Organochlorine pesticides in eel

Application Note

Environmental

Authors

Agilent Technologies, Inc.

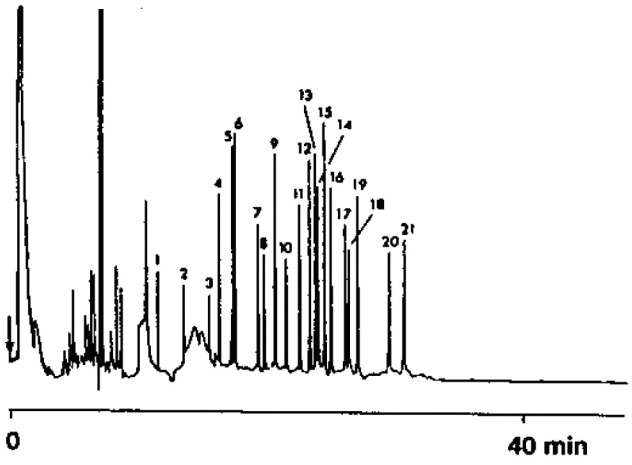
Introduction

A good separation of 21 organochlorine pesticides has been accomplished on a fused silica Agilent CP-Sil 19 CB column. The use of this phase, which differentiates between oxychlordane and heptachlor epoxide, can be recommended for residue analysis together with, or instead of, the less polar phases CP-Sil 5, 7 and 8.

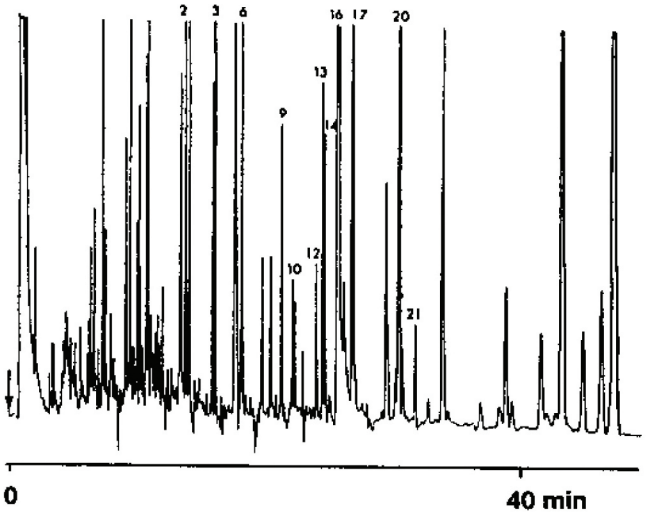
Agilent Technologies

Conditions

Technique	: GC-capillary
Column	: Agilent CP-Sil 19 CB, 0.22 mm x 16 m fused silica WCOT CP-Sil 19 CB (0.2 µm) (Custom made*)
Temperature	: 80 °C (3 min) → 150 °C, 35 °C/min (2 min) 150 °C → 216 °C, 3 °C/min
Carrier Gas	: He
Injector Mode	: Grob; the splitter was opened 0.5 min after injection (1 µL)
Detector	: ECD
Courtesy	: Mia A.T. Kerkhoff and Piet F. Otte from the Netherlands Institute for Fishery Investigations (PO Box 68, 1970 AB Ijmuiden, The Netherlands)



Chromatogram 1. First Fraction



Chromatogram 2. Second Fraction (Hollandse IJssell, Gouderak)

In the past, the packed column analysis of PCBs and organochlorine pesticides were usually performed on two or more different stationary phases. Nowadays more and more residue analyses occur on capillary columns. Just one column is often used with a nonpolar coating like CP-Sil 5 (SE-30) or CP-Sil 7 and 8 (SE-52, SE-54) (1).

Although these high efficiency columns provide excellent separations, one single stationary phase is not capable of separating a number of organochlorine compounds.

During a study in marine mammals from the North Sea, a very poor separation of oxychlordane and heptachlor epoxide on CP-Sil 7 was observed (1 ,2).

Since both compounds occur in the environment a better phase had to be selected for their detection. The complete separation of these compounds on a packed OV-17 column has been reported (4) and for that reason the suitability of a fused silica column coated with CP-Sil 19 CB was tested (5).

Sample preparation

Most compounds were purchased from commercial suppliers. The chlordane compounds were kindly supplied by the US Environmental Protection Agency (EPA). The samples were (Soxhlet) extracted with n-pentane, and the extracts were cleaned up on alumina and separated into two fractions on silica (1).

Results

Several tests with different temperature programs were performed on a fused silica CP-Sil 19 CB column. This resulted in a program in which an almost complete separation of 21 organochlorine pesticides has been achieved.

See Chromatogram 1. The analyzed compounds with their retention times and their relative retention times to dieldrin are given in Table 1.

Twenty-five extracts of fish from the North Sea and Dutch inland waters have been successfully analyzed on the same columns without any decline of the separation efficiency.

Chromatogram 2 illustrates the presence of oxychlordane and heptachlor epoxide in eels, which have been sampled in the Hollandse IJssel near a waste dumping site (Gouderak). This sample is characterized by extremely high residues of dieldrin (1.1 mg/kg) and endrin (0.18 mg/kg). In addition, the levels of cis-(0.019 mg/kg) and trans-chlordane (0.010 mg/kg), transnonachlor (0.022 mg/kg), and oxychlordane (0.024mg/kg) are higher than those determined in other Dutch inland waters.

Table 1. Organochlorine compounds analyzed on the WCOT CP-Sil 19 CB column.

	Compound	Retention time (min)	Relative retention time to dieldrin	Concentration (µg/mL)
1	HCB	10.91	0.44	0.004
2	a-HCH	12.95	0.52	0.004
3	y-HCH	14.93	0.60	0.004
4	heptachlor	15.77	0.64	0.010
5	o-chlordene	16.86	0.68	0.020
6	aldrin	17.34	0.70	0.020
7	y-chlordene	18.89	0.76	0.020
8	β-HCH	19.39	0.78	0.020
9	oxychlordane	20.26	0.82	0.020
10	heptachlor epoxide	21.17	0.85	0.010
11	o,p′-DDE	22.25	0.90	0.020
12	transchlordane	23.04	0.93	0.020
13	cischlordane	23.51	0.95	0.020
14	transnonachlor	23.72	0.96	0.020
15	p,p′-DDE	24.26	0.98	0.020
16	dieldrin	24.77	1.00	0.020
17	endrin	25.93	1.05	0.020
16	o,p′-DDD	26.27	1.06	0.020
19	o,p′-DDT	26.98	1.09	0.040
20	p,p′-DDD	29.47	1.19	0.020
21	p,p′-DDT	30.62	1.24	0.040

www.agilent.com/chem

Printed in the USA
31 October, 2011
Firt published prior to 11 May, 2010
A00055

Agilent Technologies