Multiresidue Methods Used in The Netherlands and Residue Data Obtained in 1993

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SUMMARY

In this lecture an overview will be given of the Pesticide Residue Sampling and Monitoring Program in The Netherlands and the multiresidue methods used to identify and quantitate the pesticide residues in fruit and vegetable samples. Special attention will also be given to the method validation program and the quality assurance system maintained. Finally, summarized data of the pesticide residues found during our monitoring program in 1993 will be presented.

INTRODUCTION

The Inspectorate for Health Protection (IHP) is an executive department of the Ministry of Health of the Netherlands with 13 regional Inspectorates, better known as Food Inspection Services (FIS), which are spread over the country. The IHP is the largest Special Investigation Service in the Netherlands and has a total of 850 employees, divided over the 13 regions. A regional Inspectorate is divided into an administrative department, a field-inspectors department, a microbiological and a chemical laboratory, and a labortatory for specialized tasks with regard to a certain product group.

The major task of the IHP, in addition to the tasks related to the Health Law, is the enforcement and promotion of compliance with, amongst others, the Food and Commodities Law, the Meat Inspection Law, and the Pesticides Law. Priority is given to activities related to public health, product safety, consumer protection and information and the promotion of fair trade. Beside the control of foodstuffs, the Inspectorate is also concerned with other (non-food) commodities, such as cosmetics, toys, packaging materials, household chemicals, sports articles, and in Alkmaar, tobacco and textile products. The Inspectorate also has to respond to consumer complaints about products or shops and restaurants.

One of the major tasks of the 13 regional chemical laboratories is the monitoring for pesticide residues in fruits and vegetables, in order to enforce compliance with the Residue Decree (of the Pesticide Law), in which maximum residue limits (MRLs) are fixed for approximately 400 pesticides in all kinds of commodities.

Annually, circa 10.000 samples are analyzed for pesticide residues in the 13 regional laboratories by ± 25 analists. The total cost for the pesticide analyses is about 3 million Dutch guilders (± 1.5 million US dollars)

NATIONAL SAMPLING PROGRAM

The regulatory monitoring program in the Netherlands encompasses the sampling of circa 10.000 lots of domestically produced and imported crop samples. Domestic samples are collected as close as possible to the point of production, that is at the 22 auctions where the harvested crops are traded from the grower to the buyers. Import samples are collected at the importers' and wholesalers' warehouses.

In general, the samples that are collected are "surveillance" samples, that is, there is no prior knowledge about the possible presence of pesticide residues. Occasionally, "compliance" samples may be taken as a follow-up to the detection of pesticide residues exceeding maximum residue levels in samples. Although the pesticide-use history of the samples is usually unknown, the residue data obtained in the previous years are considered when determining the number of samples of each commodity.

In the national sampling plan, the ratio of domestic/imported commodities sampled is circa 70:30. This ratio is based on both the ratio between domestically produced crops and imported crops, which is about 4:1, and on the consumption pattern, which roughly shows a 1:1 ratio between domestic and imported products.

The 10.000 samples taken annually, are collected in the 13 regions of the country and analyzed by the corresponding Inspectorate for Health Protection (IHP)/Food Inspection Service (FIS) laboratories. The division of the samples over the 13 labs is determined by the production volume and/or the trade volume at the regional auction of the domestic commodities in a region , and the annual turnover of the imported commodities traded at the wholesalers and importers. Other factors taken into account are knowledge about commodity/pesticide usage, climatic conditions, dietary importance, residue data obtained in recent years, and, last but not least, analytical lab capacity and capabilities. At the regional FIS Alkmaar, usually 1000 samples are collected and analyzed yearly.

SAMPLING PROCEDURE

The samples are taken by field inspectors of the Food Inspection Services according to the sampling procedures as laid down in the Dutch Pesticide Law. In summary, this means that each sample should be composed of at least 3, 5 or 10 grasps (at random) out of a lot that weighs <50, 50-500, or >500 kg, respectively. The whole sample should consist of at least 1-5 kg, and 10 individual pieces of the commodity. Of each sample, sub-samples are made that are representative for the whole sample and thus the original lot. This so-called laboratory sample is sealed at the sampling site and is then, accompanied by a sample information sheet, transferred to the chemical laboratory for analysis, as soon as possible.

PESTICIDE RESIDUE TOLERANCES

For about 400 pesticides, tolerances or maximum residue limits (MRLs) are set in the Residue Decree of the Dutch Pesticide Law. MRLs are set for commodity groups and/or single commodities. Part of the Residue Decree is already adapted for the harmonized EC tolerances (for 42 pesticides) and the EC commodity grouping.

The registration of pesticides and fixing of residue tolerances is determined by the College for Admission of Pesticides under the shared responsibility of the Ministry of Agriculture and the Ministry of Health. The major criteria for accepting pesticide use and fixing MRLs are Good Agricultural Practice (GAP), toxicological and environmental properties, residue data from supervised trials and the harmonization processes in the EC and Codex.

ANALYTICAL METHODS

Multiresidue methods (MRMs), that is methods capable of detecting a large number of pesticides in one procedure, are routinely used to monitor for the pesticide residues in the fruit and vegetable samples. At FIS Akmaar, a modified Luke-extraction method has been developed and applied successfully over the last decade.

The miniaturized Luke method is based on a 1-minute, "one-tube" extraction of a homogenized 15-g crop sample with 30 ml acetone in a 200-ml centrifuge tube, by means of a Polytron (or Ultraturrax) homogenizer, followed by a 1-minute partitioning step in the same tube, after addition of a dichloromethane/petroleum-ether (DCM/PE) mixture (30/30 ml). After a 3-min. centrifugation, the clear organic phase (a acetone/DCM/PE mixture) is transferred to an Erlenmeyer flask. This is the central extract from which aliquots are taken for analysis via the various multiresidue detection systems.

Sample extracts are analyzed via 4 GC-detection systems, namely GC-ITD (MS), GC-ECD, GC-NPD and GC-FPD.

The GC-ITD method is a promising multiresidue method, capable of identifying and quantitating circa 320 GC-compatible analytes fully automatically. The method has been developed at the FIS Alkmaar, using the standard instruments' software. An extensive calibration file with all 320 pesticides' retention times (plus a certain retention time window), full mass spectra and quantitation ions has been composed. The automated method searches in the sample chromatogram for the presence of each pesticide within its corresponding retention time window, by comparing the pesticides' library mass spectra with the spectra of the peaks in the sample extract. If the fit value, representing the similarity between the library and unknowns' spectrum, exceeds the predetermined value, and if the signal to noise ratio is >2, the pesticide is considered to be detected and positively identified. The concentration is then automatically calculated based on the set quantitation ion. Finally, each positive finding is afterwards visually inspected for final confirmation.

With the GC-ITD system (in the electron-impact mode), a total of circa 320 pesticides can be automatically detected during our daily routine analyses. The same number of pestides (except propargite, biphenyl and o-phenyl-phenol) can also be detected by the other 3 GC-detection systems, namely GC-ECD, GC-FPD and GC-NPD. These systems detect the classes of organochlorine-, organophosphorus-, and organonitrogen/phosphorus pesticides, that can be chromatographed on a capillary GC. These 3 methods are still used parallel with the GC-ITD, because the chance to detect a pesticide is dependant on the pesticide/commodity combination and the sensitivity/selectivity of the typical GC-detection system. Based on our 2-year experience in routine practice, it can be concluded, that at the present time, GC-ITD can not yet fully replace the array of conventional GC-detection systems. Accordingly, the combined use of all GC-detection systems rather guarantees an optimal efficiency in interpretation of chromatograms and tracing the maximum number of pesticide residues.

Next to the GC multiresidue methods, 2 HPLC methods are applied routinely in our pesticide monitoring. The multiresidue method for N-methylcarbamates, which has been developed in our FIS lab in Alkmaar, is capable of detecting 22 parent carbamates and 12 of the major metabolites via automated solid-phase extraction cleanup and HPLC with post-column "solid-phase" hydrolysis and fluorescence detection. The second HPLC method applied is a so-called selective multiresidue method for the determination of the fungicides thiabendazole and carbendazim.

A very practical aspect of the combination of the 6 multiresidue methods is the possibility of taking aliquots out of the same sample extraxt for (optional) cleanup and subsequent detection with the chromatography-detector combinations.

QUALITY ASSURANCE / QUALITY CONTROL

As part of our quality assurance system, all the analytical methods in use in our laboratory, have to be validated. Besides, all the pesticide standard solutions have to be checked as to purity and accuracy. In 1993 and 1994 this validation process has consumed a considerable amount of time. As a result, the pesticide residue department of the FIS Alkmaar has recently been officially accredited by the national IHP quality assurance unit.

Standard pesticides are checked very thoroughly, via both intra- and interlaboratory comparison studies. Two FIS labs (in Alkmaar and Amsterdam) independently prepare stock solutions (1.0 mg/ml in toluene) from the neat standard (preferably from different suppliers and/or different batch or lot numbers), by two analysts per lab. Then, corresponding diluted stock solutions (10 µg/ml in iso-octane/toluene) are made. In a second dilution step (with iso-octane), working solutions are prepared for an intralab check of the two independent stock solutions on the analytical instrument (GC or LC). If the quantitative result (detector response) of the two independant standard does not differ more than 5% (chosen criterium), the standard is "approved" within-lab. Subsequently, standard mixtures for the various multiresidue methods are composed in duplicate, again by two analysts, and compared on GC or LC as described above. The same criteria are followed.

When the standard mixtures meet the requirements in both laboratories, these are exchanged and quantified against each other in both labs. If the interlab comparison also results in response differences smaller than 5%, the standard mixtures are approved and, consequently, the original standard stock solutions of both the individual pesticides and the pesticide mixtures are then considered as accurate and "validated".

If the intercomparison checks within the lab or between the two labs do not meet the criteria, the whole procedure from weighing the neat standard to quantitative comparison is repeated. If this does not lead to a positive result, one of the two labs has to order a new neat standard, preferably from a different supplier than the one used. Working this way, we have succeeded in preparing 2 "interlab-validated" standard mixtures for GC-ECD (18 OC-pesticides) and GC-NPD (18 OP-pesticides), respectively, and all our necessary "intralab-validated" standard mixtures.

When new standard mixtures have to be prepared, the new solution is always checked against the old solution. The same criteria are kept and the same procedures are followed as outlined above. All relevant data about degradation, GC problems, etc. are archived.

Next to the standard solutions used, also the analytical multiresidue methods have been or are currently being validated. This means, that for the circa 100 most detected pesticides during routine monitoring, calibration lines have been constructed, recovery and precision data (repeatibility, n=10) for one matrix at 2 concentration levels have been collected, as well as reproducibility data (average for various relevant matrices) at one concentration level within the normal working range.

During routine analyses, standard control samples are always included in the sample series. In order to avoid degradation problems or cross influences between pesticides in matrix, the control sample is prepared freshly each day, by adding the various standard mixtures to a, for that day, representative (homogenized) matrix type. The control sample are further treated exactly the same way as the other samples in the series. The quantitative results for all the pesticides are included in Shewhart control charts for quality assurance.

It should be noted here, that (in GC analysis) it is necessary to quantitate all pesticides against a standard mixture, added to a (preferably the same) matrix, in order to avoid matrix-effected detector responses and to guarantee reliable, quantitative results.

As a final built-in quality check in our system, it is mandatory, that in case a pesticide residue concentration exceeds the tolerance level and the FIS invokes sanctions or prosecution, the complete analysis has to be repeated by a second, independant analyst. Of course, special criteria hold for the acceptable, quantitative difference between the two analytical results.

By including the automated GC-ITD method in the routine monitoring, the requirements of GC-mass spectrometric identification and confirmation are met.

PESTICIDE RESIDUE FINDINGS IN THE NETHERLANDS IN 1993.

In 1993, 752 fruit and vegetable samples were analyzed under regulatory monitoring. In these samples 3525 multiresidue methods analyses were performed, as shown in detail, per commodity, in Table 1. The GC-ITD method (not included in Table 1) was incorporated in the routine analyses as a screening method during the second half of the year for, in total, 363 samples. From 1994, this method is executed routinely as a qualitative and quantitative method, though.

Of the samples collected, 427 were domestic (56.7%) and 307 (40.8%) were imported, while of 18 samples (2.5%), the origin could not be traced. Of the 307 imported samples, 201 originated from countries of the European Union, namely Spain (110), France (37), Italy (37) and Greece (10). A detailed overview of the origin of the samples by commodity is given in Table 2.

Table 3 shows, by commodity, the number of samples collected and the types and incidence of pesticides detected. In the 752 samples 971 pesticide residues in total were detected. It should be kept in mind here, that the number of pesticides that can be detected per single sample may vary between 0 and 10 ! Also, the level at which a pesticide can be detected is dependent on the typical pesticide/ commodity combination and the detection method applied. The lower limit of residue measurement is usually well below tolerance levels. These tolerance levels generally range from 0.01 to 50 mg/kg. Residue levels at 0.01 mg/kg and above are mostly detectable. For some individual pesticides, the detection limit may deviate and range down to 0.001 mg/kg, or occasionnally, also up to 1 mg/kg. The improvement of the analytical capabilities in recent years has contributed considerably to the increase in the number of pesticides detected. Not only the scope of the methods have been extended, but also the detection limits have become lower and lower.

In Table 3, the number of exceedings of MRLs is also given, by pesticide/commodity combination. The total number of exceedings was 83, which corresponds with a violation rate of 11.0%, relative to the total number of samples. In 1993 and 1991, these violation rates were 10.2% and 13.2%, respectively. Because some samples had more than one violative pesticide residues, the number of *samples* with violative residues was 63, which is 8.4% of the samples. This percentage of violative samples was in 1992 and 1991 8.6% and 10.8%.

More detailed information (e.g. origin, pesticide levels and MRLs) about the samples with violative residues is shown in Table 4. In total 41 different pesticides caused the exceedings of MRLs. Two pesticides, pyridaphenthion and tebuconazole, were not even included in the Dutch Residue Decree. Methiocarb was the pesticide with the most violations (11x) observed.

Of the 427 domestic samples, 30 (7.0%) were violative, while 32 (10.4%) of the imported samples were violative. The origin of 1 violative sample was unknown. The higher violation rate for imported products, relative to domestic products, is a trend that has been observed for many years. On the one hand, this may be (partly) arbitrary, but, on the other hand, can also be (partly) explained by the differences in MRLs and registered pesticides between various European countries and also the differences between the types of products being analyzed from different countries. Some products collected (see Table 2) are only domestic or are only imported (e.g. citrus fruit, bananas).

Besides, the sampling year, the region of origin of the products, the time of the season, the climatic conditions, and the historic pesticide residue data all can play a role in the ultimate violation rate for products of a particular country. A fair comparison will always be difficult, although the harmonization process for pesticide registration and fixing MRLs in the European Union is an important factor and is definitely a step forward. Unfortunately, the completion of this work will probably take at least 10 years.

Table 5 contains information on residue findings by commodity, including the number of samples analyzed, the percentage of samples with no residues, with residues below MRLs and with residues exceeding MRLs. Overall, 45.1% of the samples were free of residues (compared with 55% in 1992!). Residues, which were not violative, were detected in 46.5% of the samples. The percentage of samples with over-tolerance residues amounted to 8.4%.

In Table 6a an overview, in alphabetical order, is given of all the pesticides detected in the 752 samples during the regulatory monitoring. Table 6b shows the same list of pesticides, but now in the order of incidence. In total, 80 different pesticides (metabolites not counted) were detected. The same pesticides as those in 1992 were detected most frequently, namely vinchlozoline, tolylfluanide, captan, iprodion and thiabendazole. The number of pesticides, including metabolites, that were detectable by the GC and HPLC multiresidue methods used, was circa 340.

Overall, it can be concluded, that, at the FIS lab in Alkmaar, the scope of the methods used for the determination of pesticides in fruits and vegetables has been extended considerably during the last decade and the quality of the residue data produced has increased as well. In the next years, these methods and high-quality standards will be disseminated over the other regional laboratories in the Netherlands, in order to complete the process of national harmonization of methods and quality assurance. Comparable work has to be done in the near future within the whole European Union. The exchange of knowledge and methods between experts of different countries at symposia and workshops is, therefore, a prerequisite.

TABLE 1

Total number of samples of fruits and vegetables analyzed for pesticide residues in 1993 and the multiresidue methods applied

Commodity	Number of samples	Multiresidue methods				
		A	B	C	D	E
apple	25	25	25	25	25	18
apricot	4	3	4	4	4	2
asparagus	3	3	3	3	3	3
avocado	4	3	4	4	0	0
balm	1	1	1	1	1	1
banana	14	13	14	14	14	10
beans	31	30	31	31	31	19
beet, red	3	3	3	3	3	3
blackberry	2	2	2	2	2	0
blueberry	2	2	2	2	2	0
broccoli	6	6	6	6	6	3
brussels sprouts	9	9	9	9	8	2
cabbage, chinese	8	8	8	8	8	8
cabbage, pointed	3	3	3	3	3	3
cabbage, red	2	2	2	2	2	2
cabbage, savoy	1	1	1	1	1	1
cabbage, white	1	1	1	1	1	1
carrot	28	28	28	28	28	18
carrot, winter-	2	2	2	2	2	2
cauliflower	19	19	19	19	19	17
celeriac	3	3	3	3	3	1
celery	8	8	8	8	8	8
celery, b	3	3	3	3	3	2
cherry	5	5	5	5	5	5
chervil	1	1	1	1	1	1
chicory	9	9	9	9	9	6
chicory, red	1	1	1	1	1	1
coconut	1	1	1	1	0	0
cucumber	19	19	19	19	19	17
currant	32	32	32	32	32	22
date	1	0	1	1	0	0
dill	1	1	1	1	1	1
eggplant	4	3	4	4	4	2
endive	30	30	30	30	30	21
fennel	1	1	1	1	1	1

TABLE 1 (cont.)

Total number of samples of fruits and vegetables analyzed for pesticide residues in 1993 and the multiresidue methods applied

Commodity	Number of samples	Multiresidue methods				
		A	B	C	D	E
garlic	1	1	1	1	1	0
garter	1	1	1	1	1	0
gooseberry	2	2	2	2	2	1
grape	12	12	12	12	12	1
grapefruit	7	5	7	7	7	1
kale	8	8	8	8	8	6
kiwi	6	5	6	6	6	4
kohlrabi	1	1	1	1	1	1
leek	5	5	5	5	5	4
lemon	5	5	5	5	5	3
lettuce (cabbage-)	11	11	11	11	11	8
lettuce, curled	2	2	2	2	2	0
lettuce, cut	2	2	2	2	2	2
lettuce, iceberg	22	22	22	22	22	14
lettuce, oak-leaf	6	6	6	6	6	5
lollo rosso	7	7	7	7	7	4
mandarin	8	8	8	8	8	7
mango	2	2	2	2	2	1
melon	11	11	11	11	11	8
millet	1	1	1	1	1	0
mint	1	1	1	1	1	1
nectarine	3	3	3	3	3	2
onion	5	5	5	5	5	5
orange	35	34	35	35	35	26
other fruits and nuts	3	3	3	3	3	1
others agricultural products	7	7	7	7	7	1
parsley	13	13	13	13	13	6
pea, field	1	1	1	1	1	1
peach	4	4	4	4	4	3
peas, sweet	2	2	2	2	2	1
pineapple	1	1	1	1	1	0
plum	4	4	4	4	4	2
potato	14	14	14	14	11	10
pursiane	6	6	6	6	6	6
radish	6	6	6	6	6	6

TABLE 1 (cont.)

Total number of samples of fruits and vegetables analyzed for pesticide residues in 1993 and the multiresidue methods applied

Commodity	Number of samples	Multiresidue methods A	B	C	D	E
radish, black	1	1	1	1	1	1
raisin	2	2	2	2	2	2
raspberry	3	3	3	3	3	3
rhubarb	8	8	8	8	8	8
rice	20	20	20	20	20	13
rye	4	4	4	4	4	3
shallot	1	1	1	1	1	1
spinach	11	11	11	11	11	10
strawberry	92	92	92	92	92	78
sweet pepper	29	29	29	29	29	25
tomato	33	32	33	33	33	28
tumip-tops	4	4	4	4	4	4
wheat	26	26	26	26	26	17
zucchini	6	5	6	6	6	4
Total number of samples:	752					
Total number of analyses:	3525	740	752	752	742	539

A=Organochloro-pesticides; B=Org. Phosph./sulfur-pesticides; C=Organonitrogen-pesticides; D=N-Methyl-carbamates; E=Benzimidazole-fungicides

TABLE 2

Countries of origin of samples collected and analyzed in 1993

Commodity	Country of origin* Total	NL	imp.	unk.	Country of origin (EC)* B	DK	D	E	F	GB	GR	I	Total	Country of origin* other import
currant	32	32												
potato	14	7	5	2	1				4				5	
strawberry	92	87	5					5					5	
apricot	4		4						1		2		3	1xSA
pineapple	1		1											1xDR
endive	30	24	4	2				1				3	4	
apple	25		24	1					20			1	21	1xMZ, 2xSA
asparagus	3	3												
eggplant	4		3	1				1					1	2xIL
avocado	4		4											2xIL, 2xSA
banana	14		13	1										6xCO, 4xCR, 3xPA
blueberry	2	2												
celery, b	3		3					2				1	3	
cualiflower	19	10	9					3	2			4	9	
kale	8	8												
beans	31	16	15					6				1	7	3xSG,5xEG
onion	5	5												
blackberry	2	2												
broccoli	6	1	5					2				3	5	
cabbage, chinese	8	8												
lemon	5		5					5					5	
balm	1			1										
coconut	1		1											1xCL
date	1		1											1xSA
dill	1			1										
grape	12		12						1		4	5	10	1xSA, 1xEG
lettuce, oak-leaf	6	5	1									1	1	
raspberry	3	3												
millet	1		1											1xUSA
grapefruit	7		7											1xAR, 1xCO, 1xCU, 2xIL, 2xUSA
lettuce, iceberg	22	13	8	1				7	1				8	
pea, field	1	1												
cherry	5		5								3	1	4	1xAR
chervil	1	1												
kiwi	6		6					1				2	3	3xMZ
garlic	1	1												
celeriac	3	3												
cucumber	19	17	2					2					2	
kohlrabi	1	1												
garter	1			1										
beet, red	3	3												
lettuce (cabbage-)	11	11												
gooseberry	2	2												
lettuce, curled	2	2												
lollo rosso	7	6	1									1	1	
mandarin	8		8					3					3	5xMR
mango	2		2											1xSA, 1xUSA
melon	11	1	10					7					7	1xBR, 1xIL, 1xSA
mint	1			1										
nectarine	3		3					1	1				2	1xSA
other fruits/nuts	3		3											2xIL, 1xMA
other agric. prod.	7	3	2	2										1xIL, 1xZB
sweet pepper	29	10	18	1				15					15	3xIL
peach	4		4					1	1		1	1	4	
parsley	13	11	2					1				1	2	
peas, sweet	2		2											1xZB, 1xEG

TABLE 2 (cont.)

Countries of origin of samples collected and analyzed in 1993

Commodity	Country of origin* Total	NL	imp.	unk.	Country of origin (EC)* B	DK	D	E	F	GB	GR	I	Total	Country of origin* other import
zucchini	6	2	4					4					4	
purslane	6	5		1										
leek	5	5												
plum	4		4					2					2	1xRM, 1xSA
turnip-tops	4	4												
rhubarb	8	8												
radish	6	5	1											1xIL
radish, black	1	1												
rice	20		20									1	1	1xGY, 9xSM, 9xUSA
cabbage, red	2	2												
rye	4	1	3				2			1			3	
chicory, red	1		1									1	1	
raisin	2		2											2xTR
cabbage, savoy	1	1												
celery	8	8												
orange	35		34	1				25					25	1xCU, 1xIL,7xMR
shallot	1	1												
lettuce, cut	2	2												
spinach	11	11												
cabbage, pointed	3	2	1					1					1	
brussels sprouts	9	8		1										
wheat	26	9	17			1	7		6	3			17	
tomato	33	15	18					13				1	14	2xIL, 2xMR
fennel	1		1									1	1	
carrot, winter-	2	2												
chicory	9	9												
cabbage, white	1	1												
carrot	28	26	2					2					2	
Total number of samples	752	427	307	18	1	1	9	110	37	4	10	29	201	106

* country codes:
NL=Netherlands, imp.=Import, unk.=UNknowm.
EC countries: B=Belgium, DK=Denmark, D=Germany, E=Spain, F=France, GB=Great-Britain, GR=Greece, I=Italy.
Other countries: AR=Argentina, BR=Brazil, CL=Chill, CO=Columbia, CR=Costa Rica, CU=Cuba, DR=Dominican Republic, EG=Egipt, GY=Guayana, IL=Isral, MA=Malayssa, MR=Morocco, NZ=New-Zeeland, PA=Panama. RM=Romania, SA=South-Africa, SG=Senegal, SN= Surinem, TR=Turkey, USA=United States, ZB=Zimbabwe.

Table 3
Commodities collected and frequency of ocurrence of pesticides detected in 1993

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
32	currant	Azinphos-methyl	1	
		Bupirimate	1	
		Captan	8	
		Chinomethionate	1	
		Demeton-s-methyl	3	
		Dimethoate	3	
		Iprodion	2	
		Methiocarb	1	
		Methomyl	1	
		Omethoate	2	
		Parathion	2	
		Pirimicarb	17	
		Propoxur	5	
		Tolyfluanide	21	
		Vinchlozolin	10	
14	potato	-		
92	strawberry	Bifenthrin	1	1
		Bitertanol	1	1
		Buripimate	10	
		Captan	38	2
		Carbaryl	2	
		Carbendazim (incl. benomyl)	1	
		Carbofuran	2	
		Chlorpyrifos	1	1
		Dicofol	11	
		Dimethoate	1	
		Endosulfan	7	
		Folpet	1	1
		Heptenophos	2	
		Iprodion	14	
		Malathion	3	
		Methiocarb	11	3
		Metalaxyl	2	
		Methomyl	1	
		Mevinphos	6	
		Penconazole	3	1
		Pirimicarb	9	
		Pirimiphos-ethyl	1	
		Procymidon	4	1
		Propoxur	8	
		Pyrazophos	8	1
		Tetradifon	2	
		Tolyfluanide	41	
		Triazophos	1	1
		Triadimenol	2	2
		Vinchlozolin	31	

TABLE 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
4	apricot	Azinphos-ethyl	1	
		Captan	3	
1	pineapple	-		
30	endive	Iprodion	5	
		Lindane	1	
		Methiocarb	2	1
		Mevinphos	3	
		Pirimicarb	2	1
		Propoxur	1	
		Pyrazophos	1	
		Tolclophos-methyl	4	2
		Vinchlozolin	6	
25	apple	Azinphos-methyl	3	
		Biphenyl	1	
		Bromopropylate	1	
		Captan	3	
		Carbaryl	5	
		Carbendazim (incl. benomyl)	11	
		Chlorpyrifos	5	
		Dicofol	2	
		Diphenylamine	10	
		Dimethoate	3	
		Ethion	1	
		Fluvalinate	1	1
		Phosalone	13	
		Iprodion	1	
		Lindane	1	
		Methomyl	1	
		Procymidon	1	
		Propargite	2	
		Thiabendazole	10	
		Tolyfluanide	3	
3	asparagus	Sulfur	1	
4	eggplant	Chlorothalonil	1	
		Dichloran	1	
		Dimethoate	1	
		Endosulfan	1	
		Omethoate	1	
		Permethrin	1	
		Vinchlozolin	1	
4	avocado	-		
14	banana	Chlorpyriphos	3	
		Thiabendazole	10	
2	blueberry	-		

Table 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
3	celery, b	Chlorpyriphos	1	
		Chlorothalonil	1	
		Iprodion	1	
		Methomyl	1	
19	cauliflower	Captan	1	
		Dimethoate	1	
8	kale	Lindane	1	
31	beans	Endosulfan	1	
		Malathion	1	
		Vinchlozolin	3	
5	onion	Chlorfenvinphos	1	
		Chlorothalonil	1	1
2	blackberry	Captan	1	
		Tolyfluanide	2	
		Vinchlozolin	1	
6	broccoli	Captan	1	
		Carbofuran	2	
		Chlorpyrifos	1	
		Propoxur	1	
8	cabbage, chinese	Ethiofencarb	1	
		Iprodion	1	
		Methiocarb	1	
		Permethrin	1	
		Pirimicarb	1	1
5	lemon	Azinphos-methyl	1	
		Chlorfenvinphos	3	
		Dicofol	1	
		Ethion	1	
		Imazalil	3	
		Mecarbam	2	
		Methidathion	1	
		Ortho-phenylphenol	1	
		Quinalphos	1	1
		Thiabendazole	2	
1	balm	-		
1	coconut	-		
1	date	Malathion	1	
1	dill	-		

TABLE 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
12	grape	Acephate	1	1
		Azinphos-methyl	2	
		Carbaryl	1	
		Cypermethrin	1	
		Dichloran	1	
		Dicofol	1	
		Dimethoate	2	
		Endosulfan	1	
		Fenpropathrin	3	1
		Flucythrinate	1	1
		Folpet	1	
		Iprodion	1	
		Methiocarb	2	
		Methidathion	1	
		Methomyl	1	
		Monocrotophos	1	1
		Omethoate	1	
		Parathion	1	
		Parathion-methyl	2	1
		Procymidon	3	
		Tetradifon	1	
		Vinchlozolin	1	
6	lettuce, oak-leaf	Carbofuran	1	
		Methomyl	2	
		Parathion	1	
		Permethrin	1	
		Pirimicarb	1	1
		Procymidon	1	1
3	raspberry	Pirimicarb	1	
		Tolyfluanide	1	
1	millet	-		
7	grapefruit	Biphenyl	1	
		Bromopropylate	2	
		Butocarboxim	2	
		Captan	1	
		Carbaryl	2	1
		Chlorpyrifos	2	
		Dicofol	2	
		Imazalil	1	
		Malation	2	
		Ortho-phenylphenol	1	
		Procymidon	1	
		Thiabendazole	6	
22	lettuce, iceberg	Endosulfan	1	
		Methomyl	2	2
		Procymidon	1	

Table 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
1	pea, field	-		
5	cherry	Azinphos-ethyl	1	
		Captan	1	
		Diazinon	1	
		Dimethoate	1	
1	chervil	-		
6	kiwi	Carbendazim (incl. benomyl)	1	
		Chlorpyrifos	1	
		Diazinon	1	
		Dimethoate	1	
		Iprodion	4	
		Pirimiphos-methyl	1	
		Vinchlozolin	5	
1	garlic	-		
3	celeriac	Iprodion	1	
19	cucumber	Dichlorvos	1	1
		Endosulfan	1	
		Methiocarb	1	1
		Oxamyl	2	
		Procymidon	2	
		Tolyfluanide	1	
1	kohlrabi	-		
1	garter	-		
3	beet, red	Dimethoate	1	
		Tolclophos-methyl	1	1
11	lettuce (cabbage-)	Folpet	1	
		Iprodion	6	
		Methiocarb	2	
		Metalaxyl	1	1
		Mevinphos	1	
		Pirimicarb	1	
		Tolclophos-methyl	7	
		Vinchlozolin	6	
2	gooseberry	Captan	1	
		Endosulfan	1	
		Tolyfluanide	1	
		Vinchlozolin	1	
2	lettuce, curled	-		

TABLE 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
7	lollo rosso	Demeton-s-methyl	1	
		Methiocarb	1	
		Parathion	1	
8	mandarin	Azinphos-methyl	1	
		Bromopropylate	1	
		Carbaryl	1	
		Carbendazim (incl. benomyl)	1	
		Chlorpyrifos	3	
		Dichloran	1	1
		Dicofol	5	
		Dimethoate	1	
		Imazalil	6	
		Malathion	2	
		Methidathion	5	
		Prochloraz	4	1
		Thiabendazole	5	
2	mango	Prochloraz	1	
11	melon	Dimethoate	1	
		Endosulfan	3	
		Fenitrothion	1	
		Imazalil	1	1
		Pirimicarb	1	
1	mint	-		
3	nectarine	Acephate	1	1
		Methamidophos	1	1
3	other fruits and nuts	Chlorpyriphos	1	
		Methiocarb	1	
7	other agricultural product	Chlorothalonil	1	
		Dichloran	1	
		Dimethoate	1	
		Endosulfan	1	
		Omethoate	1	
29	sweet pepper	Carbendazim (incl. benomyl)	2	
		Chlorpyrifos-methyl	9	
		Chlorothalonil	2	
		Dichlofluanide	1	
		Dicofol	2	
		Endosulfan	2	
		Heptenophos	1	
		Iprodion	8	
		Malathion	1	
		Methiocarb	9	5
		Methomyl	1	1

TABLE 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
		Procymidon	2	
		Tebuconazole	2	2
		Triadimenol	2	
		Vinchlozolin	3	
		Sulfur	1	
4	peach	Acephate	2	2
		Methamidophos	2	2
		Pirimicarb	1	
13	parsley	Chlorothalonil	2	
		Endosulfan	1	
		Heptenophos	1	1
		Iprodion	3	
		Mevinphos	2	
		Permethrin	1	1
		Tolclophos-methyl	3	1
		Vinchlozolin	1	
2	peas, sweet	Chlorothalonil	1	1
6	zucchini	Endosulfan	3	
		Methiocarb	1	1
6	purslane	Tolclophos-methyl	2	
5	leek	-		
4	plum	Ethion	2	
		Methomyl	2	
		Pirimiphos-methyl	1	
4	turnip-tops	Chlorothalonil	1	
		Tolclophos-metyl	1	
		Vinchlozolin	2	
8	rhubarb	Pirimiphos-methyl	1	1
6	radish	Carbofuran	1	
		Tolclophos-methyl	1	
		Vinchlozolin	1	
1	radish, black	-		
20	rice	Pirimiphos-methyl	2	
2	cabbage, red	-		
4	rye	Pirimiphos-metyl	3	
1	chicory, red	-		

TABLE 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
2	raisin	Bromopropylate	1	1
		Dicofol	1	1
		Procymidon	1	1
		Propargite	1	1
1	cabbagge, savoy	-		
8	celery	Captan	1	
		Chlorothalonil	1	
		Heptenophos	2	2
		Iprodion	1	
		Mevinphos	1	1
		Pirimicarb	1	1
		Tolclophos-methyl	2	1
		Vinchlozolin	1	
35	orange	Azinphos-methyl	8	
		Bromopropylate	1	
		Butocarboxim	6	
		Carbaryl	2	
		Carbendazim (incl. benomyl)	1	
		Chlorfenvinphos	1	
		Chlorpyrifos	9	
		Chlorpyrifos-methyl	1	
		Diazinon	3	
		Dicofol	9	
		Dimethoate	5	
		Endosulfan	3	
		Ethion	1	
		Fenitrothion	3	
		Fenthion	1	
		Phenthoate	2	
		Fluvalinate	1	1
		Phosmet	1	
		Imazalil	25	
		Malathion	4	
		Mecarbam	1	
		Metalaxyl	1	
		Methidathion	17	
		Monocrotophos	1	
		Ortho-phenylphenol	2	
		Parathion	1	
		Parathion-methyl	1	
		Pirimiphos-methyl	3	
		Prochloraz	1	1
		Pyridaphenthion	4	4
		Quinalphos	1	
		Tetradifon	5	
		Thiabendazole	12	
		Vinchlozolin	2	

Table 3 (cont.)

Number of samples	Commodity	Pesticides	Frecuency of occurrence	Number of exceedings of MRL
1	shallot	-		
2	lettuce, cut	Iprodion	1	
		Vinchlozolin	2	
11	spinach	Tolclophos-methyl	2	
3	cabbage, pointed	Methiocarb	1	
9	brussels sprouts	-		
26	wheat	Chlorpyriphos-methyl	1	
		Dichlorvos	1	
		Malathion	1	
		Pirimiphos-methyl	6	
		Propoxur	1	
33	tomato	Bitertanol	3	
		Bupirimate	1	
		Carbendazim (incl. benomyl)	4	
		Chlorothalonil	1	
		Dichlorvos	1	1
		Endosulfan	1	
		Methiocarb	1	
		Oxamyl	1	
		Procymidon	3	
		Tolyfluanide	2	
		Vinchlozolin	4	
		Sulfur	1	
1	fennel	-		
2	carrot, winter-	Chlorfenvinphos	2	1
		Iprodion	1	
9	chicory	Dimethoate	5	
		Pirimicarb	1	
		Vinchlozolin	1	
1	cabbage, white	-		
28	carrot	Chlorfenvinphos	12	
		Hexachlorobenzene	2	2
		Iprodion	2	
		Lindane	1	
		Procymidon	1	1
		Quintozene	2	2
		Tolclophos-methyl	1	1
Totals: 752			Total: 971	Total: 83

TABLE 4

Exceedings of Maximun Residue Tolerances (MRLs) in 1993

Commodity	Country*	Pesticide	Residue conc. (mg/kg)	MRL# (mg/kg)
strawberry	E	Bifenthrin	0.60	0* (0.05)
strawberry	E	Folpet	4.1	3
		Triadimenol	0.27	0* (0.1)
strawberry	E	Procymidon	6.4	5
		Triadimenol	1.3	0* (0.1)
		Chlorpyrifos	0.20	0.05*
strawberry	NL	Methiocarb	0.28	0.05*
strawberry	NL	Methiocarb	0.26	0.05*
strawberry	NL	Triazophos	0.11	0.01*
strawberry	NL	Captan	4.1	3
strawberry	NL	Methiocarb	0.08	0.05*
strawberry	NL	Captan	7.0	3
strawberry	NL	Penconazole	0.73	0.2
strawberry	NL	Bitertanol	0.57	0* (0.05)
		Pirazophos	0.47	0.1
endive	NL	Methiocarb	1.9	1
endive	NL	Tolclophos-methyl	0.26	0* (0.01)
endive	NL	Tolclophos-methyl	0.51	0* (0.01)
endive	NL	Pirimicarb	1.3	1
apple	F	Fluvalinate	0.07	0* (0.05)
beet	NL	Tolclophos-methyl	0.09	0* (0.05)
onion	NL	Chlorothalonil	1.1	0.5
lemon	E	Quinalphos	0.03	0* (0.02)
zucchini	E	Methiocarb	0.12	0.05*
grape	GR	Fenpropathrin	0.10	0* (0.05)
grape	I	Acephate	0.14	0* (0.02)
		Parathion-methyl	0.37	0.2
		Flucythrinate	0.13	0* (0.02)
grape	GR	Monocrotophos	0.10	0.05*
oak-leaf lettuce	NL	Procymidon	23.0	0* (0.01)
		Pirimicarb	0.57	0* (0.02)
grapefruit	IL	Carbaryl	1.2	1
iceberg lettuce	E	Methomyl	0.04	0* (0.02)
iceberg lettuce	E	Methomyl	0.04	0* (0.02)
cucumber	NL	Methiocarb	0.08	0.05*
		Dichlorvos	0.19	0.1
mandarin	E	Prochloraz	0.32	0* (0.05)
mandarin	E	Dichloran	0.06	0* (0.01)
melon	IL	Imazalil	1.2	0.5
nectarine	F	Methamidophos	0.02	0* (0.01)
		Acephate	0.16	0* (0.02)
chinese cabbage	NL	Pirimicarb	0.13	0* (0.05)
sweet pepper	E	Tebuconazole	0.24	-
sweet pepper	E	Methiocarb	0.09	0.05*

TABLE 4 (cont.)

Exceedings of Maximun Residue Tolerances (MRLs) in 1993

Commodity	Country*	Pesticide	Residue conc. (mg/kg)	MRL# (mg/kg)
sweet pepper	E	Methiocarb	0.53	0.05*
		Tebuconazole	0.18	-
sweet pepper	E	Methiocarb	0.16	0.05*
sweet pepper	E	Methiocarb	0.53	0.05*
sweet pepper	E	Methiocarb	0.20	0.05*
sweet pepper	E	Methomyl	0.08	0* (0.02)
peach	F	Methamidophos	0.04	0* (0.01)
		Acephate	0.33	0* (0.02)
peach	GR	Methamidophos	0.05	0* (0.01)
		Acephate	0.62	0* (0.02)
parsley	NL	Permethrin	11.7	2
		Tolclophos-methyl	0.85	0* (0.05)
parsley	NL	Heptenophos	0.58	0.1
sweet peas	EG	Chorothalonil	0.37	0.02*
rhubarb	NL	Pirimiphos-methyl	0.23	0* (0.02)
raisin	TR	Bromopropylate	0.54	0 (EKO)
		Dicofol	0.17	0 (EKO)
		Procymidon	0.32	0 (EKO)
		Propargite	0.25	0 (EKO)
celery	NL	Tolclophos-methyl	0.09	0* (0.05)
celery	NL	Pirimicarb	0.14	0* (0.05)
		Heptenophos	12.0	0.1
celery	NL	Heptenophos	1.2	0.1
		Mevinphos	0.21	0.1
orange	?	Fluvalinate	0.46	0* (0.05)
orange	E	Pyridaphenthion	0.14	-
orange	E	Pyridaphenthion	0.21	-
orange	E	Pyridaphenthion	0.12	-
orange	E	Prochloraz	0.45	0* (0.01)
		Pyridaphenthion	0.21	-
lettuce	NL	Metalaxyl	0.27	0.1
tomato	NL	Dichlorvos	0.50	0.1
winter-carrot	NL	Chlorfenvinphos	0.70	0.5
carrot	NL	Tolclophos-methyl	0.22	0* (0.05)
carrot	NL	Procymidon	0.13	0* (0.01)
carrot	NL	Quintozene	0.05	0* (0.01)
		Hexachlorobenzene	0.07	0.05
carrot	NL	Quintozene	0.03	0* (0.01)
carrot	NL	Hexachlorobenzene	0.10	0.05

* Country code: NL=Netherlands, E=Spain, F=France, I=Italy, GR=Greece, TR=Turkey, IL=Israel, EG=Egypt.
# 0* = Residues of this pesticide may not be present.
0.01* = This pesticide may be applied on this commodity, however, without leaving any detectable residues. The concentration depicted here is considered as the «practible» detection limit.

TABLE 5
Number of samples analyzed and number of samples with and without pesticide residues

Commodity	Number of samples	Samples without residues	Samples with residues < tolerance	Samples with residues > tolerance
apple	25		24	1
apricot	4	1	3	
asparagus	3	3		
avocado	4	4		
balm	1	1		
banana	14	4	10	
beans	31	26	5	
beet, red	3	1	1	1
blackberry	2		2	
blueberry	2	2		
broccoli	6	3	3	
brussels sprouts	9	9		
cabbage, chinese	8	5	2	1
cabbage, pointed	3	2	1	
cabbage, red	2	2		
cabbage, savoy	1	1		
cabbage, white	1	1		
carrot	28	10	13	5
carrot, winter-	2		1	1
cauliflower	19	17	2	
celeriac	3	2	1	
celery	8	4	1	3
celery, b	3	1	2	
cherry	5	2	3	
chervil	1	1		
chicory	9	3	6	
chicory, red	1	1		
coconut	1	1		
cucumber	19	14	4	1
currant	32	4	28	
date	1		1	
dill	1	1		
eggplant	4	2	2	
endive	30	12	14	4
fennel	1	1		
garlic	1	1		
garter	1	1		
gooseberry	2		2	
grape	12	1	8	3
grapefruit	7		6	1
kale	8	7	1	
kiwi	6		6	
kohlrabi	1	1		

Table 5 (cont.)

Number of samples analyzed and number of samples with and without pesticide residues

Commodity	Number of samples	Samples without residues	Samples with residues < tolerance	Samples with residues > tolerance
leek	5	5		
lemon	5		4	1
lettuce (cabbage-)	11		10	1
lettuce, curled	2	2		
lettuce, cut	2		2	
lettuce, iceberg	22	17	3	2
lettuce, oak-leaf	6	4	1	1
lollo rosso	7	5	2	
mandarin	8		6	2
mango	2	1	1	
melon	11	6	4	1
millet	1	1		
mint	1	1		
nectarine	3	2		1
onion	5	3	1	1
orange	35		30	5
other fruits and nuts	3	1	2	
others agricultural products	7	6	1	
parsley	13	8	3	2
pea, field	1	1		
peach	4	2		2
peas, sweet	2	1		1
pineapple	1	1		
plum	4	2	2	
potato	14	14		
purslane	6	4	2	
radish	6	4	2	
radish, black	1	1		
raisin	2	1		1
raspberry	3	1	2	
rhubarb	8	7		1
rice	20	18	2	
rye	4	1	3	
shallot	1	1		
spinach	11	9	2	
strawberry	92	5	76	11
sweet pepper	29	14	8	7
tomato	33	15	17	1
turnip-tops	4	2	2	
wheat	26	19	7	
zucchini	6	2	3	1
Total	752	339	350	63
	100%	45,1%	46,5%	8,4%

TABLE 6a

Frequency of occurrence of pesticides detected in samples analyzed in 1993 (*in alphabetic order*)*

Pesticide	Occurrence	Pesticide	Occurrence
Acephate	4	Lindane	4
Azinphos-ethyl	2	Malathion	15
Azinphos-methyl	16	Mecarbam	3
Bifenthrin	1	Metalaxyl	4
Biphenyl	2	Methamidophos	3
Bitertanol	4	Methidathion	24
Bromopropylate	6	Methiocarb	34
Bupirimate	12	Methomyl	12
Butocarboxim	8	Mevinphos	13
Captan	59	Monocrotophos	2
Carbaryl	13	Omethoate	5
Carbendazim (incl. benomyl)	21	Ortho-phenylphenol	4
Carbofuran	6	Oxamyl	3
Chinomethionate	1	Parathion	6
Chlorfenvinphos	19	Parathion-methyl	3
Chlorothalonil	12	Penconazole	3
Chlorpyrifos	27	Permethrin	4
Chlorpyrifos-methyl	11	Phenthoate	2
Cypermethrin	1	Phosalone	13
Demeton-s-methyl	4	Phosmet	1
Diazinon	5	Pirimicarb	36
Dichlofluanide	1	Pirimiphos-ethyl	1
Dichloran	4	Pirimiphos-methyl	17
Dichlorvos	3	Prochloraz	6
Dicofol	34	Procymidon	20
Dimethoate	27	Propargite	3
Diphenylamine	10	Propoxur	16
Endosulfan	27	Pyrazophos	9
Ethiofencarb	1	Pyridaphenthion	4
Ethion	5	Quinalphos	2
Fenitrothion	4	Quintozene	2
Fenpropathrin	3	Sulfur	3
Fenthion	1	Tebuconazole	2
Flucythrinate	1	Tetradifon	8
Fluvalinate	2	Thiabendazole	45
Folpet	3	Tolclophos-methyl	24
Heptenophos	6	Tolyfluanide	72
Hexachlorobenzene	2	Triadimenol	4
Imazalil	36	Traizophos	1
Iprodion	52	Vinchlozolin	82
* *in 752 samples*		Total	971

Table 6b

Frequency of occurrence of pesticides detected in samples analyzed in 1993 (*in order of frequency of occurrence*)*

Pesticide	Occurrence	Pesticide	Occurrence
Vinchlozolin	82	Acephate	4
Tolyfluanide	72	Bitertanol	4
Captan	59	Demeton-s-methyl	4
Iprodion	52	Dichloran	4
Thiabendazole	45	Fenitrothion	4
Imazalil	36	Lindane	4
Pirimicarb	36	Metalaxyl	4
Dicofol	34	Ortho-phenylphenol	4
Methiocarb	34	Permethrin	4
Chlorpyrifos	27	Pyridaphenthion	4
Dimethoate	27	Triadimenol	4
Endosulfan	27	Dichlorvos	3
Methidathion	24	Fenpropathrin	3
Tolclophos-methyl	24	Folpet	3
Carbendazim (incl.benomyl)	21	Mecarbam	3
Procymidon	20	Methamidophos	3
Chlorfenvinphos	19	Oxamyl	3
Pirimiphos-methyl	17	Parathion-methyl	3
Propoxur	16	Penconazole	3
Azinphos-methyl	16	Propargite	3
Malathion	15	Sulfur	3
Carbaryl	13	Azinphos-ethyl	2
Mevinphos	13	Biphenyl	2
Phosalone	13	Fluvalinate	2
Bupirimate	12	Hexachlorobenzene	2
Chlorothalonil	12	Monocrotophos	2
Methomyl	12	Phenthoate	2
Chlorpyrifos-methyl	11	Quinalphos	2
Diphenylamine	10	Quintozene	2
Pyrazophos	9	Tebuconazole	2
Butocarboxim	8	Bifenthrin	1
Tetradifon	8	Cypermethrin	1
Bromopropylate	6	Chinomethionate	1
Carbofuran	6	Dichlofluanide	1
Heptenophos	6	Ethiofencarb	1
Parathion	6	Fenthion	1
Prochloraz	6	Flucythrinate	1
Diazinon	5	Phosmet	1
Ethion	5	Pirimiphos-ethyl	1
Omethoate	5	Traizophos	1
* *in 752 samples*		Total	971