RESIDUES AND TRACE ELEMENTS

Monitoring Pesticide Residues in Egyptian Fruits and Vegetables in 1995

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Organophosphorus, dithiocarbamates, and some synthetic pyrethroids pesticides, which are commonly used in Egypt for pest control, were monitored, as well as persistent organochlorines, which had been prohibited from use several years ago. Fruit and vegetable samples (397) were collected from 8 local markets and examined for 52 pesticides. Of all analyzed samples, 42.8% contained detectable residues, of which 1.76% exceeded their maximum residue limits (MRLs). The rates of contamination with the different pesticides were 0–86%. However, violation rates among contaminated products were very low, ranging from 0 to 4.6%. In general, organochlorine pesticide residues were not detected in most samples. Dithiocarbamate residues were found in 70.4% of 98 samples analyzed for dithiocarbamates, but only one grape sample had residues exceeding the MRL established by the Codex Committee on Pesticide Residues.

The national monitoring programs for pesticide residues are the key means of ensuring compliance with regulations. They also create a database to help assess the levels of pesticide residues and the levels of residue intake. The information is invaluable in assessing human exposure to pesticide residues through the diet and assists in the country's formulation of a pesticide strategy. Monitoring of pesticide residues also provides a check on compliance with good agricultural practice in the use of pesticides.

The present study is a continuation of previous studies beginning in 1988 (1–5) within the National Residue Monitoring Program of the Ministry of Agriculture of Egypt to evaluate the actual contamination of Egyptian food by pesticides. The most common fruits and vegetables consumed during the year were obtained from markets in 6 governorates.

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Experimental

Sampling

A total of 397 samples were collected from 8 local markets in 6 governorates (Cairo, Giza, Qualubiya, Beni Suef, Minufiya, and Ismailia) in 1995. For residue analysis, 2 kg of each type of fruit or vegetable was prepared according to guidelines of the Codex Committee on Pesticide Residues (CCPR; 6).

Residues

Samples were analyzed for 52 pesticides, including organophosphorus, nitrogen, and organochlorine pesticides and certain pyrethroids. Dithiocarbamates were analyzed in only 98 of the 397 samples.

Samples were prepared according to the generally recommended method of sampling to achieve a representative part of the material to be analyzed (7, 8). Samples were analyzed immediately on their arrival at the laboratory or stored at 0°–5°C for no more than 4 days before analysis. However, samples subjected to dithiocarbamate analysis were analyzed immediately after preparation to avoid decomposition of the chemical.

Extraction and Cleanup

(**a**) *Multiresidue method.*—According to the method described by Luke (9, 10), residues were extracted from nonfatty foods by blending the sample with acetone or water–acetone and then extracting with petroleum ether and dichloromethane. The organic phase was separated, dried, and concentrated just to dryness. The residue was dissolved in hexane–acetone for gas chromatographic (GC) determination. The method allows determination of the 52 pesticide residues listed in Table 1, which also lists commodities, spike levels, average recoveries, and standard deviations.

(**b**) *Dithiocarbamates.*—Residues of dithiocarbamates should be expressed as CS_2 (carbon disulfide) for comparison with the Codex maximum residue limits (MRLs), which were established on a CS_2 basis. The analytical method is based on the evolution of CS_2 as modified by Keppel (7, 8). Dithiocarbamates release CS_2 when heated with HCl in the

Table 1. Recoveries of pesticides from spiked samples

Compound	Spiking level, mg/kg	Matrix	No. of samples	Average recovery, %	CV, %
		Organophosphorus and nitrogen compounds			
Atrazine	0.2	Apple	2	78	4.6
Bendiocarb	0.1	Apple	2	92	6.2
		Pepper	6	87	1
Carbaryl	0.5	Apple	2	92	7.8
		Pepper	6	109	1
Chlorpyrifos	0.02	Pepper	5	80	12
	0.04	Apple	2	97	11.2
Chlorpyrifos-Me	0.02	Pepper	5	100	11
Cyanophos	0.05	Pepper	6	81	3
		Apple	2	72	3.9
Diazinon	0.02	Pepper	5	90	15
	0.04	Apple	2	76	2.3
Dimethoate	0.06	Pepper	5	105	9
Fenitrothion	0.02	Pepper	5	80	11
Fenthion	0.05	Pepper	6	94	2
		Apple	2	90	4.8
Malathion	0.06	Cucumber	4	108	1
		Pepper	5	75	4
Metalaxyl	0.5	Pepper	1	110	—
Omethoate	0.05	Pepper	1	83	—
Phosalone	0.04	Pepper	2	102	—
	0.08				
Pirimicarb	0.06	Pepper	5	82	4
		Cucumber	4	90	1
Pirimiphos-Et	0.02	Pepper	2	83	—
	0.04			80	
Pirimiphos-Me	0.02	Pepper	4	91	8
		Apple	2	82	2.2
Profenofos	0.02	Apple	2	93	11.5
Prothiofos	0.02	Pepper	4	82	20
		Apple	2	83	4.3
Pyrazophos	0.02	Pepper	4	105	17
		Apple	2	87	2
Tolclophos-Me	0.02	Pepper	4	82	2
		Apple	2		3
Triazophos	0.02	Apple	2	90	6
		Organochlorine compounds			
Bromopropylate	0.05	Pepper	3	97	8
Carbosulfan	0.2	Pepper	1	90	—
Chlorothalonil	0.03	Pepper	3	91	6
Cyfluthrin	0.1	Pepper	1	114	—
Cypermethrin	0.1	Pepper	6	117	2
		Apple	3	112	6.4

Table 1. (*continued*)

Compound	Spiking level, mg/kg	Matrix	No. of samples	Average recovery, %	CV, %
DDD-*p',p'*	0.02	Pepper	6	113	5
DDE-*p',p'*	0.02	Apple	3	112	2.6
DDT-*o,p*	0.02	Pepper	3	95	5
DDT-*p',p'*	0.02	Pepper	6	76	5
		Apple	3	112	2.6
Deltamethrin	0.2	Pepper	6	101	4
Dichlofluanide	0.05	Pepper	9	107	9
		Apple	2	109	1.3
Dicofol	0.02	Pepper	2	118	8
Dieldrin	0.01	Pepper	3	99	8.3
Endrin	0.06	Orange	1	82	—
Fenvalerate	0.02	Pepper	5	94	11
		Cucumber	4	107	5
α-HCH	0.01	Pepper	6	94	3
		Apple	3	10	8.7
β-HCH	0.01	Pepper	3	110	13
γ-HCH (lindane)	0.01	Cucumber	4	85	4
		Pepper	5	83	13
δ-HCH	0.01	Pepper	3	100	14
Heptachlorepoxide	0.01	Pepper	6	94	8
		Apple	3	108	1.9
Hexachlorobenzene	0.01	Pepper	3	111	18
Iprodion	0.5	Pepper	6	111	4
		Apple	3	110	2.8
Permethrin	1.0	Pepper	6	101	9
		Apple	3	115	3.7
Procymidone	0.06	Pepper	5	77	9
		Cucumber	4	104	3
Propiconazole	0.05	Pepper	3	100	6
Tetradifon	0.03	Pepper	2	90	11
Triadimefon	0.05	Pepper	6	109	5
		Apple	3	98	3.9
Triadimenol	0.1	Apple	1	76	—
Trifluralin	0.01	Pepper	3	100	19
Vinclozolin	0.01	Pepper	5	97	15
		Cucumber	4	99	9

presence of stannous chloride as a reducing reagent. The CS_2 evolved is distilled, purified, and collected in an ethanolic solution of copper(II) acetate and diethanolamine to form a yellow complex. The absorbance of the yellow product is determined spectrophotometrically at 435 nm.

GC Determination

Detection of residues and confirmation of their presence in food samples depended on the use of chromatographic columns of different polarities. Quantitative determinations were made by the internal-standard technique. Aldrin was used as the internal standard for organochlorine pesticides and pyrethroids, which were detected by electron capture. Ditalimphos was the internal standard for organophosphates and nitrogen-containing compounds, which were detected with a nitrogen–phosphorus detector (NPD).

Quality Assurance

Methods and instruments were fully validated as part of a laboratory quality assurance system (11) and have been audited and accredited by the Center for Metrology and Accreditation, Helsinki, Finland (12).

The Codex committee's criteria for quality assurance were followed to determine the performance of the multiresidue method. Recovery, accuracy, limit of determination, and coefficient of variation (CV) were determined for every compound in the different commodities. Recoveries of several compounds from at least one commodity were determined. Results show that the method may be applied to the 52 different pesticides.

Average recoveries and CVs of the 52 pesticides were 72–118 and 1–20%, respectively, at the spiking levels shown in Table 1. Reproducibility, expressed as relative standard deviation, was less than 20%. The limits of determination for fruits and vegetables ranged from 0.01 to 0.1 mg/kg. Measurement uncertainty, including random and systematic errors (95% confidence level), was <±40%.

For recovery studies, samples were spiked with 7 indicators representing different types of pesticides (γ-HCH [lindane], vinclozoline, procymidone, fenvalerate, pirimicarb, dimethoate, and malathion).

Previously determined recoveries of ethylenebisdithiocarbamates at different levels of fortification (0.1, 1, and 10 mg/kg) from cucumber, tomato, and eggplant ranged from 80 to 110%. The relative standard deviation was less than 20%, and the limit of determination was 0.2 mg/kg (13).

Apparatus

(a) *GC systems.*—HP 5890 equipped with 2 electron capture detectors and 2 capillary columns: injector, 225°C; detector, 300°C (Hewlett-Packard, USA). Operating conditions: nitrogen carrier gas, 2.5 mL/min, 75–90 mL/min (carrier + make up); column head pressure, 82 kPa. A second HP 5890 was equipped with 2 NPDs: injector, 225°C; detector, 280°C. Operating conditions: hydrogen, 3.5 ± 0.1 mL/min; air, 100–200 mL/min; nitrogen carrier gas, 25 mL/min.

(b) *Chromatographic columns.*—PAS-5 ECD tested ultra 2 silicon, 25 m × 0.32 mm, 0.52 μm film thickness, and PAS-1701 ECD tested 1701 silicon, 25 m×0.32 mm, 0.25 μm film thickness. Temperature programs of both GC instruments were as follows: initial temperature, 90°C for 2 min, ramp (*1*) 20°C/min to 150°C, ramp (*2*) 6°C/min to 270°C hold 15 min.

(c) *UV spectrophotometer.*—Double beam, Unicam SP 1800 (Cambridge, UK).

Reagents

(a) *Solvents and chemicals.*—Acetone, dichloromethane, *n*-hexane, and petroleum ether (Pestiscan chromatography grade or similar quality); ethanol (95–96%), diethanolamine (98%), HCl, toluene, and carbon disulfide; anhydrous sodium sulfate (Riedel-de Haen, Dublin, Ireland), NaCl, NaOH, copper(II) acetate monohydrate (98%), and tin(II) chloride dehydrate.

(b) *Pesticide reference standards.*—(*1*) *Organophosphorus and nitrogen-containing compounds.*—Atrazine, bendiocarb, carbaryl, chlorpyrifos, chlorpyrifos-Me, cyanophos, diazinon, dimethoate, fenitrothion, fenthion, malathion, metalaxyl, omethoate, phosalone, pirimicarb, pirimiphos-Et, pirimiphos-Me, profenofos, prothiofos, pyrazophos, tolclophos-Me, and triazophos. (*2*) *Organochlorine and pyrethroid compounds.*—Bromopropylate, carbosulfan, chlorothalonil, cyfluthrin, cypermethrin, DDD-*p',p'*, DDE-*p',p'*, DDT-*o,p*, DDT-*p',p'*, deltamethrin, dichlofluanide, dicofol, dieldrin, endrin, fenvalerate, α-HCH, β-HCH, γ-HCH (lindane), δ-HCH, heptachlorepoxide, hexachlorobenzene, iprodion, permethrin, procymidone, propiconazole, tetradifon, triadimefon, triadimenol, trifluralin, and vinclozolin. All reference materials were certified standards from Dr. Ehrenstorfer Gmbh, Augsburg, Germany, and from the Food and Agriculture Organization of the United Nations, Rome, Italy, and were prepared in *n*-hexane–acetone. (*3*) *Dithiocarbamate compounds.*—Sodium diethyldithiocarbamate (<95%).

Results and Discussion

A total of 397 samples of fruits and vegetables (cabbage, cauliflower, carrot, courgette, cucumber, eggplant, green beans, green peas, lettuce, onion, pepper, strawberry, tomato, apple, cantaloupe, grape, guava, mango, orange, and peach) were examined for residues of 52 pesticides. Dithiocarbamates were analyzed as CS_2 in 98 samples of cucumber, green beans, green peas, strawberry, tomato, and grape. Less than half of the samples (42.8%) contained detectable residues, of which 1.76% exceeded their MRLs. Details of residues detected are provided in Table 2. The most commonly detected residues were dithiocarbamates (70.4% of 98 samples), as well as dicofol (15.1% of 397 samples), dimethoate (6.8%), tetradifon (4.5%), malathion (3.3%), profenofos (2.8%), omethoate (2.3%), chlorothalonil (2.0%), and chlorpyrifos–methyl (1.5%).

Among all samples, 22 strawberry samples (5.32%) contained 10 pesticide residues, 65 grape samples (15.73%) contained 11 pesticide residues, and 62 tomato samples (15.01%) contained 13 pesticide residues. Cauliflower, onion, and guava samples were free from pesticides residues. Samples of carrot and eggplant contained trace amounts of DDT-*p',p'* and DDE-*p',p'* residues. But in general, residues of DDT and HCH have disappeared almost completely from vegetables and fruits. Use of these pesticides in Egypt was completely prohibited by law in 1987.

Rates of contamination of vegetables and fruits by different pesticides ranged from 0 to 86% (Table 2). However, violations among contaminated products were very low, ranging from 0 to 4.6% (excluding omethoate in cabbage, because of low number of samples [1 of 5 samples]).

For comparison, rates of contamination found by the U.S. Food and Drug Administration in 1993 through its monitoring program (14) were 59 and 28.2% for fruits and vegetables, respectively, with violation rates of 1.5 and 1.8%, respectively. Our data showed slightly higher contamination and violation rates of vegetables and fruits, 37 and 2.2%, respectively, and lower contamination and violation rates of fruits and vegetables, 54 and 0.77%, respectively.

Table 2. Pesticide residues in vegetables and fruits from Egyptian markets, 1995[a]

Sample	Total No.	Pesticides found	Frequencies	Range (min–max)	Mean	Contaminated samples		MRL, mg/kg[b]	Violative compounds		Violative sam
						No.	%		No.	%	No.
						Vegetables					
Cabbage	5	Dicofol	1	0.03–0.03	0.03	3	60	5.0	—	—	—
		Dimethoate	1	0.42–0.42	0.42			2.0	—	—	—
		Omethoate	1	0.46–0.46	0.46			0.2	1	20	1
		Pirimiphos-ethyl	1	0.32–0.32	0.32			—[c]	—	—	—
Cauliflower	4	None	—	—	—	—	—	—	—	—	—
Carrot	21	Dicofol	1	0.63–0.63	0.63	2	9.5	5.0	—	—	—
		DDE-*p′,p′*	1	0.03–0.03	0.03			—[c]			
		DDT-*p′,p′*	1	0.04–0.04	0.04			—[c]			
		Total DDT	1	0.07–0.07	0.07			0.2 EMRL[d]			
		Tetradifon	1	0.05–0.05	0.05			—[c]			
		Triadimenol	1	0.28–0.28	0.28			—[c]			
Courgette	16	Chlorothalonil	1	0.02–0.02	0.02	1	6.2	5.0	—	—	—
Cucumber	47	Bromopropylate	1	0.23–0.23	0.23	18	38.3	1.0	—	—	—
		Chlorothalonil	1	0.12–0.12	0.12			5.0			
		Dicofol	3	0.04–0.10	0.06			2.0			
		Vinclozoline	2	0.02–0.31	0.17			1.0			
		Dimethoate	2	0.05–0.05	0.05			—[c]			
		Metalaxyl	1	0.24–0.24	0.24			0.5			
		Dithiocarbamates (16)[e]	9	0.10–0.30	0.15			0.5			
Egg plant	7	Dicofol	1	0.03–0.03	0.03	6	85	5.0	—	—	—
		DDE-*p′,p′*	3	0.01–0.2	0.08			—[c]			
		Dimethoate	3	0.01–0.03	0.02			—[c]			
Green beans	24	Chlorothalonil	1	0.15–0.15	0.15	10	41.6	5.0	—	—	—
		Dicofol	5	0.05–1.4	0.37			5.0			
		Omethoate	1	0.05–0.05	0.05			0.2			
		Dimethoate	3	0.05–0.14	0.08			—[c]			
		Malathion	1	0.02–0.02	0.02			2.0			
		Dithiocarbamates (11)[e]	1	0.12–0.12	0.12			0.5			
Green peas	13	Dicofol	1	0.08–0.08	0.08	2	15.3	5.0	—	—	—

Table 2. (*continued*)

Sample	Total No.	Pesticides found	Frequencies	Range (min–max)	Mean	Contaminated samples		MRL, mg/kg[b]	Violative compounds		Violative samples	
						No.	%		No.	%	No.	%
		Omethoate	1	0.09–0.09	0.09			0.1				
		Dimethoate	1	0.28–0.28	0.28			0.5				
		Dithiocarbamates (8)[e]	1	0.14–0.14	0.14			—[c]				
Lettuce	12	Omethoate	2	0.05–0.05	0.05	3	25	0.2	—	—	—	—
		Dimethoate	1	0.17–0.25	0.21			—[c]				
Onion	19	None	—	—	—	—	—	—	—	—	—	—
Pepper	8	Carbosulfan	1	0.37–0.37	0.37	3	37	—[c]	—	—	—	—
		Dicofol	2	0.25–0.79	0.52			5.0				
		Tetradifon	2	0.09–0.17	0.13			—[c]				
		Dimethoate	1	0.07–0.07	0.07			1.0				
Tomato	62	Chlorothalonil	3	0.05–0.16	0.09	29	46.7	5.0			2	3.2
		Dicofol	9	0.02–0.28	0.11			1.0				
		Cypermethrin	1	1.1–1.1	1.1			0.5	1	1.6		
		Permethrin	1	0.79–0.79	0.79			1.0				
		Ipodione	3	0.05–0.22	0.11			5.0				
		Tetradifon	2	0.04–0.09	0.07			—[c]				
		Chlorpyrifos-Me	1	0.07–0.07	0.07			0.5				
		Dimethoate	1	0.21–0.2	0.21			1.0				
		Metalaxyl	1	0.15–0.15	0.15			0.5				
		Omethoate	1	0.05–0.05	0.05			1.0				
		Pirimiphos-Me	1	0.06–0.06	0.06			1.0				
		Profenofos	5	0.04–2	0.72			2.0	1	1.6		
		Dithiocarbamates (23)[e]	23	0.1–0.7	0.23			3.0				
					Fruits							
Apple	13	Dicofol	5	0.5–0.77	0.51	6	46.2	5.0	—	—	—	—
		Propiconazol	1	0.39–0.39	0.39			—[c]				
		Tetradifon	3	0.06–0.18	0.14			—[c]				
		Chlorpyrifos-Me	1	0.15–0.15	0.15			0.5				
		Dimethoate	2	0.03–0.05	0.04			1.0				

Table 2. (*continued*)

Sample	Total No.	Pesticides found	Frequencies	Range (min–max)	Mean	Contaminated samples		MRL, mg/kg[b]	Violative compounds		Violative samples	
						No.	%		No.	%	No.	%
		Omethoate	1	0.05–0.05	0.05			2.0				
Cantaloupe	13	Dicofol	3	0.05–0.15	0.09	5	83.4	5.0	—	—	—	—
		Tetradifon	3	0.02–0.05	0.03			—[c]				
		Bromopropylate	1	0.46–0.46	0.46			0.5				
		Prothiofos	1	0.27–0.27	0.27			—[c]				
Grape	65	Dicofol	13	0.05–3.3	0.73			5.0			3	4.6
		Tetradifon	1	0.04–0.04	0.04	52	80	—[c]				
		Triadimefon	2	0.08–0.42	0.25			0.5				
		Triadimenol	1	3.04–3.04	3.04			2.0	1	1.5		
		Dimethoate	8	0.06–1.53	0.39			1.0				
		Omethoate	1	0.16–0.16	0.16			2.0				
		Malathion	11	0.02–0.2	0.09			8.0				
		Pirimicarb	1	0.11–0.11	0.11			—[c]				
		Profenofos	4	0.05–0.55	0.19			—[c]				
		Chlorpyrifos-Me	4	0.05–0.24	0.13			0.2	1	1.5		
		Dithiocarbamates (32)[e]	32	0.13–6.8	1.2			5.0	1	1.5		
Guava	12	None	—	—	—	—	—	—	—	—	—	—
Mango	10	Fenvalerate	2	0.43–0.51	0.47	2	20	—[c]	—	—	—	—
Orange	13	Dicofol	3	0.03–0.14	0.09	6	46	—[c]	—	—	—	—
		Dimethoate	2	0.02–0.07	0.05			2				
		Vinclozoline	1	0.02–0.02	0.02			—[c]				
Peach	11	Permethrin	1	0.11–0.11	0.11	3	27	2	—	—	—	—
		Dimethoate	1	0.62–0.62	0.62			2				
		Omethoate	1	0.14–0.14	0.14			2				
Strawberry	22	Dicofol	13	0.02–1.4	0.31	9	86	1.0	1	4.5	1	4.5
		Tetradifon	6	0.1–0.69	0.2		—[c]					
		Chlorothalonil	2	0.05–0.13	0.09			—[c]				
		Dimethoate	1	0.16–0.16	0.16			1.0				
		Fenitrothion	1	0.02–0.02	0.02			0.5				
		Malathion	1	0.04–0.04	0.04			1.0				

Table 2. (continued)

Sample	Total No.	Pesticides found	Frequencies	Range (min–max)	Mean	Contaminated samples No.	Contaminated samples %	MRL, mg/kg[b]	Violative compounds No.	Violative compounds %	Violative samples No.	Violative samples %
		Profenofos	2	0.06–0.14	0.1			—[c]				
		Tolclophos-Me	1	0.12–0.12	0.12			—[c]				
		Triazophos	1	0.03–0.03	0.03			0.05				
		Dithiocarbamates (8)[c]	3	0.1–0.12	0.11			3.0				

[a] Values for 90th percentile were not calculated because the frequency of the pesticide present in each commodity was less than 10 samples. It was only calculated in case of grape: dicofol = 1.27 mg/kg, malathion = 0.16 mg/kg, dithiocarbamates = 2.48 mg/kg; strawberry: dicofol = 0.56 mg/kg; and tomato: dithiocarbamates = 0.5 mg/kg.

[b] MRLs issued by the Codex Committee of Pesticide Residues, 1996.

[c] No MRL available.

[d] EMRL = extraneous maximum residue limits.

[e] Number of samples analyzed for mentioned compounds.

The number of samples analyzed per item needs to be increased for a better representation of contamination in the markets.

Dithiocarbamates were found in 69 (70.4%) of 98 samples analyzed for dithiocarbamate: 3 of 8 (37.5%) strawberry samples, 9 of 16 (56.3%) cucumber samples, 23 of 23 (100%) tomato samples, 32 of 32 (100%) grape samples. None of the samples analyzed contained dithiocarbamates in excess of the MRLs established by CCPR, except for one grape sample, with dithiocarbamates at 6.8 mg/kg. In general, the data indicate application of good agriculture practices. In addition, levels of dithiocarbamate residues found were lower than those found in a previous monitoring study in 1993 (14).

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