

Analysis of Phenols and Chlorinated Phenols in Drinking Water by GC/MS

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Key Words

- US EPA 528
- US EPA 604 and US EPA 8041
- chlorinated phenols
- phenols
- phenols in waste water
- phenols in drinking water
- TraceGOLD TG-5SiIMS
- 5% Phenyl

Introduction

US EPA method 528 is an analytical GC/MS method used for determining certain phenols and chlorinated phenols in drinking water and untreated source water.

The EPA method 528 was performed using a splitless injection mode on a Thermo Scientific TRACE GC coupled to a Thermo Scientific Ion Trap mass spectrometer.

The Thermo Scientific TraceGOLD TG-5SiIMS column provides excellent performance for the analysis of phenols and chlorinated phenols in accordance with EPA method 528. This can also be extended to EPA methods 604 and 8041, which analyses the same compounds, using FID and ECD detection rather than mass spectrometry.

Goal

To demonstrate the suitability and performance of TraceGOLD™ TG-5SiIMS for the analysis of EPA method 528; phenols and chlorinated phenols in drinking water.

Experimental details

The phenols and chlorinated phenols stated in the EPA methods 528 were run on a TRACE™ GC fitted with a TriPlus autosampler. The ion trap mass spectrometer was used in a segmented mode to allow precise control of groups of ions for improved ion statistics and ion ratios. The column used for analyses was a low polarity silylene phase with selectivity comparable to a 5% diphenyl/95% dimethyl polysiloxane phase. The data was acquired and processed using Thermo Scientific Xcalibur data handling software.

Sample preparation

A pre-mixed 1 ng/μL of phenolic standard solution prepared in dichloromethane was used for the analysis.

Column	Part Number
TraceGOLD TG-5SiIMS, 30 m × 0.25 mm × 0.25 μm,	26096-1420
Guard Column 2 m × 0.32 mm	260RG497
Press-Fit Union	64000-001



Thermo Scientific TriPlus Autosampler

Sample volume	1 μL
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TRACE GC Ultra

Oven Program	60 °C (5 min), 8 °C/min, 300 °C (10 min)
Equilibration Time	0.5 min
Injector	275 °C, Splitless (1 min)
Split Flow	30 mL/min
Column Flow	Helium, 1.5 mL/min (constant flow)
Transfer Line Temperature	300 °C

Thermo Scientific Ion Trap MS

MS Type	ITD 230 LT (250 L turbo pump)
MS Source Temperature	225 °C
MS Source Current	250 μA
Electron Energy	70 eV
Filament Delay	5 min
MS Acquisition Mode	El+, 45-450 amu Segmented Scan

Consumables

Consumables	Part Number
BTO 17 mm septa	31303211
3 mm ID Focus Liner, 105 mm long	45350032
Liner graphite seal	29033406
10 μL, 80 mm Syringe	36502019
Graphite ferrules to fit 0.32 mm id columns	29053487
Graphite/vespel 0.25 mm ID ferrules for GC/MS interface	29033496
2 mL clear vial and Si/PTFE seal	60180-599

Results

The requirement for the US EPA methods 528, 604 and 8041 were achieved with separation of all of the phenolic analytes using the TraceGOLD TG-5SiIMS on a GC/MS system. The stationary phase in the TraceGOLD TG-5SiIMS provides excellent performance due to minimal interaction of active compounds with active sites on the column, therefore no significant peak tailing was observed. Figure 1 shows the TIC chromatogram for 1 ng/ μ L of phenols, including chlorinated phenols in dichloromethane obtained using a TraceGOLD TG-5SiIMS column. Table 1 shows the peak identification of phenol compounds according to the retention times on TraceGOLD TG-5SiIMS column.

Conclusions

The TraceGOLD TG-5SiIMS column demonstrated excellent performance for the analysis of phenols and chlorinated phenols with no significant peak tailing observed for the analytes. The chromatogram illustrates the superior performance of the TraceGOLD TG-5SiIMS column for the analysis of phenols and chlorinated

phenols in accordance with EPA method 528. Furthermore, the excellent separation exhibited by the column makes it ideal for the analysis of the same compounds using different detection systems in accordance with EPA methods 604 and 8041.

References

1. EPA method 528:
www.epa.gov/nerlcwww/m_528.pdf
2. EPA method 604:
www.epa.gov/waterscience/methods/method/organics/604.pdf
3. EPA method 8041:
www.epa.gov/osw/hazard/testmethods/sw846/pdfs/8041a.pdf

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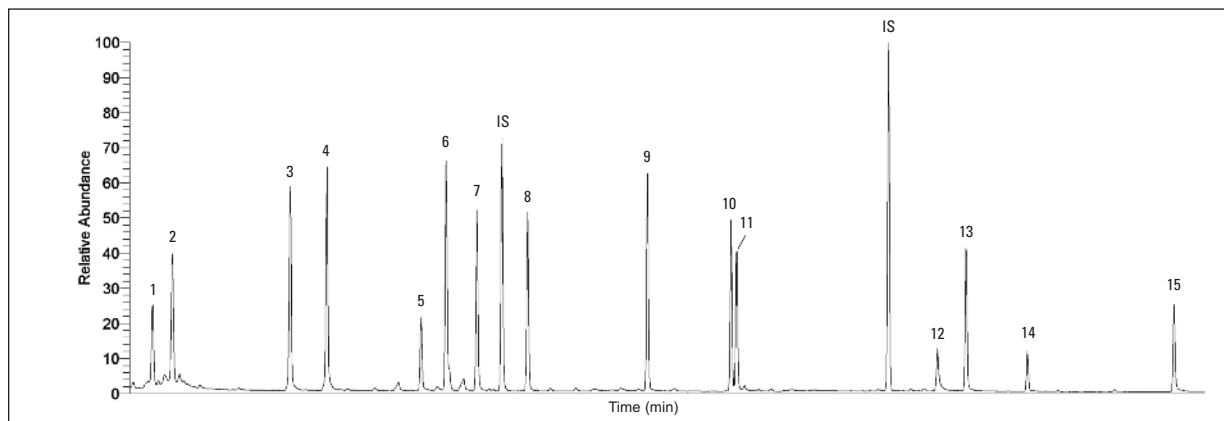


Figure 1: TIC chromatogram of 1 ng/ μ L of Phenolic compounds separated on a TraceGOLD TG-5SiIMS column

Phenols	Peak No.	Phenols	Peak No.
Phenol	1	4-Chloro-3-Methylphenol	9
2-Chlorophenol	2	2,4,5-Trichlorophenol	10
o-cresol	3	2,4,6-Trichlorophenol	11
p-Cresol	4	Internal Standard	IS
2-Nitrophenol	5	4-Nitrophenol	12
2,4-Dimethylphenol	6	2,3,4,5-Tetrachlorophenol	13
2,4-Dichlorophenol	7	2-Methyl-4,6-Dinitrophenol	14
Internal Standard	IS	Pentachlorophenol	15
2,6-Dichlorophenol	8		

Table 1: List of Phenolic compounds according to retention times

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