Detection of Natural Compounds on Multi-residue Analysis for Vegetables and Fruits

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Introduction

CDFA Multi-Residue Method(MRM) is very compatible to protect food safety from pesticide hazard, because it is efficient way to screen a large number of samples for multiple pesticides in relatively short time. Unknown peaks were observed on several vegetables and fruits during monitoring of pesticides about 89 types, 4205 samples of vegetables and fruits by CDFA MRM in 1999. This research carried out to prevent interference and waste of re-analytical cost on detection of pesticides about vegetables and fruits.

Methods

1. Experimental

1) Pesticides

Pesticide standards were purchased from Chem service(USA), Dr. Ehrenstorfer GmbH(Germany), and Wako Pure Chemical(Japan), Riedel-dehaen(Germany) The Purity of those were over 95% on analysis of GLC and HPLC.

2) Apparatus and Reagents

GC-MSD(Hewlett-Packard 5973 ,USA), GC-NPD/ECD(Hewlett-Packard 6890,USA), GC-FPD(Hewlett-Packard 5890,USA), Mixer(Robot Coupe, France), Homogenizer(Omni, USA), Vacuum manifold(Supelco, USA), Evaporator(Organomation Associates, USA), Acetonitrile, Hexane, and Acetone were special grade for pesticide residue analysis(Wako Pure Chemical, Japan), Florisil-SPE(Supelco, USA), NH_2-SPE(Varian,USA), and 0.2μm Nylon Filter(Whatsman,USA) were used.

2. Analytical Methods

CDFA Analytical procedures for monitoring of pesticides about vegetables and fruits are showed on Fig. 1.

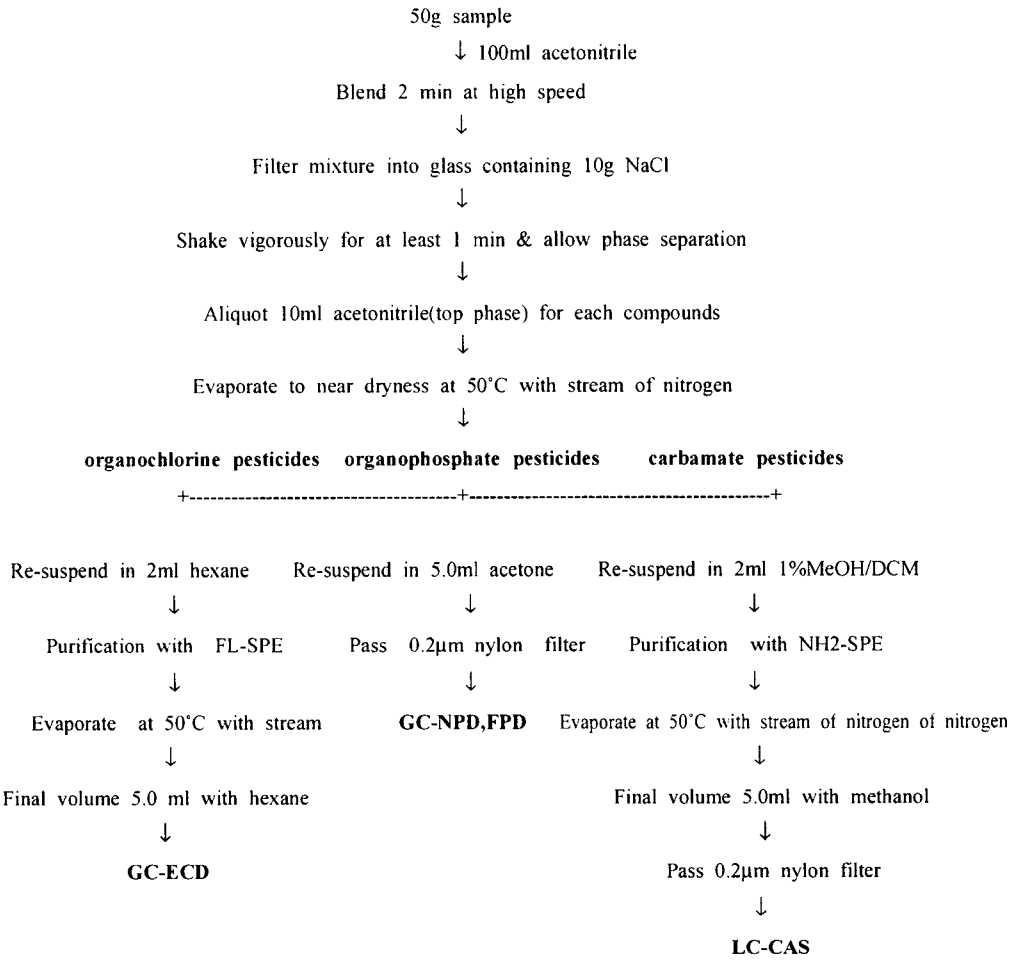

Fig. 1. Schematic diagram for multi-residue screen of pesticide.

Results

Unknown peaks were observed continuously on 7 samples, leafy radish, perilla leaf, pepper, crown daisy, lemon, grape fruit and orange during monitoring of pesticides for 89 types, 420 5 samples of vegetables and fruits in 1999.

Leafy radish, perilla leaf and pepper were observed natural compound peaks on analysis of organophosphate pesticides, and crown daisy and perilla leaf were on organochlorine pesticide s, and lemon, grape fruit and orange were on carbamate pesticides. These samples were anal ysised by GC-MSD to confirm whether unknown peaks is pesticide or not.

Single natural compound peak of leafy radish was detected on similar retention time(RT) of mevinphos by GC-NPD and GC-FPD. But those of pepper were observed 4~6 peaks on simi

lar RTs of EPN and phosmet only on GC-NPD. The natural peak shape of leafy radish was very uniform through whole samples, and GC-MSD was used to confirm that.

In crown daisy, several peaks appeared on GC-ECD, whose RTs were similar to those of v inclozolin and chlorotharonil. Re-analyzing with contrast column could avoid disturbance of th ose, because shape and RT of those had a little difference.

Perilla leaf was observed very small 3~5 peaks on similar RTs of EPN and phosmet, which peaks were detected only GC-NPD but GC-FPD.

On carbamate analysis of lemon, grape fruit and orange, 1~7 natural peaks were detected, a nd similar RT of natural compounds occurred at the that of ethiofencarb on orange and the R T of methiocab on grape fruit.

Consequently, natural peaks occurred in 6~7 vegetables and fruits , which could be confuse d pesticide peaks on CDFA multi-residual analytical method.

Pepper, perilla leaf, and crown daisy could avoid disturbance by changing column and dete ctor type, and other samples by experience of peak patterns and shapes.

Specially, constant analysis condition in our lab. as example monitoring analysis could be a dopted to CDFA method as multi-residual analytical method for various vegetables and fruits.

Reference

1. Obana, H. Hori, S. : Latest analytical methods for the residual pesticides in foods, Japan ese Journal of Toxicology and Environmental Health 42(1), 1:16(1996)
2. Pylypiw, H.M. : Rapid gas chromatographic method for the multiresidue screening of fru its and vegetables for organochlorine and organophosphate pesticides, Journal of AOAC I nternational, 76(6), 1369:1373(1993)
3. Lee, S.M. Papathakis, M.L. Feng, H.C. Hunter, G.F. and Carr, J.E. : Multipesticide resid ue method for fruits and vegetables; California Department of Food and Agriculture, Fres enius J Anal. Chem. 376:383(1991)
4. Yoshii, K. Tsumura, Y. Nakamura, Y. Ishimitsu,S etc. : Multiresidue analysis of various k inds of pesticides in cereals by SFE, GC and HPLC, Journal of Food Hygienic Society o f Japan 40(1), 68:74(1999)
5. Imanaka, M. Katota, M. Kumashiro, K. and Mori, T. : Identification of phylloquinone(Vit amin K1) as an unknown peak in electron capture detection gas chromatograms of pyreth roid insecticide residues, Journal of AOAC International, 79(2), 538:543(1996)