

生薬中の残留有機リン系農薬の分析 (第3報*)

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(受付: 平成 21 年 10 月 8 日, 受理: 平成 22 年 2 月 3 日)

Analysis of Organophosphorus Pesticide Residues in Crude Drugs (Part 3*)

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Summary

A method was developed for simultaneous determination of 28 organophosphorus pesticides in Angelica Dahurica Root (ANGELICAE DAHURICAE RADIX), Astragalus Root (ASTRAGALI RADIX), Cnidium Rhizome (CNIDII RHIZOMA), Gardenia Fruit (GARDENIAE FRUCTUS), Glehnia Root (GLEHNIAE RADIX CUM RHIZOMA), Magnolia Bark (MAGNOLIAE CORTEX), Pinellia Tuber (PINELLIAE TUBER), Poria Sclerotium (PORIA), Rehmannia Root (REHMANNIAE RADIX), Rhubarb (RHEI RHIZOMA) and Senega (SENEGAE RADIX).

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. In the case of Rhubarb, a brown sticky insoluble material was separated from the water solution and dissolved in methanol. After addition of sodium chloride to the concentrated aqueous solution, the pesticides were re-extracted with *n*-hexane. In the case of Senega, 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 Magnolia Bark were further cleaned up on a Diol mini-column and a Silica gel mini-column. The extracts of Rhubarb were further cleaned up on an ENVI-Carb/LC-NH₂ mini-column. The extracts of the other crude drugs were further cleaned up on a Silica gel mini-column.

The determination was performed by gas chromatography with FPD detection. The recoveries of organophosphorus pesticides added at the concentration of 0.4 µg/g to the crude drugs, except for Angelica Dahurica Root, Cnidium Rhizome, Glehnia Root and Magnolia Bark were mostly in the range of 70~120% (peak area method). The recoveries of methidathion, phosmet, edifenphos and phosalone added to Angelica Dahurica Root, Cnidium Rhizome and Glehnia Root were greater than 120%. The recoveries of quinalphos and iprobenfos added to Magnolia Bark were 42% and 33%, respectively. These lower recoveries may be due to reactions with components of the crude drug during extraction procedures.

The established method was applied to 111 samples of 16 kinds of crude drugs. Seven kinds of organophosphorus pesticides were detected in 6 samples of 3 kinds of crude drugs produced in Japan and 12 samples of 4 kinds of crude drugs produced in the People's Republic of China in the range of trace to 0.54 ppm.

The Agricultural Chemicals Regulation Law was revised on March 10, 2003, and states that a person who uses agricultural chemicals shall not use them contrary to the regulations determined by ministerial ordinances (Article 12). Four kinds of organophosphorus pesticides regulated by this law were detected in 4 samples of 2 kinds of crude drugs.

Key words

Angelica Dahurica Root, Astragalus Root, Cnidium Rhizome, Gardenia Fruit, Glehnia Root, Magnolia Bark, Pinellia Tuber, Poria Sclerotium, Rehmannia Root, Rhubarb, Senega, Organophosphorus pesticide, Pesticide residue, GC-PPD