Analysis of Explosives by UHPLC with Dual-Wavelength Detection

Agilent 1290 Infinity II LC System with Variable Wavelength Detector

Suitable for Agilent
1290 Infinity III LC

Application Note

Environmental

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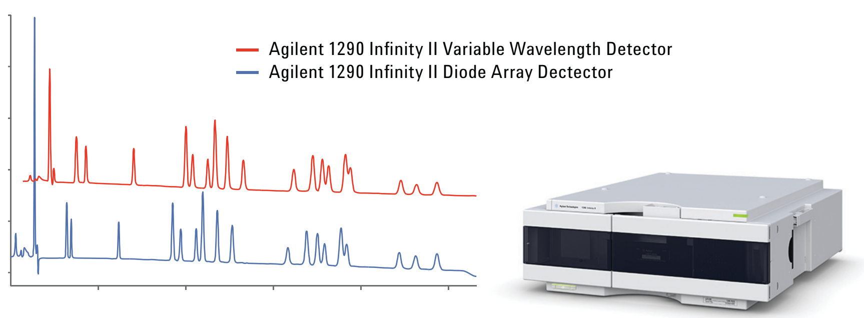
Abstract

This Application Note shows the analysis of 20 explosives with the Agilent 1290 Infinity II Variable Wavelength Detector (VWD) in dual-wavelength mode using 254 and 214 nm. The results were compared to the Agilent 1290 Infinity II Diode Array Detector (DAD) with respect to resolution, sensitivity, and linearity. Resolution and sensitivity were found to be absolutely comparable with both detectors. The 1290 Infinity II VWD achieved even higher sensitivity with lower limits of detection and quantification. If higher sensitivity is desired, the 60-mm flow cell of the 1290 Infinity II DAD offers superior performance. Also, both detectors showed high linearity with correlation coefficients better than 0.9999 for the majority of the analytes.



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Introduction

Nitroaromatics are organic compounds with one or more nitro functional groups ($-NO_2$) that are directly attached to an aromatic group. Most nitroaromatics are highly explosive, especially with an increasing number of nitro groups. It is of major importance to detect and identify these substances in environmental sites, for example, in soil from former military training grounds. Also, the analysis of explosives can be essential in crime-scene forensic investigation and homeland security. Among other environmental agencies, the Environmental Protection Agency (EPA) has published method 8330A/B[1], which has become the most commonly accepted method for sensitive UV-based analysis of nitroaromatics and nitramines[2,3,4].

The sample used for analysis was a standard mix of 20 explosives from Dr. Ehrenstorfer that also included nitropenta, which does not contain an aromatic group (Figure 1).

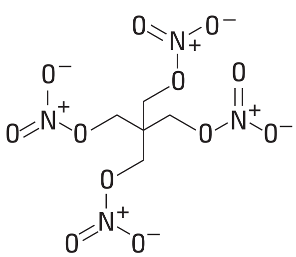

Figure 1. Nitropenta, also known as pentaerythritol tetranitrate.

Therefore, two different wavelengths were necessary for detection to enable the analysis of the explosives that were present in the standard. As the Agilent 1290 Infinity II Variable Wavelength Detector (VWD) is the most sensitive and fastest detector in its class, it is possible to acquire signals with two wavelengths in the dual-wavelength mode. Using time-programmable wavelength switching, high sensitivity, and selectivity is provided. Low detector noise (< ± 1.5 µAU) and baseline drift ($< 1 \times 10^{-4}$ AU/h) facilitates precise quantification, even of trace-level components.

This Application Note presents a comparison of the 1290 Infinity II VWD in dual-wavelength mode with the Agilent 1290 Infinity II Diode Array Detector (DAD). Resolution, sensitivity with limits of detection (LOD) and quantification (LOQ), and linearity are compared.

Experimental

The Agilent 1290 Infinity II LC System used for the experiments consisted of the following modules:

- Agilent 1290 Infinity II High Speed Pump (G7120A)
- Agilent 1290 Infinity II Multisampler (G7167B)
- Agilent 1290 Infinity II Multicolumn Thermostat (G7116B)
- Agilent 1290 Infinity II Diode Array Detector (G7117B), equipped with a 10-mm Max-Light cartridge cell
- Agilent 1290 Infinity II Variable Wavelength Detector (G7114B), equipped with a standard 10-mm flow cell

Column

Agilent Poroshell 120 SB C18, 3 × 150 mm, 2.7 µm (p/n 693975-302)

Software

Agilent OpenLAB CDS ChemStation Edition for LC & LC/MS systems, rev. C.01.07 [27]

Solvents and samples

Solvent A: Water

Solvent B: Methanol

Sample: Dr. Ehrenstorfer Nitroaromatic-Explosive Mix no. 3 (p/n 08330300), 10 ng/µL each

All solvents used were LC grade. Fresh ultrapure water was obtained from a Milli-Q Integral system equipped with a 0.22-µm membrane point-of-use cartridge (Millipak). Dr. Ehrenstorfer Nitroaromatic-Explosive Mix was purchased from LGC standards, Teddington, UK.

Chromatographic conditions

Parameter	Value
Flow rate	0.5 mL/min
Gradient	0 minutes, 20 % B 10 minutes, 30 % B 25 minutes, 38 % B 30 minutes, 90 % B
Stop time	35 minutes
Post time	10 minutes
Injection volume	1 and 2 µL
Column temperature	50 °C
DAD	Signal A) 254 nm, ref. off Signal B) 214 nm, ref. off Peak width > 0.1 minutes (2 seconds response time) (2.5 Hz)
VWD peak width	Signal A) 254 nm Signal B) 214 nm Dual-wavelength signal peak width > 0.2 minutes (4 seconds response time) (2.5 Hz)

Results and Discussion

To analyze the explosive standard containing 20 different explosives, two wavelengths are necessary: 254 nm (for highest selectivity), and 214 nm (Figure 2). A wavelength of 214 nm is needed to detect nitropenta (peak 20), which is not detected using 254 nm.

The dual-wavelength mode of the 1290 Infinity II VWD enables the detection of two wavelengths. Figure 3 shows the overlay of the 254 nm signals, collected with the 1290 Infinity II DAD (blue signal) and the 1290 Infinity II VWD (red). Both chromatograms are comparable with respect to sensitivity and resolution. This is also confirmed with the calculation of LOD and LOQ, (Figure 4, LOD data not shown).

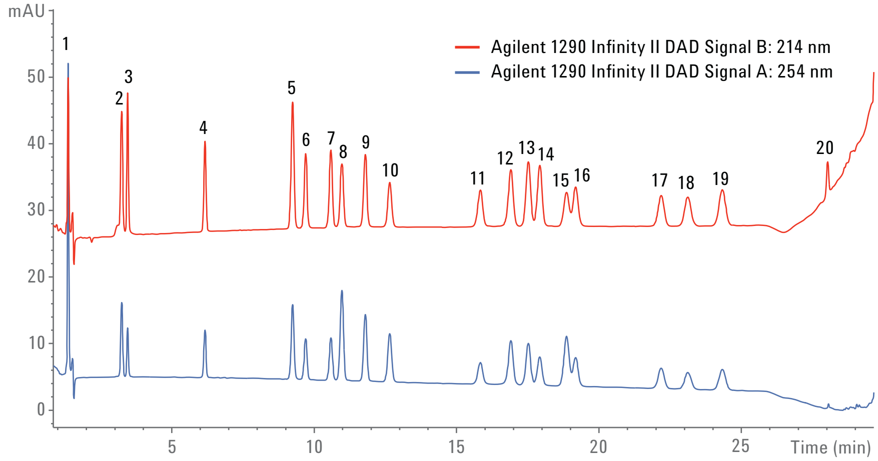

Figure 2. Detection of 20 explosives using 254 and 214 nm. Nitropenta (peak 20) is only detectable with 214 nm.

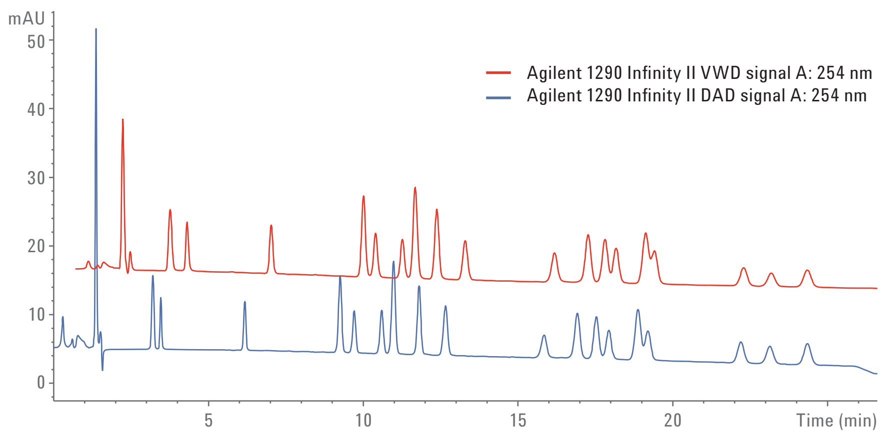

Figure 3. Agilent 1290 Infinity II DAD and Agilent 1290 Infinity II VWD signals at 254 nm. Similar sensitivity and resolution is observed.

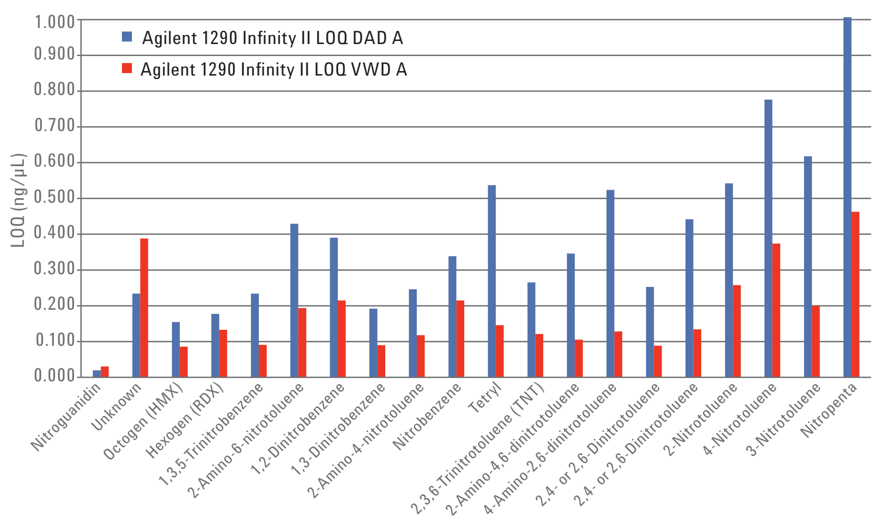

Figure 4 Comparison of limits of quantification of the Agilent 1290 Infinity II DAD and Agilent 1290 Infinity II VWD signal A with 254 nm. For most of the analytes, the 1290 Infinity II VWD showed more than double the sensitivity compared to the 1290 Infinity II DAD.

The comparison of LOD (data not shown) and LOQ showed higher sensitivity for all peaks (except for the first two) with the 1290 Infinity II VWD. For most of the peaks, the VWD showed more than twice the sensitivity. In addition, the linearity was also calculated using a 1:2 dilution series of the explosive standard from 10 ng/µL each to 0.625 ng/µL. Table 1 shows the correlation coefficients for this concentration range. The linearity was absolutely comparable between DAD and VWD with correlation coefficients over 0.9999 for most of the analytes.

Conclusion

This Application Note compared the resolution, sensitivity, and linearity of the Agilent 1290 Infinity II Diode Array Detector and the Agilent 1290 Infinity II Variable Wavelength Detector for the analysis of 20 nitroaromatic explosives. Since the explosive mix contained nitroaromatics and nitramines, two different wavelengths of 254 and 214 nm were necessary for analysis. The analysis with the VWD was facilitated by the dual-wavelength mode of the 1290 Infinity II Variable Wavelength Detector. Both detectors showed absolutely comparable resolution and sensitivity. The 1290 Infinity II Variable Wavelength Detector showed even better sensitivity with LODs and LOQs, two-times better than the 1290 Infinity II Diode Array Detector for the majority of the analytes. In addition, high linearity was observed with correlation coefficients over 0.9999 for most of the analytes for both detectors.

Table 1. Correlation coefficients for a range from 10 ng/µL to 0.625 ng/µL per analyte for the Agilent 1290 Infinity II DAD and Agilent 1290 Infinity II VWD.

Peak	Substance	Correlation coefficients DAD	Correlation coefficients VWD
1	Nitroguanidin	0.97274	0.99894
2	Unknown	0.9846	0.99835
3	Octogen (HMX)	0.99795	0.99894
4	Hexogen (RDX)	0.99999	0.99998
5	1,3,5-Trinitrobenzene	0.99999	0.99998
6	2-Amino-6-nitrotoluene	0.99998	0.99995
7	1,2-Dinitrobenzene	0.99999	0.99999
8	1,3-Dinitrobenzene	1	0.99999
9	2-Amino-4-nitrotoluene	0.99999	0.99999
10	Nitrobenzene	0.99997	0.99984
11	Tetryl	0.99974	0.99943
12	2,3,6-Trinitrotoluene (TNT)	0.99996	0.99998
13	2-Amino-4,6-dinitrotoluene	0.99999	0.99999
14	4-Amino-2,6-dinitrotoluene	0.99994	0.99999
15	2,4- or 2.6-Dinitrotoluene	0.99999	0.99999
16	2,4- or 2.6-Dinitrotoluene	0.99991	1
17	2-Nitrotoluene	0.99998	0.99997
18	4-Nitrotoluene	0.99995	1
19	3-Nitrotoluene	0.99997	0.99996
20	Nitropenta	0.99919	0.99767

References

1. US Environmental Protection Agency, EPA Method 8330B-B-29, revision 2, "Nitroaromatics and Nitramines by HPLC", October **2006**.

2. Kinghorn, R; Milner, C; Zweigenbaum, J. Analysis of Trace Residues of Explosive Materials by Time-of-Flight LC/MS, *Agilent Technologies Application Note*, publication number 5989-2449EN, **2005**.

3. Huesgen, A. G. Seamless instrument-to-instrument method transfer of the EPA method 8330A/B for nitroaromatics from an Agilent 1200 Series LC to the Agilent 1290 Infinity Binary LC using ISET, *Agilent Technologies Application Note*, publication number 5991-1194EN, **2012**.

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