Separations
Analytical Instruments b.v.

APPLICATION INFO

Fully automated sample preparation of OCP's and PCB's in soil samples.

2010

Introduction

OCP-PCB (Organ-Chlorine-Pesticides - Poly-Chlorine-Biphenyl's) are a group of chemically related compounds which are of interest to toxicologists because of their unknown or potential health properties. PCBs were widely used in commerce for almost fifty years but were removed from the market in 1979 by the Environmental Protection Agency (EPA) primarily because of their capacity to accumulate in the environment and migrate through the food chain. Because they not only accumulate but persist in the environment, they can often be found in air, water, soil and food until today. PCBs can also be found in the human body, primarily in fatty tissues. PCBs are still used in some electrical equipment, such as transformers and capacitors, but this use is also being phased out

Experimental

For the chromatographic separation a GC-MS is most used as detection system. The automated solid phase extracton (SPE) is performed by a Spark Holland **Symbiosis™** workstation which can control a **Reliance** autosampler when using volumes of 10mL. When the volumes are between 10-100 mL, the samples can be connected to the SSM valves (Sample Selection Manifolds), but the capacity of the system is lower.
Also is this not necessary for the OCP-PCB analysis of a equal kind of analysis like PAH.
The soil samples (extracted in aceton and diluted with water) are introduced by the HPD (high pressure dispenser) of the Symbiosis system on the SPE cartridge,the switching diagram shows how the samples are prepared prior to ON-line elution.
The HPD which is pumping 100% of injection solvent for the GC-MS, is connected to the cartridge ensuring a quick desorption. Afterwards it is led to **Reliance** so that the extract can be transferred to the GC-MS or other system of choice.
With the **Symbiosis™** workstation, a flexibel system is created for easy use of SPE wit high capacity (up to 72 sample per series) and low organic solvent use (about 70mL per sample for total extraction method).
In a Laboratory an analyst is capable to do 10-16 samples a day. With the **Symbiosis™** workstation the capacity increases to 72.
Also the Reproducibility will improve, less manual handling and better drying and eluting method of the **Symbiosis™** workstation.

Chromatographic conditions

Materials

SPE cartridge	PLRPS, L= 10 mm I.D.=2mm
Sample trays	1 x 2mL (1x96= 96 pos.) 6 x 10mL (6x12= 72 pos.)
Extraction solvent	Choice of Customer

Autosampler conditions

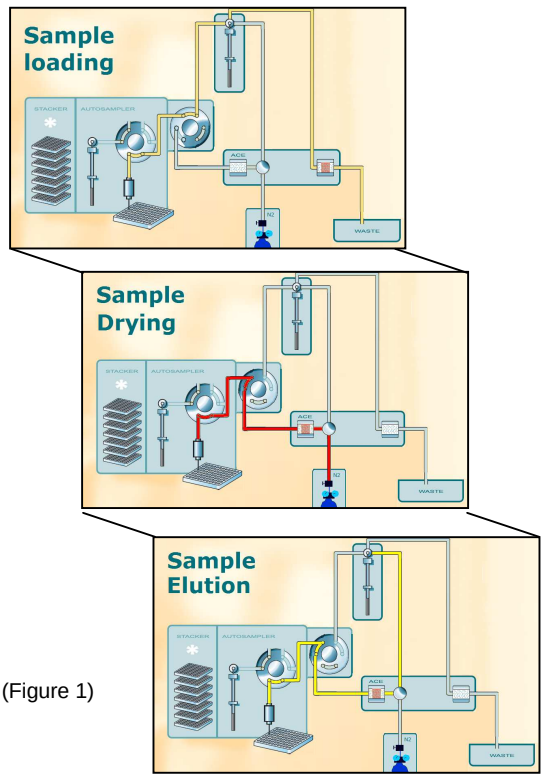
Reliance

Needle	45 µL
Wash solvent	GC-injection solvent

SPE conditions

SPE cartridge	PLRPS	
Solvation	3 mL Acetonitril 3 mL Methanol	6 mL/min.
Equilibration	3 mL Water	4 mL/min.
Sample load	10 mL	2 mL/min.
Washing	1 mL Water	2 mL/min.
Elution	50-2000uL extraction solvent	

System scheme



(Figure 1)

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Sample Handling

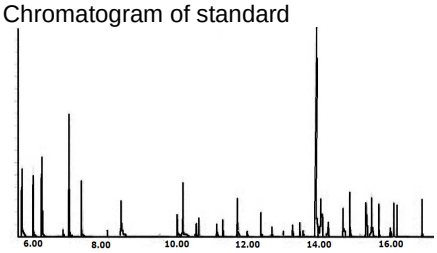
- Use amber borosilicate glassware
- Take 20 gram soil in 250mL flask
- Add 50 mL Acetone
- Take 4mL sample in a 10mLvial
- Ad 6mL of water to the vial
- The sample vial and also the 2mL collection vial (with or without insert) are placed in the **Reliance** autosampler and the system can be started.

The 50mL Acetone extract can also be used for PAH, Mineral Oil and EOX analysis. By combining the analysis the solvent use can be minimized to a minimum.

The procedure is Time saving (from Aceton extract to extract for analysis = 16min.) and also solvent saving.

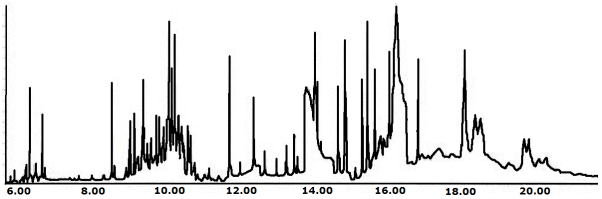
Results

Chromatogram of standard



(figure 2)

Chromatogram of real sludge sample



(figure 3)

Validation results

See validation results in figure 4 and 5

Conclusion

With the **Symbiosis™** workstation the sample preparation of Soil samples for OCP-PCB can be upgraded to a higher level, faster, with lower cost and less sample handling and solvent use. Also the influence of sample and manual labor is reduced, which results in better Performance and Reproducibility.

In this Application Info the **Symbiosis™** workstation is used for OCP-PCB, but the Workstion can also be used for the analysis of PAH's, Pesticides, residues of Medicines in Soil and Water.

Reference

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Figure 4 Validation results in Soil/Sludge during customer testing .

Recovery testing Sludge sample							
Components	**standard**	**Sample 1**	**Recovery**	**Sample 2**	**Recovery**	**Average**	**Criteria**
Hexachloorbutadieen	16130	13213	82%	14350	89%	85%	75<Tv<110
Pentachloorbenzeen	25708	22545	88%	24789	96%	92%	75<Tv<110
α-HCH	7615	5853	77%	6593	87%	82%	75<Tv<110
HCB	27007	20484	76%	22597	84%	80%	60<Tv<110
β-HCH	5360	5392	101%	5876	110%	105%	75<Tv<110
γ-HCH	5414	4172	77%	4573	84%	81%	75<Tv<110
δ-HCH	4677	1162	25%	1061	23%	24%	75<Tv<110
PCB-28	24176	23852	99%	25450	105%	102%	75<Tv<110
Heptachloor	2883	2756	96%	3032	105%	100%	75<Tv<110
PCB-52	15435	12997	84%	13820	90%	87%	75<Tv<110
Aldrin	6800	5451	80%	6152	90%	85%	75<Tv<110
Telodrin	3923	3557	91%	3903	99%	95%	75<Tv<110
Isodrin	5192	4085	79%	4531	87%	83%	75<Tv<110
Heptachloorepoxide	7269	6347	87%	6716	92%	90%	75<Tv<110
o,p-DDE	31119	25389	82%	26855	86%	84%	75<Tv<110
PCB-101	20410	15929	78%	17446	85%	82%	75<Tv<110
α-Endosulfan	2407	2110	88%	2302	96%	92%	60<Tv<110
Dieldrin	3331	3037	91%	3309	99%	95%	75<Tv<110
p,p-DDE	19347	18301	95%	19048	98%	97%	75<Tv<110
o,p-DDD	33430	31640	95%	32155	96%	95%	75<Tv<110
Endrin	2072	2226	107%	2270	110%	108%	75<Tv<110
β-Endosulfan	2334	2207	95%	2251	96%	96%	-
PCB-118	25472	20517	81%	24338	96%	88%	75<Tv<110
p,p-DDD	32579	35399	109%	34689	106%	108%	75<Tv<110
o,p-DDT	5240	5736	109%	5305	101%	105%	75<Tv<110
PCB-153	23259	17500	75%	19793	85%	80%	75<Tv<110
p,p-DDT	1605	1390	87%	1388	86%	87%	75<Tv<110
PCB-138	21918	18283	83%	20213	92%	88%	75<Tv<110
PCB-180	22002	17973	82%	20115	91%	87%	75<Tv<110

Figure 5 Validation results **Symbiosis™** workstation and GC-MS .

Component	Detection limit (mg/kg.ds)		Reproducibility (%)	Recovery (%)
	Level (2 µg/kg.ds)	Level (20 µg/kg.ds)	Level (0,0651mg/kg.ds)	Level (0,0651mg/kg.ds)
Hexachloorbutadieen	0.6	-	8.4	85
Pentachloorbenzeen	0.9	-	12.0	92
α- HCH	1.4	-	12.8	104
HCB	1.1	-	11.7	87
β-HCH	1.6	-	15.0	101
γ- HCH	1.6	-	15.6	99
δ- HCH	1.7	-	10.7	107
ε- HCH	2.2	-	10.7	103
PCB-28	1.7	-	12.7	99
Heptachloor	1.9	-	16.6	88
PCB-52	0.7	-	11.7	93
Aldrin	-	7.1	10.9	78
Telodrin	-	7.0	11.8	97
Isodrin	-	10.2	20.0	76
Heptachloorepoxide	1.3	-	12.8	100
γ-Chloordaan	1.9	-	13.4	94
o,p-DDE	0.9	-	9.0	94
PCB-101	2.4	-	13.7	86
α-Endosulfan	-	15.0	10.6	104
α-Chloordaan	1.4	-	14.7	94
Dieldrin	-	5.9	11.3	89
p,p-DDE	2.5	-	11.9	98
o,p-DDD	1.1	-	11.9	98
Endrin	-	7.5	15.0	102
β-Endosulfan	-	7.2	10.0	103
PCB-118	1.2	-	10.4	84
p,p-DDD	1.3	-	11.4	99
o,p-DDT	1.8	-	32.1	63
PCB-153	1.2	-	13.1	85
Endosulfan sulfaat	-	6.9	14.1	101
p,p-DDT	-	11.2	25.4	76
PCB-138	1.6	-	12.5	86
PCB-180	1.6	-	10.4	85