

Analysis of Carbamate Pesticides Using Agilent 6820 Gas Chromatograph/Nitrogen Phosphorus Detector

Application

Environmental and Food Analysis

Authors

Weijun Yao, 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/nitrogen phosphorus detector (GC/NPD) was employed to determine carbamate pesticides, following the China National Standard Method GB/T 5009.104-2003. Seven common carbamates were fully separated using an HP-5ms column, 30 m × 0.32 mm × 0.25 μm. Each compound showed good linearity in the 10–1000-ppb range, with a detection limit lower than 10 ppb, which is 10–1000 times lower than the maximum allowable residue level.

Introduction

The nitrogen phosphorus detector (NPD) is both sensitive and selective for organic compounds containing nitrogen and phosphorus, and is widely used in the trace analysis of organonitrogen or organophosphorus pesticides. The carbamates are types of organic synthetic pesticides, widely used to prevent plant diseases. The application of these pesticides may leave residues in agricultural products that may pose potential human health risks via the food chain. The Chinese government publishes maximum residue limits for the pesticides carbofuran, primicarb, and carbaryl in food. GB/T 5009.104-2003 (originally GB14877-1994) is a method used to determine six carbamate residues based on GC/NPD using a packed column [1]. In recent years, capillary-column methods have gradually replaced packed-column methods

because of the excellent separation capability of the capillary column. In this note, the Agilent 6820 GC/NPD is used to determine seven common carbamates including metolcarb, isoprocarb, baycarb, propoxur, carbofuran, primicarb, and carbaryl.

Experimental

All experiments were performed on an Agilent 6820 GC with NPD, deactivated splitless inlet liner (p/n 5183-4696), and Agilent advanced green septa (p/n 5183-4759). Agilent Cerity Networked Data System (NDS) is used for instrument control and data acquisition. Instrumental parameters are listed in Table 1. The standard pesticides were dissolved in acetone solution. Samples were injected manually using a 10-μL syringe (p/n 5182-3428).

Table 1. Instrumental Parameters

Instrument	Agilent 6820 GC
Software	Cerity NDS for chemical QA/QC
Inlet	Split/Splitless inlet
Inlet temperature	250 °C
Injection mode	Splitless
Injection volume	1 μL
Purge time	0.75 min
Column	HP-5ms, 30 m × 0.32 mm × 0.25 μm (p/n 19091S-413)
Carrier gas	Nitrogen, constant pressure of 5 psi, 1.0 mL/min (50 °C)
Oven temp	50 °C (1 min) Ramp 1: 20 °C/min to 100 °C Ramp 2: 5 °C/min to 150 °C (5 min) Ramp 3: 10 °C/min to 200 °C (10 min)
Detector	NPD, 325 °C, white bead (p/n G1534-60570) H ₂ , 3 mL/min Air, 60 mL/min
Detector gas	Makeup, N ₂ , 10 mL/min



Results and Discussions

All compounds were separated with a HP-5ms column. The resolution of baycarb and propoxur is 1.0, although their structures are very similar. The chromatogram of these pesticides is shown in Figure 1. The peak areas are linear with concentrations for all carbamates in the range of 10–1000 ppb. Typical calibration curves for carbofuran and primicarb are shown in Figure 2.

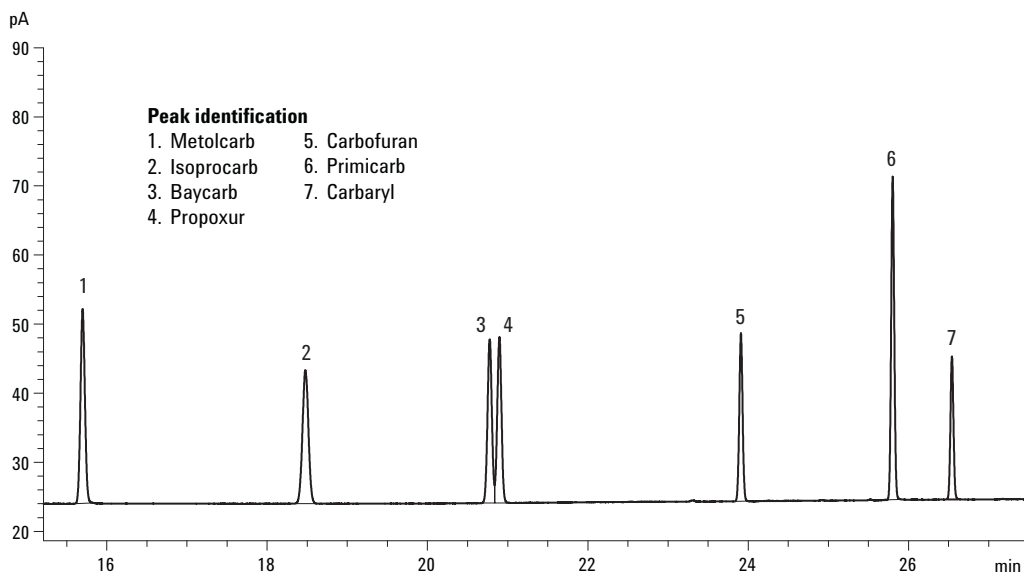


Figure 1. Chromatogram of 1-ppm carbamates using NPD detector.

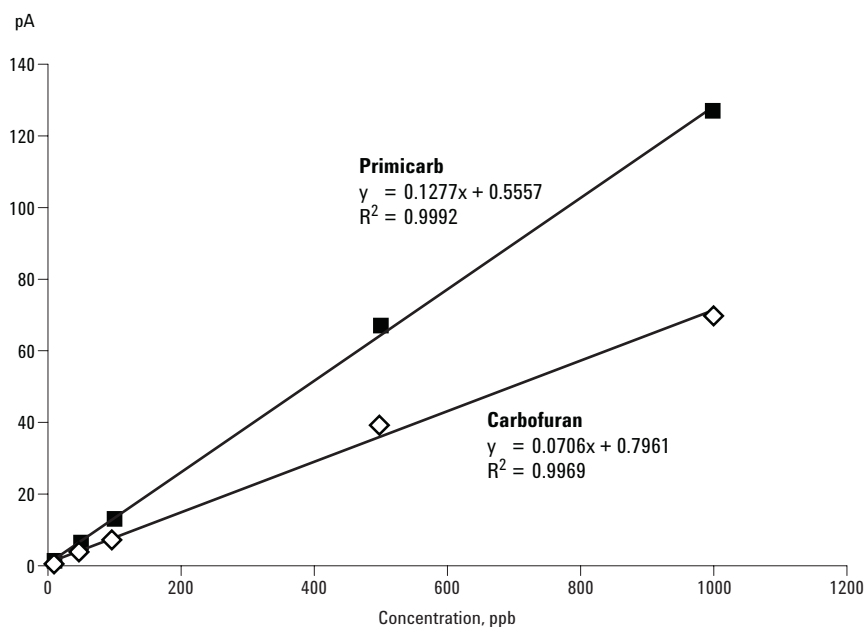


Figure 2. Calibration curves for primicarb and carbofuran.

The chromatogram for the 10-ppb carbamate standard is shown in Figure 3. The signal to noise ratios are between 6–12 for the 10-ppb carbamates. The limits of detection are 4–6 ppb; these are 10 to 1000 times lower than the maximum allowable residue limits stipulated in China National Standard (GB14928.2-94, GB14928.7-94, and GB14971-94), which is 50-ppb primicarb and 5000-ppb carbaryl in cereals [2, 3, 4]. The NPD can easily meet these requirements for the routine analysis of carbamate pesticide residues.

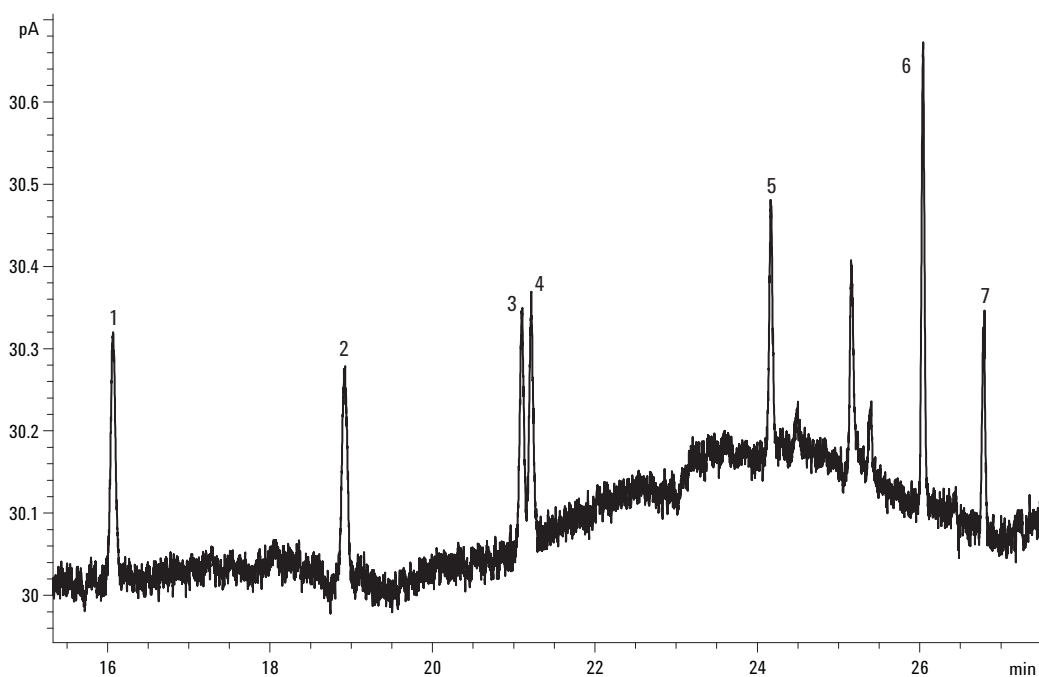


Figure 3. Chromatogram of 10-ppb carbamates standard with 1- μ L injection.

Conclusion

The Agilent 6820 GC with NPD detector can be used for the sensitive and selective measurement of carbamate pesticide residues in food. The detection limit is much lower than the maximum residue limits. Good linearity was obtained in the range of 10–1000 ppb.

References

1. GB/T 5009.104-2003, Determination of carbamate pesticide residues in plant-originated foods.
2. GB14928.2-94 Maximum residue limits of primicarb in food.
3. GB14928.7-94 Maximum residue limits of carbofuran in rice grains.
4. GB14971-94 Maximum residue limits of carbaryl in food.

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