

Determination of four volatile organic sulfur compounds by automated headspace technique coupled with gas chromatography–mass spectrometry

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ABSTRACT

A simple and efficient method based on headspace technique coupled with gas chromatography–mass spectrometry (GC/MS) was developed for the simultaneous determination of four volatile organic sulfur compounds (VOSCs) in surface water samples, including dimethyl sulfide, diethyl sulfide, dimethyl disulfide, and dimethyl trisulfide. Headspace parameters such as sample volume, ionic strength, rotational stirring, headspace analysis duration, and temperature were optimized. The linear range spanned approximately three or four orders of magnitude. The detection limits (MDLs) and method quantification limits were 0.02–0.2 µg/L and 0.06–0.5 µg/L, respectively. The reproducibility (relative standard deviation) of the method was 2.1%–6.5%, and the recovery was 90.0%–114.0%. The method was applied for the determination of VOSCs in surface water, and tap water from four plants for surface water treatment. Only dimethyl sulfide was detected in partial surface water samples (1–7 µg/L), while other VOSCs were all below MDLs.

Key words | GC/MS, headspace, surface water, volatile organic sulfur compounds

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INTRODUCTION

Odorous emission from treatment facilities has posed a problem to citizens for decades (Romero & Ventura 2000; Sheng *et al.* 2008; Lasaridi *et al.* 2010). Volatile organic sulfur compounds (VOSCs) such as dimethyl sulfide (DMS), diethyl sulfide (DES), dimethyl disulfide (DMDS), and dimethyl trisulfide (DMTS) are believed to be important causes of unpleasant odors from surface water in treatment plants (Wardencki 1998; Higgins *et al.* 2006; Kim & Park 2008; Suffet *et al.* 2009), because they are malodorous and have low odor thresholds. The odor thresholds of DMS, DES, DMDS, and DMTS were 0.004, 0.03, 2.2, and 80 µg/L, respectively (Guah 1997; Czerny *et al.* 2008). Even a small amount of VOSCs can contribute to significant odor pollution (Chen *et al.* 2010; Allard *et al.* 2012). At higher

concentrations (>0.5–20 mg/L), they can induce health problems (Aparicio *et al.* 1997; Chen *et al.* 2013).

The main analytical methods for odor analysis are gas chromatography (GC) and gas chromatography–mass spectrometry (GC/MS) due to the volatility of odor compounds (Bak *et al.* 1992; Hwang *et al.* 1995; López-García *et al.* 2002; Cheng *et al.* 2005). VOSCs are usually isolated from the sample matrix before GC or GC/MS through sample preparation techniques, such as liquid-phase extraction, gas-phase extraction/distillation, solid-phase extraction, solid-phase microextraction (SPME), purge and trap (P&T), and headspace technique (Abalos *et al.* 2002; Lestremau *et al.* 2004; Cheng *et al.* 2007; Lu *et al.* 2012; Wichaphon *et al.* 2012). Headspace technique is frequently used for VOSCs

analysis because it is simple, solvent-free, fully automated and can avoid contamination by nonvolatile constituents (Sun *et al.* 2014). Other techniques such as P&T and SPME are time-consuming and expensive. Despite the fact that headspace has been used in the analysis of VOSCs in water, a comprehensive study of all variables that may affect the efficiency of analysis has not been carried out.

The aim of this study was to develop a fast analytical method based on headspace technique to analyze VOSCs in drinking water. The headspace autosampler was connected to a GC/MS instrument that employed selective ion monitoring. In the present study, parameters such as sample volume, salt addition, stirring rate, headspace analysis duration, and temperature have been evaluated. The method was applied to determine the concentrations of VOSCs in samples from four plants for surface-water treatment in Jinan.

MATERIALS AND METHODS

Reagents and chemicals

DMS and DES (purity >99.2%) were purchased from Chem Service (USA). DMDS (purity >99.0%) was purchased from Dr. Ehrenstorfer GmbH (Germany). Reagent-grade DMTS (purity 98%) was purchased from Sigma Aldrich (USA). NaCl was purchased from Carlo Erba (Milan, Italy). Methanol (high performance liquid chromatography grade) was acquired from Merck (Germany). Water was obtained from a Milli-Q Plus purification system (Millipore, Billerica, MA, USA). Stock standard solutions were prepared by weighing and then stored at -20°C . Working solutions were prepared daily by diluting standard solutions with Milli-Q water to the required concentrations.

Instrumentation

A GC 2010 Plus was interfaced to a TQ8030 quadrupole mass spectrometer (Shimadzu, Japan). The HS-20 headspace autosampler had an incubator for heating and mixing (Shimadzu, Japan), which was coupled to the GC/MS system for direct analysis of the four VOSCs (DMS, DES, DMDS, and DMTS) in the water samples.

Sample preparation

A 20 mL headspace glass vial (7.2 cm \times 2 cm) was filled with 10 mL of water sample and immediately sealed with a polytetrafluoroethylene/silicone septum. It was placed in the headspace autosampler for heating with mechanical stirring for 20 min at 90°C to ensure equilibration of the gaseous and liquid phases in the vial. An aliquot of the gas phase above the water (1.0 mL) was injected into the GC system. Injector temperature was set at 26.1°C and the injector split ratio was 50:1.

Chromatographic conditions

A capillary column (Rxi-5Sil, 30 m \times 0.25 mm \times 0.25 μm ; Restek, USA) was used for VOSCs separation and the carrier gas was helium (purity 99.9999%) with flow rate at 36.1 cm/min. The column temperature was held at 40°C for 2 min, increased to 150°C at $15^{\circ}\text{C}/\text{min}$, and then held at this temperature for 2 min. The mass spectrometer was operated in electron ionization mode. Temperatures for transfer line and source of ionization were 280°C and 230°C , respectively. The ionization voltage was 70 eV, and the solvent cut time was 1.5 min. The mass spectrometer was calibrated weekly with perfluorotributylamine. Selected ions and retention times are shown in Table 1.

RESULTS AND DISCUSSION

Method development

This study explored the applicability of static headspace to the analysis of VOSCs in aqueous matrixes. The effects of some variables, including sample volume, ionic strength,

Table 1 | Selected ions and retention times for the target VOSCS

Analyte	Selected ions	Retention time (min)
DMS	62, 47, 45	1.7
DES	75, 90, 61	3.0
DMDS	94, 79, 45	3.5
DMTS	126, 111, 79	6.6

rotation stirring, headspace duration, and headspace temperature were investigated and optimized to achieve high sensitivity of the proposed method with Milli-Q water samples. The Milli-Q water samples were free of VOSCs through analysis of the proposed method.

Sample volume

The effect of the sample volume on the signal intensity was investigated. Samples with volumes of 1, 2, 5, 8, and 10 mL were separately transferred to 20 mL headspace vials. All samples contained the same concentration of VOSCs per vial. As shown in Figure 1, peak areas for DMS, DES, DMDS, and DMTS increased considerably with increasing sample volume from 1 mL to 10 mL. However, increasing the sample volume could lead to sample leakage, and the peak pattern deteriorated for volumes higher than 10 mL because of higher pressure. Thus, a sample volume of 10 mL was used.

Ionic strength

NaCl solid (0%, 10%, 20%, 30%, 40%, 50%, or 60% mass fraction) was added to the water samples. All samples contained the same concentration of VOSCs of 10 mL per vial. As shown in Figure 2, areas of DMS, DES, DMDS, and DMTS peaks increased markedly with increasing NaCl mass (0–20%) and then slightly at big mass (do not consider saturation). Therefore, addition of salt could affect the headspace composition. Consequently, all

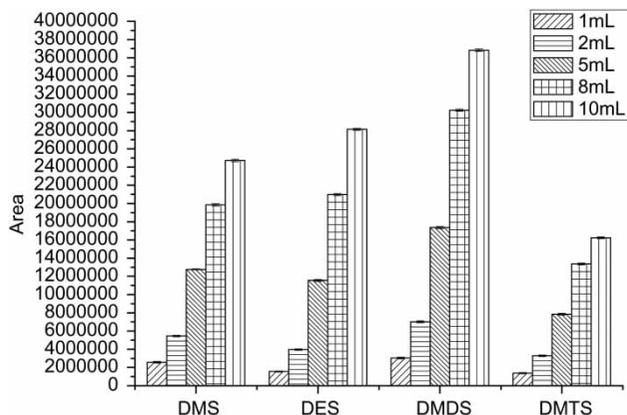


Figure 1 | Effect of sample volume on the headspace composition of samples containing dimethyl sulfide (DMS), diethyl sulfide (DES), dimethyl disulfide (DMDS), and dimethyl trisulfide (DMTS).

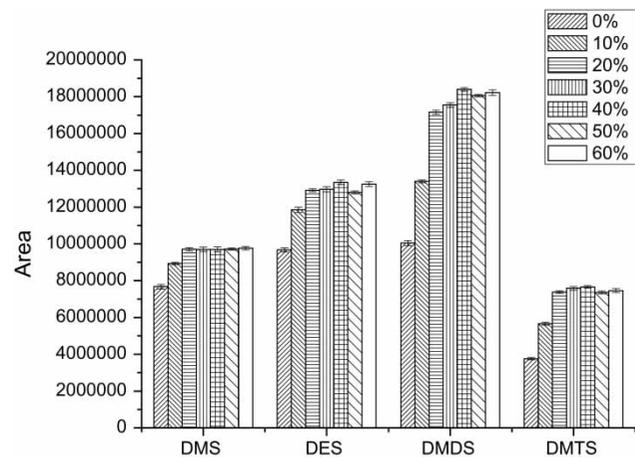


Figure 2 | Salting-out effect in the headspace composition of samples containing dimethyl sulfide (DMS), diethyl sulfide (DES), dimethyl disulfide (DMDS), and dimethyl trisulfide (DMTS).

subsequent experiments were performed by addition of 20% NaCl solid.

Rotation stirring

To investigate the influence of rotational stirring on the analysis efficiency, the stirring rate was varied from 0–115.4 r/min (0, 34.3, 57.1, 80, 95.2, and 115.4 r/min). All samples contained the same concentration of VOSCs of 10 mL per vial. The results showed that stirring the liquid sample could accelerate equilibration between the liquid and headspace. This effect was exemplified by the increase in relative peak areas as the stirring rate was increased from 0 to 34.3 r/min (Figure 3). However, the analysis

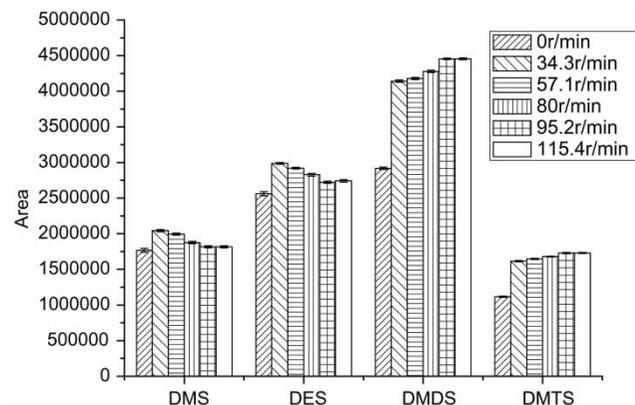


Figure 3 | Effect of rotational stirring on the headspace composition of samples containing dimethyl sulfide (DMS), diethyl sulfide (DES), dimethyl disulfide (DMDS), and dimethyl trisulfide (DMTS).

efficiency of DMDS and DMTS did not appreciably increase at stirring rates greater than 34.3 r/min, and that of DMS and DES decreased. These trends might be due to slight volatilization of more volatile species (DMS and DES) with increasing pressure.

Headspace duration

For experiments on headspace heating, a fixed temperature was chosen (90 °C), and the duration of headspace analysis was varied. Analysis times of 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 min were used for optimization. All samples contained the same concentration of VOSCs in 10 mL per vial. As shown in Figure 4, the peak area markedly increased from 10 to 20 min, and then decreased slightly from 20 to 100 min. Thus, analytes in the water and gas phases were assumed to have reached equilibrium in 20 min. Therefore, 20 min duration was used in subsequent headspace experiments. The decrease in peak area from 20 to 100 min could be due to slight volatilization of analytes. As the composition of actual samples was more complex than that of the standard samples, the matrix effect could affect the durations of headspace analysis (Guillen & Ibargoitia 1998; Fritsch & Schieberle 2005). Therefore, durations for the headspace analysis of real samples were different from those for the standard samples. The durations for the headspace analysis of real samples were the same as that of the standard samples in this study.

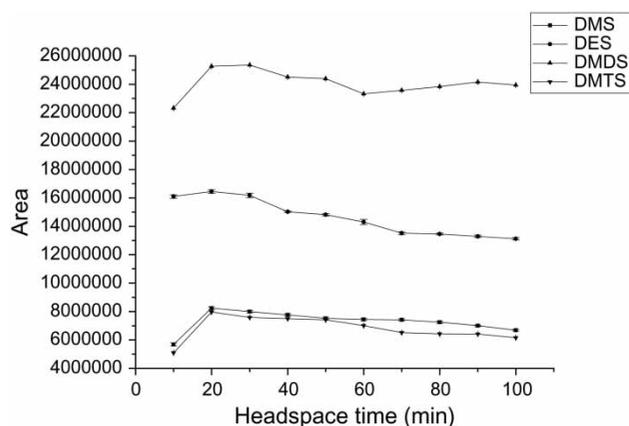


Figure 4 | Effect of analysis duration on the headspace composition of samples containing dimethyl sulfide (DMS), diethyl sulfide (DES), dimethyl disulfide (DMDS), and dimethyl trisulfide (DMTS).

Headspace temperature

Headspace analysis was performed at 50, 60, 70, 80, and 90 °C for 20 min. Figure 5 shows that the peak areas of all analytes increased from 50 to 90 °C. All samples contained the same concentration of VOSCs of 10 mL per vial. Higher temperatures typically increased the volatility of compounds, thus increasing their concentrations in the headspace. However, the instrument and capillary column could be damaged with the increase in temperatures because of the production of more water vapor. As the boiling point of water was 100 °C, 90 °C was used in all subsequent headspace experiments.

Performance of the analytical method

Quality assurance/quality control

The samples were collected in 250 mL amber glass bottles with polytetrafluoroethylene (PTFE) screw caps and without headspace to avoid the evaporation of volatile compounds. The water samples were stored at 4 °C and analysis should be within 1 day of collection. When the time between sample collection and analysis exceeded 1 day, samples were stored at -20 °C for up to 7 days. There is no organic interference in the sample storage area.

The entire journey blanks were analyzed to eliminate VOSCs peaks attributable to sources other than the samples.

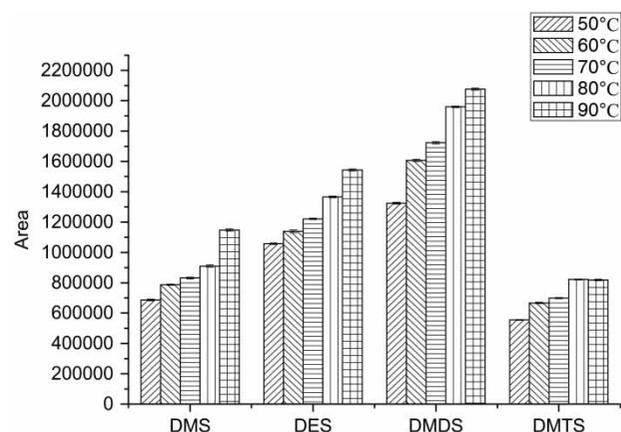


Figure 5 | Effect of the headspace temperature on the headspace composition of samples containing dimethyl sulfide (DMS), diethyl sulfide (DES), dimethyl disulfide (DMDS), and dimethyl trisulfide (DMTS).

To avoid reporting VOSCs related to column and septum bleed, VOSCs analysis was performed without sample injection. Quality control samples were analyzed in each batch of samples. In addition, at least one in ten VOSCs samples were collected and analyzed in duplicate to ensure the precision of the test.

Linearity

The optimized headspace conditions used were as follows: 10 mL water sample with 20% NaCl mass fraction in a 20 mL headspace vial; rotational stirring at 34.3 r/min; temperature of 90 °C; and analysis for 20 min. The performance of the method under these conditions was investigated by using standard aqueous solutions of the four analytes at different concentrations (0.01–100.0 µg/L, Table 2). Analysis for these substances had good linearity by external standard method with the correlation coefficients over 0.9990.

Table 2 | Performance parameters for the determination of VOSCs using HS-GC/MS

Analyte	Linear range (µg/L)	R ²	MDL _s (µg/L)	MQL _s (µg/L)
DMS	0.5–100.0	0.9992	0.2	0.5
DES	0.06–100.0	0.9995	0.02	0.06
DMDS	0.06–100.0	0.9994	0.02	0.06
DMTS	0.06–100.0	0.9990	0.02	0.06

Table 3 | Average recoveries and relative standard deviation values

VOSC _s	Surface water				Pipe-network water			
	Nominal concentration (µg/L)	Measured concentration (µg/L)	Recovery (%)	RSD (%)	Nominal concentration (µg/L)	Measured concentration (µg/L)	Recovery (%)	RSD (%)
DMS	80.0	83.4	104.2	4.7	80.0	82.1	102.6	3.9
	5.0	5.18	103.6	5.4	5.0	5.26	105.2	5.5
	1.0	1.14	114.0	2.7	1.0	1.10	110.0	3.5
DES	80.0	79.4	99.2	5.1	80.0	78.9	98.6	5.3
	5.0	5.24	104.8	6.1	5.0	5.43	108.6	6.5
	0.1	0.11	110.0	5.5	0.1	0.09	90.0	5.3
DMDS	80.0	81.2	101.5	5.0	80.0	82.6	103.2	2.4
	5.0	5.15	103.0	4.7	5.0	5.40	108.0	5.2
	0.1	0.1	100.0	2.1	0.1	0.09	90.0	3.7
DMTS	80.0	78.6	98.2	3.1	80.0	84.9	106.1	4.7
	5.0	4.98	99.6	2.9	5.0	4.95	99.0	3.0
	0.1	0.11	110.0	4.2	0.1	0.09	90.0	3.6

Method detection limits (MDLs) and method quantitation limits (MQLs)

MDLs and MQLs were calculated from the calibration plots by considering the peak area corresponding to three and ten times the signal-to-noise ratio of a procedural blank. MDLs for analysis of DMS was 0.2 µg/L, and that for analysis of the other compounds was 0.02 µg/L. MQLs for analysis of DMS was 0.5 µg/L, and that for analysis of the other compounds was 0.06 µg/L. Calibration using VOSCs showed good linearity (Table 2). However, the VOSCs with low molecular weight (MW), especially DMS, were more prone to analytical bias because of the distinctively reduced sensitivity. As such, the MDLs vary by a factor of 10 (0.2 µg/L for DMS and 0.02 µg/L for the other VOSCs). MQLs corresponded to the lowest calibration levels. MDLs and MQLs for analysis of the four target compounds are listed in Table 2.

Recovery

Recoveries for analysis of DMS, DES, DMDS, and DMTS in surface water and tap water were determined by spiking a predetermined amount of these compounds into both matrixes and then calculating the relative difference between the measured and actual concentrations. Excess sodium thiosulfate (Na₂S₂O₃) was added to the tap water matrix to remove residual chlorine before the experiment.

The matrixes were analyzed at the same time. The results indicated that the matrixes of the surface water and tap water had a negligible effect on the performance of the method. Results were based on three tests for each compound with three different concentration levels. The external standard method was used for the quantitative analysis. Recoveries (Table 3) with the optimized method using the spiked samples were appropriate.

Comparison with other methods

The static headspace method is compared with other reported methods for VOSCs measurement in Table 4. As this method did not require preconcentration processes, the analysis time for each sample was shorter than the HS-SPME method and the P&T method. In addition, the use of fiber, as in the case of the HS-SPME method, was avoided. The calibration range of this method covers three or four orders of magnitude, comparable to other methods. The correlation coefficients (>0.9990) are higher than coefficients attained with other preconcentration processes. MDLs of our method are lower than those obtained by using purge and trap preconcentration or other static headspace methods, although they are considerably higher than those achieved with the HS-SPME method. The recoveries are also higher than those obtained with other preconcentration processes.

Sample analysis

The method was applied for the analysis of several samples of surface water and tap water from four plants for surface water treatment. Only DMS was detected in surface water samples (1–7 $\mu\text{g/L}$). This result indicates that this compound is present at concentrations above its odor threshold value. Figure 6 shows the total ion chromatograms of surface water spiked with 5.0 $\mu\text{g/L}$ of a standard mixture.

CONCLUSIONS

In the present study, a headspace method coupled with GC/MS to analyze volatile sulfides, disulfides, and trisulfides in surface water and tap water samples has been developed

Table 4 | Comparison of various methods for the VOSCs analysis^a

No.	Analyte	Apparatus	Preconcentration	Analysis time per sample (min)	Calibration range ($\mu\text{g/L}$)	R^2	Detection limits ($\mu\text{g/L}$)	RSD (%)	Recovery (%)	Reference
1	DMS, DES, DMDS, and DMTS	GC/MS	Static headspace	27	0.06–100	0.9990–0.9995	0.02–0.2	2.6–6.5	98.5–110.4	This study
2	DMS, DMDS, and DMDS	GC-FPD	HS-SPME	53	0.01–10	0.9989–0.9998	0.01–0.05	3.7–10.3	90.1–111.8	Lu et al. (2012)
3	MT, DMS, and DMDS	GC-SCD	Static headspace	17	0.5–500	0.9995–0.9998	0.08–0.21	2.1–2.3	83–103	Sun et al. (2014)
4	MT, DMS, and DMDS	GC/MS	Purge and trap	58	5–500	0.993–0.998	1.2–4.8	0–8	81–100	Cheng et al. (2007)
5	DMS, EMS, DES, and DMDS	GC/MS	HS-SPME	70	0.0044–10.6	0.995–0.997	0.006–0.035	4.1–6.1	Not mentioned	Abalos et al. (2002)

^aMT denotes methanethiol.

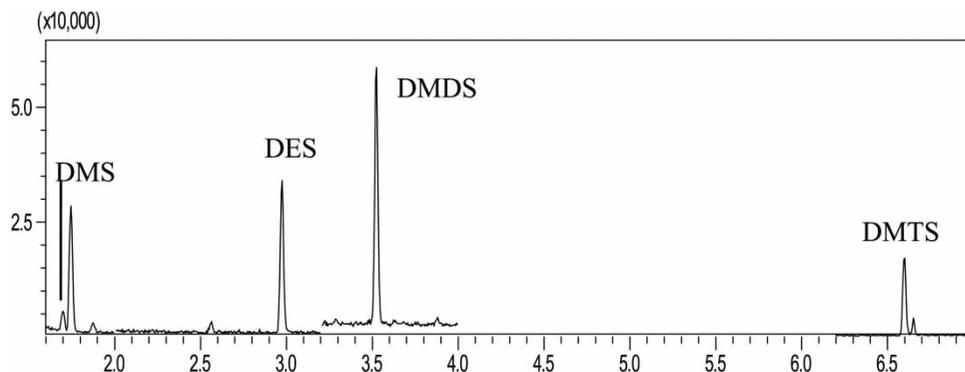


Figure 6 | Total ion current of DMS, DES, DMDS, and DMTS, respectively.

and applied. Parameters that might affect the method performance have been optimized. Headspace conditions were as follows: 10 mL aqueous samples, addition of 20% NaCl mass fraction, 20 min headspace analysis, stirring at 34.3 r/min, and 90 °C headspace temperature. The optimized method was validated by using spiked samples of surface water and tap water. Low MDLs (0.02–0.2 µg/L) and high recoveries (90.0%–114.0%; RSD of 2.1%–6.5%, $n = 3$) were obtained. DMS was found in surface water and tap water samples from four plants for surface water treatment.

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