PUF size-selective samplers for isocyanates aerosols in spray-painting survey are presented. Compared with other conventional size-selective samplers such as Andersen impactor, cyclone and the two-stage virtual impactor Respicon, the respective corresponding fractions (inhalable, extra-thoracic, thoracic, and respirable), were very similar in term of mass fraction of equivalent total NCO isocyanates. During spraying of isocyanate-based paints, large particle-borne isocyanate and prepolymer fractions dominate. Vapor HDI in commercial two-component paints tend to be a negligible part of total emission (lower than 0.5%) and denuder techniques are not necessary. In term of regulation of occupation isocyanate exposure, more attention is needed to focus on the mass fraction of large prepolymer isocyanate aerosols, less on monomer HDI vapor. Survey of isocyanate occupational exposure with there proposed size-selective personal samplers can be useful to understand the mechanism of occupation asthma developed in spray-painting shops.

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