

## 生薬中の残留有機リン系農薬の分析 (第2報\*)

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## Analysis of Organophosphorus Pesticide Residues in Crude Drugs (Part 2)

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## Summary

A method was developed for simultaneous determination of 28 organophosphorus pesticides in *Angelicae Radix*, *Atractylodis Lanceae Rhizoma*, *Atractylodis Rhizoma*, *Bupleuri Radix*, *Cimicifugae Rhizoma*, *Coicis Semen*, *Ephedrae Herba*, *Foeniculi Fructus*, *Ginseng Radix*, *Magnoliae Flos*, *Menthae Herba*, *Paeoniae Radix*, *Puerariae Radix* and *Zingiberis Rhizoma*. The pesticides were extracted with aqueous acetonitrile. The extract was cleaned up on a C18 mini-column, and the pesticide-containing fraction of the eluate was concentrated. After addition of sodium chloride to the concentrated aqueous solution, the pesticides were re-extracted with *n*-hexane. In the case of *Bupleuri Radix*, a small quantity of methanol was added to the *n*-hexane to prevent emulsification. The extract was washed with water and dried over anhydrous sodium sulfate. The extracts of *Atractylodis Lanceae Rhizoma* and *Atractylodis Rhizoma* were further cleaned up on a Diol mini-column and a Silica gel mini-column. The extracts of the other crude drugs were further cleaned up on a Silica gel mini-column. In the case of *Bupleuri Radix*, the pesticides were eluted with a mixture of acetone and *n*-hexane after the column had been washed with *n*-hexane. The analysis was performed by gas chromatography with FPD detection.

The recoveries of organophosphorus pesticides added at the concentration of 0.4  $\mu\text{g/g}$  to the crude drugs, except for *Cimicifugae Rhizoma* and *Paeoniae Radix*, were mostly in the range of 70~120% (peak area method). The recoveries of methidathion, phosmet, edifenphos, phosalone and pyridaphenthion added to *Paeoniae Radix* were greater than 120%. The recoveries of chlorpyrifos, ethion and leptophos added to *Cimicifugae Rhizoma* were only 51%, 35% and 22%, respectively, most likely due to reactions with components of the crude drug during moistening for 1 hour. The detection limits were 0.01 to 0.06 ppm.

The established method was applied to 51 samples in 15 kinds of crude drugs. Five kinds of organophosphorus pesticides were detected in 8 samples of 4 kinds of crude drugs in the range of trace to 0.22 ppm.

## Key words

*Angelicae Radix*, *Atractylodis Lanceae Rhizoma*, *Atractylodis Rhizoma*, *Bupleuri Radix*, *Cimicifugae Rhizoma*, *Coicis Semen*, *Ephedrae Herba*, *Foeniculi Fructus*, *Ginseng Radix*, *Magnoliae Flos*, *Menthae Herba*, *Paeoniae Radix*, *Puerariae Radix*, *Zingiberis Rhizoma*, Organophosphorus pesticide, Pesticide residue, GC-FPD