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PERSISTENCE OF PERMETHRIN, PRIMIPHOS METHYL AND CHLORPYRIPHOS METHYL INSECTICIDES IN WHEAT STORED UNDER SIMULATED ENVIRONMENTAL CONDITIONS

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Procedures are described for determining the persistence of permethrin, primiphos methyl and chlorpyriphos methyl insecticides applied directly to wheat grains and stored at two moisture contents and at four different temperatures in the laboratory. This was done with a view to simulate different climatic zones of Pakistan. Samples of treated wheat were withdrawn from storage at regular intervals, ground to a coarse powder, extracted with suitable solvents, cleanedup and finally analyzed for residues by gas-liquid chromatography using thermionic specific and electron capture detectors. The decline in concentration of active ingredients of the three insecticides was maximum at 40° and 13% moisture content whereas it was minimum at 25° and 10% moisture content. Amongst the three products, permethrin was found to be the most persistent followed by primiphos methyl while chlorpyriphos methyl was the least persistent.

Key words: Wheat grains, Pesticide residues, Gas chromatography.

Introduction

It is conservatively believed that nearly 10% of the food grains are annually destroyed in Pakistan by a variety of insect pests. For protection of wheat grain during storage, fumigation by phosphine is very common but recent reports of emergence of phosphine resistant strains of stored grain pests has prompted active research in the area of bioefficacy evaluation of newer grain protectant insecticides.

Several organophosphates and pyrethroids, alone or in combination, have been evaluated for recommendation as useful replacements against a range of stored grain pests [1-6] but their increasing use has necessitated studies on persistence which involves efficient means of pesticide residue determination of newer compounds. Many researchers have worked on the residual toxicity and persistence of grain protectants on wheat grains [7- 10]. Masud and Zakai [11]determined persistence of malathion and fenitrothion insecticides in treated wheat grains stored under laboratory conditions. Gas chromatographic analyses of samples revealed that malathion at fortification levels of 16 and 24 ppm and fenitrothion at 4, 8 and 12 ppm have been found to persist in the grain for 8-10 and 18 to 20 months respectively.

Present studies were aimed at developing a suitable analytical methodology for multiple pesticide residues of three insecticides namely; permethrin (Coopex), primiphos methyl (Actellic) and chlorpyriphos methyl (Reldan) in wheat grains and determination of their persistence when applied as protectants directly to wheat of two different moisture contents and stored at four different temperatures in the laboratory. These conditions were chosen to simulate different environmental conditions of Pakistan. Results of residue determinations are presented in this paper.

Materials and Methods

Wheat grain. Fresh wheat was procured from Distt. Thatta, Sindh. Prior to use, the grain was cleaned of dust, extraneous plant parts and other foreign materials to ensure uniformity. It was thereafter fumigated with phosphine for one week to make it pest free. The fumigated grain was then exposed to atmosphere for 24 hrs to remove phosphine gas.

Moisture adjustment. Laboratory evaluations were carried out at 10% and 13% moisture contents. Initial moisture content of fresh wheat determined by standard ISO air oven method [12] was 10.6%. 30 kg wheat was taken for each moisture content. For adjustment at 13% moisture content, calculated amount of water was added to grain in 5 kg glass jars which were then sealed and tempered according to the method of Winks [13]. One week was considered sufficient for tempering. For adjustment at 10% moisture content, wheat was sun dried.

Grain protectant insecticides. Three insecticides for laboratory evaluation, namely; permethrin (Coopex) 10/50 grain protectant containing 10% w/v permethrin and 50% w/v piperonyl butoxide, primiphos methyl (Actellic) 50% w/v EC, and chlorpyriphos methyl (Reldan) 21.8% w/w EC were supplied by M/s. Wellcome (Pak) Ltd., ICI (Pak) Ltd., and Dow Chemicals Pacific (Pak) Ltd. respectively. The grain was treated at the recommended dosages of 2 mg/kg, 4 mg/kg and 10 mg/kg respectively.

Insecticide admixing. Three kg lots of wheat at each moisture content and for each temperature were treated with each the above-mentioned insecticides. Three replicates for each insecticide and each moisture content were treated in the following manner.

(i). Recommended dosage of each insecticide was separately diluted with 4 ml distilled water and applied to approx. 1cm thick layer of wheat in a 68 x 46 x 9 cm galvanized iron tray in the form of a fine spray using a Quickfit spray atomizer. The contents were transferred to glass jars and tumbled vigorously for approximately 20 mins.

(ii). Control lots of wheat for each moisture content and for each temperature were treated in a manner analogous to treated wheat but using only water.

Sample storage. For sample storage, 1 kg glass jars were used. Prior to use, they were properly cleaned, dried and labelled. Treated and control wheat samples were stored in sealed glass jars at 25, 30, 35 and 40° in different ovens. Wheat sample (800 gm) was placed in each jar for subsequent periodic sub-sampling.

Sampling scheme. Sub-samples have been taken at the given intervals of time after treatment, i.e., 0, 1, 2, 3, 4 weeks and thereafter at monthly intervals upto 12 months. Prior to sampling, each lot of wheat was thoroughly mixed and then nearly 50 gm were removed from each glass jar for residue analysis. The subsamples were properly packed in polythene bags, sealed and deep frozen at 20° for subsequent residue analysis.

Residue determination. Each sub-sample was analyzed for residues in triplicate along with control samples to check reproducibility of results. In order to economize cost and time, a modified Becker procedure for organophosphorus pesticide residues in grain [14] was further modified in our laboratory to make it workable for permethrin (a pyrethroid) as well. Details of procedures employed are given hereunder.

(a) *Apparatus* (i). Chromatographic column, 450 x 25 mm i.d. fitted with pyrex glass stopcock.

(ii). Separatory funnel, glass stoppered, Pyrex, 1 litre cap.

(iii). Grinder, hand driven.

(iv). Filter paper, whatman No.542.

(v). High speed waring blender.

(vi). Rotary vacuum evaporator.

(vii). Griffine flask shaker, Griffin and George, England.

(viii) GLC apparatus. Varian AG GC-3600 fitted with thermionic specific (TSD) and 63 Ni electron capture (ECD) detectors for Actellic, Reldan and permethrin respectively. GLC columns and operating para- meters were chosen which achieved optimum balance between sensitivity and degree of resolution with good symmetrical peaks emerging with reasonable retention times. The following operating parameters were employed for the studied compounds on two different detection systems.

Actellic and Reldan. Glass column 1 meter long x 2 mm i.d. packed with 3% OV-210 on 80-100 mesh Chromosorb W-HP. Temperatures; Injector, 220°, column oven 190° detector (TSD) 250°. Attenuation 64, range 12, Bead current 3.2 Amp., Gas flows; Nitrogen carrier gas 30 ml/min. Hydrogen 54 ml/min. and Air 175 ml/min., The detector was linear in the range of 0.01-1.0 ng and 0.01-2.0 ng for Actellic and Reldan insecticides respectively.

Permethrin. Glass column 2 metre long x 2 mm i.d. packed with a mixture of 1.5% OV-17+1.95% OV-210 on 80- 100 mesh Chromosorb W-HP., Temperatures; Injector; 250°, column oven 230°, detector (ECD) 280°, Attenuation 32, range 10, Gas flow. Nitrogen (carrier) 30 ml/min. The detector was linear in the range of 0.01 to 1.0 ng.

Prior to use, each column was conditioned for 24 hrs under a slow stream of nitrogen at temperatures 50° higher than their working temperatures.

Under the above operating conditions, retention times of pesticides were as follows:

Actellic 2.2 min., Reldan 1.9 min., and Permethrin 10.9min.

The equipment was used in conjunction with data system Varian DS-651 and thinkjet printer (Hewlett Packard, USA).

(b) *Reagents* (i). Acetone, toluene, dichloromethane and n-hexane. All solvents were AR grade and distilled before use.

(ii). *Extraction solvent*; water-acetone (1:8).

(iii). *Eluting solvent mixture;* Toluene-dischloromethane (1:5). Acetone was excluded from the procedure [14] because with its use, starch was not retained at the column cleanup step. It was necessary to completely remove starch which interferred with subsequent analysis.

(iv). *Silica gel*. For column chromatography (Merck No.7734), used without pretreatment.

(v). Activated charcoal (Merck No.2183).

(vi). Sodium chloride, AR grade, saturated solution.

(vii). Sodium sulphate, anhydrous, AR grade. Heated at 400° for 4 hrs, cooled in a dessicator and transferred to a suitable bottle.

(c) *Extraction*. The sample of wheat grain was ground to a coarse powder. 15 gm of the prepared sample was homogenised with 75 ml of extraction solvent in a blender for 1 min. The contents were transferred to a 250 ml glass stoppered conical flask, shaken on an electrical shaker for 3 hrs and then filtered through whatman No.542 filter paper. The residue was washed

twice with 15+10 ml portions of extraction solvent. Combined the three filtrates and discarded the residue. The combined filtrate (extract) was transferred to 1 lit. separatory funnel, added 150 ml of water, 10 ml of saturated sodium chloride solution and 25 ml of dichloromethane. The contents were shaken for 2 mins and separated the dichloromethane layer. Repeated the process twice with two, 25 ml portions of dichloromethane. Combined the dichloromethane layers. 10 gm anhydrous sodium sulphate were added to it and allowed to stand for 30 mins. Filtered the dry extract through a fluted filter paper, rinsed the container and filtered with three more 10 ml portions of dichloromethane. Combined the filtrates and reduced its volume to approximately 10 ml in a rotary evaporator.

(d) *Cleanup. Preparation of a chromatographic column.* A slurry of a mixture of 5 gm silica gel and 15 ml eluting solvent was prepared and poured into a column pre-charged with 1 ml of dichloromethane. The mixture was allowed to settle and excess of the solvent was run through the column. Thoroughly mixed 15 gm silica gel with 1 gm activated charcoal in a 100 ml beaker and slowly stirred in 35 ml of eluting solvent. While stirring, poured the charcoal-silica gel mixture on to the silica layer in the column, at first slowly and then the remainder reapidly. During this process, the tap was left open to obtain compact column packing and allowed the eluting solvent to run of until its level stood approximately 2 cm above the column packing and then covered it slowly with about 5 gm sodium sulphate. Washed the column with 50 ml of eluting solvent.

Column chromatography. The concentrated extract was transferred quantitatively into the prepared column, rinsing the flask thrice with a small volume of dichloromethane. Collected all the eluate from the moment of transfer in a 250 ml. Erlenmeyer flask. Eluted with 200 ml eluting solvent. The eluate was concentrated to about dryness in a rotary vacuum evaporator and the residue was taken-up in a small volume of n-hexane and quantitatively transferred to a 10 ml calibrated flask and diluted as desired for gas chromatography.

(e) *Gas chromatography.* Each cleanedup sample extract was analyzed by gas chromatography alongwith its insecticide standard in n-hexane using 1 µl injections. Results were evaluated by comparing the peak heights of sample extracts with those of relevant insecticide standards. Three sample injections were carried out on each extract to check reproducibility of results. Untreated control samples processed in an analogous manner did not show any interfering peak that might be attributed to the studied compounds.

(f) *Fortification procedures.* Prior to these studies, the efficiency of analytical procedures was evaluated in model experiments with procured wheat. 15 gm of coarsely ground wheat was spiked with each of the three insecticides separately at levels of 0.01 to 10 ppm. Extraction, cleanup and subsequent gas chromatographic determination was conducted immediately afterwards. Results of recovery experiments are presented in Table 1.

Results and Discussion

Recovery data (Table 1) has established that the minimum detection limit of each of the three compounds was 0.01 ppm. Recoveries at this fortification level were in the range of 80- 79%. These results were also compared with wheat treated with the three insecticides and forzen at-20° for 24 hrs prior to residue analysis because according to Desmarchellier [15], recoveries from fortified samples are sometimes not a reliable guide to recoveries of aged deposits. In both the cases, results compared satisfactorily. The methods are efficient, sensitive and reliable.

TABLE 1. RECOVERY OF STUDIED PESTICIDES FROM FORTIFIED WHEAT SAMPLES.

Sample No.	Pesticide	ppm fortification (mg kg^{-1})	% recovery+SD*
1.	Permethrin	10.00	98.40±0.11
		1.00	96.17±0.97
		0.10	82.70±0.55
		0.01	96.80±0.56
2.	Actellic	10.00	92.72±0.36
		1.00	87.10±0.88
		0.10	93.94±1.08
		0.01	80.50±0.68
3.	Reldan	10.00	96.37±0.33
		1.00	81.30±0.08
		0.10	93.57±0.20
		0.01	87.57±0.80

*Mean of three replicates.

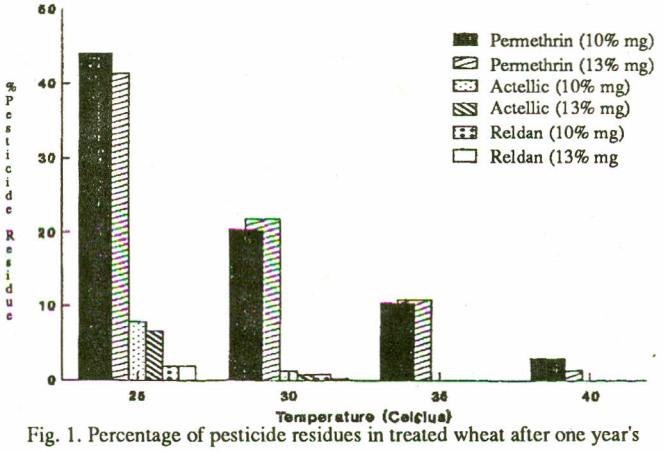

Fig. 1. Percentage of pesticide residues in treated wheat after one year's storage under different conditions.

Loss of each studied insecticide with time in relation to different temperatures and moisture contents is presented in Tables 2 to 4. Each figure in the Table is the mean pesticide residue of three replicates and is presented with standard error. Loss of three compounds under the given conditions after one year's storage is depicted in Fig. 1.

It is evident from this data that there is a gradual loss of each insecticide at increasing temperatures and moisture contents (m.c). It is minimum at 25° and 10% m.c. and maximum at 40° and 13% m. c. However, two moisture contents have not played any significant role in the break down of insecticides. Loss of active ingredient of each insecticide from treated wheat is discussed separately hereunder.

Permethrin. From an average zero day figure of 2.31 ppm, the residue levels were 1.02, 0.47, 0.24 and 0.07 ppm at the end of 16th sampling of treated wheat at 25, 30, 35 and 40° respectively at 10% m.c. while at 13% m.c., from an initial

TABLE 2. RESIDUE LEVELS (ppm) OF PERMETHRIN ON WHEAT AT FOUR DIFFERENT STORAGE TEMPERATURES AND TWO MOISTURE CONTENTS AFTER INDICATED PERIODS OF SAMPLING.

Sample No.	Sampling after treatment	Moisture content							
		10%				13%			
		Storage temperature (°C)				Storage temperature (°C)			
		25	30	35	40	25	30	35	40
1.	0 day*	2.31 ±0.1	–	–	–	2.20 ±0.06	–	–	–
2.	1 week	2.26 ±0.02	2.08 ±0.03	1.94 ±0.04	1.85 ±0.03	2.18 ±0.05	2.10 ±0.03	2.01 ±0.03	1.79 ±0.03
3.	2 weeks	2.12 ±0.005	1.96 ±0.02	1.89 ±0.02	1.83 ±0.02	2.07 ±0.02	1.91 ±0.02	1.91 ±0.04	1.75 ±0.02
4.	3 weeks	1.95 ±0.04	1.95 ±0.01	1.84 ±0.02	1.85 0.02	2.11 ±0.07	1.93 ±0.02	1.84 ±0.03	1.72 ±0.01
5.	4 weeks	1.86 ±0.03	1.98 ±0.03	1.79 ±0.01	1.73 ±0.01	1.99 ±0.06	1.88 ±.03	1.87 ±0.02	1.72 ±0.02
6.	2 months	1.62 ±0.03	1.54 ±0.04	1.59 ±0.04	1.31 ±0.02	1.68 ±0.06	1.68 ±0.07	1.53 ±0.03	1.29 ±0.01
7.	3 months	1.53 ±0.01	1.40 ±0.02	1.21 ±0.03	0.94 ±0.04	1.48 ±0.03	1.34 ±0.02	1.02 ±0.01	0.88 ±0.005
8.	4 months	1.65 ±0.07	1.38 ±0.006	1.19 ±0.01	0.86 ±0.02	1.63 ±0.04	1.35 ±0.01	1.01 ±0.004	0.82 ±0.02
9.	5 months	1.48 ±0.02	1.34 ±0.01	1.13 ±0.01	0.81 ±0.01	1.47 ±0.01	1.30 ±0.01	1.07 ±0.03	0.77 ±0.03
10.	6 months	1.42 ±0.01	1.31 ±0.01	1.05 ±0.04	0.74 ±0.01	1.41 ±0.01	1.26 ±0.01	1.02 ±0.02	0.72 ±0.02
11.	7 months	1.31 ±0.06	1.16 ±0.06	0.98 ±0.01	0.56 ±0.01	1.25 ±0.01	1.08 ±0.004	0.93 ±0.01	0.47 ±0.004
12.	8 months	1.26 ±0.01	1.05 ±0.01	0.69 ±0.01	0.43 ±0.01	1.21 ±0.01	1.02 ±0.004	0.66 ±0.01	0.34 ±0.004
13.	9 months	1.21 ±0.004	1.02 ±0.01	0.65 ±0.02	0.41 ±0.01	1.14 ±0.03	0.99 ±0.004	0.59 ±0.004	0.30 ±0.04
14.	10 months	1.17 ±0.01	0.92 ±0.01	0.53 ±0.01	0.28 ±0.01	1.10 ±0.02	0.83 ±0.01	0.51 ±0.004	0.19 ±0.004
15.	11 months	1.12 ±0.01	0.70 ±0.01	0.38 ±0.01	0.20 ±0.0	1.04 ±0.02	0.62 ±0.01	0.40 ±0.004	0.14 ±0.004
16.	12 months	1.02 ±0.004	0.47 ±0.01	0.24 ±0.01	0.07 ±0.004	0.91 ±0.004	0.48 ±0.01	0.24 ±0.01	0.03 ±0.004

*Room temperature.

TABLE 3. RESIDUE LEVELS (ppm) OF ACTELLIC ON WHEAT AT FOUR DIFFERENT STORAGE TEMPERATURES AND TWO MOISTURE CONTENTS AFTER INDICATED PERIOD OF SAMPLING.

Sample No.	Sampling after treatment	Moisture content							
		10%				13%			
		Storage temperature (°C)				Storage temperature (°C)			
		25	30	35	40	25	30	35	40
1.	0 day*	4.08 ±0.04	–	–	–	4.27 ±0.14	–	–	–
2.	1 week	4.07 ±0.10	4.03 ±0.04	4.03 ±0.03	4.01 ±0.06	4.24 ±0.01	4.21 ±0.06	4.06 ±0.01	4.04 ±0.06
3.	2 weeks	4.04 ±0.03	4.01 ±0.12	3.99 ±0.03	3.36 ±0.08	4.08 ±0.08	4.02 ±0.05	4.01 ±0.04	3.58 ±0.13
4.	3 weeks	3.65 ±0.01	3.55 ±0.04	2.99 ±0.04	2.77 ±0.09	3.57 ±0.14	3.41 ±0.17	3.24 ±0.10	2.77 ±0.01
5.	4 weeks	3.16 ±0.03	2.68 ±0.06	2.49 ±0.20	2.01 ±0.08	3.40 ±0.12	2.44 ±0.04	2.28 ±0.09	1.67 ±0.04
6.	2 months	3.07 ±0.02	2.36 ±0.02	1.32 ±0.02	0.80 ±0.02	3.01 ±0.03	2.24 ±0.04	1.12 ±0.04	0.61 ±0.01
7.	3 months	2.42 ±0.04	1.74 ±0.04	1.23 ±0.01	0.22 ±0.01	2.37 ±0.02	1.68 ±0.02	0.97 ±0.02	0.20 ±0.01
8.	4 months	2.12 ±0.02	1.08 ±0.03	0.93 ±0.02	0.18 ±0.01	2.05 ±0.04	1.11 ±0.01	0.85 ±0.01	0.14 ±0.01
9.	5 months	1.91 ±0.03	0.77 ±0.04	0.65 ±0.02	0.15 ±0.01	1.72 ±0.02	0.76 ±0.02	0.53 ±0.01	0.12 ±0.01
10.	6 months	1.61 ±0.004	0.60 ±0.02	0.53 ±0.02	0.13 ±0.01	1.57 ±0.02	0.56 ±0.01	0.41 ±0.02	0.10 ±0.01
11.	7 months	1.03 ±0.01	0.44 ±0.01	0.37 ±0.02	0.08 ±0.0	0.97 ±0.02	0.39 ±0.01	0.25 ±0.01	0.06 ±0.01
12.	8 months	0.97 ±0.01	0.39 ±0.004	0.23 ±0.004	0.06 ±0.004	0.94 ±0.02	0.31 ±0.01	0.20 ±0.004	0.04 ±0.004
13.	9 months	0.69 ±0.02	0.27 ±0.004	0.17 ±0.01	0.04 ±0.004	0.65 ±0.02	0.24 ±0.01	0.15 ±0.004	0.02 ±0.004
14.	10 months	0.51 ±0.01	0.18 ±0.01	0.11 ±0.01	0.02 ±0.004	0.45 ±0.03	0.15 ±0.01	0.08 ±0.004	0.01 ±0.0
15.	11 months	0.40 ±0.01	0.12 ±0.004	0.07 ±0.01	Trace	0.37 ±0.004	0.10 ±0.0	0.04 ±0.0	Traces
16.	12 months	0.32 ±0.01	0.05 ±0.0	Traces	Nil	0.28 ±0.004	0.03 ±0.004	Traces	Nil

*Room temperature.

TABLE 4. RESIDUE LEVELS (PPM) OF RELDAN ON WHEAT AT FOUR DIFFERENT STORAGE TEMPERATURES AND TWO MOISTURE CONTENTS AFTER INDICATED PERIODS OF SAMPLING.

Sample No.	Sampling after treatment	Moisture content							
		10%				13%			
		Storage temperature (°C)				Storage temperature (°C)			
		25	30	35	40	25	30	35	40
1.	0 day*	10.29 ±0.03	–	–	–	10.03 ±0.04	–	–	–
2.	1 week	9.94 ±0.18	10.03 ±0.04	8.05 ±0.08	5.80 ±0.20	9.99 ±0.09	9.27 ±0.18	7.69 ±0.05	6.46 ±0.29
3.	2 weeks	9.98 ±0.02	9.14 ±0.10	7.71 ±0.02	4.78 ±0.05	19.94 ±0.20	8.54 ±0.03	7.11 ±0.04	4.37 ±0.12
4.	3 weeks	9.66 ±0.04	5.72 ±0.21	3.18 ±0.04	3.06 ±0.14	9.56 ±0.17	5.56 ±0.04	3.47 ±0.04	3.28 ±0.15
5.	4 weeks	9.39 ±0.08	3.70 ±0.05	1.98 ±0.07	1.60 ±0.09	9.84 ±0.13	3.24 ±0.20	1.95 ±0.19	1.71 ±0.04
6.	2 months	3.13 ±0.04	2.27 ±0.04	1.28 ±0.004	1.10 ±0.08	2.98 ±0.03	1.53 ±0.04	1.36 ±0.04	1.16 ±0.04
7.	3 months	1.21 ±0.03	0.98 ±0.04	0.41 ±0.02	0.37 ±0.01	1.11 ±0.02	0.79 ±0.02	0.37 ±0.06	0.25 ±0.03
8.	4 months	1.16 ±0.004	0.69 ±0.01	0.34 ±0.01	0.26 ±0.02	1.04 ±0.01	0.68 ±0.02	0.33 ±0.01	0.18 ±0.01
9.	5 months	1.13 ±0.01	0.65 ±0.01	0.32 ±0.004	0.24 ±0.02	1.02 ±0.004	0.59 ±0.01	0.33 ±0.004	0.16 ±0.02
10.	6 months	1.07 ±0.02	0.62 ±0.01	0.32 ±0.004	0.20 ±0.01	1.01 ±0.01	0.56 ±0.01	0.29 ±0.004	0.21 ±0.01
11.	7 months	0.96 ±0.02	0.58 ±0.004	0.26 ±0.004	0.09 ±0.0	0.92 ±0.004	0.48 ±0.01	0.25 ±0.01	0.07 ±0.0
12.	8 months	0.88 ±0.01	0.51 ±0.01	0.20 ±0.004	0.06 ±0.0	0.86 ±0.01	0.40 ±0.01	0.18 ±0.01	0.05 ±0.0
13.	9 months	0.72 ±0.004	0.45 ±0.01	0.14 ±0.004	0.04 ±0.004	0.69 ±0.01	0.18 ±0.004	0.10 ±0.004	0.03 ±0.0
14.	10 months	0.51 ±0.004	0.32 ±0.004	0.10 ±0.004	0.02 ±0.0	0.49 ±0.01	0.12 ±0.004	0.05 ±0.004	0.01 ±0.0
15.	11 months	0.36 ±0.0	0.22 ±0.004	Traces	Nil	0.37 ±0.004	0.07 ±0.004	Traces	Nil
16.	12 months	0.19 ±0.0	0.08 ±0.01	Nil	Nil	0.19 ±0.004	0.02 ±0.004	Nil	Nil

* Room temperature.

treatment dosage of 2.20 ppm, the levels were 0.91, 0.48, 0.24 and 0.03 ppm respectively after the same period of storage.

Actellic. AT 10% moisture content, from an average treatment dosage of 4.08 ppm on the zero day, the residue levels at 25, 30, 35 and 40° at the end of 16th sampling fell down to 0.32, 0.05, traces and 0.00 ppm respectively while at 13% moisture content, from an average zero day figure of 4.27 ppm, the levels were 0.28, 0.03, traces and 0.00 ppm respectively after the same period of storage.

Reldan. At 10% moisture content, from an average treatment dosage of 10.29 ppm on the zero day, the residue levels at 25, 30, 35 and 40° at the end of 16th sampling (i.e., 12 months after storage) diminished to 0.19, 0.08, 0.00 and 0.00 ppm respectively while at 13% moisture content, from an average treatment dosage of 10.03 ppm, it decreased to 0.19, 0.02, 0.00 and 0.00 ppm after the same storage period.

Conclusion

On the basis of these studies, it can be safely concluded that permethrin is the most persistent of the three followed by Actellic and Reldan. Actellic and Reldan residues declined quickly at 40° but slowly at 25°. Therefore, the three insecticides may be used in the order: permethrin> primiphos methyl> chlorpyriphos methyl depending upon the period of storage. Depending upon the nature, intensity of stored grain pests and period of storage, the insecticides may be safely applied at a slightly higher dosage rate than applied by the authors in order to achieve satisfactory control.

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