

VALIDATION OF NEW MULTI-RESIDUE METHOD FOR THE DETERMINATION OF ORGANOPHOSPHORUS INSECTICIDES IN CHRYSANTHEMUM FLOWER BY CAPILLARY GAS CHROMATOGRAPHY

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ABSTRACT

A method for the analysis of 8 organophosphorus (OP) with wide range of physico-chemical properties in Chrysanthemum flower was developed and validated. The procedure involved the extraction of the spiked sample (5 g) with 100 mL acetone and the final volume was not subjected to any clean-up procedure. The analyte concentrations were determined through gas chromatography GC-9A, equipped with flame photometric detector (FPD). The mean recoveries of the spiked blank samples at three concentrations for different pesticides were found in the range of 91.00–99.67 % and relative standard deviation lower than 12.0%. Limit of detection (LOD) of the method ranged between 0.06 and 0.08 mg kg⁻¹ and the limit of quantitation (LOQ) were varied from 0.09 to 0.1 mg kg⁻¹. The selected parameters for method validation (precision, accuracy, calibration function, sensitivity, selectivity, limit of detection and limit of quantitation) were within the acceptable limits according to the EU criteria (SANCO) for the 8 insecticides included in the validation.

Keywords: Chrysanthemum flower; Multi-residue analysis; gas chromatography; validation

INTRODUCTION

Medicinal plants have been used therapeutically all around the world, being an important aspect of various traditional medicine systems. During the past few decades, phytotherapy has started to be increasingly used even in industrialized countries. In low and middle-income countries, phytotherapy never stopped being important, often representing the only therapeutic system to which certain people could refer. With the ever-increasing use of herbal medicines worldwide and the rapid expansion of the global market for these products, the safety and quality of medicinal plant materials and finished herbal medicinal products have become a major concern for health authorities, pharmaceutical industries and the public. Contaminants in herbal medicines are classified into physicochemical contaminants and biological contaminants. A variety of agrochemical agents and some organic solvents may be important residues in herbal medicines. (WHO guidelines 2007). However, pesticides are often used in order to improve productivity and profit margins in the production of Chinese medicinal materials. The published researches on pesticide residues in crude herbal materials indicate that the presence of organophosphorus pesticide (OPP) residues is quite common (Ozbey and Uygun 2007; Vidal *et al.* 2002; Garrido-Frenich *et al.* 2003). Hence pesticide residues in Chinese medicinal materials become pitfalls in safety and present obstacles to be acknowledged by the international community (Qing *et al.* 2009). In recent years, with the significant improvements in pesticide analytical techniques and tremendous concerns in the safety of consumers' products, the pesticides residues in foods have been more strictly monitored, the respective analytical methods of 3 pyrethroids and 12 organophosphorous (OPs) pesticides, but no MRLs for pyrethroids and organophosphorous pesticides (OPPs) have been established for all Chinese medicinal materials, (China Pharmacopoeia 2005). Among several Medicinal plants, Chrysanthemum flower (Pharmaceutical Name:

Flos Chrysanthemi, Botanical Name: *Chrysanthemum morifolium* Ramat. Common Name: Chrysanthemum flower) is one of the most popular Chinese herbal medicines. Chrysanthemums not only are these flowers beautiful additions to gardens and homes but they have also shown to reduce indoor air pollution and also have medicinal properties, it can be used to treat western symptoms such as: irritability, thirst, cough-phlegm, cough-blood, carbuncles, sores and breast-lumps and Chinese symptoms such as: lung-heat, lung-phlegm heat, yin-deficient and fire poison. Although Chrysanthemums produce their own natural insecticide they still have a plethora of pests that can attack them and cause leaves and petals to wilt, and sometimes even kill the entire plant. Invertebrates such as nematodes, leaf roller, thrips, aphids, mites, caterpillars, weevils and flies are most common (Williams and Pullman 2000; Sabir *et al.* 2012). Chrysanthemums usually attacked by a variety of chewing, sucking and rasping (scraping) pests, the most common insect pests of Chrysanthemum are Chewing insects: Budworms (*Helicoverpa spp.*), Cineraria leafminer (*Chromatomyia syngenesiae*), Chrysanthemum Leafminer (*Phytomyza syngenesiae*), Chrysanthemum gall midge (*Rhopalomyia chrysanthemi*) and European earwigs (*Forficula auricularia*); Sucking insects: Chrysanthemum aphids (*Macrosiphoniella sanborni*), Broad mite (*Polyphagotarsonemus latus*), Cyclamen mite (*Phytonemus pallidus*), Two-Spotted Spider Mites (*Tetranychus urtica*); Rasping insects: Western Flower Thrips (*Frankliniella occidentalis*) (Marjan *et al.* 2006). Late season infestations of pest populations on Chrysanthemums can cause severe damage and reduced flower quality, therefore the application of organophosphorus pesticides is became necessary to reduce the damage and to improve the flower quality and this may lead to some residues on the flower. To be able to detect such residues, we have to develop a valid, simple, reliable, rapid, efficient method. Method validation is an important requirement in the practice of chemical analysis. The analyst must generate information to show that a method intended for these

purposes is capable of providing adequate specificity, accuracy and precision, at relevant analyte concentrations and in appropriate matrices (Hill and Reynolds 1999). Most of pesticide residue methods are carried out with modern, sophisticated and expensive instruments (GC/MS, LC/MS), such kind of instruments are rarely found and usually not available in each pesticide residue laboratories, especially in the developing countries. In the literature, no method was found to determine the residues of organophosphorus insecticides in Chrysanthemum flower, therefore, the present study deals with developing a rapid and efficient multiresidue analytical method for the determination of 8 insecticides in Chrysanthemum flower using common

gas chromatography equipped with flame photometric detector (FPD).

MATERIALS AND METHODS

Sample Preparation

All crops were purchased at a local market in Hangzhou (China) and we confirmed that the concentrations of pesticide residues in foods were below detectable levels with the proposed method. About 500 g of food was chopped in a DC- 200 high speed blender (Shanghai, China) for 2 min to obtain thoroughly mixed homogenates. Wide range of physico-chemical properties of representative insecticides were selected to assess the laboratory performance of the method, (Table 1).

Table 1. Summary of physico-chemical properties of selected representative insecticides

Active ingredient	Water solubility		log Pow		Vapour pressure		Hydrolysis (pH 9) half-life
	mg/l	°C		°C	mPa	°C	
Dimethoate	23.3	20	0.704	20	1.10	25	12 d
Malathion	145	25	2.7	25	5.30	30	11.8 h
Methamidophos	200	20	-0.8	20	2.30	20	120 h
Omethoate	39.8	25	-0.74	20	3.30	20	4.4 d
Parathion	55–60	25	3.43	20	1.30	20	100 d
Parathion - methyl	70	25	2.8	25	1.30	20	33 d
Phosmet	25	25	2.95	20	0.065	25	0.02 d
Trichlorphon	120	20	0.43	20	0.21	20	0.5 h

The standard pesticides were obtained from Sigma–Aldrich (Germany) with purity more than 96 %. The stock and working solutions were prepared in pesticide residue grade acetone solvent, obtained from Lian You factory (Hangzhou, China).

Extraction

An aliquot of 5 g of sample homogenate was weighed into a high speed blender jar Model DC- 200 model tool factory (Shanghai, China) and the fortification standard was added into the sample and allowed to equilibrate for 12h before extraction. The spiked sample was extracted with a 100 ml of acetone for 2 min. The extract was transferred into 250 mL conical flask, and the blender jar was washed three times with 100 mL acetone into the same conical flask and shaken with a mechanical shaker model Haake SWB20 (Germany) for 30 minutes. The extract was filtered through glass funnel containing anhydrous sodium sulfate (analytical grade) obtained from Lan qi reagents and chemical factory (China) and the filtrate was collected into 250 mL round bottom flask. The conical flask washed with 100 mL acetone in three portions and added to the filtrate. The filtrates and washing solutions were combined before being concentrated to ca 2 mL with rotary vacuum evaporator model SBXZ (Shanghai, China) at 40 °C. The final volume of the extract was adjusted to 10.0 mL test tube with acetone and was not subjected to any clean-up procedure, the final volume analyzed was 1 µL. The analyte concentrations were determined through gas chromatography GC-9A, equipped with FPD detector connected to a C-R3A reporting integrator from

Shimadzu Corp. (Kyoto, Japan) and a 30-m AT-1701 (Altech) capillary column, with an internal diameter of 0.53 mm and film thickness of 1.2 µm. The analytical conditions were injector temperature: 250°C, the carrier gas (N₂) with a flow rate of 35 mL min⁻¹. Hydrogen and air was used as a detector gases with flow rate of 90 and 500 mL min⁻¹ respectively. The detector temperature set at: 275°C, and the injections were done in the on-column injection mode with an oven temperature program of 120 °C for 1 min, 20 °C min⁻¹ – 180 °C, and 40 °C min⁻¹ – 250 °C for 2 min. Recoveries were determined by adding the tested pesticides to a control Chrysanthemum sample and the pesticide residues were calculated by external standard method. Analytical validation of the method was performed to check the credibility of the data in the quantitative analyses by applying the following factors: recovery, accuracy, precision, determination coefficient (R²), detection (LOD) and quantification (LOQ) limits (Hill and Reynolds 1999; Aysal *et al.* 2007). The selected parameters for method validation for the analysis of pesticide residues in herbal products were mainly taken from the European Directorate General for Health and Consumer Affairs (SANCO/12495/2011).

RESULTS AND DISCUSSION

Selection of representative analytes

In order to develop a universal analytical method and to assess the laboratory performance of the method, representative insecticides (different polarities, solubilities, volatilities and pK_a values) that have to be

simultaneously extracted and analysed should be selected (FAO/WHO) 2003; Alder *et al.* 2006) Table 1 demonstrates the physico-chemical properties of the selected representative compounds with wide range of **properties:** water solubility from 23.3 to 200 mg/l; logPow: from -0.8 to 3.43; vapour pressure: from 0.065 to 5.30 mPa; and hydrolytic stability from 0.02 to 100 days at pH 9 were selected.

Pesticide extraction from samples

A number of solvents have been used for multiresidue extractions and the most common include acetone (P. Van Zoonen 1996; Hans-J SJ 2000) ethyl acetate (Hernando *et al.* 2001; Berrada *et al.* 2006; Georgakopoulos *et al.* 2007) and acetonitrile (Lee *et al.* 1991; Fillion *et al.* 2000; Anastassiades *et al.* 2003). Acetone was selected as the solvent for extraction of

pesticides because of its effectiveness for very polar insecticides (logKow < 0, methamidophos, omethoate), polar and nonpolar pesticides from a diverse range of matrices. Its other advantages include low toxicity and cost, miscibility with water and ease of evaporation.

Clean up

Inadequate or extensive clean-up of extracts may result in the partial loss of some compounds as well as increased labour, cost demands and can lead to adverse effects related to the quality of the generated data (Darinka *et al.* 2003). In this experiment, the selective detector (FPD) was used for the determination of the residues, we found that clean up was not necessary to perform the method. Interference from co-extractives was not found to interfere with the tested pesticides (Figure 1).

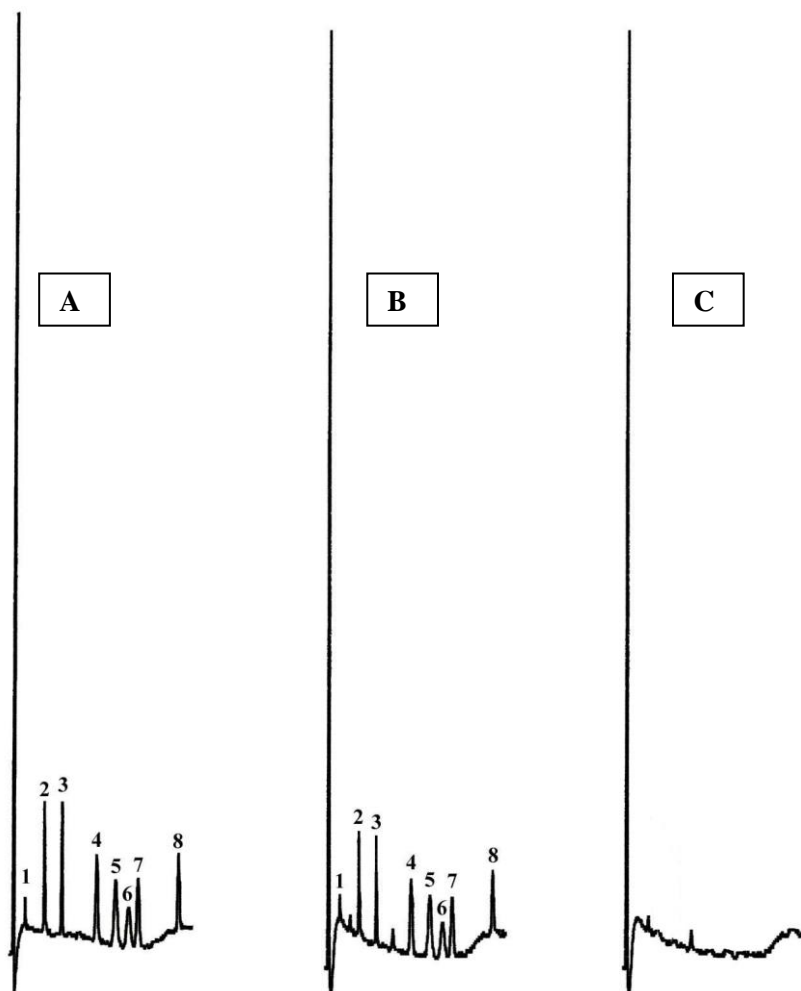


Figure 1. Representative gas chromatograms of (A) Insecticide standards (B), Chrysanthemum sample fortified with OP insecticide 0.1 mg kg⁻¹. Peak identities are Omethoate (1), Methamidophos (2), Trichlorphon (3), Dimethoate (4), Parathion methyl (5), Malathion (6), Parathion (7), Phosmet (8). (C) Control Chrysanthemum.

Method validation

Validation is performed to verify that the method is fit for purpose, which means that the desired commodities and analytes are evaluated to achieve acceptable recoveries, reproducibilities, and detection

limits. Sample preparation was rapid and straightforward. Cost and waste, consumption of solvents and reagents was low, that is with agreement with EU directives with regard to sample definition (Annex I to Directive 90/642/EEC 1993) and laboratory

sample size (Commission directive 2002) for residue analysis should be respected. In order to prevent the degradation of the analytes, samples were extracted by blending for 2 minutes only before shaking, It has been reported that for well homogenized samples extraction by vortex mixing or shaking, instead of high-speed blending (Turrax) suffices for effective extraction (Anastassiades *et al.* 2003).

Recovery test

Recovery experiments using spiked blank samples at three concentrations showed that mean recoveries for different pesticides were found in the range of 91.00–99.67 % and relative standard deviation (RSD) were all lower than 12.0% (Table 2), the results met the EU criteria (recovery 70–120%, RSD \leq 20% (SANCO 2011) for the 8 insecticides included in the validation.

Table 2. Recovery (%) and reproducibility (CV %) of the for 8 representative insecticides at different spiking levels (n =9).

Insecticide	Fortification (mg kg ⁻¹)	Batch 1		Batch 2		Batch 3		Overall	
		mean	% CV	mean	% CV	mean	% CV	mean	% CV
Dimethoate	2.00	94	10	89	2	88	3	91	5
	0.20	93	11	92	10	92	9	92	10
	0.10	104	8	90	3	110	4	101	10
Malathion	2.36	86	4	86	5	85	6	86	5
	0.24	106	9	105	12	105	11	106	10
	0.12	83	4	100	4	102	10	95	11
Methamidophos	1.19	108	12	101	11	113	9	107	11
	0.12	99	17	98	9	102	12	100	13
	0.06	83	6	103	13	92	10	92	11
Omethoate	2.21	94	9	86	9	97	9	92	9
	0.22	100	10	100	11	108	6	103	9
	0.11	81	9	98	6	82	8	87	11
Parathion	1.89	89	14	86	9	85	10	86	11
	0.19	99	10	101	14	101	6	100	10
	0.10	77	5	94	12	89	10	87	10
Parathion- methyl	2.14	86	6	92	13	84	7	87	9
	0.21	98	12	110	11	100	13	103	12
	0.11	99	6	83	12	83	6	88	10
Phosmet	2.26	98	8	97	10	95	12	97	10
	0.23	103	10	103	11	102	7	103	10
	0.12	85	5	105	10	87	5	92	12
Trichlorphon	3.28	100	9	101	9	107	9	103	9
	0.33	104	7	103	13	93	14	100	11
	0.17	79	7	85	9	97	7	87	11

Precision

Recovery data from the fortified controls was used to measure repeatability and reproducibility variation of the method at 0.1-2 mg kg⁻¹ and expressed as % CV. Each analytical batch comprised of 10 negative control samples, and the test was repeated on three occasions at each concentration. All the recoveries obtained were within the acceptable range (70-120) see Table 2.

Accuracy

Accuracy was described as the overall recovery (table 2), it is the mean recovery obtained for each compound, at each fortification level, for all observations.

Calibration and matrix function

Organophosphorus insecticide standard solutions (0.1 to 2 mg kg⁻¹) were prepared in acetone and assayed on three separate occasions. Standard curves were

constructed for each insecticide by plotting peak height versus concentration; line was fitted by using linear regression. The FPD response for all pesticides was linear in the range assayed with correlation coefficients $r^2 > 0.990$ (Table 3). In order to determine the matrix effect on linearity response, Chrysanthemum flower control extracts were fortified with concentrations as above prior to GC analysis. Matrix standard curves for the tested insecticide were also constructed as above. Results in Table 3 demonstrate that, curves are linear over the tested concentration range. Two tailed paired t-test was used to compare the standard curve in matrix against that in solvent, results indicated that t-critical was greater than t-calculated at the 95% confidence level. Therefore there was no significant difference in recovery of OPs between the standard curve in matrix against that in solvent (Table 3).

Table 3. t-test, Mean Values of Slope, Intercept and Regression Coefficient (r^2) for Organophosphorus Insecticides In Standard Solutions and Matrix

Insecticide	Slop		Intercept		R^2		t-test	
	Std	Matrix	Std	Matrix	Std	Matrix	tcalc	t-critical
Omethoate	0.965	0.956	0.639	18.314	0.992	0.993	1.8898	4.303
Methamidophos	7.238	7.268	2.406	2.503	0.993	0.994	1.3093	4.303
Trichlorphon	1.977	1.977	2.564	2.864	0.991	0.990	2.5000	4.303
Dimethoate	2.496	2.496	1.687	1.887	0.991	0.993	4.000	4.303
Parathion	1.874	1.882	1.228	1.510	0.993	0.994	0.2500	4.303
parathion-methyl	0.967	0.941	0.763	1.084	0.996	0.997	2.0000	4.303
Phosmet	1.513	1.490	1.383	1.704	0.994	0.994	1.7321	4.303
Trichlorphon	1.061	1.066	1.497	1.813	0.991	0.990	0.6547	4.303

Sensitivity

Slope, intercept and correlation coefficient (r^2) was used to express the sensitivity of the method, for each compound, over the range 0.1 to 2 mg kg⁻¹ (Table 3). All the results obtained were within the acceptable limits.

Selectivity

Peak retention time was used to determine the selectivity of the method, no interference from co-extractives was found to interfere with the tested pesticide, good resolution was obtained by the method; (figure 1). Dual column confirmation was used, pesticides eluted with different retention times on 2 different column phases, but retention times remain constant from both columns (Mid-polar AT-1701 capillary column (Altech), 30m, 0.53mm id, 1.2µm and

Non-polar HP-5 capillary column (Agilent) 30m, 0.32mm id, 0.25µm film thickness. 1 = Omethoate, 2 = methamidophos, 3 = trichlorphon, 4= dimethoate, 5 = parathion-methyl, 6 = malathion, 7 = parathion, 8 = phosmet (Table 4).

Limit of Detection (LOD) and Limit of Quantitation (LOQ):

The limits of detection (LOD) and quantification (LOQ) for all pesticides were calculated by considering a value 3 and 10 times the background noise obtained for blank samples, respectively. LOD and LOQ for all OP insecticides at 0.1 mg kg⁻¹ are shown in Table 4. LODs of the method ranged between 0.06 mg kg⁻¹ for Parathion and 0.08 mg kg⁻¹ for Phosmet, LOQs were varied from 0.09 to 0.1 mg kg⁻¹ for various analytes studied.

Table 4. Method Detection Limit (MDL) and Limit of Quantitation of OPs Insecticide on Gas Chromatograph by FPD.

Name	Retention Time	Method Detection Limit (MDL) (mg kg ⁻¹)	Limit of Quantification (LOQ) (mg kg ⁻¹)
Omethoate	1.03	0.07	0.09
Methamidophos	2.24	0.07	0.10
Trichlorphon	3.38	0.06	0.09
Dimethoate	5.63	0.07	0.10
Parathion - methyl	6.86	0.06	0.09
Malathion	7.70	0.07	0.10
Parathion	8.30	0.06	0.09
Phosmet	11.09	0.08	0.11

CONCLUSIONS

The present multi-residue method, developed for the simultaneous determination of 8 insecticides in Chrysanthemum flower involves a rapid extraction procedure and a specific GC–FPD determination with satisfactory recoveries and LODs. The method described in this study has shown suitable sensitivity for monitoring a wide range of pesticide residues in Chrysanthemum flower.

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التثبت من (اختبار فاعلية) طريقة جديدة للمتبقيات العديدة لتقدير المبيدات الفسفورية العضوية الحشرية في زهرة الأبقوان باستخدام الكروماتوجرافي الغازي المجهز بالعمود الشعري
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إن عملية التثبيت هي عملية التوصل إلى خصائص الأداء وحدود طريقة الفحص والتعرف على المؤثرات التي يمكن أن تغير هذه الخصائص وإلى أي مدى. وتهدف عملية التثبيت من الفحص أو المعايرة إلى توفير ضمان لاعتمادية البيانات. ولا يمكن ضمان اعتمادية وإمكانية مقارنة البيانات إلا من خلال تنفيذ نظام لضمان الجودة بما في ذلك تطبيق أسلوب التثبيت من الطريقة وفقاً لإجراءات ومعايير للأداء مقبولة دولياً. عند تطوير طريقة جديدة، مثل هذه الطريقة نحتاج إلى عملية تثبت شاملة لتحديد مجالها ومحددات تطبيقها، ولتحديد خصائص الأداء ذات العلاقة، ولإظهار قوتها في التطبيق تحت ظروف مختلفة. في هذا البحث تم تطوير طريقة جديدة لتحليل ثمانية مبيدات فسفورية عضوية (OP) ذات مجال واسع في الخصائص الفيزيائية والكيميائية في زهرة الأبقوان والتحقق من صحتها. تتضمن الخطوات المتبعة استخلاص العينات المقواه (٥جم) باستخدام ١٠٠ مل أسيتون وعدم تعريض المستخلص النهائي لعملية التنقية. تم تحديد تركيزات المبيدات بواسطة جهاز الكروماتوجرافي الغازي طراز GC-9A المزود بكاشف الإشغال الضوئي (FPD) للكشف عن تركيزات المبيدات تحت الدراسة حيث كانت معدلات الاسترجاع لعينات الشاهد والمقواه بثلاث تركيزات للمبيدات المختلفة من ٩١.٠٠ - ٩٩.٦٧ % وبانحراف معياري نسبي اقل من ١٢.٠%. تراوحت حدود الكشف (LOD) للطريقة المستخدمة بين ٠.٠٦ و ٠.٠٨ ملجم كجم^{-١} واختلف الحد الكمي (LOQ) من ٠.٠٩ إلى ٠.١ ملجم كجم^{-١}. المحددات المختارة للتحقق من الطريقة المستخدمة (الدقة، الضباطة، المعايرة، الحساسية، الانتقائية، حد الكشف والحد الكمي) كانت ضمن الحدود المقبولة وفقاً لمعايير الاتحاد الأوروبي (SANCO) للمبيدات الحشرية الثمانية تحت الدراسة.