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Research Article

# Determination of pesticides residue in cannabis, cannabis extract and cannabis oil by gas chromatography tandem mass spectrometry technique

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## ABSTRACT

Pesticide residues analysis in cannabis has become a major interest in Thailand due to recent legalization and decriminalization of cannabis for medicinal uses since February 2019. To meet regulatory and quality control standards, cannabis raw materials and products should be tested for pesticides with action limits set by Thai Herbal Pharmacopeia. In this study, pesticides found in cannabis, cannabis extract, and cannabis oil samples submitted by government agencies have been reported. A quantitative and sample preparation method was established and validated following the EN 15662 QuEChERS for cannabis and EURL-FV (2012-M6) for cannabis extract and cannabis oil analysis. The identification of 122 pesticides in the sample was performed by scheduled selected reaction monitoring technique via GC-MS/MS using matrix-matched calibration curves. The method LOD and LOQ in 3 products were 0.03 and 0.05 mg/kg respectively with recoveries situated between 70% and 120%, and within laboratory relative standard deviations below 20%. According to the requirements of international pharmacopoeias and food regulatory agencies, the developed method was compiled since cannabis was found in both categories, as herbal remedies and foods. Overall, 8 pesticides belonging to different chemical classes were identified in 69 of 85 samples (81.2%) ranging from less than 0.05 to 77.5 mg/kg. The occurrence of most pesticides was exceeded the action limit. Generally, the samples of illegal cannabis contained toxic pesticides used in cultivation which were not safe for the production of cannabis-derived pharmaceutical drugs. Therefore, the strategic control for organic or medical-grade cannabis plantation should be enforced.

## 1. INTRODUCTION

Medicinal cannabis is a plant medicine in use for over 6000 years[1]. In Thailand, cannabis is prohibited under the Narcotic Drugs Act (1979), though the possession of marijuana and krathom (*Mitragyna speciosa*) within legal limits was allowed for treatment of certain diseases, for first aid or in cases of emergency since February 2019. Became the first South-East Asian country who legalized medical cannabis, the study in regard to cannabis has been a top priority for Thai government. In recent years, the therapeutic

use of medicinal cannabis has increased despite the limited number of clinical studies[2]. Over 500 compounds and more than 120 cannabinoids have been identified from cannabis plant material.

Cannabidiol (CBD) has been applied in the treatment and management of epilepsy[3], as an antipsychotic[4], in anxiety management[5], and as an analgesic and antioxidant[6]. On the other hand, tetrahydrocannabinol (THC), responsible for the psychoactive properties[7], has been approved to control nausea and vomiting in cancer treatments, appetite stimulation in AIDS patients[8] and in the treatment of glaucoma[9], migraine[10], anxiety, and as an analgesic.

Cannabis has been used in the region and Thailand has a long tradition with the use of cannabis. The plant was popular in the traditional Thai medicine for centuries. Since the legalization, medical cannabis can be prescribed to patients showing symptoms of some 38 conditions. According to the professionals of medicine who have permission to deliver cannabis derivative drugs, cannabis oils are the most common form of application, with pills and drops being alternatives. Moreover, traditional medicine authorities have registered 16 cannabis-based medical formulas[11-12] for production and application approval. The cannabis-laced traditional medicine is used to treat certain conditions such as pain, insomnia, hemorrhoids, mental illness[13] and skin disease[14] and to boost health and appetite for patients in cancer treatments.

With the high demand for cannabis for pharmaceutical industry[15], the quality and safety of cannabis and its related products have become a major concern for consumers[16]. The Department of Medical Sciences, Ministry of Public Health has issued the Thai Herbal Pharmacopoeia to be the guidelines for ensuring the quality, safety, and efficacy of medicinal herbs including cannabis. The guidelines require control of contaminants including pesticides, toxic elements, mycotoxins, and pathogens, as well as residual solvents in regard to cannabis oils. Accordingly, appropriate analytical methods are required to determine these contaminants in cannabis and cannabis products for quality control.

In this work, the main focus was on the analytical challenges and method development for pesticide residue detection in cannabis and cannabis product samples in order to meet the various guidelines[17]. The international reference method was improved to eliminate matrix interferences highly presented in cannabis[18]. The aim of the work was to find a suitable, reliable, and accurate method for routine analysis. The final selected methodology was fully validated and applied to routine received samples to check the compliance of products with the national and international regulation.

## 2. MATERIALS AND METHODS

### 2.1. Chemicals, materials and standards

Acetonitrile (HPLC), acetone (HPLC), ethyl acetate (HPLC), toluene (ACS.) and glacial acetic acid (AR) were purchased from J.T. Baker, USA. N-hexane (PG) was supplied by RCILabscan, Thailand. QuEChERS extract pouch, EN method containing 4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate and 0.5 g disodium hydrogenate sesquihydrate (p/n 5982-0650) and dispersive SPE 2 ml, Fat + Pigments, AOAC: A mixture of 50 mg PSA, 50 mg C18EC, 50 mg GCB and 150 mg $MgSO_4$, Agilent Bond Elut (p/n 5982-5421) and were from Agilent Technologies, USA. D-sorbitol, purity ≥ 98% was provided by Merck KGaA, Germany. Reverse osmosis water was generated by Millipore Milli-Q system, USA.

Certified reference material, CRM of pesticides standards mixture (kit of 122 components) with certified value and uncertainty approximately 100±3 µg/ml were purchased from C.P.A. Chem Ltd., France. The CRM was produced by gravimetric measurement and dissolving individually and mixed solution (solution of 10 µg/ml approximately) from each pesticide standard and working solution (1 µg/ml) were prepared in acetone and stored in amber screw-capped glass vials in the dark at -20 °C. Matrix matched calibration curve of standard (5, 10, 20, 50, 100, 250 and 500 ng/ml) was freshly prepared before use by serial dilution of stock solution to the appropriate concentrations and matrices. The residue concentrations were calculated using the calibration curve generated from the peak area response versus the working solution concentrations.

### 2.2. Instrumentation

An analytical method was developed for the Thermo Scientific TRACE™ 1300 Gas Chromatograph and the TSQ 9000, Triple Quadrupole Mass Spectrometer. The GC-MS/MS system was equipped with PAL RTC autosampler using TraceFinder 4,1 EFS software. The analytical conditions of the GC-MS/MS method are provided in Table 1. The MS system was

**Table 1.** Instrument condition.

| TRACE 1300 GC | | TSQ 9000 MS | |
|---|---|---|---|
| Injection Volume: | 2 µl | Source Temp.: | 280 ˚C |
| Injector: | PTV Temp. 80 ˚C 0.1 min. 5 ˚C/s to 300 ˚C. | Emission Current: | 50 µA |
| Carrier Gas: | He, constant pressure, 15 psi | Ionization Mode: | EI, 70 eV |
| Column Type: | DB-5MS 20 m, 0.25 mm ID, 0.25 µm df | Collision Gas: | Argon |
| Column Oven: | Initial 70 ˚C, hold 1 min. Ramp 50 ˚C/min to 150 ˚C. Ramp 6 ˚C/min to 200 ˚C. Ramp 16 ˚C/min to 280 ˚C. Hold 8.5 min. Ramp 50 ˚C/min to 300 ˚C. Hold 0.5 min. | Cycle time: | 30 min |
| Transfer Line: | 280 ˚C | Acquisition Mode: | Timed-SRM |

operated in electron ionization mode (EI, 70 eV). The analytes were separated in a fused silica capillary column DB-5ms (30 m x 0.25 mm i.d., 0.25 µm film thickness) from Agilent.

The column oven temperature was programmed as follows: an initial temperature of 70 °C was held for 1 min, then increased by 50 °C/min to 150 °C and then increased by 6 °C/min to 200 °C and then increased by 16 °C/min to 280 °C and was held for 8.5 min. The oven temperature was increased by 50 °C/min to final temperature at 300 °C and was held for 0.5 min. The total run time was 25 min. The injection volume was 2 µl using programmable temperature vaporizing (PTV) as an injector. The PTV temperature was set at 80 °C and held for 0.1 min then was increased by 5 °C/s to 300 °C. The purge $N_2$ gas flow was set at 30 ml/min. By using the Thermo AutoSRM software, the two most intense transitions and their optimal collision energies were selected. The most intense product was selected as the quantifier ion and the second most intense was set as the qualifier ion. Depending on the type of matrix interference, alternatives transition could be selected for quantitative and qualitative ions. The settings on the mass spectrometry detector, including the retention time (RT), quantitative peak, confirmation peak and CE are reported.

## 2.3. Sample and sample preparations

### *2.3.1. Cannabis*

A sample blank of dried cannabis (~10% moisture w/w) was obtained from The Office of Narcotics Control Board, Thailand. The sample was homogenized into a powder using a cryogenic blender (POWTEQ HM100, China) and passed through a 2-mm sieve as a blank sample to optimize and validate the method. The blank sample was screened for pesticide residues before method validation. Samples (1 g) were weighed into 50 ml PTFE centrifuge tubes and spiked with appropriate amount of standard solution (10 µg/ml). Then, 9 ml of Milli Q water was added to hydrate for 30 min. After when, 10 ml of MeCN and ceramic homogenizer was added into the tube. Solutions were vortexed for 30 seconds and shaken manually for 1 min. The QuEChERS extraction kit (p/n 5982-0650) was added directly to the tube, which was shaken immediately for 1 min to prevent coagulation of $MgSO_4$. After centrifugation at 4000 rpm, room temperature for 5 min (HERMLE Z366, Germany), the upper ACN layer (1.3-1.5 ml) was transferred to and purified in a dispersive SPE 2 ml centrifuge tube containing $MgSO_4$, GCB, PSA and C18EC. Then, the centrifuge tube was shaken for 30 seconds and was placed immediately into centrifuge (4000 rpm, 5 min, at room temperature). The extract (1 ml) was dried under gentle $N_2$ gas and the residue re-dissolved with 1 ml n-hexane:EtOAc, 3:1 and vortexed for 1 min. For the GC-MS/MS analysis, 3 µl an analytical protectant (D-sorbitol, 3%) solution was added before GCinjection to obtain good signal and peak shape.

### *2.3.2. Sesame oil*

The cannabis extract and cannabis oil are non-polar and high fatty sample and were generally prepared with edible oil (sesame oil, coconut oil, etc.) with 1-3% concentration of pure extract. Pesticide free sesame oil was purchased from supermarket and was selected to be representative matrix for method validation. The samples were shaken for homogenization and were stored at room temperature before analysis. As sesame oil is a processed product sample, it should be analyzed within the stated shelf life. Homogenous sample (1 g) was transferred into a 50 ml centrifuge tube and was dissolved by 5 ml n-hexane. Ten milliliters of acetonitrile saturated with n-hexane were added, and the tube was closed tidily and was shaken vigorously at 80 rpm

for 20 min (JISICO, Korea). The tube was centrifuged for 5 min at 4000 rpm and was left for 5 min longer. An aliquot of about 6-8 ml of under layer was transferred into 15 ml capped graduated tube and placed into freezer for at least 1 hour.

The separation of the frozen co-extractives (frozen lipids) was operated immediately by filtration through a clean cotton wool into a 15 ml screw capped centrifuge tube. Approximately 1.5 ml of ACN extract solution was purified using the same dispersive SPE and analytical procedure than cannabis sample preparation. Final extract was transferred into auto-sampler vials with addition of analytical protectant to be used for gas chromatographic analysis.

*2.3.3. Samples analyzed*

In this study, pesticide residues in cannabis, cannabis extract and cannabis oil samples submitted by government agencies have been analyzed. The validated method was applied to the routine received samples. A total of 85 samples have been analyzed during 2019-2020 including 77 dried cannabis samples, 7 cannabis extracts and 1 cannabis oil. The samples were submitted by government units mainly from the Office of Narcotic Control Board. Some samples were delivered from intra-department such as the Medicinal Plant Research Institute and the Bureau of Drugs and Narcotic. Several samples were sent from departments and hospitals in the Ministry of Public Health such as the Department of Medical Services, the Department of Thai Traditional and Alternative Medicine, Chao Phya Abhaibhubejhr Hospital and Pra Ajarn Fan Acharo Hospital, as well as the Government Pharmaceutical Organization (GPO) (Table 2.).

## 2.4. Method validation

The method was developed and validated in accordance with the European standard (SANTE/12682/2019), which determined the limit of detection (LOD), limit of quantification (LOQ),

**Table 2.** Number of analyzed sample submitted by government agencies during 2019.

| Agency | Numbers of sample |
|---|---|
| Office of narcotics control board | 52 |
| The Government pharmaceutical organization, GPO | 13 |
| Medicinal plant research institute, Department of medical sciences | 12 |
| Bureau of drug and narcotic, Department of medical sciences | 3 |
| Chao Phya Abhaibhubejhr Hospital | 1 |
| Phra Achan Fan Acharo Hospital | 1 |
| Princess mother national institute on drug abuse treatment, Department of medical services | 1 |
| Buri Ram provincial health office | 1 |
| Herb and Thai traditional medicine development division, Department of Thai traditional and alternative medicine | 1 |

and linearity of the calibration curve. Matrix-matched calibration curve using pesticide-free extract was performed to compensate for matrix effects and minimize quantification errors. Calibration curves were obtained by spiking standards, ranging from 5 to 500 ng/ml, into blank matrix extract solutions and analytical protectant was added prior to injection. Accuracy, expressed as a percentage of recovery and precision were determined based on 10 replicated samples spiked at 0.05, 0.2, and 1 mg/kg. The LOQs were evaluated by determining the lowest concentration spike for samples where accuracy and repeatability were satisfactory (within 70–120% and less than 20%, respectively) (SANTE/12682/2019). The estimation of LOD was calculated from the variability of the blank signals read from the calibration curve. The standard deviation (SD) of blank amount was used for determining the LODs which LOD was 3xSD. The single analytical LOD of all pesticides was selected to facilitate the method application in routine work and should be above all LODs calculated. Generally, LODs were estimated as one third or half of the LOQs.

Compound identification and confirmation of 122 pesticides when samples were analyzed, in all cases, the results were performed according to SANTE (DG-SANTE, 2019) guidelines for each analytical instrument. Firstly, same retention time as the standard (±0.1 min), secondly for GC-MS/MS, 2 product ions analyte

peaks in the extracted ion chromatograms must fully overlap and finally in all cases, ion ratios from sample extracts should be relatively within ±30% of the average of calibration standards from the same sequence.

## 3. RESULTS AND DISCUSSION

### 3.1. Method development

An analytical method was based on EN 15662 QuEChERS for cannabis and modified QuEChERS EURL-FV (2012-M6) for cannabis extract and cannabis oil. The list of pesticides of interest was selected mainly by Thai Herbal Pharmacopeia, THP 2019 and Food Act B.E.2522 (No. 387) B.E. 2560 (2017) Re: Food Containing Pesticide Residues (Pesticide Residues in Food). A total of 122 pesticides were the representative group of GC amenable insecticides, herbicides, acaricides, nematocides, fungicides, and plant growth regulators belonging to different chemical families (organophosphates, carbamates, pyrethroids, neonicotinoids, etc.). The QuEChERS extract solution was cleaned up by using variations of salts/materials with potential capability to eliminate most of the coextracted metabolites from the matrix. The diversity of dispersive solid-phase extraction salts (dispersive SPE) were tested for the best clean up property. The dispersive SPE chosen included primary and secondary amine (PSA) aiming at the removal of organic acids and also showed high specificity towards some polyphenols and other glycosides. Graphitized carbon black (GCB) was used to reduce the content of chlorophylls and pigments and C18EC was added to minimize the lipophilic non-polar compounds and waxes from the cannabis and cannabis product samples. For fatty samples, freezing out (for removal of lipids, waxes, sugars, and other matrix co-extractives with low solubility in acetonitrile) was required where-with the major part of fat and waxes precipitated. Since the precipitates were not separated by decantation, they may be separated either by a quick centrifugation followed by filtering the still cold extract through a piece of cotton wool. The extract could be used for further cleanup by dispersive SPE. The total ion chromatogram (TIC) of 122 pesticides in GC-MS/MS under SRM mode was showed in Figure 1. and Figure 2. Displaying eight examples of the chromatogram, ion overlay, quantitation and confirmation ion and calibration curves for pesticides detected in real samples at 0.03 mg/kg concentration level. The choice of solvent for needle wash, toluene/n-hexane, to avoid the plunger of the syringes to become stuck or jammed, was important because cannabis extract contains viscous elements.

### 3.2. Method validation

#### *3.2.1. LOD* and *LOQ*

The LOD was evaluated as the lowest concentration that can be determined to be statistically different from a blank.

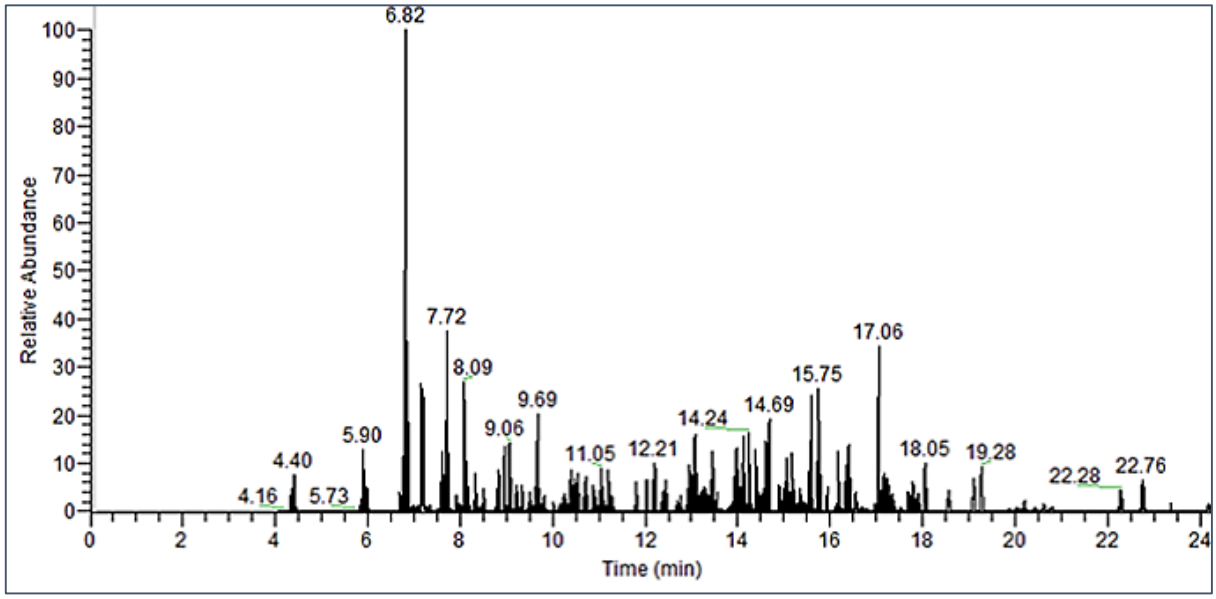


**Figure 1.** The total ion chromatogram (TIC) of the target pesticides in GC-MS/MS under selected reaction monitoring (SRM) mode.

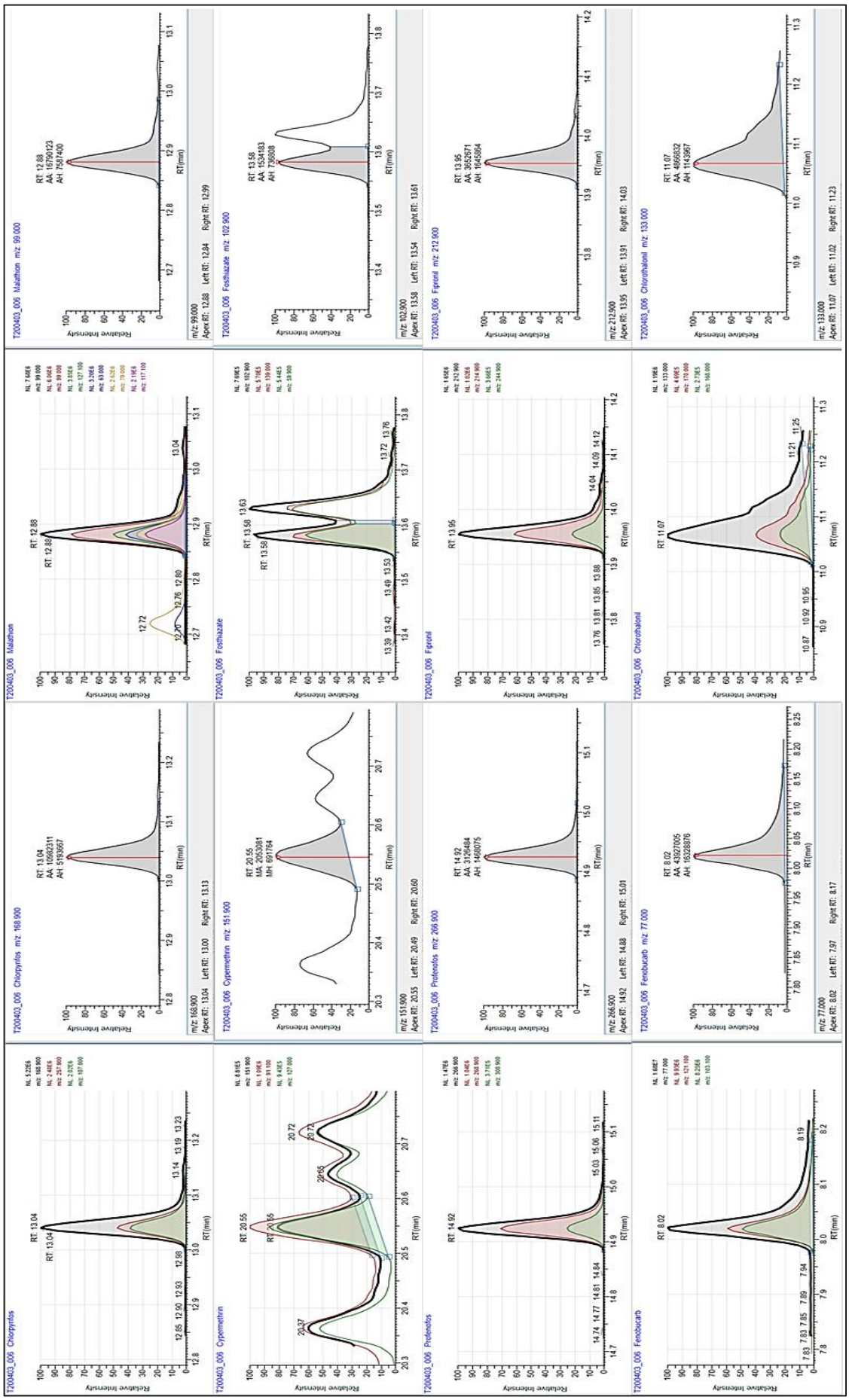

**Figure 2.** Matrix matched chromatogram, ion overlay including quantitation and confirmation ion and RT for chlorpyrifos, cypermethrin, profenofos, fenobucarb, malathion, fosthiazate, fipronil and chlorothalonilat 0.03 mg/kg concentration level.

This concentration was calculated by three standard deviations of 10 replicate injections of blank extract. The injection of analytes at 0.03 mg/kg gave a signal to noise ratio greater than 3 for all compounds and the method detection limit (MLD) at this level was confirmed by showing detected for all pesticides. The LOQ for each analyte was the lowest concentration at which the identification criteria were met, according to the SANTE guidance document. The lowest concentration level validated using matrix-matched calibrations was 0.05 mg/kg

*3.2.3. Accuracy and precision*

For the validation purposes, the total of 122 compounds were validated for analysis by GC-MS/MS. Percentages of recovery and %RSD at three levels, at LOQ, 4xLOQ and 20xLOQ, for each pesticide under this study are shown in Table 3. Relative standard deviations (%RSD) were less than 20% and recoveries were between 70% and 120% for all pesticides showing accuracy and precision within the SANTE accepted boundaries.

*3.2.4. Sample analyzed results*

The validated method was applied to the simultaneous determination of 122 pesticide residues and the level of analytes concentration in 85 samples submitted by government agencies during 2019-2020. As can be seen in Table 4. The results showed only 8 out of 122 pesticides can be detected. Overall, pesticides were identified in 69 (81.2%) of 85 samples ranging from less than 0.05 to 77.5 mg/kg. In general, each positive sample contained at least one of the studied pesticides. For a set of tested cannabis and cannabis product samples, the most commonly detected pesticides were chlorpyrifos, cypermethrin and profenofos. The overall detection rates of these three pesticides were high, and multi-pesticides (up to 5) were detected in one cannabis sample. The main reason for the high detection rates of pesticides in samples may be attributed to the abuse of a variety of mixed pesticides by illegal growers in surrounding countries of Thailand in order to increase the amount of high-value products. The lack of good agricultural practice (GAP) due to unauthorized production in hidden areas, and the lack of regulatory control from authority led to toxic cannabis disseminated to consumers. Most of these samples could not be used for the production of cannabis-derived pharmaceutical drugs. For this reason, the strategic control for organic or medical-grade cannabis plantation should be enforced. The cannabis and cannabis products should be regularly monitored for multiresidue pesticides, and more stringent management and regulation of pesticides need to be implemented in future cannabis production.

*3.2.5. Estimation of measurement uncertainty*

Although, it is especially difficult to determine uncertainties for multiresidue methods, the estimation of uncertainty of an analytical result is a one of the ISO/IEC: 17025 requirements. In this study, the bottom-up approach (EURACHEM/CITAC guide Quantifying Uncertainty in Analytical Measurements) was followed to estimate the expanded uncertainties for 122 pesticides. Identification of the sources of uncertainty was performed and an uncertainty budget was assessed by the measurement functions. The uncertainties of each element were determined using validation data in the estimation and combined uncertainty.

Taking an example of the estimation of Measurement uncertainty (MU) of chlopyrifos found in cannabis and sesame oil samples at the level of 0.05 μg/kg, the sample weight, the sample taken, the final volume of extract, the $C_0$, the standard concentration, the method precision, and the recovery were uncertainty sources. The largest sources were from $C_0$, sample weight and method precision, respectively (Figure 3.). The uncertainty of the sample taken and the final volume of the sample was minimal in both cases. The reported uncertainty in an expanded uncertainty calculated using a coverage factor of 2 which gave a level of confidence of approximately 95% showed 0.050±0.011 mg/kg and 0.050±0.005 mg/kg of uncertainties which were 22% and 10% of reported results. The laboratory has demonstrated that the expanded MU is not exceeding the 50% default value used by the regulatory authorities for enforcement decisions.

## 4. CONCLUSIONS

A sample preparation method for the multiresidue pesticides analysis in cannabis

**Table 3.** Percentage of recovery (%), relative standard deviation (%RSD) for the validated method for each analyte by GC-MS/MS system in cannabis and sesame oil.

| Pesticide | Cannabis (mg/kg) | | | | | | Sesame oil (mg/kg) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0.05 | | 0.2 | | 1 | | 0.05 | | 0.2 | | 1 | |
| | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD |
| DDD-p,p′ | 92.5 | 1.7 | 94.1 | 7.5 | 94.1 | 6.1 | 106.6 | 2.71 | 94.8 | 3.06 | 102.1 | 5.94 |
| DDE-p,p′ | 102.4 | 2.3 | 103.6 | 5.4 | 98.7 | 5.1 | 105.3 | 3.63 | 70.8 | 3.08 | 111.2 | 7.15 |
| DDT-p,p′ | 99.5 | 5.9 | 95.9 | 5.6 | 91.8 | 14.8 | 107.9 | 3.31 | 95.9 | 2.00 | 102.3 | 5.93 |
| Acephate | 77.9 | 8.2 | 83.7 | 9.1 | 95.2 | 9.1 | 112.5 | 4.22 | 114.3 | 13.02 | 113.2 | 13.46 |
| Alachlor | 105.7 | 4.2 | 101.4 | 6.0 | 98.5 | 5.0 | 94.3 | 12.55 | 73.4 | 13.08 | 104.6 | 9.90 |
| Aldrin | 104.2 | 4.9 | 104.5 | 4.4 | 97.9 | 5.0 | 102.8 | 3.76 | 60.3 | 5.48 | 112.4 | 10.82 |
| Ametryn | 105.4 | 5.1 | 101.4 | 6.6 | 100.3 | 5.2 | 113.5 | 11.23 | 65.3 | 11.12 | 113.2 | 8.00 |
| Atrazine | 101.2 | 6.9 | 102.1 | 7.4 | 98.1 | 5.5 | 106.0 | 10.85 | 87.8 | 9.01 | 111.0 | 3.35 |
| Azinphos-ethyl | 95.2 | 6.6 | 92.3 | 12.9 | 96.4 | 13.3 | 112.0 | 5.79 | 116.4 | 2.95 | 83.8 | 6.34 |
| Azinphos-methyl | 84.5 | 8.8 | 99.9 | 6.3 | 98.6 | 14.8 | 105.7 | 17.50 | 114.4 | 2.49 | 81.9 | 10.26 |
| Bendiocarb | 103.3 | 4.5 | 97.1 | 9.2 | 99.6 | 11.2 | 109.1 | 9.05 | 81.3 | 6.08 | 116.4 | 8.36 |
| BHC, Alpha | 107.4 | 4.8 | 104.1 | 6.2 | 96.4 | 5.6 | 103.0 | 2.02 | 61.1 | 6.98 | 116.9 | 10.69 |
| BHC, Beta | 99.6 | 3.3 | 100.3 | 8.9 | 96.4 | 6.2 | 102.2 | 6.64 | 84.9 | 6.41 | 103.6 | 16.93 |
| BHC, delta | 98.6 | 4.8 | 95.4 | 9.0 | 99.8 | 7.4 | 119.5 | 15.39 | 68.8 | 19.72 | 114.7 | 8.73 |
| BHC, gamma | 99.6 | 3.3 | 99.0 | 8.9 | 96.4 | 6.2 | 102.2 | 6.64 | 82.8 | 13.29 | 101.9 | 12.14 |
| Bifenazate | 106.6 | 7.8 | 104.8 | 7.1 | 100.5 | 7.6 | 101.9 | 17.64 | 86.7 | 14.35 | 110.2 | 8.37 |
| Bifenthrin | 106.5 | 4.8 | 104.4 | 5.5 | 98.6 | 4.7 | 101.5 | 3.23 | 69.4 | 3.47 | 114.2 | 8.19 |
| Bromacil | 103.0 | 4.3 | 97.9 | 9.2 | 101.2 | 5.3 | 107.1 | 7.47 | 85.3 | 10.76 | 99.3 | 6.78 |
| Bromophos-ethyl | 105.7 | 5.3 | 100.6 | 7.0 | 99.0 | 5.6 | 109.6 | 6.01 | 102.2 | 2.40 | 113.2 | 9.41 |
| Bromopropylate | 103.2 | 4.3 | 103.8 | 5.5 | 98.6 | 4.6 | 104.7 | 4.70 | 85.8 | 3.07 | 99.7 | 5.82 |
| Buprofezin | 97.8 | 4.7 | 103.5 | 6.4 | 97.1 | 5.0 | 115.7 | 10.72 | 100.3 | 8.76 | 105.3 | 5.37 |
| Butachlor | 108.7 | 5.2 | 102.1 | 5.9 | 98.0 | 5.4 | 115.7 | 4.51 | 74.8 | 9.04 | 114.2 | 6.03 |
| Cadusafos | 100.3 | 4.1 | 100.3 | 8.1 | 100.7 | 5.9 | 112.3 | 4.11 | 63.8 | 6.02 | 112.7 | 8.96 |
| Carboxin | 104.2 | 5.2 | 99.4 | 7.8 | 96.0 | 6.3 | 94.2 | 18.62 | 93.1 | 0.06 | 107.9 | 2.53 |
| Chlordane-alpha | 106.1 | 4.7 | 102.3 | 5.5 | 97.1 | 4.4 | 100.9 | 3.49 | 91.2 | 3.01 | 119.1 | 10.75 |
| Chlordane-gamma | 101.2 | 3.6 | 103.3 | 5.5 | 96.6 | 4.9 | 104.5 | 4.04 | 94.2 | 3.69 | 115.8 | 10.32 |
| Chlordane (Oxy) | 99.3 | 6.9 | 101.2 | 5.7 | 95.3 | 4.9 | 107.4 | 5.52 | 75.9 | 6.50 | 110.3 | 5.54 |
| Chlorfenapyr | 104.8 | 3.8 | 102.3 | 7.2 | 98.8 | 5.0 | 113.5 | 4.25 | 102.1 | 5.20 | 98.4 | 6.60 |
| Chlorfenvinphos | 106.2 | 4.6 | 102.8 | 8.0 | 97.0 | 6.0 | 108.0 | 5.25 | 89.9 | 3.57 | 112.0 | 6.07 |
| Chlorobenzilate | 104.9 | 5.3 | 104.1 | 5.8 | 99.2 | 5.2 | 108.8 | 4.08 | 109.0 | 2.36 | 99.5 | 4.81 |
| Chloroneb | 106.3 | 4.9 | 100.3 | 5.8 | 99.6 | 6.0 | 95.1 | 12.85 | 68.9 | 12.91 | 108.1 | 10.97 |
| Chlorothalonil | 90.9 | 7.6 | 91.6 | 11.8 | 99.5 | 13.4 | 109.6 | 6.25 | 76.7 | 6.29 | 102.4 | 8.17 |

**Table 3.** Percentage of recovery (%), relative standard deviation (%RSD) for the validated method for each analyte by GC-MS/MS system in cannabis and sesame oil. (cont.)

| Pesticide | Cannabis (mg/kg) | | | | | | Sesame oil (mg/kg) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0.05 | | 0.2 | | 1 | | 0.05 | | 0.2 | | 1 | |
| | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD |
| Chlorpropham | 100.0 | 7.7 | 99.8 | 7.3 | 100.1 | 8.3 | 111.9 | 17.14 | 76.7 | 14.48 | 97.2 | 13.73 |
| Chlorpyrifos | 101.7 | 4.9 | 103.2 | 6.2 | 99.0 | 5.9 | 104.7 | 2.23 | 61.6 | 5.16 | 113.1 | 8.20 |
| Chlorpyrifos-methyl | 102.8 | 4.4 | 93.6 | 11.6 | 97.6 | 8.5 | 103.1 | 7.88 | 84.5 | 2.90 | 104.4 | 5.94 |
| Chlorthal-dimethyl | 101.7 | 3.6 | 101.3 | 6.7 | 99.0 | 5.8 | 107.4 | 1.79 | 61.8 | 12.88 | 115.0 | 9.76 |
| Cyanophos | 97.0 | 5.8 | 92.2 | 10.2 | 95.0 | 12.3 | 101.2 | 11.68 | 75.2 | 9.64 | 106.9 | 6.22 |
| Cyfluthrin | 104.1 | 5.6 | 103.3 | 9.4 | 103.8 | 7.7 | 96.9 | 10.70 | 115.0 | 6.80 | 81.0 | 8.00 |
| Cyhalothrin, lambda | 104.5 | 4.5 | 102.9 | 5.8 | 98.2 | 4.8 | 106.1 | 7.32 | 87.7 | 2.99 | 98.4 | 10.24 |
| Cypermethrin | 108.9 | 6.1 | 103.3 | 6.6 | 99.0 | 5.1 | 101.7 | 13.80 | 98.0 | 16.10 | 86.9 | 13.34 |
| DEET | 109.2 | 5.5 | 102.6 | 5.8 | 100.1 | 5.7 | 109.7 | 5.56 | 86.3 | 3.16 | 108.5 | 6.74 |
| Deltamethrin | 111.1 | 5.4 | 99.6 | 8.2 | 96.9 | 6.1 | 99.2 | 15.53 | 103.3 | 2.02 | 85.2 | 5.33 |
| Demeton-S-methyl | 95.8 | 6.3 | 90.7 | 9.2 | 94.9 | 12.3 | 104.3 | 3.98 | 113.8 | 0.39 | 99.8 | 19.26 |
| Diazinon | 106.6 | 4.4 | 102.0 | 6.2 | 98.8 | 5.0 | 99.6 | 3.51 | 63.7 | 19.41 | 106.0 | 16.44 |
| Dichlorvos | 104.9 | 3.0 | 99.0 | 6.9 | 99.5 | 6.7 | 113.0 | 4.02 | 70.1 | 17.94 | 110.1 | 7.30 |
| Dicofol | 106.9 | 5.2 | 100.7 | 6.9 | 96.7 | 5.1 | 105.8 | 8.04 | 83.3 | 17.83 | 94.0 | 5.23 |
| Dicrotophos | 88.2 | 9.2 | 88.5 | 12.9 | 100.8 | 9.0 | 111.4 | 17.91 | 117.0 | 9.90 | 85.7 | 17.48 |
| Dieldrin | 100.6 | 4.9 | 101.8 | 5.0 | 96.6 | 5.2 | 115.2 | 5.86 | 94.3 | 15.67 | 115.5 | 8.35 |
| Dimethoate | 84.8 | 9.1 | 89.0 | 13.8 | 99.9 | 6.7 | 107.4 | 12.48 | 89.3 | 19.73 | 94.7 | 7.11 |
| Dioxathion | 110.1 | 6.0 | 107.7 | 8.5 | 100.7 | 7.7 | 111.7 | 9.67 | 81.4 | 13.79 | 119.4 | 11.96 |
| Disulfoton | 103.3 | 4.6 | 102.1 | 6.5 | 98.6 | 5.1 | 102.5 | 6.83 | 113.3 | 0.50 | 99.8 | 19.19 |
| Ditalimfos | 100.8 | 4.7 | 97.3 | 10.5 | 97.7 | 7.2 | 103.5 | 4.60 | 97.5 | 5.64 | 105.7 | 5.22 |
| Endosulfan I | 103.7 | 3.8 | 102.0 | 4.7 | 97.4 | 4.6 | 111.5 | 8.19 | 74.2 | 17.82 | 115.1 | 10.25 |
| Endosulfan II | 106.0 | 6.1 | 99.4 | 5.1 | 99.6 | 4.4 | 101.8 | 9.46 | 96.4 | 6.85 | 102.8 | 5.32 |
| Endosulfan sulfate | 99.6 | 3.8 | 98.0 | 7.5 | 95.1 | 5.6 | 96.0 | 6.66 | 91.1 | 4.74 | 101.2 | 7.21 |
| Endrin | 96.6 | 4.7 | 92.7 | 7.1 | 94.1 | 6.1 | 97.4 | 13.41 | 85.3 | 4.42 | 117.1 | 11.60 |
| EPN | 99.5 | 4.5 | 95.8 | 9.5 | 100.9 | 6.7 | 106.8 | 7.04 | 115.1 | 2.80 | 90.0 | 4.07 |
| Ethion | 107.1 | 4.7 | 104.4 | 7.8 | 100.1 | 5.6 | 110.8 | 3.75 | 114.8 | 1.20 | 94.5 | 4.04 |
| Ethoprophos | 107.7 | 4.7 | 103.8 | 6.3 | 101.6 | 6.2 | 111.0 | 5.05 | 68.4 | 8.88 | 105.9 | 6.49 |
| Etrimfos | 99.3 | 4.1 | 98.5 | 9.0 | 95.7 | 6.9 | 106.2 | 3.74 | 60.4 | 8.81 | 112.1 | 8.82 |
| Fenchlorfos | 98.1 | 5.1 | 94.9 | 11.2 | 98.2 | 7.9 | 105.3 | 5.31 | 67.9 | 6.85 | 108.2 | 10.39 |
| Fenitrothion | 92.2 | 4.7 | 92.7 | 11.4 | 101.3 | 10.4 | 96.8 | 12.07 | 82.9 | 5.98 | 97.5 | 8.04 |
| Fenobucarb | 103.2 | 4.1 | 100.8 | 6.5 | 100.7 | 6.0 | 91.9 | 6.74 | 86.2 | 3.69 | 105.9 | 8.04 |
| Fenpropathrin | 98.7 | 4.0 | 102.5 | 5.5 | 97.7 | 6.7 | 97.4 | 6.43 | 82.0 | 3.54 | 104.5 | 7.10 |

**Table 3.** Percentage of recovery (%), relative standard deviation (%RSD) for the validated method for each analyte by GC-MS/MS system in cannabis and sesame oil. (cont.)

| Pesticide | Cannabis (mg/kg) | | | | | | Sesame oil (mg/kg) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0.05 | | 0.2 | | 1 | | 0.05 | | 0.2 | | 1 | |
| | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD |
| Fenthion | 97.6 | 5.0 | 98.3 | 8.9 | 99.5 | 6.9 | 108.9 | 7.59 | 85.6 | 6.45 | 92.0 | 3.96 |
| Fenvalerate | 107.7 | 5.0 | 102.1 | 6.4 | 97.1 | 7.2 | 106.8 | 13.60 | 114.2 | 14.60 | 79.4 | 7.10 |
| Fipronil | 104.1 | 5.0 | 101.6 | 7.2 | 99.1 | 4.7 | 109.9 | 8.38 | 110.2 | 2.01 | 89.2 | 5.89 |
| Folpet | 101.1 | 4.2 | 100.9 | 8.9 | 99.8 | 7.5 | 107.4 | 5.37 | 103.5 | 3.16 | 101.7 | 5.30 |
| Fosthiazate | 91.2 | 5.4 | 91.7 | 11.6 | 85.6 | 13.4 | 101.9 | 11.30 | 70.4 | 8.60 | 97.1 | 17.29 |
| Heptachlor | 83.1 | 6.2 | 88.6 | 15.1 | 88.8 | 7.8 | 101.6 | 1.74 | 63.4 | 4.09 | 109.5 | 10.40 |
| Heptachlor epoxide, cis | 95.8 | 4.0 | 100.2 | 5.8 | 97.1 | 4.3 | 104.4 | 6.02 | 60.5 | 5.97 | 112.9 | 9.16 |
| Heptachlor epoxide, trans | 104.5 | 5.1 | 103.5 | 4.6 | 97.2 | 4.3 | 100.0 | 3.27 | 61.4 | 18.94 | 110.4 | 10.27 |
| Heptenophos | 98.6 | 4.3 | 95.2 | 10.8 | 100.1 | 8.5 | 108.4 | 3.66 | 86.2 | 3.68 | 106.1 | 6.17 |
| Hexachlorobenzene | 109.8 | 5.3 | 102.2 | 4.8 | 100.3 | 4.3 | 103.5 | 7.75 | 96.4 | 5.99 | 118.3 | 12.87 |
| Hexazinone | 106.5 | 5.5 | 103.0 | 6.5 | 98.4 | 5.5 | 101.6 | 6.19 | 102.7 | 3.77 | 110.0 | 8.04 |
| Isofenphos | 102.6 | 4.4 | 103.9 | 5.8 | 97.9 | 4.8 | 110.9 | 4.79 | 102.5 | 3.24 | 116.4 | 7.78 |
| Isoprocarb | 109.9 | 4.3 | 101.8 | 7.4 | 99.6 | 6.0 | 100.4 | 4.56 | 76.3 | 4.72 | 106.3 | 7.54 |
| Isoxathion | 97.3 | 3.2 | 89.9 | 8.7 | 96.5 | 10.3 | 116.5 | 10.08 | 102.7 | 2.59 | 79.2 | 4.29 |
| Malathion | 98.8 | 4.6 | 96.9 | 11.5 | 99.7 | 8.0 | 111.0 | 4.91 | 62.9 | 4.30 | 116.8 | 5.20 |
| Metalaxyl | 109.4 | 5.4 | 101.0 | 6.8 | 98.2 | 5.0 | 105.5 | 2.61 | 70.7 | 18.62 | 107.1 | 9.77 |
| Methacrifos | 102.2 | 4.2 | 97.8 | 8.0 | 99.3 | 7.8 | 107.9 | 7.28 | 90.8 | 6.81 | 118.9 | 10.08 |
| Methamidophos | 102.0 | 10.5 | 91.0 | 10.1 | 96.9 | 14.1 | 102.1 | 15.88 | 85.2 | 17.03 | 98.1 | 6.34 |
| Methidathion | 85.7 | 7.3 | 88.2 | 13.3 | 100.7 | 8.9 | 97.4 | 17.63 | 102.9 | 14.29 | 104.0 | 8.27 |
| Methoxychlor | 92.1 | 14.9 | 94.9 | 4.6 | 99.6 | 9.6 | 102.0 | 4.78 | 85.9 | 2.22 | 100.5 | 6.73 |
| Metolachlor | 103.1 | 7.1 | 101.0 | 7.2 | 99.3 | 4.9 | 108.6 | 2.01 | 60.8 | 11.18 | 117.6 | 8.36 |
| Metribuzin | 109.6 | 7.5 | 103.1 | 6.2 | 92.9 | 8.7 | 107.8 | 6.16 | 76.5 | 6.80 | 109.3 | 4.72 |
| Mevinphos | 88.9 | 8.6 | 90.4 | 12.8 | 99.4 | 13.6 | 112.0 | 4.33 | 79.3 | 6.85 | 109.7 | 7.25 |
| Monocrotophos | 79.6 | 11.9 | 96.9 | 3.9 | 98.7 | 11.7 | 102.4 | 11.35 | 119.0 | 10.93 | 95.9 | 15.09 |
| Naled | 79.0 | 9.9 | 96.1 | 6.7 | 97.3 | 11.7 | 101.3 | 16.89 | 107.4 | 12.52 | 116.7 | 14.41 |
| Omethoate | 92.1 | 13.8 | 92.2 | 9.5 | 96.5 | 14.2 | 97.6 | 17.88 | 102.4 | 14.03 | 94.9 | 13.15 |
| Parathion | 104.4 | 5.9 | 94.3 | 9.4 | 100.1 | 7.1 | 104.5 | 13.63 | 100.6 | 2.36 | 93.3 | 4.47 |
| Parathion-methyl | 90.6 | 7.1 | 90.1 | 13.3 | 98.8 | 13.2 | 93.9 | 17.69 | 87.9 | 16.04 | 94.6 | 12.40 |
| Permethrin | 105.8 | 5.3 | 100.8 | 6.0 | 108.7 | 7.5 | 104.3 | 8.70 | 87.3 | 9.00 | 102.0 | 2.75 |
| Phenthoate | 98.1 | 3.9 | 98.4 | 8.9 | 98.2 | 6.2 | 91.0 | 18.67 | 79.3 | 14.43 | 104.4 | 3.37 |
| Phorate | 110.0 | 5.4 | 102.9 | 7.3 | 99.5 | 5.3 | 107.2 | 8.77 | 106.9 | 5.58 | 83.4 | 4.76 |
| Phosalone | 97.9 | 5.3 | 92.7 | 10.8 | 99.6 | 10.6 | 111.4 | 7.05 | 107.0 | 1.88 | 92.3 | 4.15 |

**Table 3.** Percentage of recovery (%), relative standard deviation (%RSD) for the validated method for each analyte by GC-MS/MS system in cannabis and sesame oil. (cont.)

| Pesticide | Cannabis (mg/kg) | | | | | | Sesame oil (mg/kg) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0.05 | | 0.2 | | 1 | | 0.05 | | 0.2 | | 1 | |
| | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD | %Rec | %RSD |
| Phosmet | 77.4 | 8.8 | 93.9 | 8.7 | 96.4 | 11.9 | 104.3 | 15.80 | 102.6 | 3.22 | 92.9 | 7.32 |
| Phosphamidon | 91.8 | 5.1 | 93.4 | 12.0 | 99.2 | 12.5 | 107.6 | 9.13 | 60.5 | 11.60 | 108.1 | 4.11 |
| Picoxystrobin | 107.4 | 4.8 | 103.7 | 5.7 | 99.4 | 4.9 | 101.0 | 13.42 | 117.0 | 2.68 | 99.1 | 11.23 |
| Pirimiphos-ethyl | 105.2 | 4.6 | 102.4 | 6.0 | 98.1 | 5.3 | 100.4 | 10.69 | 91.3 | 15.77 | 98.5 | 4.76 |
| Pirimiphos-methyl | 101.3 | 6.2 | 103.2 | 6.4 | 99.4 | 5.9 | 101.9 | 6.78 | 76.9 | 10.29 | 106.6 | 7.02 |
| Profenofos | 98.1 | 4.1 | 95.8 | 10.6 | 99.5 | 7.1 | 110.5 | 7.36 | 102.3 | 3.72 | 102.9 | 7.62 |
| Propachlor | 105.2 | 4.3 | 101.6 | 6.7 | 98.5 | 6.0 | 103.9 | 7.04 | 92.4 | 5.71 | 104.3 | 6.11 |
| Propargite | 107.0 | 7.1 | 102.7 | 8.2 | 97.6 | 5.6 | 110.8 | 6.31 | 68.7 | 8.66 | 108.7 | 5.96 |
| Propetamphos | 103.7 | 5.2 | 101.4 | 7.3 | 100.1 | 6.1 | 109.5 | 5.78 | 70.2 | 5.56 | 107.6 | 4.38 |
| Prothiofos | 105.2 | 5.1 | 102.6 | 6.7 | 98.4 | 5.4 | 106.0 | 7.36 | 91.6 | 7.74 | 105.7 | 7.22 |
| Pyrimethanil | 109.4 | 5.2 | 98.7 | 9.0 | 101.4 | 6.7 | 107.9 | 6.36 | 86.7 | 16.84 | 105.4 | 5.69 |
| Quinalphos | 101.8 | 5.1 | 99.3 | 7.9 | 98.2 | 5.7 | 104.6 | 6.30 | 71.1 | 4.43 | 110.4 | 2.75 |
| Quintozene | 104.2 | 6.2 | 104.9 | 7.0 | 95.3 | 6.3 | 102.8 | 10.31 | 66.8 | 9.84 | 102.6 | 10.33 |
| Simazine | 107.3 | 8.1 | 103.1 | 11.5 | 100.4 | 10.5 | 108.4 | 7.06 | 81.7 | 16.32 | 98.5 | 10.31 |
| Tebufenpyrad | 107.3 | 4.8 | 104.6 | 5.7 | 99.0 | 5.1 | 107.2 | 5.80 | 73.1 | 18.39 | 108.7 | 6.25 |
| Tecnazene | 110.1 | 4.7 | 101.1 | 5.2 | 100.8 | 5.5 | 99.2 | 5.35 | 76.8 | 6.52 | 103.4 | 9.10 |
| Terbacil | 100.9 | 5.4 | 95.6 | 12.8 | 97.3 | 8.1 | 82.3 | 17.31 | 112.2 | 11.33 | 110.1 | 6.09 |
| Terbufos | 106.6 | 4.9 | 103.6 | 5.8 | 100.4 | 5.5 | 111.7 | 9.55 | 92.8 | 5.90 | 80.3 | 5.86 |
| Tetrachlorvinphos | 100.0 | 4.7 | 91.7 | 11.6 | 96.7 | 11.9 | 101.8 | 8.04 | 119.1 | 2.45 | 103.0 | 4.76 |
| Tetradifon | 109.3 | 8.2 | 100.6 | 5.9 | 99.7 | 4.9 | 104.7 | 6.95 | 112.4 | 11.62 | 103.4 | 5.21 |
| Thiometon | 103.7 | 3.4 | 98.8 | 7.8 | 99.9 | 6.1 | 105.0 | 8.29 | 89.4 | 8.33 | 96.5 | 8.39 |
| Tolclofos-methyl | 102.4 | 3.9 | 101.1 | 7.1 | 100.1 | 5.6 | 103.5 | 15.64 | 91.1 | 16.49 | 101.8 | 13.30 |
| Tolyfluanid | 79.9 | 6.0 | 87.2 | 8.4 | 86.5 | 11.6 | 109.7 | 4.22 | 69.3 | 16.73 | 110.1 | 9.61 |
| Triadimefon | 103.6 | 4.7 | 104.4 | 6.6 | 99.0 | 4.9 | 104.2 | 4.56 | 81.3 | 15.61 | 105.9 | 4.90 |
| Triazophos | 99.0 | 4.8 | 95.2 | 9.6 | 100.8 | 9.5 | 102.1 | 11.54 | 100.3 | 5.02 | 104.7 | 2.01 |
| Trifluralin | 99.5 | 3.8 | 101.7 | 6.2 | 99.7 | 5.4 | 92.2 | 16.61 | 95.7 | 12.20 | 99.5 | 7.41 |

**Table 4.** Pesticides detected from cannabis and cannabis product samples submitted by government agencies.

| Pesticides | Detected (n) | Positive sample (%) | Max. (mg/kg) | Min. (mg/kg) | Median (mg/kg) |
|---|---|---|---|---|---|
| Chlorpyrifos | 68 | 80.0 | 77.50 | < 0.05 | 5.18 |
| Cypermethrin | 19 | 22.4 | 11.40 | 0.22 | 1.02 |
| Profenofos | 17 | 20.0 | 16.60 | < 0.05 | 1.06 |
| Fenobucarb | 4 | 4.7 | 3.37 | 2.57 | 3.00 |
| Malathion | 2 | 2.3 | 0.50 | 0.18 | 0.34 |
| Fosthiazate | 2 | 2.3 | 0.81 | 0.59 | 0.70 |
| Fipronil | 2 | 2.3 | 0.21 | 0.20 | 0.20 |
| Chlorothalonil | 1 | 1.2 | 0.43 | 0.43 | 0.43 |

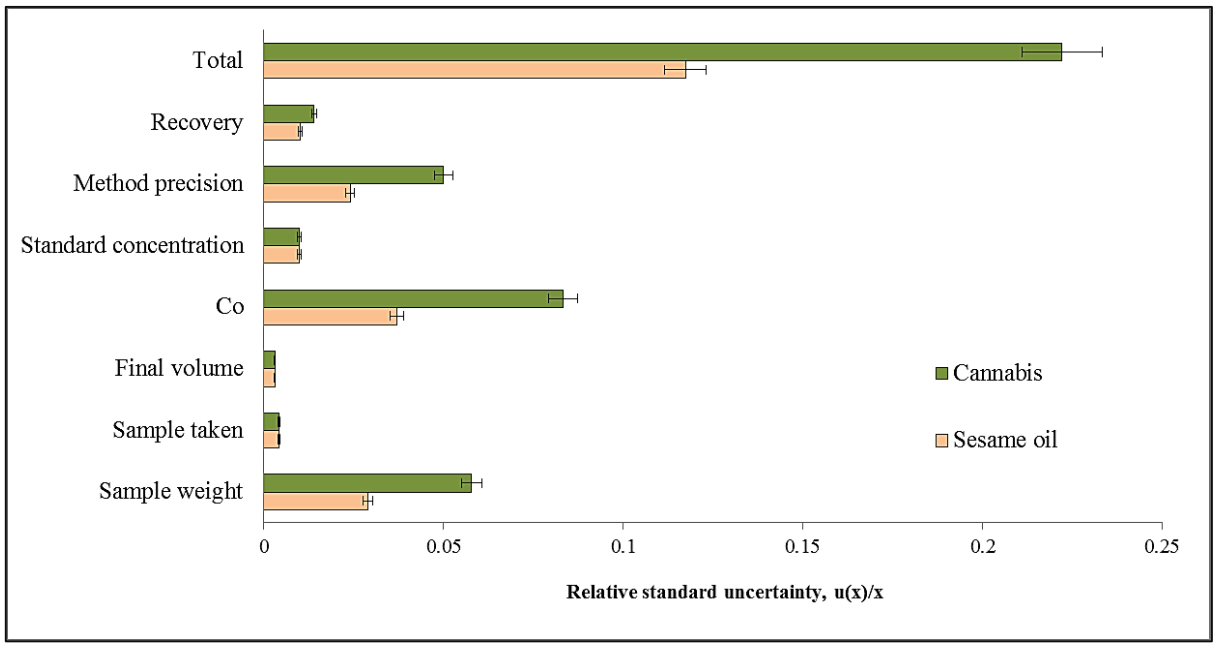


**Figure 3.** Contribution of the different sources to the overall combined uncertainties of chlorpyrifos in cannabis and sesame oil.

and cannabis products combining several sorbents for clean-up was developed. The EN15662 QuEChERS extraction was modified and the dispersive SPE purification technique with three different sorbents (C18EC, GCB, and PSA) was used to eliminate the matrix interference, prior to gas chromatography tandem mass spectrometry technique. Therefore, 122 pesticides were quantified using matrix-matched calibration curves to overcome matrix effects. Pesticide compounds were quantified with high accuracy and met SANTE/12682/2019 standards for recovery (70% to 120%). The method had good repeatability and met regulations for RSD (≤ 20%) and low LOQs (0.05 mg/kg). LOQs for almost all pesticides were less than the action limits for these pesticides in medicinal herbs and were complied with the guidelines of the Thai Herbal Pharmacopoeia as well as the requirements of Codex Alimentarius and Thai maximum residue limits (MRLs) for the category herbs and dry herbs categories. These results demonstrated this method's applicability and effectiveness this method in detecting and quantifying GC amenable pesticides in cannabis and similar samples. In future research, the investigation on matrix effects in Thai traditional herbal formulas containing cannabis will be performed to evaluate whether this method can be applied to these types of commodities.

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