

Environmental Monitoring of Low-Level Sulphur in Ambient Air



Application Note

AN0058

INTRODUCTION

The presence of sulphides in ambient air is an environmental hazard. The detection of sulphur compounds at trace levels is critically important through air monitoring. Monitoring sulphur compounds is commonly done in industrial emissions testing and environmental monitoring at sewage treatment and landfill sites. Compounds such as carbon disulphide, methyl mercaptan, ethyl mercaptan, dimethyl sulphide, propanethiol, ethyl sulphide, dimethyl disulphide and butyl mercaptan are volatile compounds that require thermal desorption as a means of analysis.

Thermal desorption is a technique that provides a trace level analysis through both offline and real time monitoring of vapour changes in the sampling environment.

This application note describes environmental monitoring of low level sulphurs in ambient air through thermal desorption gas chromatography.

EXPERIMENTAL

A Scion 456 GC with a pulsed flame photometric detector (PFPD) was used in combination with thermal desorption. Ambient air samples were collected using a low temperature on-line sulphur monitoring system. The pre-concentration unit was cryogenically cooled and enriched with samples thermally analysed.

Analytical conditions for thermal desorption can be found in Table 1a with the GC-PFPD parameters in Table 1b.

Table 1a. Analytical conditions of the thermal desorption unit

Conditions	
Flow Rate	50mL/min
Cold Trap	-10°C
Split Flow	40mL/min
Flat Rate	100°C/min
Trap	300°C
Resolution Time	4 minutes
Purge Flow	100mL/min, 1 minute
Cold Trap Purge	50mL/min, 2 minutes

Table 1b. Analytical conditions of the GC-FID

Conditions	
Column	GC-GASPRO 30m x 0.32mm
Oven Programme	80°C (1min), 25°C/min to 260°C (3mins)
Carrier	3.5mL/min
PFPD	250°C, 540V
Flow	Air 20mL/min, H ₂ 15.5mL/min, Air 2 10mL/min

An ambient air gas standard was diluted to 0.5, 1.0, 2.0, 5.0 and 8.0nmol/mol using a stainless steel ultra-clean valve. The gas standard included the following target compounds carbon disulphide, methyl mercaptan, ethyl mercaptan, dimethyl sulphide, propanethiol, ethyl sulphide, dimethyl disulphide and butyl mercaptan.

RESULTS

Figure 1 shows the 5.0nmol/mol gas standard during repeatability testing. The 5.0nmol/mol standard was analysed with eight consecutive injections. Due to excellent sensitivity to sulphide by the PFPD, the response for carbon disulphide and dimethyl disulphide is much greater than the other target compounds. Identification of corresponding target analytes can be found in Table 2.

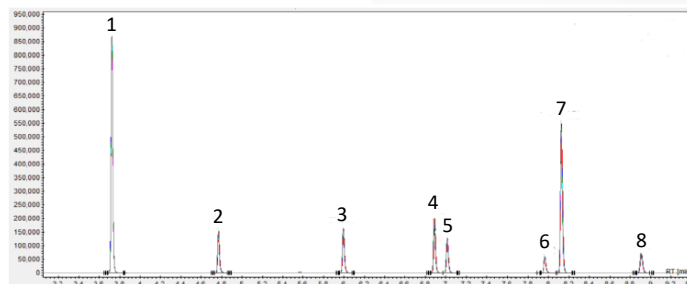


Figure 1. 5nmol/mol gas standard of ambient air components

Table 2. Peak identification of components in Figure 1.

Peak Number	ID
1	Carbon Disulphide
2	Methyl Mercaptan
3	Ethyl Mercaptan
4	Methyl Sulphide
5	Propanethiol
6	Ethyl Sulphide
7	Dimethyl Disulphide
8	Butyl Mercaptan

Excellent linearity was observed for all target compounds in the gas standards. The average correlation coefficient was 0.9999 with each compound achieving an R² value of 1 with the exception of methyl mercaptan and dimethyl disulphide which had an R² value of 0.9999. Figure 2 shows the calibration curve of carbon disulphide and is representative of all target compounds.

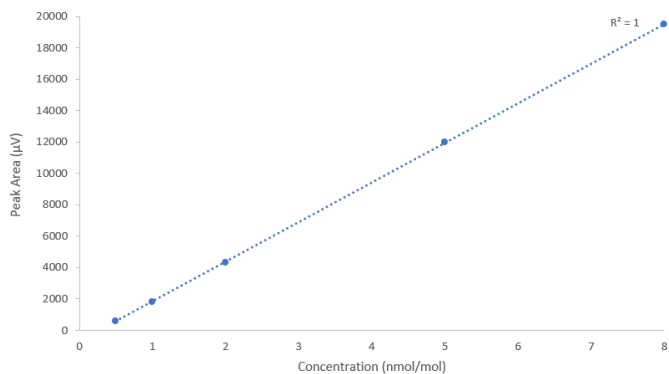


Figure 2. Calibration curve of carbon disulphide

Repeatability was demonstrated with eight replicates of the 0.5nmol/mol standard. Table 3 details the RSD% value as well as the calculated method detection limit (MDL) for each target compound.

Table 3. Repeatability values and method detection limits (n=8)

Compound	RSD %	MDL (nmol/mol)
Carbon Disulphide	2.07	0.08
Methyl Mercaptan	2.87	0.06
Ethyl Mercaptan	1.54	0.08
Methyl Sulphide	2.50	0.06
Propanethiol	1.18	0.07
Ethyl Sulphide	1.18	0.10
Dimethyl Disulphide	1.89	0.07
Butyl Mercaptan	2.00	0.07

The eight target compounds show excellent repeatability at the lowest concentration analysed of 0.5nmol/mol. Additionally, exceptionally low detection limits were obtained for each target compound. These results highlight the sensitivity and robustness of the system.

CONCLUSION

The Scion GC:PFPD analyser for the environmental monitoring of low level sulphurs in ambient air is the ideal solution for all environmental monitoring requirements. In under 15 minutes, including sample preparation and sample analysis, eight target compounds were easily separated and resolved with excellent repeatability and low detection limits.