投稿/原著

Improvement of the Official Method to Analyze Organochlorine Pesticides in *Glycyrrhizae* Radix

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Summary

The official method to analyze organochlorine pesticides in crude drugs, described in The Japanese Pharmacopoeia 15th edition, does not provide adequate recovery of organochlorine pesticides from *Glycyrrhizae* radix. Here, we examined the reason for this. Our results indicated that the low recovery in the official method is due to absorption of the pesticides on the brown tarry precipitate that is formed during the concentration step. Therefore, in the present study, we modified the official method by preparing the test solution for gas chromatography without the concentration step. The modified method gave satisfactory recovery and relative standard deviation (RSD) for residue analysis. Since it is identical to the official method, except for omission of the concentration step, it can be readily adopted for analysis of organochlorine pesticides in *Glycyrrhizae* radix. The improved method was applied to four samples of *Glycyrrhizae* radix, and their contents of BHCs and DDTs were found to be below the limit of detection.

Key words

Crude drug, Organochlorine pesticide, Glycyrrhizae radix, Gas chromatography/electron capture detector

INTRODUCTION

Ensuring the safety of crude drugs is important, because patients generally take crude drugs for long periods, sometimes years. In Japan, maximum residue levels (MRLs) have been set for a range of pesticides, including α -BHC, β -BHC, γ -BHC, δ -BHC (BHCs), p, p'-DDE, o, p'-DDT, p, p'-DDD, and p, p'-DDT (DDTs) (Fig. 1), in 14 crude drugs, including Glycyrrhizae radix10. The official method to analyze organochlorine pesticides, using a gas chromatograph equipped with an electron capture detector (GC/ECD)10, was established based on a collaborative study2. However, the official method gives inadequate recovery of BHCs and DDTs from Glycyrrhizae radix (between 41% and 69%)²⁾. Glycyrrhizae radix is a very important crude drug because it is imported in large quantities (2000~10,000 tons/year) and is frequently used in traditional Kampo formulae1).

We examined the application of the official method to the analysis of BHCs and DDTs in *Glycyrrhizae* radix in order to find the reason for the low recovery, and slightly modified the original method to obtain acceptable recovery and RSD.

MATERIALS AND METHODS

Pesticide standards

Pesticide standards were obtained from Wako Pure Chemical Industries (Japan) and Riedel de Haën (Germany). Each compound was dissolved in acetone to make a 0.5 mg/mL standard stock solution. These were diluted to prepare solutions spiked at $5 \mu \text{g/mL}$.

Reagents

Acetone, hexane, diethyl ether, sodium chloride, and anhydrous sodium sulfate of pesticide analysis grade, sul-