EPA 8330A Analysis of Explosives Using Agilent Poroshell 120 EC-CN and EC-C18 Columns

Application Note

Environmental

Author

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Abstract

A group of 14 nitroaromatics and nitramines was analyzed according to EPA Method 8330A for the trace analysis of explosive residues by HPLC/UV. The analysis was improved by using superficially porous Agilent Poroshell 120 columns as compared to the traditional 5 µm columns suggested by the United States Environmental Protection Agency (EPA). Two complimentary phases were used; Poroshell 120 EC-CN and Poroshell 120 EC-C18. As recommended by the EPA, the Poroshell 120 EC-CN column was a good confirmation column when paired with a Poroshell 120 EC-C18 column due to its orthogonal selectivity.

Introduction

There is significant interest in transferring LC methods to superficially porous particles from larger 5 µm totally porous particles. The high efficiency of superficially porous particles is similar to sub-2 µm totally porous particles. This is attributed primarily to a shorter mass transfer distance and a narrower particle size distribution. Furthermore, the larger particle size results in lower backpressure, allowing for these columns to be implemented in methods on nearly any LC system. The benefits of transferring from larger particle columns are very significant time and cost savings, because superficially porous particles are optimally run at faster flow rates and achieve similar resolution with a much shorter column length [1,2].

In this application note, a method from the EPA for the trace analysis of explosive residues by HPLC/UV was transferred from 5 µm columns to shorter 2.7 µm superficially porous Poroshell 120 columns.

Agilent Technologies

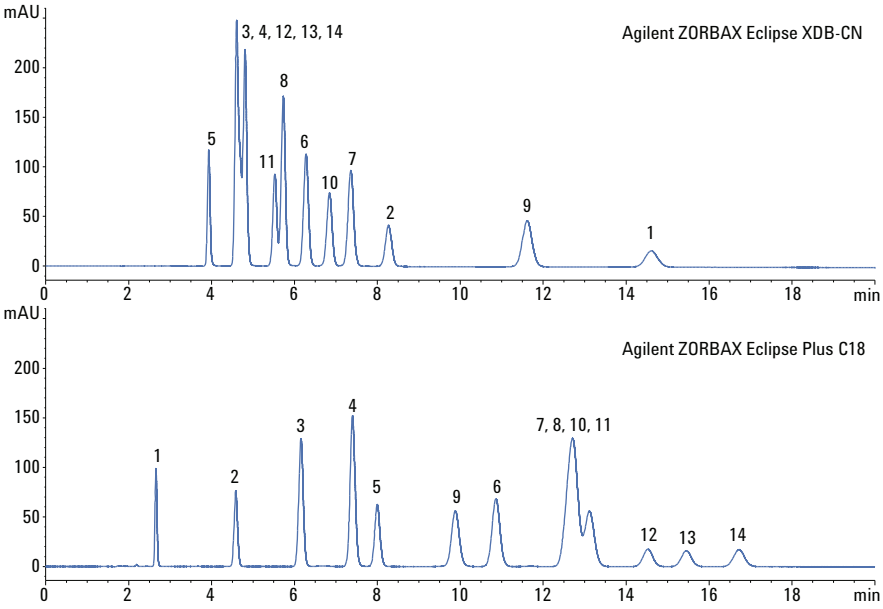
Materials and Methods

The instrument setup was optimized for lowest possible extra-column volume with short 0.075 mm id capillaries found in the Agilent Ultra Low Dispersion Kit (p/n 5067-5189) and with an Agilent LC System Rack (p/n 5001-3726) [3].

The EPA 8330A stock standard was purchased from Cerilliant Corp. The 14 explosive residues were obtained in an acetonitrile solution at a concentration of 200 µg/mL each. Methanol was purchased from Honeywell International Inc. Water was 18 Mohm.cm Milli-Q.

Results and Discussion

Figure 1 shows EPA Method 8330A for the trace analysis of explosive residues by HPLC/UV, performed as specified by the EPA with 4.6 × 250 mm, 5 µm columns. Two phases were used, Agilent ZORBAX Eclipse XDB-CN, and Agilent ZORBAX Eclipse Plus C18 for confirmatory analysis. Figure 1 shows the two phases had orthogonal selectivity. The nitrotoluene isomers coeluted on the CN column and were separated on the C18 column, while the dinitrotoluene compounds coeluted on the C18 and were separated on the CN column.



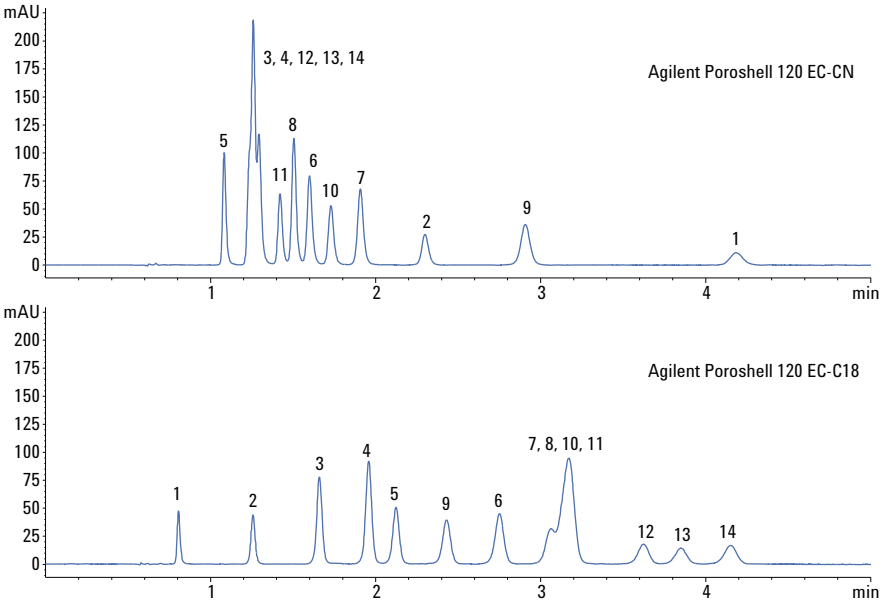
Peak id
1. HMX
2. RDX
3. 1,3,5-Trinitrobenzene
4. 1,3-Dinitrobenzene
5. Nitrobenzene
6. 2,4,6-Trinitrotoluene
7. 2-Amino-4,6-dinitrotoluene
8. 2,4-Dinitrotoluene
9. Tetryl
10. 4-Amino-2,6-dinitrotoluene
11. 2,6-Dinitrotoluene
12. 2-Nitrotoluene
13. 4-Nitrotoluene
14. 3-Nitrotoluene

Conditions

Columns:	Agilent ZORBAX Eclipse XDB-CN, 4.6 × 250 mm, 5 µm (p/n 990967-905)
	Agilent ZORBAX Eclipse Plus C18, 4.6 × 250 mm, 5 µm (p/n 959990-902)
Samples:	Explosive residues
Sample concentration:	20 µg/mL of each compound
Eluent:	A, H_2O; B, CH_3OH
Injection volume:	20 µL
Flow rate:	1 mL/min
Isocratic:	50% B
Temperature:	25 °C
Detector:	254 nm
Instrument:	Agilent 1290 Infinity LC

Figure 1. EPA 8330A analysis of explosives using EPA method parameters (isocratic) and Agilent ZORBAX Eclipse columns (5 µm).

Figure 2 shows that the 4.6 × 250 mm, 5 µm analyses can be transferred to 2.1 × 100 mm, 2.7 µm Poroshell 120 columns. Poroshell 120 EC-CN columns have very similar selectivity to ZORBAX Eclipse XDB-CN columns, while Poroshell 120 EC-C18 columns have similar selectivity to ZORBAX Eclipse Plus C18 columns. This allows for simplified method transfer. The flow rate (0.4 mL/min) and injection volume (1 µL) were scaled proportionally to the difference in column volume, while all other method parameters remained the same. The result was nearly the same but accomplished in a fraction of the time, saving significant time and solvent compared to the original analysis. This provided increased productivity as well as substantial cost savings.



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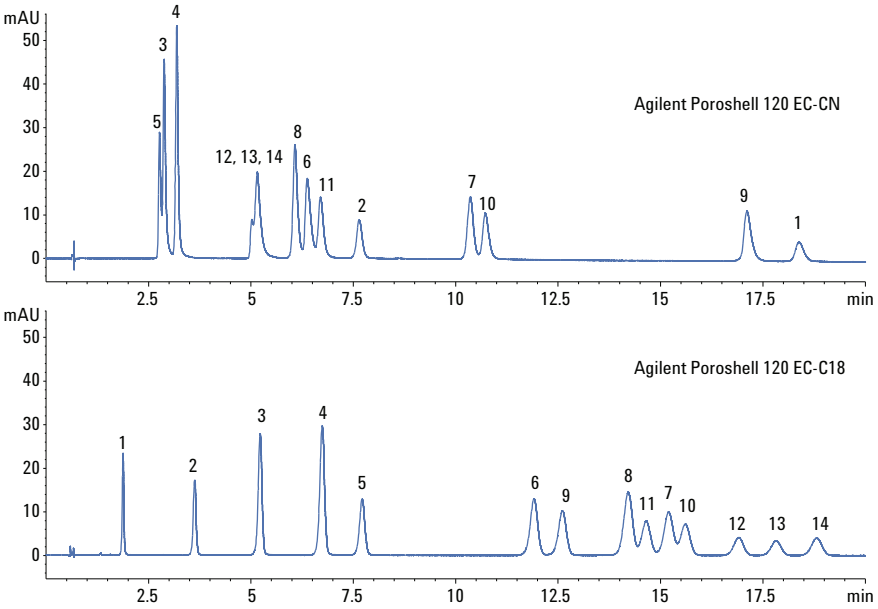
11. 2,6-Dinitrotoluene
12. 2-Nitrotoluene
13. 4-Nitrotoluene
14. 3-Nitrotoluene

Conditions

Columns:	Agilent Poroshell 120 EC-CN, 2.1 × 100 mm, 2.7 µm (p/n 695775-905)
	Agilent Poroshell 120 EC-C18, 2.1 × 100 mm, 2.7 µm (p/n 695775-902)
Samples:	Explosive residues
Sample concentration:	20 µg/mL of each compound
Eluent:	A, H_2O; B, CH_3OH
Injection volume:	1 µL
Flow rate:	0.4 mL/min
Isocratic:	50% B
Temperature:	25 °C
Detector:	254 nm
Instrument:	Agilent 1290 Infinity LC

Figure 2. EPA 8330A analysis of explosives using EPA method parameters (isocratic) and Agilent Poroshell 120 superficially porous columns.

Figure 3 shows the two Poroshell 120 columns with independently optimized gradient analyses; Poroshell 120 EC-CN (7% B for 7 minutes, 7-35% B in 13 minutes) and Poroshell 120 EC-C18 (25-36% B in 20 minutes). As can be seen, full resolution of the 14 nitroaromatics and nitramines was difficult, even with an optimized separation. This supports the need for orthogonal columns for confirmatory analyses.



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11. 2,6-Dinitrotoluene
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Conditions

Columns:	Agilent Poroshell 120 EC-CN, 2.1 × 100 mm, 2.7 µm (p/n 695775-905)
	Agilent Poroshell 120 EC-C18, 2.1 × 100 mm, 2.7 µm (p/n 695775-902)
Samples:	Explosive residues
Sample concentration:	20 µg/mL of each compound
Eluent:	A, H_2O; B, CH_3OH
Injection volume:	1 µL (ADVR on)
Flow rate:	0.4 mL/min
Gradient:	Poroshell 120 EC-CN, 7% B for 7 min, 7 to 35% B in 13 min; Poroshell 120 EC-C18, 25 to 36% B in 20 min
Temperature:	Agilent Poroshell 120 EC-CN, 20 °C; Agilent Poroshell 120 EC-C18, 25 °C
Detector:	254 nm
Instrument:	Agilent 1290 Infinity LC

Figure 3. EPA 8330A analysis of explosives using gradient elution with Agilent Poroshell 120 superficially porous columns; injection performed with Automatic Delay Volume Reduction.

Conclusions

Superficially porous Agilent Poroshell 120 columns were successfully substituted for traditional 5 µm columns for EPA Method 8330A for the trace analysis of explosive residues by HPLC/UV. The smaller dimension Poroshell 120 columns could be used to improve productivity and to save time and money over larger 5 µm columns. Additionally, it is found that the two Poroshell 120 phases (EC-CN and EC-C18) provided orthogonal selectivity, allowing for easy confirmatory analyses.

References

1. A. Gratzfeld-Hüsgen, E. Naegele. "Maximizing efficiency using Agilent Poroshell 120 columns". Application Note, Agilent Technologies, Inc. Publication Number 5990-5602EN (2010).

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3. A. Mack. "Optimizing Performance of an Agilent ZORBAX RRHD Eclipse Plus C18 Column by Enhancing an Agilent 1290 Infinity LC System for Ultra-Low Dispersion". Application Note, Agilent Technologies, Inc. Publication Number 5990-9502EN (2011).

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