SHIMADZU APPLICATION NEWS

LA190-E013

● HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

No.L291

GPC Cleanup in Analysis of Pesticide Residue in Food

When analyzing pesticide residues in agricultural products, fats and pigments contained in the sample should be removed during pretreatment, because they can contaminate the GC or GCMS injection port, and also interfere with the peaks of the target substances. However, the conventional solvent extraction method, which is time-consuming and involves complex procedures, is not suited to processing multiple samples.

GPC (Gel Permeation Chromatography) is a technique that separates the chemical constituents of a sample according to molecular size. It efficiently separates fats and pigments, which have larger molecular sizes than pesticides, from pesticide components, allowing automated purification. Due to this advantage, GPC is adopted as a purification (cleanup) method in the residual pesticides pesticide residue rapid analysis method prescribed by the Japanese Ministry of Health, Labour and Welfare (Chemical Hygiene No. 43, April 8, 1997).

This Application News introduces the principle of GPC cleanup and application examples of the Shimadzu GPC cleanup system.

■ Principle of GPC Cleanup Method

Fig. 1 shows the principle of the GPC cleanup method. The packing material contained in the GPC column has pores of a specific size. Substances with smaller molecular sizes such as pesticides (gray circles in the figure) will permeate deep into the pores, while larger substances such as fats and pigments (slant-lined circles in the figure) do not permeate. As a result, fats and pigments are eluted quickly from the column and pesticides are eluted later. Purification is conducted by separating out the elution portion of pesticides.

* In actuality, separation is conducted by molecular size, as well as adsorption to the packing material.

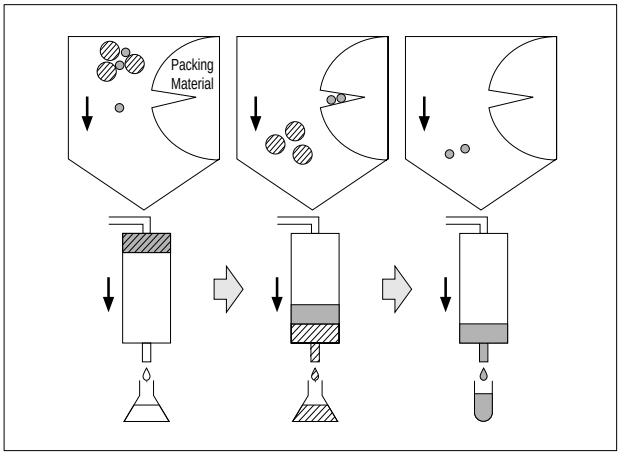

Fig.1 Principle of GPC Cleanup

■ Pretreatment for the pesticide residue rapid analysis method

Fig. 2 shows a flowchart for the the pesticide residue rapid analysis method. The official methods (analyzing individual pesticides by different methods) use liquid-liquid or solid phase extraction to remove fats and pigments from the sample, while the rapid analysis method uses GPC. The rapid analysis method simultaneously processes all pesticides in the time required for the official method to process a single pesticide.

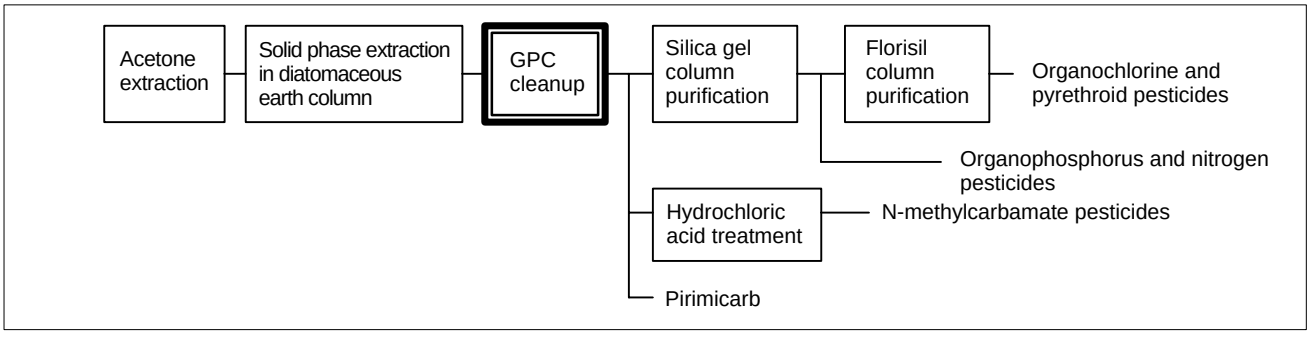

Fig.2 Flow Diagram of Pretreatment for Rapid Analysis Method

■ Preparative Conditions

Rice was extracted according to the rapid analysis method and purified using the Shimadzu GPC cleanup system. Table 1 shows the preparative conditions and Fig. 3 the chromatogram obtained. In Fig. 3, the chromatogram of fluvalinate and chinomethionate obtained by GPC is overlaid with the sample chromatogram. In principle, pesticides subjected to the rapid analysis method elute between these two substances, so the elution time between these two substances are taken out.

Table 1 Preparative Conditions

Instrument	: Shimadzu GPC Cleanup System
Column	: CLNpak EV-G + CLNpak EV-2000
Mobile Phase	: A : Ethyl acetate, B : Cyclohexane A / B =1 / 4(v / v)
Flow Rate	: 4.0ml / min.
Detection	: SPD-10A*VP* at 254nm

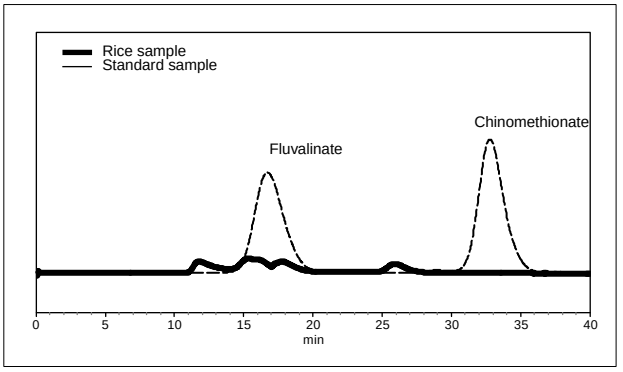

Fig.3 GPC Chromatogram of Rice

■ Example of Organophosphorus Pesticide Analysis

Fig. 4 shows the chromatogram for a soybean extract spiked with organophosphorus pesticides. The sample was purified by the Shimadzu GPC cleanup system and a silica gel minicolumn, dissolved in acetone and analyzed by GC. Table 2 shows the analytical conditions.

Fig. 5 shows the chromatogram for the same soybean extract obtained by GC analysis after pretreatment in accordance with the official method. Almost identical results were obtained from the two methods.

Table 2 Analytical Conditions

Instrument	: GC-2010
Column	: Rtx-1(0.53mm × 15m, df=1.5μm)
Col.Temp.	: 80˚C(1min.)→8˚C/min.→250˚C(10min.)
Inj.Temp.	: 230˚C
Det.Temp.	: 280˚C
Carrier Gas	: He, 16.5mL/min., Splitless mode(1min.)
Detection	: FPD-2010

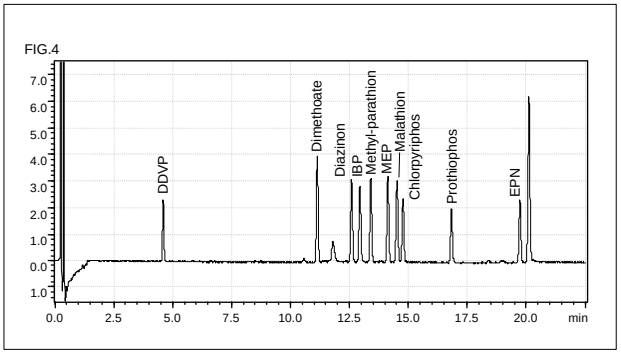

Fig.4 Chromatogram of Organophosphorus Pesticides Obtained by Rapid Analysis Method

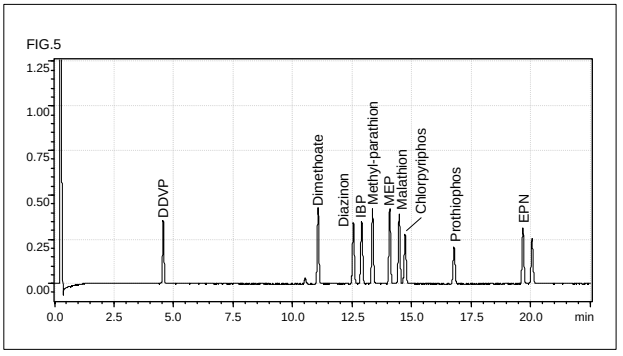

Fig.5 Chromatogram of Organophosphorus Pesticides Obtained by Official Method

Reference:
1) Pesticide residue Rapid Analysis Method Development Committee: Food Sanitation Research, Vol. 47, p. 35 (1997)
2) Isao Saito: LCtalk Vol. 35, p. 3 (1995)

SHIMADZU

SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

Printed in Japan 3100-02405-10A-IK