Qualitative and Quantitative Determination of Organophosphorus Pesticide Residues in Beans Sold in Karu Market, Nasarawa State, Nigeria

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ABSTRACT

The presence of organophosphorus pesticide residues in food commodities poses significant health risks to consumers and these pesticides are widely used in Nigeria to enhance crop yields. This study determined the presences and levels of organophosphorus pesticide residues in beans sold in Karu Market, Nasarawa State, Nigeria. Bean samples were collected from the market and analyzed using gas chromatography-mass spectrometry (GC-MS) for residues of organophosphorus pesticides, the QuEChERS (quick, easy, cheap, effective, rugged, and safe) method was used for extraction and clean-up of pesticide residues. The analysis detected residues of pesticides such as Ethoprophos, Dichlofenthion, Methyl parathion, Chlorpyrifos, Ronnel, Phosphorodithioic acid, O-(2, 4-dicorophenyl) O-ethyl S-propyl ester and Azinphos-methyl in the bean samples, brown and white beans respectively. Concentrations of some residues exceeded maximum residue limits (MRLs) set by regulatory bodies. Findings highlight potential health risks associated with consumption and underscore the need for regular monitoring and regulation of pesticide use in agricultural practices.

Keywords: Organophosphorus, pesticides, market, significant, health, risks, consumers.

INTRODUCTION

The identification and quantification of pesticide in the food are becoming the public interest. Several researchers [1- 9] analyzed pesticide residues in fruit and vegetables in Greece, India, Spain, China, Japan and other developed country. The use of pesticides has increased because they have rapid action to control the pests and diseases, and are less labor intensive than other pest control methods [10]. Pesticide being toxic can become a potential hazard to the manufacturers, the users and the environment. Pesticide can produce negative impacts, both socially and economically [11]. Extensive use of pesticides has resulted in contamination of vital supplies, air, water, and food, the risk to humans may be short term as well as long term depending on the persistence of the pesticide and the exposure period. Pesticide residue in food has become a consumers' safety issue and the consumers have the right to know how much pesticide get incorporated in the food they eat.

These pesticides can leave behind their presence even when used in with good agricultural practices as residue on the crops they used on [12]. Pesticides are numerous and are classified based on the pest they control, chemical structure and their mode of action. Organophosphate (OP) pesticides refer to group of insecticides or nerve agent acting on the enzyme acetyl cholinesterase. The term is used often to describe virtually all organic phosphorus (V) containing compound especially when dealing with neurotoxin compound [13]. These insecticides are esters, amides or sample derivatives of phosphoric or thiosulphuric acid. Some of the less toxic compound are used as systematic insecticides in animals against internal and external parasites [14]. While organophosphate pesticides (OPPs) are insecticides or nerve agent acting on the enzyme acetyl cholinesterase. Insecticides against internal and external parasites in animals are being treated with some of the less toxic compound of organophosphates [12]. Their toxicity lies in the way they function they can be permanently bound to the group hydroxylating the enzyme, which then prevents acetylcholine esterase from decomposing and gain more amount of acetylcholine (concentrated) at the synapses then leading to a state of hyper arousal, paralysis of the muscles and the main respiratory center. Long-term exposure to pesticides is increasingly

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suspected of being linked to a broad spectrum of medical problems such as cancer, neurotoxic effects, reproductive health concerns and endocrine disruption, particularly for specific [15, 16]. Apart from farmers, those working on the farm and those living within the vicinity of the farm (they are at risk of greatest exposure) that these pesticides are used, consumers are also at risk of these pesticide because their major mode of exposure is through consumption [17] of the agriculture products that this class of pesticides are used on.

Preservation of food is unavoidable due to many reasons. Some foods are available in specific seasons and not in others. Hence, the preservation, processing and storage are vital for the continuous supply of foods during seasons and off-seasons and to areas where they are less available [18]. Beans is a food crop that requires much preservation due to its unavailability in most part of Nigeria, and to effectively control weevils and beetles when storing these products [18, 19], hence, it is most likely to contain high levels of pesticide residues [20]. In the past, cereal farmers and grain merchants have often sought ways to preserve their products, beans inclusive, with the application of red dry peppers rather than pesticides, but today, one means of beans preservation is the use of poisonous chemicals and indiscriminate application not minding the consequences [19]. As a result, most of the crops, including beans have high pesticide residues [20]. To this end, the use of chemicals for beans preservation is alarming with regard to food quality and safety in the country [18]. Several reports show the presence of pesticide residues in several food samples, in Nigeria: beans in Lagos markets [20], and Northeastern Nigeria (Jos, Maidugari) [21, 22]; beans in Ghana [23]; and other food like vegetables in Southwestern Nigeria [24; 25], grains in Bangladesh [26].

Bean - white and brown beans are common leguminous crops and the most important staple plant protein food crops in sub-Sahara Africa [27]. Heavy losses in cowpea production are reported in Africa as a result of high incidence of insect pests and diseases [28]. Farmers who applied insecticides recorded a tenfold increase in yield as compared to uncontrolled plots. Insect pests inflict injury on cowpea plants in the course of their feeding on crop parts and these pests indirectly transmit diseases by acting as vectors of pathogens [29, 30]. As a result, most of the crops have high pesticide residues and here is limited study carried out to identify and measure the level of this different pesticides present. This study aimed to determine the occurrence and concentration levels of selected organophosphorus pesticide residues in white and brown beans sold in major markets in Karu, Nasarawa State, Nigeria.

MATERIALS AND METHODS

Study Area

The research was conducted in the Karu Urban Area, located within Nasarawa State, adjacent to Nigeria's Federal Capital Territory. The area experiences a tropical climate with an average annual rainfall of 1,100–2,000 mm and supports intensive agricultural activity. Major markets sampled were Orange Market, Karu Market, and Mararaba Market, all known for grain trading and storage.

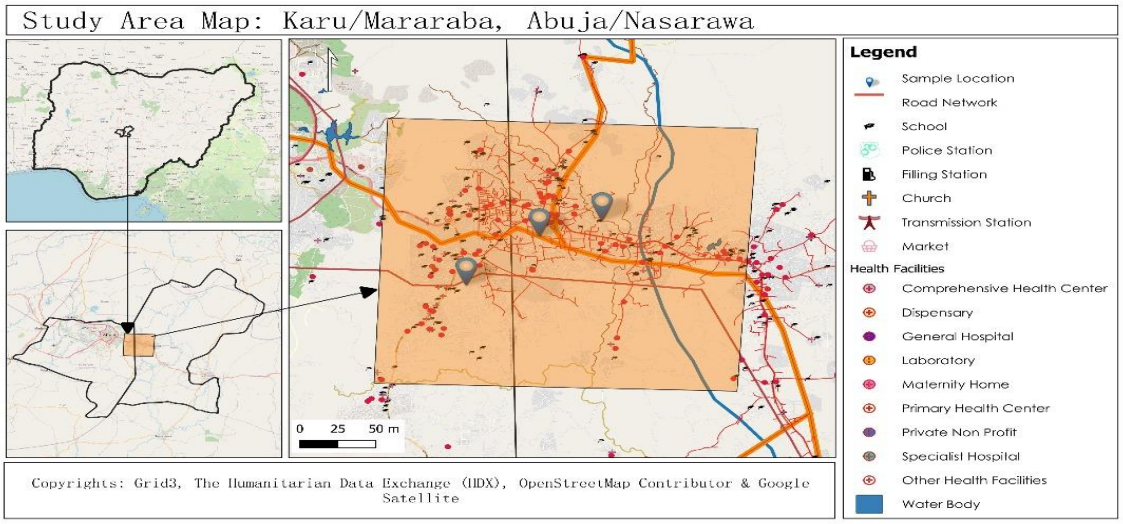

Figure 1: Map of sampling locations in Nasarawa state.

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Sample Collection and Preparation

Six (6) samples of white and brown beans **(***Vigna unguiculata* **(L.) Walp**, of two (2) each were collected randomly from the three markets. Each sample (1 kg) was stored in sealed bags, labelled, and transported to the Analytical Laboratory, Ahmadu Bello University, Zaria for pesticide residue analysis. Samples were cleaned, milled, and homogenized. Precautionary measures were taken to prevent cross-contamination during handling [31]. Clean air tight polythene bags were used to store chopped sample in refrigerator at -20°C until extraction and cleanup process started, milled, and homogenized.

Chemicals and reagents

The standard of Dichlorvos, Oxydisulfoton, Ethoprophos, Dichlofenthion, Methyl parathion, Chlorpyrifos, Ronnel, Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester Azinphos-methyl, were obtained from Sigma Aldrich. Standards of all the pesticides contained >99.6% purity. Methanol, acetone, gradient grade acetonitrile, sodium chloride (NaCl), anhydrous magnesium sulphate ($MgSO_4$) and Primary Secondary Amine (PSA) were purchased too.

Preparation of pesticide standard solution

Pesticide standard stock solutions were prepared separately in acetone at a concentration of 1000 µg/ml and stored at -20°C until use. A mixed standard solution of 50 i in acetone containing all the aforementioned pesticides was prepared by adding the appropriate volume of each individual stock solution in a 50 ml volumetric flask and made to volume by addition of acetone. An intermediate mixed standard solution of 10 µg/ml in acetone was prepared from the mixed standard solution of 50 µg/ml. Then working standard solutions of 0, 50, 100, 150, 200, and 250 µg/ml in acetone were prepared by transferring the appropriate amount from 10 mg/L intermediate mixed standard solution into ten separate 10-mL volumetric flasks. All the standard solutions were kept in a freezer at -20°C until use. Extraction and clean up QuEChERS extraction method is one of the latest extraction and clean up techniques for pesticide residue analysis in food matrices which is an anagram for Quick, Easy, Cheap, Effective, Rugged and Safe. This technique was first introduced by Anastassiades *et al*. [32]. For this study, QuEChERS extraction technique was used for the extraction and clean-up of samples which was modified [34]. The chopped samples were grounded thoroughly with the fruit blender. A representative 10-g portion of thoroughly homogenized sample was weighted in a 50 mL polypropylene centrifuge tube. Then 10 mL of acetonitrile (MeCN) was added into the centrifuge tube. The centrifuge tube was closed properly and shaken vigorously for 30 s by the use of a vortex mixer. Then, 4 g of anhydrous $MgSO_4$ and 1 g of NaCl were added into the centrifuge tube, and it was shaken immediately by the vortex mixer for 1 minute to prevent the formation of magnesium sulfate aggregates. Afterwards, the extract was centrifuged for 5 min at 5000 rpm. An aliquot of 3 mL of the MeCN layer was transferred into a 15 mL micro centrifuge tube containing 600 mg anhydrous $MgSO_4$ and 120 mg Primary Secondary Amine (PSA). Then it was thoroughly mixed by vortex for 30 s and centrifuged for 5 minutes at 4000 rpm. (Laboratory Centrifuges, Sigma-3K30, Germany). After centrifuge, a 1 mL supernatant was filtered by a 0.2 µm PTFE filter, and then it was taken in a clean HPLC vial for injection).

Instruments and equipment for GC-MS determination

The concentrations of OPPs in the sample extracts were determined using an Mass Hunter\GCMS\2\5977\ Agilent HP-5-60 to 325 ^oC GC column (30 m × 320 m x 0.25 m film thickness) attached to a gas chromatograph (6890N Agilent technologies) and a mass selective detector (Agilent 5977) (GC-MS). The volume of the sample injected in the split less mode was 1µL. The initial oven temperature was maintained at 100 ^oC for 2 minutes, then increased to 180 ^oC at a rate of 15 ^oC/minute, ramped up to 300 ^oC at a rate of 3 oC/minute, and held for 9 minutes. The carrier gas was helium with a flow rate of 0.8 mL/min. The operation mode of the mass spectrometer was electron impact ionization with the use of automatic gain control. The storage window was programmed at full scan mode in the range of m/z 200–500, and the selected ion monitoring (SIM) mode was employed in acquiring data by Agilent Chem station software.

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RESULTS AND DISCUSSION

From the six (6) beans sample collected from three (3) markets, nine (9) Organphosphorus pesticides namely: Dichlorvos, Oxydisulfoton, Ethoprophos, Dichlofenthion, Methyl parathion, Chlorpyrifos, Ronnel, Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester Azinphos-methyl were detected using a Mass Hunter\2\5977 GC-MS as shown in **Table 1.** Ibigbami *et al*., [35] in their studies detected varying concentration of Dichlorvos, mevinfos, diazinon, chlorpyrifos, dimethoate and parathion in the bean samples, the percentage of pesticides recovered ranged from 88% to 92%. Abugu *et al*. [36] in their study of eleven organochlorine pesticides (OCP) and seven organophosphate pesticide (OPP) residues were detected in white specie, while twelve OCPs and seven OPPs were found in brown specie. And found that five and six OCP residues were above recommended MRLs in white and brown species respectively. Omokpariola *et al*., [37] studied the levels of organochlorine and organophosphate pesticide residues in selected cereal and legumes crops (beans, cowpea, millet, maize, sorghum and rice) purchased from major markets in Awka, South East Nigeria as well as its health risk. According to studies, [39] about 80% of pesticides used in agriculture have a direct negative impact on the environment. The fate of pesticides in the environment may be completely unknown; however, the biological transport mechanism of pesticides in the environment is more likely to be determined by their physicochemical properties [40]. Factors, such as the chemical structures of pesticides, especially the number of benzene-ring linkages, the position and type of halogen appendages on a benzene ring, the physicochemical properties of their post biodegradation chemical adduct, their solubility, mobility, volatility, and persistence, are predictive of their behaviour and toxic activities in the environment [40]. The instrument was calibrated with concentrations ranging from 0 mg/L to 250 mg/L as shown in the calibration curves in **Table 1 – 3.**

Table1: Qualitative and Quantitative results of Organophosphorus pesticide used for calibration.

Quantitation Results Report

Agilent Technologies

Data File	: BLANK_0.D
Operator	: ABDULLAHI
Acq. Method	: ORGANOPHOSPHORUS PESTICIDE METHOD
Acq. Date-Time	: 8/4/2025 2:50:33 PM
Sample Name:	: CALIBRATION 0 ug/ml
Vial	: 1
Multiplier	: 1
Sample Info	:
DA Method File	:
Tune File	: atune.u
Tune Date	:
Batch Name	: AKINWOLA ADESHOLA.batch.bin
Last Calib Update	: 9/1/2025 7:58:15 PM
Reference Library	:

Compound	RT	Qlon	Resp.	Conc.	Units		Dev(Min)
Internal Standards							
System Monitoring Compounds							
Target Compounds							Qvalue
Dichlorvos	5.150	187.1	118	0.0022	µg/mL		99
Oxydisulfoton	6.372	97.1	8974	2.7321	µg/mL	#	35
Ethoprophos	9.820	158.1	246	0.0006	µg/mL	#	1
Dichlofenthion	0.000		0	N.D.			
Methyl parathion	14.541	93.1	924	0.0087	µg/mL	#	1
Chlorpyrifos	0.000		0	N.D.			
Ronnel	16.818	125.1	9825	0.3821	µg/mL		40
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	0.000		0	N.D.			
Azinphos-methyl	25.795	132.1	69	0.0008	µg/mL	#	1

(#) = Qualifier Out of Range; (m) = Manual Integration; (+) = Area Summed; (*) = Surrogate Percent Recovery Out of Range; (d): Zeroed Peak

Quantitation Results Report

Agilent Technologies

Data File	: Cal_50.D
Operator	: ABDULLAHI
Acq. Method	: ORGANOPHOSPHORUS PESTICIDE METHOD
Acq. Date-Time	: 4/16/2025 1:33:15 PM
Sample Name:	: Calibration 50 ug/ml
Vial	: 2
Multiplier	: 1
Sample Info	:
DA Method File	:
Tune File	: ATUNE.U
Tune Date	:
Batch Name	: AKINWOLA ADESHOLA.batch.bin
Last Calib Update	: 9/1/2025 7:58:15 PM
Reference Library	:

Compound	RT	Qlon	Resp.	Conc.	Units		Dev(Min)
Internal Standards							
System Monitoring Compounds							
Target Compounds							Qvalue
Dichlorvos	5.867	187.1	2311172	43.7402	µg/mL		99
Oxydisulfoton	6.522	97.1	130810	39.8230	µg/mL		99
Ethoprophos	10.206	158.1	20850048	57.0651	µg/mL		92
Dichlofenthion	14.131	279.0	48155	49.0468	µg/mL		92
Methyl parathion	14.456	93.1	4344133	40.7069	µg/mL		96
Chlorpyrifos	16.186	314.1	6569143	44.0342	µg/mL		97
Ronnel	17.217	125.1	917032	35.6696	µg/mL		99
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	18.139	309.1	38762	49.8268	µg/mL		92
Azinphos-methyl	25.335	132.1	2382321	27.9660	µg/mL		90

(#) = Qualifier Out of Range; (m) = Manual Integration; (+) = Area Summed; (*) = Surrogate Percent Recovery Out of Range; (d): Zeroed Peak

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Table2 Qualitative and Quantitative results of Organophosphorus pesticide used for calibration.

Quantitation Results Report

Agilent Technologies

Data File : Cal_100.D
Operator : ABDULLAHI
Acq. Method : ORGANOPHOSPHORUS PESTICIDE METHOD
Acq. Date-Time : 4/16/2025 12:47:35 PM
Sample Name: : Calibration 100 ug/ml
Vial : 2
Multiplier : 1
Sample Info :
DA Method File :
Tune File : ATUNE.U
Tune Date :
Batch Name : AKINWOLA ADESHOLA.batch.bin
Last Calib Update : 9/1/2025 7:58:15 PM
Reference Library :

Compound	RT	QIon	Resp.	Conc.	Units	Dev(Min)
Internal Standards						
System Monitoring Compounds						
Target Compounds						QValue
Dichlorvos	5.873	187.1	5419976	102.5760	µg/mL	100
Oxydisulfoton	6.522	97.1	336678	102.4965	µg/mL	97
Ethoprophos	10.219	158.1	31368117	97.8718	µg/mL	92
Dichlofenthion	14.131	279.0	104366	106.2978	µg/mL	95
Methyl parathion	14.488	93.1	10336033	96.8542	µg/mL	92
Chlorpyrifos	16.212	314.1	15344068	102.8541	µg/mL	96
Ronnel	17.217	125.1	2461768	95.7548	µg/mL	99
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	18.158	309.1	80801	99.8203	µg/mL	99
Azinphos-methyl	25.347	132.1	8239900	96.7279	µg/mL	90

(#) = Qualifier Out of Range; (m) = Manual Integration; (+) = Area Summed; (*) = Surrogate Percent Recovery Out of Range; (d): Zeroed Peak

Quantitation Results Report

Agilent Technologies

Data File : Cal_150.D
Operator : ABDULLAHI
Acq. Method : ORGANOPHOSPHORUS PESTICIDE METHOD
Acq. Date-Time : 4/16/2025 12:01:59 PM
Sample Name: : Calibration 150 ug/ml
Vial : 2
Multiplier : 1
Sample Info :
DA Method File :
Tune File : ATUNE.U
Tune Date :
Batch Name : AKINWOLA ADESHOLA.batch.bin
Last Calib Update : 9/1/2025 7:58:15 PM
Reference Library :

Compound	RT	QIon	Resp.	Conc.	Units	Dev(Min)
Internal Standards						
System Monitoring Compounds						
Target Compounds						QValue
Dichlorvos	5.880	187.1	8304113	157.1598	µg/mL	99
Oxydisulfoton	6.522	97.1	540422	164.5233	µg/mL	99
Ethoprophos	10.238	158.1	37881803	135.9644	µg/mL	90
Dichlofenthion	14.131	279.0	158749	161.6878	µg/mL	96
Methyl parathion	14.513	93.1	16326720	152.9901	µg/mL	94
Chlorpyrifos	16.231	314.1	23882464	160.0886	µg/mL	96
Ronnel	17.223	125.1	4105656	159.6968	µg/mL	98
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	18.152	309.1	124263	151.5047	µg/mL	93
Azinphos-methyl	25.360	132.1	16595447	194.8135	µg/mL	93

(#) = Qualifier Out of Range; (m) = Manual Integration; (+) = Area Summed; (*) = Surrogate Percent Recovery Out of Range; (d): Zeroed Peak

Table 3: Qualitative and Quantitative results of Organophosphorus pesticide used for calibration.

Quantitation Results Report

Agilent Technologies

Data File : Cal_200.D
Operator : ABDULLAHI
Acq. Method : ORGANOPHOSPHORUS PESTICIDE METHOD
Acq. Date-Time : 4/16/2025 11:16:20 AM
Sample Name: : Calibration 200 ug/ml
Vial : 2
Multiplier : 1
Sample Info :
DA Method File :
Tune File : ATUNE.U
Tune Date :
Batch Name : AKINWOLA ADESHOLA.batch.bin
Last Calib Update : 9/1/2025 7:58:15 PM
Reference Library :

Compound	RT	QIon	Resp.	Conc.	Units	Dev(Min)
Internal Standards						
System Monitoring Compounds						
Target Compounds						QValue
Dichlorvos	5.886	187.1	10298627	194.9071	µg/mL	99
Oxydisulfoton	6.522	97.1	625433	190.4035	µg/mL	98
Ethoprophos	10.238	158.1	42512897		µg/mL	91
Dichlofenthion	14.138	279.0	192142	195.6991	µg/mL	100
Methyl parathion	14.526	93.1	21519962	201.6536	µg/mL	98
Chlorpyrifos	16.243	314.1	29873745	200.2492	µg/mL	98
Ronnel	17.223	125.1	5101519	198.4326	µg/mL	100
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	18.158	309.1	163548	198.2225	µg/mL	100
Azinphos-methyl	25.379	132.1	24965836	293.0732	µg/mL	99

(#) = Qualifier Out of Range; (m) = Manual Integration; (+) = Area Summed; (*) = Surrogate Percent Recovery Out of Range; (d): Zeroed Peak

Quantitation Results Report

Agilent Technologies

Data File : Cal_250.D
Operator : ABDULLAHI
Acq. Method : ORGANOPHOSPHORUS PESTICIDE METHOD
Acq. Date-Time : 4/16/2025 10:30:46 AM
Sample Name: : Calibration 250 ug/ml
Vial : 2
Multiplier : 1
Sample Info :
DA Method File :
Tune File : ATUNE.U
Tune Date :
Batch Name : AKINWOLA ADESHOLA.batch.bin
Last Calib Update : 9/1/2025 7:58:15 PM
Reference Library :

Compound	RT	QIon	Resp.	Conc.	Units	Dev(Min)
Internal Standards						
System Monitoring Compounds						
Target Compounds						QValue
Dichlorvos	5.899	187.1	11162098	211.2487	µg/mL	100
Oxydisulfoton	6.535	97.1	723013	220.1103	µg/mL	100
Ethoprophos	9.277	158.1	361669	0.8466	µg/mL	97
Dichlofenthion	14.150	279.0	239663	244.0996	µg/mL	100
Methyl parathion	14.551	93.1	23979294	224.6989	µg/mL	100
Chlorpyrifos	16.262	314.1	36370616	243.7990	µg/mL	100
Ronnel	17.236	125.1	5826523	226.6329	µg/mL	100
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	18.165	309.1	207615	250.6257	µg/mL	100
Azinphos-methyl	25.385	132.1	21783478	255.7156	µg/mL	100

(#) = Qualifier Out of Range; (m) = Manual Integration; (+) = Area Summed; (*) = Surrogate Percent Recovery Out of Range; (d): Zeroed Peak

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Analytical Method Validation

Calibration curve for determination of the analytes

The calibration curves were obtained using analytical solutions of the mixture of the pesticides prepared in pure solvent and prepared in the extract of the matrix in the concentration range [31]. A correlation coefficient of > 0.999 is generally considered acceptable. The y-intercept should be less than a few percent of the response obtained from the target level [32]. Accordingly, the calibration curves were obtained from a running of five point calibration solutions having a concentration range of 50 to 250 µg/ml. The lowest concentration level in the calibration curve was established as a practical determination limit for the instrument. Linearity was evaluated by the calculation of a five-point linear plots of the peak height (as observed in **Figure 1 - 5** representative chromatogram from the five concentration range) against concentration based on linear regression and squared correlation coefficient, r^2, which should be > 0.990. The linearity range and r^2 values are given in **Table 1 - 3**. Prior to the injection of the sample extract, standard solutions of different concentrations of each pesticide group were prepared and injected with suitable instrument parameters. The samples were calibrated (retention time, peak area etc.) against five-pointed calibration curve of standard solution of concerned pesticide. Each peak was characterized by its retention time. Sample results were expressed in µg/ml automatically by the GC-MS software [25].

Linearity of the Standard Curves: Calibration curves have been produced for quantification. Linearity has been observed all along the area of concentration studied depending on the target pesticide chemicals. These ranges of concentrations were selected in function of the sensitivity of the gas chromatography towards each pesticide from the correlation coefficient (r^2) of the linear regression. The calibration curves were obtained by injecting five different concentrations of the pesticide standards in a range of 0, 50 - 250 µg/ml. The r^2 values obtained from the plot of known concentrations of OPPs against their peak areas ranged between 0.9997 and 0.9999 as show in **Figure 1 – 5.**

Limits of Quantification: The limit of determination (LODs) of the OPPs compound ranged from 0.03 to 0.20 µg g^{-1}. Limits of detection (LOD) and limits of quantification (LOQ) of the method were measured by spiked serial dilution of working standards prepared for calibration curves and calculated by considering a value 3 and 10 times of background noise, respectively. LOD was determined considering it as 3 times the signal-to noise ratio, while LOQ was determined as 10 times the signal-to-noise ratio. This means that LOD and LOQ were determined as the lowest concentrations yielding a signal-to-noise (S/N) ratio of 3 and 10, respectively.

Recovery Studies: The cowpea sample was spiked with a solution containing a mixture of the 20 OPPs pesticide standards. A pesticide standard was spiked into a laboratory blank sample to give 0.25 mg/g, and recovery was based on 3 replicates. The spiked samples were left for 1 hour before extraction to allow the insecticide residue to partition into the matrices and the percentage recovery was obtained according to the following formula:

$$\text{Percent recovery} = \frac{\text{Conc in spike sample} - \text{Conc in the unspike sample}}{\text{Amount added}} \times 100$$

Precision

The precision of a method is the measure of agreement or closeness of analyte concentrations to each other when the analyses were performed using identical conditions that is, the same method, same sample, same operator, and same laboratory conditions over a short period of time. This is known as repeatability [32]. Reproducibility is data collection using the sample and the same method but a different operator, another set of laboratory conditions, and a different period of time (days or even weeks).The precision data is shown in **Table 4**.

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Table 4: Precision and Accuracy Studies.

Dichlorvos

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Dichlorvos	Calibration	5.899	11162098	211.2487	250.0000	84.50
Cal_200.D	Dichlorvos	Calibration	5.886	10298627	194.9071	200.0000	97.45
Cal_150.D	Dichlorvos	Calibration	5.880	8304113	157.1598	150.0000	104.77
Cal_100.D	Dichlorvos	Calibration	5.873	5419976	102.5760	100.0000	102.58
Cal_50.D	Dichlorvos	Calibration	5.867	2311172	43.7402	50.0000	87.48
BLANK_0.D	Dichlorvos	Blank	5.150	118	0.0022		

Oxydisulfoton

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Oxydisulfoton	Calibration	6.535	723013	220.1103	250.0000	88.04
Cal_200.D	Oxydisulfoton	Calibration	6.522	625433	190.4035	200.0000	95.20
Cal_150.D	Oxydisulfoton	Calibration	6.522	540422	164.5233	150.0000	109.68
Cal_100.D	Oxydisulfoton	Calibration	6.522	336678	102.4965	100.0000	102.50
Cal_50.D	Oxydisulfoton	Calibration	6.522	130810	39.8230	50.0000	79.65

Ethoprophos

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Ethoprophos	Calibration	9.277	361669	0.8466	250.0000	0.34
Cal_200.D	Ethoprophos	Calibration	10.238	42512897	ND	200.0000	
Cal_150.D	Ethoprophos	Calibration	10.238	37881803	135.9644	150.0000	90.64
Cal_100.D	Ethoprophos	Calibration	10.219	31368117	97.8718	100.0000	97.87
Cal_50.D	Ethoprophos	Calibration	10.206	20850048	57.0651	50.0000	114.13
BLANK_0.D	Ethoprophos	Blank	9.820	246	0.0006		

Dichlofenthion

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Dichlofenthion	Calibration	14.150	239663	244.0996	250.0000	97.64
Cal_200.D	Dichlofenthion	Calibration	14.138	192142	195.6991	200.0000	97.85
Cal_150.D	Dichlofenthion	Calibration	14.131	158749	161.6878	150.0000	107.79
Cal_100.D	Dichlofenthion	Calibration	14.131	104366	106.2978	100.0000	106.30
Cal_50.D	Dichlofenthion	Calibration	14.131	48155	49.0468	50.0000	98.09
BLANK_0.D	Dichlofenthion	Blank			ND		

Methyl parathion

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Methyl parathion	Calibration	14.551	23979294	224.6989	250.0000	89.88
Cal_200.D	Methyl parathion	Calibration	14.526	21519962	201.6536	200.0000	100.83
Cal_150.D	Methyl parathion	Calibration	14.513	16326720	152.9901	150.0000	101.99
Cal_100.D	Methyl parathion	Calibration	14.488	10336033	96.8542	100.0000	96.85
Cal_50.D	Methyl parathion	Calibration	14.456	4341133	40.7069	50.0000	81.41
BLANK_0.D	Methyl parathion	Blank	14.541	924	0.0087		

Chlorpyrifos

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Chlorpyrifos	Calibration	16.262	36370616	243.7990	250.0000	97.52
Cal_200.D	Chlorpyrifos	Calibration	16.243	29873745	200.2492	200.0000	100.12
Cal_150.D	Chlorpyrifos	Calibration	16.231	23882464	160.0886	150.0000	106.73
Cal_100.D	Chlorpyrifos	Calibration	16.212	15344068	102.8541	100.0000	102.85
Cal_50.D	Chlorpyrifos	Calibration	16.186	6569143	44.0342	50.0000	88.07
BLANK_0.D	Chlorpyrifos	Blank			ND		

Ronnel

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Ronnel	Calibration	17.236	5826523	226.6329	250.0000	90.65
Cal_200.D	Ronnel	Calibration	17.223	5101519	198.4326	200.0000	99.22
Cal_150.D	Ronnel	Calibration	17.223	4105656	159.6968	150.0000	106.46
Cal_100.D	Ronnel	Calibration	17.217	2461768	95.7548	100.0000	95.75
Cal_50.D	Ronnel	Calibration	17.217	917032	35.6696	50.0000	71.34
BLANK_0.D	Ronnel	Blank	16.818	9825	0.3821		

Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	Calibration	18.165	207615	250.6257	250.0000	100.25
Cal_200.D	Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	Calibration	18.158	163548	198.2225	200.0000	99.11
Cal_150.D	Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	Calibration	18.152	124263	151.5047	150.0000	101.00
Cal_100.D	Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	Calibration	18.158	80801	99.8203	100.0000	99.82
Cal_50.D	Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	Calibration	18.139	38762	49.8268	50.0000	99.65
BLANK_0.D	Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	Blank			ND		

Azinphos-methyl

Data File	Compound	Sample Type	RT	Resp.	Final Conc	Exp. Conc	Accuracy
Cal_250.D	Azinphos-methyl	Calibration	25.385	21783478	255.7156	250.0000	102.29
Cal_200.D	Azinphos-methyl	Calibration	25.379	24965836	293.0732	200.0000	146.54
Cal_150.D	Azinphos-methyl	Calibration	25.360	16595447	194.8135	150.0000	129.88
Cal_100.D	Azinphos-methyl	Calibration	25.347	8239900	96.7279	100.0000	96.73
Cal_50.D	Azinphos-methyl	Calibration	25.335	2382321	27.9660	50.0000	55.93
BLANK_0.D	Azinphos-methyl	Blank	25.795	69	0.0008		

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Quantitative Analysis Complete Report

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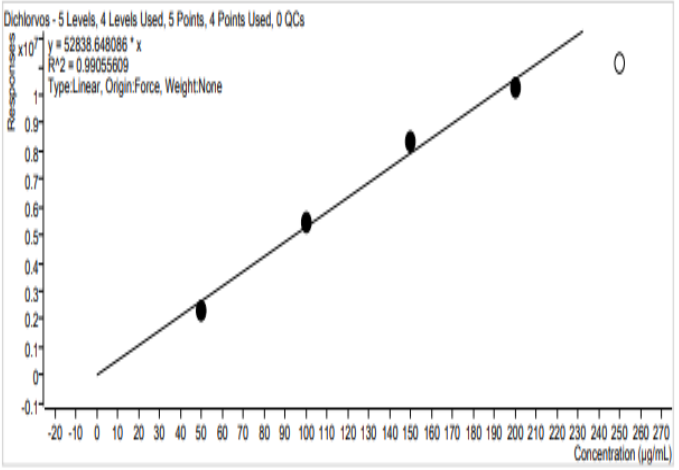
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00

Dichlorvos



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Quantitative Analysis Complete Report

Agilent Technologies

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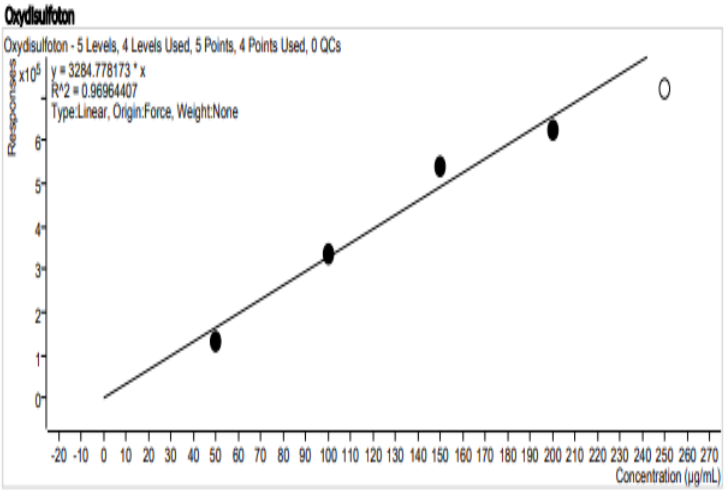
Quant Batch Version	B.07.00	Quant Report Version	B.07.00

Oxydisulfoton



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D:\MassHunter\GCMS\2\5977\akinwola\Cal_100.D	Calibration	L4	x	336678	100.0000	3366.7829
D:\MassHunter\GCMS\2\5977\akinwola\Cal_150.D	Calibration	L3	x	540422	150.0000	3602.8162
D:\MassHunter\GCMS\2\5977\akinwola\Cal_200.D	Calibration	L2	x	625433	200.0000	3127.1670
D:\MassHunter\GCMS\2\5977\akinwola\Cal_250.D	Calibration	L1		723013	250.0000	2892.0536

Figure 1a &b: Calibration for Dichlorvos & Oxydisulfonton**.**

Quantitative Analysis Complete Report

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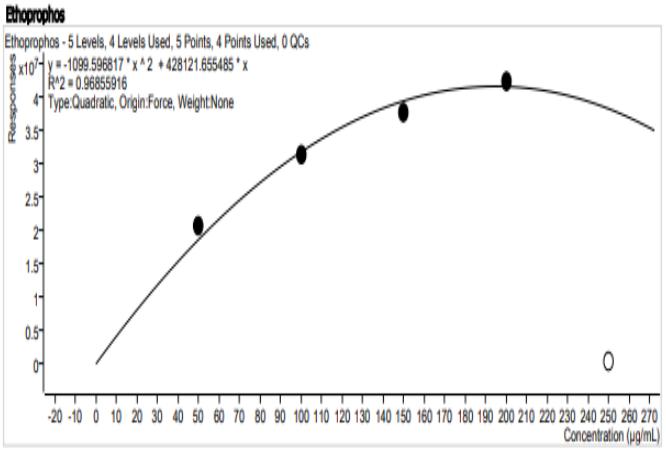
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00

Ethoprophos



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D:\MassHunter\GCMS\2\5977\akinwola\Cal_150.D	Calibration	L3	x	37881803	150.0000	252545.3528
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Quantitative Analysis Complete Report

Agilent Technologies

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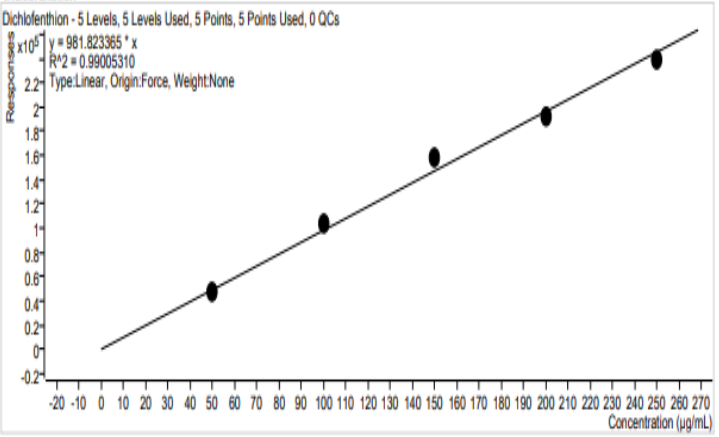
Quant Batch Version	B.07.00	Quant Report Version	B.07.00

Dichlofenthion



Calibration STD Path	Cal Type	Level	Enabled	Resp.	Exp. Conc	Resp. Factor
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D:\MassHunter\GCMS\2\5977\akinwola\Cal_100.D	Calibration	L4	x	104366	100.0000	1043.6571
D:\MassHunter\GCMS\2\5977\akinwola\Cal_150.D	Calibration	L3	x	158749	150.0000	1058.3255
D:\MassHunter\GCMS\2\5977\akinwola\Cal_200.D	Calibration	L2	x	192142	200.0000	960.7098
D:\MassHunter\GCMS\2\5977\akinwola\Cal_250.D	Calibration	L1	x	239663	250.0000	958.6506

Figure 2a &b: Calibration for Ethoprophos & Dichlofenthion**.**

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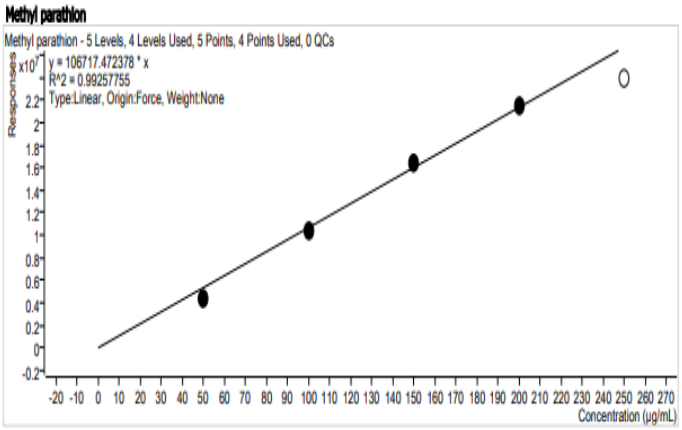
Quant Batch Version	B.07.00	Quant Report Version	B.07.00

Methyl parathion



Calibration STD Path	Cal Type	Level	Enabled	Resp.	Exp. Conc	Resp. Factor
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D:\MassHunter\GCMS\2\5977\akinwola\Cal_200.D	Calibration	L2	x	21519962	200.0000	107599.8100
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Quantitative Analysis Complete Report

Agilent Technologies

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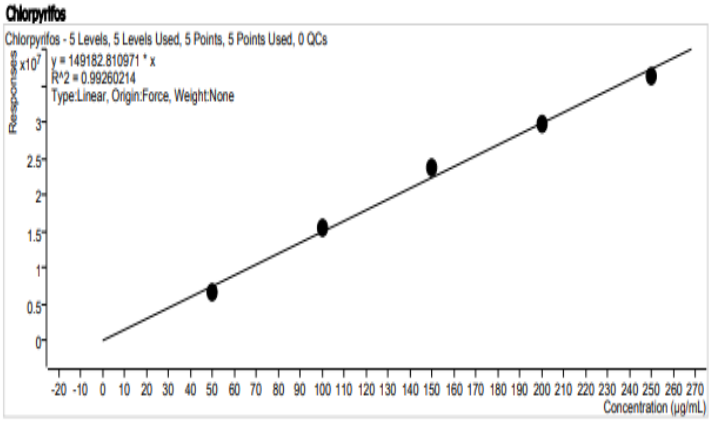
Chlorpyrifos



Calibration STD Path	Cal Type	Level	Enabled	Resp.	Exp. Conc	Resp. Factor
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D:\MassHunter\GCMS\2\5977\akinwola\Cal_250.D	Calibration	L1	x	36370616	250.0000	145482.4647

Figure 3a &b: Calibration for Methyl parathion & Chlorpyrifos**.**

Quantitative Analysis Complete Report

Agilent Technologies

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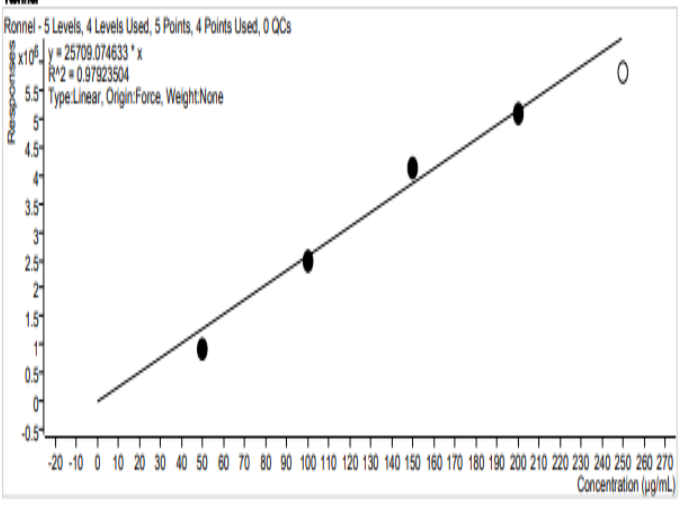
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00

Ronnel



Calibration STD Path	Cal Type	Level	Enabled	Resp.	Exp. Conc	Resp. Factor
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Quantitative Analysis Complete Report

Agilent Technologies

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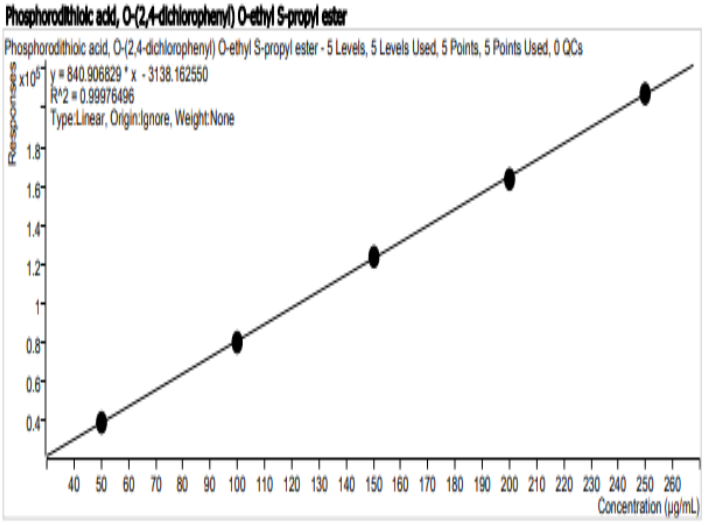
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester



Calibration STD Path	Cal Type	Level	Enabled	Resp.	Exp. Conc	Resp. Factor
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D:\MassHunter\GCMS\2\5977\akinwola\Cal_150.D	Calibration	L3	x	124263	150.0000	828.4209
D:\MassHunter\GCMS\2\5977\akinwola\Cal_200.D	Calibration	L2	x	163548	200.0000	817.7424
D:\MassHunter\GCMS\2\5977\akinwola\Cal_250.D	Calibration	L1	x	207615	250.0000	830.4589

Figure 4a &b: Calibration for Ronnel & Phosphorodithioic acid**.**

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Quantitative Analysis Complete Report

Agilent Technologies

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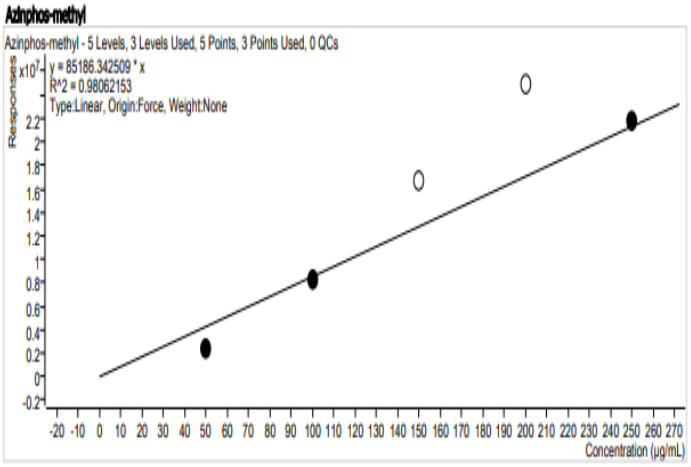
Azinphos-methyl



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D:\MassHunter\GCMS\2\5977\akinwola\Cal_200.D	Calibration	L2		24965836	200.0000	124829.1788
D:\MassHunter\GCMS\2\5977\akinwola\Cal_250.D	Calibration	L1	x	21783478	250.0000	87133.9137

Quantitative Analysis Complete Report

Agilent Technologies

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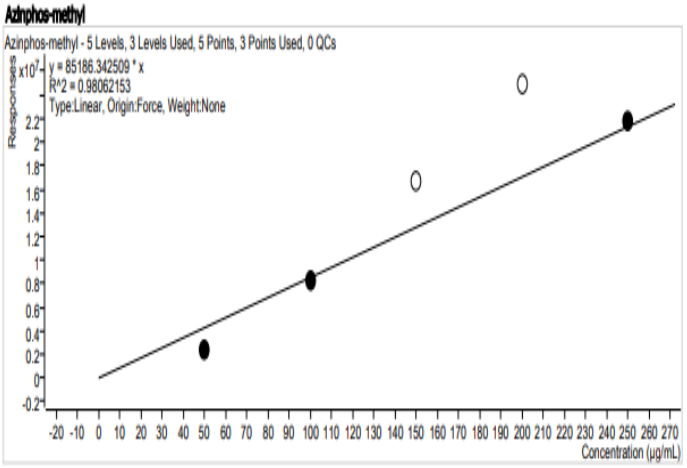
Azinphos-methyl



Calibration STD Path	Cal Type	Level	Enabled	Resp.	Exp. Conc	Resp. Factor
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D:\MassHunter\GCMS\2\5977\akinwola\Cal_150.D	Calibration	L3		16595447	150.0000	110636.3151
D:\MassHunter\GCMS\2\5977\akinwola\Cal_200.D	Calibration	L2		24965836	200.0000	124829.1788
D:\MassHunter\GCMS\2\5977\akinwola\Cal_250.D	Calibration	L1	x	21783478	250.0000	87133.9137

Figure 5a &b: Calibration for Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester Azinphos-methyl.

Table 5: Beans sample that were analyzed.

Quantitative Analysis Complete Report

Agilent Technologies

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Quant Batch Version	B.07.00	**Quant Report Version**	B.07.00

Data File	Sample Name	Sample Type	Position	Inj Vol	Level	Acq. Method
Cal_250.D	Calibration 250 ug/ml	Calibration	dummy	2.5	L1	ORGANOPHOSPHORUS PESTICIDE METHOD
Cal_200.D	Calibration 200 ug/ml	Calibration	dummy	2	L2	ORGANOPHOSPHORUS PESTICIDE METHOD
Cal_150.D	Calibration 150 ug/ml	Calibration	dummy	1.5	L3	ORGANOPHOSPHORUS PESTICIDE METHOD
Cal_100.D	Calibration 100 ug/ml	Calibration	dummy	1	L4	ORGANOPHOSPHORUS PESTICIDE METHOD
Cal_50.D	Calibration 50 ug/ml	Calibration	dummy	0.5	L5	ORGANOPHOSPHORUS PESTICIDE METHOD
BLANK_0.D	CALIBRATION 0 ug/ml	Blank	dummy	2	L6	ORGANOPHOSPHORUS PESTICIDE METHOD
AKINWOLA ADESHOLA7.D	WHITE BEANS (ORANGE MARKET)	Sample	dummy	2		ORGANOPHOSPHORUS PESTICIDE METHOD
AKINWOLA ADESHOLA8.D	BROWN BEANS (ORANGE MARKET)	Sample	dummy	2		ORGANOPHOSPHORUS PESTICIDE METHOD
AKINWOLA ADESHOLA9.D	WHITE BEANS (MARABA MARKET)	Sample	dummy	2		ORGANOPHOSPHORUS PESTICIDE METHOD
AKINWOLA ADESHOLA10.D	WHITE BEANS (KARU MARKET)	Sample	dummy	2		ORGANOPHOSPHORUS PESTICIDE METHOD
AKINWOLA ADESHOLA11.D	BROWN BEANS (KARU MARKET)	Sample	dummy	2		ORGANOPHOSPHORUS PESTICIDE METHOD
AKINWOLA ADESHOLA12.D	BROWN BEANS (MARABA MARKET)	Sample	dummy	2		ORGANOPHOSPHORUS PESTICIDE METHOD

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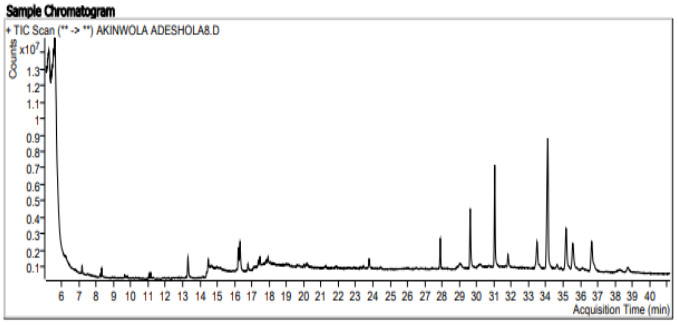
Table 6: Quantitative results of Organophosphorus pesticide in brown and white beans samples from Orange market.

Quantitative Analysis Complete Report

Agilent Technologies

Batch Path	D:\MassHunter\GCMS\2\5977\akinwola\QuantResults\AKINWOLA ADESHOLA.batch.bin		
Analysis Time	9/1/2025 7:58:38 PM	Analyst Name	MUSRL-HP\Admin
Report Time	9/1/2025 8:02:09 PM	Reporter Name	Admin
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00
Acq. Time	8/8/2025 7:26:30 PM		AKINWOLA ADESHOLA8.D
Sample Type	Sample	Sample Name	BROWN BEANS (ORANGE MARKET)
Dilution	1	Acq. Method	ORGANOPHOSPHORUS PESTICIDE METHOD

Sample Chromatogram



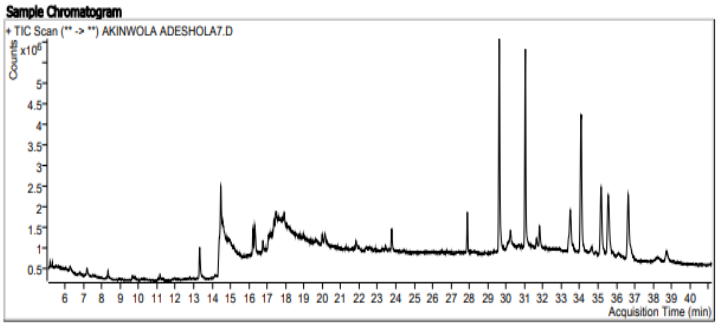
Compound	Transition	RT	Resp.	Final Conc	Units
Dichlorvos	187.1	5.411	121	0.0023	µg/mL
Oxydisulfoton	97.1	6.805	2571	0.7827	µg/mL
Ethoprophos	158.1	9.388	122	0.0003	µg/mL
Dichlofenthion	279.0	14.604	154	0.1571	µg/mL
Methyl parathion	93.1	14.503	13721	0.1286	µg/mL
Chlorpyrifos	314.1			ND	µg/mL
Ronnel	125.1	17.480	11324	0.4405	µg/mL
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	309.1	17.378	1041	4.9701	µg/mL
Azinphos-methyl	132.1	25.706	118	0.0014	µg/mL

Quantitative Analysis Complete Report

Agilent Technologies

Batch Path	D:\MassHunter\GCMS\2\5977\akinwola\QuantResults\AKINWOLA ADESHOLA.batch.bin		
Analysis Time	9/1/2025 7:58:38 PM	Analyst Name	MUSRL-HP\Admin
Report Time	9/1/2025 8:02:08 PM	Reporter Name	Admin
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00
Acq. Time	8/8/2025 6:01:50 PM		AKINWOLA ADESHOLA7.D
Sample Type	Sample	Sample Name	WHITE BEANS (ORANGE MARKET)
Dilution	1	Acq. Method	ORGANOPHOSPHORUS PESTICIDE METHOD

Sample Chromatogram



Compound	Transition	RT	Resp.	Final Conc	Units
Dichlorvos	187.1			ND	µg/mL
Oxydisulfoton	97.1	6.264	14006	4.2638	µg/mL
Ethoprophos	158.1	9.878	1001	0.0023	µg/mL
Dichlofenthion	279.0	14.255	144	0.1462	µg/mL
Methyl parathion	93.1	14.484	51972	0.4870	µg/mL
Chlorpyrifos	314.1			ND	µg/mL
Ronnel	125.1	17.455	7440	0.2894	µg/mL
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	309.1	17.366	1084	5.0208	µg/mL
Azinphos-methyl	132.1	26.056	550	0.0065	µg/mL

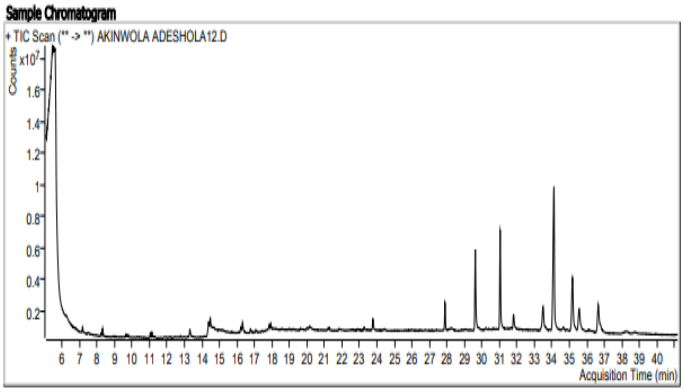
Table 7: Quantitative results of Organophosphorus pesticide in brown and white beans samples from Mararaba market.

Quantitative Analysis Complete Report

Agilent Technologies

Batch Path	D:\MassHunter\GCMS\2\5977\akinwola\QuantResults\AKINWOLA ADESHOLA.batch.bin		
Analysis Time	9/1/2025 7:58:38 PM	Analyst Name	MUSRL-HP\Admin
Report Time	9/1/2025 8:02:12 PM	Reporter Name	Admin
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00
Acq. Time	8/9/2025 9:28:42 AM		AKINWOLA ADESHOLA12.D
Sample Type	Sample	Sample Name	BROWN BEANS (MARABA MARKET)
Dilution	1	Acq. Method	ORGANOPHOSPHORUS PESTICIDE METHOD

Sample Chromatogram



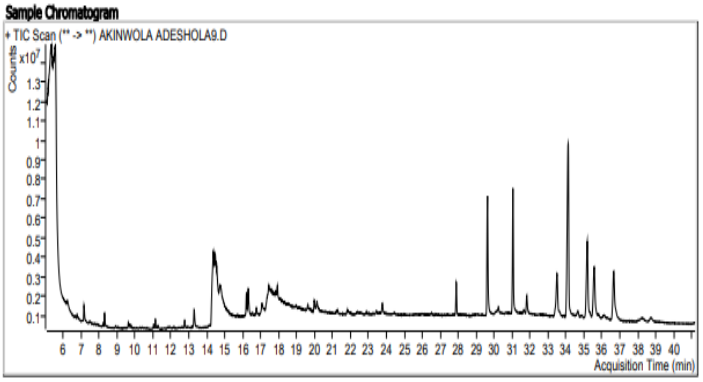
Compound	Transition	RT	Resp.	Final Conc	Units
Dichlorvos	187.1	5.373	220	0.0042	µg/mL
Oxydisulfoton	97.1	6.251	16369	4.9833	µg/mL
Ethoprophos	158.1	9.718	697	0.0016	µg/mL
Dichlofenthion	279.0	14.923	145	0.1480	µg/mL
Methyl parathion	93.1	14.458	20596	0.1930	µg/mL
Chlorpyrifos	314.1			ND	µg/mL
Ronnel	125.1	17.843	14812	0.5761	µg/mL
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	309.1	18.581	268	4.0510	µg/mL
Azinphos-methyl	132.1	26.011	343	0.0040	µg/mL

Quantitative Analysis Complete Report

Agilent Technologies

Batch Path	D:\MassHunter\GCMS\2\5977\akinwola\QuantResults\AKINWOLA ADESHOLA.batch.bin		
Analysis Time	9/1/2025 7:58:38 PM	Analyst Name	MUSRL-HP\Admin
Report Time	9/1/2025 8:02:09 PM	Reporter Name	Admin
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00
Acq. Time	8/8/2025 8:14:29 PM		AKINWOLA ADESHOLA9.D
Sample Type	Sample	Sample Name	WHITE BEANS (MARABA MARKET)
Dilution	1	Acq. Method	ORGANOPHOSPHORUS PESTICIDE METHOD

Sample Chromatogram



Compound	Transition	RT	Resp.	Final Conc	Units
Dichlorvos	187.1	6.881	193	0.0036	µg/mL
Oxydisulfoton	97.1	6.779	8053	2.4515	µg/mL
Ethoprophos	158.1	9.280	503	0.0012	µg/mL
Dichlofenthion	279.0	14.503	191	0.1949	µg/mL
Methyl parathion	93.1	14.369	75202	0.7047	µg/mL
Chlorpyrifos	314.1			ND	µg/mL
Ronnel	125.1	17.455	31579	1.2283	µg/mL
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	309.1	18.670	318	4.1101	µg/mL
Azinphos-methyl	132.1	25.541	807	0.0095	µg/mL

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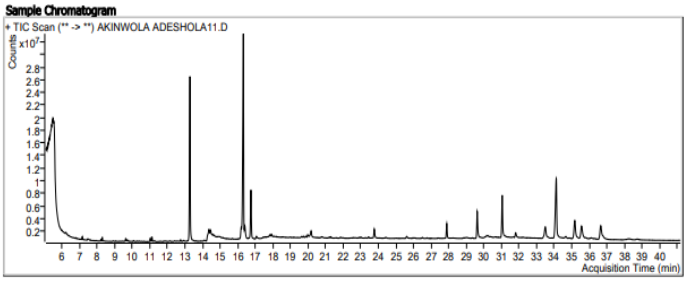
Table 8: Quantitative results of Organophosphorus pesticide in brown and white beans samples from Karu market.

Quantitative Analysis Complete Report

Agilent Technologies

Batch Path	D:\MassHunter\GCMS\2\5977\akinwola\QuantResults\AKINWOLA ADESHOLA.batch.bin		
Analysis Time	9/1/2025 7:58:38 PM	Analyst Name	MUSRL-HP\Admin
Report Time	9/1/2025 8:02:11 PM	Reporter Name	Admin
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00
Acq. Time	8/9/2025 8:40:44 AM		AKINWOLA ADESHOLA11.D
Sample Type	Sample	Sample Name	BROWN BEANS (KARU MARKET)
Dilution	1	Acq. Method	ORGANOPHOSPHORUS PESTICIDE METHOD

Sample Chromatogram



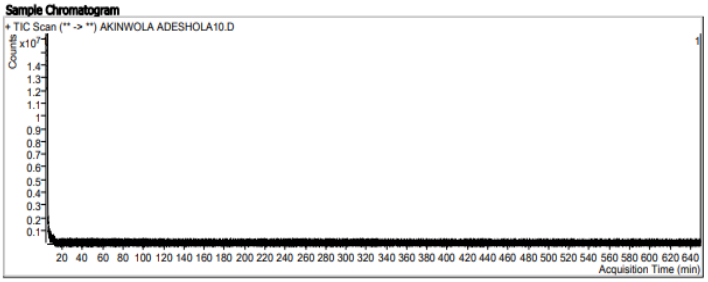
Compound	Transition	RT	Resp.	Final Conc	Units
Dichlorvos	187.1	5.182	3995	0.0756	µg/mL
Oxydisulfoton	97.1	6.690	1050	0.3197	µg/mL
Ethoprophos	158.1			ND	µg/mL
Dichlofenthion	279.0	14.471	160	0.1630	µg/mL
Methyl parathion	93.1	14.331	6405	0.0600	µg/mL
Chlorpyrifos	314.1			ND	µg/mL
Ronnel	125.1	16.755	47800	1.8593	µg/mL
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	309.1	17.385	186	3.9527	µg/mL
Azinphos-methyl	132.1	25.490	148	0.0017	µg/mL

Quantitative Analysis Complete Report

Agilent Technologies

Batch Path	D:\MassHunter\GCMS\2\5977\akinwola\QuantResults\AKINWOLA ADESHOLA.batch.bin		
Analysis Time	9/1/2025 7:58:38 PM	Analyst Name	MUSRL-HP\Admin
Report Time	9/1/2025 8:02:10 PM	Reporter Name	Admin
Last Calib Update	9/1/2025 7:58:15 PM	Batch State	Processed
Quant Batch Version	B.07.00	Quant Report Version	B.07.00
Acq. Time	8/8/2025 9:02:30 PM		AKINWOLA ADESHOLA10.D
Sample Type	Sample	Sample Name	WHITE BEANS (KARU MARKET)
Dilution	1	Acq. Method	ORGANOPHOSPHORUS PESTICIDE METHOD

Sample Chromatogram



Compound	Transition	RT	Resp.	Final Conc	Units
Dichlorvos	187.1	6.550	150	0.0028	µg/mL
Oxydisulfoton	97.1	6.684	1253	0.3814	µg/mL
Ethoprophos	158.1	9.649	596	0.0014	µg/mL
Dichlofenthion	279.0			ND	µg/mL
Methyl parathion	93.1	14.496	107	0.0010	µg/mL
Chlorpyrifos	314.1			ND	µg/mL
Ronnel	125.1			ND	µg/mL
Phosphorodithioic acid, O-(2,4-dichlorophenyl) O-ethyl S-propyl ester	309.1			ND	µg/mL
Azinphos-methyl	132.1	24.841	245	0.0029	µg/mL

Concentration of Identified Pesticide in Beans Sample Analyzed.

After the validation of the method, beans samples were analyzed and the levels of pesticide residues found are outlined in **Table 6 - 8**. Both the brown and white obtained from Orange market were found to contain the OPPs except for Chlorpyrifos that was not detected in brown beans, while Dichlorvos and Chlorpyrifos and were also not seen in the white beans sample, their concentration ranges from 0.0003 to 4.9701 mg/kg and 0.0023 to 5.0208 mg/kg brown and white beans respectively as seen in **Table 6**. Both the brown and white obtained from Mararaba market were found to contain the OPPs except for Chlorpyrifos that was not detected, the concentration of the other OPPs ranges from 0.0016 to 4.9833 mg/g and 0.0012 to 4.1101 mg/kg brown and white beans respectively as seen in **Table 7**. Ethoprophos, Dichlofenthion, Methyl parathion, Chlorpyrifos, Ronnel, Phosphorodithioic acid, O-(2, 4-dicorophenyl) O-ethyl S-propyl ester and Azinphos-methyl From this study Ethoprophos and Chlorpyrifos were not detected in the brown beans and then concentration of the seven OPPs ranges 0.0756 to 3.9527 mg/kg and the white beans did not contain Dichlofenthion, Chlorpyrifos, Ronnel, Phosphorodithioic acid, O-(2,4-dicorophenyl) O-ethyl S-propyl ester as other OPPs their concentration ranges from and 0.0010 to 0.3814 mg/kg white beans as seen in **Table 8**. Of all the compounds Phosphorodithioic acid, O-(2, 4-dicorophenyl) O-ethyl S-propyl ester has the highest concentration of 5.0802 mg/kg in white beans from Orange market followed by to 4.9701 mg/kg Oxydisulfoton in brown beans from Mararaba. The levels of Organphosphorus found in this study are within range as compared to some of the OPP residues in the bean samples in a study by [43]. Compared to the MRL set by the EU, the level of in the bean samples studied were was higheer or equal to the stipulated value of 0.01.

CONCLUSIONS AND RECOMMENDATIONS

Pesticide residues in or on plants may be unavoidable even when pesticides are used in accordance with Good Agricultural Practice. Pesticides have negative effects on society, so it is important to reevaluate their viability. To meet the need for food globally, alternative routes must be pursued to provide healthier food. Therefore, one of the primary findings of this study is that contamination is universal because no individual of any species throughout the entire sample appeared to be free of numerous pesticide residues in grains.. However, before the determination of target contaminant the methods used for the analysis was validated to test whether or not they were fit for the intended purpose. The study investigates the presence organophosphate pesticide residue in six selected grains sold in major markets of Karu, Nasarawa state, North-cental Nigeria. This study will help to understand the residual contamination of bean in the study area and will help to increase public awareness as

well. Most of the samples analysed in this study indicates presence of one or more of the pesticide residues, with most occurring at concentrations higher than their respective EU/UK maximum residue limits (MRLs). The repeatability and reproducibility of the data in both analysis showed that the method is fit for the analysis, therefore, these results depicted that the analysis method is appropriate for the targeted pesticides. For this study on beans from Karu LGA market, established Nigerian work on beans and grains shows that GC–MS after QuEChERS (or similar) can give robust qualitative and quantitative OPP data, and prior results from other states indicate that detectable OPP residues, sometimes exceeding MRLs, are likely.

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