Analysis of Organochlorine and Pyrethroid Pesticides with Agilent 6820 Gas Chromatograph/Micro-Electron Capture Detector

Application

Environmental and Food Analysis

Author

Chuanhong Tu

Agilent Technologies Co., Ltd. (Shanghai)

412 YingLun Road

Waigaoqiao Free Trade Zone

Shanghai, 200131 P. R. C.

Abstract

The Agilent 6820 gas chromatograph (GC) with micro-electron capture detector (µECD) was used to analyze organochlorine and pyrethroid compounds. All compounds demonstrated good linearity among a wide concentration range. The sensitivity provided by µECD was much better than the requirements of the routine pesticide residue analysis.

Introduction

The electron capture detector (ECD) is a type of detector with high sensitivity and selectivity for halogenated compounds. However, there are some drawbacks in ECD design. The ECD is inherently nonlinear, with a limited linear range. Due to the narrow linear range, sample concentration or dilution, and re-analysis have to be employed, resulting in lower productivity. In addition, in traditional ECD design, a large flow cell is necessary to be compatible with both packed and capillary columns, leading to lower detector sensitivity [1].

To address these problems, the µECD, developed by Agilent Technologies, uses a smaller flow cell. The µECD is optimized for capillary columns and designed for improved sensitivity. It was successfully used with an Agilent 6890 series GC with better detector sensitivity and a wider linear range.

In this note, the Agilent 6820 GC with µECD was used to determine organochlorine and pyrethroid pesticides following the Chinese National Standard Method GB/T 5009.146-2003 [2]. System sensitivity and limits of detection (LOD) were examined for organochlorine and pyrethroid compounds.

Experimental

All experiments were performed on an Agilent 6820 GC with split/splitless inlet and µECD. Single-tapered deactivated liner (p/n 5183-4696) and Agilent green septa (p/n 5183-4759) were used. Cerity Networked Data System (NDS) software was used for instrument control, signal acquisition, and data processing. Samples were manually introduced into the GC with a 10-µL micro-syringe (p/n 5182-3428). Experimental conditions are listed in Table 1. All organochlorine and pyrethroid compounds were diluted with hexane.

Table 1. Instrumental Parameters

Instrument	Agilent 6820 GC
Software	Cerity NDS Chemical for QA/QC
Inlet	Split/Splitless; 250 °C; splitless mode; purge time: 0.75 min
Injection volume	2 µL
Column	HP-1, 30 m × 0.32 mm × 0.25 µm (p/n 19091Z-413)
Carrier	Nitrogen, 6.0 psi, constant head pressure mode, 1.2 mL/min (60 °C)
Oven	60 °C (1 min), 30 °C/min to 180 °C, 5 °C/min to 250 °C (5 min), 3 °C/min to 280 °C (10 min)
Detector	µECD; 330 °C; make-up: nitrogen, 60 mL/min

Results

µECD Sensitivity

A chromatogram of organochlorine and pyrethroid pesticides on an HP-1 column is shown in Figure 1. The concentrations, for four benzene hydrochlorides (BHCs), heptachlor, aldrin, heptachlor epoxide, and six pyrethroids are 10 ppb; for p,p'-DDE, dieldrin, endrin, endosulfan I, and endosulfan II, 20 ppb; and for p,p'-DDD, endrin aldehyde, endosulfan sulfate, and p,p'-DDT, 60 ppb. Except for endrin and endosulfan II, the other 20 compounds were fully separated. Among pyrethroid pesticides, two permethrin, four cypermethrin and two fenvalerate isomers were separated.

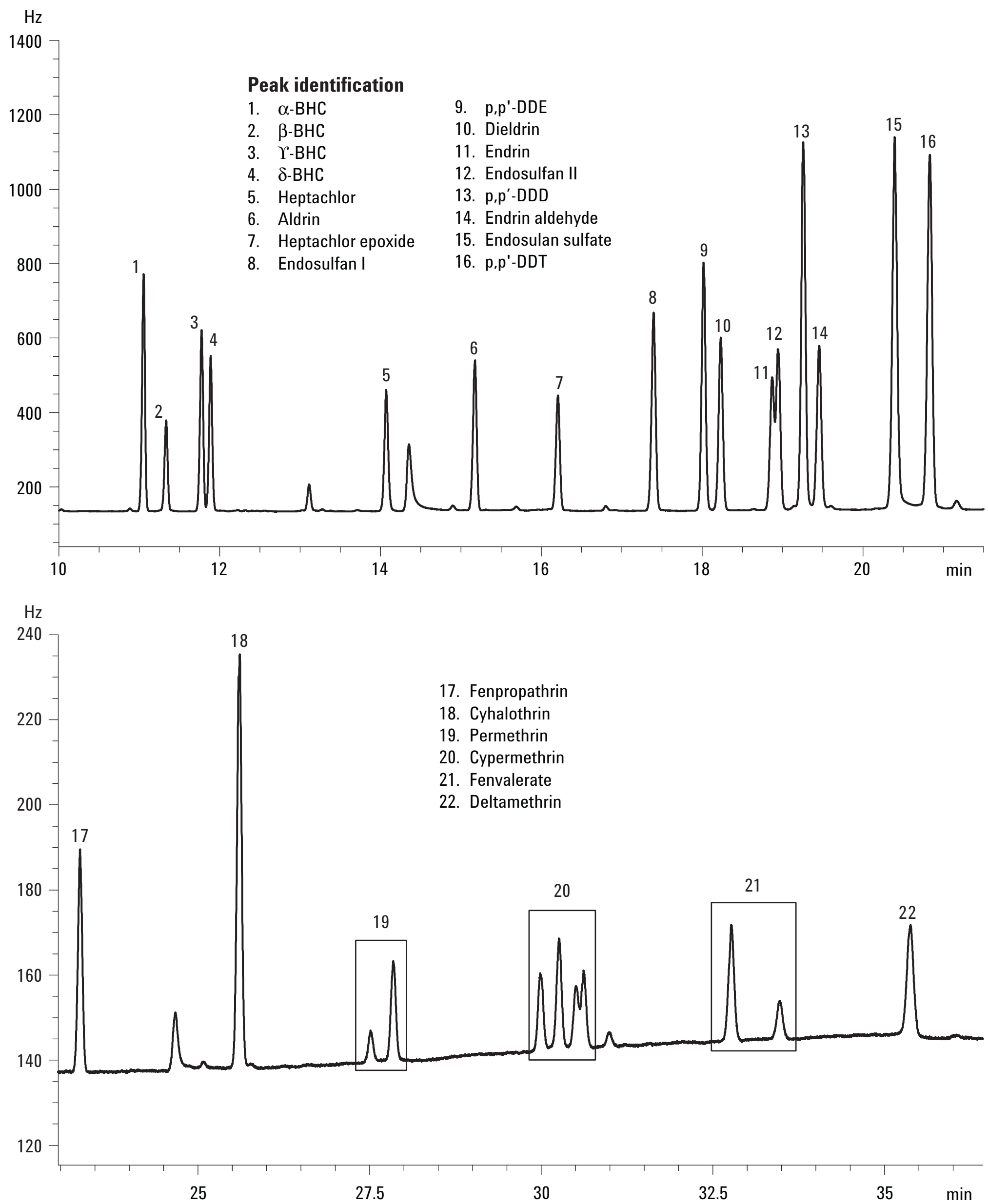

Figure 1. Chromatogram of organochlorine and pyrethroid pesticides on HP-1 column.

The signal-to-noise ratios are larger than 20 for organochlorine pesticides at concentrations of 1–6 ppb. For 5 ppb pyrethroids, the signal-to noise ratios are larger than 10. They can be easily quantitated. Therefore, the µECD provides more than enough sensitivity to meet the requirements of quantitative analysis of pesticides residues.

Linear Range and Response Factors

The calibration curves of γ-BHC and permethrin, typical of organochlorine and pyrethroid pesticides, are shown in Figures 2 and 3, respectively. The linear range and response factors (RFs) are listed in Table 2. The RFs are the ratios of compound concentrations to peak areas. The relative standard deviations (RSDs) of RFs are less than 20%, better than the precision requirements for response factors in the contract laboratory program of the USEPA (the United States Environmental Protection Agency). The linear correlation coefficients for all compounds are better than 0.995.

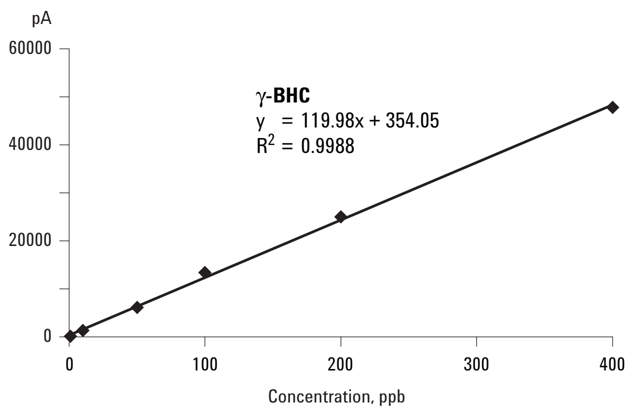

Figure 2. Calibration curve of γ-BHC, a typical organochlorine pesticide.

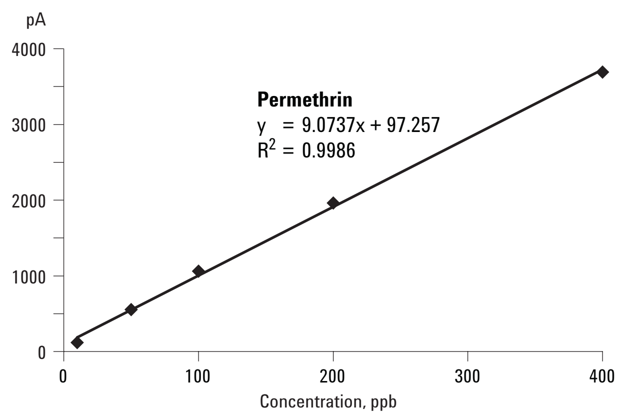

Figure 3. Calibration curve of permethrin, a typical pyrethroid.

Table 2. Linearity Results for Organochlorine and Pyrethroid Pesticides

Compound	Average RF	%RSD of RF	Linear range (ppb)	R^2
α-BHC	0.0064	7.5	1–400	0.9983
ß-BHC	0.0159	18.1	1–400	0.9984
γ-BHC	0.0078	9.4	1–400	0.9988
δ-BHC	0.0087	8.8	1–400	0.9986
Heptachlor	0.0097	13.2	1–400	0.9987
Aldrin	0.0078	8.0	1–400	0.9982
Heptachlor epoxide	0.0100	11.7	1–400	0.9983
Endosulfan I	0.0109	11.7	2–800	0.9983
p,p'-DDE	0.0088	11.0	2–800	0.9948
Dieldrin	0.0123	13.0	2–800	0.9981
Endrin	0.0152	16.3	2–800	0.9991
Endosulfan II	0.0121	15.8	2–800	0.9987
p,p'-DDD	0.0379	8.7	6–2400	0.9983
p,p'-DDT	0.0175	15.8	6–2400	0.9972
Endrin aldehyde	0.0139	10.6	6–2400	0.9989
Endosulfan sulfate	0.0140	10.0	6–2400	0.9988
Fenpropathrin	0.0500	18.0	1–400	0.9951
Cyhalothrin	0.0225	8.6	1–400	0.9982
Permethrin	0.1007	10.2	10–400	0.9986
Cypermethrin	0.0809	7.2	10–400	0.9991
Fenvalerate	0.0728	6.9	10–400	0.9990
Deltamethrin	0.0461	15.0	10–400	0.9996

Conclusion

The Agilent 6820 GC/ ECD system shows good sensitivity and wide linear range for organochlorine and pyrethroid pesticides, and are much better than routine pesticide residue analysis requirements.

References

1. Channel, I., and Chang, I. L., "Analysis of Organochlorine Pesticides and PCB Congeners with the Agilent 6890 Micro-ECD", Agilent Technologies, Publication (23) 5965-8556E www.agilent.com/chem
2. China National Standard Method GB/T 5009. 146-2003, Multiresidue analytical methods for organochlorine and pyrethroid pesticides for plant-originated food, August, 2003

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