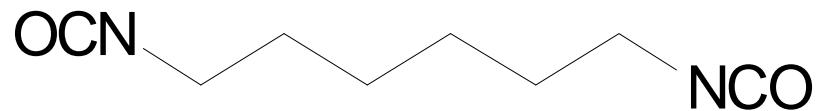
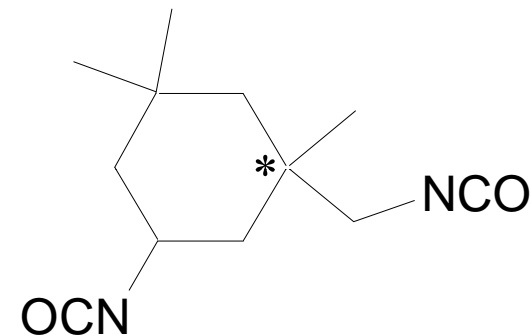


Introduction

- One exposure situation that has been given a lot of attention is during hot work, such as welding and thermal cutting, on coated metal parts.
- Hot work on metal parts coated with a polyurethane based coating can release diisocyanates, e.g. isophorone diisocyanate (IPDI) and hexamethylene diisocyanate (HDI) as well as monoisocyanates, e.g. isocyanic acid (ICA) and methylisocyanate (MIC).
- Gas filters are seldom tested for specific substances, instead protection against a certain compound is based on the boiling point of the substance of interest.



1,6-Hexamethylene diisocyanate, 1,6-HDI

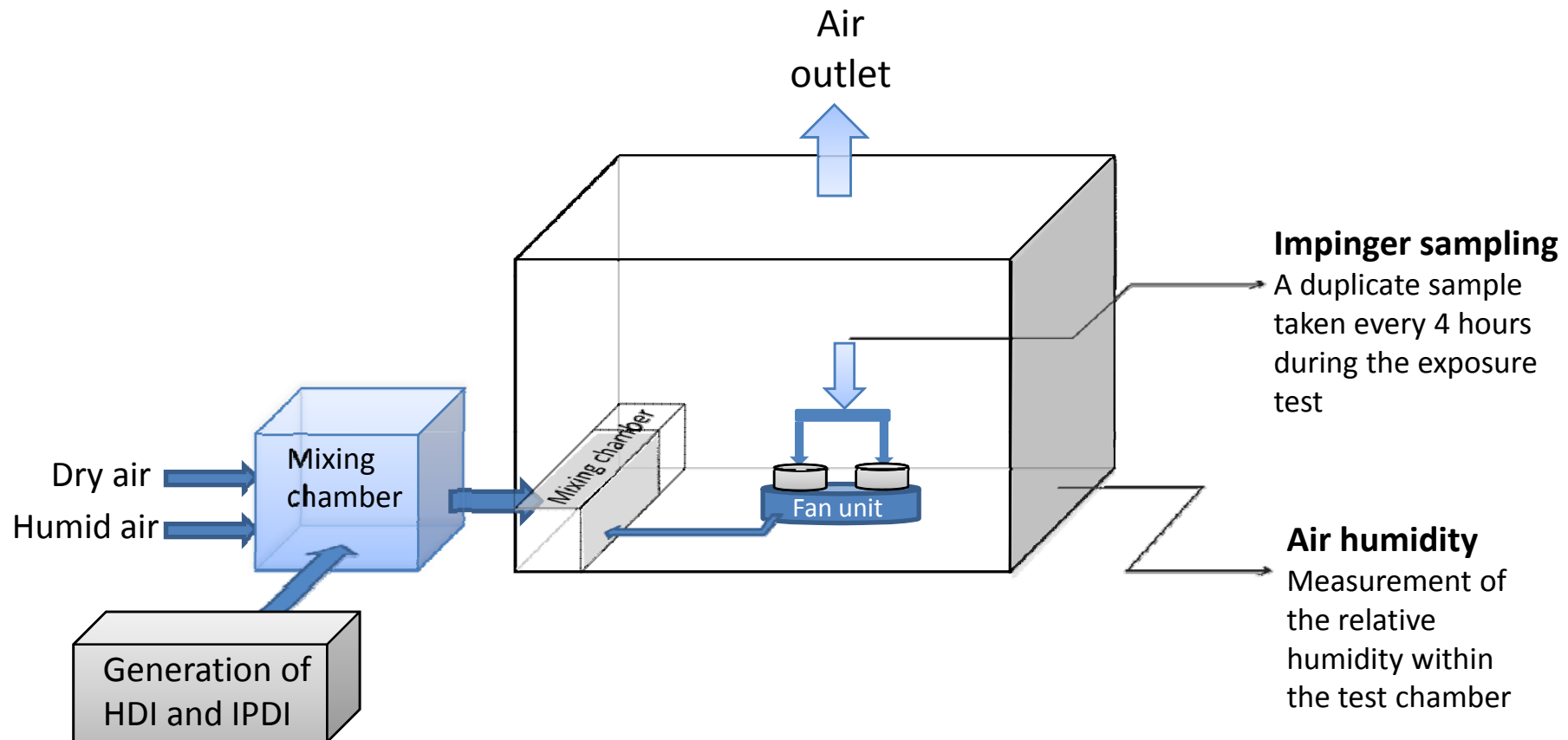


Isophorone diisocyanate, IPDI

Filter test system

- HDI and IPDI

- A climate chamber with a total volume of 0.3 m³ was used for exposure of filters.
- The chamber has a stainless steel framework and glass walls.



Experimental setup for filter tests

- The filter performance was tested at two different relative humidity, 60 and 80 % RH.
- The filters were exposed for 16 hours and 120 hours respectively.
- The effects of pre-exposure to humid air was investigated.

- Two types of filters were tested: Sundström SR 515 and SR 599.
- Two filter cartridges were tested during each exposure
- test For each filter type, four tests with a 16 hours exposure time was performed, two at 60 % RH and two at 80 % RH.
- Two tests using 120 hours of exposure time was performed at 80 % RH.

- ➡ A total of 12 filters of each type were tested

Experimental setup for filter tests

Air sampling

- To monitor the isocyanate concentration going in to the filters, Impinger air samples were collected at regular intervals.
- During the 16 hour exposures, 5 pairs of impinger samples were collected.
- During the 5 day of exposures, 6 pairs of air samples were collected.

➡ A total of 128 impinger samples were collected and analysed.

Collection efficiency

- The collection efficiency of the filters was estimated by analysis of their isocyanate content after exposure.
- The collection efficiency of the filters was estimated by analysis of the isocyanates' corresponding amine content after exposure.
- Triplicate samples from each layer in the filter cartridges + particle filters were analysed and duplicate samples from each pre-filter were analysed.

➡ A total of 624 filter samples were extracted and analysed.

Experimental setup for filter tests

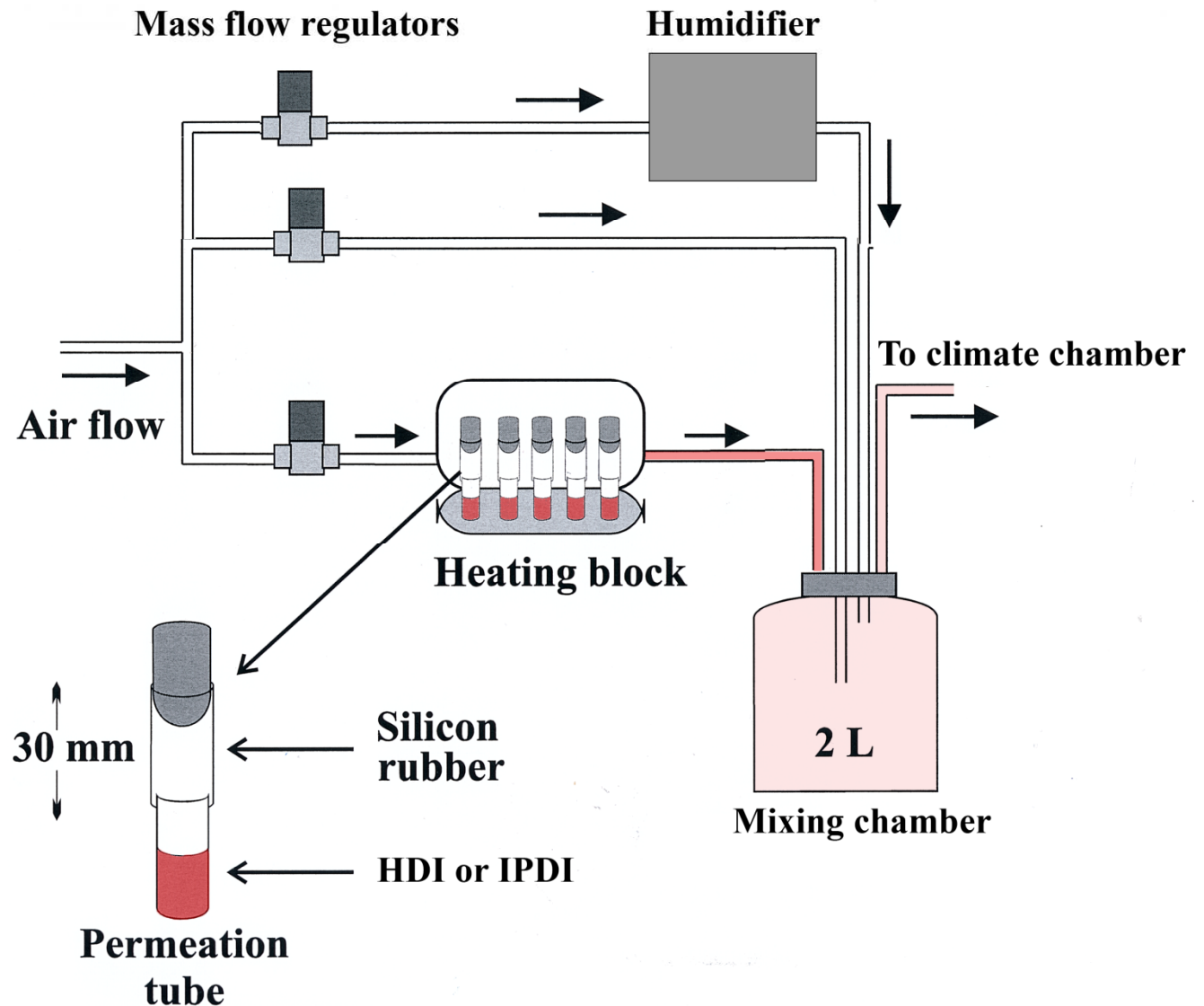
- Custom made glass adapters and connections



Generation of HDI and IPDI

- Generation of HDI and IPDI using permeation of the pure substance through silicone tubing.

- Can generate stable concentrations of airborne isocyanates over long periods of time.



Sundström SR 515 and SR 510

SR 510, Particle filter:

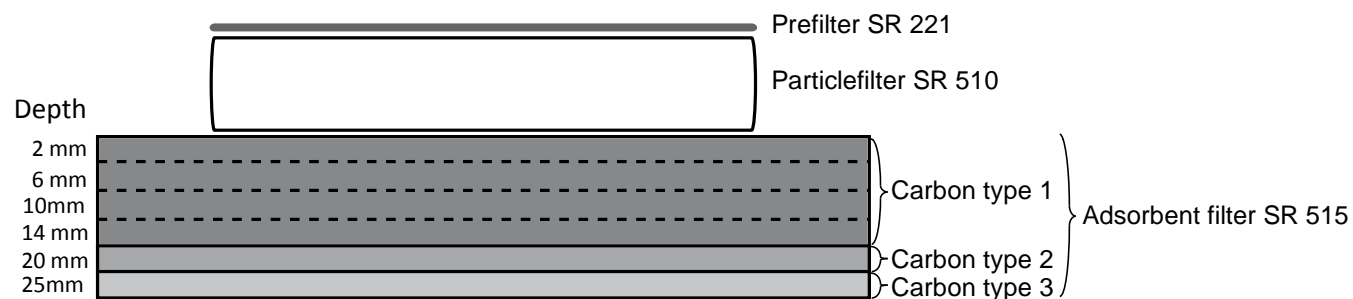
- A particle filter, stated to protect against all types of solid and liquid particles. Consists of a folded glass fibre sheet.

SR 515, Adsorbent filter:

- An adsorbent filter, stated to protect against organic substances with a boiling point above 65 °C, inorganic substances and acidic gases.
- Consists of **three** different types of activated carbon in three layers.



Extraction method - SR 515

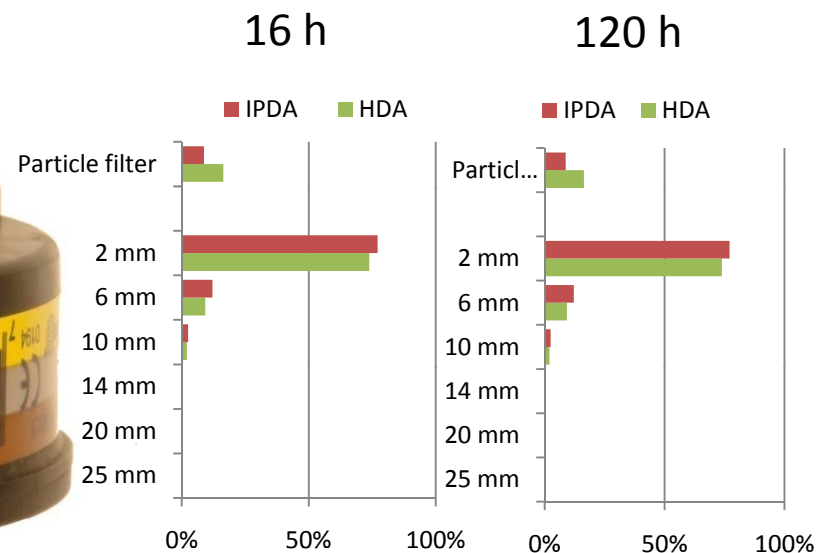


- The adsorbent filter was sawed opened after exposure to isocyanates.
- The activated carbon was removed in **six** layers using a suction device.
- Samples from each layer were hydrolysed and the isocyanates were extracted as their corresponding amine.
- The extracted amines were analysed using Liquid chromatography-tandem mass spectrometry (LC-MS/MS).
- To correctly quantify the extracted HDA and IPDA, the standards had to be prepared in blank adsorbents (prefilter, particle filter or different types of carbon)
- There were great differences in recovery for the different carbon types, affecting HDA and IPDA differently.



Results - Sundström SR 515 and SR 510

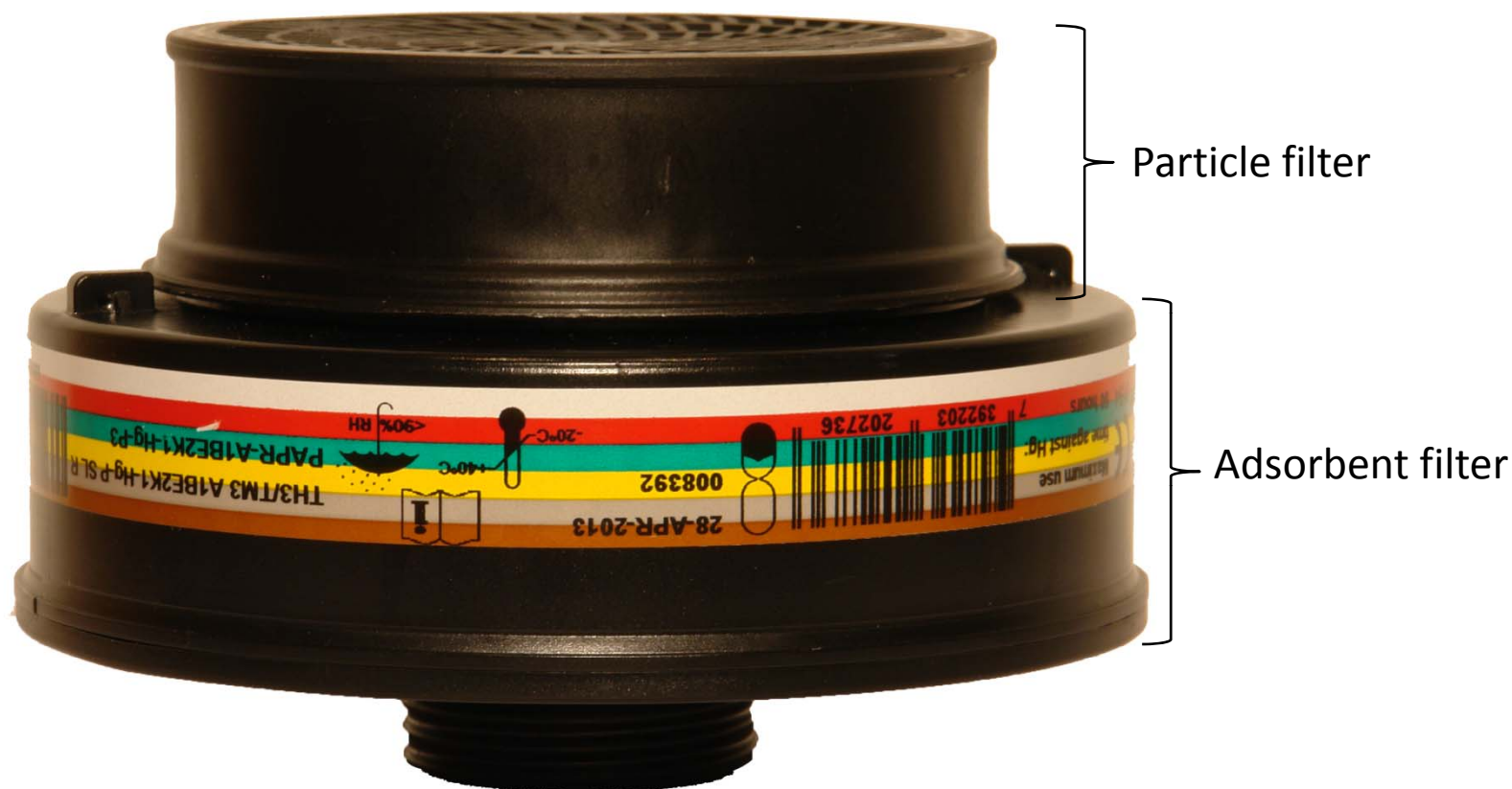
- Exposure to HDI and IPDI ($\sim 25-180 \mu\text{g}/\text{m}^3$) at 60 and 80 % RH and 20 °C
- NO Administrative norms: HDI 35 $\mu\text{g}/\text{m}^3$, IPDI 45 $\mu\text{g}/\text{m}^3$



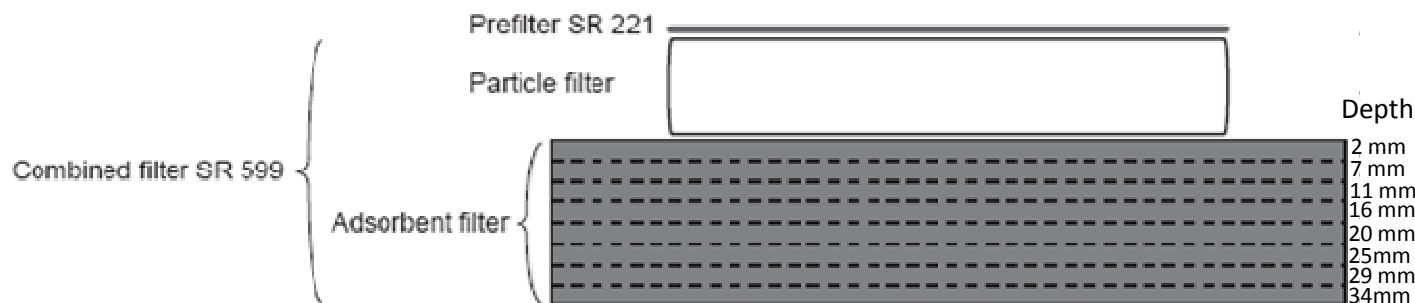
Sundström SR-599

SR 599 is a combination filter and consists of two parts:

- A particle filter, stated to protect against all types of solid and liquid particles. Consists of a folded glass fibre sheet
- An adsorbent filter, stated to protect against organic substances with a boiling point above 65 °C, inorganic substances, acidic gases, ammonia, certain amines and mercury.
- Consists of one type of activated carbon.



Extraction method - SR 599

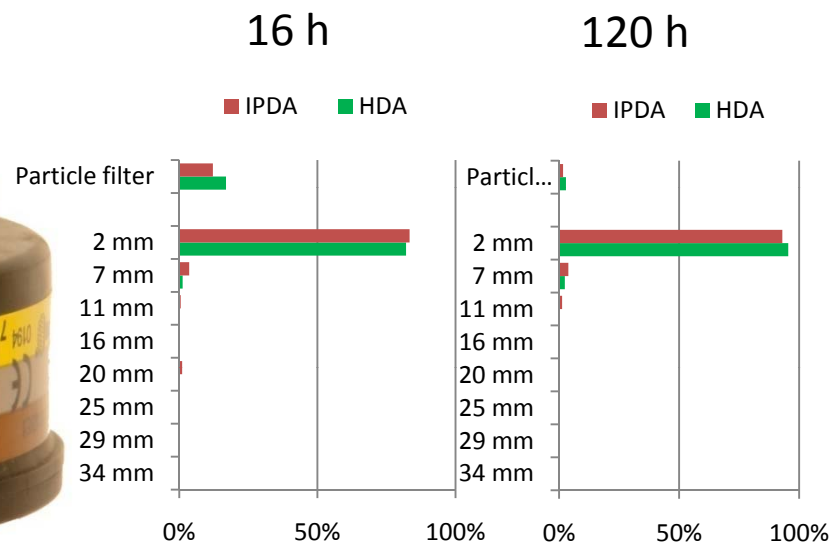


- The adsorbent filter was sawed opened after exposure to isocyanates.
- The activated carbon was removed in **eight** layers using a suction device.
- Samples from each layer were hydrolysed and the isocyanates were extracted as their corresponding amine.
- The extracted amines were analysed using Liquid chromatography-tandem mass spectrometry (LC-MS/MS).
- To correctly quantify the extracted HDA and IPDA, the standards had to be prepared in blank adsorbents (prefilter, particle filter or different types of carbon)
- There were great differences in recovery for the different carbon types, affecting HDA and IPDA differently.



Results - Sundström SR 599

- Exposure to HDI and IPDI ($\sim 25\text{-}180 \mu\text{g}/\text{m}^3$) at 60 and 80 % RH and 20 °C
- NO Administrative norms: HDI 35 $\mu\text{g}/\text{m}^3$, IPDI 45 $\mu\text{g}/\text{m}^3$



Conclusions

- Filter tests for HDI and IPDI

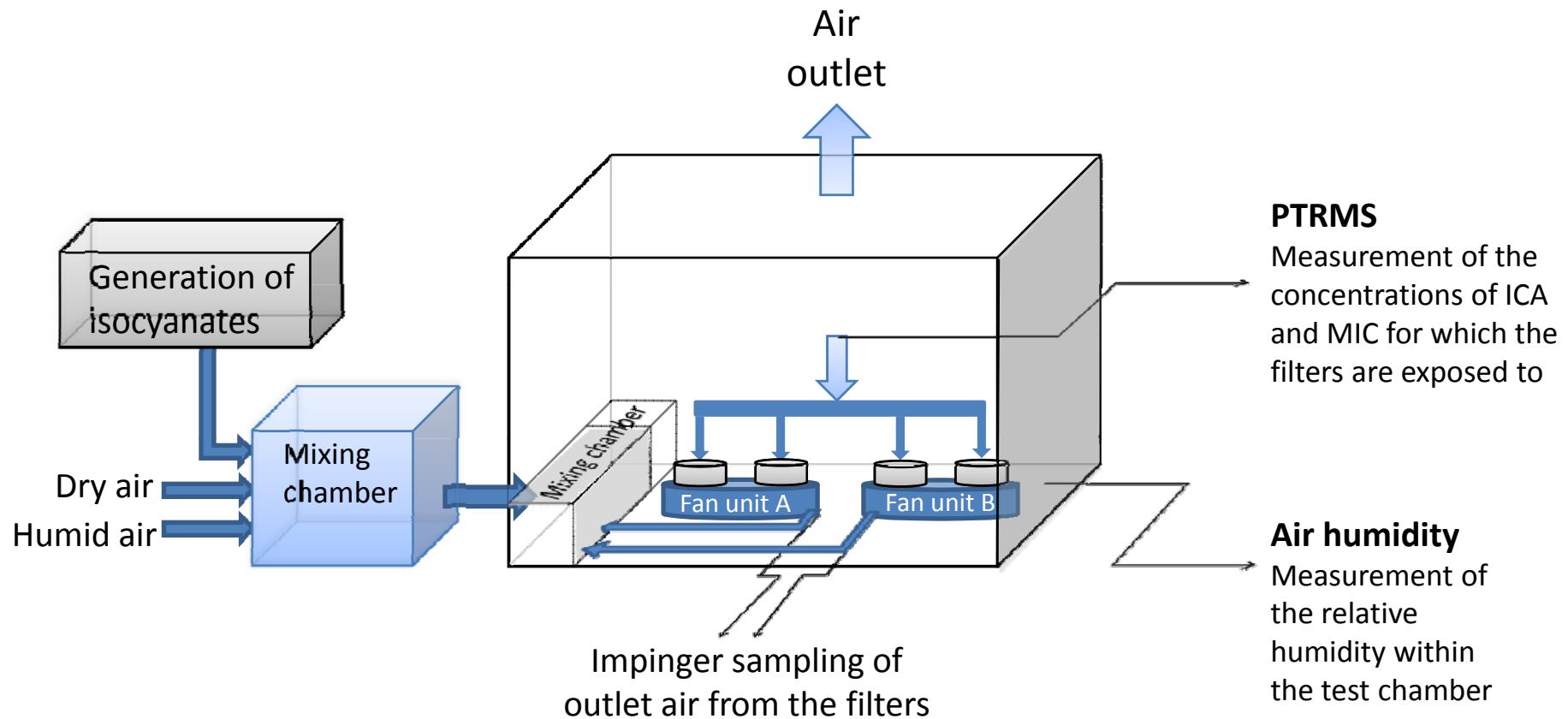
- Determination of amines in hydrolysed activated carbon samples from gas filters can be used for estimating different filters efficiency for isocyanates.
- No breakthrough was detected for any of the tested filters during continuous exposure to HDI and IPDI (25-180 $\mu\text{g}/\text{m}^3$) for up to 16 h.
- The penetration depth of the isocyanates in the filter cartridges was affected by the exposure duration.

Results from this study will, in shortly, be submitted for publication together with Total E&P.

Filter test system

- ICA and MIC

- A climate chamber with a total volume of 0.3 m³ was used for exposure of filters.
- The chamber has a stainless steel framework and glass walls.



Experimental setup for filter tests

- The filter performance was tested at two different relative humidity, 20 and 80 % RH.
 - The filters were exposed for 48 h.
 - The effects of pre-exposure to humid air and the test gas were investigated.
-
- Two types of filter were tested: Sundström SR 515 and SR 599.
 - For each filter type: ten tests were performed at 25 °C, five at 20 % RH and five at 80 % RH.
 - Four filter cartridges were tested during each exposure test at 25 °C.
 - ➡ A total of 40 filter cartridges of each type were tested at 25 °C
 - Ten tests were performed at 35 °C, five at 20 % RH and five at 80 % RH.
 - Two filter cartridges of each type were tested during each exposure test at 35 °C.
 - ➡ A total of 20 filter cartridges of each type were tested at 35 °C

Experimental setup for filter tests

Air sampling

- To continuously monitor the isocyanate concentration going in to the filters a proton transfer reaction mass spectrometer (PTRMS) was used.
- To monitor the breakthrough concentration of isocyanates, impinger samplers (DBA-toluene) or EasySamplers (DBA-dry sampler) were used to collect samples in the outgoing air from the filters.
- Impinger samples were taken every four hours during a 48 h exposure.
 - ➔ A total of 650 impinger samples were collected and analysed.
 - ➔ A total of 130 samples were collected with EasySamplers and analysed.

Protection factor

- To estimate the efficiency of the filters a protection factor for each test gas substance was calculated.
- The protection factor is the ratio between the ambient concentration of test substance and the concentration of the test substance in the air after passing through the filters

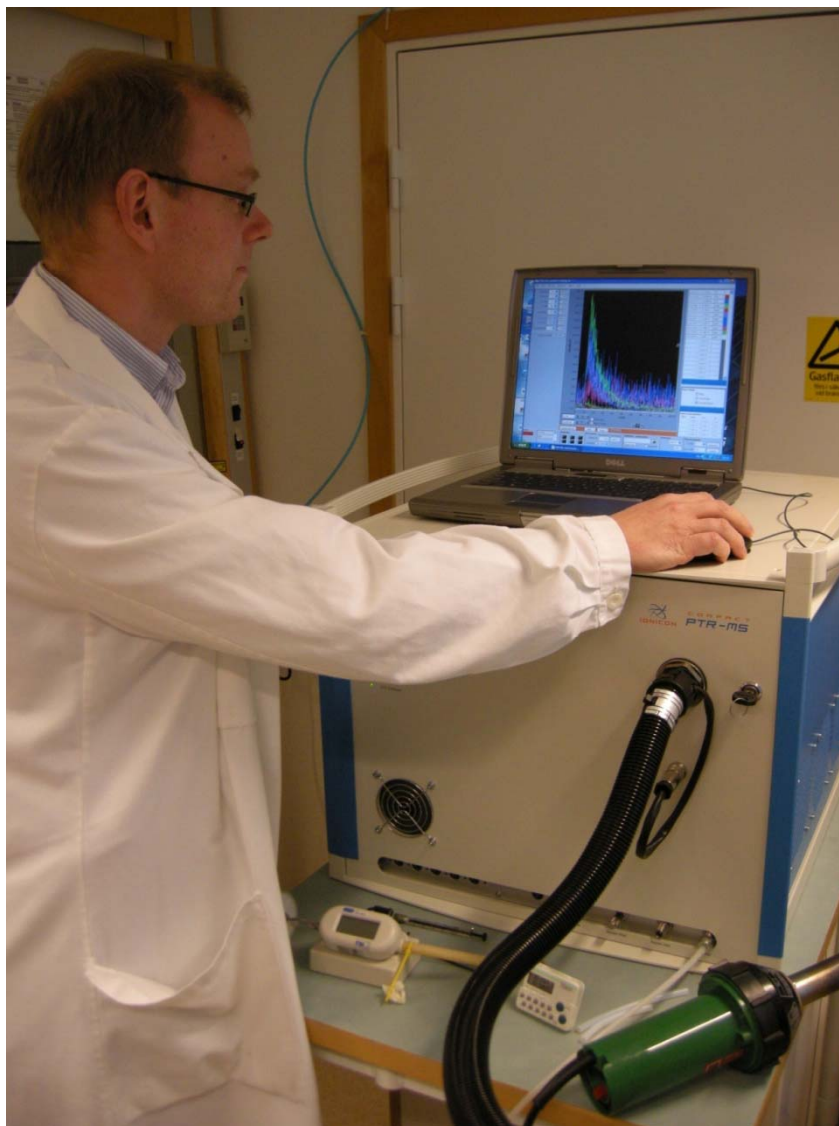
$$\textit{Protection factor} = \frac{C_{\textit{ambient}}}{C_{\textit{after filter}}}$$

Experimental setup for filter tests

- Custom made glass adapters and connections



Proton transfer reaction mass spectrometer - PTRMS



Sundström SR 515 and SR 510

SR 510, Particle filter:

- A particle filter, stated to protect against all types of solid and liquid particles. Consists of a folded glass fibre sheet.

SR 515, Adsorbent filter:

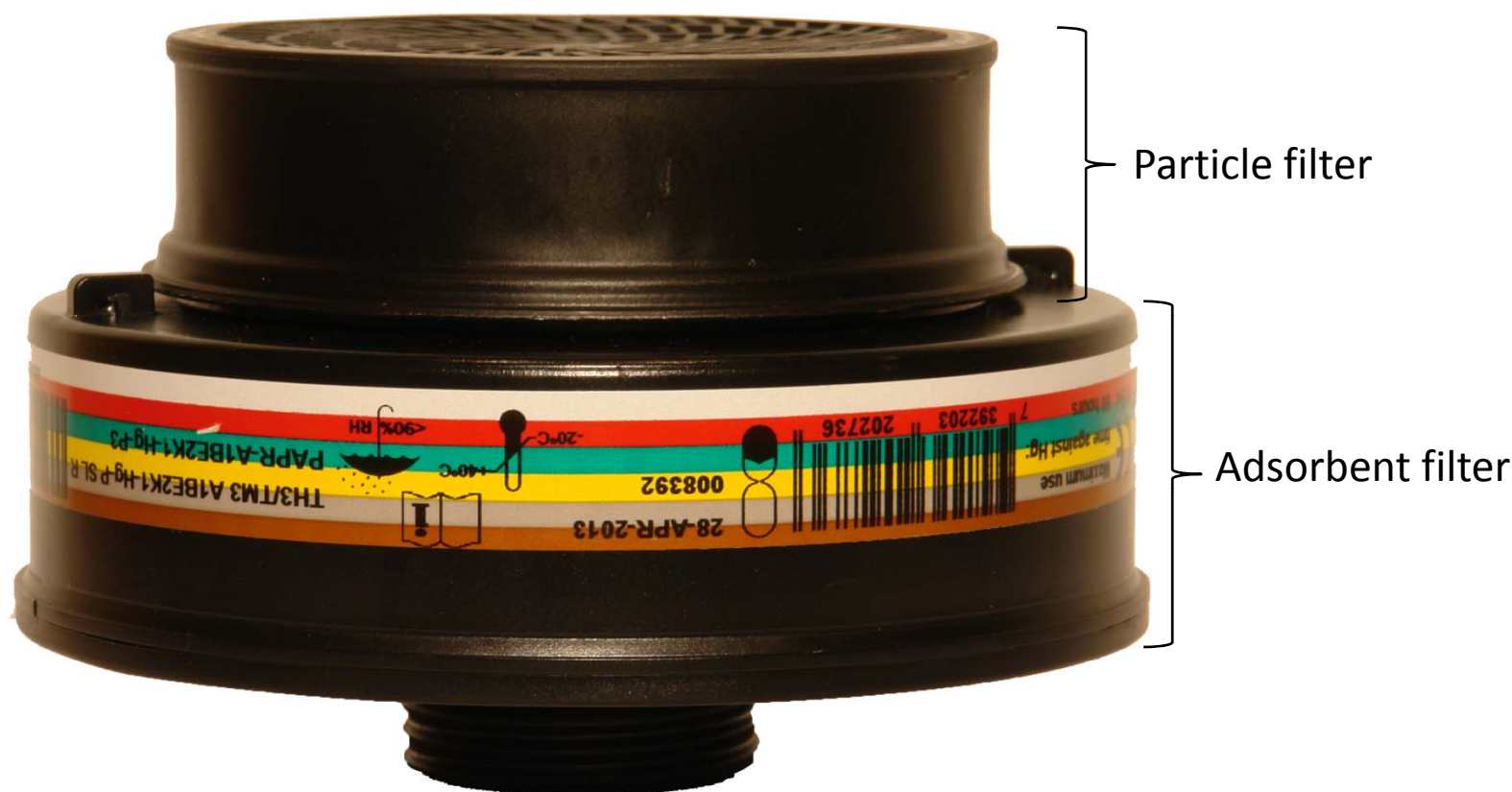
- An adsorbent filter, stated to protect against organic substances with a boiling point above 65 °C, inorganic substances and acidic gases.
- Consists of **three** different types of activated carbon in three layers.



Sundström SR-599

SR 599 is a combination filter and consists of two parts:

- A particle filter, stated to protect against all types of solid and liquid particles. Consists of a folded glass fibre sheet
- An adsorbent filter, stated to protect against organic substances with a boiling point above 65 °C, inorganic substances, acidic gases, ammonia, certain amines and mercury.
- Consists of one type of activated carbon.



Results – Exposure tests at 25 °C

- Both filter types, SR 515 and SR 599

- NO administrative norm (8h work day): ICA: 9 $\mu\text{g}/\text{m}^3$
MIC: 12 $\mu\text{g}/\text{m}^3$

- Test gas concentrations: ICA: ~180-360 $\mu\text{g}/\text{m}^3$
MIC: ~240-470 $\mu\text{g}/\text{m}^3$

- Results from tests at 25 °C, 80 %RH and 20 %RH:

Filter type	Protection factor	
	ICA	MIC
SR-515; n=40	760	6200
SR-599; n=40	800	7500

Results – Exposure tests at 35 °C and 20 %RH

- Both filter types, SR 515 and SR 599

- NO administrative norm (8h work day): ICA: 9 $\mu\text{g}/\text{m}^3$
MIC: 12 $\mu\text{g}/\text{m}^3$

- Test gas concentrations: ICA: ~180-360 $\mu\text{g}/\text{m}^3$
MIC: ~240-470 $\mu\text{g}/\text{m}^3$

- Results from tests at 35 °C and 20 %RH:

Filter type	Protection factor	
	ICA (n = 10)	MIC (n = 10)
SR-515	950	7000
SR-599	1000	7200

Results – Exposure tests at 35 °C and 80 %RH

- Both filter types, SR 515 and SR 599

- NO administrative norm (8h work day): ICA: 9 $\mu\text{g}/\text{m}^3$
MIC: 12 $\mu\text{g}/\text{m}^3$

- Test gas concentrations: ICA: ~180-360 $\mu\text{g}/\text{m}^3$
MIC: ~240-470 $\mu\text{g}/\text{m}^3$

- Results from tests at 35 °C and 80 %RH:

Filter type	Protection factor	
	ICA	MIC (n = 10)
SR-515	*	9000
SR-599	*	10000

**The sampling and analysis of ICA did not work at 35 °C, 80 % RH
No sampling technique available at high humidity?**

Conclusions

- Filter tests for ICA and MIC

- A robust system for continuous generation of stable air concentrations of ICA and MIC was developed.
- The use of the PTRMS for real time monitoring of air concentrations greatly increased the quality of the exposure data as well as saving time on collecting numerous of air samples and subsequent analysis.
- No trend of impaired performance for the two different types of filters studied was observed throughout the 48 h exposure tests. Not even if the filters were pre-exposed to high concentrations of ICA and MIC for 48.

Results from this study will, in shortly, be submitted for publication together with Total E&P.