DETERMINATION OF PHENOLIC COMPOUNDS IN WATER (HJ 676-2013)

Technology Advantage: Agilent Intuvo 9000 GC with FID

Introduction

HJ 676-2013 is a method approved by the Chinese Ministry of Environmental Protection for the determination of 13 phenolic compounds in water by gas chromatography with flame ionization detection (FID). The method specifies procedures for sample extraction, analysis, identification, and quantitation [1].

This application brief demonstrates that the Agilent Intuvo 9000 GC system can easily achieve the performance specification for the analysis of phenols, as outlined in method HJ 676-2013.

Instrumentation

- Intuvo 9000 GC system with FID
- Agilent DB-5ms UI 30 m × 0.32 mm, 0.25 µm column
- All instrumental settings were the same as those listed in the method.
- The Guard Chip was operated in oven track mode.

Sample Preparation

- A stock mixture of 13 phenols at a concentration of 250 µg/mL was used to prepare standards for the study.
- Standards were prepared in a 1:1 mixture of dichloromethane and ethyl acetate (v/v) at the concentrations listed in the method, ranging from 1 to 250 µg/mL.

For more information, visit:
www.agilent.com

Agilent Technologies

Results and Discussion

Figure 1 shows an example chromatogram of the 13 phenols at a concentration 25 µg/mL. Table 1 lists the peak number identifications. Peak shapes should be noted for 2,4-dinitrophenol, 4-nitrophenol, and pentachlorophenol. These compounds are among the most acidic within the group of phenols and the most difficult to analyze. The Intuvo demonstrates good performance for the analysis of these compounds, indicating a highly inert flowpath from inlet to detector.

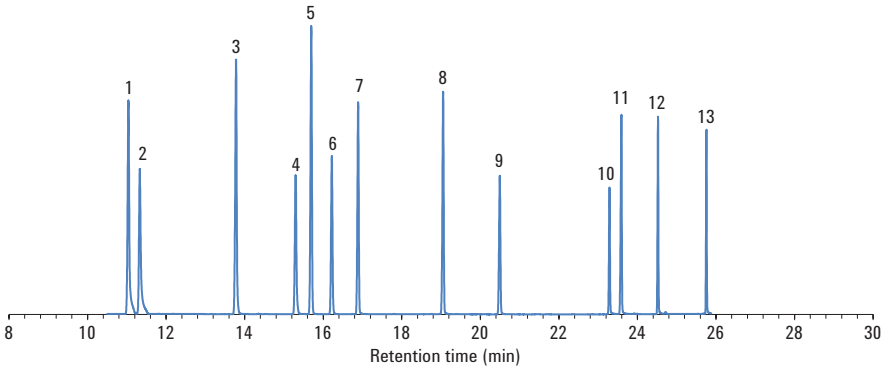

Figure 1. Chromatogram of the 13 target phenols.

Compound identification is based upon the retention time of a target eluting within a range of retention times. The time range is defined as three standard deviations of the average retention time for the calibration standards (t ±3s). Table 1 lists the average retention time, standard deviation, retention time window, and retention time range. Retention time precision is quite good. For two closely eluting compounds, phenol and 2-chlorophenol, the retention time windows do not overlap. This allows confident identification and quantitation for these two compounds.

Quantitation is based upon instrument response calibration. Peak area was used to construct a calibration curve fitted with a straight line. For a calibration to be valid, the correlation coefficient must be greater than or equal to 0.995, and the calculated concentration of the middle calibration standard must be within ±20 % of the actual concentration. Table 2 lists the correlation coefficient, calculated concentration of the middle standard, and associated error. All targets pass the method calibration requirements.

The method requires a repeatability test. For 10 injections, the percent relative standard deviation (RSD) in peak area should be within ±25 %. Figure 2 shows an overlay of 10 repeat injections of the 25 µg/mL standard. For all 13 phenols, the area RSD is well within the 25 % limit,

Table 1. Retention time windows for target identification.

No.	Target	Average RT (min)	Std. dev. (min)	RT Window (min)	Range (min)
1	Phenol	11.036	0.015	10.992 – 11.080	0.09
2	2-Chlorophenol	11.335	0.014	11.292 – 11.377	0.08
3	3-Methylphenol	13.774	0.015	13.728 – 13.819	0.09
4	2-Nitrophenol	15.301	0.018	15.246 – 15.355	0.11
5	2,4-Xylenol	15.691	0.013	15.653 – 15.729	0.08
6	2,4-Dichlorophenol	16.216	0.010	16.185 – 16.246	0.06
7	4-Chlorophenol	16.893	0.020	16.833 – 16.952	0.12
8	4-Chlorine-3-methylphenol	19.049	0.011	19.016 – 19.801	0.06
9	2,4,6-Trichlorophenol	20.498	0.017	20.448 – 20.548	0.10
10	2,4-Dinitrophenol	23.307	0.030	23.217 – 23.396	0.18
11	4-Nitrophenol	23.593	0.027	23.510 – 23.675	0.16
12	2-methyl-4,6-dinitrophenol	24.539	0.027	24.458 – 24.619	0.16
13	Pentachlorophenol	25.758	0.008	25.733 – 25.783	0.05

Table 2. Calibration metrics for target compounds.

No.	Target	Correlation coefficient	Calculated concentration of middle standard (µg/mL)	Percent error in calculated middle standard concentration
1	Phenol	0.9998	24.7	-1.4
2	2-Chlorophenol	0.9998	51.0	1.9
3	3-Methylphenol	0.9998	24.9	-0.5
4	2-Nitrophenol	0.9998	50.6	1.3
5	2,4-Xylenol	0.9998	24.9	-0.4
6	2,4-Dichlorophenol	0.9998	51.0	2.0
7	4-Chlorophenol	0.9998	51.1	2.1
8	4-Chlorine-3-methylphenol	0.9998	25.1	0.3
9	2,4,6-Trichlorophenol	0.9998	50.9	1.9
10	2,4-Dinitrophenol	0.9998	124.2	-0.6
11	4-Nitrophenol	0.9999	50.8	1.6
12	2-methyl-4,6-dinitrophenol	0.9999	125.0	0.0
13	Pentachlorophenol	0.9999	50.8	1.6

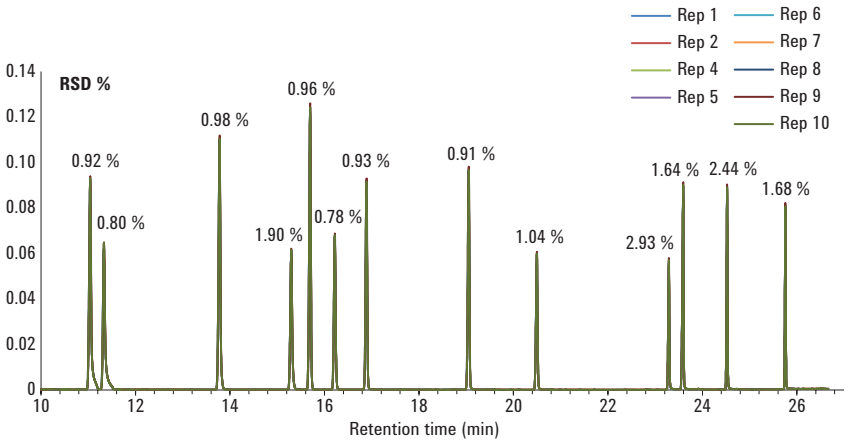

Figure 2. Overlay of 10 replicate injections with peak area percent RSD labeled.

Conclusion

The Agilent Intuvo 9000 GC system demonstrates outstanding performance for the analysis of phenols, and meets the requirements specified in HJ 676-2013.

Reference

1. Water Quality – Determination of Phenolic Compounds Liquid-Liquid Extraction Gas Chromatography. National Environmental Protection Standard of the People's Republic of China. HJ 676-2013.

www.agilent.com
Information, descriptions and specifications in this publication are subject to change without notice.

Published in USA, December 1, 2017
5991-8319EN

Agilent Technologies