



Development of A Multi Residue Method for Determination of Pesticide Residues in Some High Oil Content Agricultural Products

Mostafa K. Khalil¹, Mohamed E. Amer¹ and L.H. Khalil²

¹Central Laboratory of Residue Analysis of Pesticides and Heavy Metals in Foods (QCAP Lab), Agricultural Research Center (ARC), Ministry of Agriculture and Land Reclamation (MALR), Giza, Egypt.

²Inorganic and Analytical Chemistry Department, Faculty of Science, Ain Shams University, Cairo, Egypt.

Received: 02 Jan 2024

Accepted: 05 Feb. 2024

Published: 30 Sept. 2024

ABSTRACT

Pesticides are chemical compounds used to enhance food production. Unfortunately, pesticides have toxic effects on humans and animals through the consumption of agricultural products. Sesame and peanuts were fatty heavy matrices that couldn't be easily extracted by most known extraction methods. The developed method was optimized and validated for the simultaneous determination of 377 pesticide residues in sesame and 388 pesticides in peanuts depending on a modification in the most common "Quick Easy Cheap Effective Rugged and Safe (QuEChERS)" extraction method. The detection and quantification of these pesticide residues was carried out using both high performance liquid chromatography triple quadrupole mass spectrometry (LC-MS/MS) and gas chromatography triple quadrupole mass spectrometry (GC-MS/MS). The studied method was validated according to SANTE guideline 11312/2021 with different validation parameters such as limit of quantitation (LOQ), linearity, matrix effect, precision and trueness. LOQs of the studied method were ranged from 0.01–0.05 mg/kg for all targeted pesticides. The linearity range for LC-MS/MS was in the range of 0.001–0.1 µg/ml and for GC-MS/MS was in the range of 0.002–0.5 µg/ml with $R^2 > 0.99$. The suppression and enhancement effects on the pesticide concentrations due to the sesame and peanuts components, called the matrix effect which were compensated during the analysis by using the standard addition technique. The precision and trueness of the method were determined from recovery experiments on 6 replicates of spiked blank samples in each commodity at 3 validation levels 0.01, 0.05, and 0.1 mg/kg respectively. Acceptable recoveries in the range of 70–120% and relative standard deviations (RSD %) less than 20% for all the tested pesticides were obtained. The developed method can be easily used in the routine analysis of pesticide residues in sesame and peanuts.

Keywords: Pesticide residues, Sesame, Peanuts, LC-MS/MS, GC-MS/MS, QuEChERS.

1. Introduction

Recently, in the agricultural production process, a wide variety of pesticides have been employed to manage undesirable plants, fungi, rodents, and insects. Some trace levels of pesticide residues may penetrate the food chain and reach humans, potentially posing a health risk, even though the majority of them evaporate from products or break down in soil, water, and the atmosphere (Shettima *et al.*, 2023 and Panseri *et al.*, 2013).

Because of the growing worldwide population and the demand for increased food supplies, it was expected that the use of pesticides for crop protection is going to increase. While there are many advantages to protect plants, improper use of certain pesticides can have major adverse effects on health since they are poisonous (Liu *et al.*, 2016).

Corresponding Author: Mostafa K. Khalil, Agricultural Research Center (ARC), Central Laboratory of Residue Analysis of Pesticides and Heavy Metals in Foods (QCAP Lab), Ministry of Agriculture and Land Reclamation (MALR), Giza, Egypt.
E-mail: mostafakamal476@gmail.com

For this reason, new analytical methods and food monitoring programs for pesticides in the national area are implemented globally to prevent harm to consumer health, enhance the management of agricultural resources, and avert financial damages (Liu *et al.*, 2016).

Over the years, several public and private laboratories around the world have investigated food and environmental samples for pesticide residues. However, the techniques for analyzing common pesticides are far from optimal (Anastassiades *et al.*, 2003).

Humanity has been familiar with sesame since ancient times. Sesame oil is used in many Asian countries' cultural preparations, despite its less widespread distribution than other oil seed crops (rapeseed, soy). Sesame oil and seeds have received recognition as health foods in recent years (Papadakis *et al.*, 2006).

Sesame is consumed in many countries of the world to make edible oil and processed products including chikki (sesame brittle), cake, crackers, and tahini (Shinde *et al.*, 2021). China, Nigeria, Tanzania, India, Sudan, and Myanmar are the largest producer's countries of sesame (Martinchik, 2021).

One of the most essential legumes and a widely consumed food which is rich in nutrients is peanuts that contain high amount of protein, unsaturated fatty acids, vitamins, minerals, and enzymes. As a result, peanuts consumption has added and gained popularity as an additional food source for health (Pang *et al.*, 2021). Besides being processed into a variety of products, such as peanuts sauce, peanuts butter, peanuts pickles, and numerous additional snack items that were sold in stores, peanuts can be consumed as raw food (Hou *et al.*, 2017). As part of a diet low in saturated fat and cholesterol, taking in 1.5 ounces (43 g) of most nuts including peanuts per day might reduce the risk of heart disease according to the US Food and Drug Administration (USFDA) (Campos-Mondragón *et al.*, 2009).

It is still complicated to separate pesticides and other chemical pollutants from fatty food matrices prior to instrumental analysis in the process of analysis, despite major experiments to enhance the analytical technique. It is well known that certain pesticides are used abundantly on various items while they are being grown. These food samples contain high fat content and complicated matrices that restrict the analysis's determination stage, making them challenging to examine these fatty matrices, especially sesame and peanuts (Park *et al.*, 2012).

As multiple of the pesticides typically studied are fat-soluble and non-polar compounds that seek to concentrate and remain in the fat portion, it is actually highly challenging to avoid co-extraction of fatty content. Since multi-class pesticides have a wide range of highly varied physicochemical properties, outstanding recoveries must be achieved in a preferably fat-free extract in order to prevent damage to the various elements of the instruments employed and any interferences with the chromatographic analysis. Before going on to the next steps of the analytical process, there needs to be an extra cleanup. Any small amounts of lipids can damage detectors, chromatographic C₁₈ columns and/or suppress signals. The presence of pesticide residues in fatty matrices leads to apply different clean-up steps with detection techniques that consume large time and huge amounts of chemicals which increase the analysis cost in the multi-residue procedures (García-Reyes *et al.*, 2007).

There are different analytical methods for the determination of pesticide residues in sesame. Bhatnagar and Gupta established an analytical method for chemical substances' residues of chlorpyriphos, quinalphos, and lindane in sesame seeds and oil in 1998 (Bhatnagar, A. and A. Gupta, 1998). Om *et al.* analyzed seven varieties of sesame seeds to determine of small number of detected pesticides such as benomyl, mancozeb, and metalaxyl residues while looking into the contamination of imported and indigenous food products in 1998 (Om *et al.*, 1998). In two other studies, Papadakis *et al.* and Wang *et al.* in 2006 and 2009 determined the chlorinated hydrocarbons in sesame seeds using gas Chromatography mass spectroscopy (GC/MS) (Papadakis *et al.*, 2006) and gas chromatography with electron capture detector (GC/ECD) (Wang *et al.*, 2009) without touching on determination of polar pesticides.

A modified single-step edition of the QuEChERS method was published by Hua *et al.* in 2019 to analyze pesticide residues by LC-MS/MS in four oil seed matrices, including sesame seed, about ten years later. Fortunately, the technique only utilized a wide range of 38 pesticides, and none of them were able to be analyzed using GC-MS (Hua *et al.*, 2019). As a way to analyze 37 target components of sesame seed, sesame oil, sesame paste, and sesame meal simultaneously, the additional multi-residue method was developed (Meng *et al.*, 2023).

As far as we understand, few pesticides for peanuts have been researched. A low-temperature clean-up method for residue determination was developed and validated for only a limited number of 14 organophosphorus pesticides in soybean oil, peanuts oil and sesame oil by old fashion technique as gas chromatography with flame photometric detector (GC-FPD) (Li *et al.*, 2007). There is a method for the determination of pesticide residues in nuts was presented by using ethyl acetate and sodium sulfate to extract pesticides and inject them on a high-pressure liquid chromatography (HPLC) (Cortés *et al.*, 2008). Wang *et al.*, in 2009 established a method based on automated gel permeation chromatography (GPC) for the determination of residues of 38 typically used pesticides in high-oil peanuts (Wang *et al.*, 2009). High performance liquid chromatography-ultraviolet detector (HPLC-UV) was used to determine only pyraclostrobin which was extracted by acetonitrile and cleaned-up with gel permeation chromatography and solid phase extraction-amino sorbent (SPE-NH₂) (Di *et al.*, 2012). Yang *et al.* developed a method For the detection of quizalofop-p-ethyl, chlorpyrifos, acetochlor and imidacloprid in peanuts by ultra-high performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) (Song *et al.*, 2021).

Many methods, including solvent partitioning, solid phase extraction (SPE), dispersive solid phase extraction (DSPE), matrix solid-phase dispersion (MSPD), gel permeation chromatography (GPC), Soxhlet extraction, accelerated solvent extraction (ASE), micro extraction techniques, and the QuEChERS method, would be covered and discussed the determination of pesticide residues in different fat matrices (Madej *et al.*, 2018).

Most of the last procedures were unsuitable for routine analysis and failed as complicated, time-consuming, tedious or lacking in multi-pesticide application. In the present days, QuEChERS (quick, easy, cheap, effective, rugged and safe) is a commonly employed extraction and clean-up method for various food samples and multi-residue pesticide analysis. A potential drawback of the multi-step techniques is the chance of loss of analyte and the need for an extensive amount of expensive and hazardous chemical solvents. A unique movement in pesticide analysis is the use of inexpensive, simple-to-apply ingredients and a less amount of organic solvents in a way that is safe for the environment (Dimitrova *et al.*, 2018). The main step and obstacle in this type of analysis is the sample processing and extraction method (García-Reyes *et al.*, 2007).

The methods that are commonly applied to the multi-residue determination of pesticide residues in food are gas chromatography combined with mass spectrometry using electron impact ionization (EI) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) using the electrospray ionization (ESI) mode. This is due to the fact that can evaluate several pesticides from different chemical categories in highly complicated matrices using a single approach, which proves their excellent sensitivity and selectivity. As polar compounds were analyzed by liquid chromatography-tandem mass spectrometry (Abdelwahed *et al.*, 2021), non-polar compounds were analyzed using gas chromatography combined with mass spectrometry (Soliman *et al.*, 2019). The most widespread and commonly employed technique to evaluate residues of pesticides in food is the QuEChERS methodology (Anastassiades *et al.*, 2003).

In our study, the QuEChERS modern technology was applied to determine multi-class pesticide residues from fatty matrices. The pesticide residue levels in sesame and peanut seeds were investigated. The samples were obtained from the major markets in Egypt in the agricultural seasons 2020-2023. Modified QuEChERS method was developed and validated according to SANTE guideline to determine 377 pesticides divided 229 LC-compounds, 59 GC-compounds, and 89 LC-GC-compounds in sesame in addition to 388 pesticides divided into 242 LC-compounds, 63 GC-compounds, and 83 LC-GC-compounds in peanuts.

2. Materials and Methods

2.1. Chemicals and reagents

All reference standards were purchased from Dr. Ehrenstorfer (Augsburg, Germany) with a purity of more than 99%. Aldrin was used as an injection standard for the GC-MS/MS analysis, purchased from Dr. Ehrenstorfer (Augsburg, Germany). Acetonitrile and Methanol LC-MS grade were purchased from J.T. Baker (Center Valley, PA, USA). Ammonium formate was bought from Sigma-Aldrich (Oakville, ON, Canada). Toluene, Acetone and n-hexane were all obtained from Merck (Darmstadt, Germany). QuEChERS Reagent (1) Kit was obtained from Agilent (Santa Clara, CA, USA)

and consisted of salt packets which included 4 ± 0.02 g anhydrous magnesium sulfate, 1 ± 0.05 g sodium chloride, 1 ± 0.05 g trisodium citrate dihydrate, and 0.5 ± 0.03 g disodium hydrogen citrate sesquihydrate. QuEChERS Reagent (2) Kit was purchased from Agilent (Santa Clara, CA, USA) that contains 900 mg of anhydrous magnesium sulfate and 150 mg of primary secondary amine bonded phase silica (PSA) for dispersive solid phase extraction (d-SPE) in 15 ml centrifuge tubes. Deionized pure water was purified by Millipore MilliQ water purification system (Billerica, MA, USA).

2.2. Apparatus

Sample homogenization was done using ultra-turrax® homogenizer T25 digital (IKA, Germany), the polytetrafluoroethylene (PTFE) membrane syringe filter with $0.45 \mu\text{m}$ pore size and the 50 ml polypropylene centrifuge falcon tubes with screw caps were obtained from Supelco (Bellefonte, USA). The used Z32 HK centrifuge was supplied from Hermle (Gosheim, Germany). The injection glass vials were brought along with Teflon-coated caps from Agilent Technologies (Santa Clara, CA, USA). The used volumetric flasks (5, 10 and 20 ml), the graduated glass pipettes (5 ml), the bottle top dispenser (5–50 ml) and the micropipettes (variable 2–20 μl , 10–100 μl and 100–1000 μl) under ISO grade-A quality were purchased from Hirschman (Eberstadt, Germany). The used Hei-VAP rotary evaporator was obtained from Heidolph (Schwabach, Germany). The benchtop pH meter, analytical balance and precision balance were supplied from Mettler Toledo (Greifensee, Switzerland). Grindomix Knife Mills GM 300 blender to get $300 \mu\text{m}$ particular size samples was used (Schwabach, Germany).

2.3. Standards preparation

Stock solutions of $1000 \mu\text{g/ml}$ were prepared for each pesticide separately in a suitable solvent Toluene. A $10 \mu\text{g/ml}$ intermediate standard solution mixture of LC amenable pesticides and another mixture of GC amenable pesticides were prepared in toluene, followed by a separate dilution for each mixture to prepare a $2.5 \mu\text{g/ml}$ spiking mixture in toluene. A standard solution of Aldrin ($100 \mu\text{g/ml}$) was prepared in n-hexane to be later used as an injection standard (IS) for GC-MS/MS analysis. The pesticide calibration mixture levels for GC-MS/MS were as following 0.002, 0.01, 0.05, 0.1 and $0.5 \mu\text{g/ml}$ in pesticides blank green beans extract (extracted using QuEChERS method) with solvent compositions of (n-hexane: acetone, 9:1) containing $0.1 \mu\text{g/ml}$ Aldrin added to all the calibration levels. For LC-MS/MS, the calibration levels were prepared in methanol as following 0.001, 0.002, 0.01, 0.05 and $0.1 \mu\text{g/ml}$. The stock, intermediate standard and IS were stored in a freezer at -20°C until the time of the analysis while calibration and spiking mixtures were stored in a refrigerator at 4°C .

2.4. Sample preparation

Different samples were collected from the Egyptian local market such as Sesame and peanuts without shells were homogenized by grinding by Grindomix electric blender to grind the representative samples and subsampled in plastic bottles to be stored in a freezer at -20°C .

Spiked samples were used in method development and validation by using pre-analyzed blank samples which have been fortified with a spiking mixture containing the target compounds. Pesticides were extracted from samples using an extraction procedure based on the QuEChERS methodology.

A schematic diagram of the sample extraction method is shown in Figure (1). The procedure was as following, A 2 ± 0.03 g homogenized sample was weighted and added into a 50 ml polypropylene falcon tube. We added about 10 ml from acetonitrile: acetone (9:1 v/v) into the sample. Ultra-Turrax homogenizer was operated at 5,000 rounds per minute (rpm) for 1 minute for each sample to blend well and let the extracting solvent a chance to penetrate the sample tissues.

QuEChERS Reagent (1) was added to the tube and shaken vigorously for 1 min at 500 rpm by Geno/Grinder device to homogenize the sample and facilitate the single-phase extraction steps. The sample was centrifuged for 5 min at 4000 rpm. About 1 ml of the supernatant portion was filtered by a syringe filter and transferred into a vial for LC/MS-MS injection.

The remaining supernatant about 5 ml was transferred into QuEChERS Reagent (2) to make DSPE. The tube was shaken vigorously by hand and then centrifuged at 4000 rpm for 2 minutes. A 2 ml of supernatant was transferred into a 50 ml glass flask and concentrated using a rotary evaporator at 280 rpm and $39 \pm 1^\circ\text{C}$ until dryness.

The thin film in the dried flask was reconstituted by using 2 ml n-hexane: acetone (9:1 v/v) containing $0.1 \mu\text{g/ml}$ of the Aldrin injection standard followed by ultra-sonication for a few seconds to

dissolve the last thin film of the sample. Finally, the sample was filtered through a 0.45 µm syringe filter and transferred into a vial for GC/MS-MS injection.

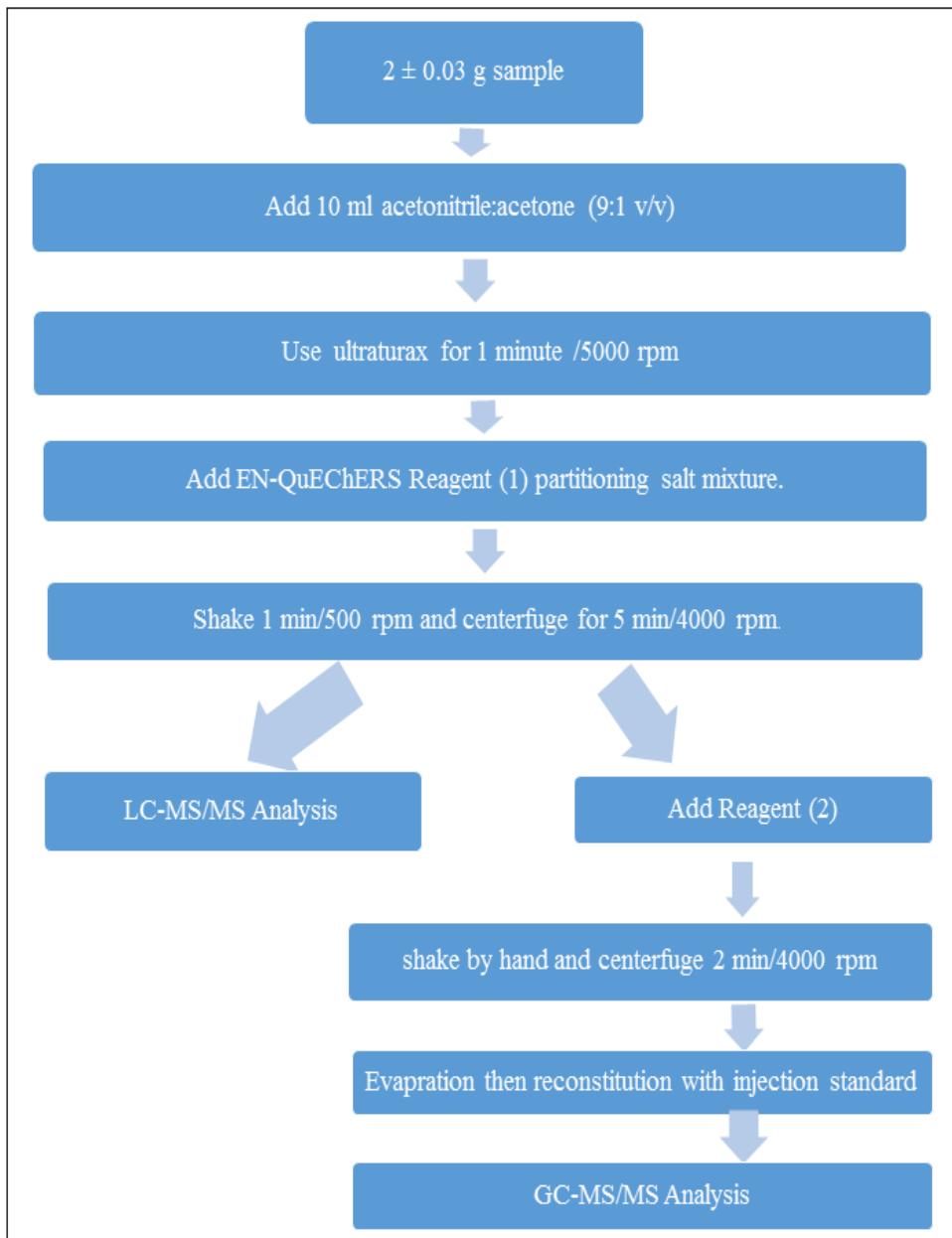


Fig. 1: Scheme of the developed extraction method for sesame and peanuts

Instrumentation

2.5. LC-MS/MS analysis

Agilent HPLC from 1200 Series instrument coupled with an API 4000 Qtrap MS/MS from ABSciex and an electro spray ionization (ESI) interface in the positive mode, source temperature was set at 400 °C, and ion spray potential was operated at 5500 V. Chromatographic separation was performed on an Agilent C₁₈ column ZORBAX Eclipse XDB 4.6 × 150 mm with 5.0 µm particle size. The ESI source, N₂ nebulizer, curtain gas, declustering potential (DP), collision energy (CE), the MRM transitions, and other specific parameters were optimized according to the manufacturer's recommendations using Harvard apparatus syringe pump to introduce individual pesticide solutions into the MS instrument as described in Table (1S) in Appendix.

The injection volume was set at 5 μ l. As shown in Table (1), a gradient elution program was done at 500 μ l/min flow rate of mobile phase, in which one reservoir contained 10 mM ammonium formate solution in Methanol: Water(1:9 v/v) at pH 4 and the other contained LC-MS Methanol grade. The run time was 20 min. Analyst software 1.6 was employed for instrument control and data acquisition/processing.

Table 1: The LC gradient elution program for determination of pesticides in sesame and peanuts.

Total Time (min)	Flow Time(μ l/min)	Mobile phase Buffer (%)	Methanol (%)
0	500	50	50
2	500	50	50
3	500	25	75
10	500	25	75
10.5	500	10	90
11	500	3	97
17.5	500	3	97
17.6	500	50	50
20	500	50	50

2.6. GC-MS/MS analysis

The analysis was carried out using a 7890A Agilent Gas Chromatography system equipped with a 7000B triple quadruple mass spectrometer (Agilent Technologies, USA). Chromatographic separations were accomplished using the HB-5MS Ultra-inert capillary column (5% biphenyl-95% dimethylsiloxane, 30 m column length \times 0.18 mm id \times Film thickness 0.25 μ m) that was obtained from Agilent Technologies (USA). The inlet operating conditions were 1 μ l injection volume, 1.3 ml/min flow rate, and GC oven temperatures programming as described in Table (2) where the samples were injected in a splitless mode and the run time was 25 min. The Electron impact (EI) mode was at (+70 eV); the monitoring was from m/z 50 to 400. The ion source, Injector, and Quadrupole analyzer temperatures were fixed at 300, 250, and 150 $^{\circ}$ C, respectively. Mass Hunter software was employed for instrument control and data acquisition/processing.

Table 2: The GC oven temperature programming for determination of pesticide residues.

Rate ($^{\circ}$ C/min)	Temperature value ($^{\circ}$ C)	Hold time (min)	Runtime (min)
Initial	70	1	1
Ramp 1	50	0	2.6
Ramp 2	6	0	20.933
Ramp 3	20	310	1.567
			25

3. Results and Discussions

3.1. Optimization of extraction method

Optimization of the extraction method was carried out by testing and changing the most effective parameters that have high effects on the extraction performance. It is necessary to achieve high extraction efficiency at optimum extraction method conditions. Many factors can influence the analytical procedures but in our extraction method, there were four main factors such as fat content reduction, extracting solvent, homogenization time by Ultra-Turrax homogenizer and GC-injection solvent.

3.1.1. Reducing the fat content during the extraction:

In several studies, sesame has an average oil content ranging from 44 to 53% due to the presence of a large number of different saturated and unsaturated fats such as oleic acids, linoleic acid, linolenic acid, palmitic acid, and stearic acid (Kurt, C., 2018 and Onsارد, E., 2012). One of the main oilseeds in terms of nutritional value is the peanuts, which has a protein level of 27%–29% and an oil content of 47% and 50% (Boukid, F. 2022 and Zhang *et al.*, 2020). We measure the fat content of sesame and Peanuts by using Soxhlet as shown in Table (3).

Table 3: Fat content percentages for sesame and peanuts.

Samples	Sample weight (g)	Fat weight (g)	Fat Content %
Peanuts (1)	24.37	10.72	44
Peanuts (2)	24.87	12.82	51
Sesame (1)	21.68	11.82	55
Sesame (2)	20.7	12.67	61

We measure the remaining matrix components in our studied samples by evaporation the solvent after addition of QuEChERS Reagent (1) and Reagent (2) as shown in Figure (2).

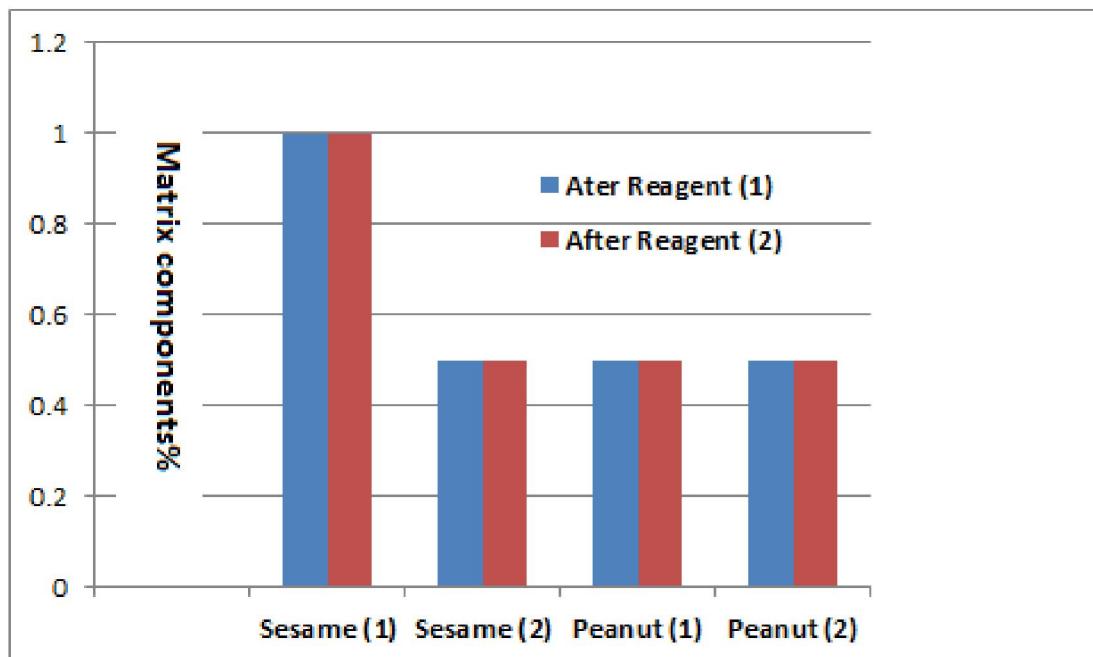


Fig. 2: Matrix components% after using QuEChERS Reagent (1) and Reagent (2)

3.1.2. Optimization of the extracting solvent

Due to the presence of fat content in sesame and peanuts, we have to optimize a solvent that can extract difficult non-polar analytes from non-polar fatty matrices such as sesame and peanuts. When creating regular samples, these matrices presented obstacles during the acetonitrile or ethyl acetate extraction procedure due to their higher lipid content (Rejczak, T. and zimski, 2015). High matrix effects and a decrease in LC-MS/MS sensitivity brought on by the accumulation of co-extracted lipids in the ion source were the issues identified during this investigation (Elshabrawy *et al.*, 2019 and Shinde *et al.*, 2021).

Because acetonitrile is appropriate for a wide variety of pesticides (physicochemical characteristics) and facilitates a very limited amount of matrix-fat co-extraction, it was chosen as the extraction solvent (Attallah *et al.*, 2018). Because of its low viscosity and intermediate polarity, it is highly beneficial in reversed-phase liquid chromatography (LC) and SPE applications. For the extraction of multiple pesticide residues, acetonitrile was considered to be the best solvent since it provided better recoveries from sample matrices with the lowest co-extractives (Abdelwahed *et al.*, 2019 and Anastassiades *et al.*, 2003).

Identifying the extraction solvent is a crucial stage in our study's extraction procedure. We optimize the extracting solvent by adding 1 ml of acetone to 9 ml acetonitrile for the reason to extract non-polar compounds such as Organochlorines (OCs), organophosphorus (Ops) and Polychlorinated biphenyl (PCBs) which improve sensitivity and selectivity of the compounds. The test was carried out by 3 replicates for 4 experiments that have different ratios from acetonitrile and acetone as extracting solvent mixture.

In our test, the experiments have different ratios between acetonitrile and acetone where experiment (1) represents 100% acetonitrile, experiment (2) represents acetonitrile: acetone (9.5: 0.5 v/v), experiment (3) represents acetonitrile: acetone (9: 1 v/v) and experiment (4) represent acetonitrile: acetone (8:2 v/v).

Among the four experiments for the four extracting solvents, found that acetonitrile: acetone (9:1 v/v) gave an acceptable recovery range in both of LC-MS/MS and GC-MS/MS in sesame and peanuts at (70-120%) with the highest number of pesticides as shown in Figure (3, 4).

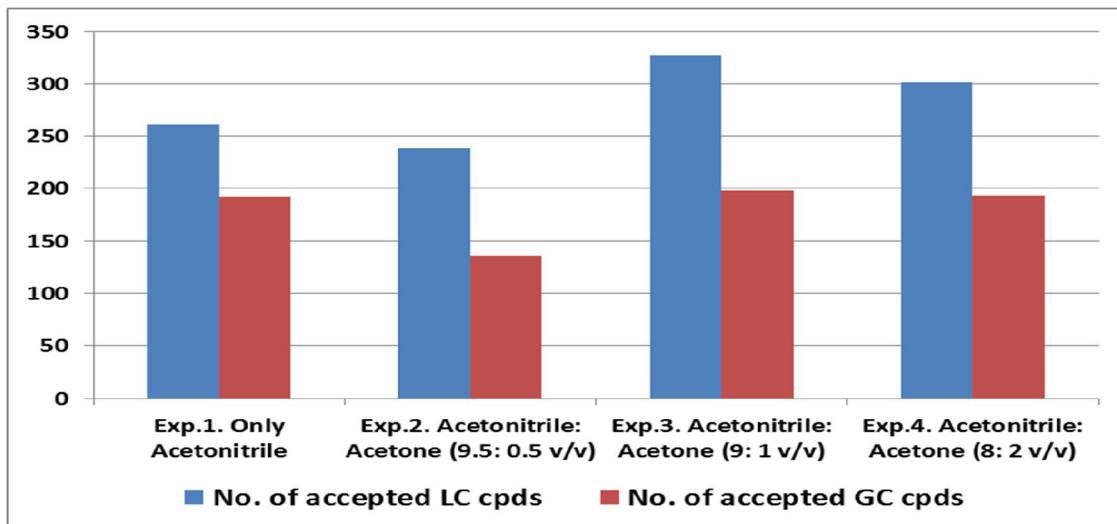


Fig. 3: Optimization of solvent extraction for sesame samples

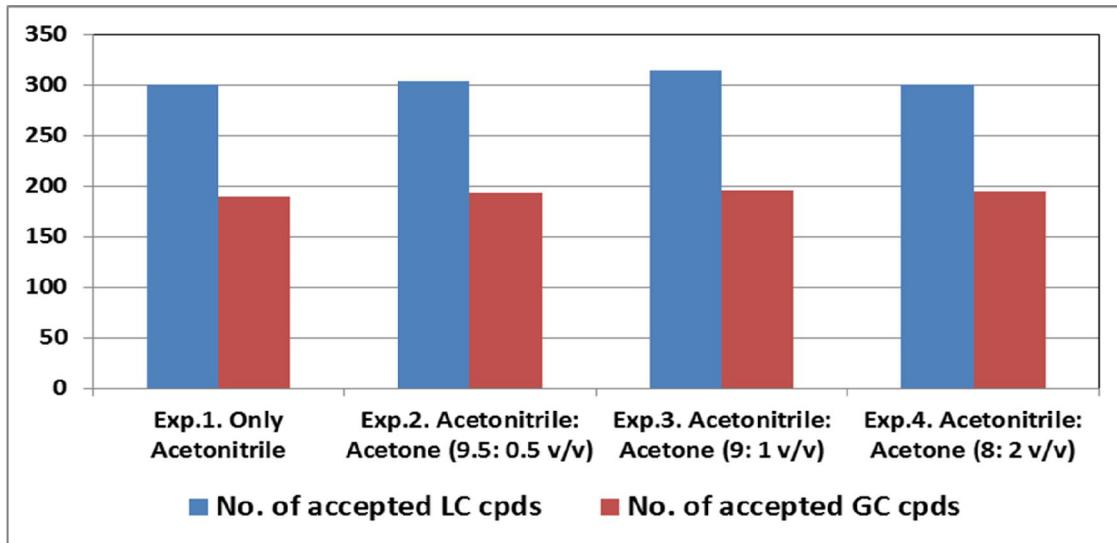


Fig. 4: Optimization of solvent extraction for peanuts samples

3.1.3. Optimization of homogenization time by Ultra-Turrax homogenizer

We used Ultra-Turrax homogenizer to enable the best possible results by grinding the sample with extraction solvent because of the presence of a high fatty matrix of sesame and peanuts. The Ultra-Turrax helps the solvent to penetrate the tissue to extract the analytes.

In these experiments, we compare the time of homogenization (1, 2 and 3 minutes) for sesame samples. The data obtained showed that 1 min Ultra-Turrax homogenization is the optimum one as shown in Figure (5).

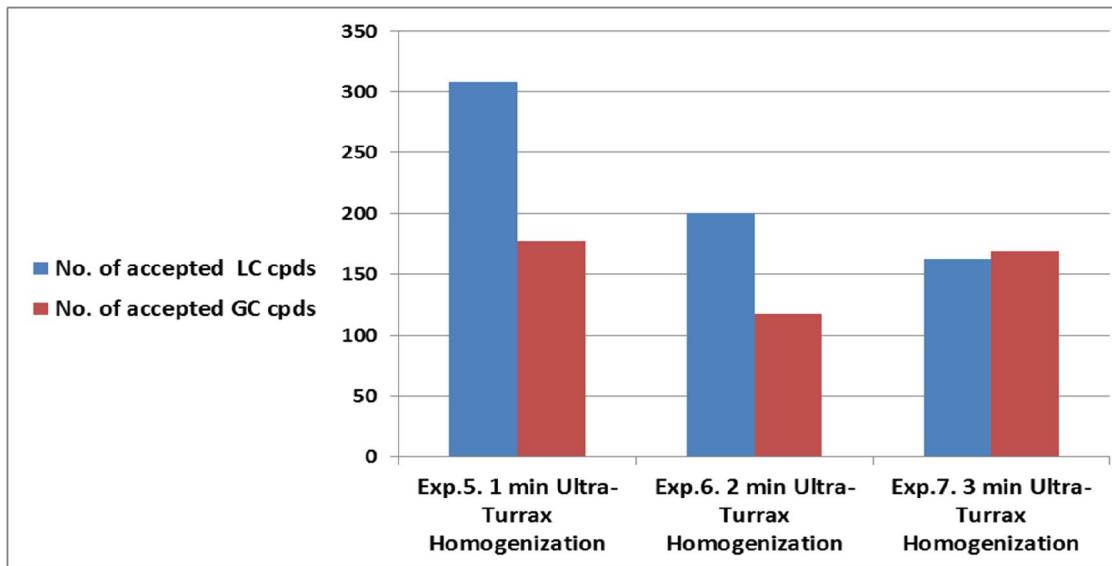


Fig. 5: Optimization of homogenization time by using Ultra-Turrax blending

3.1.4. Optimization of the GC-injection solvent:

It is very difficult to use acetonitrile as a GC injection solvent because it has a high vapour pressure. It is not compatible with relatively non-polar GC stationary phases and can form droplets rather than a continuous film upon condensation in the GC column, causing peak distortions for early eluting analytes.

So, we should evaporate the sample with a rotary evaporator and then make reconstitution with a suitable solvent as an injection standard solution. As shown in Figure (6), using the GC-Injection standard in n-hexane: Acetone (9: 1 v/v) is better than using the GC-Injection standard in n-hexane: Acetone (9: 1 v/v) in green Beans extract. The test was carried out by 3 replicates.

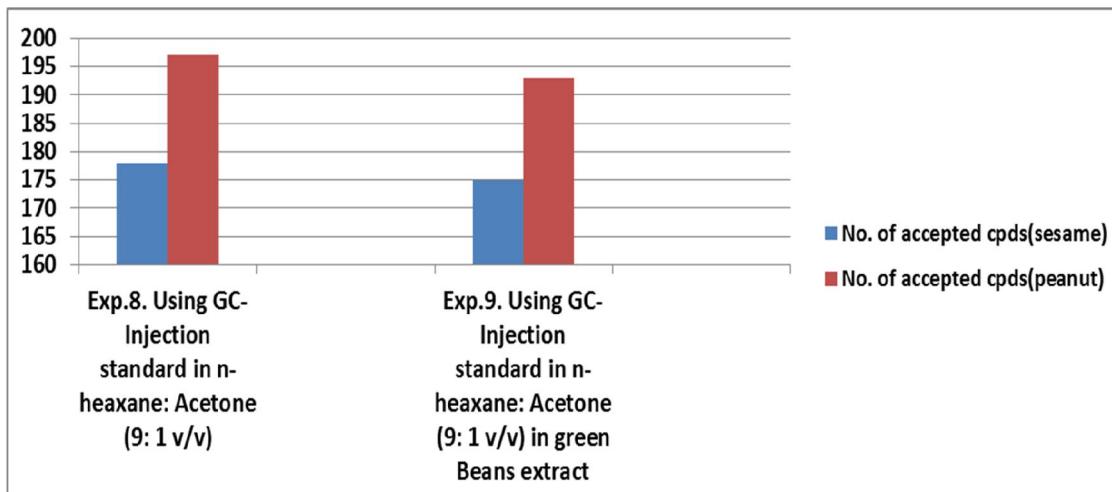


Fig. 6: Comparison of GC- Injection standard for determination of pesticide residues

3.2. Method validation

The protocol that was established received validation in line with the method validation standards outlined in the SANTE guideline 11312/2021 paper. The linearity check between the calibration levels and the calibration's sensitivity were measured in addition to making compensation for the matrix effect. By comparing a single-point standard addition solution with the calibration that was used, the matrix effect was quantified. The aforementioned commodities were spiked at different levels in order to

compute the following parameters: matrix effect, recovery and precision, limit of quantitation (LOQ), linearity and repeatability.

3.2.1. Matrix effect

By employing post-extraction spiking, matrix effects such as signal suppression or enhancement were studied in relation to the co-elution of matrix components and contrasted with solvent standards. When the extract of blank control samples was applied to create matrix-matched calibration standards, the resulting standards were around the same concentration range as the solvent standard solutions. By using a standard addition technique to get an expected concentration of 0.05ug/ml for any tested pesticides, the matrix effect was evaluated to compensate the matrix-induced enhancement and/or suppression in LC-MS/MS and/or GC-MS/MS. The calculations were done as the following equation:

$$\text{Matrix effect \%} = \frac{\text{Found concentration of standard in matrix}}{\text{Expected concentration of standard in matrix } 0.05} \times 100 \dots \dots \dots (1)$$

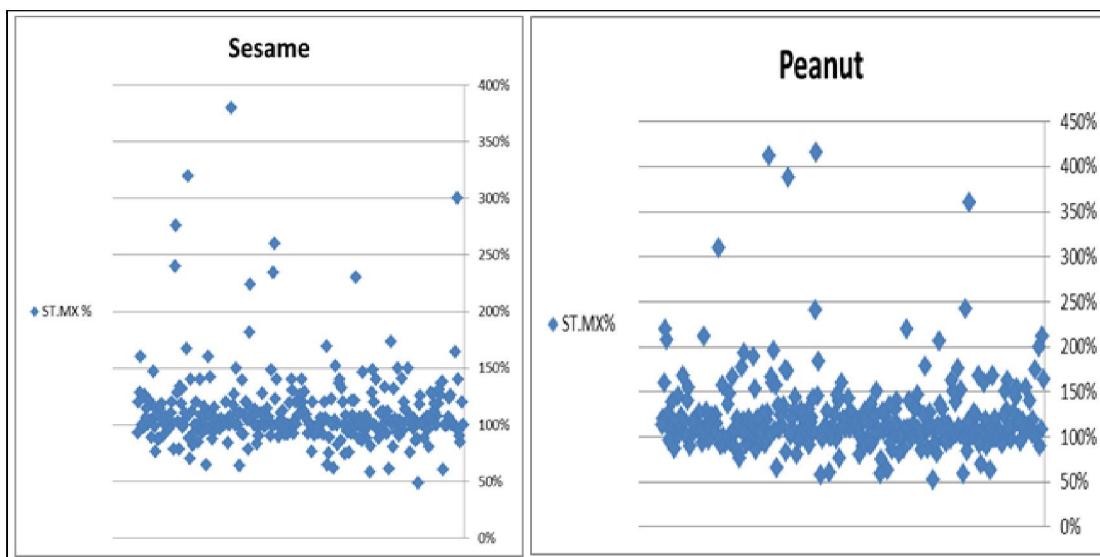


Fig. 7: Matrix Effect % of all analytes on LC-MS/MS at expected concentration 0.05 mg/kg

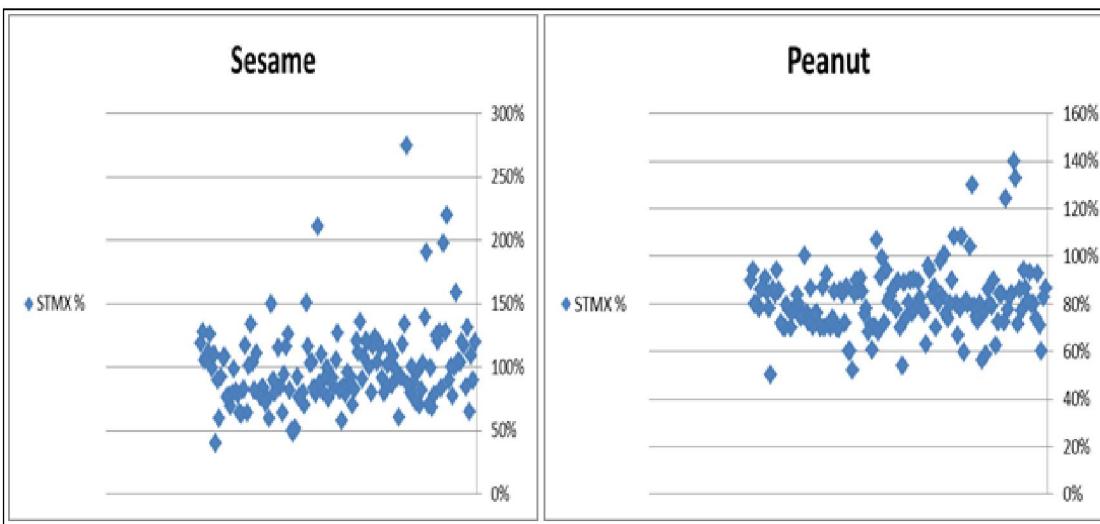


Fig. 8: Matrix Effect % of all analytes on GC-MS/MS at expected concentration 0.05 mg/kg

3.2.2. Recovery and precision

The percentage recovery at three separate fortification levels (0.01, 0.05, and 0.1 µg/ml) was used to calculate the recovery. The study examined precision using two different metrics which was the repeatability at three different fortification levels with six replicates in the same day, extraction method, analyst and instrument as shown in **Tables (4, 5)**.

The numerical precision values are defined as the relative standard deviations of six repeat measurements of the analytes by using the equation:

Where SD = standard deviation of replicates.

3.2.3. Limit of Quantification

The minimal amount that a reliable analytical method might successfully detect and quantify is designated as the limit of quantitation, or LOQ. Six recovery replicates on blank peanuts and sesame samples were performed to estimate the LOQs, and the samples were spiked at the indicated lowest quantitation validation level of 0.01 µg/ml.

According to the procedural manual of Codex Alimentarius Commission (CAC), 28th Edition in 2023, the most acceptable default limit of tolerance (LOQ) for pesticides in food and feed was 0.01 mg/kg. The minimum quantitation limit of this method is 0.01 mg/kg except for 84 analytes for sesame and 77 analytes for peanuts which have LOQs at 0.05 mg/kg on LC and/or GC as shown in Tables (4, 5).

3.2.4. Linearity and calibration curve

The linearity of calibration curves was assessed over the range from 0.001–0.1 µg/ml for LC-MS/MS and 0.002–0.5 µg/ml for GC-MS/MS for the analytes which have correlation coefficients of linear functions > 0.99 as shown in Figure (9, 10). Figure (9) Calibration curve of acetamiprid in LC-MS/MS as an example on LC-MS/MS compounds while Figure (10) calibration curve of chlorophrophoram in GC-MS/MS as an example on GC-MS/MS compounds.

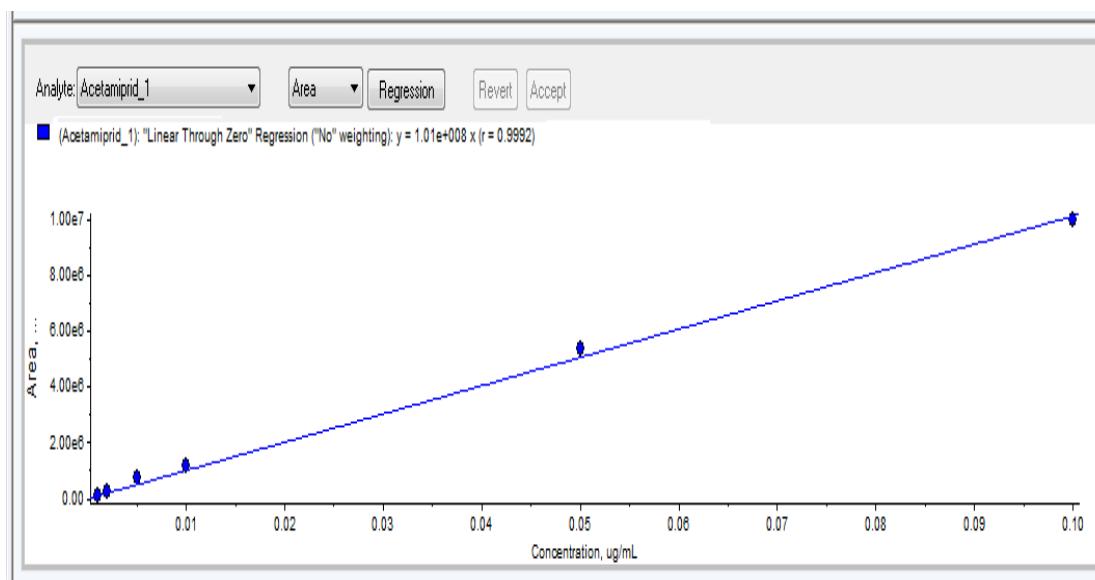


Fig. 9: Calibration curve of acetamiprid in LC-MS/MS with $R^2 = 0.9992$

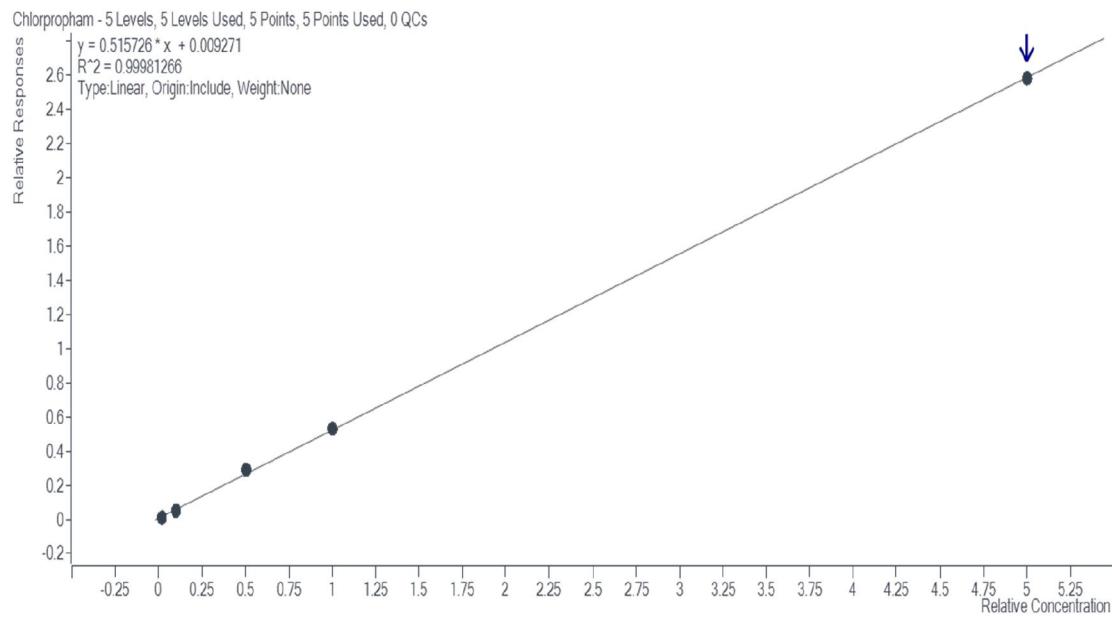


Fig. 10: Calibration curve of chlorophropham in GC-MS/MS with $R^2 = 0.99981266$

3.2.5. Repeatability

The test findings were gathered in a single laboratory over a short time, using the same method on the same sample. No variations in the materials and equipment used, or the analysts concerned, occurred during this time. Six replicates of spiked on blank samples at three different concentration levels (0.01, 0.05, and 0.1 mg/kg) were fortified in order to perform repeatability tests, from which recovery percentage and RSD% were obtained. The results of the study demonstrated that all pesticides had satisfactory repeatability ($RSD\% < 20\%$) over the analyzed validation levels. The relative standard deviations were less than 20, and the corresponding percentages of average recoveries were within the allowable range of 70–120% as shown in Tables (4, 5).

Table 4: Compilation of average pesticide recovery results in LC-MS/MS and GC-MS spiked at 0.01, 0.05, and 0.1 mg/kg (6 replicates) in the sesame matrix.

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Abamectin	LC	106	13%	117	16%	103	18%
Acephate*	LC	110	8%	99	15%		
Acetamiprid	LC	117	5%	105	10%	87	9%
Acrinathrin*	LC	93	15%	112	12%		
Aldicarb Sulfone*	LC	96	6%	86	11%		
Aldicarb Sulfoxide*	LC	82	9%	73	19%		
Aldicarb	LC	118	9%	116	14%	103	19%
Ametryn	LC	118	3%	115	12%	120	8%
Amidosulfuron*	LC	112	10%	91	16%		
Aminocarb	LC	114	4%	120	5%	115	5%
Anilofos*	LC	120	5%	119	9%		
Atrazine	LC	114	7%	115	19%	117	5%
Azaconazol	LC	118	5%	118	15%	105	16%
Azamethiphos	LC	120	16%	115	11%	87	9%
Azimsulfuron*	LC	119	8%	111	16%		
Azinophos-ethyl	LC	120	4%	113	20%	117	11%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Azoxystrobin	LC	118	10%	114	13%	113	10%
Beflubutamid*	LC	119	7%	119	15%		
Benalaxyl	LC	116	5%	115	17%	109	7%
Bendiocarb	LC	119	8%	115	10%	105	14%
Benfuracarb	LC	116	6%	107	10%	119	11%
Bensulfuron-methyl	LC	119	5%	116	14%	71	18%
Benthiavalicarb							
isopropyl	LC	120	9%	118	12%	118	19%
Benzoximate*	LC	120	3%	119	8%		
Bifenazate	LC	116	6%	105	9%	119	12%
Bitertanol	LC	118	8%	114	11%	110	11%
Boscalid	LC	114	11%	111	11%	117	9%
Bromacil	LC	119	9%	118	16%	81	10%
Bromuconazole	LC	100	17%	97	16%	118	18%
Bupirimate	LC	119	8%	115	15%	112	8%
Buprofezin	LC	109	3%	110	17%	116	14%
Butachlor*	LC	118	8%	115	13%		
Butocarboxim*							
sulfoxide	LC	82	11%	71	13%		
Butocarboxim	LC	93	10%	81	17%	89	10%
Butralin	LC	106	5%	110	11%	116	5%
Butylate	LC	113	9%	109	9%	120	19%
Carbaryl	LC	119	8%	117	14%	120	15%
Carbendazim	LC	106	4%	105	9%	78	13%
Carbetamide	LC	119	6%	110	8%	73	13%
Carbofuran	LC	119	6%	119	8%	115	11%
Carbofuran-3OH*	LC	119	5%	107	14%		
Carboxin	LC	118	5%	118	17%	117	17%
Chlorbromuron	LC	114	5%	111	11%	119	14%
Chlorbufam	LC	119	9%	114	15%	120	15%
Chlorfenvinphos	LC	119	4%	115	16%	117	11%
Chlorfluazuron	LC	98	11%	111	15%	90	9%
Chloridazon*	LC	97	10%	98	11%		
Chloroxuron	LC	118	7%	117	15%	115	11%
Chlorpyrifos	LC	107	4%	111	14%		
Chlorpyrifos-methyl	LC	117	5%	116	12%	116	9%
Chlorsulfuron*	LC	98	5%	117	15%		
Chlorthiophos	LC	99	6%	96	8%	118	17%
Chromafenozone	LC	118	9%	117	13%	111	4%
Cinidon Ethyl	LC	107	5%	108	12%	106	14%
Cinosulfuron*	LC	113	5%	101	6%		
Clethodim	LC	116	7%	118	13%	116	18%
Clodinafop-propargyl	LC	119	5%	116	14%	109	4%
Clofentazine	LC	114	9%	113	19%	107	12%
Clomazone	LC	118	3%	112	12%	119	14%
Cloquintocet mexyl*	LC	118	8%	115	11%		
Clothianidin*	LC	105	7%	96	15%		
Coumaphos*	LC	119	8%	117	11%		
Coumatetralyl	LC	107	6%	108	14%	102	8%
Cyflufenamid	LC	116	19%	119	18%	119	9%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Cyhalothrin-L*	LC	116	15%	114	12%		
Cymoxanil	LC	119	5%	117	9%	96	19%
Cypermethrin*	LC	106	8%	95	15%		
Cyproconazole	LC	118	4%	113	13%	107	13%
Cyprodinil	LC	102	12%	91	16%	115	9%
Demeton-S-methyl	LC	112	6%	119	17%	119	18%
Demeton-S-methylsulfone*	LC	109	2%	102	6%		
Desmedipham	LC	107	5%	117	14%	109	11%
Diafenthiuron	LC	84	8%	77	16%	100	9%
Diazinon	LC	117	5%	113	10%	117	9%
Dichlofuanid	LC	91	7%	101	12%	82	19%
Dichlorvos	LC	117	10%	111	10%	106	6%
Diclofop methyl*	LC	118	11%	87	17%		
Dicrotophos*	LC	111	4%	96	16%		
Diethofencarb	LC	120	7%	115	18%	114	7%
Difenoconazole	LC	115	4%	117	10%	119	13%
Diflufenican	LC	113	6%	109	8%	117	10%
Dimethachlor	LC	118	4%	116	8%	116	8%
Dimethenamid	LC	118	2%	105	19%	115	6%
Dimethoate	LC	118	8%	102	9%	71	14%
Dimethomorph	LC	119	6%	116	13%	117	12%
Diniconazole	LC	98	13%	114	20%	120	8%
Dinotefuran	LC	86	9%	82	18%	101	9%
Diphacinone*	LC	104	10%	114	14%		
Disulfoton sulfoxide	LC	118	4%	115	16%	99	12%
Disulfoton	LC	119	6%	119	11%	119	10%
Disulfoton_Sulfone	LC	119	14%	116	8%	118	10%
DMF	LC	120	8%	109	9%	102	13%
Dodemorph	LC	71	15%	71	11%	75	8%
Dodine	LC	87	7%	84	14%	84	8%
Edifenphos	LC	116	5%	109	17%	117	6%
Emamectin	LC	101	6%	112	10%	76	10%
EPN*	LC	105	17%	110	6%		
Epoxiconazole	LC	120	7%	118	10%	119	16%
Ethiofencarb Sulfone*	LC	111	5%	113	13%		
Ethiofencarb Sulfoxide*	LC	112	7%	102	10%		
Ethiofencarb	LC	119	4%	100	16%	113	15%
Ethion	LC	120	6%	113	7%	119	7%
Ethirimol*	LC	92	3%	89	11%		
Ethofumesate	LC	119	6%	115	11%	118	11%
Ethoprophos	LC	119	4%	115	6%	115	6%
Etoxazole	LC	102	18%	114	18%	119	12%
Etrimfos	LC	116	7%	116	11%	120	16%
Famoxadone	LC	108	10%	108	11%	116	9%
Fenamidone	LC	118	8%	118	17%	117	14%
Fenamiphos sulfone	LC	116	20%	119	9%	72	14%
Fenamiphos	LC	118	11%	120	7%	120	10%
Fenarimol	LC	115	6%	108	11%	107	19%
Fenbuconazole	LC	117	10%	118	12%	112	12%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Fenfuram	LC	117	7%	113	12%	118	15%
Fenhexamid	LC	117	12%	107	20%	100	17%
Fenoxyprop-p-ethyl	LC	119	6%	110	14%	119	5%
Fenpropathrin	LC	118	4%	118	11%	118	18%
Fenpropidin	LC	110	9%	99	15%	93	14%
Fenpropimorph	LC	92	5%	91	9%	107	7%
Fenpyrazamine	LC	118	11%	117	12%	119	10%
Fenpyroximate	LC	106	8%	115	16%	109	8%
Fenthion Sulfone	LC	119	4%	115	9%	115	15%
Fenthion sulfoxide	LC	117	15%	115	10%	93	18%
Fenthion	LC	120	6%	117	14%	120	14%
Fenvalerate*	LC	99	19%	98	17%		
Fipronil*	LC	119	8%	112	19%		
Flamprop*	LC	112	4%	111	18%		
Flonicamid	LC	111	9%	87	10%	98	16%
Fluazifop-p-butyl*	LC	119	11%	116	8%		
Flubendiamide	LC	119	4%	117	17%	113	16%
Flufenacet	LC	112	4%	119	9%	114	12%
Flufenoxuron	LC	113	7%	106	14%	119	8%
Flumetsulam	LC	76	10%	87	18%	101	14%
Flumeturon*	LC	86	7%	89	17%		
Fluopicolide	LC	119	8%	115	9%	112	13%
Fluquinconazole	LC	115	7%	111	12%	114	6%
Fluroxypyrr meptyl	LC	105	5%	113	6%	119	9%
Fluroxypyrr	LC	102	2%	104	7%	120	8%
Flusilazole	LC	119	8%	116	10%	119	11%
Flutolanil	LC	115	8%	117	12%	119	14%
Flutriafol	LC	120	15%	111	6%	116	19%
Forasulam*	LC	100	8%	98	20%		
Formetanate	LC	117	6%	114	9%	104	18%
Formothion	LC	118	6%	119	9%	107	6%
Fosthiazate	LC	120	7%	114	19%	107	5%
Fuberidazole	LC	115	6%	105	9%	72	13%
Furathiocarb	LC	118	6%	118	9%	85	11%
Halosulfuron methyl	LC	118	9%	116	7%	116	2%
Haloxyfop Ethyl*	LC	115	12%	118	11%		
Heptenophos	LC	118	4%	113	12%	103	13%
Hexaconazole	LC	115	10%	110	17%	116	19%
Hexazinone	LC	120	6%	115	9%	77	9%
Hexythiazox	LC	111	5%	105	9%	97	13%
Imazalil	LC	119	11%	113	11%	96	17%
Imazamethabenz-methyl	LC	117	13%	116	14%	90	18%
Imibenconazole	LC	107	6%	112	14%	117	16%
Imidacloprid	LC	117	6%	102	10%	79	19%
Indoxacarb	LC	117	9%	115	6%	117	18%
Iprobenfos	LC	115	8%	117	7%	104	16%
Iprovalicarb	LC	119	7%	119	9%	120	14%
Isofenphos	LC	97	5%	113	13%	119	19%
Isofenphos-methyl	LC	119	7%	117	9%	119	15%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Isoprothiolane	LC	118	8%	117	8%	119	11%
Isoproturon	LC	117	3%	120	18%	105	18%
Isoxaben	LC	118	8%	117	20%	119	17%
Karbutilate	LC	116	3%	119	9%	71	18%
Kresoxim-methyl	LC	119	5%	119	18%	110	8%
Lenacil	LC	119	9%	120	18%	94	6%
Linuron	LC	118	7%	115	12%	117	10%
Lufenuron	LC	103	10%	82	16%	105	17%
Malaoxon	LC	118	6%	118	12%	116	6%
Malathion*	LC	115	4%	109	20%		
Mandipropamid	LC	118	1%	109	9%	101	6%
Mecarbam	LC	119	4%	114	5%	119	8%
Mefenacet	LC	120	8%	111	20%	120	15%
Mefenpyr diethyl	LC	115	3%	118	8%	118	9%
Mepanipyrim	LC	115	8%	114	12%	118	17%
Mepronil	LC	116	4%	119	19%	118	5%
Metalaxyl	LC	120	13%	116	4%	115	11%
Metamitron*	LC	105	6%	114	6%		
Metazachlor	LC	116	9%	114	12%	114	4%
Metconazole	LC	115	5%	116	16%	102	16%
Methabenzthiazuron	LC	118	14%	119	14%	110	8%
Methacrifos	LC	108	6%	105	19%	93	16%
Methamidophos	LC	86	7%	71	12%	87	19%
Methidathion	LC	117	13%	116	8%	111	6%
Methiocarb Sulfoxide*	LC	118	4%	102	16%		
Methiocarb*	LC	93	9%	102	17%		
Methomyl	LC	112	10%	91	9%	109	5%
Methoprotryne	LC	118	9%	118	8%	115	16%
Methoxyfenozide	LC	117	3%	120	8%	104	12%
Metobromuron	LC	114	4%	118	10%	113	8%
Metolachlor	LC	117	12%	116	8%	119	14%
Metosulam*	LC	114	11%	97	15%		
Metoxuron	LC	117	4%	108	8%	73	18%
Mevinphos	LC	111	5%	119	8%	98	6%
Molinat	LC	112	5%	105	8%	116	12%
Monocrotophos	LC	98	7%	86	11%	120	10%
Monolinuron	LC	113	4%	112	12%	82	8%
Monuron*	LC	114	5%	117	14%		
Myclobutanil	LC	114	9%	116	16%	92	12%
Napropamide	LC	116	5%	114	8%	116	12%
Neburon	LC	114	6%	107	14%	120	19%
Nitenpyram*	LC	79	6%	81	17%		
Novaluron	LC	115	6%	118	18%	119	17%
Nuarimol*	LC	119	4%	108	14%		
Ofurace	LC	119	10%	119	11%	93	6%
Omethoate	LC	91	7%	73	13%	114	18%
Oxadiargyl*	LC	119	10%	119	11%		
Oxadiazon*	LC	111	5%	117	13%		
Oxamyl	LC	99	6%	93	16%	105	7%
Oxasulfuron	LC	114	6%	117	8%	75	12%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Oxycarboxin*	LC	119	3%	114	7%		
Oxydemeton methyl	LC	94	7%	82	12%	77	15%
Paclobutrazol	LC	118	10%	118	19%	103	10%
Paraoxon-ethyl	LC	116	5%	117	13%	119	12%
Paraoxon-methyl	LC	119	5%	117	13%	105	8%
Penconazole	LC	119	9%	114	11%	120	13%
Pencycuron*	LC	72	6%	72	13%		
Pendimethalin*	LC	105	11%	119	15%		
Permethrin	LC	76	8%	106	15%	119	14%
Phenmedipham	LC	117	5%	117	19%	118	12%
Phenthoate	LC	116	8%	118	8%	120	18%
Phorate sulfone	LC	119	6%	120	7%	118	14%
Phorate sulfoxide	LC	120	9%	117	19%	106	16%
Phorate	LC	115	5%	112	10%	120	8%
Phosalone*	LC	118	4%	112	14%		
Phosphamidon	LC	119	7%	116	11%	81	10%
Phoxim	LC	118	13%	120	8%	118	12%
Picolinafen	LC	112	4%	114	16%	113	11%
Picoxytrobin*	LC	120	2%	120	9%		
Piperonyl butoxide*	LC	119	8%	119	7%		
Pirimicarb desmethyl	LC	115	4%	113	8%	80	11%
Pirimicarb	LC	115	15%	117	12%	119	13%
Pirimiphos-ethyl	LC	119	4%	119	9%	115	7%
Pirimiphos-methyl	LC	112	10%	113	11%	119	10%
Prochloraz	LC	119	7%	111	9%	110	16%
Profenofos	LC	110	2%	110	10%	115	16%
Profoxydim-Li	LC	111	15%	108	16%	107	15%
Promecarb	LC	119	13%	113	9%	119	6%
Prometon	LC	118	7%	113	9%	119	6%
Prometryn	LC	117	5%	103	10%	110	8%
Propachlor	LC	119	8%	115	18%	119	9%
Propanil	LC	119	8%	113	9%	116	11%
Propaqizafop	LC	113	5%	111	15%	118	13%
Propargite	LC	117	4%	114	11%	119	17%
Propazine	LC	117	6%	115	11%	116	12%
Propetamphos	LC	119	10%	116	8%	118	18%
Propiconazol	LC	111	4%	111	6%	117	16%
Propoxur	LC	120	5%	118	9%	119	14%
Propyzamide	LC	119	4%	120	14%	115	11%
Proquinazid	LC	84	5%	87	9%	86	16%
Prosulfocarb	LC	120	5%	109	10%	119	7%
Prothioconazole*	LC	94	16%	109	6%		
Pyraclostrobin*	LC	109	8%	106	11%		
Pyraflufen Et	LC	119	18%	119	12%	118	5%
Pyrazofos	LC	120	5%	114	9%	114	7%
Pyrazosulfuron-ethyl	LC	120	11%	119	9%	108	19%
Pyrethrins	LC	117	4%	119	9%	117	20%
Pyridaben	LC	114	5%	118	11%	119	14%
Pyridalyl	LC	92	5%	90	9%	101	8%
Pyridaphenthion	LC	119	7%	117	12%	119	15%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Pyrifenoxyfen	LC	118	3%	117	10%	118	18%
Pyrimethanil	LC	103	8%	107	15%	103	14%
Pyriproxyfen	LC	119	2%	115	8%	116	11%
Pyroxasulam*	LC	105	4%	95	10%		
Quinalphos	LC	118	8%	118	13%	116	10%
Quinoxylfen	LC	92	7%	94	15%	102	14%
Quizalofop-ethyl	LC	114	4%	116	12%	119	19%
Rimsulfuron*	LC	120	9%	106	19%		
Rotenone	LC	119	2%	116	5%	118	14%
Sebuthylazine desethyl	LC	108	14%	109	10%	88	13%
Sebuthylazine	LC	117	4%	103	10%	118	12%
Simazine	LC	113	13%	117	10%	98	9%
Simetryn	LC	101	8%	111	12%	118	13%
Spinetoram	LC	98	18%	95	19%	118	16%
Spinosad-A*	LC	119	17%	113	11%		
Spinosad_D*	LC	115	15%	111	10%		
Spirodiclofen	LC	109	7%	113	11%	119	12%
Spiroxamine	LC	112	5%	95	14%	98	10%
Sulfotep	LC	119	6%	118	16%	113	8%
Tebuconazole	LC	119	8%	115	17%	119	6%
Tebufenozide	LC	114	11%	116	13%	118	10%
Tebufenpyrad	LC	102	5%	107	10%	119	15%
Tebutam	LC	114	13%	117	8%	119	9%
Tebuthiuron	LC	116	14%	114	9%	99	15%
Tepraloxydim	LC	92	14%	103	11%	119	16%
Terbufos*	LC	120	6%	119	7%		
Terbumeton*	LC	120	4%	118	16%		
Terbutylazine*	LC	116	15%	118	11%		
Terbutrym	LC	118	4%	111	12%	113	9%
Tetrachlorvinphos	LC	113	5%	113	9%	118	17%
Tetraconazole	LC	113	14%	113	14%	117	14%
Tetramethrin-NH4	LC	114	4%	118	11%	119	9%
Thiabendazole	LC	111	3%	95	12%	72	15%
Thiacloprid*	LC	112	5%	104	14%		
Thifensulfuron-methyl*	LC	85	14%	83	13%		
Thiobencarb	LC	113	9%	113	13%	113	8%
Thiocyclam HO	LC	100	16%	98	9%	117	19%
Thiodicarb*	LC	114	8%	106	16%		
Thiofanox	LC	118	20%	95	19%	112	15%
Thiophanate-methyl	LC	101	4%	119	9%	71	13%
Tolclofos-methyl	LC	116	5%	118	11%	113	5%
Tolylfluanid*	LC	115	7%	108	15%		
Tralkoxydim	LC	118	4%	116	8%	119	7%
Tri allate	LC	96	7%	108	7%	118	7%
Triadimefon	LC	119	14%	113	12%	119	11%
Triadimenol	LC	93	18%	120	14%	104	10%
Triasulfuron*	LC	117	9%	104	11%		
Triazophos	LC	119	5%	118	18%	111	8%
Triazoxide	LC	116	9%	105	14%	94	19%
Trichlorfon*	LC	101	14%	93	13%		

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Triclopyr-2-butotyl	LC	114	8%	105	11%	119	10%
Tricyclazole	LC	116	5%	95	14%	76	16%
Trietazine	LC	113	8%	115	15%	117	14%
Trifloxystrobin	LC	110	4%	115	12%	117	11%
Triticonazole	LC	112	2%	119	13%	117	6%
Vamidothion*	LC	112	6%	104	9%		
Zoxamide	LC	119	3%	118	10%	119	13%
τ- Fluvalinate	LC	115	3%	114	14%	118	19%
Acrinathrin	GC	78	7%	81	12%	107	14%
Alachlor	GC	110	5%	100	11%	100	4%
Amitraz	GC	80	10%	94	7%	82	11%
Atrazine	GC	110	10%	97	13%	101	7%
Azinphos-ethyl*	GC	82	8%	108	6%		
Azinphos-methyl*	GC	99	7%	101	7%		
Benalaxyl	GC	84	14%	84	9%	103	9%
Bifenthrin*	GC	84	13%	81	11%		
Bifinazate*	GC	103	18%	99	17%		
Bitertanol	GC	84	7%	86	10%	77	10%
Boscalid	GC	74	7%	75	4%	107	9%
Bromophos-ethyl	GC	89	15%	91	13%	95	3%
Bromophos-methyl*	GC	100	8%	96	20%		
Butralin	GC	87	11%	99	18%	74	4%
Cadusafos	GC	95	8%	91	4%	82	10%
Carbofuran	GC	96	15%	89	19%	89	12%
Carbosulfan*	GC	102	9%	107	7%		
Chlорfenapyr	GC	84	9%	86	5%	96	6%
Chlorfenvinphos	GC	95	7%	95	8%	86	14%
Chlorpropham	GC	84	12%	92	7%	110	4%
Chlorpyrifos	GC	72	3%	85	16%	99	4%
Chlorpyrifos-methyl	GC	93	10%	91	17%	102	5%
Chlorthal-dimethyl*	GC	98	11%	103	17%		
Coumaphos*	GC	96	14%	97	14%		
Cyanophos	GC	103	8%	104	4%	107	4%
Cyfluthrin	GC	89	5%	77	5%	97	3%
Cypermethrin	GC	86	17%	84	7%	97	5%
Cyproconazole	GC	84	14%	93	13%	98	10%
Cyprodinil	GC	84	10%	87	1%	93	7%
Cyromazine*	GC	98	6%	106	10%		
DDD pp`-	GC	80	5%	79	14%	104	5%
DDE pp`-	GC	75	6%	75	2%	82	4%
DDT pp`-	GC	85	11%	85	4%	74	12%
Demeton-S- methyl	GC	102	8%	95	4%	99	12%
Diazinon	GC	90	9%	81	6%	100	5%
Dichlofenthion	GC	90	11%	86	10%	96	4%
Dicofol*	GC	79	9%	87	8%		
Diethofencarb*	GC	91	13%	90	15%		
Difenconazol	GC	77	5%	75	2%	87	10%
Dimethachlor	GC	94	4%	85	9%	97	6%
Dimethoate	GC	100	8%	100	9%	96	6%
Diphenylamine	GC	92	14%	90	19%	102	11%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Disulfoton sulfone*	GC	84	8%	78	6%		
Ditalimfos	GC	74	4%	79	10%	85	9%
Endosulfan alpha-	GC	101	15%	88	14%	93	10%
Endosulfan-sulfate	GC	107	15%	108	7%	120	2%
EPN	GC	86	7%	97	7%	91	10%
Ethion	GC	86	9%	81	8%	89	10%
Ethofumesate	GC	89	15%	93	20%	111	3%
Ethoprophos	GC	88	10%	78	7%	93	7%
Ethoxyquin	GC	86	10%	76	8%	88	13%
Etofenprox*	GC	79	7%	79	7%		
Fenarimol	GC	104	8%	104	10%	93	8%
Fenazaquin	GC	80	13%	77	4%	85	16%
Fenbuconazole	GC	83	7%	94	6%	108	5%
Fenitrothion	GC	97	18%	97	19%	91	16%
Fenoxy carb	GC	98	17%	102	18%	107	5%
Fenpropathrin	GC	92	18%	82	16%	96	13%
Fenpropidin	GC	79	18%	83	7%	72	12%
Fenvalerate	GC	77	9%	81	12%	87	10%
Fluazifop-p-butyl	GC	101	8%	88	18%	90	10%
Flucythrinate	GC	88	18%	98	7%	74	8%
Fludioxonil	GC	77	7%	94	10%	101	13%
Fluquinconazole	GC	82	15%	91	10%	113	9%
Formothion I	GC	95	15%	98	5%	113	10%
HCH delta- (Lindane)	GC	93	16%	97	14%	114	5%
HCH alpha-	GC	95	8%	95	4%	116	5%
HCH beta-	GC	109	6%	102	10%	95	6%
HCH delta- (Lindane)	GC	101	9%	96	11%	104	4%
Heptachlor exo-epoxide isomer B	GC	95	12%	89	14%	105	6%
Heptachlor-endo-Epoxide (trans-)	GC	90	19%	100	5%	116	7%
Hexachlorobenzene (HCB)	GC	77	12%	76	5%	76	9%
Hexaconazole	GC	92	18%	85	19%	102	18%
Hexazinone	GC	98	8%	99	4%	109	6%
Iprobenfos	GC	75	4%	76	6%	84	11%
Isofenphos-methyl	GC	108	8%	101	3%	94	3%
lambda-Cyhalothrin I	GC	96	5%	98	9%	97	12%
Linuron*	GC	89	9%	96	11%		
Malathion	GC	98	5%	91	11%	95	7%
Mefenacet*	GC	100	10%	109	6%		
Mefenpyr-diethyl	GC	101	11%	97	14%	107	7%
Metazachlor	GC	97	14%	97	5%	110	5%
Methacrifos	GC	86	16%	84	8%	89	18%
Methidathion	GC	87	8%	92	4%	107	12%
Methiocarb	GC	105	7%	95	6%	76	12%
Metribuzin	GC	112	6%	100	9%	78	15%
Mevinphos	GC	90	15%	87	4%	79	13%
Myclobutanil	GC	87	9%	90	12%	105	3%
Napropamide	GC	84	5%	96	6%	114	10%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Nuarimol	GC	81	8%	83	5%	110	15%
Oxadixyl	GC	92	7%	85	12%	115	3%
Parathion-ethyl	GC	106	9%	102	19%	95	9%
Parathion-methyl	GC	104	11%	100	8%	97	6%
PCB 028*	GC	76	5%	81	8%		
PCB 052*	GC	85	7%	83	13%		
PCB 101*	GC	73	17%	82	15%		
PCB 118*	GC	102	18%	85	14%		
PCB 138*	GC	90	17%	85	13%		
PCB 153*	GC	94	18%	81	13%		
Penconazole	GC	94	8%	93	8%	107	5%
Pendimethalin	GC	71	16%	85	14%	92	5%
Permethrin	GC	76	6%	97	10%	82	6%
Phenthioate	GC	88	15%	84	15%	87	5%
Phenylphenol ortho-(2-phenylphenol) (opp)*	GC	106	2%	106	12%		
Phorate	GC	92	6%	103	19%	94	9%
Phosalone	GC	83	8%	86	10%	112	11%
Phosmet*	GC	96	6%	99	8%		
Piperonyl-butoxide	GC	97	5%	101	4%	84	11%
Pirimicarb	GC	85	7%	81	6%	100	7%
Pirimiphos-ethyl	GC	74	6%	98	18%	91	7%
Pirimiphos-methyl	GC	105	9%	95	16%	95	5%
Procymidone	GC	103	11%	107	7%	119	3%
Profenofos	GC	92	16%	91	14%	107	11%
Prometon	GC	100	7%	93	7%	92	4%
Prometryn	GC	94	9%	100	20%	104	6%
Propachlor	GC	97	9%	101	6%	101	8%
Propazine	GC	103	11%	92	15%	101	6%
Propiconazole	GC	95	8%	103	15%	105	4%
Prosulfocarb	GC	89	8%	89	6%	108	12%
Prothiofos	GC	86	13%	89	18%	92	3%
Pyrazofos	GC	85	11%	80	5%	98	11%
Pyridaben	GC	80	8%	98	9%	80	4%
Pyrimethanil	GC	93	11%	89	16%	108	7%
Pyriproxyfen	GC	80	8%	79	7%	99	8%
Quinalphos	GC	99	8%	101	8%	96	8%
Sulfotep	GC	98	14%	82	12%	101	7%
tau-Flvalinate*	GC	94	10%	95	13%		
Tebuconazole	GC	102	11%	105	13%	79	14%
Tecnazene	GC	95	19%	97	8%	87	11%
Tefluthrin	GC	90	8%	80	7%	99	3%
Terbumeton	GC	91	3%	95	8%	111	7%
Terbutylazine	GC	109	6%	87	16%	115	9%
Terbutryn	GC	87	5%	87	9%	101	7%
Tetraconazole	GC	105	8%	100	18%	109	5%
Tetradifon	GC	100	17%	93	19%	78	10%
Tetramethrin I	GC	84	14%	86	11%	72	16%
Thiometon	GC	100	5%	97	8%	97	6%
Tolclofos-methyl	GC	86	3%	82	7%	114	5%

Pesticides	Instrument	0.1 ml/ kg Level		0.05 ml/ kg Level		0.01 ml/ kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Tolylfluanid	GC	93	10%	86	12%	119	7%
Triadimefon	GC	94	7%	89	10%	109	5%
Triadimenol	GC	98	9%	93	7%	93	9%
Triazophos	GC	93	3%	87	14%	112	16%
Trifloxystrobin	GC	93	11%	93	6%	108	7%
Triflumizole	GC	92	3%	94	15%	94	13%
Trifluralin	GC	98	15%	89	14%	99	4%
Triticonazole	GC	86	14%	93	5%	83	13%
Vinclozolin	GC	94	17%	93	19%	108	6%

Table 5: Compilation of average pesticide recovery results in LC-MS/MS and GC-MS spiked at 0.01, 0.05, and 0.1 mg/kg (6 replicates) in the peanuts matrix.

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Abamectin	LC	109	9%	116	10%	103	8%
Acephate*	LC	91	7%	108	9%		
Acetamiprid	LC	111	3%	103	9%	85	8%
Aldicarb Sulfone*	LC	91	5%	88	10%		
Aldicarb Sulfoxide*	LC	74	5%	71	8%		
Aldicarb	LC	119	14%	106	6%	95	13%
Ametryn	LC	113	6%	109	4%	116	16%
Amidosulfuron*	LC	86	9%	72	20%		
Aminocarb	LC	116	6%	109	7%	116	10%
Anilofos	LC	116	5%	113	7%	119	8%
Atrazine	LC	119	5%	120	7%	118	17%
Azaconazol	LC	115	7%	116	5%	120	14%
Azamethiphos	LC	94	18%	117	7%	102	13%
Azimsulfuron*	LC	112	7%	93	15%		
Azinophos-ethyl	LC	115	4%	107	18%	119	18%
Azinophos-methyl	LC	115	9%	114	9%	120	6%
Azoxystrobin	LC	119	3%	109	9%	119	14%
Barban*	LC	119	12%	117	18%		
Beflubutamid*	LC	119	8%	108	11%		
Benalaxyl	LC	119	3%	117	5%	120	8%
Bendiocarb	LC	108	6%	106	12%	111	20%
Benfuracarb	LC	118	7%	119	18%	119	17%
Bensulfuron-methyl	LC	117	6%	119	11%	86	16%
Benthiavalicarb isopropyl	LC	112	5%	113	11%	115	14%
Benzoximate	LC	114	5%	112	9%	119	11%
Bitertanol	LC	118	11%	105	16%	117	12%
Boscalid	LC	119	8%	112	9%	108	11%
Bromacil	LC	117	5%	112	11%	110	11%
Bromuconazole	LC	113	10%	107	15%	119	15%
Bupirimate	LC	114	10%	116	9%	117	6%
Buprofezin	LC	111	8%	102	7%	117	11%
Butachlor	LC	106	5%	109	7%	120	13%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Butocarboxim sulfoxide	LC	71	3%	71	5%	106	8%
Butocarboxim	LC	118	6%	106	9%	110	15%
Butralin*	LC	106	2%	96	13%		
Butylate	LC	111	7%	105	3%	119	12%
Carbaryl	LC	119	7%	118	10%	118	14%
Carbendazim	LC	105	4%	94	6%	97	8%
Carbetamide	LC	113	4%	107	7%	99	8%
Carbofuran*	LC	119	3%	118	15%		
Carbofuran-3OH	LC	110	4%	99	11%	73	16%
Carboxin	LC	106	8%	102	7%	119	17%
Chlorbromuron	LC	109	6%	102	10%	115	11%
Chlorbufam	LC	117	12%	111	5%	119	20%
Chlorfenvinphos	LC	114	5%	104	7%	117	10%
Chlorfluazuron	LC	89	11%	100	17%	70	20%
Chloridazon	LC	90	7%	86	11%	106	14%
Chloroxuron*	LC	115	11%	98	8%		
Chlorpyrifos*	LC	104	4%	105	3%		
Chlorpyrifos-methyl	LC	110	8%	98	8%	118	14%
Chlorsulfuron	LC	76	5%	71	6%	113	19%
Chlorthiophos*	LC	116	5%	98	7%		
Chromafenozide	LC	115	5%	113	7%	106	14%
Cinidon Ethyl*	LC	114	7%	107	14%		
Cinosulfuron*	LC	96	10%	92	10%		
Clethodim	LC	115	5%	108	9%	113	17%
Clodinafop-propargyl	LC	117	6%	109	9%	119	8%
Clofentazine	LC	109	2%	101	9%	110	6%
Clomazone	LC	109	6%	109	7%	116	9%
Cloquintocet mexyl*	LC	118	4%	118	10%		
Clothianidin*	LC	88	5%	85	9%		
Coumaphos*	LC	113	5%	110	7%		
Coumatetralyl	LC	110	6%	102	4%	100	13%
Cyazofamid	LC	116	6%	119	10%	119	15%
Cyflufenamid	LC	119	9%	117	9%	119	13%
Cyhalothrin-L	LC	107	19%	106	20%	105	9%
Cymoxanil*	LC	114	10%	104	8%		
Cypermethrin*	LC	86	17%	108	15%		
Cyproconazole	LC	104	9%	119	12%	118	19%
Cyprodinil	LC	98	9%	91	9%	108	20%
Demeton-S-methyl	LC	115	9%	104	10%	98	11%
Demeton-S-methylsulfone	LC	95	6%	92	5%	80	11%
Desmedipham	LC	114	6%	114	7%	115	10%
Diafenthuron	LC	89	4%	76	11%	119	16%
Diazinon	LC	115	5%	106	8%	119	8%
Dichlofenthion	LC	116	13%	114	18%	101	18%
Dichlofluanid*	LC	83	6%	71	8%		
Dichlorvos	LC	117	8%	106	4%	106	9%
Dicrotrophos	LC	112	7%	94	7%	80	13%
Diethofencarb	LC	120	4%	119	7%	119	13%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Difenconazole	LC	118	6%	108	12%	119	9%
Diflufenican	LC	116	5%	99	5%	116	13%
Dimethachlor	LC	117	5%	114	9%	118	18%
Dimethenamid	LC	115	7%	116	5%	118	10%
Dimethoate	LC	111	5%	96	5%	93	11%
Dimethomorph	LC	117	7%	108	8%	92	19%
Diniconazole	LC	95	16%	105	8%	115	20%
Dinotefuran	LC	71	2%	72	4%	97	7%
Diphacinone*	LC	89	8%	95	12%		
Disulfoton sulfoxide	LC	120	5%	113	9%	116	13%
Disulfoton	LC	115	6%	109	8%	117	14%
Disulfoton_Sulfone	LC	118	6%	110	12%	116	7%
Diuron	LC	111	6%	117	14%	104	5%
DMF	LC	105	6%	106	13%	110	9%
Dodemorph	LC	72	15%	58	15%	71	3%
Dodine	LC	79	5%	82	16%	82	12%
Edifenphos	LC	120	5%	110	7%	116	11%
Emamectin	LC	99	7%	87	12%	92	15%
Epoxiconazole	LC	113	7%	117	5%	118	14%
Ethiofencarb	LC	110	7%	97	8%	75	10%
Sulfone	LC	110	7%				
Ethiofencarb	LC	102	8%	99	8%	73	11%
Ethiofencarb	LC	119	8%	112	11%	119	20%
Ethion*	LC	118	3%	108	6%		
Ethirimol	LC	104	8%	95	9%	103	11%
Ethofumesate	LC	114	8%	118	9%	118	12%
Ethoprophos	LC	115	8%	108	6%	118	13%
Etoxazole	LC	118	5%	102	20%	117	17%
Etrimesfos	LC	116	1%	113	8%	117	6%
Famoxadone	LC	119	8%	111	9%	105	3%
Fenamidone	LC	113	9%	102	12%	104	14%
Fenamiphos sulfone	LC	117	9%	119	10%	113	17%
Fenamiphos	LC						
Sulfoxide*	LC	115	16%	109	19%		
Fenamiphos	LC	115	6%	107	9%	117	12%
Fenarimol	LC	111	10%	108	9%	111	19%
Fenbuconazole	LC	110	5%	116	7%	118	7%
Fenfuram	LC	111	8%	98	7%	113	18%
Fenhexamid	LC	102	17%	101	12%	119	5%
Fenitrothion	LC	106	16%	82	25%	120	16%
Fenoxyprop-p-ethyl*	LC	111	8%	104	7%		
Fenoxy carb	LC	120	3%	116	6%	118	17%
Fenpropathrin*	LC	109	3%	106	6%		
Fenpropidin	LC	100	14%	79	8%	101	10%
Fenpropimorph	LC	92	5%	88	6%	103	8%
Fenpyrazamine	LC	119	12%	118	18%	118	13%
Fenpyroximate	LC	109	5%	100	9%	119	7%
Fenthion Oxon Sulfone*	LC	114	11%	113	7%		

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Fenthion Sulfone	LC	115	4%	108	7%	120	11%
Fenthion sulfoxide	LC	119	10%	117	9%	104	13%
Fenthion	LC	104	9%	108	5%	99	8%
Flonicamid	LC	96	15%	86	14%	114	14%
Fluazifop-p-butyl*	LC	114	4%	111	13%		
Flubendiamide	LC	112	5%	119	9%	106	14%
Flufenacet	LC	120	4%	112	9%	115	16%
Flufenoxuron	LC	103	8%	102	18%	103	17%
Flumetsulam*	LC	76	10%	71	18%		
Flumeturon*	LC	118	14%	117	14%		
Fluopicolide	LC	119	7%	116	12%	119	16%
Fluquinconazole	LC	109	6%	110	10%	113	11%
Fluroxypyrmethyl	LC	106	5%	98	6%	119	8%
Fluroxypyrr	LC	100	4%	92	6%	119	20%
Flusilazole	LC	110	5%	113	11%	119	14%
Flutolanil	LC	119	8%	118	6%	119	14%
Flutriafol	LC	106	8%	109	19%	101	20%
Forasulam*	LC	95	8%	80	6%		
Formetanate	LC	98	7%	91	8%	97	16%
Formothion	LC	109	5%	104	7%	118	10%
Fosthiazate	LC	116	7%	116	8%	119	12%
Fuberidazole	LC	102	8%	96	12%	96	13%
Furathiocarb	LC	119	6%	116	6%	114	10%
Halosulfuron-methyl*	LC	112	17%	115	10%		
Haloxyfop Ethyl	LC	117	7%	111	9%	115	10%
Heptenophos	LC	100	6%	100	9%	119	20%
Hexaconazole	LC	108	12%	99	9%	102	16%
Hexazinone	LC	116	6%	114	5%	111	11%
Hexythiazox	LC	97	8%	100	8%	94	14%
Imazalil	LC	118	6%	108	8%	107	20%
Imazamethabenz-methyl	LC	117	4%	104	13%	97	7%
Imazamethpyr	LC	77	12%	73	9%	118	15%
Imibenconazole*	LC	116	8%	105	7%		
Imidacloprid	LC	99	12%	94	8%	72	15%
Indoxacarb	LC	100	13%	112	9%	102	9%
Iprobenfos	LC	113	6%	109	5%	106	6%
Iprovalicarb	LC	111	5%	113	9%	110	6%
Isofenphos	LC	103	6%	107	6%	116	7%
Isofenphos-methyl	LC	117	7%	111	8%	113	11%
Isofenphos-oxon	LC	119	7%	117	10%	118	5%
Isoprothiolane	LC	113	7%	99	6%	120	16%
Isoproturon	LC	119	6%	112	4%	119	20%
Isoxaben	LC	116	6%	119	8%	113	10%
Karbutilate	LC	115	4%	111	8%	100	11%
Kresoxim-methyl	LC	119	4%	110	8%	117	8%
Lenacil	LC	107	7%	110	6%	118	17%
Linuron	LC	111	7%	105	5%	120	17%
Lufenuron*	LC	102	16%	113	12%		
Malaoxon	LC	116	7%	118	7%	120	4%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Malathion	LC	113	7%	119	9%	120	10%
Mandipropamid	LC	115	6%	105	14%	117	15%
Mecarbam	LC	112	6%	110	12%	119	7%
Mefenacet	LC	116	4%	112	15%	119	8%
Mefenpyr diethyl	LC	111	6%	105	10%	116	9%
Mepanipyrim	LC	109	4%	100	11%	117	10%
Mepronil	LC	118	7%	116	16%	120	7%
Metaflumizone*	LC	116	8%	90	18%		
Metalaxyl*	LC	119	5%	116	16%		
Metamitron	LC	94	11%	96	8%	85	15%
Metazachlor	LC	116	4%	118	12%	111	14%
Metconazole	LC	106	16%	107	18%	102	17%
Methabenzthiazuron	LC	117	4%	117	8%	116	9%
Methamidophos	LC	72	7%	77	11%	74	18%
Methidathion	LC	115	7%	116	11%	119	13%
Methiocarb Sulfone	LC	107	7%	99	12%	93	9%
Methiocarb Sulfoxide	LC	114	4%	102	7%	72	18%
Methiocarb	LC	117	7%	112	10%	119	15%
Methomyl	LC	116	6%	118	14%	117	19%
Methoprottryne	LC	111	8%	104	5%	118	18%
Methoxyfenozide	LC	117	6%	107	8%	119	13%
Metobromuron	LC	116	4%	111	6%	117	12%
Metolachlor	LC	111	5%	104	11%	112	15%
Metosulam*	LC	94	10%	84	14%		
Metoxuron	LC	105	6%	99	9%	102	8%
Metribuzin	LC	114	9%	100	19%	92	13%
Mevinphos*	LC	110	6%	97	8%		
Molinat	LC	115	4%	98	7%	118	10%
Monocrotophos	LC	87	5%	83	9%	74	11%
Monolinuron	LC	114	3%	111	9%	119	14%
Monuron	LC	114	4%	106	7%	94	7%
Myclobutanil	LC	119	7%	102	16%	118	17%
Napropamide	LC	117	6%	106	7%	119	13%
Neburon	LC	105	6%	98	7%	109	16%
Nitenpyram*	LC	75	9%	72	11%		
Novaluron	LC	113	4%	106	12%	119	16%
Nuarimol*	LC	100	9%	105	17%		
Ofurace	LC	115	8%	108	6%	100	15%
Omethoate*	LC	75	5%	72	6%		
Oxadiargyl*	LC	109	7%	111	12%		
Oxadiazon	LC	111	5%	108	8%		
Oxamyl	LC	93	7%	85	5%	75	10%
Oxasulfuron*	LC	97	12%	84	9%		
Oxycarboxin	LC	112	3%	95	13%	87	8%
Oxydemeton methyl	LC	84	5%	86	14%	73	13%
Pacllobutrazol	LC	110	15%	115	11%	91	13%
Paraoxon-ethyl	LC	118	4%	118	7%	114	9%
Paraoxon-methyl	LC	104	5%	101	15%	89	7%
Parathion-ethyl*	LC	111	8%	112	18%		
Penconazole	LC	116	6%	101	10%	105	14%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Pendimethalin*	LC	115	7%	94	18%		
Permethrin	LC	112	18%	93	10%	71	3%
Phenmedipham	LC	119	5%	111	8%	113	10%
Phenthroate	LC	112	5%	104	6%	113	5%
Phorate sulfone	LC	119	10%	114	13%	119	15%
Phorate sulfoxide	LC	119	6%	119	4%	119	12%
Phorate	LC	118	8%	102	10%	119	16%
Phosalone	LC	113	8%	111	4%	117	19%
Phosmet	LC	113	7%	113	14%	120	19%
Phosphamidon	LC	97	7%	97	8%	89	12%
Phoxim	LC	110	7%	104	8%	119	13%
Picolinafen	LC	102	7%	98	12%	119	20%
Picoxystrobin	LC	115	5%	116	9%	117	10%
Piperonyl butoxide*	LC	107	3%	111	6%		
Pirimicarb desmethyl	LC	111	4%	108	7%	103	7%
Pirimicarb	LC	116	5%	117	3%	118	14%
Pirimiphos-ethyl	LC	116	4%	112	5%	117	15%
Pirimiphos-methyl	LC	118	5%	108	9%	112	20%
Prochloraz	LC	115	6%	103	6%	100	14%
Profenofos	LC	111	4%	99	11%	118	13%
Profoxydim-Li	LC	108	11%	93	11%	112	16%
Promecarb	LC	103	6%	100	6%	119	13%
Prometon	LC	109	6%	107	9%	111	14%
Prometryn	LC	107	7%	103	7%	107	5%
Propachlor	LC	116	4%	117	12%	120	12%
Propanil	LC	119	10%	106	11%	119	16%
Propaquizafop	LC	110	6%	98	15%	117	13%
Propargite	LC	108	4%	108	13%	117	8%
Propazine	LC	116	6%	112	6%	120	14%
Propetamphos	LC	100	6%	111	12%	104	16%
Propiconazol	LC	112	7%	110	7%	102	7%
Propoxur	LC	102	3%	113	13%	119	11%
Propyzamide*	LC	114	6%	114	8%		
Proquinazid	LC	91	8%	78	6%	81	12%
Prosulfocarb*	LC	112	5%	110	8%		
Prothioconazole*	LC	93	8%	94	16%		
Pyraclostrobin	LC	117	4%	101	10%	117	9%
Pyraflufen Et	LC	112	5%	114	6%	116	8%
Pyrazofos	LC	113	6%	110	7%	119	11%
Pyrazosulfuron-ethyl	LC	113	4%	104	7%	72	19%
Pyrethrins*	LC	101	9%	119	13%		
Pyridaben	LC	118	6%	113	5%	119	7%
Pyridalyl	LC	90	3%	83	6%	96	11%
Pyridaphenthion	LC	118	7%	114	6%	119	18%
Pyrifenoxy	LC	111	15%	120	7%	118	15%
Pyrimethanil	LC	97	10%	106	17%	114	19%
Pyriproxyfen	LC	103	4%	109	6%	118	9%
Pyroxsulam*	LC	80	4%	82	8%		
Quinalphos	LC	113	6%	106	9%	110	10%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Quinoxifen	LC	87	3%	87	7%	101	18%
Quizalofop-ethyl	LC	106	4%	92	15%	119	14%
Rimsulfuron*	LC	80	10%	77	16%		
Rotenone	LC	117	8%	101	8%	120	19%
Sebutylazine desethyl	LC	101	13%	100	4%	120	14%
Sebutylazine	LC	109	7%	104	7%	97	12%
Simazine	LC	107	6%	119	9%	115	16%
Simetryn	LC	117	7%	109	6%	116	7%
Spinetoram	LC	107	11%	98	16%	116	10%
Spinosad_A*	LC	110	15%	98	15%		
Spinosad_D*	LC	116	17%	116	12%		
Spirodiclofen	LC	104	7%	105	7%	118	12%
Spiroxamine	LC	92	3%	85	8%	96	8%
Sulfotep	LC	116	7%	107	7%	117	14%
Tebuconazole	LC	119	5%	109	9%	111	14%
Tebufenozide	LC	110	8%	99	9%	114	17%
Tebufenpyrad	LC	101	9%	92	11%	104	8%
Tebutam	LC	113	6%	108	11%	120	19%
Tebuthiuron	LC	116	5%	110	9%	97	7%
Terbufos	LC	112	6%	108	7%	116	14%
Terbumeton	LC	117	6%	103	13%	113	20%
Terbutylazine	LC	112	4%	93	12%	118	8%
Terbutryn	LC	102	4%	103	10%	112	11%
Tetrachlorvinphos	LC	115	5%	104	10%	119	16%
Tetraconazole	LC	117	7%	114	9%	119	19%
Tetramethrin-NH4	LC	116	2%	114	7%	117	10%
Thiabendazole	LC	96	8%	91	8%	80	12%
Thiacloprid	LC	114	6%	102	8%	80	5%
Thiamethoxam*	LC	87	6%	89	13%		
Thiobencarb	LC	108	5%	102	10%	107	15%
Thiofanox	LC	108	16%	94	30%	109	18%
Thiophanate-methyl	LC	102	9%	114	5%	111	12%
Tolclofos-methyl	LC	106	5%	94	11%	108	17%
Tolylfluanid*	LC	98	3%	97	5%		
Tralkoxydim	LC	114	4%	111	7%	112	5%
Tri allate	LC	97	7%	95	17%	104	14%
Triadimefon	LC	115	6%	110	17%	116	18%
Triadiimenol	LC	116	14%	105	13%	108	20%
Triasulfuron*	LC	106	10%	106	7%		
Triazophos	LC	118	6%	118	6%	117	6%
Triazoxide	LC	109	17%	108	17%	94	20%
Trichlorfon	LC	93	8%	87	6%	80	19%
Triclopyr-2-butotyl*	LC	101	8%	104	15%		
Tricyclazole	LC	98	7%	90	4%	74	12%
Trietazine	LC	100	4%	102	6%	110	20%
Trifloxystrobin	LC	106	5%	116	8%	120	18%
Triflumizole	LC	118	10%	117	20%	116	6%
Triflumuron	LC	113	14%	119	10%	119	7%
Triticonazole	LC	116	17%	119	6%	107	9%
Vamidothion	LC	106	7%	101	7%	75	12%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Zoxamide	LC	113	4%	111	8%	117	10%
τ- Fluvalinate	LC	98	7%	110	11%	109	9%
Acrinathrin	GC	79	5%	109	5%	89	10%
Alachlor	GC	93	11%	111	12%	112	6%
Amitraz	GC	97	17%	107	7%	117	13%
Atrazine	GC	100	8%	104	5%	112	3%
Azinphos-methyl*	GC	88	9%	103	7%		
Benalaxyl	GC	85	7%	102	13%	116	11%
Bifenthrin*	GC	83	7%	108	9%		
Bifinazate*	GC	89	19%	110	14%		
Bitertanol	GC	86	2%	106	8%	93	10%
Boscalid	GC	80	8%	94	10%	96	3%
Bromophos-ethyl	GC	96	10%	100	11%	103	4%
Bromophos-methyl	GC	88	7%	90	9%	103	7%
Butralin	GC	97	12%	93	18%	99	3%
Cadusafos	GC	84	12%	109	5%	96	6%
Carbofuran	GC	106	13%	103	13%	92	8%
Carbosulfan*	GC	93	17%	112	5%		
Chlорfenapyr	GC	83	2%	89	17%	97	7%
Chlorfenvinphos	GC	87	4%	94	6%	104	12%
Chlorpropham	GC	102	10%	107	7%	93	8%
Chlorpyrifos	GC	89	10%	86	10%	113	4%
Chlorpyrifos-methyl	GC	83	6%	87	7%	125	6%
Chlorthal-dimethyl*	GC	103	8%	93	4%		
Coumaphos*	GC	86	13%	91	18%		
Cyanophos	GC	101	9%	107	2%	106	15%
Cyfluthrin	GC	97	8%	111	7%	119	16%
Cypermethrin	GC	84	8%	107	3%	119	20%
Cyproconazole	GC	79	5%	101	7%	116	13%
Cyprodinil	GC	80	7%	79	4%	90	10%
Cyromazine*	GC	87	8%	116	2%		
DDD pp'-	GC	82	11%	103	9%	119	8%
DDE pp'-	GC	84	10%	105	7%	95	5%
DDT pp'-	GC	76	7%	105	6%	116	14%
Demeton-S- methyl	GC	93	10%	108	6%	118	14%
Diazinon	GC	103	10%	99	11%	107	1%
Dichlofenthion	GC	78	6%	98	5%	120	4%
Dicofol*	GC	90	12%	92	7%		
Diethofencarb*	GC	84	14%	97	13%		
Difenocconazol	GC	81	7%	105	9%	119	17%
Dimethachlor	GC	89	7%	95	2%	108	3%
Dimethoate	GC	93	7%	98	6%	93	4%
Diphenylamine	GC	85	18%	96	15%	112	9%
Disulfoton sulfone	GC	79	5%	93	5%	110	3%
Ditalimfos	GC	81	15%	95	9%	114	10%
Endosulfan alpha-	GC	99	11%	101	18%	103	7%
Endosulfan-sulfate	GC	84	5%	107	13%	103	7%
EPN	GC	82	5%	103	7%	93	12%
Ethion	GC	78	7%	101	10%	96	7%
Ethofumesate	GC	91	11%	95	9%	119	13%
Ethoprophos	GC	89	8%	105	8%	110	4%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Ethoxyquin	GC	88	6%	91	6%	108	3%
Etofenprox*	GC	77	8%	99	8%		
Fenarimol	GC	102	14%	107	7%	117	17%
Fenazaquin	GC	87	16%	99	10%	120	19%
Fenbuconazole	GC	83	6%	107	5%	93	14%
Fenitrothion	GC	94	11%	92	12%	108	5%
Fenoxy carb	GC	95	10%	103	11%	111	4%
Fenpropothrin	GC	94	13%	98	16%	118	8%
Fenpropidin	GC	79	6%	101	12%	89	11%
Fenvalerate	GC	89	12%	111	8%	103	4%
Fluazifop-p-butyl	GC	81	6%	103	10%	119	9%
Flucythrinate	GC	76	8%	112	4%	118	9%
Fludioxonil	GC	104	3%	96	4%	117	17%
Fluquinconazole	GC	97	10%	93	7%	109	10%
Formothion I	GC	87	17%	97	8%	108	14%
HCH delta- (Lindane)	GC	92	16%	87	10%	109	6%
HCH alpha-	GC	88	8%	102	12%	113	4%
HCH beta-	GC	86	16%	100	9%	118	5%
HCH delta- (Lindane)	GC	85	9%	105	7%	111	6%
Heptachlor exo- epoxide isomer B	GC	88	18%	89	19%	116	2%
Heptachlor-endo- Epoxide (trans-)	GC	82	9%	93	8%	108	5%
Hexachlorobenzene (HCB)	GC	83	9%	87	11%	75	7%
Hexaconazole	GC	98	14%	104	8%	109	12%
Hexazinone	GC	78	8%	92	11%	108	9%
Iprobenfos	GC	82	5%	91	11%	86	5%
Isofenphos-methyl lambda-Cyhalothrin I	GC	101	8%	100	3%	102	5%
Linuron*	GC	95	17%	100	11%		
Malathion	GC	85	6%	92	4%	119	2%
Mefenacet*	GC	84	9%	99	9%		
Mefenpyr-diethyl	GC	85	9%	101	12%	116	7%
Metazachlor	GC	81	6%	86	7%	117	5%
Methacrifos	GC	93	16%	93	4%	114	11%
Methidathion	GC	77	8%	95	9%	116	5%
Methiocarb	GC	85	9%	111	4%	106	14%
Metribuzin	GC	99	8%	112	3%	93	14%
Mevinphos	GC	87	8%	99	7%	86	9%
Myclobutanil	GC	88	11%	108	8%	109	16%
Napropamide*	GC	84	10%	110	4%		
Nuarimol	GC	90	8%	106	6%	114	10%
Oxadixayl	GC	84	4%	105	7%	117	8%
Parathion-ethyl	GC	91	15%	112	8%	105	4%
Parathion-methyl	GC	96	9%	85	9%	101	12%
PCB 028*	GC	99	9%	83	4%		
PCB 052*	GC	89	5%	83	9%		

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
PCB 101*	GC	73	19%	86	9%		
PCB 118*	GC	74	4%	92	19%		
PCB 138*	GC	70	4%	79	6%		
PCB 153*	GC	72	9%	78	7%		
Penconazole	GC	87	9%	96	9%	118	13%
Pendimethalin	GC	86	11%	94	19%	108	12%
Permethrin	GC	86	11%	105	12%	112	10%
Phenthioate	GC	82	9%	89	6%	112	6%
Phenylphenol ortho-(2-phenylphenol) (opp)*	GC	102	7%	104	7%		
Phorate	GC	82	5%	99	4%	112	16%
Phosalone	GC	79	10%	104	6%	102	58%
Phosmet*	GC	91	7%	96	14%		
Piperonyl-butoxide	GC	79	4%	103	14%	118	7%
Pirimicarb	GC	92	6%	92	7%	98	10%
Pirimiphos-ethyl	GC	84	13%	95	18%	97	6%
Pirimiphos-methyl	GC	87	13%	86	3%	106	17%
Procymidone	GC	104	9%	99	8%	117	17%
Profenofos	GC	87	6%	87	17%	104	17%
Prometon	GC	86	7%	100	6%	105	4%
Prometryn	GC	97	14%	99	14%	120	4%
Propachlor	GC	88	20%	97	7%	105	6%
Propazine	GC	84	7%	102	14%	105	17%
Propiconazole	GC	92	4%	101	6%	117	19%
Prosulfocarb	GC	74	5%	87	5%	117	6%
Prothiofos	GC	78	6%	102	7%	96	7%
Pyrazofos	GC	81	8%	106	9%	83	12%
Pyridaben	GC	78	9%	104	5%	117	6%
Pyrimethanil	GC	91	14%	91	4%	109	14%
Pyriproxyfen	GC	77	5%	98	12%	89	8%
Quinalphos	GC	77	7%	94	1%	112	3%
Sulfotep	GC	96	17%	88	13%	112	6%
tau-Flvalinate	GC	82	9%	107	7%	92	17%
Tebuconazole	GC	83	10%	95	12%	95	6%
Tecnazene	GC	97	13%	104	10%	106	3%
Tefluthrin	GC	97	10%	106	3%	105	4%
Terbumeton	GC	86	8%	97	6%	116	5%
Terbutylazine	GC	87	10%	105	6%	107	9%
Terbutryn	GC	85	7%	88	3%	113	11%
Tetraconazole	GC	87	9%	91	8%	117	17%
Tetradifon	GC	86	14%	95	17%	117	18%
Tetramethrin I	GC	84	5%	97	13%	118	8%
Thiometon	GC	96	15%	92	15%	104	5%
Tolclofos-methyl	GC	89	10%	92	12%	116	3%
Tolylfluanid	GC	88	6%	93	10%	112	15%
Triadimefon	GC	98	9%	102	4%	119	12%
Triadimenol	GC	94	8%	96	11%	117	11%
Triazophos	GC	91	13%	106	9%	83	6%
Trifloxystrobin	GC	82	9%	101	4%	113	6%
Triflumizole	GC	90	10%	103	11%	115	7%

Pesticides	Instrument	0.1 ml/kg Level		0.05 ml/kg Level		0.01 ml/kg Level	
		Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)	Average recovery (%)	RSD (%)
Trifluralin	GC	91	12%	110	5%	110	2%
Triticonazole	GC	85	11%	99	14%	101	17%
Vinclozolin	GC	98	19%	100	11%	120	12%

Where * means that these compounds have LOQ equal to 0.05 mg/kg.

4. Conclusion

This study developed a large-scale multi-residue approach in sesame and peanuts for the first time by using LC-MS/MS and GC-MS/MS to determine 377 pesticide residues in sesame and 388 pesticide residues in peanuts. This analytical method provided satisfactory and accepted results according to the rules in Eurachem and SANTE guidelines 11312/2021 and EU MRL specifications. The main benefits of the studied method are a simple-to-use methodology, a wide range of applicability, compensation of the matrix effects through standard addition technique and high accuracy and precision performance. In conformance with the SANTE/11312/2021, the linearity, matrix effect, recovery, and repeatability were examined in sesame and peanuts matrices. The studied method is very useful in the routine determination of pesticide residues in both peanuts and sesame.

Acknowledgments

The authors gratefully acknowledge the use of the facilities, equipments, and resources of the Central Laboratory of Residue Analysis of Pesticides and Heavy Metals in Foods during the period of the development of this paper. The authors would like to thank Dr. Osama El-Sayed Hussein for assisting in the LC/MS/MS results evaluation. The authors would like to thank Dr. Mostafa Soliman and Dr. Mahmoud S Elshabrawy for assisting in the GC/MS/MS results evaluation. The authors also would like to thank Dr. Emad R. Attallah, Dr. Mohamed R. Abd-elmoataal, Dr. Sherif M. Taha and Sherif Alsherbeny Ali for their help. We would like to thank Dr. Mahmoud Hamdy Abdelwahed for reviewing the paper manuscript.

Reference

- Abdelwahed, M.H. ,M.A. Khorshed, A.M. Elmarsafy, M.S. Elshabrawy and E.R. Souaya, 2021. Polar reversed-phase liquid chromatography coupled with triple quadrupole mass spectrometer method for simple and rapid determination of maleic hydrazide residues in some fruits and vegetables. *J Food Analytical Methods*, 14(1): 172-185.
- Abdelwahed, M. H., M.A. Khorshid, A.M. El-Marsafy and E. Souaya, 2019. Validation of short run time LC-ESI (+) MS/MS method for determination of twenty recommended pesticide residues in food based on QuEChERS extraction technique. *International Journal of Environmental Analytical Chemistry*, 99(5): 409-427.
- Anastassiades, M., S.J. Lehota, D. Štajnbaher, and F.J. Schenck, 2003. Fast and easy multiresidue method Employing acetonitrile extraction/partitioning and “dispersive solid-phase extraction” for the determination of pesticide residues in produce. *Journal of AOAC international*, 86(2): 412-431.
- Attallah, E.R., M. Hamdy Abdelwahed, and M.M. Abo-Aly, 2018. Development and validation of multi-residue method for determination of 412 pesticide residues in cotton fiber using GC-MS/MS and LC-MS/MS. *The Journal of The Textile Institute*, 109(1): 46-63.
- Bhatnagar, A.A. Gupta, 1998. Chlorpyriphos, quinalphos, and lindane residues in sesame seed and oil (*Sesamum indicum L.*). *J Bulletin of environmental contamination toxicology*, 60(4): 596-600.
- Boukid, F., 2022. Peanut protein—an underutilised by-product with great potential: a review. *International Journal of Food Science Technology*, 57(9): 5585-5591.
- Campos-Mondragón, M.G.A.C. De La Barca, A. Durán-Prado, L. Campos-Reyes, R. Oliart-Ros, J. Ortega-García, L. Medina-Juárez and O. Angulo, 2009. Nutritional composition of new peanut (*Arachis hypogaea L.*) cultivars. *J Grasas y aceites*, 60(2): 161-167.

- Cortés, J.M., R.M. Toledo, J.S. Villén and A. Vazquez,, 2008. Analysis of pesticides in nuts by online reversed-phase liquid chromatography– gas chromatography using the through-oven transfer adsorption/desorption Interface. *Journal of agricultural*, 56(14): 5544-5549.
- Di, W.U., X. Jing, P. Hongji, J. Hongyun, Z. Xiyue, L. Xueting and F. Lijia,2012. Determination of pyraclostrobin residues in peanut and peanut hull by gel permeation chromatography-solid phase extraction-high performance liquid chromatography. *J Chinese Journal of Pesticide Science*, 14(6): 681-684.
- Dimitrova, R.T., I.I. Stoykova, T.T. Yankovska-Stefanova, S.A. Yaneva, and T.T. Stoyanchev, 2018. Development of analytical method for determination of organochlorine pesticides residues in meat by GC-ECD. *J Revue Méd. Vét*, 169(4-6): 77-86.
- Elshabrawy, M.S., M.A. Khorshid, M. Hamdy Abdelwahed, and M.M. Abo-Aly, 2023. Optimization and evaluation of four multi-residue methods for the determination of pesticide residues in orange oil using LC-MS/MS and GC-MS/MS: a comparative study. *International Journal of Environmental Analytical Chemistry*, 103(16): 4061-4078.
- García-Reyes, J.F., C. Ferrer, M.J. Gómez-Ramos, A.R. Fernández-Alba and A. Molina-Díaz, 2007. Determination of pesticide residues in olive oil and olives. *J Trac trends in analytical chemistry*, 26(3): 239-251.
- Hou, F., P. Teng, F. Liu and W. Wang, 2017. Tebuconazole and azoxystrobin residue behaviors and distribution in field and cooked peanut. *Journal of Agricultural Food Chemistry*, 65(22): 4484-4492.
- Hua ,J., A. Fayyaz, H. Song, M. Tufail and Y. Gai, 2019. Development of a method Sin-QuEChERS for thedetermination of multiple pesticide residues in oilseed samples. *J Quality Assurance Safety of Crops Foods*, 11(6): 511-516.
- Kurt, C., 2018. Variation in oil content and fatty acid composition of sesame accessions from different origins. *J Grasas y aceites*, 69(1): e241-e241.
- Li, L., Z. Zhou, C. Pan, C. Qian, S. Jiang, F. Liu, 2007. Determination of organophosphorus pesticides in soybean oil, peanut oil and sesame oil by low-temperature extraction and GC-FPD. *J Chromatographia*, 66(7): 625-629.
- Liu, Y., D. Shen, S. Li, Z. Ni, M. Ding, C. Ye and F. Tang, 2016. Residue levels and risk assessment of pesticides in nuts of China. *J Chemosphere*, 144 : 645-651.
- Madej, K., T.K. Kalenik and W. Piekoszewski, 2018. Sample preparation and determination of pesticides in fat-containing foods. *J Food chemistry*, 269: 527-541.
- Martinchik, A.N., 2011. Nutritional value of sesame seeds. *J Voprosy pitaniia*, 80(3): 41-43.
- Meng, Z., D. Liu, S. Li, Z. Xu, Q. Deng and Y. Liu, 2023. A fast multi-residue analysis of twenty-four classes of pesticide in sesame (*Sesamum indicum* L.) and their migration into processed products. *J Food Research International*, 173(14):113322.
- Om, A.S., K.W. Chung and Y. Ko, 1998. Pesticide residues in marketed sesame. *J Bulletin of environmental contamination toxicology*, 61(6): 716-721.
- Onsارد, E., 2012. Sesame proteins. *International Food Research Journal*, 19(4):1287-1295.
- Pang, K., C. Cheng, H. Zhao, Y. Ma, B. Dong and J. Hu,2021. Simultaneous analysis and risk assessment of Quinalofop, Acifluorfen, bentazone and its metabolites residues in peanut and straw under field conditions of China. *Microchemical Journal*, 164(6): 105994.
- Panseri, S., P. Biondi, D. Vigo, R. Communod and L. Chiesa,2013. Occurrence of organochlorine pesticides residues in animal feed and fatty bovine tissue. *J Food Industry*, 13: 261-283.
- Papadakis, E.N., Z. Vryzas and E. Papadopoulou-Mourkidou, 2006.Rapid method for the determination of 16 organochlorine pesticides in sesame seeds by microwave-assisted extraction and analysis of extracts by gas chromatography–mass spectrometry. *Journal of Chromatography A*, 1127(1-2): 6-11.
- Park, J. Y., J.-Y. Park, A. Yang, J.-H. Park, A.A. El-Aty, J.-H. Oh, J.-A. Do, K. Kwon, K.-H. Shim, O.-J. Choi and J.-H. Shim, 2012. Separation of multi-class pesticide residues from fatty food matrices prior to analysis using gas chromatography. *Journal of the Korean Society for Applied Biological Chemistry*, 55(4): 541-549.
- Rejczak, T. and T. Tuzimski,2015. A review of recent developments and trends in the QuEChERS sample preparation approach. *J Open Chemistry*, 13(1): 000010151520150109.

- Shettima, S.A., A.M. Gashinge, A.A. Baffa, A.K .Akinlabi and A.S. Abdulkadir, 2023. Investigation Into The Pesticides Residues In Sesame, Millet, Rice, and Guinea Corn Grown Around The Hadejia-Komadugu-Yobe River Area in Yobe State, Nigeria. *Journal of Chemical Society of Nigeria*, 48(3): 447-458.
- Shinde, R., A. Pardeshi, M. Dhanshetty, M. Anastassiades, and K. Banerjee, 2021. Development and validation of an analytical method for the multiresidue analysis of pesticides in sesame seeds using liquid-and gas chromatography with tandem mass spectrometry. *Journal of Chromatography A*, 1652(6): 462346.
- Soliman, M., M.A. Khorshid, A.M. El-Marsafy, M.M. Abo-Aly and T. Khedr, 2019. Determination of 10 pesticides, newly registered in Egypt, using modified QuEChERS method in combination with gas and liquid chromatography coupled with tandem mass spectrometric detection. *International Journal of Environmental Analytical Chemistry*, 99(3): 224-242.
- Song, Y., Z. Nan, Y. Zheng, Y. Jie, W. Ling, S. Li and M. Wei, 2021. Detection of 4 pesticides residues in peanut using QuEChERS-ultra high performance liquid chromatography-tandem mass spectrometry. *Chinese Journal of Pesticide Science*, 23(2): 414-420.
- Wang, G. Q., D.-F. Zhang, S.-F. Wang, Y.-A. Sun and X.-L. Sun, 2009. Determination of organochlorine pesticide residues in sesame seeds by GC-ECD. *J Chromatographia*, 69(11): 1347-1351.
- Wang, J.H. , F. Cai, Y.L. Wang and X.L. Wang, 2009. Pesticide multiresidue analysis of peanuts using automated gel permeation chromatography clean-up/gas chromatography-mass spectrometry. *J Food Additives Contaminants: Part A*, 26(3): 333-339.
- Zhang, D., X. Li, Y. Cao, C. Wang and Y. Xue, 2020. Effect of roasting on the chemical components of peanut oil. *J. Lwt*, 125: 109249.

Appendix:

Table 1S: The LC mass parameters for determination of pesticide residues in sesame and peanut.

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Abamectin_1	890.5	305.3	17.5	81	37	8
Abamectin_2	890.5	567.4	17.5	81	29	10
Acephate_1	184	143	3.3	51	13	10
Acephate_2	184	125	3.3	51	25	5
Acetamiprid_1	223	126	4.41	46	31	6
Acetamiprid_2	223	90	4.41	46	51	6
Aclonifen_1	265.1	248	4.9	61	25	14
Aclonifen_2	265.1	182.1	5	61	39	10
Acrinathrin_1	559	181	16.6	56	45	16
Acrinathrin_2	559	208	16.6	56	37	12
Aldicarb Sulfone_1	223.1	86.2	3.3	66	25	4
Aldicarb Sulfone_2	223.1	76.2	3.4	66	25	14
Aldicarb Sulfoxide_1	207.3	132	3.2	56	11	10
Aldicarb Sulfoxide_2	207.3	89	3.3	56	19	6
Aldicarb_1	208.2	116	6.6	50	20	10
Aldicarb_2	208.2	89	6.6	50	20	10
Ametryn_1	228	186	10	50	35	10
Ametryn_2	228	116	10	50	35	10
Amidosulfuron_1	370.1	261	7.5	51	23	10
Amidosulfuron_2	370.1	218	7.5	51	37	8
Aminocarb_1	209	137	5.9	21	35	10
Aminocarb_2	209	152.1	5.9	21	21	10
Anilofos_1	367.9	125.1	13.1	61	45	8
Anilofos_2	367.9	198.9	13.1	61	21	14
Atrazine_1	216.1	174	8.9	76	25	12
Atrazine_2	216.1	104	8.9	76	41	10
Azaconazol_1	300	159	9	56	65	18
Azaconazol_2	300	231	9	51	27	8
Azamethiphos_1	325	139	7.3	56	43	20
Azamethiphos_2	325	183	7.3	61	51	16
Azimsulfuron_1	425	182	8.1	6	29	8
Azimsulfuron_2	425	156	8.1	6	43	42
Azinophos-ethyl_1	346.3	132	11.3	50	20	10
Azinophos-ethyl_2	346.3	104	11.3	50	20	10
Azinphos-methyl_1	318	132	9.2	50	20	10
Azinphos-methyl_2	318	104	9.2	50	20	10
Azoxystrobin_1	404	372	9.1	50	20	10
Azoxystrobin_2	404	344.2	9.1	50	35	10
Barban_1	258	178	10.3	105.333	15	12
Barban_2	258	143	10.3	105.333	29	12
Beflubutamid_1	356.1	91.1	12.3	51	53	14
Beflubutamid_2	356	162	12.3	71	39	24
Benalaxyll_1	326.3	208.2	13.9	50	20	10
Benalaxyll_2	326.3	148.1	13.9	50	20	10
Bendiocarb_1	224	167	7.6	50	20	10
Bendiocarb_2	224	109.1	7.6	50	20	10
Benfuracarb_1	411	190	15.8	140.667	17	12
Benfuracarb_2	411	102	15.8	140.667	43	10
Bensulfuron-methyl_1	411	149	9	50	35	10
Bensulfuron-methyl_2	411	182.1	9	50	35	10
Benthiavalicarb isopropyl_1	382.1	116.1	10.4	46	39	18
Benthiavalicarb isopropyl_2	382.1	180.1	10.4	46	53	24
Benzoximate_1	364	199	14.9	46	13	10
Benzoximate_2	364	105	14.9	41	43	14
Bifenazate_1	301.125	198.1	10.6	46	13	12
Bifenazate_2	301.125	170.2	10.6	46	29	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Bispyribac_1	431.2	275.1	9.2	51	19	18
Bispyribac_2	431.2	413	9.2	51	23	12
Bitertanol_1	338.3	269.2	14.6	36	13	18
Bitertanol_2	338.3	99.1	14.6	36	23	6
Boscalid_1	343	307.2	9.9	61	29	20
Boscalid_2	343	139.9	9.9	61	27	8
Bromacil_1	261.2	205	7.8	41	19	12
Bromacil_2	261.2	188	7.8	41	41	10
Bromoconazole_1	378	159	11	50	35	10
Bromoconazole_2	378	70	11	50	35	10
Bupirimate_1	317	166.2	12.3	50	35	10
Bupirimate_2	317	210.2	12.3	50	35	10
Buprofezin_1	306.2	116	16.2	50	20	10
Buprofezin_2	306.2	201	16.2	50	35	10
Butachlor_1	312.3	238	16.3	41	11	18
Butachlor_2	312.3	162	16.3	41	33	10
Butocarboxim sulfoxide_1	207.1	132.1	3.3	51	11	8
Butocarboxim sulfoxide_2	207.1	75	3.2	51	19	2
Butocarboxim_1	208	116	6.59	26	11	8
Butocarboxim_2	208	75	3.2	26	31	12
Butralin_1	296	240.1	16.9	51	21	14
Butralin_2	296	222.2	16.9	51	31	14
Butylate_1	218.1	57.1	16.1	46	31	8
Butylate_2	218.1	156.2	16.1	46	17	6
Carbaryl_1	202.1	145.1	8	50	20	10
Carbaryl_2	202.1	127.1	8	50	35	10
Carbendazim_1	192.1	160.1	5.3	50	20	10
Carbendazim_2	192.1	132.1	5.4	50	50	10
Carbetamide_1	237.2	192.1	7.1	56	13	10
Carbetamide_2	237.2	118.1	7.1	56	19	6
Carbofuran_1	222.1	165	7.7	50	20	10
Carbofuran_2	222.1	123	7.7	50	20	10
Carbofuran-3OH_1	238.3	163.1	4.4	66	21	10
Carbofuran-3OH_2	238.3	181.1	4.4	66	15	12
Carbosulfan_1	381.2	118	18.1	50	35	10
Carbosulfan_2	381.2	160	18.1	50	20	10
Carboxin_1	236	142.9	8.1	50	20	10
Carboxin_2	236	93.1	8.1	50	35	10
Chlorbromuron_1	294.9	205.9	10.2	61	31	30
Chlorbromuron_2	293	182.1	10.2	61	27	30
Chlorbufam_1	224	172	9.7	31	11	8
Chlorbufam_2	224	153.9	9.7	31	25	8
Chlofenvinphos_1	359	155	13.8	61	19	10
Chlofenvinphos_2	359	99	13.8	66	45	6
Chlorfluazuron_1	540	383	16.6	50	35	10
Chlorfluazuron_2	540	158	16.6	50	35	10
Chloridazon_1	222	92	4.9	41	43	4
Chloridazon_2	222	104	4.9	71	47	10
Chloroxuron_1	293	182	10.2	61	27	30
Chloroxuron_2	293	204	10.2	56	55	16
Chlorpyrifos_1	349.9	197.9	16.6	66	25	14
Chlorpyrifos_2	349.9	115.1	16.6	66	33	8
Chlorpyrifos-methyl_1	322	124.9	15.5	50	35	10
Chlorpyrifos-methyl_2	322	290	15.3	50	20	10
Chlorsulfuron_1	358.1	141.1	7.1	66	27	6
Chlorsulfuron_2	358.1	167.1	7.1	66	25	8
Chlorthiophos_1	360.9	304.9	16.6	51	29	12
Chlorthiophos_2	360.9	192	16.8	51	47	28

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Chromafenozide_1	395.3	174.9	10.8	46	17	12
Chromafenozide_2	395.3	339.1	10.8	46	11	10
Cinidon Ethyl_1	394.1	348.1	16.1	81	29	12
Cinidon Ethyl_2	394	107	16.1	81	55	12
Cinosulfuron_1	414.1	183	6.1	51	29	6
Cinosulfuron_2	414.1	157	6.1	51	37	6
Clethodim_1	360	268	15.7	50	20	10
Clethodim_2	360	164	15.7	50	20	10
Clodinafop-propargyl_1	350	266	12.5	50	20	10
Clodinafop-propargyl_2	350	91.1	12.6	50	35	10
Clofentazine_1	303	138	15	51	21	8
Clofentazine_2	303	102.1	15	51	53	4
Clomazone_1	240.1	125	9.6	61	27	18
Clomazone_2	240.1	89.1	9.6	61	75	6
Cloquintocet mexyl_1	336.2	238	16.2	71	25	8
Cloquintocet mexyl_2	336.2	192.1	16.2	71	47	28
Clothianidin_1	250	132	4.2	50	30	10
Clothianidin_2	250	169	4.2	50	30	10
Coumaphos_1	363	226.9	13.7	71	43	32
Coumaphos_2	363	307	13.7	66	29	10
Coumatetralyl_1	293.2	175	9.9	66	31	10
Coumatetralyl_2	293.2	91.1	9.9	66	55	4
Cyazofamid_1	325	108	11.3	46	29	18
Cyazofamid_2	325	261	11.3	51	17	12
Cyclohexamid_1	282.4	264.1	18.5	71	15	18
Cyclohexamid_2	282.4	91	18.5	71	69	4
Cyflufenamid_1	413.198	241	14.3	86	33	14
Cyflufenamid_2	413.198	203.1	14.3	86	55	12
Cyfluthrin_1	451.2	191	16.5	46	21	14
Cyfluthrin_2	451.2	127	16.5	46	45	10
Cyhalothrin-L_1	467.2	225	16.5	66	23	14
Cyhalothrin-L_2	467.2	141	16.5	66	40	10
Cymoxanil_1	199.2	128	5.3	56	13	8
Cymoxanil_2	199.2	83	5.3	56	35	6
Cypermethrin_1	433.1	191	16.8	106	23	12
Cypermethrin_2	433.1	127	16.9	106	40	10
Cyproconazole_1	292.1	69.9	11.1	46	37	12
Cyproconazole_2	292.1	125	11.1	46	41	8
Cyprodinil_1	226	93	14.1	50	50	10
Cyprodinil_2	226	118.1	14.1	50	50	10
Cyromazine_1	167	125	5	6	25	8
Cyromazine_2	167	108	5	6	5	8
Deltamethrin_1	523.2	281.1	16.9	111	21	8
Deltamethrin_2	523.2	181	16.9	111	35	10
Demeton-S-methyl_1	231	89	7.9	36	11	6
Demeton-S-methyl_2	231	61	7.9	36	47	8
Demeton-S-methylsulfone_1	263	169.1	3.5	71	23	10
Demeton-S-methylsulfone_2	263	108.9	3.5	71	41	6
Desmedipham_1	301.2	136	8.9	66	33	8
Desmedipham_2	301.2	182.1	8.7	66	13	12
Diafenthiuron_1	385	329.1	16.7	81	29	22
Diafenthiuron_2	385	278.1	16.7	81	43	18
Diazinon_1	305.1	97	14.3	50	50	10
Diazinon_2	305.1	169.1	14.3	50	35	10
Dichlofenthion_1	315.1	259.1	16.4	11	23	16
Dichlofenthion_2	315.1	287	16.4	11	17	18
Dichlofluanid_1	350.1	224	11	50	20	10
Dichlofluanid_2	350.1	123	11	50	35	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Dichlorvos_1	221	127	7.6	76	25	10
Dichlorvos_2	221	109	7.6	76	27	6
Diclofop methyl_1	358	281	16.1	41	23	18
Diclofop methyl_2	358	120	16.1	26	41	6
Dicrotophos_1	238.1	112	3.7	51	21	18
Dicrotophos_2	238.1	193	3.7	51	15	8
Diethofencarb_1	268.3	226.1	9.6	56	13	14
Diethofencarb_2	268.3	124	9.6	56	43	6
Difenoconazole_1	406	251	15.2	50	50	10
Difenoconazole_2	406	337.1	15.2	50	35	10
Diflufenican_1	395.1	266	15.4	50	35	10
Diflufenican_2	395.1	246	15.4	50	50	10
Dimethachlor_1	256	224	9.3	36	25	36
Dimethachlor_2	256	148	9.3	31	43	18
Dimethenamid_1	276.1	244.1	10.3	46	21	14
Dimethenamid_2	276.1	168.2	10.3	46	37	24
Dimethoate_1	230	199	4.8	50	20	10
Dimethoate_2	230	171	4.8	50	20	10
Dimethomorph_1	388	301	10.1	50	35	10
Dimethomorph_2	388	165	10.1	50	50	10
Diniconazole_1	326.1	70	15.2	91	57	12
Diniconazole_2	326.1	159	15.2	91	43	12
Dinotefuran_1	203	129	3.3	56	17	8
Dinotefuran_2	203	157	3.3	56	13	10
Diphacinone_1	341.2	263.1	8.8	56	19	18
Diphacinone_2	341.2	235.1	8.8	56	27	14
Disulfoton sulfoxide_1	291	185	8.3	36	19	12
Disulfoton sulfoxide_2	291	213.1	8.3	36	15	14
Disulfoton_1	275	169	15.6	46	23	8
Disulfoton_2	275	89	15.4	46	13	4
Disulfoton_Sulfone_1	307	96.9	8.4	56	39	4
Disulfoton_Sulfone_2	307	153	8.4	56	19	10
Diuron_1	233.2	72	8.3	71	37	12
Diuron_2	233.2	160	8.3	71	35	10
DMF_1	150	107	7.3	31	29	18
DMF_2	150	106	7.3	91	43	6
Dodemorph_1	282.4	116	10.3	1	35	4
Dodemorph_2	282.4	98.1	10.3	1	49	4
Dodine_1	228.305	57	13.1	26	43	8
Dodine_2	228.305	60	13	26	41	10
Edifenphos_1	311	109	13.3	71	43	8
Edifenphos_2	311	283	13.1	71	19	18
Emamectin_1	886.5	82	15.8	91	125	12
Emamectin_2	886.5	158.1	15.8	91	53	8
EPN_1	324	296	15.4	46	21	12
EPN_2	324	157	15.4	46	39	24
Epoxiconazole_1	330.2	121.1	11.5	51	31	8
Epoxiconazole_2	330.2	101.1	11.5	51	71	4
Esfenvalerate_1	439.2	231.1	18.4	56	17	16
Esfenvalerate_2	439.2	164	18.8	56	15	12
Ethiofencarb Sulfone_1	258.1	200.9	3.9	46	11	10
Ethiofencarb Sulfone_2	258.1	107.2	3.9	46	27	16
Ethiofencarb Sulfoxide_1	242.1	185.1	4	41	13	8
Ethiofencarb Sulfoxide_2	242.1	107	4	41	29	16
Ethion_1	226	164	8.3	41	13	12
Ethion_2	226.1	107.1	8.3	36	21	16
Ethion_1	385	143	16.2	50	35	10
Ethion_2	385	171	16.2	50	20	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Ethirimol_1	210.1	98.1	8.7	66	43	12
Ethirimol_2	210.1	140.1	8.7	66	35	20
Ethofumesate_1	287.2	121.1	9.6	71	23	6
Ethofumesate_2	287.2	259.1	9.6	71	15	16
Ethoprophos_1	243.1	131	11.8	50	20	10
Ethoprophos_2	243.1	97	11.8	50	35	10
Ethoxyquin_1	218	160	9.6	66	49	8
Ethoxyquin_2	218	174	9.6	71	49	8
Etoxazole_1	360	141	16.6	50	30	10
Etoxazole_2	360	113	16.6	50	45	10
Etrimfos_1	293.2	265.1	14	61	25	16
Etrimfos_2	293.2	125	13.9	61	35	8
Famoxadone_1	392	331	13.2	50	20	10
Famoxadone_2	392	238	13.3	50	20	10
Fenamidone_1	312	236	9.8	46	29	14
Fenamidone_2	312	92	9.8	41	73	12
Fenamiphos sulfone_1	336	188	7.6	177.333	37	12
Fenamiphos sulfone_2	336	108	7.6	177.333	53	10
Fenamiphos Sulfoxide_1	320	233	7.4	177.333	33	16
Fenamiphos Sulfoxide_2	320	171	7.5	177.333	31	12
Fenamiphos_1	304.1	217	12.1	50	35	10
Fenamiphos_2	304.1	276.1	12.1	50	20	10
Fenarimol_1	331.1	268.1	11.3	50	35	10
Fenarimol_2	331.1	189	11.3	50	50	10
Fenbuconazole_1	337.3	125	11.4	56	39	6
Fenbuconazole_2	337.3	69.9	11.4	56	47	2
Fenfuram_1	202	109	8.2	61	35	16
Fenfuram_2	202	120	8.2	61	23	4
Fenhexamid_1	302	97	11.1	50	35	10
Fenhexamid_2	302	143	11.1	50	50	10
Fenitrothion_1	278.2	125	8.4	71	31	8
Fenitrothion_2	278	109	7.7	71	45	10
Fenoxyprop-p-ethyl_1	362	288.1	15.9	50	35	10
Fenoxyprop-p-ethyl_2	362	244.2	15.9	50	35	10
Fenoxy carb_1	302.3	88.1	12.1	46	29	6
Fenoxy carb_2	302.3	116.1	12.2	46	17	8
Fenpropathrin_1	350.2	97	16.6	50	35	10
Fenpropathrin_2	350.2	125.1	16.6	50	20	10
Fenpropidin_1	274.2	147.2	8.7	76	43	22
Fenpropidin_2	274.2	117.1	8.7	76	85	6
Fenpropimorph_1	304.4	147.2	15.48	46	41	10
Fenpropimorph_2	304.4	117.1	15.48	46	81	6
Fenpyrazamine_1	332	230	10.6	282	27	22
Fenpyrazamine_2	332	189	10.6	282	37	16
Fenpyroximate_1	422	366	17	50	20	10
Fenpyroximate_2	422	215.1	17	50	35	10
Fenthion Oxon Sulfone_1	295	78	7.68	71	59	6
Fenthion Oxon Sulfone_2	295	217.1	7.68	71	27	14
Fenthion Oxon Sulfoxide_1	279.1	149.1	4.9	51	19	8
Fenthion Oxon Sulfoxide_2	279.1	205.1	4.8	51	11	12
Fenthion Sulfone_1	311.001	109.001	13.3	194	33	10
Fenthion Sulfone_2	311.001	125.001	7.9	194	29	10
Fenthion sulfoxide_1	295.2	109.1	8.2	71	43	8
Fenthion sulfoxide_2	295.2	78.9	7.7	71	67	12
Fenthion_1	279	247.1	13.7	66	19	16
Fenthion_2	279	169	13.7	66	35	10
Fenalvalerate_1	437.3	167.1	17	46	15	10
Fenalvalerate_2	437.3	125	17	46	30	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Fipronil_1	454	368	11.5	50	35	10
Fipronil_2	454	255	11.5	50	50	10
Flamprop_1	322	77	16.43	61	71	12
Flamprop_2	322	172	16.43	61	18	6
Flonicamid_1	230.1	203.1	3.7	41	25	4
Flonicamid_2	230.1	148	3.7	41	37	4
Fluazifop-p-butyl_1	384.1	282.1	15.8	50	35	10
Fluazifop-p-butyl_2	384.1	328	15.8	50	20	10
Flubendiamide_1	683.058	408	11.7	46	13	12
Flubendiamide_2	683.058	274	11.7	46	53	14
Flucythrinate_1	469	199	16.2	56	27	14
Flucythrinate_2	469	181	16.2	51	69	10
Flufenacet_1	364.1	152	11.2	46	31	6
Flufenacet_2	364.1	194.1	11.2	46	17	8
Flufenoxuron_1	489.1	158	16.2	50	35	10
Flufenoxuron_2	489.1	141.1	16.2	50	50	10
Flumetsulam_1	326.2	109.2	3.9	76	79	6
Flumetsulam_2	326.2	129	3.9	76	35	8
Flumeturon_1	233	72	8.3	61	57	2
Flumeturon_2	233	160	8.3	61	39	8
Fluopicolide_1	383.007	173	10.2	61	31	10
Fluopicolide_2	383.007	145	10.2	61	73	8
Fluopyram_1	397	208	16.9	40	35	7
Fluopyram_2	397	173	17	40	42	7
Fluquinconazole_1	376.2	307.1	11	66	37	20
Fluquinconazole_2	376.2	349.1	11	66	29	22
Fluroxypyrr meptyl_1	367	254.9	16.4	31	19	10
Fluroxypyrr meptyl_2	367	209	16.4	31	39	32
Fluroxypyrr_1	255.2	91	16.4	61	75	6
Fluroxypyrr_2	255.2	208.9	16.4	61	23	14
Flusilazole_1	316.1	165.1	11.8	50	35	10
Flusilazole_2	316.1	169.1	11.8	50	35	10
Flutolanil_1	324	262	9.9	50	30	10
Flutolanil_2	324	242	9.9	50	45	10
Flutriafol_1	302	109	8.4	154	39	10
Flutriafol_2	302	123	8.8	154	41	10
Foramsulfuron_1	453	182	7.8	36	45	10
Foramsulfuron_2	453	139	7.8	46	75	26
Forasulam_1	360	129	5.1	137.333	35	10
Forasulam_2	360	192	5.2	134	23	14
Formetanate_1	222.1	165.1	8.8	46	25	24
Formetanate_2	222	165.1	8.8	41	49	8
Formothion_1	258	198.9	15.3	57	42	6
Formothion_2	258	125	15.2	57	31	4
Fosthiazate_1	284.2	228	8.3	51	15	14
Fosthiazate_2	284.2	104	8.3	51	29	6
Fuberidazole_1	185.1	157.2	7	101	35	24
Fuberidazole_2	185.1	65	7	101	69	4
Furathiocarb_1	383.1	195.1	16	51	27	8
Furathiocarb_2	383.1	252	16	51	21	10
Halosulfuron methyl_1	435	182	9.2	96	45	10
Halosulfuron methyl_2	435	83	9.2	26	77	14
Haloxyfop Ethyl_1	434	316.1	15.8	71	33	10
Haloxyfop Ethyl_2	434	288	15.8	71	49	46
Heptenophos_1	251	127	8.9	51	31	8
Heptenophos_2	251	109	8.9	51	45	6
Hexaconazole_1	314	70	14.4	50	50	10
Hexaconazole_2	314	159	14.4	50	50	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Hexazinone_1	253.1	171.2	7.77	56	27	26
Hexazinone_2	253.1	71.1	7.77	56	51	6
Hexythiazox_1	353.1	228	16.5	66	23	16
Hexythiazox_2	353.1	167.9	16.5	66	37	10
Imazalil_1	297	159	9.9	50	35	10
Imazalil_2	297	201	9.8	50	50	10
Imazamethabenz-methyl_1	289	144.2	7.8	61	49	8
Imazamethabenz-methyl_2	289	89.1	7.8	61	91	14
Imazamethpyr_1	290	177	5.2	51	39	10
Imazamethpyr_2	290	245	5.5	71	29	16
Imibenconazole_1	411	125	16.1	101	49	8
Imibenconazole_2	411	171	16.1	96	27	10
Imidacloprid_1	256.2	175.1	4	50	35	10
Imidacloprid_2	256.2	209.2	4	50	35	10
Indoxacarb_1	528	203.1	15.2	86	57	12
Indoxacarb_2	528	56	15.2	86	79	10
Iprobenfos_1	289	247.1	12.8	46	13	12
Iprobenfos_2	289	205.2	12.8	46	17	8
Iprodione_1	330.2	56.2	16.7	76	65	10
Iprodione_2	330.2	244.9	18.3	76	21	16
Iprovalicarb_1	321.2	119.1	11.1	46	23	6
Iprovalicarb_2	321.2	203.1	11.1	46	13	14
Isofenphos_1	346	245	14.9	26	19	12
Isofenphos_2	346	217	14.9	21	45	10
Isofenphos-methyl_1	332	231.1	12.7	31	21	8
Isofenphos-methyl_2	332	273	13	31	9	16
Isofenphos-oxon_1	330.1	229	10.8	26	19	10
Isofenphos-oxon_2	330.1	200.9	10.8	26	37	28
Isoprothiolane_1	291	189	10.4	50	30	10
Isoprothiolane_2	291	231	10.3	50	30	10
Isoproturon_1	207.3	72	8.8	76	33	12
Isoproturon_2	207.3	165.1	8.8	76	21	12
Isoxaben_1	333	165	9.9	56	37	10
Isoxaben_2	333	107	9.9	51	85	4
Karbutilate_1	280.4	181.1	7.5	61	17	12
Karbutilate_2	280.4	72.1	7.5	61	55	2
Kresoxim-methyl_1	314.1	206.1	13.1	56	11	14
Kresoxim-methyl_2	314.1	116.1	13.1	56	19	6
Lenacil_1	235.1	153.1	8.9	41	23	6
Lenacil_2	235	135	8.9	46	53	16
Linuron_1	249.1	160	9.8	50	20	10
Linuron_2	249.1	182.1	9.8	50	20	10
Lufenuron_1	511	141	15.8	50	35	10
Lufenuron_2	511	158	15.8	50	20	10
Malaoxon_1	315.1	127	7.6	50	20	10
Malaoxon_2	315.1	99	7.6	50	35	10
Malathion_1	331	127	10.3	66	19	8
Malathion_2	331	285	10.3	66	11	16
Mandipropamid_1	412.274	328.1	9.6	61	19	8
Mandipropamid_2	412.274	356.1	9.6	61	15	10
Mecarbam_1	330.2	227.1	11.4	36	13	14
Mecarbam_2	330.2	96.9	11.4	36	55	4
Mefenacet_1	299.2	148.1	10.8	36	21	8
Mefenacet_2	299.2	120.1	10.8	36	35	6
Mefenpyr diethyl_1	373	327	14.4	61	23	12
Mefenpyr diethyl_2	373	160	14.4	61	59	22
Mepanipyrim_1	224.1	106.1	11.5	46	37	6
Mepanipyrim_2	224.1	77.1	11.5	46	59	12

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Mepronil_1	270.3	119.1	10.4	56	33	8
Mepronil_2	270.3	91.1	10.4	56	61	4
Metaflumizone_1	507	178	15.7	197.333	33	14
Metaflumizone_2	507	116	15.7	197.333	73	14
Metalaxyl_1	280.2	220.2	8.8	50	20	10
Metalaxyl_2	280.2	160.1	8.8	50	35	10
Metamitron_1	203.1	175.1	5	76	25	12
Metamitron_2	203.1	77.1	5	76	47	12
Metazachlor_1	278.3	134	8.7	36	29	8
Metazachlor_2	278.3	210	8.7	36	15	14
Metconazole_1	320	70	14.6	66	95	10
Metconazole_2	320	125	14.6	61	61	10
Methabenzthiazuron_1	222	165	8.8	36	33	22
Methabenzthiazuron_2	222	150	8.8	36	55	14
Methacrifos_1	241	209	9.1	46	15	8
Methacrifos_2	241	125	9.1	41	33	14
Methamidophos_1	142.2	94	3.3	51	21	16
Methamidophos_2	142.2	112	3.3	51	19	8
Methidathion_1	303	145	9.2	36	13	8
Methidathion_2	303	85.1	9.2	36	31	6
Methiocarb Sulfone_1	275.1	122	4.3	36	35	6
Methiocarb Sulfone_2	275.1	201.3	4.3	71	21	30
Methiocarb Sulfoxide_1	242.1	185	4	71	19	12
Methiocarb Sulfoxide_2	242	122	4	71	39	10
Methiocarb_1	243	169.1	9.9	50	20	10
Methiocarb_2	243	121.1	9.9	50	35	10
Methomyl_1	163.2	88	3.7	46	13	4
Methomyl_2	163.2	106	3.7	46	15	8
Methoprottryne_1	272	240	9.8	91	27	16
Methoprottryne_2	272	170	9.8	91	39	10
Methoxyfenozide_1	369	149	10.1	50	20	10
Methoxyfenozide_2	369	133	10.1	50	35	10
Metobromuron_1	259.2	148.1	8.6	61	21	10
Metobromuron_2	259.2	170.2	8.6	61	25	10
Metolachlor_1	284	252	12.4	61	21	16
Metolachlor_2	284	176	12.4	66	35	10
Metosulam_1	418	175	7.5	50	50	10
Metosulam_2	418	190	7.5	50	35	10
Metoxuron_1	229.2	72	6.2	66	37	12
Metoxuron_2	229.2	156.2	6.2	66	35	10
Metribuzin_1	215.2	187.1	7.9	71	25	18
Metribuzin_2	215.2	60	7.9	71	67	10
Metsulfuron-methyl_1	382.3	167.2	16	51	21	10
Metsulfuron-methyl_2	382.3	199	16.3	51	35	10
Mevinphos_1	225.2	127.1	5.3	46	21	8
Mevinphos_2	225.2	193	5.3	46	11	12
Molinat_1	188	83	11.5	56	31	14
Molinat_2	188	126	11.5	56	19	8
Monocrotophos_1	224	127	3.6	50	20	10
Monocrotophos_2	224	98	3.6	50	20	10
Monolinuron_1	215.2	126	8.3	51	25	8
Monolinuron_2	215.2	148.1	8.3	51	21	10
Monuron_1	199	72	7.6	46	29	10
Monuron_2	199	126	7.6	51	45	14
Myclobutanil_1	289	70	10.3	50	50	10
Myclobutanil_2	289	125	10.3	50	50	10
Napropamide_1	272.1	129.1	11.7	46	25	20
Napropamide_2	272.1	171.1	11.7	46	31	26

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Neburon_1	275	88.2	12.6	61	29	4
Neburon_2	275	114	12.6	61	25	4
Nicosulfuron_1	411	182	7	56	31	10
Nicosulfuron_2	411	106	7	61	51	6
Nitenpyram_1	271	237	3.5	56	31	36
Nitenpyram_2	271	126	3.5	46	57	10
Novaluron_1	493.103	158	15.4	71	29	10
Novaluron_2	493.103	141.1	15.3	71	65	10
Nuarimol_1	315	252	9.6	50	35	10
Nuarimol_2	315	81.1	9.6	50	35	10
O,S-TEPP_1	291.3	64.9	8.3	61	43	10
O,S-TEPP_2	291.3	93	12.9	61	23	6
Ofurace_1	282	254.2	7.6	36	21	10
Ofurace_2	282	160.1	7.6	36	37	24
Omethoate_1	214	183	3.3	50	15	10
Omethoate_2	214	143	3.3	50	20	10
Oxadiargyl_1	340.8	223.1	14.3	81	21	14
Oxadiargyl_2	340.8	151	14.3	81	33	10
Oxadiazon_1	345.3	220	16.2	91	29	14
Oxadiazon_2	345.3	303	16.2	91	17	20
Oxamyl_1	237	72	3.3	50	35	10
Oxamyl_2	237	90.1	3.3	50	20	10
Oxasulfuron_1	407	150	6.6	51	39	22
Oxasulfuron_2	407	107	6.6	56	83	12
Oxycarboxin_1	268	175	5.1	50	20	10
Oxycarboxin_2	268	147	5.1	50	35	10
Oxydemeton methyl_1	247	169	3.4	66	21	16
Oxydemeton methyl_2	247	108.9	3.4	66	37	6
Paclobutrazol_1	296.2	70.1	10.2	117.333	49	10
Paclobutrazol_2	294.1	70	10.2	144	53	10
Paraoxon-ethyl_1	276.2	219.9	8.4	61	23	14
Paraoxon-ethyl_2	276.2	94	8.4	61	53	4
Paraoxon-methyl_1	248	90	6.4	76	37	16
Paraoxon-methyl_2	248	202.2	6.4	76	27	12
Parathion-ethyl_1	292.2	235.9	12.7	71	21	16
Parathion-ethyl_2	292.2	93.8	12.7	71	49	6
Parathion-methyl_1	264.2	125	9.7	81	25	10
Parathion-methyl_2	264.2	232	9.6	81	40	10
Penconazole_1	284	70	13.2	50	35	10
Penconazole_2	284	159	13.2	50	35	10
Pencycuron_1	329.1	125	15.1	50	35	10
Pencycuron_2	329.1	99	15.1	50	35	10
Pendimethalin_1	282.1	212	16.7	50	20	10
Pendimethalin_2	282.1	194	16.7	50	35	10
Permethrin_1	408	183	17.8	50	30	10
Permethrin_2	408	153	17.8	50	45	10
Phenmedipham_1	301.3	136	8.9	71	27	8
Phenmedipham_2	301.3	168.1	8.9	71	13	12
Phenthroate_1	321	79	12.6	50	50	10
Phenthroate_2	321	247.2	12.6	50	20	10
Phorate sulfone_2	293.1	96.9	8.5	56	43	6
Phorate sulfone_21	293.1	170.9	8.5	56	17	10
Phorate sulfoxide_1	277.1	199	8.4	46	15	12
Phorate sulfoxide_2	277.1	97	8.4	46	45	4
Phorate_1	261.2	75	15.2	36	15	12
Phorate_2	261.2	199	15.2	36	11	12
Phosalone_1	368	182	14.8	50	20	10
Phosalone_2	368	111	14.8	50	50	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Phosmet_1	318	159.9	9.2	46	17	10
Phosmet_2	318	133	9.2	46	51	8
Phosphamidon_1	300.1	127.1	6.8	50	35	10
Phosphamidon_2	300.1	174	6.8	50	20	10
Phoxim_1	299	129	14.4	56	17	6
Phoxim_2	299	153	14.4	56	13	8
Picolinafen_1	377	145	16	66	85	14
Picolinafen_2	377	238	16	66	63	30
Picoxystrobin_1	368	205	11.9	31	15	10
Picoxystrobin_2	368	145	11.9	26	39	24
Piperonyl butoxide_1	356	177	16.4	50	30	10
Piperonyl butoxide_2	356	119	16.4	50	45	10
Pirimicarb desmethyl_1	225.1	72.1	5.9	46	31	12
Pirimicarb desmethyl_2	225.1	168.2	5.9	46	21	10
Pirimicarb_1	239.2	72	8.5	50	20	10
Pirimicarb_2	239.2	182.3	8.5	50	20	10
Pirimiphos-ethyl_1	334.1	198	16.3	50	35	10
Pirimiphos-ethyl_2	334.1	182.3	16.3	50	35	10
Pirimiphos-methyl_1	306.2	108	15.2	50	50	10
Pirimiphos-methyl_2	306.2	164	15.2	50	20	10
Prochloraz_1	376	70.1	14.7	50	50	10
Prochloraz_2	376	308	14.7	50	30	10
Profenofos_1	373	302.9	15.9	50	35	10
Profenofos_2	373	144.1	15.9	50	50	10
Profluralin_1	348.1	55.1	16.7	26	31	8
Profluralin_2	348.1	276.1	16.7	26	13	20
Profoxydim-Li_1	466	180	17.1	76	43	10
Profoxydim-Li_2	466	280	17.1	96	21	18
Promecarb_1	208	109.1	10.2	50	20	10
Promecarb_2	208	151.2	10.2	50	20	10
Prometon_1	226	184	9.6	66	29	8
Prometon_2	226	142	9.6	51	43	16
Prometryn_1	242	158	11.6	50	35	10
Prometryn_2	242	200	11.6	50	35	10
Propachlor_1	212.1	169.9	8.9	51	25	24
Propachlor_2	212.1	94.1	8.9	51	43	6
Propamocarb HCl_1	189	102	3.2	50	20	10
Propamocarb HCl_2	189	144.1	3.2	50	20	10
Propanil_1	218.124	162.1	10	56	23	10
Propanil_2	218.124	127.1	10	56	37	8
Propaquizafop_1	444.1	100.1	16.1	51	31	4
Propaquizafop_2	444.1	299	16.1	51	37	10
Propargite_1	368.208	231	16.6	46	15	14
Propargite_2	368.208	174.9	16.6	46	23	12
Propazine_1	230	188	10	66	29	6
Propazine_2	230	146	10	71	55	12
Propazine-2-hydroxy_1	212	170	6.04	66	29	26
Propazine-2-hydroxy_2	212	128	6.04	66	39	18
Propetamphos_1	282	138	10.5	51	27	22
Propetamphos_2	282	156	10.5	51	13	8
Propiconazol_1	342.1	159	14	50	35	10
Propiconazol_2	342.1	69	14	50	35	10
Propoxur_1	210.1	111.1	7.7	50	20	10
Propoxur_2	210.1	168	7.7	50	20	10
Propyzamide_1	256.2	190.1	10.7	51	19	12
Propyzamide_2	256.2	173	10.7	51	31	10
Proquinazid_1	373.167	331.1	17.8	46	21	20
Proquinazid_2	373.167	288.9	17.8	46	37	16

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Prosulfocarb_1	252.1	91	15.9	31	33	12
Prosulfocarb_2	252.1	128	15.9	31	19	4
Prothioconazole Desthio_1	312	125	5.2	140.667	51	10
Prothioconazole Desthio_2	312	70	5.1	140.667	53	10
Prothioconazole_1	344	189	14.1	63.333	29	14
Prothioconazole_2	344	125	14.1	63.333	55	10
Pymetrozine_1	218.2	105.1	3.4	50	35	10
Pymetrozine_2	218.2	79	3.4	50	50	10
Pyraclostrobin_1	388	163	14.2	41	33	10
Pyraclostrobin_2	388	194	14.2	46	33	12
Pyraflufen Et_1	413	339	12.9	61	31	20
Pyraflufen Et_2	413	253	12.9	71	47	14
Pyrazofos_1	374	222	15	50	35	10
Pyrazofos_2	374	194.2	15	50	50	10
Pyrazosulfuron-ethyl_1	415.3	182.1	10.4	71	23	12
Pyrazosulfuron-ethyl_2	415.3	83.1	10.4	71	63	14
Pyrethrins_1	329.1	161.1	16.8	66	15	12
Pyrethrins_2	329.1	77.1	16.8	66	85	12
Pyridaben_1	365.2	147.2	17.5	46	35	10
Pyridaben_2	365.2	309.1	17.5	46	17	8
Pyridalyl_1	490	109	19.3	56	39	8
Pyridalyl_2	492	111	19.3	56	41	8
Pyridaphenthion_1	341.2	189.3	10.4	61	31	12
Pyridaphenthion_2	341.2	92.1	10.4	61	57	4
Pyridate_1	379	207.1	17.7	56	27	8
Pyridate_2	379	351.1	17.9	56	17	14
Pyrifenoxy_1	295	263	11.4	50	35	10
Pyrifenoxy_2	295	93	11.4	50	50	10
Pyrimethanil_1	200.1	107.2	10.4	126	35	20
Pyrimethanil_2	200.1	82	10.4	126	59	10
Pyriproxyfen_1	322.2	96	16.4	50	20	10
Pyriproxyfen_2	322.2	227.3	16.4	50	20	10
Pyroxasulam_1	435	195	7	190.667	35	14
Pyroxasulam_2	435	166	7.1	190.667	51	14
Quinalphos_1	299.2	96.9	13.2	51	43	6
Quinalphos_2	299.2	163	13.2	51	33	10
Quinmerac_1	222	203.9	4.1	41	25	32
Quinmerac_2	222	140.9	4.1	41	55	16
Quinoxifen_1	308	196.9	16.7	71	45	10
Quinoxifen_2	308	162.1	16.7	71	63	10
Quizalofop-ethyl_1	373	299	16	50	20	10
Quizalofop-ethyl_2	373	255	16	50	50	10
Rimsulfuron_1	432	182	7.7	61	39	28
Rimsulfuron_2	432	325	7.7	56	29	10
Rotenone_1	395.2	213.1	12	76	33	14
Rotenone_2	395.2	192.3	12	76	35	12
Sebuthylazine desethyl_1	202.1	79.1	7.5	61	37	10
Sebuthylazine desethyl_2	202.1	146.1	7.5	61	29	20
Sebuthylazine_1	230	104	10	41	49	14
Sebuthylazine_2	230	174	10.2	66	49	12
Simazine_1	202.2	131.9	7.8	61	27	8
Simazine_2	202.2	68.1	7.8	61	47	2
Simetryn_1	214.1	124.1	8.8	66	31	4
Simetryn_2	214	144	8.8	66	33	20
Spinetoram_1	748	142	15.6	27.333	39	14
Spinetoram_2	748	98	15.6	20.667	89	12
Spinosad_1	732	142	14.5	50	30	10
Spinosad_2	732	98	14.5	50	30	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Spinosad_3	746	142	15.5	50	30	10
Spinosad_4	746	98	15.4	50	30	10
Spirodiclofen_1	411.2	71.1	17	61	31	4
Spirodiclofen_2	411.2	313.2	17	61	15	8
Spirotetramate_1	374	302	16.12	90	24	15
Spirotetramate_2	374	330	16.12	90	19	17
Spiroxamine_1	298.3	144.2	9.5	56	29	8
Spiroxamine_2	298.3	100.3	9.5	56	45	6
Sulfotep_1	323	171	13.1	51	25	28
Sulfotep_2	323	295	13.1	56	19	12
Tebuconazole_1	308	70.1	13.3	50	35	10
Tebuconazole_2	308	125	13.3	50	35	10
Tebufenozide_1	353	133	12	50	20	10
Tebufenozide_2	353	297.2	12	50	20	10
Tebufenpyrad_1	334.3	147.2	16	66	35	10
Tebufenpyrad_2	334.3	145	16	66	37	8
Tebutam_1	234	91	12.3	31	21	14
Tebutam_2	234.2	199.2	12.3	46	13	8
Tebuthiuron_1	229.3	172.1	7.9	51	25	12
Tebuthiuron_2	229.3	116.2	7.9	51	37	6
Tepraloxydim_1	342	250	7.8	46	19	24
Tepraloxydim_2	342	166	7.8	46	31	12
Terbufos_1	289.2	57	16.2	41	31	8
Terbufos_2	289.2	103.1	16.2	41	13	6
Terbumeton_1	226.2	170.1	10	61	27	28
Terbumeton_2	226	114	10	51	41	16
Terbutylazine_1	230	174.01	10.2	50	20	10
Terbutylazine_2	230	104.01	10.2	50	50	10
Terbutryl_1	242.2	186	11.9	66	29	30
Terbutryl_2	242.2	96	11.9	66	47	14
Tetrachlorvinphos_1	367	127	12.5	66	47	18
Tetrachlorvinphos_2	367	241	12.5	66	31	38
Tetraconazole_1	372	159	10.9	50	35	10
Tetraconazole_2	372	70	10.9	50	35	10
Tetramethrin-NH4_1	349.3	164.1	16	31	33	12
Tetramethrin-NH4_2	349.3	135.1	16	31	25	8
TFNA-AM_1	191.1	148.1	5	46	29	4
TFNA-AM_2	191.1	98.1	4.8	46	43	4
Thiabendazole_1	202.1	175	6.5	50	35	10
Thiabendazole_2	202.1	131	6.5	50	50	10
Thiacloprid_1	253.2	126	4.9	50	20	10
Thiacloprid_2	253.2	186	4.8	50	20	10
Thiamethoxam_1	292	211	3.6	50	20	10
Thiamethoxam_2	292	181.2	3.6	50	35	10
Thifensulfuron-methyl_1	388.2	167	6.2	61	23	12
Thifensulfuron-methyl_2	388.2	69	6.2	61	125	12
Thiobencarb_1	258.3	125	15.2	61	25	8
Thiobencarb_2	258.3	89	15.2	61	69	16
Thiocyclam HO_1	182	73	3.6	46	35	12
Thiocyclam HO_2	182	137.1	3.6	46	23	10
Thiodicarb_1	355.1	88.1	7.9	50	35	10
Thiodicarb_2	355.1	108	7.9	50	20	10
Thifanox_1	219.1	76	8.5	26	9	4
Thifanox_2	219	61	8.4	21	13	10
Thiophanate-methyl_1	343	151	7.3	50	20	10
Thiophanate-methyl_2	343	192.2	7.3	50	20	10
Tolclofos-methyl_1	301.1	175	15.1	50	35	10
Tolclofos-methyl_2	301.1	269	15.1	50	35	10

ID	Q1 Mass (DA)	Q3 Mass (DA)	Time (min)	DP (volt)	CE (volts)	CXP (volts)
Tolylfluanid_1	364	238	12.9	50	20	10
Tolylfluanid_2	364	137	12.9	50	35	10
Tralkoxydim_1	330.3	284.2	16.7	51	17	18
Tralkoxydim_2	330.3	138.1	16.7	51	29	8
Tri allate_1	304	143	16.8	71	43	8
Tri allate_2	306	145	16.8	81	35	10
Triadimefon_1	294.1	197	10.5	50	20	10
Triadimefon_2	294.1	225	10.5	50	20	10
Triadimenol_1	296.1	70	10.9	41	19	12
Triadimenol_2	298.1	70	10.9	41	37	12
Triasulfuron_1	402	141	6.7	56	39	20
Triasulfuron_2	402	167	6.7	61	37	26
Triazophos_1	314.2	162	10.6	50	20	10
Triazophos_2	314.2	119	10.6	50	50	10
Triazoxide_1	248	124	8.9	197.333	43	10
Triazoxide_2	248	150	8.9	197.333	47	12
Trichlorfon_1	257.2	109.1	4.9	61	25	6
Trichlorfon_2	257.2	220.9	4.9	61	17	14
Triclopyr-2-butotyl_1	356.2	237.7	16.2	66	15	14
Triclopyr-2-butotyl_2	356.2	281.9	16.2	66	17	18
Tricyclazole_1	190.2	136.1	5.8	66	39	8
Tricyclazole_2	190.2	163.1	5.8	66	33	10
Trietazine_1	230	99.1	12	61	39	12
Trietazine_2	230	132	12	61	37	18
Trifloxystrobin_1	409	186	15.4	50	20	10
Trifloxystrobin_2	409	206.2	15.4	50	20	10
Triflumizole_1	346.3	278	15.6	46	15	18
Triflumizole_2	346.3	72.8	15.6	46	25	22
Triflumuron_1	358.9	156	14.1	140.667	23	12
Triflumuron_2	360.9	158	14.1	90.667	23	12
Triforine_1	432.4	387.8	8.7	61	15	12
Triforine_2	435.2	344.7	15.8	46	33	22
Triticonazole_1	318.3	70	11.32	61	53	12
Triticonazole_2	318.3	125	11.32	61	35	8
Vamidothion_1	288.1	146.1	4.3	56	19	6
Vamidothion_2	288.1	118	4.3	56	35	18
Xylylcarb_1	180.2	108.1	5.3	46	39	6
Xylylcarb_2	180.2	123.1	5.1	46	15	8
Zoxamide_1	336	187	14.1	41	35	26
Zoxamide_2	336	159.1	14.1	41	63	22
τ- Fluvalinate_1	503.3	181.1	17	81	35	12
τ- Fluvalinate_2	503.3	208.1	17	81	17	14