APPLICATION NOTE

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Rapid Monitoring of Dimethyl Sulfate (DMS) in Chemical Plants using GC-IMS Technology

Introduction

Toxic industrial chemicals (TICs) or materials (TIMs) are substances that exhibit harmful effects on humans and are common in manufacturing facilities, chemical plants, maintenance areas or general storage areas. Exposure to those chemicals are seriously harmful especially after multiple low-level exposures. Dimethyl sulfate (DMS) is commonly used as a reagent for the methylation of phenoles, amines, and thiols. In industry DMS is preferred, compared to other methylating agents, because of its low cost and high reactivity. It is known that DMS is carcinogenic, mutagenic, highly poisonous, corrosive, environmentally hazardous and volatile. The substance can be absorbed through skin, mucous membranes, and gastrointestinal tract.

With its GC-IMS device G.A.S. developed a highly selective and sensitive measuring system for rapid monitoring of Dimethyl Sulfate in chemical plants.

Monitoring of Toxic Industrial Chemicals (TICs)

Experimental Conditions

Table 1: GC Conditions	
Column	MCC-OV5
Temperature	60 °C
Flow rate	20 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	negative

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

In order to validate the GC-IMS performance regarding sensitivity and reproducibility a permeation oven was used.

Table 4: Calibration Conditions	
Calibrated concentrations	5, 25, 50, 75, 100, 120, 140 ppb
Permeation rate	164 ng/min
Permeation oven	Customized MCZ CMK5
Oven temperature	70 °C

Results

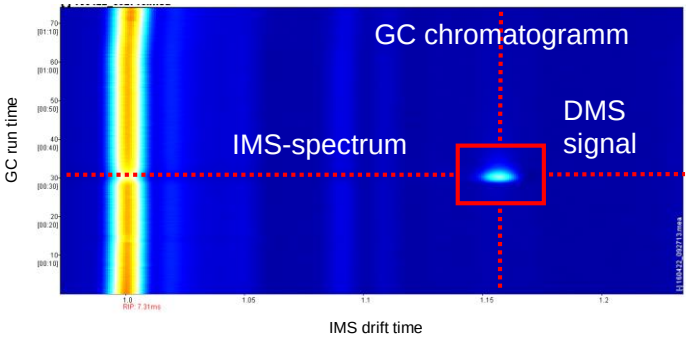

Figure 1: 2-dimensional representation of the DMS signal (red rectangle). GC-runtime: y-axis; IMS drift time: x-axis. Intensity is depicted in false colours: blue: low intensity; red: high intensity.

Under the applied GC and IMS conditions (Table 1) DMS elutes after 30 sec and exhibits a drift time of 8.46 ms (Figure 1).

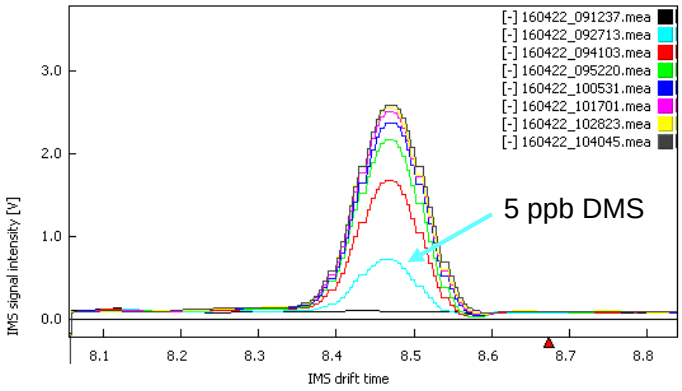

Figure 2: IMS spectra of DMS with increasing concentration (5 to 140 ppb).

The detected IMS-signals of DMS in the range of 5 to 140 ppb are depicted in Figure 2 and corresponding chromatograms in Figure 3.

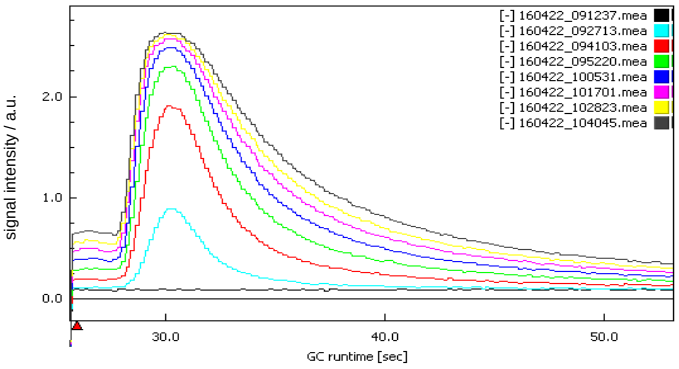

Figure 3: Chromatograms of DMS with increasing concentration (5 to 140 ppb).

Monitoring of Toxic Industrial Chemicals (TICs)

Based on the highly reproducible DMS specific signals the GC-IMS measurement system was calibrated in the range of 5 to 140 ppb by calculation of the maximum peak value in each IMS spectrum. As it can be derived from the signal height, the measured concentration of 5 ppb does not represent the limit of detection under these parameters. Extrapolation of the data yields to a detection limit of approx. 200 ppt.

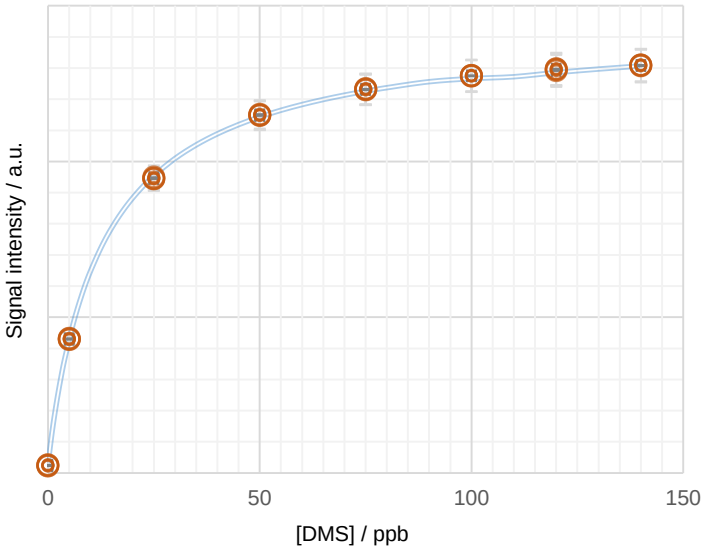

Figure 3: Calibration curve of DMS in the range of 5 to 140 ppb.

In order to prevent undiscovered system failures or false-negative measurement results the system comes with comprehensive self-monitoring check-up features. An integrated hardware 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 measurement system at-site can be checked by a test substance to verify the accurate readiness and by that the reliability of the measurements results.
False-positive results, based on potentially interferring compounds, which might be present on chemical plants (anti-freezing agents, e.g.), 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 could be decreased to a value of 2 min, including a cleaning step of the instrument's gas touching parts in order to avoid carry-over. The calibration curve for DMS was established exhibiting a high reproducibility in the range of 5 to 140 ppb. The calculated detection limit (extrapolated) of the instrument is approx. 200 ppt.

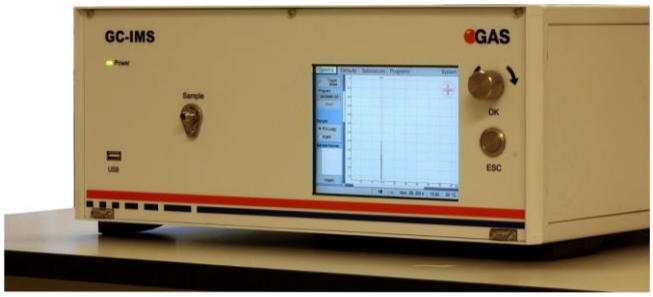

Figure 4: GC-IMS instrument for monitoring of TICs in chemical plants.

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