Using the Agilent 1290 Infinity Series LC Method Development System and Agilent MassHunter Walkup Software for Multiple-Method Analysis of Environmental Samples

Application Note

Environmental

Author

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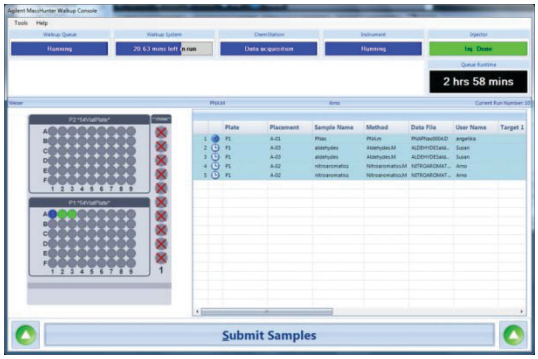
Abstract

The Agilent 1290 Infinity Series LC Method Development Solution in combination with the Agilent MassHunter Walkup Software provides easy and convenient access to an LC system for different users with different applications. For the analysis of the submitted samples, predefined methods including appropriate column, mobile phases, and chromatographic methods are available. If needed, the information about the results are send remotely by, for example, e-mail after completion of the run. The super-users take care of the LC and/or LC/MS system, providing the chromatographic methods, keeping the LC and LC/MS system running, and, in case of errors, taking appropriate action.



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Introduction

Frequently, several different applications have to be run on the same system in a laboratory using different columns, different mobile phases, different column temperatures, and different chromatographic parameters. Changing the column or changing mobile phases is time consuming, and the risk of bad column connections increases with the number of installations. Change of the mobile phase is often required due to different pH requirements or the need for a different organic mobile phase. The Agilent 1290 Infinity Series LC Method Development Solution[1] in combination with the Agilent MassHunter Walkup[2,3] offers a convenient solution.

Column and mobile phases are included in the chromatographic method by the appropriate valve position and other parameters such as flow rate, gradient percentage, and so forth. The super-user defines the chromatographic methods for all needed applications and the user selects the appropriate method for his samples. After submitting his samples, the analyst starts using the correct column and mobile phases. The results are stored and sent to the user by, for example, email.

The super-user establishes the definition of user profiles, pass words, and other system relevant parameters. The single user or a user group can have different access levels, reflecting their chromatographic expertise.

In the following, we show different tasks of an environmental laboratory, analysis of nitro aromatics, analysis of aldehydes, and analysis poly-nuclear aromatics (PNAs). Different users submitted their samples selecting different chromatographic methods.

Experimental

Table 1. Instrumentation used.

Module	Product number
Quaternary pump	G4204A
Auto sampler	G4226A
ALS cooler	G1330B
Column compartment 1 equipped with high pressure column switching valve + valve drive option # 58	G1316C
Column compartment 2 equipped with low pressure column switching valve + valve drive option # 58	G1316C
Solvent selection valve for the aqueous phases	G1160A
Solvent selection valve for the organic phases	G1170A
Diode array detector, with 10-mm path length cell	G4212A
Method development valve kit (1,200 bar)	G4230B
Low dispersion capillary kit	5067-4646

Chromatographic conditions for nitroaromatics

Sample	Dr. Ehrenstorfer Nitroaromatic-Explosive Mix no. 3 (p/n 08330300), 10 ng/µL each,
Column	Agilent Poroshell 120 C18, 4.6 × 150 mm, 2.7 µm (p/n 683975-902)
Mobile phase	A = Water, B = Methanol
Gradient	0 minutes 20 % B, at 10 minutes 30 % B, at 30 minutes 95 % B, at 31 minutes 95 % B
Flow rate	0.8 mL/min
Stop time	40 minutes
Post time	5 minutes
Injection volume	3 µL of undiluted sample was injected
Column temperature	35 °C
DAD	254/214, 235/10 nm Ref 400/80 nm Flow cell: 10 mm Peak width: < 0.013 minutes (20 Hz)

Chromatographic conditions for aldehydes

Sample	Sigma-Aldrich (Catalog No. 47651-U) diluted in acetonitrile. In the mixture, each analyte had a concentration of 30 µg/mL.
Column	Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 150 mm, 1.8 µm (p/n 959759-902)
Mobile Phase	A: Water, B: Acetone
Flow rate	0.25 mL/min
Gradient	at 0 minutes 45 %, at 12 minutes 53 % B, at 28 minutes 67 % B, at 32 minutes 67 % B, at 33 minutes 95 % B
Stop time	34 minutes
Post time	20 minutes
Injection volume	2.1 µL
Temperature	45 °C
Detection	360 nm/10 nm Ref: off Peak width > 0.025 minutes (0.5 second response time, 10 Hz)

Chromatographic conditions for PNAs

Sample	SS EPA 610 PAH Mix in Methanol/Methylene Chloride (1:1), (Supelco Analytical)
Column	Agilent ZORBAX Eclipse PAH, 4.6 × 150 mm, 5 µm (p/n 959993-918)
Solvents	A: Water, B: Acetonitrile
Gradient	at 0 minutes 40 % B, at 20 minutes 95 % B
Flow rate	1.5 mL/min
Stop time	30 minutes
Post time	5 minutes
Injection volume	3 µL
DAD	230 nm, reference 400 nm

Analyzed compounds

Nitroaromatics

1. Nitroguanidin
2. Octogen (HNX)
3. Hexogen (RDX)
4. 1,3,5 Trinitrobenzene
5. 2-Amino-6-Nitrotoluene
6. 1,2-Dinitrobenzene
7. 1,3-Dinitrobenzene
8. 2-Amino-4-Nitrotoluene
9. Nitrobenzene
10. Tetyl
11. 2,4,6-dinitrotoluene
12. 4-Amino-2,6-dinitrotoluene
13. 2,6-Dinitrotoluene
14. 2-Nitrotoluene
15. 4-Nitrotoluene
16. 3-Nitrotoluene
17. Nitropenta

PNAs

1. Napthalene
2. Acenaphtylene
3. Acenaphtene
4. Fluorene
5. Phenanntrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fuoranthene
13. Benzo(a)pyrene
14. Dibenzo(a,h)anthracene
15. Benzo(g,h,i)perylene
16. Indeno(1,2,3-cd)pyrene

Aldehydes

1. Formaldehyde-2,4-dinitrophenylhydrazone
2. Acetaldehyde-2,4-dinitrophenylhydrazone
3. Acrolein-2,4-dinitrophenylhydrazone
4. Acerone-2,4-dinitrophenylhydrazone
5. Propionaldehyde-2,4-dinitrophenylhydrazone
6. Crotonaldehyde-2,4-dinitrophenylhydrazone
7. Methacrolein-2,4-dinitrophenylhydrazone
8. 2-Butanone-2,4-dinitrophenylhydrazone
9. Butyraldehyde-2,4-dinitrophenylhydrazone
10. Benzaldehyde-2,4-dinitrophenylhydrazone
11. Valeraldehyde-2,4-dinitrophenylhydrazone
12. m-Tolualdehyde-2,4-dinitrophenylhydrazone
13. Hexaldehyde-2,4-dinitrophenylhydrazone

Acquisition and Evaluation Software

OpenLAB CDS ChemStation version C.01.05 and MassHunter Walkup software C.01.00

Results and Discussion

The 1290 Infinity LC Series Method Development Solution can be used with up to eight columns of different length and internal diameter. Having installed two solvent selection valves and a 1290 Infinity Quaternary Pump, 28 different solvents are available. In our example, we used three different columns and four different mobile phases for the analysis of nitro-aromatics[4], aldehydes[5], and PNAs[6]. The super-user had set up three different methods for the three applications, see Figure 1. This is done in the administration screen, which is typically accessible only by the super-user.

Having defined the appropriate method in the administration tool, the methods are accessible for submitting samples. Typically, three steps are needed to submit samples, see Figures 2, 3, and 4. Figure 2 shows the first step. The user has to fill in name, password, vial type, and number of samples.

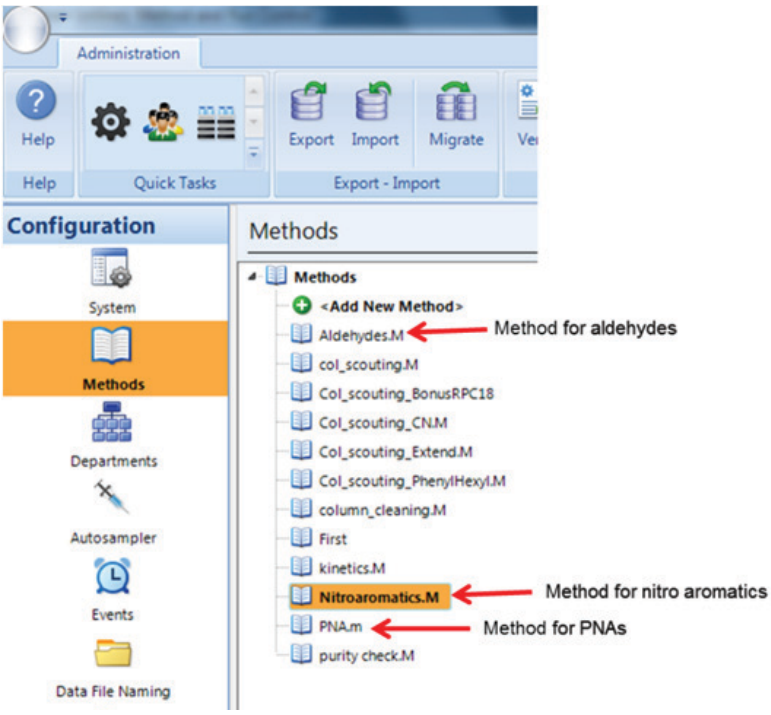

Figure 1. Method definition by the super user in the walk up administration screen.

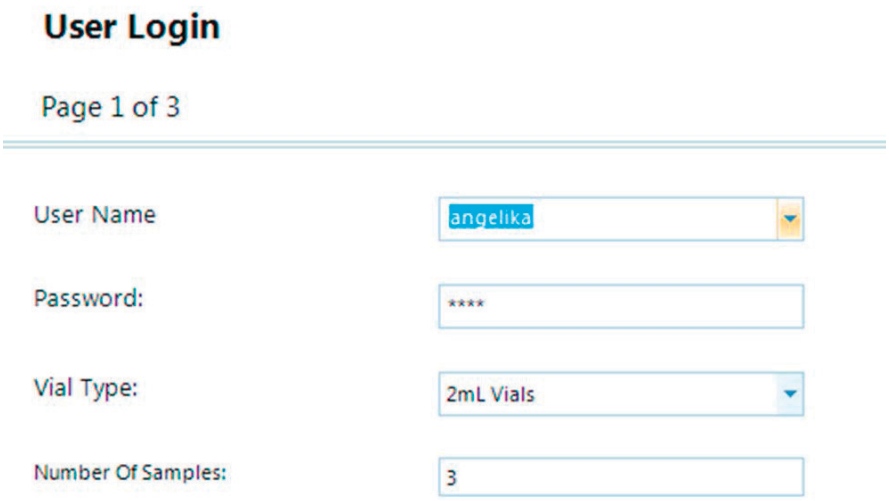

Figure 2. Start screen for submitting samples.

Figure 3 shows what additional sample and method information has to be filled in.

In the third step, the sample vial has to be put into the indicated auto sampler position, see Figure 4.

When step 3 is completed, the sample submission is finished and the analysis starts. Typically, the super-user has set up a pre-run method and equilibration methods. An individual equilibration and post cleaning method has to be set up for each method with different column and mobile phase combinations. A blank run before the analysis of standards and real life samples is required, otherwise, the column is not sufficiently conditioned.

In our example, three samples were provided by one user, see Figure 5.

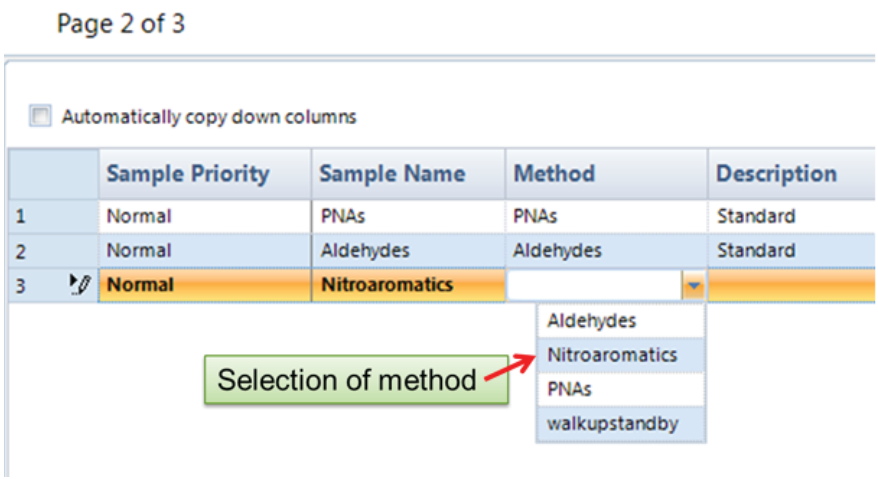

Figure 3. Submitting samples for the analysis of nitroaromatics using the appropriate method.

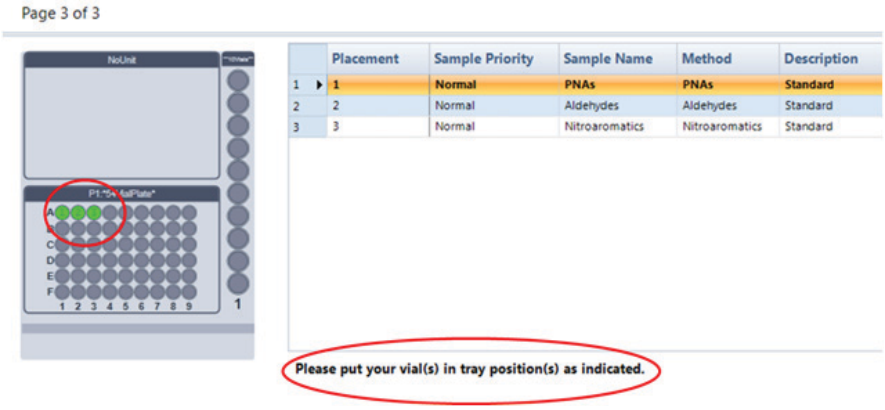

Figure 4. Final step during sample submission.

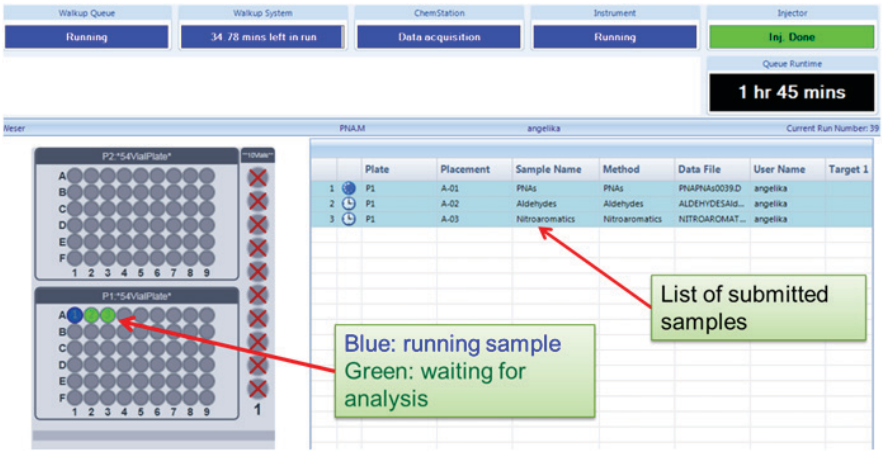

Figure 5. One user provides samples for analysis.

The data were stored in different folders according to the user group, see Figure 6. Typically, the users get informed about the results remotely through email or other tools, and can then evaluate the data in detail. Figure 6 shows the chromatograms of the nitroaromatics analysis.

The software sends an area percent report to the user if not otherwise defined. Figure 7 gives an example. If calibrated reports are needed, a calibration run has to be performed by the super-user and stored with the appropriate walk-up method.

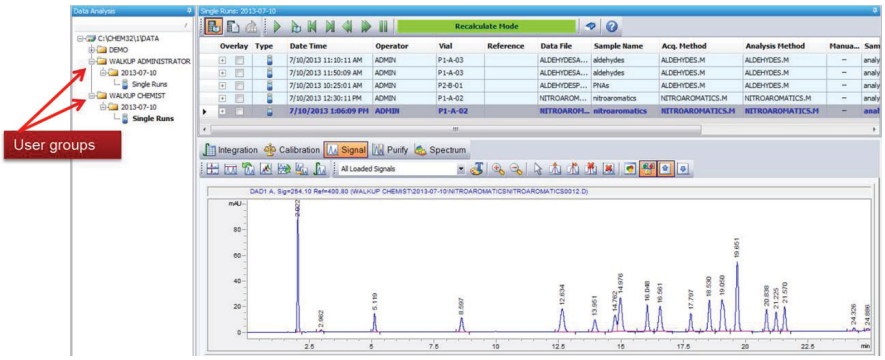

Figure 6. Data storage of nitroaromatics analysis accordingly to the user group.

Acq. Operator : ADMIN

Sample Operator : angelika

Acq. Instrument : Weser Location : P1-A-04

Injection Date : 7/10/2013 14:06:04

Inj Volume : 3.000 µL

Acq. Method : C:\CHEM32\1\METHODS\PNA.M

Area Percent Report

Sorted By : Signal
Calib. Data Modified : Monday, July 01, 201314:18:55
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
Signal 1: DAD1 A, Sig=230,10 Ref=400,60

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	3.445	VB	0.4236	22.90172	1.1204	?
2	7.349	VB	0.0868	68.71390	3.3616	?
3	8.200	BB	0.0961	255.53972	12.5013	?
4	9.543	VV	0.1028	291.95688	14.2829	?
5	9.825	VB	0.1119	26.40264	1.2917	?

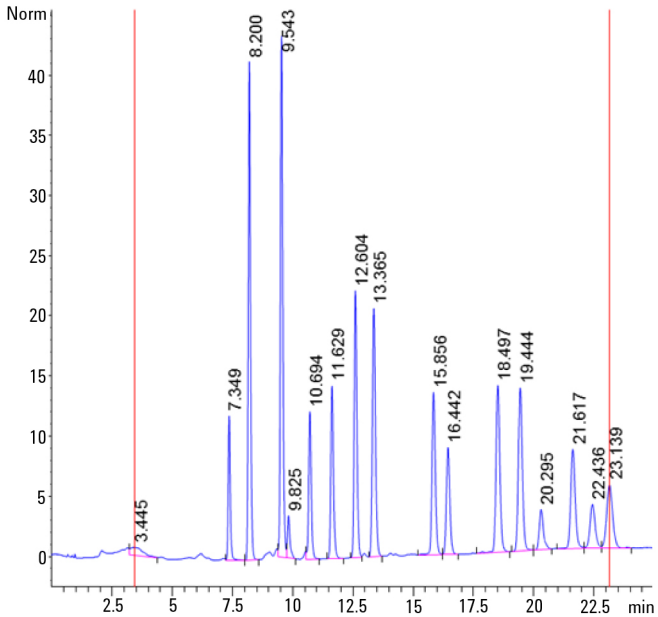

Figure 7. Area % report of the analysis of PNAs.

Figure 8 shows an overlay of all created chromatograms. These applications are high-resolution methods using UV detection. It is also possible to add a mass spectrometer for highly selective analysis or to set up fast screening methods for high sample throughput. The analysis of the nitroaromatics required two wavelengths to be able to detect Compound 17.

Conclusions

The combination of the Agilent 1290 Infinity Series LC Method Development Solution and the Agilent MassHunter Walkup Software enables users or user groups to access an UHPLC system for multimethod applications. All method parameters such as columns, mobile phases, gradients, and all further chromatographic conditions are combined in one method and are available without the need to change hardware, for example, columns. Up to eight columns and up to 28 mobile phases are possible with the system setup described in this Application Note. The super-user creates all needed methods, which are then accessible by the users. Submitting samples is done in three steps and includes selecting the appropriate method and putting the sample(s) into the indicated vial position. The reports can be obtained remotely for example, through email.

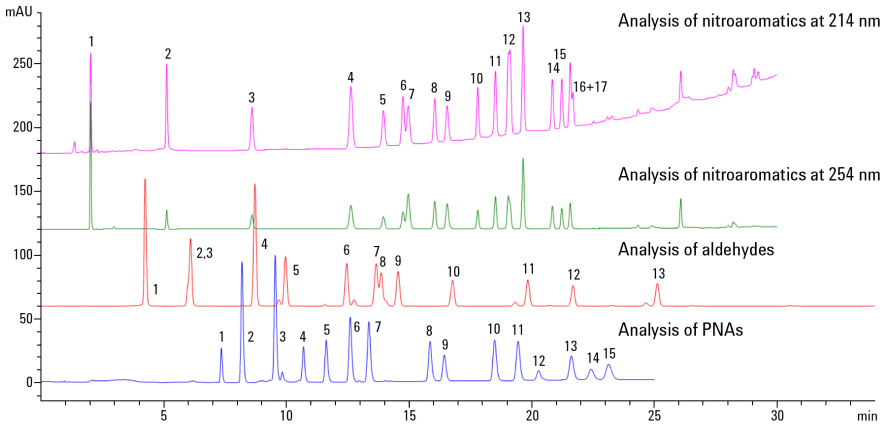

Figure 8. Overlay of created chromatograms of PNAs, aldehydes, and nitroaromatics.

References

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