SampleQ

Automated In-vial Extraction of Surface Waters for the Analysis of PAH and OCP by GC-MS/MS.

Keywords: GC-MS(/MS), sample preparation, automation, PAH, OCP, water, environment Authors: Mathias van Lancker, Joeri Vercammen



Introduction

Environmental contamination has been a trending topic for years as it raises public concern. The quality of surface water is one of the key aspects that needs to be monitored and laboratories constantly require methods to analyse contaminants in a fast and costeffective way. These laboratories desire solutions that reduce solvent disposal and optimize the sample preparation without compromising the quality of the analytical results. This application note focuses on the development and validation of an automated in-vial

This application note focuses on the development and validation of an automated in-vial extraction method for the analysis of low-level polycyclic aromatic hydrocarbons (PAHs) and organochlorine pesticides (OCPs) in surface waters followed by subsequent GC-MS/MS analysis. For which SampleQ has developed a new automated in-vial extraction method taking into account the following parameters as method performance qualification:









Reproducibility and Sensitivity				
The instrument reproducibility				
is calculated as relative				
standard deviation of the				
component amount.				
Measurements are repeated 7				
times on day 1 and 7 times on				
day 2 by using calibration				
standard 2 (5 ng/L). Sensitivity				
is calculated using the following				
formula:				

Component	Avg (ng/L)	SD (ng/L)	RSD (%)	MDL (ng/L)
Naphthalene	4,68	0,87	14,4	1,93
Alpha-BHC	4,54	0,22	4,9	0,59
Beta-BHC	4,59	0,07	1,6	0,2
Heptachlor	5,1	0,07	1,5	0,2
Aldrin	4,5	0,22	4,9	0,58
Heptachlor Epoxide	4,5	0,09	2	0,24
Endosulfan 1	4,19	0,09	2,1	0,23
Dieldrin	4,08	0,15	3,7	0,4
Endrin	6,01	0,89	14,9	2,37
Endosulfan 2	4,34	0,2	4,7	0,54
4,4'-DDT	4,95	0,21	4,1	0,54
Benzo(a)pyrene	4,24	0,44	10,3	1,15
Benzo(ghi)perylene	4,61	0,37	8,1	0,99

With:

- $MDL = t_{0.99} \times SD$
- MDL = Method Detection Limit
- $t_{0.99}$ = t-distribution with a 99% confidence level and n-1 degrees of freedom = 2,650
- SD = Standard deviation on the amount (ng/L)

Component	R2	Linearity		
Naphthalene	0,9974	In order to check the linearity of the GC method, a series of 6 calibration standards were prepared, with all components of interest. Calibration curves were automatically generated using the Chromeleon software. All calibration curves were based on the average of two injections for each calibration point. The results show a coefficient of determination (R2) of at least 0,9927 with a linear fit for all components.		
Alpha-BHC	0,9986			
Beta-BHC	0,9986			
Heptachlor	0,9998			
Aldrin	0,9999			
Heptachlor Epoxide	0,9999			
Endosulfan I	0,9987			
Dieldrin	0,9996			
Endrin	0,9992			
Endosulfan II	0,9969	Linearity reproducibility and consitivity results were		
4,4'-DDT	0,9996	Linearity, reproducionity and sensitivity results were in		
Benzo(a)pyrene	0,9988	application note #10591. ¹		
Benzo(ghi)perylene	0,9927			

Chromatography

The following chromatograms show typical results of a 2,5 ng/L standard for naphthalene, alpha-BHC and Beta-BHC.







References

 Bijsterbosch C, Wissel C, Cojocariu C, Dobbeleer I. Application Note 10591: "Automated sample preparation followed by sensitive analysis by GC-MS/MS for environmental contaminants in surface waters". 2018. https://assets.thermofisher.com/TFS-Assets/CMD/Application-Notes/an-10591-gcms-ms-enviro-contaminants-water-an10591-en.pdf.



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