

Determination of Phenols in Drinking Water with Agilent Bond Elut Plexa SPE and HPLC

Application Note

Environmental

Author

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Abstract

A method for simultaneous determination of 11 phenols in drinking water was developed and validated. The analytes were extracted by solid phase extraction (SPE) with an Agilent Bond Elut Plexa cartridge, and separated by HPLC using an Agilent Poroshell 120 column. Overall recoveries ranged from 87% to 108%, with RSD values between 1.4% and 6.7%. The method was simple and effective for the extraction, enrichment, and analysis of multiple phenol compounds in drinking water.


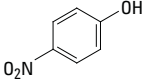

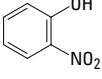
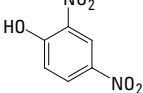
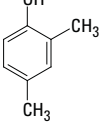
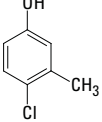
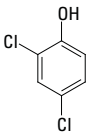
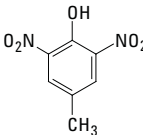
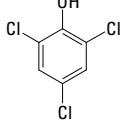
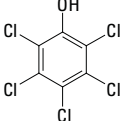
Introduction

Phenols are harmful to people, dangerous to the environment, and can impart a metallic taste and odor to water when it is chlorinated. Frequently, these compounds find their way into the environment as water pollutants, making analytical determination of phenols necessary. This application note describes the development of a sample extraction, cleanup, and enrichment method with an Agilent Bond Elut Plexa cartridge and a separation method using an Agilent Poroshell 120 column, with quantification by HPLC [1]. In line with EPA 604, 11 phenol compounds were chosen [2]. The structures and pKa for the compounds used in this study are shown in Table 1.



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Table 1. Phenols Used in This Study

| No. | Name | pKa | Structure |
|-----|----------------------------|------|---|
| 1 | Phenol | 9.89 |  |
| 2 | 4-nitrophenol | 7.08 |  |
| 3 | 2-chlorophenol | 8.56 |  |
| 4 | 2-nitrophenol | 7.22 |  |
| 5 | 2,4-dinitrophenol | 4.09 |  |
| 6 | 2,4-dimethylphenol | 10.6 |  |
| 7 | 4-chloro-3-methylphenol | 9.56 |  |
| 8 | 2,4-dichlorophenol | 7.68 |  |
| 9 | 4,6-dinitro-2-methylphenol | |  |
| 10 | 2,4,6-trichlorophenol | 7.41 |  |
| 11 | pentachlorophenol | 4.92 |  |

Experimental

Reagents and chemicals

The standards solution and other chemicals were purchased from Sigma-Aldrich (Saint Louis, MO, USA). All reagents and solvents were HPLC or analytical grade. Methanol, acetonitrile, water, and tetrahydrofuran were from Honeywell (Muskegon, MI, USA). Formic acid (FA) was from Fluka (Sleinheim, Germany).

Standard solutions

The standard solution (500 µg/mL for phenol, 2-nitrophenol, 2-chlorophenol, 2,4-dichlorophenol, and 2,4-dimethylphenol; 1500 µg/mL for 2,4-dinitrophenol and 2,4,6-trichlorophenol; 2500 µg/mL for 4-chloro-3-methylphenol, 4,6-dinitrophenol-2-methylphenol, 4-nitrophenol, and pentachlorophenol) was made in methanol. The QC spiking solutions were made fresh daily in 1:1 acetonitrile:water with 0.1% formic acid.

Equipment and materials

| | |
|------------------------|--|
| Instrument | Agilent 1200 series HPLC system |
| Column | Agilent Poroshell 120 SB-C18, 2.1 x 100 mm, 2.7 µm (p/n 685775-902) |
| Solid phase extraction | Agilent Bond Elut Plexa cartridge, 60 mg, 3 mL (p/n 12109603) |
| Vacuum manifold | Agilent Vac Elut 20 Manifold Tall Glass Basin (p/n 12234104) |

HPLC conditions

| | | |
|--------------|---|-------|
| Mobile phase | A: 0.1% Formic acid in water B: 0.1% Formic acid in acetonitrile | |
| Gradient | time (min) | B (%) |
| | 0 | 15 |
| | 10 | 50 |
| | 11 | 90 |
| | 12 | 90 |
| | 12.5 | 15 |
| | 14 | 15 |
| Flow rate | 0.4 mL/min | |
| Detection | UV, 280 nm | |
| Injection | 40 µL | |

Sample preparation

Drinking water was purchased from a local supermarket. A 250 mL water sample was prepared by adjusting the pH to 2.0 with phosphoric acid solution (0.1 mol/L). The Agilent Bond Elut Plexa cartridge was preconditioned with 3 mL tetrahydrofuran and 3 mL methanol, then equilibrated with 3 mL water.

The 250 mL sample was passed through the cartridge at a rate of 5 mL/min under vacuum. After the sample passed completely through the cartridge, the cartridge was washed with 2 mL water and discarded. The cartridge was then dried under negative pressure below 2.0 kPa for 3 minutes. Finally, the cartridge was eluted with 3 mL tetrahydrofuran. The collected eluent was reduced to about 0.2 mL under nitrogen at room temperature. The resulting solution was brought up to 0.5 mL accurately with water. Then the solution was filtered through a 0.45- μ m filter membrane (p/n 5185-5836) and analyzed by HPLC. The procedure used for the SPE extraction is shown in Figure 1.

Results and Discussion

Matrix blank material was prepared by taking the drinking water through the entire extraction and sample cleanup procedure. External-standard calibration curves were made by spiking matrix blanks at respective concentrations. Linear regressions were calculated for the phenols using the areas under the peaks and the spiked concentrations. The precision of the method was determined as recoveries of spiked phenols in drinking water after the SPE procedure, and the analysis was performed with six replicates at each level. The chromatograms of the blank, the spiked standard, and the standard are shown in Figure 2. The linearity, recovery and reproducibility data are shown in Table 2.

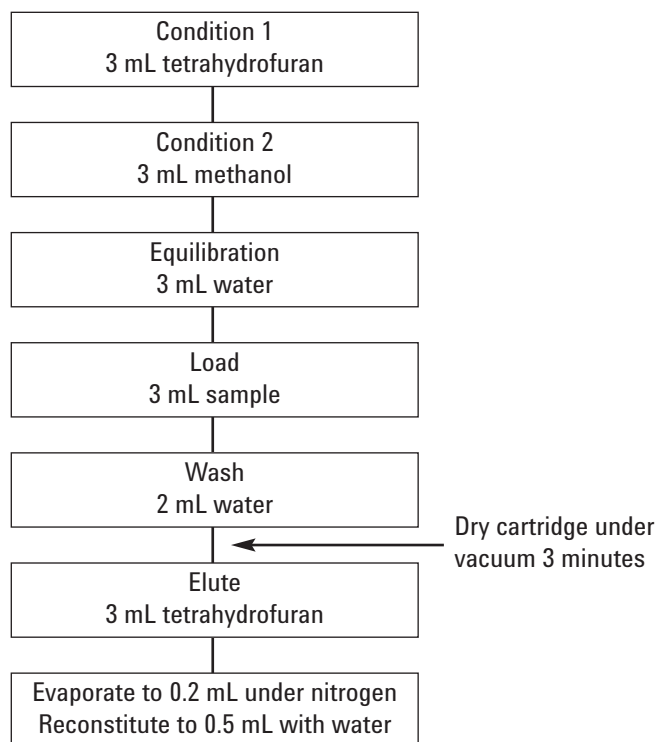


Figure 1. SPE procedure for phenols in water.

Table 2. Linearity, Recoveries and RSDs of Phenols in Drinking Water with SPE

| Compound | Regression equation | Correlation coefficient | Spiked level (μ g/L) | Recovery (%) | RSD (n=6, %) |
|----------------------------|-------------------------|-------------------------|---------------------------|--------------|--------------|
| Phenol | $Y = 75.621x - 0.1425$ | 0.9995 | 2.5 | 96.7 | 5.1 |
| 4-Nitrophenol | $Y = 82.313x - 0.0897$ | 0.9992 | 12.5 | 98.2 | 1.4 |
| 2-Chlorophenol | $Y = 103.267x - 0.2578$ | 0.9986 | 2.5 | 93.7 | 5.6 |
| 2-Nitrophenol | $Y = 68.304x + 0.0752$ | 0.9993 | 2.5 | 92.6 | 4.8 |
| 2,4-Dinitrophenol | $Y = 75.366x - 0.2335$ | 0.9997 | 7.5 | 99.3 | 2.8 |
| 2,4-Dimethylphenol | $Y = 66.689x - 0.1226$ | 0.9984 | 2.5 | 99.5 | 6.2 |
| 4-Chloro-3-methylphenol | $Y = 76.407x + 0.0571$ | 0.9991 | 12.5 | 87.3 | 3.7 |
| 2,4-Dichlorophenol | $Y = 59.018x - 0.2639$ | 0.9979 | 2.5 | 91.5 | 2.3 |
| 4,6-Dinitro-2-methylphenol | $Y = 125.735x + 0.3428$ | 0.9996 | 12.5 | 90.6 | 1.9 |
| 2,4,6-Trichlorophenol | $Y = 70.028x - 0.1112$ | 0.9989 | 7.5 | 96.4 | 2.9 |
| Pentachlorophenol | $Y = 62.135x + 0.1457$ | 0.9991 | 12.5 | 108.2 | 6.7 |

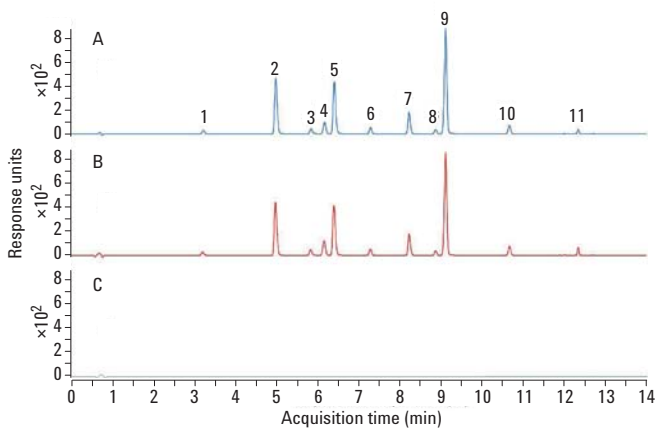


Figure 2. Chromatograms: A- Standard solution with matrix blank; B- Spiked standards in drinking water with SPE procedure; C-Drinking water blank sample with SPE procedure. 1) Phenol, 2) 4-Nitrophenol, 3) 2-Chlorophenol, 4) 2-Nitrophenol, 5) 2,4-Dinitrophenol, 6) 2,4-Dimethylphenol, 7) 4-Chloro-3-methylphenol, 8) 2,4-Dichlorophenol, 9) 4,6-Dinitro-2-methylphenol, 10) 2,4,6-Trichlorophenol, 11) Pentachlorophenol.

Conclusion

Agilent Bond Elut Plexa provides a simple and effective single cartridge method for the extraction and enrichment of multiple phenol compounds in drinking water. The recovery and reproducibility results based on solution standards are acceptable for phenol residues determination in drinking water. The impurities from water were minimal and did not interfere with any of the phenols analyzed.

References

1. William J. Long and Anne E Mack (2010) Fast Analysis of Environmental Phenols with Agilent Poroshell 120 EC-C18 columns. Agilent Technologies, Inc. Publication 5990-6156EN.
2. US Environmental Protection Agency. Method 604 – Phenols.

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