APPLICATION NOTE

Author:

Dr. C. Rosin, Scientist R&D / Applications
G.A.S. Dortmund, Germany

Rapid Monitoring of Isocyanates in Chemical Plants using GC-IMS Technology

Introduction

Toxic industrial chemicals (TICs) or materials (TIMs) are substances that exhibit harmful effects on humans but are commonly used in manufacturing facilities, pharmaceutical chemical plants. Exposure to those chemicals are seriously harmful especially after multiple low-level exposures. Due to their electrophilic properties, isocyanates are reactive towards many nucleophilic substances like alcohols, amines or even water. Several isocyanates are extremely poisoning and exposure can result in lung edema, emphysema, hemorrhages, bronchial pneumonia and death, e.g.

With its GC-IMS device G.A.S. developed a highly selective and sensitive measuring system for rapid monitoring of *trans*-4-methyl cyclohexyl isocyanate (4-MCI) in industrial areas.

Monitoring of Toxic Industrial Chemicals (TICs)

Experimental Conditions

Table 1: GC Conditions	
Column	MCC-OV5
Temperature	60 °C
Flow rate	50 mL/min
Carrier gas	N_2

Table 2: IMS Conditions	
Radiation source	Tritium
Temperature	45 °C
Flow rate	150 mL/min
Carrier gas	N_2
Mode	positive

Table 3: Sampling Conditions	
Sample loop volume	1 mL
Temperature	45 °C
Purge rate (continuous)	~160 mL/min

In order to evaluate a measurement system for monitoring of 4-MCI in chemical plants a special headspace dilution method was developed for calibration of the substance in the low ppb range.

Table 4: Calibration Conditions	
Calibrated concentrations Values in ppb	34, 46, 64, 88, 122, 168, 232, 320, 441, 608, 839
Temperature	25 °C

Results

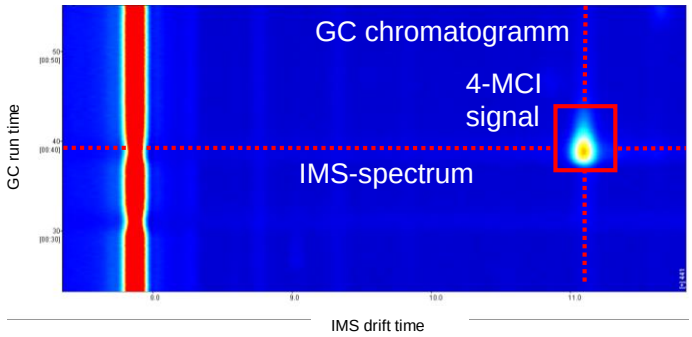

Figure 1: 2-dimensional representation of the 4-MCI signal (red rectangle). GC-runtime: y-axis; IMS drift time: x-axis. Intensity is depicted in false colours: blue: low intensity; red: high intensity.

Under the applied GC and IMS conditions (Table 1) 4-MCI elutes after 39 sec and exhibits a drift time of 11.10 ms (Figure 1).

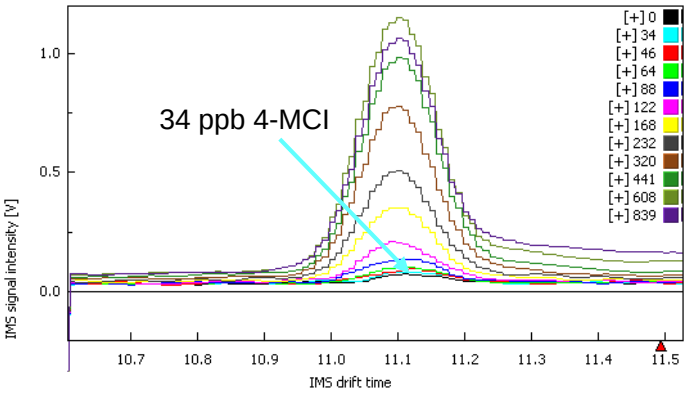

Figure 2: IMS spectra of 4-MCI with increasing concentration (34 to 839 ppb).

The detected IMS-signals of 4-MCI in the range of 34 to 839 ppb are depicted in Figure 2; corresponding chromatograms shown in Figure 3.

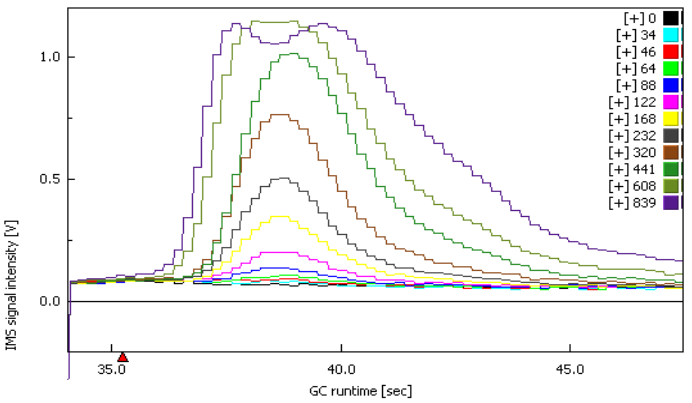

Figure 3: Chromatograms of 4-MCI with increasing concentration (34 to 839 ppb).

Monitoring of Toxic Industrial Chemicals (TICs)

Based on the highly reproducible 4-MCI specific signals the GC-IMS measurement system was calibrated in the range of 34 to 839 ppb by calculation of the maximum peak value in each IMS spectrum. The detection limit of the system (Figure 2), defined as 3σ of the baseline, was calculated to approx. 10 ppb. It has to be mentioned that below 64 ppb the slope decreases which leads to less accurate results below this value.

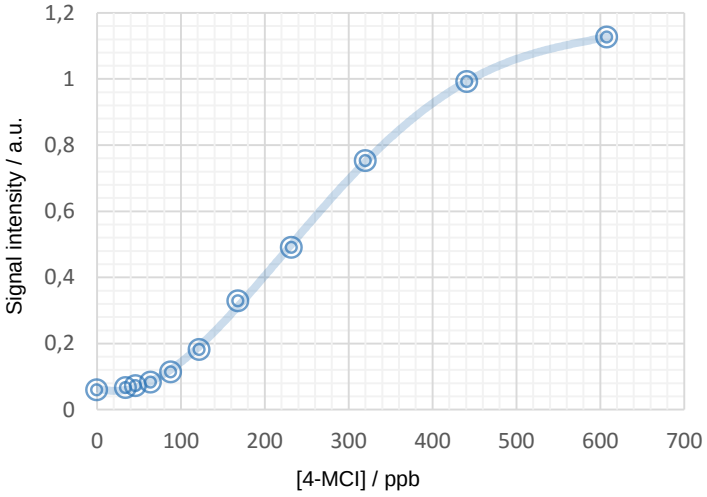

Figure 3: Calibration curve of 4-MCI in the range of 34 to 839 ppb.

In order to prevent hidden system failures or false-negative measurement results the GC-IMS comes with comprehensive self-monitoring check-up features. An integrated watchdog monitors hardware failures, a sample flow controller assures sample specifity.

Further to that, the status, same as important parameters such as correct temperatures, pressures, carrier and drift gas flows, are continuously monitored. Furthermore the whole device can be checked at-site by use of a test substance to verify the accurate readiness and by that the reliability of the measurements results.

False-positives caused by interfering compounds, which might be present on a chemical plant, are reduced to a minimum due to the 2-dimensional GC- and IMS-separation. Despite the separation a full measurement run time only takes 2 min. This includes the GC- and IMS-separation/detection and also a cleaning period at the end of the measurement to avoid carry-over effects.

Conclusions

The developed method uses the GC-IMS made by G.A.S. with outstanding field performance. The instrument continuously samples the ambient air, which shall be monitored. The run time only takes 2 min. including a cleaning step of the instrument's gas touching parts in order to avoid carry-over. The calibration curve for 4-MCI was established exhibiting a high reproducibility in the range of 34 to 839 ppb.

Figure 4: GC-IMS instrument for monitoring of TICs in chemical plants embedded into a pressurized enclosure system. The pressurized enclosure system ensures safety class Ex II 2 G Ex px II T4 (German Explosion Protection Ordinance).

G.A.S. Gesellschaft für analytische Sensorsysteme mbH
Otto-Hahn-Straße 15, D-44227 Dortmund, Germany
Phone: +49 231 9742 6550 / Fax: +49 231 9742 6555
info@gas-dortmund.de / www.gas-dortmund.de