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ANNEX

to

Monitoring of Pesticide Residues

in Products of Plant Origin

in the European Union, Norway and Iceland

1999 Report

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2. COUNTRY COMMENTS

2.1. BELGIUM

Pesticide Residue Monitoring in Fruit, Vegetables and Cereals in Belgium - 1999

This report shows the results of pesticide residue monitoring on fresh fruits and vegetables, cereals, and processed foods on the Belgian market in 1999.

The samples were taken by trained officials from the Food Inspection Service from the Ministry of Social Affairs, Public Health and the Environment (1092 samples), or from the Service for Plant Quality and Plant Protection from the Ministry of Middle Class and Agriculture (495 samples). Sampling was done mostly according to directive 79/700/EEC, at auctions, importers, wholesalers, processors and retail. Of these samples, 552 can be considered to be compliance samples, following alerts on chlormequat in Belgian pears and methamidophos in Spanish peppers (fresh and frozen).

In selecting the commodities, the methods of analysis and the number of samples, several factors were taken into account : the average consumption, the Belgian production figures, the rate of exceedences and findings of previous years, the analytical and budgetary possibilities. For less important commodities, a rolling programme is preferred. The different groups of fruits and vegetables were included as much as possible. The co-ordinated programme of the European Commission was of course included in the national programme. In total 8 different sorts of fruit, 11 different sorts of vegetables, potatoes, wheat grains, and processed pear based foodstuffs were analysed.

Samples were refrigerated and analysed within 2-10 days. Two officially recognized laboratories were involved. A multiresidue method was performed on most samples. Dithiocarbamates and benzimidazoles were also frequently analysed. Bromide ion was measured in all commodities that are frequently grown in greenhouses (lettuce, lamb's lettuce, celery and peppers). Especially on leafy vegetables and stem vegetables, a whole range of analyses were carried out, including propamocarb and N-methylcarbamates. Some single residue methods were performed on specific commodities, e.g. daminozide in apples, and propham/chlorpropham on potatoes, imidacloprid in apples, cyproconazole in leek. Chlormequat was sought for in pears, pear products, apples and carrots. Acephate, methamidophos and triazophos were only measured in some commodities for the European co-ordinated programme. In frozen peppers, only methamidophos was sought.

The two officially recognized laboratories involved in the monitoring programme were accredited for the most important analytical methods and commodities, and accreditation was gradually extended. The certificates of accreditation of the « Wetenschappelijk Instituut Volksgezondheid-Louis Pasteur (Afdeling Voedingswaren) » and the « Laboratorium voor Fytofarmacie » can be found on the website of the accreditation body BELTEST (<http://beltest.fgov.be/>). Both laboratories had participated in the European Commission's Proficiency Tests and in the Workshops on Co-ordinated Analytical Quality Control. The laboratories take into account annex II of Commission Recommendation 1999/333/EC.

A total of 1587 samples were analysed. For the random monitoring programme, 262 fruit, 706 vegetable, 38 potato, and 29 cereal samples were counted. For the compliance monitoring, 442 pear samples, 24 samples pear syrup, 37 samples babyfood and 49 samples fresh and frozen sweet peppers were analysed. The 394 leafy vegetable samples (lettuce, lamb's lettuce, endive, spinach

and witloof) represented 38% of the random monitoring samples and 25% of the total amount of samples.

Out of a list of 138 different pesticide residues, a total of 61 were found at least once during this random monitoring programme. The ten most frequently found pesticide residues, in descending order of number of findings, are: iprodione, bromide ion, dithiocarbamates, tolclofos-methyl, propamocarb, vinclozolin, tolylfluanid, procymidone, carbendazim and imazalil. In 54% of the samples, no residues were found.

Pesticides were found most often in lemons (85%) and lettuce grown in greenhouses (79%). A maximum of 7 different pesticide residues could be found in one sample (lettuce). No samples with pesticides were found for cauliflower, and for pineapple, carrot, spinach and witloof, in less than 10% of the samples pesticide residues were found.

The latter was also true for babyfood containing pears. During compliance sampling of pears and pear syrup, chlormequat was detected in more than 80% of the samples.

Slightly more exceedences are related to national MRLs than to EC-MRLs, when the compliance monitoring was not taken into account. Note that exceedences were counted **not** taking into account the uncertainty on the analytical result. A lot of exceedences are related to MRLs at the limit of determination. High percentages for exceedences were found for celery and table grapes. For the compliance monitoring on Spanish sweet peppers, more than half of the samples were not conform European legislation.

In nine lots of sweet peppers with methamidophos, 10 units were analysed separately. Variation factors ranged from 1.6 to 7.0, with a mean value of 3.4.

The results are presented in Tables A to F, in the format recommended by the Commission.

Table A1 gives, separately for monitoring and compliance samples, data on the number and origin of samples, and results as to the MRL. Table A2 is a list of pesticides which were included in analytical methods used during this monitoring programme. It gives the number of samples in which the pesticide is sought, the number of findings and the reporting level. Not only the pesticides mentioned in EC-directives are included, but all pesticides. The reporting level is not lower than the legal limit of determination. When laboratories had a higher determination limit than that in the legislation, this higher level is the reporting level. For instance the reporting levels are higher for dithiocarbamates, iprodione, and imazalil. Table B gives the detailed results of the co-ordinated programme. Table C contains the detailed results of the national programme, including the co-ordinated programme. Only pesticides which were found at least once, are listed. Please note that the exceedences counted in Table B and C are mathematical exceedences, not taking into account the uncertainty of the analytical result. Table D1 gives a list of exceedences of EC harmonised MRLs, which were higher than 1.5 x MRL. Similarly, table D2 lists exceedences of national MRLs. Table E gives information on multiple residues in single samples of the Food Inspection Service. Table F gives the results for the variability exercise.

2.2. DENMARK

Pesticide Residue Monitoring of Fruits, Vegetables and Cereals in Denmark -1999

Introduction

This report to the Commission of the European Communities includes national and co-ordinated monitoring results from the Danish monitoring programme 1999.

A total of 2287 samples of fruit and vegetables were analysed at regional or central food control laboratories, all accredited to pesticide analysis.

Danish produce as well as commodities imported from other member states and non-member states were examined.

Sampling

Sampling plans were designed by the Danish Veterinary and Food Administration, the Institute of Food Research and Nutrition on the basis of dietary consumption pattern, production and import data, and monitoring results from previous years.

Samples were taken by authorised personnel from local food control units mainly at wholesalers and importers. 25% of the Danish grown samples were taken at producers.

46% of the total 2287 samples were fruit, 45% were vegetables and 8% were cereals. 38% were of domestic origin, 62% were grown in other EU or third countries. Sampling included 4% commercially frozen produce and 3% organically grown samples.

Analysis and quality assurance

The analytical methods are developed and validated by the Danish Veterinary and Food Administration and the analyses performed by three regional laboratories and one central laboratory, all accredited for pesticide analysis in compliance with EN45001 by the Danish body of accreditation, DANAK (certificate numbers 288, 304, 315, and 350). All laboratories participate in the relevant FAPAS performance verification scheme. Laboratories analysing fruit and vegetables participated in the EU proficiency testing.

Guidelines concerning Quality Control Procedures for Pesticide Residue Analysis has been applied to some extent for all methods. The concept of Lowest Calibration Level has not been implemented for all multi methods. Mass selective confirmation was performed for specialized methods with cereals.

The fruit and vegetables were analysed for approximately 129 pesticides using a GC multi method based on extraction with acetone/cyclohexane/ethyl acetate, a HPLC method for fungicides (benomyl-group, thiabendazole, biphenyl and orto-phenylphenol) and a spectrophotometric method for the sum of dithiocarbamates (maneb-group). The cereals were analysed for 28 pesticides using a GC multi method and two LC-MS/MS methods (glyphosate and AMPA, resp. chlormequat and mepiquat).

In the analytical methods 1-2 spiked samples were included in each analytical batch. The GC multi methods included spiked samples containing 8-15 pesticides at 2 concentration levels alternating between all pesticides. Quantification was performed with a matrix-matched standard or a pure solvent standard. The calibration standard was matched to the expected concentration within a factor of 2. The reporting levels entered in Table A are the general levels of quantification based on signal to noise response in combination with sample interference and recovery experiments. However, lower quantification levels are achievable for many pesticide/crop combinations. Samples with pesticide residues exceeding the MRL were confirmed by analyses at another laboratory except findings of dithiocarbamate. Due to the methodology applied it is not possible to distinguish between dithiocarbamates included in the MRL definition and other dithiocarbamates for which no MRL has been fixed. Thus it is not possible to report confirmed exceedings from the maneb-group.

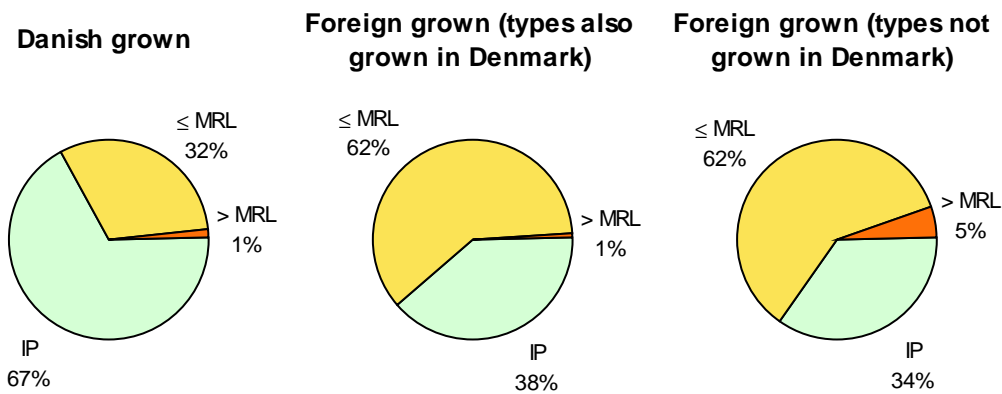
Results

Pesticide residues were found in 61% of fruit samples, 13% of vegetable samples and 17% of cereal samples (excluding organically grown samples and findings of glyphosate, AMPA, chlormequat and mepiquat).

50 (27%) of the cereal samples were analysed for glyphosate, AMPA, chlormequat and mepiquat. Residues were found in 86% of these samples.

64 samples of organically grown vegetables and 29 samples organically grown cereals were analysed. Pesticide residues were found in two samples of grapes and in 4 cereal samples.

Residues exceeding the maximum residue limits (MRLs) were found in 45 samples corresponding to 2% of all samples.



Figur 1:

Pesticide residues in fruit.

Excluding results from frozen or organic grown samples

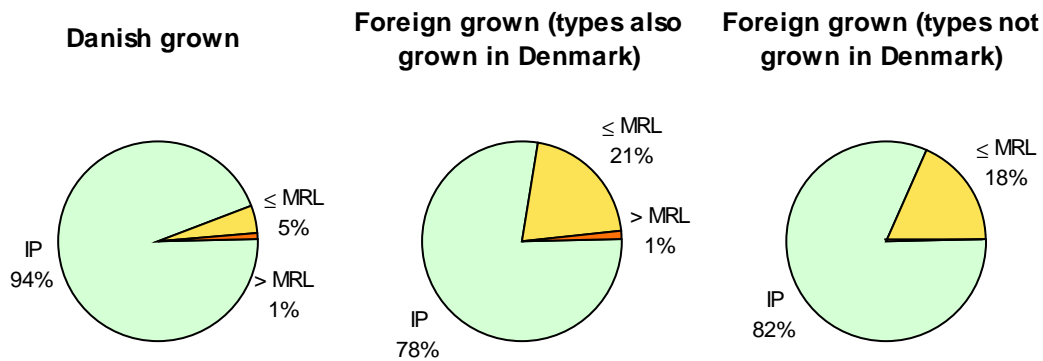


Figure 2: *Pesticide residues in vegetables.*

Excluding results from frozen or organic grown samples

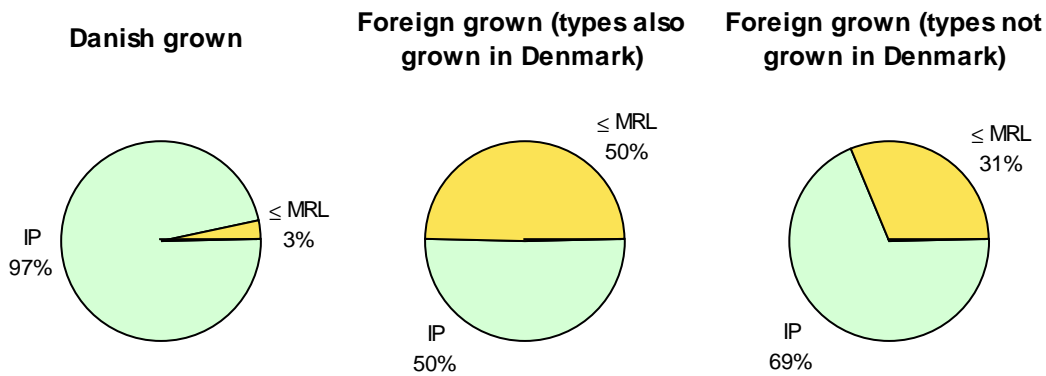


Figure 3: Pesticide residues in cereals.

Excluding results from organic grown samples and excluding results for glyphosate, AMPA, chlormequat and mepiquat.

A toxicological evaluation was performed for samples with residues exceeding the MRL, with multiple residues and with residues where no MRL was set. The evaluation concluded that the detected residues did not constitute any health hazard to the consumer. However, findings of chlorinated pesticides such as hexachlorbenzene, DDT, Lindane and HCH are undesirable, due to accumulation in the organism. Three samples had residues that exceeded the Acute Reference Dose, ArfD. One of these (methamidophos in cauliflower) was evaluated as toxicologically unacceptable. The following up of violations lies with the local authorities (The Municipal Food Control Units). Most frequently the action for confirmed exceedings was to order the companies to update their own-check procedures.

Substances have been reported according to their residue definition. Thus o,p'-DDE (found in one sample) and AMPA (found in nine cereal samples) has been reported separately from DDT and glyphosate. Due to the methodology applied results for the maneb-group may include dithiocarbamates not included in the MRL-definition.

Residues of more than one pesticide were found in 16% of all samples

The pesticides most frequently detected in fruit and vegetables were in descending order: Imazalil, benomyl-group, maneb-group, thiabendazole, endosulfan, chlorpyrifos, ortho-phenylphenol, procymidone, methidathion, and iprodione. Nine substances were found in cereal samples (in descending order of frequency): chlormequat, glyphosate, AMPA, mepiquat, pirimiphos-methyl, malathion, chlorpyrifos-methyl, fenitrothion, and propiconazole.

2.3. GERMANY

Pesticides Residue Monitoring in Food for the Year 1999

Summary of the Results from the Federal Republic of Germany

In Germany there is a difference between Monitoring Programme and official food control. Parts of the data generated in both programmes correspond with the provisions of Directives 86/362/EEC, 86/363/EEC, 90/642/EEC and the Commission Recommendation of 3 March 1999 (1999/333/EC) concerning a coordinated Community monitoring programme. Therefore, the report includes the respective data from both programmes:

- Monitoring programme 2651 samples
- official food control 10349 samples.

The report contains the results of the tests carried out in food of animal origin, grain, fruit and vegetables during the 1999 survey period. It includes all of the data transmitted by 49 accredited laboratories of the food control laboratories of the Laender to the 'Federal Institute for Health Protection of Consumers and Veterinary Medicine, BgVV'.

Samples were taken on the level of producers, manufacturers, wholesalers and retailers, and restaurants according to a national sampling protocol published as official legal regulations. The active ingredients tested for were ingredients which had already been included in the Annexes to Directives 86/362/EEC, 86/363/EEC or 90/642/EEC.

In the course of the 1999 survey period, a total of 13.000 (1998: 11.920; 1997: 12.362; 1996: 11.870) samples were tested for pesticide residues in the Federal Republic of Germany. In 7446 (57.3%) samples residues of pesticides were not determinable and in 5156 (39.7%) samples, residues of pesticides were detected in compliance with official, national and EU MRLs. Only 398 (3.06%) samples contained residues of pesticides exceeding MRLs.

Table 1 shows the number of samples divided into domestic production and imports and takes into account the three Directives. The overview given in this table indicates that residues of imported samples exceeded MRLs more frequently than those of domestic foods. The reason could surely be seen in the fact that parts of the imported foods originated from countries with other climatic conditions which require an increased use of pesticides.

Table 1 Overview of the results of the tests performed on domestic and foreign produce.

Food item	Domestic samples				Import samples			
	total amount	without residues	residues up to MRL	residues above MRL	total amount	without residues	residues up to MRL	residues above MRL
Cereals	301	171	126	4	108	79	28	1
Animal origin	5613	2937	2671	5	770	393	377	0
Fruit/vegetable	2420	1935	419	66	3788	1931	1535	322
Total	8334	5043	3216	75	4666	2403	1940	323

The food samples were analysed for a total of 74 different pesticides from which 61 were detected at least in one sample. Residues of 29 individual pesticides exceeded MRLs.

2.4. GREECE

SUMMARY OF PESTICIDE RESIDUE MONITORING RESULTS OF GREECE FOR THE YEAR 1999

1. EU COORDINATED PROGRAMME.

- 171 samples of the products specified in the 99/333/EC Commission Recommendation were examined by the coordinated laboratory. All of these were analyzed for GLC amenable pesticides (acephate, chlorpyrifos, chlorpyrifos-methyl, deltamethrin, diazinon, endosulfan, imazalil, iprodione, lambda-cyhalothrin, mecarbam, metalaxyl, methamidophos, methidathion, permethrin, pirimiphos-methyl, thiabendazole, triazophos, vinclozolin). 110 of them were also analyzed for benomyl group and 43 for maneb-group.
- 147 samples (86% of the total number of samples) contained not detectable residues of the pesticides sought.
- 17 samples (9.9%) contained detectable residues at or below the MRL.
- In 7 samples (4.1%) residues of a pesticide were detected exceeding the EU MRLs.

2. NATIONAL PROGRAMME

- 1422 samples were examined by 6 laboratories.
- 920 samples (64.7% of the total number of samples) contained not detectable residues of the pesticides sought.
- 400 samples (28.1%) contained detectable residues at or below the MRL.
- In 68 samples (4.8%) residues of one or more pesticides at concentrations exceeding the EU or national MRLs were detected (64 exceeded the EU and 4 the national MRL).
- In 83 samples (5.8%) residues of pesticides for which no EU or national MRLs are established were detected.
- The total number of pesticides analyzed for was 123.
- The 10 most frequently pesticides found were: chlorpyrifos (101 samples), fenthion (91 samples), endosulfan (76 samples), benomyl group (42 samples), phosalone (38 samples), maneb group (36 samples), chlorpyrifos methyl (30 samples), methamidophos (28 samples), chlorothalonil (27 samples) and captan (26 samples).
- The reporting levels are generally those established by the Council or Commission in the EU MRL Directives, or those indicated by FAO/WHO Codex Alimentarius. In one case (methamidophos) the EU LOD of 0.01 mg/kg could not be achieved and the limit routinely achievable (0.02 mg/kg) is reported. In some cases the reporting levels are those routinely achieved in the laboratory.
- Samples were randomly taken from points of entry, wholesalers and retailers.

QUALITY ASSURANCE

The EU guidelines are followed as close as possible. The screening of samples by GLC involves the use of matrix-matched standard mixtures containing the most commonly encountered pesticides, including at least two pesticides with a poor or variable GC response or recovery. Bracketing calibration is used. Although most quantitations are carried out using calibration curves with 3-5 levels covering a limited concentration range, a single calibration point is sometimes used, for example for confirmation of a residue, which has already been quantified on a different GC system. Routine recovery checks and blanks are carried out approximately every 20 samples rather than on the basis of batches, because of the uneven flow of samples to the laboratory. All the laboratories are in the phase of preparation for accreditation.

Table 1. Overview of the results from the Greek pesticide monitoring programme 1999.

	Co – ordinated Programme		National Programme		
	Number	% Total	Number	% Total	
Total samples analysed	171	100%	1422	100 %	
Samples with non detectable residues	147	86%	920	64.7 %	
Samples with detectable residues at or below the MRL	17	9.9%	400	28.1 %	
Samples with detectable residues of one or more pesticides exceeding the EU or national MRLs	7	4.1%	68		4.8%
			EU MRLs	National MRLs	
			64	4	
Samples with detectable residues of pesticides for which no EU or national MRLs are established	0	0%	83	5.8%	

2.5. SPAIN

[Translated Extract from the 1999 Spanish national monitoring reports for fruit and vegetables and cereals]

FRUIT AND VEGETABLES

Introduction

The surveillance programme is implemented by the Autonomous Communities on the basis of annual plans along the following lines, in accordance with previously agreed criteria:

- 1 - Establishing in advance the total number of samples in proportion to production of each vegetable product in each Autonomous Community;
- 2 - Surveillance of residues of active pesticide substances as listed in Community Directives 76/895, 93/58/EEC, 94/30/EEC, 95/30/EEC, 96/32/EC and 98/82, and of the remaining pesticides which may be used following authorisation and establishment of MRLs in Spain. Table I shows those active substances which are subject to monitoring and the analytical method used.

The samples corresponding to the 1999 plan were taken by the plant health departments of the Autonomous Communities in accordance with the principles laid down in Directive 79/700.

Analysis of the samples under this surveillance programme was carried out in the Autonomous Communities' residue analysis laboratories and in the agri-foodstuffs testing laboratory of the Ministry of Agriculture, Fisheries and Food.

The analysis methods used make it possible to work with a detection limit of between 0.01 and 0.05 mg/kg. They were selected from among internationally recognised methods adapted and validated by the "laboratories working party", comprising qualified technicians from the laboratories taking part in the National Residues Surveillance Plan.

Results and discussion

Tables II-V of this report set out the results of the residues surveillance programme corresponding to the 1999 plan.

Of the total number of 2 898 samples analysed, 1 352 were fruit, 1 304 were vegetables and 242 were other vegetable products.

Of the fruit samples, there were 622 citrus fruits, 356 stone and pome fruit, 155 grapes, 82 strawberries, 21 almonds, 95 olives and 21 tropical fruits.

The vegetables analysed belonged to the following groups: 170 roots and tubers, 702 fruiting vegetables, 120 brassica vegetables, 153 leaf vegetables, 60 green legumes and 99 stem vegetables.

The 242 samples of other vegetable products included seven mushrooms, 22 dry legumes, 127 potatoes and 86 sunflowers.

The results may be summarised as follows:

- in 1 662 samples (equivalent to 57.44%), no residues exceeding the officially established detection limit (harmonised by the EU or national) were found;

- in 963 samples (equivalent to 33.22%), the residues found were less than 50% of the MRLs laid down;
- in 174 samples (6.00%), residues were between 50-100% of MRLs;
- only in 99 samples (3.4%) did residue levels exceed the MRLs laid down (RD 280/1994 and MOs 27/II/96, 5/XII/96, 26/VII/97, 14/V/98 and 31/V/99).

Table II summarises the results, differentiating between fruit, vegetables and other vegetable products.

Fruit: in 540 samples (39.94%), no residues were detected; in 658 samples (48.66%), the residues found were less than 50% of MRLs; in 116 (8.57%), residues were at levels between 50-100% of MRLs; and in 38 (2.81%), residue levels exceeded the MRLs laid down.

Vegetables: analysis of these products revealed that a high percentage (890 samples, 68.25%) contained no residues; in 303 samples (23.23%), the residues found were less than 50% of MRLs; in 55 (4.21%), residues were at levels between 50-100% of MRLs; and in 56 (4.28%), residue levels exceeded the MRLs laid down.

Other vegetable products: In the majority of samples analysed (231, or 95.86%), no residues were detected; in only two samples (0.82%) were residues less than 50% of MRLs; in three (1.23%), residues were at levels between 50-100% of MRLs; and in five (2.06%), levels exceeded the MRLs laid down.

Table IV establishes a relationship; of those pesticides and vegetable products exceeding the MRLs in force, the most frequent were chlorpyrifos, detected in 21 samples: oranges (6), table grapes (1), tomatoes (1), peppers (2), cauliflower (1), beet (1), lettuce (1), celery (2), artichokes (1), carrots (1), potatoes (2) and sunflowers (2); methamidophos in 21 samples: peppers (8), melons (5), watermelon (3), lettuce (3), peaches (1) and table grapes (1); fenthion in six samples: oranges (1), apples (1), peaches (1), apricots (1), mangoes (1) and table grapes (1); dithiocarbamate in four samples: peaches (1), lettuce (2) and potatoes (1); imazalil in six samples: citrus fruits (4), cabbages (1); pyrazophos in 3 samples: cabbages (1), lettuce (1) and artichokes (1). The remaining pesticides which exceeded MRLs were found occasionally in one or two samples.

The number of samples in the 1999 plan which exceeded MRLs (3.41%) is higher than for 1998 (2.38%), due in part to the difficulties of farmers in adapting to the changes to MRLs in Spanish legislation to bring them into line with those laid down in Directive 98/82/EC. In these cases, with products already marketed or acquired by farmers and labelled prior to the changes to the authorised limits, there was insufficient notification of the change.

Table V lists those products analysed by the Autonomous Communities, indicating the pesticides detected and their level in relation to the MRLs laid down.

In Tables IV and V, the MRLs corresponding to those harmonised by the EU have been underlined.

Horticultural products in which more samples containing residues have been found, and the plant protection products most frequently detected, are as follows:

- Citrus fruits: chlorpyrifos, dicofol, phosmet, fenitrothion, imazalil, malathion, metidathion and pirimiphos-methyl, with imazalil being the most frequent;
- Pome fruit: captan + folpet, carbendazim and imazalil;
- Table grapes: chlorpyrifos, dimethoate, fenitrothion and primiphos-methyl;
- Strawberries: carbendazim, iprodione, procymidone and tetradifon;
- Tomatoes: clortalonil, endosulfan, methamidophos and procymidone;
- Peppers: methamidophos and procymidone;

- Lettuce: dimethoate, dithiocarbamates and procymidone.

Levels found were mostly below 50%; only a small percentage were between 50-100% of MRLs.

Table VI at the end of the report compares results for the last four years.

Measures adopted in cases where MRLs are exceeded

In samples where MRLs were exceeded, new samples were taken from the same sampling sites in order to confirm previous results; in almost all cases, these later samples were negative. In those lots where results exceeded the MRLs, appropriate action was taken; the most significant case, in terms of the number of samples detected, was the presence of methamidophos residues in peppers.

On the basis of the results of the samples inspected, and of alerts in other Member States, the Junta de Andalucía drew up a special plan for monitoring methamidophos in peppers and other horticultural products which were affected. In the case of peppers, 1 739 samples were analysed; the results are set out in Annex I. As a result of these controls, 1 000 000 kg of peppers were destroyed.

Current MRLs for methamidophos in peppers are the result of one of the amendments laid down by Directive 98/82/EC, as referred to in the above conclusions, having been reduced from 1.00 mg/kg to 0.01 mg/kg.

CEREALS

Introduction

The surveillance programme is implemented by means of annual plans carried out by the autonomous regions, using an agreed common approach.

When organising the 1999 annual plan, the same approach was adopted as for earlier plans. It consisted of the following:

1. Establishing in advance the total number of samples in proportion to the production of each crop in each autonomous community.
2. Applying the surveillance programme not only to the pesticides referred to in Directives 86/362, 93/57, 94/29, 95/39/EC, 96/33/EC and 98/33/EC but also to other pesticides that may be used because the Spanish authorities have authorised their use in cereals.

Table I lists the active substances monitored.

The samples provided for under the 1999 plan were taken by the plant health departments of the autonomous communities in accordance with the instructions given in Directive 79/700/EEC. They were analysed in the laboratories operated by the autonomous communities and in the agri-foodstuffs testing laboratory of the Ministry of Agriculture, Fisheries and Food in Madrid.

Results and discussion

Tables III-VII contain the results of the 1999 surveillance plan for residues in cereals. These relate to the samples and analyses carried out on the crops harvested in that year.

Of the 250 samples analysed, 85 were of wheat, 101 of barley, 22 of maize, 36 of rice and 6 of oats.

The following results were obtained:

In 232 samples, equivalent to 92.8% of the total, no residues were detected above the officially-established limits of determination (harmonised by the EU or set by Spain).

In 17 samples (6.8% of the total), residues were at below 50% of the MRLs set.

In one sample (0.40% of the total), the residue was at between 50 and 100% of the MRLs. In only one sample (0.40%) was a residue found that exceeded the MRL set (R.D. [Royal Decree] 280/1994 and O.M. [Ministerial Order] 27/2/96, 5/10/96, 26/7/97, 14/5/98 and 31/5/99).

When carrying out the 1999 plan, residues were detected in fewer samples (7.60%) than in the 1998 plan (14.81%).

Table III contains the results for the various cereal products analysed, which can be summarised as follows:

Wheat: no residues were detected in 77 samples (90.58%). Residues were found only in 8 samples (9.4%), where levels were below 50% of the MRLs. No sample was found with residues exceeding the MRLs.

Barley: no residues were detected in 93 samples (92.07%), while residues at levels below 50% of the MRLs were found in 7 samples (6.93%). In one sample a result of between 50-100% of the MRL was obtained. Residue levels exceeding the MRLs were not found in any sample.

Maize: no residues were detected in 21 samples (95.45%). Residues below 50% of the relevant MRL were found only in one sample (4.5%).

Rice: no residues were found in 34 samples (94.4%). Residues were detected in 2 samples, one (2.7%) with a level below 50% of the MRL, the other (2.7%) with a level exceeding the MRL.

Oats: no residues were detected in any of the samples.

Table IV shows the pesticides detected in each plant product, the number of cases being below that occurring in previous plans.

The most frequently occurring pesticides were as follows: malathion in 11 samples: 6 of wheat and 5 of barley; pirimiphosmethyl in 6 samples: 1 of wheat, 3 of barley, 1 of maize and 1 of rice.

In Tables II, VI and VII, the MRLs harmonised by the EU have been underlined, while the other MRLs are those set in Spain by the *Comisión Conjunta de Residuos de Productos Fitosanitarios* (Joint Committee on Residues of Plant Protection Products).

Table V shows the pesticide that exceeded the MRLs. This was molinate, which was found in a rice sample at a concentration of 0.10 mg/kg, the relevant MRL being 0.05 mg/kg.

The batch concerned was traced and a second sample taken to confirm the result. An analysis of this second sample produced a negative result.

At the end of the report, in Table VIII, the results of the last four years are compared.

Analytical quality assurance and accreditation

The analytical quality assurance measures taken in laboratories include the following: checks on instrumentation, use of internationally recognised methods, duplication of analyses where there is uncertainty or where levels exceed the MRLs, confirmation of results using CG - MS or an alternative method if the first is unsuitable.

During implementation of the 1999 plan, a Spanish intercalibration exercise was carried out involving those laboratories integrated into the residues surveillance programme.

It was organised by the University of Almería, using samples of strawberries treated with the following pesticides; carbendazim, cypermethrin, fenitrothion, iprodione, malathion, methamidophos, pirimiphos-methyl, procimidone and thiabendazole. Overall results were satisfactory.

Also in that year, the EU organised Proficiency Test II, involving most of the laboratories in the surveillance programme.

The laboratories in Madrid, Valencia and Zaragoza are currently accredited by the official body ENAC, while accreditation for the remaining laboratories is pending.

2.6. FRANCE

French Pesticide monitoring Results for 1999

The French pesticide monitoring programme for pesticide residues in food was carried out by the General Direction of concurrence and repression of frauds (DGCCRF) .

The objectives of the program were to assume that

- the pesticides was used following the good agricultural practice.
- The health of consumers was protected

The crops or processed foods were collected by the inspectors following the requirements (directive 79/700) at the production, wholesalers (including MIN) and retailers. Then they are sent to the laboratories. The programme of monitoring has been investigated by the central administration of the service and included European and national action.

This program takes in account

- proportion of the crops in the diet
- requirements from EU coordinated program
- some special actions concerning problems with certain crops (lettuce, pears)

Laboratories.

Seven laboratories carried out the monitoring programme but only six reported the results in the format.

All samples were analysed by the multi residue method and some of their by single method

For multi-residue methods the samples were extracted by solvent (acetone or ethyl acetate) then partitioned by liquid-liquid. The purification for organo-halogenated molecules were performed by cartridge, florisil or biobeads. The determination is conducted by ECD, NPD, FPD or hall detector. GC/MS is used as confirmation for samples above MRL's when the techniques is applicable.

Additional methods were used for the determination of methyl carbamate, maneb groups, inorganic bromides, chlormequat (HPLC with conductivity, thin layer chromatography), benomyl group, hydrazide maleique and so on.

228 pesticides can be researched and we have found 115.(Approximately 50%)

Two laboratories were accredited by COFRAC. The accreditation can be occurred only if the methods was validated by collaborative study. The validation " in house" required a lot of investigations. For the new molecules the accreditation seems difficult to be obtained.

The two laboratories were assuming approximately 50% of the control

All laboratories were involved in some proficiency tests with BIPEA and CHECK organisation. At least 4 tests were carried out by year

Results

4553 samples were analysed and the repartition were 30% for the fruits 10% for cereals and 60% on vegetables. In 1999 an investigation was pointed out on chlormequat in pears with 151 samples and 50% was recognised as above the MRL of 3 mg/kg. This active ingredient is not authorised in France and in this case we applied 0 tolerance.

Deleting this enforcement for chlormequat in pears the level on anomalies for fruit should be about 5% (73/1422). Including this operation the number of default were increasing to 10%(152/ 1573)

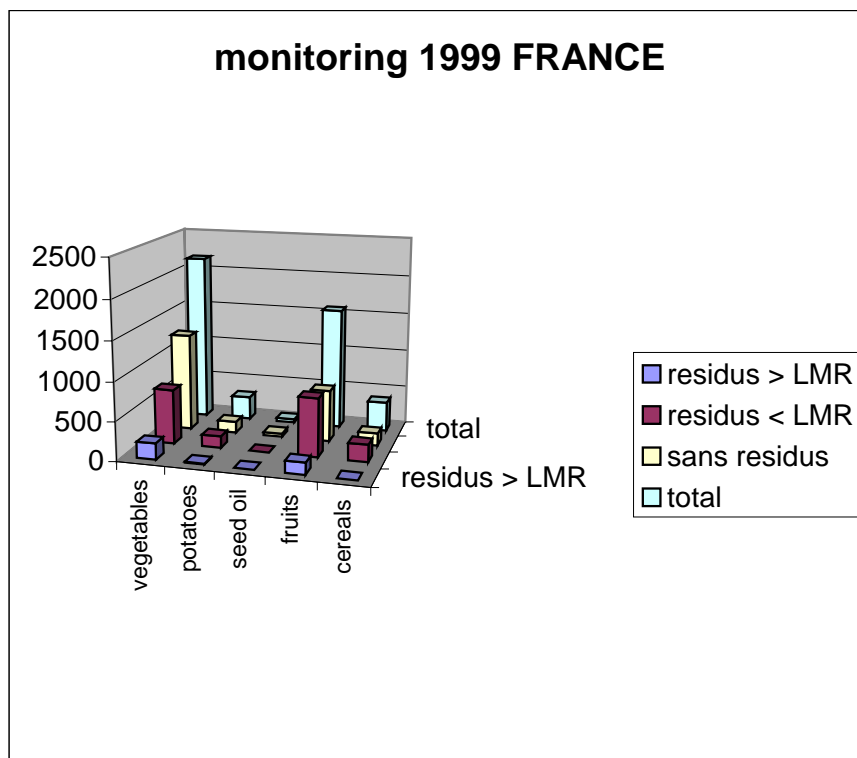
For cereals no samples were above the MRL's.

For vegetables the numbers of samples above the MRL's were affected by the enforcement in the production of lettuce indoor. Excluding lettuce 5% of the samples were above the MRL. Including lettuce the anomalies were increasing still 9.9%. The number of samples was very high (711) and if we take in account the diet we have normally to sampling only 230.

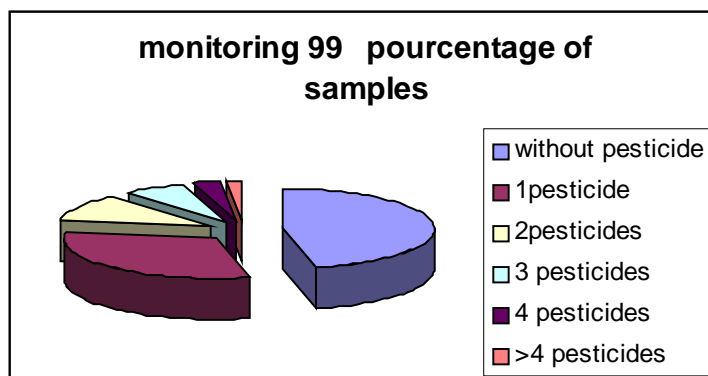
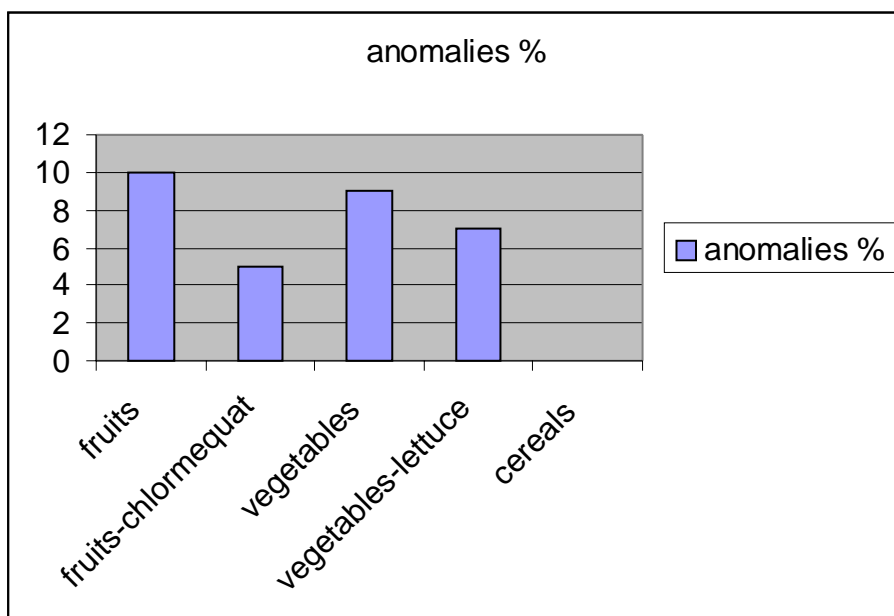
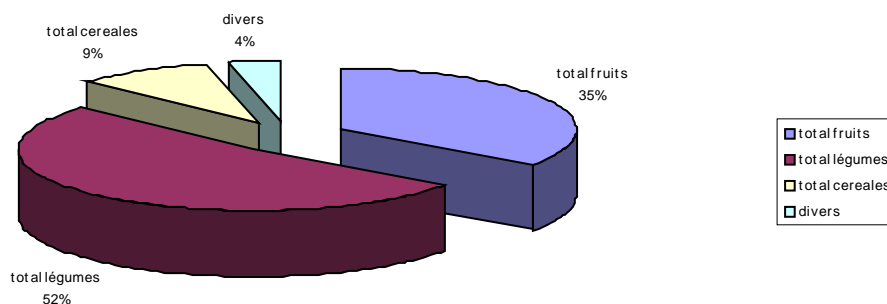
We introduced also some cases where the labelling was not following the regulation. The treatment after harvest for potatoes must be declared and some fruits coming outside of France were declared without treatment and contained pesticides

The pesticides which are found the most frequently were iprodione, dithiocarbamates, vinclozoline, benomyl groups, imazalil, procymidone, thiabendazole, chlorpyriphos, chlorpropham, phosalone.

46,4 % of the samples were found without researched residues, 31% with one pesticide, 11,6% with two pesticides 6,5% with three pesticides, 2,7% with four pesticides and less than 2% more than 4 pesticides



répartition des analyses de résidus de pesticides pour l'année 1999



2.7. IRELAND

Irish Pesticide Monitoring Results for 1999.

The Irish pesticide monitoring programme for pesticide residues in food is undertaken by the Department of Agriculture, Food and Rural Development through its Pesticide Control Service. The objectives of the programme are to ensure that

- pesticides are correctly applied to food
- and that
- consumers are not exposed to unacceptable pesticide residues in food.

In 1999 some 538 samples were analysed for pesticide residues of which 232 were fruit/vegetables while 39 were cereals.

Fruit and vegetables:

54 different types of fruit and vegetables were analysed as part of the monitoring programme. 35.3 % of the samples were of domestic origin and the remaining 64.6 % were either of imported or unknown origin. The samples were analysed for residues of up to 92 pesticides and their metabolites. 46.5% of samples were found to contain pesticide residues while 5.6% of the total number of samples analysed contained residues in excess of the MRL.

Enforcement action is taken by Ireland only in respect to those pesticide MRL's which are defined in the EU residue directives, 76/895, 86/362, 86/363, 90/642 and their amending directives.

Details of the pesticide residues detected are provided in the attached tables. In the fruit and vegetable samples analysed residues of 39 different pesticides were detected. MRL's exist for 34 of these compounds and have yet to be established for the remaining 5.

The pesticides most commonly encountered in fruit and vegetables were Thiabendazole (7.8%), iprodione (7.8%), carbendazim (6.9%), chlorpyrifos (6.4%), endosulfan (6.0%), captan (4.8%), dicofol (4.3%), chlorfenvinphos (3.5%), etc were the residues most commonly detected in the pesticide residue monitoring programme.

The distribution of pesticides found in 1999 is consistent with the results of previous years.

Thiabendazole is primarily detected in pome and citrus fruit;

iprodione was detected in lettuce, berries, stone fruit and in fruiting vegetables; Chlorpyrifos was primarily detected in citrus fruit ;

endosulfan was primarily detected in berries and in cucumbers ;

captan was mainly found in pome fruit ;

chlorfenvinphos was found in root and tuber vegetables.

In all 27 samples (11.6%) contained residues of 2 pesticides, 12 samples (5.2%) contained residues of 3 pesticides, 7 samples (3%) contained residues of 4 pesticides while 2 samples (0.9%) contained residues of 5 pesticides.

Cereals:

Cereal samples, of which 59% were of domestic origin and the remainder were imported or of unknown origin, were analysed for residues of 79 pesticides. Pesticide residues were detected in 3 samples (7.7%) none of which exceeded an MRL. Of the 3 pesticides detected in the cereal samples MRL's have been established for are in place for 2 of these. The pesticides detected were pirimiphos-methyl, etrimphos and malathion.

MRL violations:

13 samples of fruit and vegetables were found to contain pesticide residues in excess of the EU MRL's. 2 of these samples were of domestic origin while the remainder were imported (8 EU production and 3 from outside the EU). 9 of the 13 exceedances related to MRL's which were set at the LOD which suggests that the pesticides were applied to crops illegally.

An assessment of the dietary risk associated with the dietary intake of the pesticide /crop combinations in those cases where the MRL was exceeded did not indicate any risk to the consumer for 11 of the 13 samples concerned.

In the case of two of the samples

- chlorfenvinphos in parsnips
- methamidophos in peaches

a more substantial risk to the consumer was present but on evaluation was also considered to be within acceptable limits.

Quality Control Practices in the laboratory:

- The laboratory was not accredited during 1999.
- The laboratory participates in both EU proficiency and in FAPAS proficiency programmes.
- The work of the laboratory takes into account the guidelines set out in the EU Quality Control Criteria as agreed by the EU residue analysts in co-operation with the EU commission.

2.8. ITALY

Summary of the results from Italy for the year 1999

National Programme (results concerning harmonised and not harmonised pesticides).

The results of general national programme are shown in Table 1.

Table 1

	Nr. of samples	Regular samples				Irregular samples	Irregular samples %
		Without residues	%	With residues	%		
Fruit	4003	2232	55.8	1722	43.0	49	1.2
Vegetables	3799	3104	81.7	644	17.0	51	1.3
TOTAL	7802	5336	68.4	2366	30.3	100	1.3

Samples were analysed using multi residual method, able to determine the presence of 100 or more pesticides. The samples were taken at random on the market and based on dietary consumption and production.

66 laboratories of the National Health Service supplied the data for the official control on pesticide residues but only a part is already *accredited* according to the EN 45001. 30-40 laboratories participated to the third EU Proficiency test in 1999.

Each sample was analysed for, an average, 65 different pesticides (ranging from few compounds to 325 different pesticides). An estimated 508.780 individual determinations were carried out.

In about 12.6% of the analysed samples, residues of more than one pesticide were found.

National programme (results concerning only harmonised pesticides).

This report of the Ministry of Health to the European Commission contains the results of the tests carried out on food of vegetables origin and cereals during the 1999 survey period, concerning only the pesticides listed in the Annexes II of the Directives 86/362/EEC and 90/642/EEC. The results are shown in Table 2.

80.3% of the samples contained no pesticide residues. Detectable residues at or below the MRL were found in 18.8% of the samples.

In 0.9% of the samples the residues exceeded MRLs.

Table 2

	Nr. of samples	Regular samples				Irregular samples	Irregular samples %
		Without residues	%	With residues	%		
Fruit	3876	2754	71.0	1096	28.3	26	0.7
Vegetables	3728	3315	88.9	364	9.8	49	1.3
Cereals	334	303	90.7	31	9.3	0	0.0
TOTAL	7938	6372	80.3	1491	18.8	75	0.9

In about 5.3 % of the analysed samples, residues of more than one pesticide were found.

The pesticides which were found most often are in decreasing order: procimidone, maneb group, chlorpyrifos, imazalil, benomyl group, thiabendazole, iprodione, chlorpyrifos-methyl, endosulfan, vinclozolin.

EU co-ordinate monitoring exercise (Recommendation 1999/333/EC, point 1)

The number of samples analysed for each compound (active substance/product) ranges between 21 and 94 for melons, between 49 and 217 for peppers, between 32 and 161 for cauliflowers, between 8 and 126 for wheat.

Two samples of peppers presented residues exceeding the MRL (for chlorpyrifos and methamidophos).

2.9. LUXEMBOURG

Pesticide residues monitoring in fruits and vegetables and cereals carried out in Luxembourg in 1999

Introduction

This report summarizes the results of the coordinated and the national pesticide monitoring programme in fruits, vegetables and cereals on the luxembourgish market in 1999. This report has been prepared according to the recommendation of the commission as far as technically possible. It not only contains pesticides mentioned in the dir. 90/642/CEE and 86/362/CEE, but also some other pesticides.

Sampling

The samples were collected according to the annual sampling plan prepared from the National Food Control Administration.

Samples of fresh fruits, vegetables and cereals were generally collected from the central market in Luxembourg by the local police agents. Imported products' sampling was done by the food controller in the wholesalers warehouses.

Sampling is done mostly according to directive 79/700/EEC

The size of a laboratory sample was at least 1 kg.

The sampling plan is based on a rolling plan (the section of commodities is subject to annual variations).

Analysis

The samples were analysed by the division of food control which is a part of the National Health Laboratory.

All the samples were analysed by a gaschromatographic multiresidue method (modified german official method DFG S19).

This method consists of an acetone/cyclohexane/ethylacetate extraction and a clean-up on a biobeads S-X3 gel permeation column. The extract was analysed by GC with selective detectors (ECD, NPD and FPD). When a pesticide residue exceeded the MRL, the identity of the pesticide was confirmed by GC-MS. With this method more than 200 pesticides are covered.

Dithiocarbamates were also determined on each sample by using an CS₂ evolution method. The CS₂ is determined by headspace analysing using GC with flame photometric detector (EC method, document 1729/VI/80 final 2, not published).

Accreditation and quality assurance

The only official laboratory involved in the monitoring pesticide residue programme is actually in a preparative phase for an accreditation according to EN 45001.

The analytical results are governed by a quality assurance system. The laboratory takes into account, as far as it is possible for a laboratory of its size, of the document "Quality control Procedures for Pesticide Residus Analyses- Guidelines for Residus Monitoring in the European Union".

Our laboratory has participated on the Workshop on coordinated Analytical Control in November 1999 in Athens/Greece.

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Reporting levels

The lowest residue levels are the same as the limits of determination (generally in the range of 0,01-0,1 mg/kg). The limits of determination are determined by recovery tests.

Results

A total of 231 samples were analysed out of which 107 fruits, 89 vegetables and 20 cereals. Luxembourgish products were concerned for 18.6 % of the analysed samples and 81.4 % came from EU or third countries.

In 119 samples no pesticide residues were detected. Eleven samples contained a residue level higher than the maximum EU or national level.

Out of a list of 91 different pesticide residues a total of 32 were found at least once during the monitoring programme.

The ten most frequently found pesticide residues, in descending order of number of findings are iprodione, procymidone, endosulfan, folpet, thiabendazol, chlorpyrifos, tolcofosmethyl, bromproylate, vinclozolin, dicofol.

Summary of results are shown in the following table:

	Total nr. of samples analysed	Nr. of samples without residues	Nr. of samples with residues at or below the MRL	Nr. of exceedings of EC-MRLs	Nr. of exceedings of N-MRLs
Co-ordinated programme fruits, vegetables and cereals	65	50	13	2	0
National * programme fruits, vegetables and cereals	231	119	101	10	1

* In the national programme of fruits, vegetables and cereals the co-ordinated programme is included.

2.10. NETHERLANDS

SUMMARY

During 1999 about 1500 samples were analysed, both domestic and non-domestic products in the national and coordinated monitoring program. During the last 10 years the percentage of imported products was increased according to the importance on the market. Imported products in general show higher percentages of MRL-violations. Domestic products show residues above the reporting limit in about 45 % of the samples, whereas non-domestic products contain residues in 70 % of the cases.

In addition to the monitoring program, more than 500 samples of pears were analysed for chlormequat and 160 samples of sweet pepper for methamidophos because of incidents in the market.

In 1999 higher violation rates were observed than in the years before. This is probably not caused by worse agricultural practice, but more by continuous improvement and expansion of the scope of analytical procedures and a different sampling strategy.

1 INTRODUCTION

Pesticide residue control has been a task of the Dutch Inspectorate for Health Protection/Food Inspection Service for many years. Therefore, a suitable infrastructure was present for the EU-monitoring as required by directives 90/642/EEC (products of plant origin), 86/362/EEC (cereals) and Recommendation 1999/333/EU (the harmonised specific program 1999).

During 1999 the Inspectorate for Health Protection was under reorganisation. In the new system samples are analysed by only one centralised laboratory and taken by the 5 new regional inspectorates at auctions, importers, wholesale distribution centres, industries processing agricultural products and customs point of entry. Due to the reorganisation considerably less samples were analysed than in the past. In the first half of the year new staff had to be incorporated into the new unit. Especially from August to the end of the year the activity was quite low because of the move to another building. It is the intention to increase again the number of samples to 2500 in 2001 and 3000 in 2002.

2 SAMPLING

The samples are taken without prior information about the presence of pesticides in the sample. Therefore, they represent the situation in the market for the product at that time. However, sampling is directed relatively more to products that need attention because of the violation rate in previous years.

The sampling procedure, i.e. the number of items taken a lot is regulated by the Dutch Food and Commodity Law. This regulation is the implementation of the EC-directive 79/700/EEC.

3 ANALYSIS AND QUALITY

The general strategy is detecting as many pesticides as possible in one by using Multi-Residue-Methods (MRMs). Our method consists of an acetone extraction and a partition step of the residues into dichloromethane/petroleum ether followed by a chromatographic separation and selective detection of residues. The main detection method is gas chromatography (GC) followed by Ion Trap Detection (ITD). For analytes not detectable sensitively enough by ITD, a few additional methods are used:

- GC with Electron Capture Detection (ECD)
- GC with Nitrogen/Phosphorus (NPD) and Phosphorus/Sulphur detection (FPD)

For pesticides not amenable to GC, High Pressure Liquid Chromatography (HPLC) are used

- with fluorimetric detection for the detection of fungicides
- with post-column derivatisation and fluorimetric detection of N-methylcarbamates

These MRMs detect about 325 analytes of the 450 pesticides that have an MRL. The remaining pesticides must be controlled by Single-Residue-Methods (SRMs), which are not performed in routine but on a survey base.

Table A2 gives information about the scope of the MRMs (6.1). The validity of the analytical results is governed by a quality assurance system complying EN45001. The multiresidue methods are within the scope of the accreditation of the laboratory. The centralised laboratory implemented the EU Guideline on Quality Control (6.1). In order to check system performance and to avoid false negative results, a number of reference pesticides standard mixtures is run in each batch of samples at the lowest calibration level (LCL), which is also the reporting limit.

4 REGISTRATION AND COMPILATION

The 5 inspectorates for health protection have a uniform data-system for the storage of sample data and analytical results. The applied MRMs and SRMs are recorded and the results are stored. Because of the registration of MRMs also the **absence** of a residue can be established.

5 MONITORING RESULTS

During 1999 about 1500 samples were analysed in the national and coordinated monitoring program, both Dutch and non-domestic products. During the last 10 years the percentage of imported products has been increased according to the importance on the market (Figure 1). Imported products in general show higher percentages of MRL-violations (Figure 2). Dutch production shows residues above the reporting limit in about 45 % of the samples, whereas non-domestic products contain residues in 70 % of the cases (Figure 3 and 4).

In addition to the monitoring program more than 500 samples of pears were analysed for chlormequat and 160 samples of sweet pepper for methamidophos because of incidents in the market.

In 1999 higher violation rates were observed than in the years before. This is probably not caused by worse agricultural practice, but more by continuous improvement and expansion of the scope of analytical procedures and a different sampling strategy.

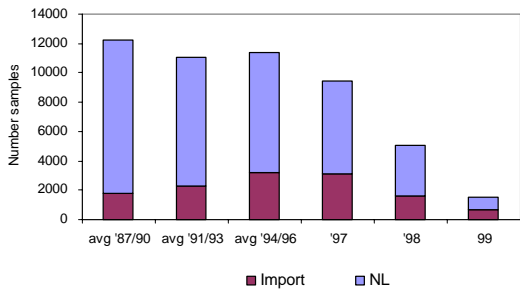


Figure 1. Total number of samples, not including pears and sweet peppers.

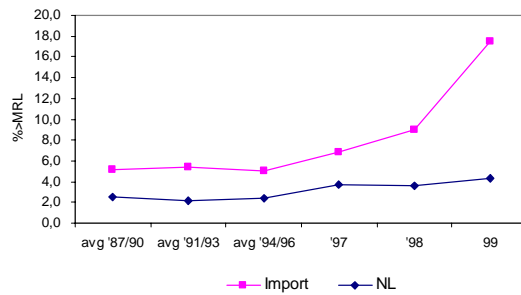


Figure 2. Violation percentage, not including pears and sweet peppers.

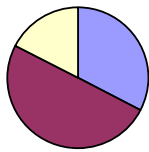


Figure 3. Pesticide occurrence in foreign products.



Figure 4. Pesticide occurrence in Dutch products.

Tables A.2 and A.3 show summarised statistical data on both residues and products. Apart from the incidents with chlormequat on pears and methamidophos on sweet peppers no conspicuous violation rates of pesticides are observed in 1999. The increase of the occurrences of some modern pesticides is obvious (figure 5).

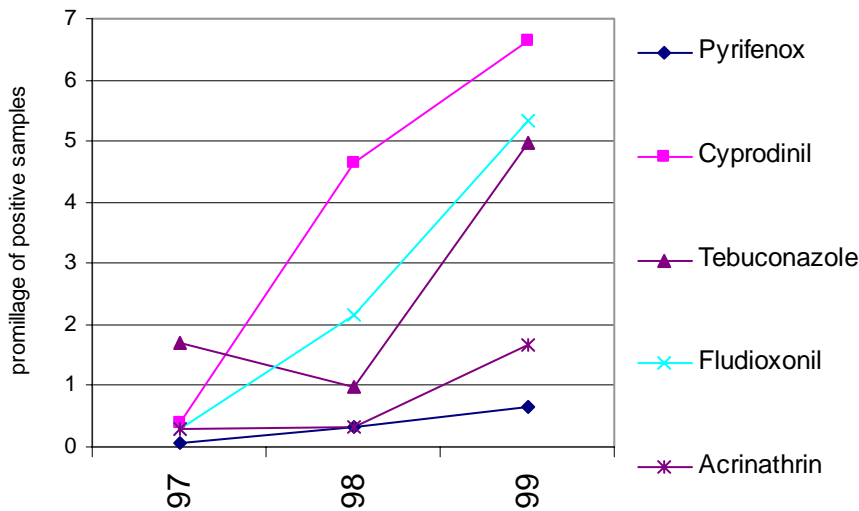


Figure 5. Change in occurrence of modern pesticides.

Also the type of products with MRL violations show variations. In addition to pears and sweet peppers more violations are encountered in:

- melons, beans and grapes

Considerably fewer violations were found in:

- mandarins, carrots and potatoes.

Some pesticide/product combinations contribute considerably to the overall violation rates of the products. Some of these combinations of products and analytes that exceed the MRLs most often are given in Table 1. It is remarkable that all these cases regard violations of zero-tolerances and ,

except for the cucumber, imported products. Probably these violations could have been avoided, if the registrant of the pesticide had applied for an import tolerance in the Netherlands.

Table 1. Combinations of products and analytes that exceed the MRLs most often.

Product	Pesticide	number of samples	% samples >MRL	MRL * zero tolerance
Grape	Pyrimethanil	73	11,0	0,05 *
Melon	Thiabendazole	22	27,3	0,05 *
Grape	Penconazole	73	6,8	0,02 *
Sweet pepper	Profenofos	121	4,1	0,02 *
Cucumber	Thiabendazole	49	10,2	0,05 *
Sweet pepper	Prothiofos	121	3,3	0,02 *
Tangerines	Fenthion	43	9,3	0,05 *

6 LITERATURE

- 6.1 Analytical Methods for Pesticide residues in Foodstuffs, Sixth Edition. Ministry of Public Health, Rijswijk, 1996.
- 6.2 Quality Control Procedures for Pesticide Residue Analysis, EU Document 7826/VI/97

2.11. AUSTRIA

PESTICIDE RESIDUE MONITORING IN AUSTRIA, 1999

A total of 597 samples were analysed under the co-ordinated and national pesticide monitoring programs. Most of the samples were fresh fruits and vegetables. Beside that other products like cereals were analyzed.

The national pesticide monitoring is done according to a nation-wide sampling plan designed by the Institute of Applied Statistics and System Analysis (Joanneum Research, Graz) in co-operation with the Federal Minister of Social Security and Generations. The plan was based on data concerning dietary consumption, production and import of fruits and vegetables and results of former measurements. The analytical and budgetary possibilities were taken into account, too.

The co-ordinated programme of the European Commission was of course also done.

The samples were taken by trained officials from the local Food Inspection Service („Lebensmittelaufsicht“).

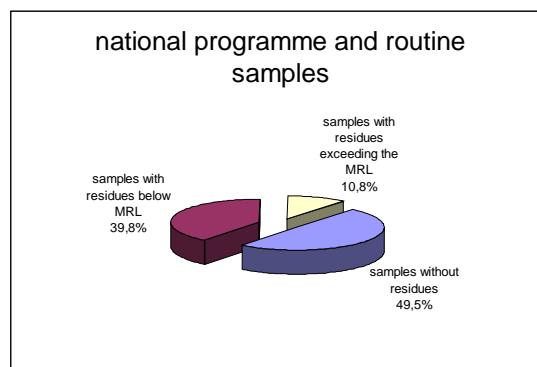
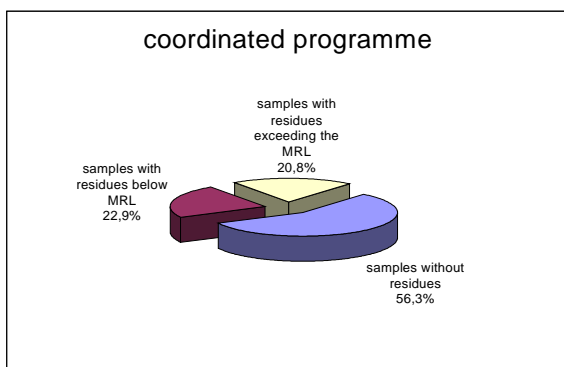
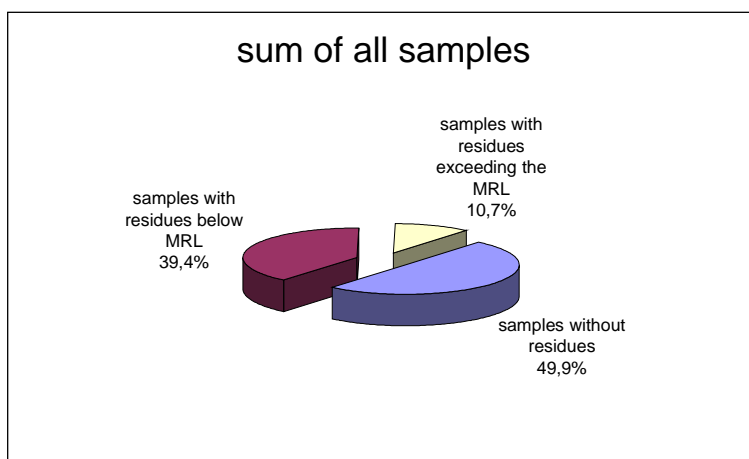
The analysis of the co-ordinated programme, the national monitoring programme and routine samples also were made by two federal laboratories for food control (Bundesanstalt für Lebensmitteluntersuchung und –forschung in Wien und Bundesanstalt für Lebensmitteluntersuchung in Innsbruck). Three federal laboratories for food control (Bundesanstalten für Lebensmitteluntersuchung in Graz, Linz und Salzburg) made the analyses of some samples of the national monitoring programme.

The analytical methods were adopted from published methods of the Dutch federal laboratories („Analytical Methods for Pesticide Residues in Foodstuffs“, 6th Ed., General Inspectorate for Health Protection, Ministry of Public Health, Welfare and Sport, The Netherlands) and validated in the laboratories. The fruits and vegetables were analysed up to a maximum of 92 pesticides. The methods used were a GC multimethod with ECD-, NPD- and FPD-detection. GC/MS-methods are primarily applied for confirmation purposes of the other GC methods, a HPLC-method for benzimidazolfungicides and a spectrophotometric method for the sum of dithiocarbamates.

All laboratories involved in the co-ordinated programme and the national monitoring programme including the routine samples got the accreditation in the year 1998.

Summary of results are shown in the following table and figures:

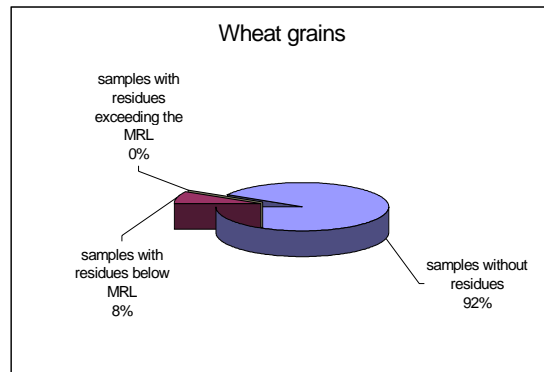
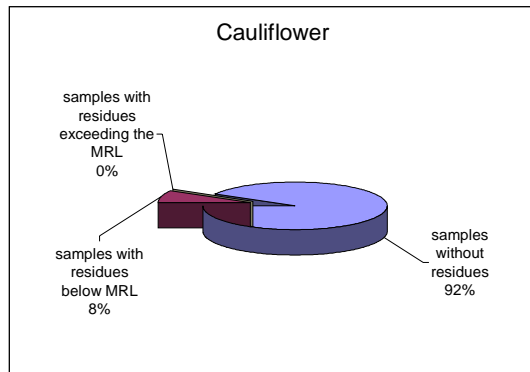
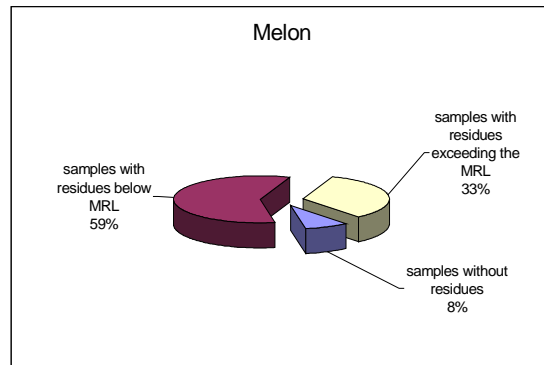
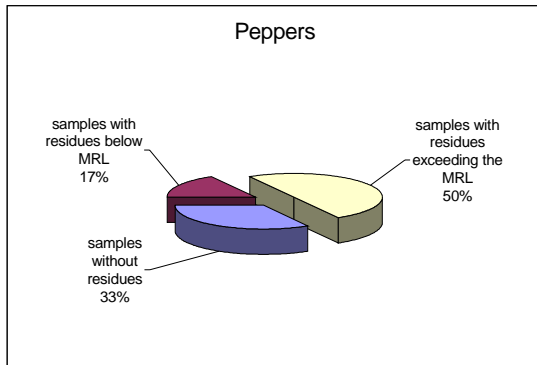
	national programme (including routine samples)				co-ordinated programme	
	fruit and vegetables		cereals			
	number	%	number	%	number	%
samples analysed	546		2		48	
without residues	269	49,3	2	100	27	56,3
with residues below MRL	218	39,9	0	0	11	22,9
with residues above MRL	59	10,8	0	0	10	20,8



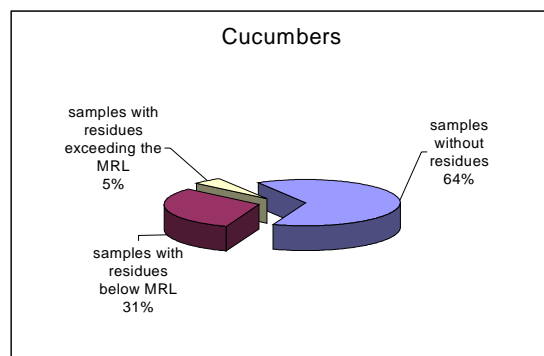
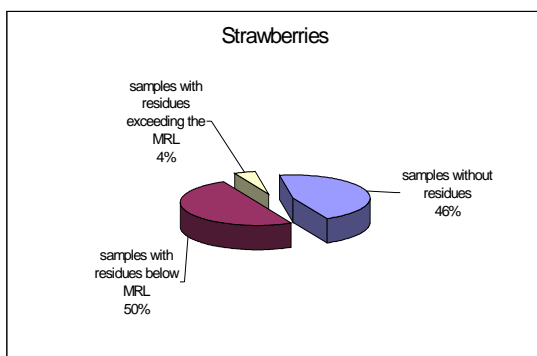
The most frequently found residues were in the co-ordinated and national programme and in the routine samples: Endosulfan, Procymidon, Methamidophos, Iprodion, Fenvalerate, Azinphosmethyl, Dicofol, Maneb-group, Acephate, Lamda-cyhalothrin

The figures below shall demonstrate the individual statistics of the different commodities analysed in the co-ordinated programme and of the five specific commodities analysed in the national monitoring programme:

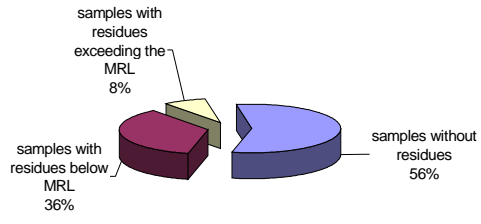
Co-ordinated programme



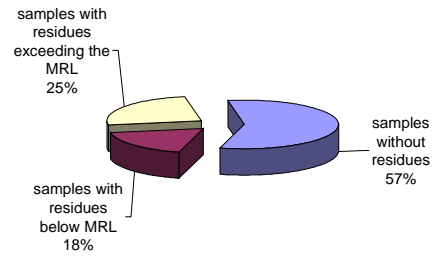
national programme



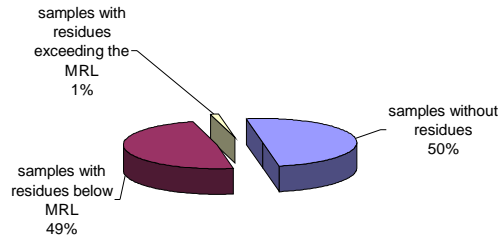
Apricots



Peppers



Peaches



2.12. PORTUGAL

Pesticide residue monitoring in foodstuffs of vegetable origin Portugal – 1999

Summary results

Throughout 1999, a total of 648 samples of fruit, vegetables and cereals and 109 samples of processed samples were analysed. Samples were collected at wholesale outlets, wholesalers warehouses and at farm gate. A small percentage of samples were taken at retail outlets. Most samples were taken by inspectors for food control (DGFCQA and regional bodies). A small number of samples of the fresh commodities and the samples of processed products were taken by DGPC (check sampling). The results reported include the contribution of four laboratories: the central laboratory (DGPC) and three regional laboratories, located in Porto, Algarve and Madeira.

Analyses were conducted using a multi-residue method by GC with selective detectors included in a European Norm, which is in-house validated for 98 active substances and relevant metabolites. The benzimidazole carbamate group was determined by HPLC after ethyl acetate extraction and acid-base partition. Dithiocarbamates were determined using the spectrophotometric method.

The EC recommended procedures on internal analytical quality control were followed as much as possible. Two of the laboratories participated in the EU proficiency test and three of them in FAPAS proficiency tests. However, none of the laboratories is accredited yet.

Residues of the pesticides sought were detected in 34,1% of the total samples of the fresh commodities analysed and infringements to national or EC MRL were found in 4,9% of the sampling. The higher contribution to this percentage of infringements is due to the presence of methamidophos in peppers at levels higher than the national MRL (0,1 mg/kg). The number of infringements would have been slightly higher if directive 98/82/EC had been transposed into the national legislation by 1 August. The unit-to-unit variation exercise conducted for methamidophos in fresh sweet peppers showed variability factors ranging from about 1,8 to 3,2.

For the 109 samples of processed products analysed, residues occurred in 38 of them, although in most cases at very low levels. In two of these samples infringements to the MRL set for the raw commodity were reported.

2.13. FINLAND

PESTICIDE RESIDUE MONITORING OF FRUIT, VEGETABLES AND CEREALS IN FINLAND 1999

SUMMARY

A total of 2460 samples were analyzed under the EC co-ordinated and national pesticide monitoring programs including fresh or frozen fruit and vegetables, cereals, dried fruits, herbs and spices.

The number of import food samples was 1881. The samples were collected from wholesalers' warehouses. Domestic samples - 579 in total - were taken directly from farms and wholesalers or from retail shops.

Pesticide residues above the reporting limits were found in 47.6 % of the samples. The occurrence of residues was more frequent in import food samples (58.2%) than in domestic samples (13.0 %). Residues exceeding the Maximum Residue Limits (MRL) were found in 135 samples (5.5 %). All violations were found from imported foods. Out of the 345 samples analysed under the EC co-ordinated program 54 (15.6 %) exceeded the MRLs.

	Co-ordinated program	All pesticide monitoring (including co-ordinated)
Total number of samples	345	2460
Samples without residues	113	1290
Samples with residues at or below MRLs	179	1035
Samples with residues exceeding MRLs	54 (54 E + 0 N)*	135 (114 E + 21 N)*

* E = EC-MRL, N = National MRL

Total number of pesticides analyzed for was 182 but residues of only 101 of them were found in the samples. The 10 most frequently found residues were endosulfan, thiabendazol, imazalil, chlorpyrifos, malathion, procymidon, iprodion, methidathion, pirimiphos-methyl and bromopropylate.

The samples were analyzed in two laboratories; Finnish Customs Laboratory and Environmental Laboratory of Helsinki. Both laboratories have obtained accreditation for pesticide residue analysis and have participated proficiency tests in 1999.

The samples for variability study of methamidophos residues in pepper were taken from lots of single growers. Twenty units were collected randomly from different boxes of the lot. The sample was mixed and ten units were homogenized and analyzed as a composite sample. The remaining 10 units were analyzed individually. Two different lots were examined and the variability factors were 4.84 and 3.74.

SAMPLING

The samples were collected according to the annual sampling plan prepared by the National Food Administration. Following criteria have been taken in to consideration to determine the number of samples:

- proportion of the commodities in diet
- known residue problems of certain commodities
- sampling should cover domestic production and imports from EU area and third countries
- the programme shall include the samples of the EU co-ordinated program

Most of the import food samples were collected from wholesalers' warehouses in Helsinki area by trained sampling officers of the Customs. Domestic samples were taken directly from farms throughout the country or from wholesalers and retail shops in Helsinki by local health inspectors.

Roughly 10 % of the samples were compliance samples taken as a follow up of violations and the distribution of the lots was prohibited until the analysis was completed.

ANALYSIS

Almost all samples were analysed by the gas chromatographic multiresidue method based on acetone extraction and partition with petroleum ether and dichloromethane. Additional clean up on PSA/SAX cartridges was carried out in case of certain difficult sample matrices. A total of 145 different active ingredients or metabolites and break down products are covered by the method. Additional methods were used for certain commodities to detect N-methylcarbamates, dithiocarbamates, carbendazim, inorganic bromide, phenoxy herbicides, chlormequat, diquat and hydrogen phosphide. Total number of pesticides analyzed for was 182.

The performance of the methods and analytical instruments is checked daily with control samples and/or test solutions. The identification of pesticides by gas chromatography is always based on retention in two different columns. Exceedences of MRLs are confirmed by mass spectrometry as far as possible and by repeating the analysis.

Official control analyses of pesticide residues were carried out by 2 laboratories; Finnish Customs Laboratory and Environmental Laboratory of Helsinki. Both laboratories have achieved accreditation for pesticide residue analysis and have participated international proficiency tests in 1999.

RESULTS

Under the national monitoring program 2460 samples were collected - 1881 import food samples and 579 domestic samples. The results of 47 different commodities including 2097 samples are collected in table C. The remaining 363 samples were miscellaneous commodities or processed products which had only few detected residues.

Pesticide residues above the reporting limits were found in 47.6 % of the samples. The occurrence of residues was more frequent in import food samples (58.2 %) than in domestic samples (13.0 %). Pesticide residues were detected most frequently in citrus fruits, bananas, boysenberries and peppers (table 1.).

Residues exceeding the MRLs (EC + national) were found in 135 samples (5.5 %). The harmonized EC MRLs were exceeded by 114 samples (4.6%). All violations were found from imported foods. An exceedence is reported when the analytical result is higher than the MRL plus the analytical

uncertainty. After an exceedence administrative measures like destruction of the lot and compliance sampling of next lots are taken. In addition to the reported exceedences, in 43 cases the residues were found to be slightly above the MRLs but within the analytical uncertainty margin. Out of the 345 samples analysed under the EC co-ordinated program 54 samples (15.6 %) exceeded the MRLs. In pepper samples methamidophos exceeded the MRL in 54 cases, acephate in two cases and endosulfan in one case. One melon sample had thiabendazol slightly above the MRL. Cauliflower and wheat grains had no residues included in to the co-ordinated program.

More than one quarter (29 %) of all analysed samples contained residues of two or more pesticides in a single sample. The highest number was 7 pesticides in same sample. The occurrence of multiple residues was common on citrus fruits, peppers, apples, table grapes, strawberries and pears (figure 2).

The samples for variability study of methamidophos residues in pepper were taken from lots of single growers. Twenty units were collected randomly from different boxes of the lot. The sample was mixed as whole units and ten peppers were separated randomly, homogenized and analyzed as a composite sample. The remaining 10 units were analyzed individually. Two different lots were examined and the variability factors were 4.84 and 3.74.

2.14. SWEDEN

The Swedish Monitoring of Pesticide Residues in Food of Plant Origin: 1999

Summary of results

Surveillance monitoring

In 1999, a total of 3 438 surveillance samples of fruits, vegetables, juices, fruit drinks, cereal grains, cereal products, vegetable oils, and oil seeds were analysed for pesticide residues. National and EU harmonised Maximum Residue Limits (EC-MRLs) were exceeded by 73 samples (2.1 %). None of the samples of domestically grown foods contained residues above the MRLs. No detectable residues were found in the 47 samples of foods for infants and young children.

Cauliflower, melons, peppers and wheat grains, in all 383 samples, were analysed in the 1999 EU co-ordinated programme. EC-MRLs were exceeded by 36 (9.4%) of these samples.

The highest violation rate was found in a (composite) sample of sweet peppers which contained 1.5 mg/kg of methamidophos. The MRL for methamidophos in peppers is 0.01mg/kg. The variability factor, calculated as the highest value divided by the mean of individual units in the sample, varied between 2.3–6.6. The highest residue in a single unit was 4.2 mg/kg

Compliance sampling

As a routine, exceedances of MRLs in surveillance samples are followed up by compliance sampling. A total of 40 enforcement samples of fruits and vegetables were collected and 16 lots (38 tons) were prohibited from being sold.

Sampling

The number of samples collected of each food was roughly proportional to the food's consumption rate and amounted to at least 100 samples for each of the more important foods. The samples were taken by trained inspectors belonging to the Board of Agriculture according to written instructions from the National Food Administration.

Analysis

In all, by using both multi-residue methods and single residue methods it was possible to determine 202 pesticides corresponding to 243 analytes. A total of about 452 000 residues (analyte/commodity combinations) were sought.

The majority of the pesticide residues were measured and reported from the limit of quantitation (determination), generally in the range of 0.01 - 0.2 mg/kg. For certain pesticides, e.g. cypermethrin, daminozide, imazalil, iprodione, methamidophos, procymidone and thiabendazole the EC-MRLs set at the LODs were not achievable in our routine monitoring.

Quality control

The analyses were carried out at one contracted laboratory which was accredited by the Swedish accreditation authority SWEDAC for all analytical methods used. The laboratory has participated in two proficiency tests in 1999. One of these tests was organised by the European Commission (EU-PT 3).

The quality control included daily checks of the instruments' sensitivity. Most of the GC-determinations were carried out using standards in matrix extracts. Recovery checks were done on a regular basis. The EC guidelines "Quality Control Procedures for Pesticide Residue Analysis" has been implemented as far as practicable.

Chronic intake

The chronic dietary intakes, based on monitoring data, were calculated for 16 pesticides in 19 major foods. Ten of these pesticides exceeded 1 % of ADI. Dimethoate/omethoate encountered for the highest figure, 5 % of ADI, when a conservative approach was applied in the calculations.

Acute intake

The national estimated short-term intake (NESTI) was calculated for residues of methamidophos in those 14 samples of peppers for which the actual variability factor was determined. The estimated short-term intake, based on the highest residue in one of these peppers, exceeded the acute reference dose for both toddlers and adults 360% and 130%, respectively.

2.15. UNITED KINGDOM

THE UK PESTICIDES MONITORING PROGRAMME BRIEF DESCRIPTION OF PROGRAMME, RESULTS AND MONITORING

1. Background

The purpose of the UK monitoring is fourfold: to back up the statutory approvals process for pesticides by checking that no unexpected residues are occurring; to check that residues do not exceed statutory EU and UK maximum residue levels (MRLs); to check that human dietary intakes of residues are at acceptable levels; and to carry out enforcement programmes. This monitoring, together with that carried out by local authorities and the food industry provided a wide-ranging overview of the residues present in food. Only 1.6% of samples analysed contained residues above MRLs, and the majority of these would not lead to an exceedance of the acceptable daily intake (ADI).

The results from the programme of work were submitted to the European Commission in fulfilment of obligations for reporting results of surveillance for pesticide residues established by Directives 86/362/EEC, 86/363/EEC and 90/642/EEC. Results of the co-ordinated recommended monitoring programme carried out across the EU were again included in the report.

2. Selection criteria

Samples for the main commodity rolling programmes have generally been obtained at monthly intervals from two population centres in each of six regions of the UK. The centres selected are changed each year. Generally, the choice of foodstuffs to be analysed in the programmes represents a balance between the levels of consumption of those foodstuffs, information on the possible levels of residues and the need to ensure that as wide a range of commodities as possible is included in the surveillance. Where practicable samples are taken, prepared and analysed according to CAC guidelines (*Recommended Method of Sampling for the Determination of Pesticide Residues (Vol. 2, section 3, Codex Alimentarius, 1993)* and Portion of Commodities to which Codex Maximum Residue Limits Apply and which is Analysed (Vol. 2, Codex Alimentarius, 1993). Samples are generally analysed unwashed and unpeeled, with the exception of root crops which are routinely washed. Only one variety, from a single source, is included in each sample.

In determining its surveillance programme, the Working Party considers data from other sources including publications produced by other UK Ministry of Agriculture departments. The UK also considers data published on monitoring carried out by other governments, as well as data and intelligence from industry and other sources.

3. Enforcement

When the level of pesticide residues found during Working Party surveys indicates use of a non-approved pesticide, or the use of a pesticide other than in accordance with the conditions of its approval, the source of the samples involved is investigated to establish the cause of the residues. Further targeted surveillance is normally carried out to check whether the result represented an isolated incident. For UK-produced commodities, enforcement involves the taking of samples from a grower's premises for analysis. If evidence of misuse is found the grower is prosecuted. In the case of imported produce from third countries, sampling is normally carried out at the point of entry while the consignment is detained. If unacceptable residues are found, the whole consignment is refused entry to the UK, and other EU member states are notified. If problems are identified in retail samples of produce of EU origin, the details are reported to the Member State concerned, to allow the relevant authorities to follow up the case.

4. The 1999 European Union recommended monitoring programme

Surveys were carried out in support of the Commission recommendation for a harmonised, specific, EU programme. Samples of cauliflower, melon, sweet peppers and wheat grain were analysed for a range of pesticides indicated in the recommendation. No residues were detected in the 71 samples of cauliflower analysed. Residues were detected in 30 of the 72 melon samples, and 10 contained multiple residues. MRLs were exceeded in six of the samples. Residues were detected in 8 of 71 samples of sweet peppers analysed, and 5 contained multiple residues. MRLs were exceeded in two of the samples. Residues were detected in 11 of 62 samples of wheat grain analysed, and one contained multiple residues. No MRLs were exceeded.

5. Implications of 1999 results for consumers

1.6% of samples analysed contained residues in excess of an MRL. This confirmed that in the vast majority of cases pesticides were being applied in accordance with their label requirements, and also provided an assurance to consumers that, where they were being exposed to residues, they were at safe levels. In all cases where MRLs were exceeded, or where there are potential intake issues, a separate consumer risk assessment was carried out to establish whether the level of residues present would lead to an exceedance of the ADI or, where appropriate the ARfD, by a high level consumer. The risk assessments indicated that in the great majority of cases there would be no exceedance. Both the ADI and the ARfD incorporate safety margins and the risk assessments assume food intake at the 97.5 percentile level in food surveys. Therefore, even where exceedances are calculated, the risk to consumers is unlikely.

6. Methods of Work and Quality Control

Full details of the Working Party's methods of work are contained in *Surveillance: The Estimation of Dietary Exposure to Pesticides - Report of the Working Party on Pesticide Residues: 1991-1993* which is published as Food Surveillance Paper Number 50 available from the Stationery Office (ISBN 0 11 243015 5).

6.1 Quality Control

Analytical methods were deemed acceptable if the level of recovery of added pesticide was between 60% - 140%, providing that routinely the mean recovery was in the range 70% - 110%. Results which exceeded MRLs, or which were unusual in occurrence, or were otherwise of particular significance were required to be accompanied by acceptable recovery data and, wherever practicable, to have been confirmed using mass spectrometry. The residues data provided in this report have not been corrected for recovery and are expressed, unless otherwise stated, on the basis of the fresh weight of the sample and as defined by the MRL.

6.2 Proficiency Testing Programme

All laboratories carrying out work have taken part in proficiency testing exercises, including the Dutch Chek Monitoring Programme and other international programmes. Recognising the importance of proficiency testing, MAFF started a scheme in 1990 for laboratories known as the Food Analysis Performance Assessment Scheme (FAPAS). All of the laboratories which submit data to the Working Party participate in FAPAS. Since 1997, all laboratories have allowed the FAPAS Secretariat to release their individual results and details of their performance to PSD. This allows an independent check of the laboratories' performance to be made. Additionally, all of the laboratories meet the requirements of a recognised accreditation scheme, such as the United Kingdom Accreditation Service (UKAS) or the requirements of Good Laboratory Practice (GLP).

THE UK PESTICIDES MONITORING PROGRAMME

Country	Total no. of samples analysed *	No. of samples with residues detected <MRL	No. of samples where 1 or more MRL exceeded
UK	1372	475	37

= Extract from UK programme. Results of monitoring:
Fruit and vegetables
Cereal products

2.16. NORWAY

Pesticide Residue Monitoring in Fruit, Vegetables and Cereals in Norway – 1999

A total of 3091 samples of fresh fruit, vegetables including potatoes and cereals were analysed in 1999. More than 100 different commodities both imported and domestically produced were included in the monitoring programme.

Samples were taken at wholesaler's warehouses in different parts of Norway. The number of surveillance samples of each commodity roughly reflects their share of the market, but more samples were taken of commodities suspected to contain residues. The samples were taken by trained inspectors as random samples by the municipal food control authorities. For samples with residues exceeding the MRL compliance samples were collected as a follow-up.

The analyses were done by the Norwegian Crop Research Institute, Pesticide Laboratory, except AMPA and glyphosate (analysed by MILJØ-KEMI, Denmark).

All samples of fruit and vegetables were analysed using a GC multi-residue method, based on extraction with acetone and petroleum ether, covering 126 pesticides, isomers and breakdown products. Six pesticides were analysed by HPLC using the same extraction method or a slightly modified method with addition of sodium hydroxide. HPLC was also used for analysis of N-methylcarbamates. The dithiocarbamates were determined with a spectrophotometric method. The reporting level is the limit of determination for all methods.

The Pesticide Laboratory was accredited on April 1st 1997. The accreditation is based on the European Standard EN 45001 and also includes no. 2 and 7 of the OECD Principles of Good Laboratory Practice, section II. The analyses were carried out in accordance with the Quality Assurance Manual of the Pesticide Laboratory. The laboratory participate regularly in international intercalibrations / proficiency tests as a part of the quality assurance programme. The routine quality assurance includes analysis of quality control (QC) samples, check of the sensitivity of the instruments and calibration with all pesticides in the monitoring programme. Check of the recovery is carried out regularly. When a pesticide residue exceeds the MRL, quantitative determination is carried out on three replicate samples using three-level calibration. Recovery is checked and the identity of the pesticide confirmed by GC/MS.

The monitoring programme covered 156 pesticides including some isomers and breakdown products (Table A 2).

About 42 % of the samples were domestic products (Table A 1). Among the domestic products violations occurred most frequently in celery and parsley root. Among the common imported products mango, papaya, plum and carrot had the highest number of samples exceeding the MRL. Two exceedings were found among the samples of cereal products.

In addition to fresh fruit and vegetables 95 samples of oil seeds, baked potatoes and French-fries were analysed. Only one of these samples contained pesticide residues.

The variability exercise concerning methamidophos in peppers was not carried out in Norway during 1999 (Table F). Owing to low recovery of methamidophos there was only reported one sample of peppers with a detectable residue in 1999. The laboratory has since 1999 implemented a new multi-method with better recovery of methamidophos and other pesticides.

2.17. ICELAND

Monitoring of pesticide residues in fresh fruits and vegetables in Iceland in 1999

An official monitoring programme for pesticide residues in fruits and vegetables was initiated in Iceland in 1991. Samples are taken at wholesaler's warehouses in Reykjavík. Since fruits for commercial purposes are not grown in Iceland, all fruits and a greater part of the vegetables are imported.

Around 300 samples are analysed each year. Approximately 80% are imported commodities and fruits count for 50% of all samples examined. Samples are analysed by a GC/MS multimethod based on ethyl acetate extraction, covering 40 pesticides.

As a main rule the maximum residue limits (MRL's) conform to EU directives, but in few cases, where EU has not set any limits, they are in accordance with either Codex Alimentarius or regulations from other Nordic countries. When a pesticide residue exceeds MRL, a new sample is analysed to confirm the results. No actions are taken unless the value exceeds the MRL plus a standard deviation.

In 1999 a total of 301 samples were examined for pesticides in fruits and vegetables. Imported samples were 241 and domestic samples 60. In 53% of all samples, no pesticide residues which were looked for were detected. 44% of the samples contained pesticide residues and violations of maximum residue limits (MRL's) were found in 3% of all samples. Commodities originated from 21 countries, most came from The Netherlands, United States and Spain. The pesticides most frequently found were thiabendazole, imazalil, orthophenylphenol, chlorpyrifos, diphenylamine, Metidation, Vinklozolin, prokymidon, iprodion, karbaryl and tolylfluanid.

The laboratory of the Environmental and Food Agency has taken part in FAPAS and will continue to do so in the future. The laboratory has not been accredited yet but is planning to initiate the preparation work as soon as possible. It is estimated that it could take at least 2-3 years to receive accreditation.

Notification of the co-ordinated programme to the Efta Surveillance Authority is not in the report, we did not participate in that specific exercise.