Multi-Pesticide Residue Method for Analysis of Organochlorine and Organophosphorus Pesticide

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Abstract

Pesticide residues were extracted with 70% acetone and transferred to dichloromethane. Extracts were applied to open-column chromatography with florisil and alumina-N. The final extract was analyzed by gas chromatography with electron-capture detector(GC/ECD) and nitrogen-phosphorus detector(GC/NPD). Recoveries of the 17 organochlorine pesticides were ranged from 60.8 to 84.9% and those of 15 organophosphorus pesticides, from 70.5 to 100.0%(except phosmet and azinphos-methyl). The minimum detectable levels of this analytical method were low(0.021-0.058 mg/kg).

Introduction

Pesticides could be deposited in the enviroment and many living organisms involving human would be threatened. Residual pesticides were found in soil, food, and drinking water. In this aspect, not only the design of new pesticides which did not induce environmental problems but also the development of the

precise analytical method to detect the pesticides was demanded. The multi-pesticide residue method was an efficient way to screen a large number of pesticide samples in a relatively short time period. We proposed another multi-residue method for the analysis of organochlorine and organophosphorus pesticide.

Preparation of analytical sample and analytical method for multi-pesticide

Organopesticide samples 10 - 20 g and 70% acetone 100 ml were mixed for 10 min and filtered with Whatman No. 4 filter paper. This solution was evaporated and the residual solution (water layer) was extracted with dichloromethane 100 ml and was shaked for 10 min. The organic layer(lower layer) was filtered with Whatman No. 1PS filter paper, added anhydrous sodium sulfate, and filtered. This filtrate was evaporated and dichloromethane 10 ml were added. This solution was purified by open-column chromatography. The packing material for analytical column was anhydrous sodium sulfate, alumina-N, and florisil. Analytical column(20 cm x 11 mm, id) was prepared. A small plug of cotton was placed at the bottom of the column and added 1 cm layer of anhydrous Na2SO4. Alumina-N 5 g and florisil 5 g were introduced into the column and tapped with 1 cm layer of anhydrous Na2SO4. The column was prewashed with ether : benzene(2:8) 20 ml, acetone 20 ml, and methanol 20 ml. The concentrated sample extract was loaded to the column. The pesticide sample was eluted with following elution solvents ; ether : benzene (2:8) 10 ml, hexane : benzene (1:1) 30 ml, dichloromethane 20 ml, acetone 20 ml, and methanol 20 ml. The eluate was collected in a 250 ml round-bottomed flask. Carefully the eluate was evaporated to dryness with rotary evaporator at 40 oC and redissolved the residue in the proper volume of acetone, and then 1 l of that was injected to GC/ECD and GC/NPD.

Conclusion

1. 32 kinds of organopesticides were detected sharply and the recoveries of the 17 organochlorine pesticides were ranged from 60.8 to 84.9% and those of 15

organophosphorus pesticides, from 70.5 to 100.0%(except phosmet and azinphos-methyl).

2. The minimum detectable levels of this analytical method were low.

3. The major advantage of this method was that cleanup procedure was no longer the rate determining step in the overall procedure because the next sample was undergoing cleanup during GC analysis.

4. The analytical method in this paper could be applied to determine the multi-pesticides simultaneously.

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