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**COMMISSION STAFF WORKING DOCUMENT**

**Monitoring of Pesticide Residues  
in Products of Plant Origin  
in the European Union, Norway, Iceland and Liechtenstein**

**2005**

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## 2. COUNTRY COMMENTS

### BELGIUM

#### Summary of results

In 2005, a total of 1496 samples of fruits, vegetables, cereals and processed products of plant origin (including baby food) were taken on the Belgian market and analysed for the presence of pesticide residues (table 1).

Table 1: Overview of the results of the pesticide residue monitoring programme 2005.

| Samples             | analysed    | without residues | with residues at or below MRL | with residues exceeding MRL | with residues exceeding MRL (%) | Products origin |      |       |      |       |      |
|---------------------|-------------|------------------|-------------------------------|-----------------------------|---------------------------------|-----------------|------|-------|------|-------|------|
|                     |             |                  |                               |                             |                                 | BE              |      | EU    |      | Other |      |
|                     |             |                  |                               |                             |                                 | total           | >MRL | total | >MRL | total | >MRL |
| Fruits & vegetables | 1322        | 583              | 635                           | 104                         | 7,9 %                           | 821             | 60   | 254   | 25   | 247   | 19   |
| Cereals             | 24          | 21               | 4                             | 0                           | 0 %                             |                 |      |       |      |       |      |
| Processed products  | 150         | 143              | 26                            | 0                           | 0 %                             |                 |      |       |      |       |      |
| <b>TOTAL</b>        | <b>1496</b> | <b>747</b>       | <b>685</b>                    | <b>104</b>                  | <b>7%</b>                       |                 |      |       |      |       |      |

**Fruit & vegetables** : 1322 samples of fresh or frozen fruit and vegetables were analysed in 2005. No residues were found in 44% of them. 104 samples exceeded the national or EU harmonized Maximum Residue Levels (MRL). Relatively high percentages of MRL exceeding were found in berries and small fruit (grapes, raspberries, currants), stone fruit (peaches, nectarines), leaf vegetables (lamb's lettuce, fresh herbs), fruiting vegetables (paprika) and stem vegetables (celery). Note that in this report exceedings were counted without taking into account the analytical uncertainty of the result. The percentage of exceedings in 2005 (7,9%) is higher than in 2004 (4,8%) (figure 1). This may be explained by the improvement of the analytical performance of the laboratories (increased number of pesticides searched for and higher sensitivity of detection). More exceeding were observed on imported products (8,8%) than on Belgian products (7,3%). Out of a list of 200 different pesticide residues sought in fruit and vegetables, a total of 72 were found at least once during the monitoring programme. The most frequently found pesticide residues are fungicides : iprodion, maneb-group, propamocarb, carbendazim, tolyfluanid, pirimicarb, thiabendazole and procymidone.

| Fruit                           | % > MRL |
|---------------------------------|---------|
| Berries and small fruit         | 11,3%   |
| Stone fruit                     | 11,1%   |
| Citrus                          | 7,0%    |
| Miscellaneous fruit             | 7,0%    |
| Pome fruit                      | 1,0%    |
| Vegetables                      | % > MRL |
| Leaf vegetables and fresh herbs | 12,7%   |
| Fruiting vegetables             | 10,1%   |
| Stem vegetables                 | 9,5%    |
| Root vegetables                 | 9,2%    |
| Potatoes                        | 3,3%    |
| Legume vegetables               | 3,2%    |

Table 2 : MRLs exceeding by group of products in 2005

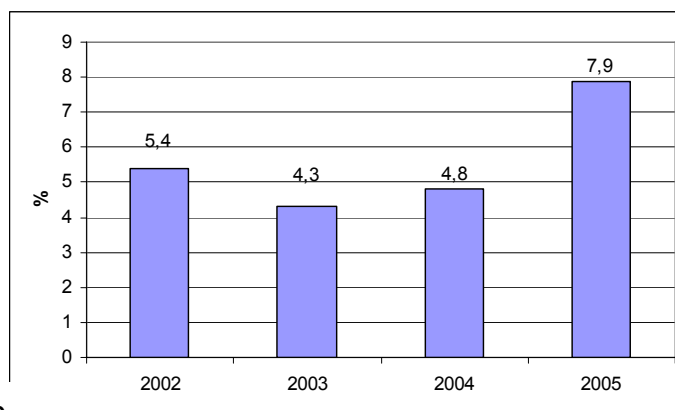


Figure 1: MRLs exceeding (%) in fruit and vegetables in Belgium from 2002 to 2005

**Cereals:** 24 samples of cereals were analysed

in 2005. Out of 31 pesticide residues sought for, dichlorvos, malathion and bromide were detected but none of them exceeded the MRL.

**Processed products:** 150 samples of processed products were analysed. Chlormequat was found in pears syrup and flour; a trace of lindane was found in chocolate. No exceeding of MRL was observed.

### **Organisation of monitoring programmes and sampling**

- **Responsibilities:** The Federal Agency for the Safety of the Food Chain, under the responsibility of the Minister of Public Health, is the competent authority for the monitoring of pesticides residues in foodstuffs (<http://www.afsca.be>)
- **Drafting of the monitoring plan:** Several factors were taken into account: the importance of the foodstuff in diets, the exceeding in previous years, the RASSF messages, the pesticides authorised in Belgium, the analytical and budgetary possibilities and all other useful information. All the groups of fruits and vegetables are included in the programme and a rolling programme is applied for less important commodities. The coordinated programme of the European Commission was included in the national programme.
- **Sampling:** Sampling was done by trained officials according to directive 2002/63/EG, at auctions, importers, wholesalers, processors and exceptionally in retail.
- **Measures taken:** In case of infringement, the responsible company/person is identified. When the exceeding of MRL is in the analytical uncertainty, a warning is issued. When the exceeding is higher, an official report is made and sent to the responsible company/person and also to the legal service. The legal service proposes a fine. If the fine is not paid, or in case of repeated offences, the matter is taken to court. When the dietary intake calculations indicate a risk for the consumer (evaluated following document SANCO/3346/2001) then a national and international rapid alert is issued and measures to protect consumers are taken (tracing and recall of the foodstuffs for destruction). In 2005, two RASFF messages were issued by Belgium: one for carbaryl on apricots imported from Chile and one for mepronil on lambs lettuce grown in Belgium.

### **Quality insurance**

- The three officially recognised laboratories involved in the monitoring programme 2005 are ISO 17025 accredited for the most important analytical methods and commodities, and accreditation was gradually extended. Multi-residues methods and sometimes specific individual methods were performed on samples. All certificates of accreditation can be found on the website of the Belgian Organisation for Accreditation (<http://belac.be/>). The laboratories take into account EU Quality control procedures (ref. SANCO/10476/2003).
- Two laboratories participated in the European Commission's Proficiency Tests 7 with good results.

### **Other information**

- An MRL is not a toxicological endpoint. An exceeding of MRL represents rarely a hazard for the consumer. MRLs exceeding reflects the non respect of the good agricultural practice (product authorised on the crop, dose, delay before harvest, ...)
- The scope of the monitoring programme is to enforce the legislation. It does not provide for a total random analysis, as more samples are taken of food items expected to contain pesticides or to exceed MRLs. For example, grapes were followed closely, due to the number of exceedings observed in the past.

## 1. SUMMARY

In 2005, totally 753 samples of fruits, vegetables, cereals and processed products and 50 baby food products of domestic and non-domestic origin were analysed in the national and co-ordinated monitoring program. National or EU harmonised MRLs were exceeded in 24 samples (3,2 %). Pesticide residues above the reporting limits were found in 37 % of the surveillance products. Regarding products of plant origin, domestic production made up 27,5 % of the samples, 57 % of the samples came from other EU countries and 15,5 % were from the third countries. Beans, carrots, cucumbers, mandarines, oranges, pears, potatoes, rice, spinach – totally 185 samples, were analysed in the of EU co-ordinated programme for the year 2005. The level exceeding MRL was found out in 18 samples of fresh fruits and vegetables, in 2 samples of coffee and baby food products and in 1 sample of wheat flour and rice. Most of violative samples were from EU countries (13), 7 samples were of the Czech origin and 3 samples of third country.

68 different pesticide residues out of 147 sought in fruit and vegetables were found at least once during the monitoring programme. The most frequently found pesticide residues, in decreasing order of frequency (found/sought) are: O-phenylphenol, imazalil, carbendazim, thiabendazole, benomyl group, procymidone, chlorpyrifos, beta enodsulphan, chlorothalonil, cyprodinil. Out of 48 pesticide residues sought for, pirimiphos-methyl, imazalil, procymidone and metalaxyl were detected in cereals.

## 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

### Responsibilities

Czech Agriculture and Food Inspection Authority (CAFIA) is the competent authority for controls of pesticide residues in foodstuffs and provides the national and EU co-ordinated monitoring programmes in co-operation with the Ministry of Agriculture.

### Design of Programmes

The sampling plan for pesticide residues monitoring is always drawn up for one calendar year. The plan is elaborated by the Headquarters of CAFIA as internal provision and it is distributed to the CAFIA regional inspectorates which are responsible for its implementation. The plan is based on Commission Recommendation concerning a coordinated Community monitoring programme which sets the minimum number of samples for the Czech Republic. Within the monitoring of pesticide residues, the Czech Republic analyses approximately 800 samples per year. Within pesticide residue monitoring, especially commodities mainly presented in the consumer basket are collected. In selection of commodities for the monitoring a number of findings in the past (in the Czech Republic and other member states) is also taken into account. Fresh fruit and vegetables and baby food comprise the primary proportion of samples taken.

### Sampling

Sampling is performed in accordance with sampling procedures referred to in Commission Directive No. 2002/63/EC that has been incorporated into the national regulation for sampling for determination of pesticides in and on fruit and vegetables.

Samples are taken by authorised and for this purpose trained employees of the CAFIA, i.e. inspectors of the CAFIA regional inspectorates. The main sampling points are importers, wholesalers and retail shops for domestic and non-domestic products.

## Enforcement

A ban on sale/distribution of inspected foodstuffs is imposed when exceeding the MRL or it is ordered to withdraw unsatisfactory foodstuffs from circulation. Furthermore, the person inspected is ordered to take such measures that minimise the risk of further occurrence of limit-exceeding findings. A fine is imposed on the inspected person within administrative procedure. When determining its amount the importance, manners, duration and consequences of illegal act are taken into consideration.

## 3. QUALITY ASSURANCE

### Accreditation

All analyses were carried out in the laboratory of Czech Agriculture and Food Inspection Authority (CAFIA) in Prague. Laboratory is accredited by Czech Accreditation Institute (CAI) according to the ISO/IEC 17025 standard for all methods used for monitoring and/or enforcement analysis.

### Analytical methods

Pesticide residues reported by the laboratory in the year 2005 were analysed by 5 analytical methods which covered together 154 analytes (incl. metabolites) – 2 multi-residue methods (one MRM is based on ethyl acetate extraction, GPC clean-up and capillary gas chromatography (GC-ECD/NPD/MSD), second MRM is based on acetonitrile extraction followed by LC-MS/MS - liquid chromatography coupled either with ion-trap mass spectrometry or with triple quadrupole) and 3 single-methods (GC-MSD for dithiocarbamates, GC-ECD for inorganic bromine and LC-MS/MS for chlormequate).

### Proficiency testing

During the year 2005 the laboratory took part in 3 proficiency tests focused on pesticide residues – in two FAPAS rounds and also in EU-PT 07.

### EU Quality Control procedures

Most of requirements from the EU quality control guidelines (SANCO/10476/2003) have been fully implemented till now, however some requirements only partly. Improvement of QC procedures has been achieved since the year 2004 and the process will continue in light of new edition of guidelines also in the future.

### Uncertainty

A concentration-dependent uncertainty concept based on the Horwitz equation is applied for uncertainty estimation. For calculation of expanded uncertainty (U), the  $RSD_R$  obtained from the equation is multiplied by a factor of 1.4, which is derived from laboratory experiments, validation and QC data. Expanded uncertainty is taken into account for evaluation of results. Enforcement action is taken only if the result after subtraction of U is still above the MRL.

## 4. OTHER INFORMATION

### Details of homogeneity exercise

Homogeneity experiments were realised on 19 samples – oranges (9 x), pears (6 x), cucumbers (3 x) and potatoes (1 x). The homogeneity factors are showed in the table below:

| Product   | Pesticides      | Homogeneity factor | Product | Pesticides   | Homogeneity factor |
|-----------|-----------------|--------------------|---------|--------------|--------------------|
| Cucumbers | Tetradifon      | 6,9                | Oranges | Chlorpyrifos | 2,1                |
| Cucumbers | Alfa endosulfan | 1,0                |         | Imazalil     | 1,3                |

| Product   | Pesticides            | Homogeneity factor |  | Product | Pesticides     | Homogeneity factor |
|-----------|-----------------------|--------------------|--|---------|----------------|--------------------|
|           | Procymidone*          | 3,5                |  | Oranges | Chlorpyrifos   | 2,1                |
|           | Carbendazim           | 3,2                |  |         | Imazalil       | 1,6                |
|           | Endosulfan sulphate** | -                  |  |         | Tebuconazole** | -                  |
| Cucumbers | Alfa endosulfan       | 1,3                |  | Pears   | Imazalil       | 1,9                |
|           | Beta endosulfan       | 1,3                |  | Pears   | Carbaryl       | 1,2                |
|           | Endosulfan sulphate** | -                  |  |         | Tebuconazole   | 1,5                |
|           | Suma endosulfan       | 1,3                |  | Pears   | Carbendazim    | 1,4                |
| Oranges   | Imazalil              | 1,7                |  |         | Imazalil       | 1,4                |
|           | Tebuconazole          | 2,7                |  |         | Thiabendazole  | 2,0                |
| Oranges   | Imazalil              | 1,6                |  |         | Diphenylamine  | 2,4                |
|           | Thiabendazole         | 3,2                |  | Pears   | Carbendazim    | 1,5                |
| Oranges   | Imazalil              | 1,5                |  | Pears   | Tebuconazole   | 1,5                |
| Oranges   | Dicocfol              | 1,6                |  | Pears   | Tolyfluanid    | 4,4                |
|           | Captan                | 1,3                |  |         | Carbendazim    | 2,7                |
|           | Tetradifon            | 3,7                |  | Potato  | Thiabendazole  | 2,5                |
|           | Methidathion          | 1,8                |  | Oranges | Imazalil       | 2,2                |
|           | Imazalil              | 2,4                |  | Oranges | Imazalil       | 1,5                |
|           | Thiabendazole         | 1,5                |  |         | Tebuconazole   | 1,5                |
| Oranges   | Imazalil              | 1,9                |  |         |                |                    |

\* concentration in composite sample below reporting limit

\*\* no positive findings in single items

### Risk assessment

All non-complying samples relevant for transmission via RASFF (Rapid Alert System for Food and Feed) were ceded to the Ministry of Health of the Czech Republic which is responsible for health risk assessment in the Czech Republic.

One of the unsatisfactory samples was after evaluation sent to the RASFF system on 18.5.2005 and uploaded to CIRCA database by European Commission as Information notification with reference number 2005.BJD.

## DENMARK

### 1. SUMMARY OF RESULTS

In 2005 a total of 1495 samples of fruit, vegetables and cereals (fresh, organic, frozen or processed) were analysed: 425 samples of domestic origin and 1095 samples of foreign origin, including 473 samples from third countries. The samples included 1360 samples of fruit and vegetables and 132 samples of cereals. In addition, 3 samples of other processed foods were analysed.

Frozen food comprised 5% of the fruit and vegetable samples and 3% were organically produced. About 24% of the cereal samples were organically produced.

Residues exceeding the maximum residue levels were found in 40 samples (3%). All samples with exceedances were fruit and vegetables.

Pesticides were found on average in 51% of fruit and vegetables, and in 27% of the cereal samples. The residues found in cereals were mainly the plant growth regulator chlormequat (found in 14% of the samples). Chlormequat and Imazalil were the pesticides most common found in fruit and vegetables (respectively 40% and 11%). Multiple residues were found in 30% of the samples of fruit and vegetables. One chilli sample had the highest number of pesticide residues, 10. Multiple residues were found in cereals in 4% of the samples. In organic food, residues of chlormequat were found in one pear sample from Italy.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

Pesticide monitoring in Denmark were in 2005 the responsibility of the Danish Veterinary and Food Administration under the Ministry of Family and Consumer Affairs. The Danish Institute for Food and Veterinary Research designed the monitoring programmes and consolidated the collected data in cooperation with the Danish Veterinary and Food Administration. The Regional Veterinary and Food Control Authorities were responsible for implementing the sampling plans and for enforcement actions.

Sampling plans were based on the dietary consumption pattern, production and import data, and monitoring results from previous years. All samples included in the centrally coordinated monitoring in 2005 were designed as surveillance sampling.

Samples were taken by authorised personnel from the 10 Danish Regional Veterinary and Food Control Authorities. Directive 2002/63/EC on sampling procedures for control of pesticide residues is implemented in Danish legislation.

Enforcement actions included warnings, fines and orders to improve internal control programmes.

### QUALITY ASSURANCE

The analytical methods are developed and validated by the Danish Institute for Food and Veterinary Research (DFVF). All samples are analysed at the Laboratory at the Regional Veterinary and Food Control in Copenhagen. DFVF performs confirmatory analysis. Both laboratories are accredited for pesticide analysis in compliance with EN45001/ISO17025 by the Danish body of accreditation, DANAK (certificate numbers 315 and 350). The laboratories participated in the relevant FAPAS performance verification scheme and in the EU-proficiency test.

All samples of fruit and vegetables were analysed for at least 190 pesticides, including metabolites. In addition, part of the samples was analysed for dithiocarbamates and imazalil.

From 15<sup>th</sup> of June Imazalil and benomyl group were included in a new multiresidue method on LC-MS/MS. All cereal samples were analysed for 124 pesticides, isomers or metabolites. 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 exceedances for the maneb-group.

"Guidelines concerning Quality Control Procedures for Pesticide Residue Analysis" has been applied for all methods. Mass selective confirmation was performed for part of the GC multimethods and for the LC/MS-MS methods for cereals and fruit and vegetables.

Analytical uncertainty is not applied in monitoring reports, but is always applied in case of enforcement actions.

### 3. OTHER INFORMATION

Residues without maximum residue limits and residues with acute reference doses were individually evaluated by toxicologists at the Danish Institute for Food and Veterinary Research. Residues of pesticides with a low acute reference dose were detected in 3 samples of fruit and vegetables in quantities that could lead to an exceedance of the ARfD but none of the samples were toxicologically unacceptable.

## GERMANY

### 1. Summary of the Results from the Federal Republic of Germany

The report contains the results of the tests carried out in fresh and frozen fruit, vegetables and cereals during the 2005 survey period. It includes all data transmitted by the laboratories of the official food control of the 16 Laender to the 'Federal Office for Consumer Protection and Food Safety, BVL'.

In 2005 in the Federal Republic of Germany a total of 14244 samples (13744 surveillance and 500 follow-up enforcement samples) were tested for pesticide residues. In 5130 (37.3 %) surveillance samples no residues of pesticides could be quantified and in 7727 (56.2%) surveillance samples, residues of pesticides were quantified in compliance with official, national and EU MRLs. 887 (6.5 %) surveillance samples contained residues of pesticides exceeding MRLs.

Table 1 shows the number of surveillance samples divided into domestic production and imports (including samples with origin not known) and takes into account the three product groups. The overview given in this table indicates that residues of imported samples and samples with unknown origin exceeded MRLs more frequently than those of domestic foods.

Non-harmonised MRLs are one reason for this result. In some countries of origin the MRLs for particular pesticides are higher (less strict) than they are in Germany. Therefore residues may be treated as MRL exceeding in Germany, while they are permitted in the country of origin.

Table 1 Overview of the results of the tests performed on domestic and foreign produce (surveillance sampling only)

| Food item           | Domestic samples |                     |                       |                          | Import samples<br>and samples with origin not known |                     |                       |                          |
|---------------------|------------------|---------------------|-----------------------|--------------------------|---|---------------------|-----------------------|--------------------------|
|                     | total<br>amount  | without<br>residues | residues<br>up to MRL | residues<br>above<br>MRL | total<br>amount                                     | without<br>residues | residues<br>up to MRL | residues<br>above<br>MRL |
| Cereals             | 253              | 151                 | 100                   | 2                        | 170   | 105                 | 60                    | 5                        |
| Fruit/<br>vegetable | 4744             | 2130                | 2432                  | 182                      | 8295  | 2514                | 5083                  | 698                      |
| Baby food           | 273              | 223                 | 50                    | 0                        | 9   | 7                   | 2                     | 0                        |
| <b>Total</b>        | <b>5270</b>      | <b>2504</b>         | <b>2582</b>           | <b>184</b>               | <b>8474</b>   | <b>2626</b>         | <b>5145</b>           | <b>703</b>               |

The food samples were analysed for a total of 636 different pesticides from which 301 were detected at least in one sample. Residues of 154 individual pesticides exceeded MRLs.

### 2. Organisation of Monitoring Programmes and Sampling

In Germany there is a difference between a Food Monitoring Programme and official food control. Parts of the data generated in both programmes correspond with the provisions of Directives 76/895/EEC, 86/362/EEC, 90/642/EEC and the Commission Recommendation of 1 March 2005 (2005/178/EC) concerning a coordinated Community monitoring programme. Therefore, the report includes the respective data from both programmes:

- Monitoring programme 1783 samples

- official food control 12461 samples.

The monitoring is based on a representative collection of data. With regard to bio-statistical aspects, the samples are taken randomly according to defined sampling plans. The sampling plans to be used for this purpose are laid down every year by the Federal Government together with the Federal Laender.

In the framework of official residue control, the samples are taken in a risk-oriented manner in order to check if there are any infringements against food legislation (e.g. surpassing of MRLs). The kind and extend of the sampling are decided on by the Federal Laender. When infringements are stated, the Laender authorities apply adequate measures (follow-up examinations, fines and, if necessary, transfer of the case to public prosecution).

796 samples of 13744 (5.8 %) were from products produced under the rules of organic farming. In only 204 (25.6%) samples residues of pesticides could be quantified. The sampling strategies for these products vary between the Laender. Some have special programs, other take samples randomly

The Federal Laender are responsible for the sampling, which is carried out by correspondingly trained official inspectors according to Commission Directive 2002/63/EC. Samples were taken on the level of producers, manufacturers, wholesalers and retailers.

### **3. Quality Assurance**

All the 33 laboratories transmitting data to the 'Federal Office for Consumer Protection and Food Safety, BVL' have been accredited and participated in proficiency tests.

Analytical methods used in the framework of food surveillance and of food monitoring comply with the requirements set out in the 'Guidance document on residue analytical methods' (SANCO/825/00), including the criterion that the average recovery rates must be in the range of 70-110% and the relative standard deviation  $\leq 20\%$ . If these requirements are met, measuring results are directly included in the assessment, without consideration of actual recovery rates and deviations. If the requirement is not fulfilled, specific rules may be agreed in justified cases.

The analytical uncertainty is not considered in this report. The numerical measured values of residues are compared to the MRLs, only.

However, food control authorities in Germany take into account the analytical uncertainty before administrative consequences follow. In these cases they use the subsequent procedure.

If measured residues indicate that maximum residue levels are exceeded, the analytical uncertainty is considered. For food of plant origin an over-all reduction of 60% of the measured value, over all ranges of concentration, is generally applied. For food of animal origin the result is reduced by twice the theoretical standard deviation as derived from the Horwitz curve for the concentration range in question.

It is taken for sure that maximum residue levels are exceeded when measured values, reduced by the respective deviation, are still above the respective limit value.

## 1. Summary of results

In 2005, a total of 448 surveillance samples of fruits, vegetables, cereals, processed products (cereal products) and baby food were analysed for residues of 204 analytes. National or EU harmonised Maximum Residue Limits (EC-MRLs) were exceeded by 14 samples (3,3 %).

Pears, beans, potatoes, carrots, oranges, mandarins, spinach, rice and cucumber, in all 129 samples, were analysed in the 2005 EU co-ordinated programme.

2 of these samples exceeded the EC-MRLs for the pesticides that were included in the co-ordinated programme.

A total of 14 samples of cereals were analysed. The residue was found only in the 1 sample of the cereals.

A total of 16 samples of processed products (cereal products) were analysed and no residues were found.

A total of 29 samples of the baby food were analysed and no residues were found.

The most frequently found pesticide residues, in decreasing order of frequency (found/sought) are: chlorpyrifos, maneb group, imazalil, procymidone, thiabendazole, benomyl group, tolylfluanid, 2-phenylphenol, iprodione, captan.

## 2. Organisation of monitoring programmes and sampling

### COMPETENT AUTHORITIES

In 2005, the Veterinary and Food Board (VFB) of the the Ministry of Agriculture and the Health Protection Inspectorate (HPI) of the Ministry of Social Affairs were the Competent Authority for the control on plant protection products residues in foodstuffs of plant origin, including baby-food and processed products.

### DRAFTING OF THE MONITORING PLAN

The national monitoring plan is drawn up by the Agricultural Research Centre (ARC) in consultation with the VFB, HPI and Plant Production Inspectorate (PPI) according Commission Directives, including the co-ordinated monitoring programme of the European Commission.

The monitoring plan specify the number and type of sample to be taken, the region and the sampling period.

The plan is based on the results of the previous year sampling activities, the results of the annual monitoring for the plant protection products residues in fruit and vegetables, the main food groups consumed in Estonia and on the Rapid Alert Systems in place.

### SAMPLING: PERSONNEL, PROCEDURES, SAMPLING POINTS

Sampling was done by trained officials inspectors according to Directive 2002/63/EC.

- HPI employees(inspectors) in their two laboratories buy samples at retail shops as planned by the ARC. The cost of the samples covered by the Ministry of Agriculture.

- VFB inspectors of the county veterinary centres carry out sampling for residues of foodstuffs of plant origin in the context of food control activity according to the provisions of the law and by the monitoring plan. Samples are taken from domestic and non-domestic commodities of plant origin at wholesale level.

-The number of samples from the organic sector are taken by the inspectors of the county centres of the PPI.

## ENFORCEMENT ACTION

The laboratories do not compare the results of analysis with the MRL, only submit the laboratory certificate to the inspector in charge. The evaluation of the analysis results is the responsibility of the inspector. Where MRLs are exceeded, enforcement action may be taken by the inspector of HPI and VFB – the marketing of the product is prohibited, retailers and consumers are informed and procedures are put in place for product recall.

### 3. Quality assurance

#### STATUS OF ACCREDITATION OF LABORATORIES; NUMBER OF LABORATORIES

Two laboratories of the HPI (Tallinn and Tartu) and one laboratory of the ARC (Laboratory for Residues and Contaminants in Saku) participated in the monitoring programme (Table G) and they are accredited by the Estonian Accreditation Centre (EAK) for all analytical methods used for official control of pesticide residues in food of plant origin. All certificates of the accreditation can be found on the website of the Estonian Accreditation Centre (<http://www.eak.ee>)

#### PARTICIPATION IN PROFICIENCY TESTS

Two laboratories have participated in the European Commission's Proficiency Test 7 with good results and all three laboratories have participated in the proficiency tests organised by FAPAS (UK) (see Table G).

#### IMPLEMENTATION OF EU QUALITY CONTROL PROCEDURES

The EC guidelines SANCO/10476/2003 “Quality Control Procedures for Pesticide Residue Analysis”, third edition, 2003 have been implemented as far as practicable (see Table G).

### 4. Other information

#### BACKGROUND ON LEGISLATION

Estonia has implemented all EC-MRLs. For some other pesticide/commodity combinations national limits are in force (mostly for cereals).

- **Rapid Alert System**

The Trade, Import and Export Department within the VFB is the national contact point (NCP) for food and feed for the Community RASFF system. This department acts as the coordination point for the official control services of the VFB, HPI, PPI Tax and Customs Board.

## GREECE

### 1. SUMMARY OF RESULTS

A total number of 2490 samples were analysed by 8 laboratories, including 1831 samples of fresh and frozen fruit and vegetables, 184 samples of cereals, 463 samples of processed products and 12 samples of baby food. From the above 2490 samples analyzed, 222 samples were also examined for pesticides specified in the 2005/178/EC Commission Recommendation.

➤ **fresh and frozen fruit and vegetables:** 1831 samples

1550 were domestic samples (84.7% of the total number of samples), while 281 samples (15.4%) were imported ones:

- 1499 samples (81.9%) contained not detectable residues of the pesticides sought.
- 312 samples (17.0 %) contained detectable residues at or below MRLs.
- In 20 samples (1.1%) contained residues of one or more pesticides at concentrations exceeding the EU-MRLs were detected.

The total number of pesticides sought in fresh fruits and vegetables was 124. The 10 most frequently pesticides found in fresh fruits and vegetables were: maneb group (20.1%), chlorpyrifos (4.6%), endosulfan (3.2%), captan (2.7%), diphenylamine (2.7%), phosalone (2.4%), procymidone (2.1%), methomyl (1.9%), cypermethrin (1.6%), iprodione (1.6%) and ethoprophos (1.5%). From the 1550 samples analysed in 2005, the 31 samples were from organic production but only in one of the 31 samples detectable residues at or below MRLs of the pesticides sought were found.

➤ **Cereals:** 184 samples

All 184 samples (100%) were imported ones:

- 180 samples (97.8%) contained not detectable residues of the pesticides sought.
- 3 samples (1.6 %) contained detectable residues at or below MRLs.
- 1 sample (0.5%) contained residues of one or more pesticides at concentrations exceeding the EU-MRLs.

The total number of pesticides sought in cereals was 49. The 3 most frequently pesticides found in cereals were: malathion (4.8%), pyrimiphos methyl (3.2%) and chlorpyrifos (1.6%).

➤ **Processed products:** 463 samples

All 463 samples were domestic ones (olive oil):

- 289 samples (62.4 %) contained not detectable residues of the pesticides sought.
- 166 samples (35.9%) contained detectable residues at or below the MRL.
- In 8 samples (1.7 %) residues of one or more pesticides at concentrations exceeding the EU or national MRLs were detected.

➤ **Baby Food:** 12 samples

- All 12 samples (100 %) contained not detectable residues of the pesticides sought.

The reporting levels are generally those routinely achieved in the laboratories.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING (max. 0.5 pages)

- Responsibilities

The annual monitoring plan (including national and EU co-ordinated monitoring programme) was organised by the **central** competent authority. The responsibilities of each of the 8 laboratories involved regarding the number of samples of each commodity that should be analysed by each laboratory and the areas of sampling were well defined by this program. The responsible for the EU co-ordinated program laboratories as well as the responsible for the homogeneity exercise laboratory were also clearly stated.

- *Design of Programmes (priorities, targeting)*

The annual national monitoring plan takes into account the most important parameters such as: the number of samples for each commodity (depending on the produce, the cultivation area and the daily dietary intake contribution of each commodity), the sampling location and the personnel and analytical capacity of each laboratory.

- *Sampling: personnel, procedures, sampling points*

The responsible for sampling authorities, with the designated personnel, follow the methods of sampling (Commission Directive 2002/63/EC) as close as possible. Samples were taken from points of entry, wholesalers, retailers and farm gates.

- *Enforcement action*

In the case of MRL infringement, the relevant to the case enforcement actions specified by our National law are taken.

### 3. QUALITY ASSURANCE

- Status of accreditation of laboratories; number of laboratories

*From the 8 laboratories involved in the pesticide monitoring program 2005, the one has been accredited within 2002, whereas, the procedures for the accreditation of the rest 7 laboratories are in a much progressed stage, but still not finalised.*

- Analytical methods used

- The Dutch Manual (5th edition 1988) for gas chromatographic analysis with NPD, ECD, TSD and PFPD

- *Multiresidue method 5 for organophosphorus compounds, Ministry of Welfare, Netherlands, FIFTH EDITION*

- The proposed EU method for dithiocarbamates

- A French UV method for benzimidazoles

- Multiresidue analysis for N-methyl-carbamates to determine the following pesticides: Aldicarb sulfoxide (Standak), Aldicarb sulfone, Oxamyl (Vydate), Methomyl (Lannate), Aldicarb (Temik), Propoxur (Baygon), Carbofuran (Furadan), Carbaryl (Serin), Methiocarb (Mesurol). EPA Methods 5 and 531.1 and AOAC international protocol 29A05, described a direct inject method which employs gradient liquid chromatography with fluorescent detection, accomplished by post-column hydrolysis and derivatization of the eluted carbamates.

- For olive oil, a method developed in the one lab and published in Journal of Chromatography

- Participation in proficiency tests

From the 8 laboratories involved in the pesticide monitoring programme 2005, four (4) have participated in one proficiency test (PT 7) organised in 2005 by EU.

- Implementation of EU quality control procedures

The EC guidelines SANCO/10476/2003 “Quality Control Procedures for Pesticide Residue Analysis”, third edition, 2003 are followed as close as possible.

- Analytical uncertainty

The pesticide residues figures found are compared with the MRLs. However, in a case of an exceedance of the MRL, before any enforcement action is taken, the analytical uncertainty (95 % confidence interval) is subtracted from the measured value. If this figure still exceeds the MRL, enforcement action relevant to the case is taken.

### 4. OTHER INFORMATION

➤ Details of homogeneity exercise

The homogeneity exercise was performed for the combinations as recommended by 2005/178/EC Commission Recommendation.

## SPAIN

The Spanish Pesticide Monitoring Programme for pesticide residues in food was carried out by the Autonomous Communities. The sampling in origin is coordinated by the Spanish Ministry of Agriculture, Fishery and Food (MAFF). The results of consumption's sampling, including baby food sampling, were gathered by the Spanish Food Safety Agency (SFSA), which is accountable to the Ministry of Health. The SFSA is the contact point between Spain and the European Commission.

The objectives of the programme were to assume that:

- The pesticides were used accordingly with the Good Agriculture Practice.
- The health of consumers was well protected.

The samples were collected by the inspectors following the requirements of the directive 2002/63/CE (Real Decreto 290/2003) at productions, wholesalers and retail level. Most of the samples were taken from domestic production because more than 95% of the Spanish food is produced in the own country. After being taken, samples are sent to the laboratories.

### Summary of the results of the Spanish monitoring 2005 :

The total number of samples in the Co-ordinate Programme and the National Spanish Programme 2005 was 4663; 4160 (89,2%) samples were taken from fruits and vegetables, 345 (13,5%) from cereals, 156 (3,3%) from baby food and 2 samples from other processed products.

For fruits, vegetables and other vegetables the number of samples that exceeded the MLRs was 157 (about 3,8 %), 3 samples (0,9 %) for cereals and no samples for baby food were above the MRL.

Pesticides most frequently found were (refer to the percentage of the total number of samples analyzed for each pesticide):

- **For fruits and vegetables:** Ditiocarbamatos, endosulfan (+), chlorpiryfos, imazalil, procymidone imidachloprid, malathion
- **For cereals:** Malathion, deltamethrin, pirimiphos-methyl, maneb group, triziclazol, flusilazol, chlorpiryfos-methyl
- **For baby food:** Only imazalil (in two samples) and diazinon (1 sample) were found with maximum levels found of 0,004 and 0,008 respectively

| Products   | Sample Analysed | Sample Without detectable residues | Sample with residues at or below LMR | Samples With residues >LMR | Samples With residues >LMR E | Samples With residues >LMR N |
|--|-----------------|------------------------------------|--------------------------------------|----------------------------|------------------------------|------------------------------|
| <b>Fruits and vegetables</b>                     | 4160            | 2542                               | 1461                                 | 157                        | 130                          | 27                           |
| <b>Cereals</b>                                   | 345             | 312                                | 30                                   | 3                          | 3                            | 0                            |
| <b>Processed products (other than baby food)</b> | 2               | 2                                  | 0                                    | 0                          | 0                            | 0                            |
| <b>Baby food</b>                                 | 156             | 153                                | 3                                    | 0                          | 0                            | 0                            |
| <b>TOTAL</b>                                     | <b>4663</b>     | <b>3009</b>                        | <b>1494</b>                          | <b>160</b>                 | <b>133</b>                   | <b>27</b>                    |

## **Organisation of monitoring programmes in Spain:**

There are two subprograms (“in origin” and “in market”) mainly distinguished for the place where samples are taken. The responsible of the coordination for the “in origin” subprogram is the Subdirección General de Medios de Producción Agrícolas from the MAFF. The responsible of the coordination for the “in market” subprogram is the SFSA.

The programme takes into account:

- Proportion of the crops accordingly with the production
- Requirements from EU co-ordinated programme
- Some special actions concerning problems with certain crop
- Products more consumed
- Alerts regarding exceeding of MRLs
- Results of previous years

The responsible personal of the sampling are the inspectors from the Autonomous Communities. Inspectors from agricultural departments take samples following the Manual Proceeding made at national level and co-ordinated by S. G. Medios de Produccion Agrícolas.

Samples are taken from wholesales, in farm gate and retail. Most of the samples are from national production taking into account than more of the 95 % of the national production is for our consumption.

## **Quality assurance**

29 laboratories carried out the analyses of the monitoring programme. Until now most of their reports are not yet accordingly with the European format that has to be made at Central Services.

11 laboratories took part in Proficiency EUPT-7 (2005) from European Commission and all laboratories are participating in the Spanish proficiency Test (Test-Qual) and five laboratories have made several exercises of intercalibration organized by FAPAS.

Nine laboratories are accredited and some others are in very advance phase to obtain the accreditation. The accreditation can be occurred only if methods are validated by collaborative studies. The validation "in house" requires a lot of investigation. For the new molecules the accreditation seems difficult to obtain. The nine laboratories accredited assumed approximately the 52% of the samples. It is important to emphasize that requirements for accreditation of ENAC (organism responsible of accreditation in Spain) are very hard and it is only awarded for each pesticide and food item.

For all the samples is applied the method of multirresidues and maneb-group, and the method of benzimidazol and methyl carbamate is applied to the samples that had registration or some problems were found.

For multirresidues methods the samples are extracted by a solvent (acetone or ethyl acetate) then partitioned by liquid-liquid. The purification for organic-halogenated and organic phosphate molecules were performed by cartridge set-pack, florisil or GPC. ECD, NPD, FPD conduct the determination or hall detector GC/MS is used, as confirmation for samples above MRL's when the techniques are applicable.

Additional methods are used for determination of methyl carbamate, maneb groups, inorganic bromides, benomyl group and so on.

## FRANCE

### 1. SUMMARY OF RESULTS

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The 2005 programme resulted in the analysing of 4 479 samples of fruits, vegetables, cereals and processed products marketed in France, including 1 018 as part of targeted surveillance. The breakdown of samples according to origin was as follows: 72.2% were of French origin, 17% were of European Union origin and 10.8% were imported from third countries. The analyses detected and analysed 237 active substances. The most often found molecules were insecticides and fungicides.

Concerning only the results of the fruits and vegetables monitoring plan (3 098 samples), 55.4% of the samples did not contain residues. Contamination levels under the maximum residue limit (MRL) were detected for 38% of the samples. 93.4% of the fruits and vegetables analysed therefore complied with regulations. MRLs were exceeded in 6.7% of cases.

- Among vegetables, 63.9% did not contain residues and globally 7% were non-conforming. There was more than 6% of non-conformity for lettuces (10.2% : 20 exceedances out of 196 samples), and peppers (23.7% : 23 exceedances out of 99 samples). For carrots, MRLs were exceeded in 4.3% of cases (7 exceedances out of 164 samples).
- As for fruits, 38% did not contain residues and globally 6% were non-conforming. Exceedances mainly concerned strawberries (16.1% : 18 exceedances out of 112 samples), peaches (19% : 12 exceedances out of 63 samples), lemons (6.1% : 5 exceedances out of 82 samples). In contrast, pears, grapefruits, apples, kiwis and grapes had few MRL exceedances—respectively 2.4% (1 exceedance out of 42 samples), 3% (7 exceedances out of 231 samples), 3.1% (1 exceedance out of 32 samples) and 3.7% (2 exceedances out of 54 samples).

Cereals and cereal products had few MRL exceedances : 1.7% of non-conformity out of 271 samples. Tea and herbal infusions had 10.3% MRL exceedances out of 97 samples. 2% of processed products were non conforming (out of 101 samples). Among organic products, 2.8 were non-conforming (out of 176 samples).

Baby food did not contain residues in a quantity exceeding regulations in force (100% compliance).

21% of samples of fruits, vegetables, cereals and processed products analysed as part of the 2005 programme (follow up enforcement and surveillance samples) contained more than 2 residues at contents often under MRLs. This ‘multiple residues’ phenomenon concerned above all lettuces, citrus fruits, apples, strawberries, tomatoes, carrots, grapes and pears.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

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The monitoring programme for plant pesticide residues was carried out by the Direction Générale de la Concurrence, de la Consommation et de la Répression des Fraudes (DGCCRF—General Directorate for Competition Policy, Consumer Affairs and Fraud Control). The aims of this multi-year Community programme are to:

1. monitor compliance with MRLs, and therefore compliance with reported good agricultural practices,
2. collect data to assess actual dietary exposure of consumers to pesticide residues.

This programme takes into account :

- ❑ the results of the 2004 monitoring programme,
- ❑ the requirements of the European Union coordinated programme,

- ❑ the dietary proportion of plant products,
- ❑ the specific and sometimes targeted inspections of certain fruits and vegetables (specific exercise).

Each department received a crop sampling plan. Inspections were made at every marketing level, with adjustments. The inspection of cereals grains generally took place at the storage stage, at silos, or at the processing stage, at plants. Cereal products, fruits and vegetables were sampled at retailers or wholesalers. Samplings at growers were less frequent. For all products entering the French territory, specific action was deployed at points of arrival. A priori, domestic and imported production was not broken down, although a 70/30 ratio has prevailed for several years.

Inspectors are trained and carried out crop sampling by following the updated monitoring programme. Quantities to be sampled and procedures for are in accordance with Directive 2002/63 provisions.

In 2005, the French targeted programme focused on lettuces, grapes, strawberries, exotic fruits, cucumbers, spinaches, peppers, turnips, apples, witloofs, celeries, tomatoes, potatoes and carrots.

### 3. QUALITY ASSURANCE

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In 2005, seven DGCCRF laboratories took part in the monitoring programme and targeted surveillance programme. Five of them are COFRAC accredited and for the remaining two, the step for accreditation is on hand. All laboratories apply the “guidance concerning quality control procedures for pesticide residue analysis” and participate in proficiency tests organised by the European Union (EUPT) and by independent suppliers (BIPEA, FAPAS, CHEK). The Rennes laboratory drew up the summary of the results.

The laboratories used multi-residue techniques and sometimes specific adapted methods. The techniques for multi-residue analyses used solvents for extraction (acetone or ethyl acetate), then liquid-liquid partition. Relevant extracts were purified on adsorbents (Florisil or Bio-beads) or by gel permeation. Determination and quantification were performed by GLC or HPLC with appropriate detectors such as ECD, FPD, NPD, ELCD, UV and fluorimetry, or, more and more, as mass-spectrometry. GC/MS and HPLC/MS were used for confirmation most of the time. Specific methods were used for the determination of dithiocarbamates, bromides, maleic hydrazide, chlormequat and benzimidazoles and some other active substances.

Uncertainties of measurements are taken into account for the interpretation of the results on the analytical reports and the action to be taken : if the difference between the value measured in excess and the MLR remains lower than the uncertainty of measurement, the report of the laboratory indicates a conclusion “to follow” in order to warn the operator. When the product is still available on the market (or at following harvest if control takes place at the production step) a second sample is then carried out for control. Nevertheless, uncertainties are not integrated into the present statements of results to the attention of the European Union, because Excel table does not allow this nuance.

## IRELAND

### 1. SUMMARY OF RESULTS

- Fruit and Vegetables

821 routine samples of fruit and vegetables including organic and processed products were analysed for pesticide residue content in 2005. Of the samples taken 23 % were of domestic origin, 42% were imports from other EU countries, 32% were imports from countries outside of the EU while a further 4% were of unknown origin. Samples were analysed for residues of up to 148 pesticides and metabolites. (an increase of 30 from last year). 44% of samples analysed contained no detectable pesticide residues. Of the remaining 56% which contained one or more detectable residues 5.1% contained residues in excess of the MRLs.

Of the positive samples iprodione (11.3%), thiabendazole (10.4%), benomyl group (carbendazim) (7.4%), diphenylamine (7.1%), chlorpyrifos (5.5%), captan (5.4%), malathion (3.9%), cyprodinil (3.1%), procymidone (2.9%), tolyfluanid (2.9%), cypermethrin (2.7%), methidathion (2.3%), prochloraz (2.2%), chlorothalonil (2.2%), folpet (2.2%), fludioxinil (2.2%), chlorfenvinphos (2.1%), and, azoxystrobin (2.0%), carbaryl (2.0%), tebuconazole (1.7%), were the most commonly detected pesticides in the routine monitoring programme.

- Cereal

67 routine cereal samples of both domestic and imported origin were analysed for residues of 148 pesticides and metabolites. Nine of the samples analysed contained pesticide residues and there were no MRL exceedances. The most common pesticide found was chlorpyrifos methyl (4.5%)

- 50 baby food samples were analysed by the Food Safety Authority of Ireland.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

- The 2005 Irish monitoring programme was carried out by the Pesticide Control Service of the Department of Agriculture and Food. This programme was agreed at the end of 2004 following discussions between the Pesticide Control Service and the Food Safety Authority of Ireland (FSAI).
- The programme was designed by taking account of the following
  - the current consumption data for Irish adults (IUNA 1996-1998 survey)
  - the co-ordinated EU monitoring programme for 2005
  - priority given to commodities eaten in the raw state i.e. lettuce
  - the capacity of the laboratory to implement the programme
  - samples that gave rise to MRL breaches in 2004 are targeted
  - the availability of organic produce on the market.
- Samples are taken in accordance with the EU sampling Directive 2002/63. Two sampling officers from the Pesticide Control Service carry out the sampling of food of plant origin. Sampling is normally taken at wholesale level but occasionally it may be carried out at retail level.
- Enforcement action

An MRL is considered to be exceeded when the residue found is numerically greater than the indicated MRL. However enforcement action will not take place unless the residue found is greater than the sum of the MRL and the uncertainty of the analytical method.

12 samples were targeted as a follow up to MRL exceedences in 2004 or in response to a RASFF notification. Enforcement action was taken on two samples, one an Irish lettuce containing iprodione at 58 mg/Kg and the other on the unauthorised use of tecnazene on potatoes. Both samples were destroyed.

A RASFF information was issued on a consignment of grapes containing dimethoate.

### 3. QUALITY ASSURANCE

- The Pesticide Control Laboratory of the Department of Agriculture and Food is the only laboratory involved in the official control of pesticide residues in food of plant origin. This laboratory is accredited to ISO 17025 standard.
- The main method used for fruit and vegetables consists of extracting the samples with acetone, partitioning into Dichloromethane and Petroleum ether; cleanup by gel permeation chromatography and detection by gas chromatography with mass selective/ single ion monitoring, electron capture and pulsed flamed photometric detections. In the case of pesticides that are not amenable to gas chromatography such as the benzimidazoles, a selective HPLC with diode array detection is used. In the case of the cereal samples a variation of the fruit and vegetable method is used with ethyl acetate as the extracting solvent. In general, multi residue methods are used to detect as many pesticides as possible in one method.
- Participation in proficiency tests  
The Pesticide Control participated in the EU Proficiency test (PT 07) which was organised by the University of Alimeria as well as the FAPAS schemes for fruit and cereal.
- Implementation of EU quality control procedures  
The laboratory incorporates the majority of the EU Quality procedures for pesticide residues into its work practices.
- Analytical uncertainty  
The Laboratory uses the 50% figure to take into consideration inter-laboratory variations for MRL breaches.

### 4. OTHER INFORMATION

- No homogeneity exercises were carried out in 2005.
- All relevant EU Directives have been transposed into Irish Legislations
- Details of risk assessment  
In all cases where an MRL breach is exceeded a deterministic risk assessment is carried out based on the Irish consumption data. A study of the Irish children diet is now complete and will be available in 2006.

## ITALY

### 1. SUMMARY OF RESULTS

Of a total of 7132 samples (Tab.1 e 2), 2288 samples (32,1%) with residues not exceeding permitted levels were found, while 94 (1,3 %) were found with residues exceeding permitted levels; no residues were detected in 4750 samples (66,6%). The percentage of irregular samples is equal to 1,3% of which 1,5% for fruit and vegetables, 0,2 % for wine products and 0,4 % for cereals.

Oil product samples analysed resulted regular.

The percentage of residues within legal limits is highest for fruit and vegetables (32,7%), than for processed products (32,5 %) and cereals (14,6 %).

| SUMMARY OF DATA - YEAR 2004 |                     |         |                    |       |
|-----------------------------|---------------------|---------|--------------------|-------|
|                             | Fruit and Vegetable | Cereals | Processed products | Total |
| Nr. Of samples              | 6.330               | 254     | 548                | 7.132 |
| Regular samples             | 6.238               | 253     | 547                | 7.038 |
| Irregular samples           | 92                  | 1       | 1                  | 94    |
| Irregular samples %         | 1,5                 | 0,4     | 0,2                | 1,3   |

Tab. 1

| PESTICIDE RESIDUES IN REGULAR SAMPLES              |                     |         |                    |       |
|--|---------------------|---------|--------------------|-------|
|  | Fruit and Vegetable | Cereals | Processed products | Total |
| Nr. Of samples without residues                    | 4.165               | 216     | 369                | 4.750 |
| Nr. Of samples without residues %                  | 65,8                | 85      | 67,3               | 66,6  |
| Nr. Of samples with residues within legal limits   | 2.073               | 37      | 178                | 2.288 |
| Nr. Of samples with residues within legal limits % | 32,7                | 14,6    | 32,5               | 32    |

Tab. 2

### 2. ORGANIZATION OF MONITORING PROGRAMMES AND SAMPLING

#### RESPONSIBILITIES

The Ministry of Health – General Directorate for Food Safety and Nutrition – coordinates and defines Italian official control programmes on foodstuffs, including the annual plans regarding pesticide residues.

#### STRUCTURE OF THE PLAN

The annual official control plans on residues of plant protection products are defined by Ministerial Decree 23 December 1992, transposing Directive 90/642/EEC, integrated by the Ministerial Decree 30 July 1993 regarding the programming of official controls for importation from Third Countries.

The National Program Pesticide Residues (P.N.R.A.) foresees a detailed programme implementing the checks to be carried out by the Regions and Autonomous Provinces of Trento and Bolzano, with indication of the minimum number and the typology of samples to be analysed. The division of the number of samples to be taken for each Region/Province is calculated according to the data on consumption and production of a given foodstuffs in the Region or autonomous Province concerned. The Decree contains some tables reporting the number of samples to be taken for each

Region/Province for the following foodstuffs: vegetables, fruits, cereals, wine, oils, meat, milks and derivatives, eggs. The plan foresees also priority of a research of residues of plant protection products both in animal and vegetable origin foodstuffs.

As regards products of vegetable origin imported from Third Countries, the sampling is performed by Uffici di Sanità Marittima, Aerea e di confine terrestre of Ministry of Health, in at least 3% of a lot present at importation with a priority given to fruit and vegetable origin products.

### SAMPLING, STAFF, PROCEDURES AND SAMPLING SPOTS

Based on the programmes of the Regions and Autonomous Province, inspectors of a Local Health Units provides for implementation of sampling of foodstuffs to be tested for PPP residues.

The sampling spots indicated in P.N.R.A concerning products of plant origin are the collection centers and cooperatives for products coming from within the Region or Autonomous Province, specialised and non-specialised wholesale markets, wholesale stores, hypermarkets and supermarkets for products coming from outside the Region or Autonomous Province.

The sampling methods are those established by the Decree of the Ministry of Health of 23 July 2002, transposing Directive 2002/63/EC of 11 July 2002 regarding the methods of sampling for the Official control for pesticide residues in plant and animal origin products.

### MEASURES TAKEN

In case of irregular samples, the administrative or criminal sanctions foreseen by Law n° 283 of 30 April 1962 and Legislative Decree of 3 March 1993, n° 123 (transposing Directive 89/397/CE on official control of foodstuffs) are applied; contaminated foodstuffs are confiscated on a precautionary basis and/or destroyed.

## 3. QUALITY ASSURANCE

### ACCREDITATION

Official control public laboratories participating in 2005 in the national programme on pesticide residues in vegetables were 36, whereas the coordinated EC programme on pesticide residues in vegetables counted on the participation of 34 laboratories.

Several laboratories have been certified in accordance with norm EN 17025 and some other laboratories are being certified. Concerning the coordinated EC programme, the number of certified laboratories all over the country is equal to 18.

Table G laboratories is enclosed, showing the list of certified and non-certified laboratories, participating in the coordinated EC programme in 2005, in accordance with EC Recommendation 1.3.2005 (n.2005/178/CE).

### ANALYTIC METHODS

Analytic methods used mainly include GC multi-residue methods, associated with selective detectors (ECG, NPD, MS) and HPLC-UV.

### PARTECIPATION IN PROFICIENCY TESTS

Roughly 30 laboratories regularly take part to quality assurance tests organized by FAPAS, by the National Health Institute and by the ARPA regional laboratories (Regional Agencies for Environment Protection).

13 laboratories attended the 7<sup>th</sup> Proficiency Test (year 2005) organized by “European Commission’s Proficiency Test on pesticide Residues in Fruit and Vegetable”.

## CYPRUS

### 1. SUMMARY OF RESULTS

In 2005 a total of 406 samples of plant origin (248 vegetable samples and 158 fruit samples) were analysed. 12 samples were of organic farming as shown in table A1- Organic. Sampling rate was 53 samples /100 000 inhabitants. The main emphasis was on the local production including exports and only 14.5% of the tested samples were from imports. In addition 394 samples were analysed for QC and method development/validation. In 62.6% of the samples no residues were detected (ND) and in 29.8% the residues were at the level of or below the MRL. The percentage of samples above MRLs (national or EC) was 7.6%, of which 4.2% were considered as real legal violations. 1.7% of the samples were classified as “critical”. In parag § 4 term definitions are provided.

Additionally 24 baby food samples of fruit and vegetable origin and 24 samples of cereals were analysed .

Out of 138 pesticides sought in the samples of fruits and vegetables 42 were found to be present. The most frequently found pesticides were Cypermethrin (in 20.7% of the samples analysed), and Chlorpyrifos (in 17.7% of the samples analysed). Out of the 122 pesticides sought in cereals only 2 pesticides were found (Pirimiphos methyl and Diazinon).

In 13.5 % of the samples of fruits, vegetables and cereals analysed, two to five pesticides were detected. No pesticides were detected in the 24 baby food samples.

### 2. ORGANIZATION OF MONITORING PROGRAMS AND SAMPLING

Ministry of Health is the competent authority for the enforcement of the Pesticide Residues (PR) Legislation and the execution of the national monitoring and surveillance programs. The enforcement of Legislation and sampling is allocated to the Department of Medical and Public Health Services (MPHS). The Pesticide Residue Lab (PR-SGL) of the State General Laboratory is the Official Laboratory for the Monitoring & Surveillance of PR in Food of Plant and Animal Origin. The PR-SGL Lab and the MPHS design and implement a multisectoral program for local market, including imports and exports. The sampling regime is based on a combination of “at random” sampling and target oriented sampling focusing towards problematic pesticides/food combination. This combination is in a way bias towards problematic products and might end up with higher violation rates. Nevertheless it can provide higher degree of consumer protection and cost-effectiveness. Main criteria used in the sampling design are: violations, pattern of actual pesticide usage, info from RASFF, toxicological data, consumption data especially by children and the needs of exports control. Sampling is done by 72 well qualified Health Inspectors (with BSc or at least 3 years of education) of the MPHS, according to the harmonized Regulation KΑΠ 473/2004. It is focused at the key points of food chain: producers, market, import processing, primary storage etc. The MPHS exercise the regulatory functions. Enforcement actions are taken as follows: a) for all critical and violating samples a notification is issued to the Dept. of Agriculture, b) “Violating” samples, for which adequate evidence can be provided , are legally prosecuted by the Dept. of MPHS. Every effort is made to withdraw violating products from the market and prevent them from been exported/consumed. Imported products non complying with the EU harmonized MRLs are not permitted to enter Cyprus. In our legislation there is a provision which allows to use 0.01 mg/Kg in cases where specific limits are not provided either in the EU or other national legislation. The action taken, when samples found to exceed this low level, follows a defined procedure which considers the particular problem, the LOQ and the related uncertainties.

### 3. QUALITY ASSURANCE

The PR Lab of the SGL is accredited by the Greek Accreditation body ESYD since 2002 according to EN 45001, from June 2003 according to ISO/IEC 17025 and from July 2006 according to

ISO/IEC 17025/2005. The following validated methods are mainly used: 1) "Multiresidue method for fruits, vegetables and milk including PCBs in milk": Ethyl acetate extraction, GPC-clean up, determination/confirmation by GCMS/ITD, GC/FPD, GC/ECD. It covers GC amendable Pesticides within polarity ranging from  $Kow = -0,9$  up to non polar. 2) GC/FPD-S determination of dithiocarbamates as CS<sub>2</sub> extracted in isooctane after reaction with aqueous hydrochloric acid in the presence of tin (II) chloride 3) "Multiresidue method for the determination of organochlorine and PCBs in fish and meat products": Soxhlet extraction, GPC clean up, GC/ECD–dual column detection.. The analytical uncertainty has been estimated during validation process at different concentration levels and is taken into consideration for "decision-making" especially to discriminate between "real legal violations" and "above MRLs".

LC/MS analytical system has been introduced and used in 2005 for measurement of polar compounds and a limited number of samples (9) have been analysed as shown in table A2 part 1 .

The PR-Lab applies Quality Control procedures, which are in line with the provisions of "EU-Quality control procedures" concerning the determination, confirmation and method quality. The lab participated in the following Proficiency tests: a) European Commissions Proficiency Test on Pesticide Residues (PT7), b) OP and Pyrethroids Pesticide Residues in wheat flour, FAPAS Test 0941, c) Pesticide Residues in vegetable oil - FAPAS (Ser.9, Rd.37), d) Organochlorine PCBs and Arochlors in tuna-homogenate IAEA – Intercomparison Run 435

#### 4. OTHER INFORMATION

Homogeneity exercise : According to the Recommendation 2005/178/EC the analysis of individual units of a sample of potatoes containing Endosulfan, was executed. The results are shown in table F.

Background on legislation: The main legal instrument for the protection of Public Health in Cyprus is the Harmonized Food (Control and Sale) Law of 1996-2005, and the EU harmonized Pesticide Residues and sampling Regulations.

The management of results: To increase the cost-effectiveness, the use of the term "critical" sample has been introduced since 2002. Critical are those samples which are either above the MRL but when the uncertainty is subtracted cannot be considered as legal violation or are at or below the MRL and when uncertainty is added exit the MRL. The samples are ranked in the following categories: "without detectable residues", with residues below or at MRL", "above the MRLs" "critical" and "violating samples". Therefore the % of above MRLs recorded in all Tables comprises the legal violations and part of the critical samples. Decision on actions are taken according to a standardized SOP process and in compliance with the legislation.

Risk Assesment : For samples containing pesticides, which exceeded MRL, the PSTI values were estimated according to Sanco Document SANCO/3346/2001rev6(Proposal on notification criteria for pesticide residue findings to the Rapid Alert System for Food and Feed' (RASFF) .

LATVIA

## 1. Summary of results

In a year 2005, in total 115 samples of cereals, fruits and vegetables were analyzed for 56 pesticide residues. From that number of samples 31 were of domestic origin, 42 samples were of origin of other EU MS, and 35 samples were taken from products that were imported from third countries. The EC maximum residue limits were exceeded in 4 samples from all food samples analyzed (4,6%) and in 90 samples the residues were under LOD. No residues were detected in the samples of organic products and in the samples of baby food.

All samples both in national and in EU coordinated programs were taken only for surveillance purposes. In total 17 products were analyzed- beans, potatoes, carrots, oranges, mandarins, spinach, rice, cucumbers, apples, watermelons, melons, aubergines, lemons, paprika, table grape, head cabbage and dills.

The EU maximum residue limits were exceeded in 4 samples: in 2 samples of carrots the procymidone exceeded MRLs, in a sample of oranges dimethoate was in exceeding amounts and in a sample of spinach chlorpyrifos was detected in exceeding amounts. In these cases the control institution issued warnings except for oranges where a Rapid Alert was notified.

In 15 samples multiple residues were found, but the number of pesticide compounds analyzed did not exceed 3 pesticides per product. Three pesticide residues were found in beans, cucumber, pears, mandarins and oranges. The pesticides that were found most frequently are: imazalil, thiabendazole, procymidone, chlorothalonil, cypermethrin, chloropyrifos, benomyl group, bromopropylate, phosalone, pyrethrins.

## 2. Organization of monitoring programs and sampling

The monitoring program of pesticide residues was elaborated in the Ministry of Agriculture in cooperation with Food and Veterinary Service, State Plant Protection Service and Latvian Food Centre.

The Food and Veterinary Service is responsible authority for practical application of monitoring program and it determines how many samples in each region should be taken and shares its responsibility with Sanitary Border Inspection that carries out the sampling at the border points. There were no enforcement sampling carried out in 2005, only surveillance sampling.

The commodities were selected taking into account the average consumption, the proportions of products produced and products imported, the budgetary possibilities, the findings in previous controls, usage of plant protection products, capacity of laboratory and coordinated EU coordinated monitoring program for 2005.

The fruits and vegetables subjected to sampling for monitoring purposes were either fresh, processed, frozen, either domestic or imported ones.

Sampling is conducted according to the requirements laid down in Directive 2002/63/EC and samples are taken in 27 district offices of Food and Veterinary Service at all stages of distribution chain, including at the farm gate in the case of domestic products. Samples were taken by trained inspectors from Food and Veterinary Service and Sanitary Border Inspection.

Samples were analyzed in the Food and Environmental Investigation Laboratory of the National Diagnostic Centre of Food and Veterinary Service which is the only laboratory carrying out the monitoring exercises.

### **3. Quality assurance**

The Food and Environmental Investigation Laboratory has introduced a quality assurance system according to the requirements of ISO 17025 standard. All methods for determination of pesticide residues are included into the scope of accreditation of laboratory. The laboratory has implemented the EU Guideline on Quality Control Procedures.

Laboratory has successfully participated in European Commission Proficiency Testing programs for detection of pesticides residues in fruits and vegetables (from EUPT-5 till EUPT-8). The laboratory has been accredited according to ISO/IEC 17025 and has for the most part implemented EC Quality control procedures that are laid down in SANCO 10476/2003 "Quality Control Procedures for Pesticide Residue Analysis".

The method applied for control of pesticides residues in Latvia consists of an acetone or ethyl acetate extraction and sample clean up using gel filtration chromatography. The gas chromatography with NPD and ECD detectors is applied for analysis of main part of pesticides. Liquid chromatography – tandem massspectrometry is used for pesticides not amenable to GC. Dithiocarbamates are analysed as CS<sub>2</sub> using GC-ECD after decomposing with tin chloride solution.

The analytical uncertainty of results is calculated based on relative standard deviation of recovery rates and results of proficiency testing if available. The estimated range of uncertainty is from 10 to 30%.

### **4. Other information**

The monitoring program is carried out in accordance with Recommendation 2005/178/EC of 1 March 2005 concerning a co-ordinated Community monitoring programme for 2005 to ensure compliance with maximum levels of pesticide residues in and on cereals and certain other products of plant origin.

The MRLs that were applied in monitoring programme are laid down in Directives 76/895/EEC, 86/362/EEC and 90/642/EEC. No national MRLs for pesticide/product combinations were applied in this monitoring program.

## 1. SUMMARY OF FINDINGS

In 2005, 364 samples on pesticide residues were selected from fruit, vegetables and grain and tested; of these 278 samples were tested in accordance with the monitoring programme and 86 follow-up enforcement samples. 133 samples from the produce originating in Lithuania were tested; of these 127 samples were tested in accordance with the monitoring programme.

Sampling in accordance with the monitoring programme:

Fruit and vegetables:

Total number of samples – 256 were tested. 113 samples from fruit and vegetables of local origin were tested, 109 samples from EU countries and 34 samples from third countries. Of 256 samples of fruit and vegetables tested pesticide residues were not detected in 178 samples; in 77 samples the detected level was below the tolerable level or at the level, but not above MRL; in 1 sample the MRL was exceeded. The pesticide dimethoate was detected in apples from Poland, at 0,23 mg/kg.

Cereals:

Total number of samples – 22 were tested. 14 samples from cereals of local origin were tested, 3 samples from EU countries and 5 samples from third countries. Of 22 samples of cereals tested pesticide residues were not detected in 20 samples; in 2 samples the detected level was below the tolerable level or at the level, but not above MRL.

Follow-up enforcement sampling:

Of 55 samples of fruit and vegetables tested no pesticide residues were detected in 26 samples, in 24 samples the detected level was below the tolerable level or at the level, but not above MRL; in 5 samples the MRL was exceeded. Pesticide fenvalerate was detected in 4 samples of raisins from Iran (0,73 mg/kg, 0,69 mg/kg, 1,14 mg/kg and 0,40 mg/kg). In lemons from Turkey pesticide paration-methyl at 0,47 mg/kg;

Cereals. Of 31 samples of cereals tested no pesticide residues were detected in 29 samples, in 2 samples the detected level was below the tolerable level or at the level, but not above MRL.

## 2. IMPLEMENTATION OF MONITORING PROGRAMME, SELECTION OF SAMPLES

State Food and Veterinary Service (hereinafter – SFVS) and its regional divisions (10 county SFVS and 4 city SFVS) are responsible for the implementation of the monitoring programme. Inspectors trained for the function take samples from foodstuffs and are responsible for a correct selection, marking of the samples and delivery to the laboratory. SFVS control is accredited by EN ISO/ICE 17020:2004 standard.

Priorities at sample selection:

Recommendations of the EU Commission, recommendations of the Ministry of Health, risk of pesticide contamination, data of earlier laboratory analysis, information of Plant Protection Service on the amounts of pesticides used, budget allocations for the implementation of the programme.

Samples are taken from the products obtained in Lithuania, transported from the EU countries and third countries (import). Samples from EU countries products are taken on the market and of imported (third countries) – within the customs area, at the place of unloading. From local products samples are taken directly at the manufacturer or on the market. Local samples make one third of all samples. Samples are selected in accordance with the provisions of Commission Directive 2002/63/EC, which has been transposed into a national legal act approved by the order of the Minister of Health. Food inspectors of the SFVS observe the approved sample selection procedures and use the required sampling equipment and packaging.

In 2005, no testing of infant food was carried out in accordance with the monitoring programme, but sampling was conducted under other programmes, where infant food was tested for nitrates and patulin.

### 3. QUALITY ASSURANCE

#### Laboratory used and accreditation

The analyses were carried out in the National Veterinary Laboratory (NVL). This laboratory is accredited since 2000 according to EN 45001 and in the year 2003 the laboratory was reaccredited according to EN ISO/IEC 17025 by German Accreditation Body DAP for all methods used for official control of pesticide residues in food of plant origin. The visit of assessors of DAP was conducted on 25-30 of July 2004.

#### Analytical procedures

Most of samples were analysed by multi-residue method EN 12393-(1-3): 2000 "Non - fatty alimentary products. Multi - stage detection of pesticide residues using the method of gas chromatography". By this method, the samples were extracted with ethylacetate or acetone-cyclohexane, cleaned up on gel permeation column and determined by capillary gas chromatography.

Dithiocarbamates were determined by method EN 12396-(1-3):2000 "Non-fatty alimentary products. Determination of residues of Dithiocarbamates and thiuram pesticides. Spectrophotometric method. Pesticides surveyed: CS2 - Maneb group (Maneb, Mancozeb, Metiram, Propineb, Zineb) and Thiuram.

Diphenylamine, thiabendazole, o-phenilphenol, benzimidazoles, benomyl group were determined by HPLC –multimethod.

#### Participation in proficiency tests

NVL participated in Proficiency tests organised by FAPAS, England, Series 19, round 42, Pesticide residues in food, determination of pesticide residues in Apple puree. 2004 November.

#### Quality control procedures

Quality control procedures include daily checks of instruments sensitivity, possible matrix effects by injection of test solution. Confirmation by mass spectrometry is used for results above reporting limits. Implementation of EU Quality control procedures for pesticide residues analysis (SANCO/10476/2003) is in process.

#### Analytical uncertainty

Uncertainties of analytical results were estimated in process of in-house validation at the level of MRL. Level of uncertainty depend on individual and is in the level of 20 to 45 % of analytical results.

### 4. Other information.

The results of the tests performed on apples, raisins and lemons has obligated the State Food and Veterinary Service to make modification in control plans for foodstuffs imported from third countries. Monitoring of foodstuffs is carried out in accordance with the EU laws, with an emphasis on the risk factors of product contamination.

## LUXEMBOURG

### 1. SUMMARY OF RESULTS

In 2005, 108 samples of plant origin were examined. 20% of the products (mainly wine grapes and barley) were domestic samples.

Chlorpyrifos at or above 0.3 mg/kg (MRL) were found in 4 (out of 7) orange samples without posing a risk to the consumer, however. This exceedance may be due to higher MRLs for mandarins than for oranges. There was no follow-up sampling.

On one wine grape sample 6 different fungicides were found. Legislative tools to limit the number of fungicides applied are missing, however.

Table: Summary of the 2005 monitoring programme (co-ordinated and national programme)

| N° of samples of plant origin analysed | N° of samples without residues | N° of samples with residues at or below the MRL | N° of samples exceeding MRL |
|--|--------------------------------|---|-----------------------------|
| 108                                    | 61                             | 45  | 2                           |

The list of most frequently found pesticides is closely linked to the commodities analysed: folpet was mainly found in wine grapes, chlorpyrifos in citrus fruits, tolylfluanid in pears, chlorpropham in potatoes. The listing depends also on the number, on the up-to-dateness of the pesticides analysed and on the reporting levels. These parameters were rather conservative.

Organic samples and baby food were included in the programme, although reporting levels were mostly above 0.01 mg/kg.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

Pesticide residues were analysed at the laboratory of food control, being a division of the National Laboratory of Health (Ministry of Health).

Due to limited personal resources (one technician), the annual programme consisted mostly of the EC programme. Instead of the by the EC programme recommended rice samples, local grown barley samples were analysed. Domestic wine grapes were analysed on a national programme base. The objective to collect at least one organic sample per commodity was not achieved, as not enough emphasis was placed on during the sampling year.

The samples were collected by a food inspector of the food control unit. Imported products were sampled at wholesaler distribution points and retailers. Wine grapes were obtained directly from winegrowers, barley from breweries. Due to the sampling requirements of directive 2002/63/EC (number of samples and sample sizes) no samples could be taken at the central market in the City of Luxembourg any more.

Warning letters were sent to the selling points of the two positive samples.

### 3. QUALITY ASSURANCE

The laboratory of food control is the only laboratory doing pesticide analysis in Luxembourg. It was accredited in 2003.

Pesticides were analysed by a gas chromatographic multi-residue method with ECD, NPD and MSD (DFG S19, modified).

Pesticides of the Maneb group were analysed separately with the Keppel-method (CS<sub>2</sub> product).

For deciding whether a MRL was exceeded or not, an analytical uncertainty of 30% was taken.

In 2005, the laboratory participated in the proficiency test organized by the EC (pesticide residues in grapes).

Not all EU quality control procedures could be fully implemented (e.g. frequencies of verification and calibrations). Due to limited resources it becomes more and more difficult to strike a balance between the steadily increasing demands concerning

- the annual programme,
- method performance (lowering reporting limits, number of pesticides, single residue and non-GC-methods [necessary e.g. for aldicarb, benomyl, imidacloprid, spiromamine]),
- and quality control.

## 1. Summary of results

### Plant Protection and Soil Conservation Service (PPSCS):

The pesticide residues in raw agriculture commodities of plant origin were controlled by the 8 laboratories of **PPSCS in 2005**.

Altogether 2,069 domestic and 1,624 import samples (from other EU Member States and third countries) were analysed. The domestic samples were taken at the markets (1248) or from the fields at the time of harvest (821), and 62.3% of them did not contain any detectable pesticide residues. The maximum residue limits (MRL) were exceeded in 1.2% of the samples. From the 1,624 import samples, 74 samples contained residues above the MRL (4.6%), while in 37.9% the residues were below the LOQ. The import samples, derived from 36 countries, were taken at the Border Inspection Points and in the wholesale premises represented 44 crops, and they were tested for 160 active substances.

Green paprika from Morocco contained most frequently residues above MRL indicating that the change of methamidophos MRL was not properly communicated to the growers and they continued applying the pesticide according to the previous use patterns.

In 2005, as part of their official inspections in the Food Safety Programme, the county Services, carried out country-wide inspections with well organised sampling scheme in wholesale premises and in logistic centres of chains of stores, three times – two times together with the National Public Health and Medical Officials' Service.

**358 samples** were tested by the Analytical Laboratories. **3.9 %** of the samples were over MRL. Large quantity of lemon and mandarin contained detectable volume of pesticide residues.

7 cases with high pesticide residue content were reported through the Hungarian Food Safety Office to DG SANCO through Rapid Alert System for Food and Feed.

**National Food Investigation Institute (NFII)** tested residues of 24 pesticides in 28 baby food samples. None of the samples contained residues above the MRL and no residue could be detected in 18 samples.

## 2. Organisation of Monitoring programmes and Sampling

### Responsibilities (Two organisations under the supervision of Ministry of Agriculture and Regional Development, MARD)

**CSPPSC** - supervising 8 regional laboratories. CSPPSC is responsible for coordination the tests of pesticide residues in unprocessed agriculture commodities.

**NFII** is responsible for testing pesticide residues in baby food.

### Plan of Programmes

CSPPSC plans the sampling scheme considering the *consumption habits* of the Hungarian population, the NFII compiles monitoring plan according to *production data of previous year*. Number of tested samples was about the same as those in the previous years. Regulation for baby food is strict, due to this fact importance of monitoring the products is increasing.

### Sampling

The sampling procedure is specified in the following national decrees:

- Decree 7/2001(I.17.) MARD on the implementation of phytosanitary measures
- Decree 82/2004 (IX.18) Ministry of Health (MH) taking samples – fruit and vegetable

- 5/2002 joint Decree (II.22) MARD-MH on MRL in plants, products of plant origin (continuously harmonised by amendments with the Community legislation).

The analytical network of PPSCS prepared the programme for official sampling. This programme covered most of the products representing the main consumption habits of Hungary. Plant health inspectors at the border, plant protection inspectors within the country and Veterinary Services in industry and trade take random samples.

#### **Enforcement action**

In every case where detected residues of samples were above MRL.

### **3. Quality assurance**

#### **Status of accreditation of laboratories**

Laboratories work according to OECD-GLP systems, have accreditation, i.e. their procedures follow the quality assurance practice based on requirements of MSZ EN ISO 17025 standard and joint decree 31/1999 (VIII. 6.) MH-MARD and 9/2001 (III. 30.) MH-MARD.

#### **Analytical methods**

Test methods are recorded in the Standard Operation Procedures (SOP) of the Services. The local inspections made by quality assurance inspectors and the audits of testing plans and reports contribute to the improvement of working efficiency.

#### **International proficiency tests**

128 laboratories took part in the 7<sup>th</sup> **European Proficiency test** in 2005, including all the Analytical Laboratories of the *national plant protection organisation and NFII*. 5 of these laboratories ranked among the first 17.

#### **Analytical uncertainty**

Depending on the concentration

| Concentration        | Repeatability     |                   | Between Laboratory Reproducibility |                   | Trueness                 |
|----------------------|-------------------|-------------------|------------------------------------|-------------------|--------------------------|
|                      | CV <sub>A</sub> % | CV <sub>L</sub> % | CV <sub>A</sub> %                  | CV <sub>L</sub> % | Range of mean Recovery % |
| ≤1 ug/kg             | 35                | 36                | 53                                 | 54                | 50 - 120                 |
| >1 ug/kg≤0.01 mg/kg  | 30                | 32                | 45                                 | 46                | 60 - 120                 |
| >0.01mg/kg≤0.1 mg/kg | 20                | 22                | 32                                 | 34                | 70 - 120                 |
| >0.1 mg/kg≤1 mg/kg   | 15                | 18                | 23                                 | 25                | 70 - 110                 |
| > 1 mg/kg            | 10                | 14                | 16                                 | 19                | 70 - 110                 |

### **4. Other Information**

#### **Hungarian legislation**

All the relevant Hungarian legislation was listed in our report for 2004. **Government Decree 302/2005 (XII. 25.)** allocated new tasks to NPPSCS from 1<sup>st</sup> January 2006 by extending its pesticide monitoring tests to processed food of plant origin, including baby food and drinks. The heavy metal content of unprocessed products of plant origin will be tested in three regional Soil Conservation Laboratories. NFII is responsible for monitoring all kind of food made of or containing component of animal origin.

## 1. Summary of Results

For the purposes of the monitoring programme for pesticide residues for 2005, a total of 65 samples were tested using a multi-residue method capable of identifying and quantifying 164 pesticide residues.

Commodities of local origin accounted for 81.5% (53 samples), while commodities originating from other European Union (EU) Member States accounted for 18.5% (12 samples) of the total number of sampled commodities. Sampled commodities included oranges, apples, peaches, grapes, strawberry, tomatoes, melon, cauliflower, kohlrabi, and broad beans.

The number of samples with residues at or below Minimal Risk Level (MRL) (national or EC) or for which no MRL is set, represented 15.4% (10 samples) of the total number of samples analysed. 4.6% (3 samples) of the samples had pesticide residues that were in excess of EC MRLs.

In total, 11 different pesticide residues were detected (above LOD). The frequency of detection was as follows:

- chlorothalonil: 6.2% (4 samples);
- fenitrothion: 4.6% (3 samples);
- dimethoate: 3.1% (2 samples);
- iprodione: 3.1% (2 samples);
- chlorpyrifos-ethyl: 1.5% (1 sample);
- cyprodinil: 1.5% (1 sample);
- dichlofluanid: 1.5% (1 sample);
- fludioxonil: 1.5% (1 sample);
- imazalil: 1.5%(1 sample);
- penconazole: 1.5% (1 sample);
- thiobendazole: 1.5% (1 sample).

MRL infringements were found in oranges originating from Italy for chlorpyrifos-ethyl and dimethoate residues, and for peaches grown in Malta that exceeded the MRL for dimethoate. The results for pesticide residue analysis for 2005 indicate a decrease in the relative number of samples of produce of local origin that were positive for dimethoate residues compared to 2004 and 2003.

## 2. Organisation of Monitoring programmes and Sampling

The Rural Affairs and Paying Agency within the Ministry for Rural Affairs and the Environment was responsible for the drafting of the National Monitoring Programme for fresh produce of plant origin for 2005. Priorities for drafting the programme included the following:

- the dietary importance of the food commodity (to adults, infants and children);
- domestic production/imports of commodities;
- past findings that may indicate a historical residues problem;
- in the light of new risks (e.g. knowledge on use of banned pesticides) or other country monitoring schemes.

The sampling was performed by 3 officers from the Rural Affairs and Paying Agency. The sampling procedures were those described in the Schedule 1 to the European Community Methods of sampling for official Control of pesticide residues in and on Products of Plant and Animal origin Regulations (LN479/04). Sampling points included organised markets for local produce, and import points for imported produce. The samples were sealed and double coded. The relevant details of the samples were inserted in a database to ensure the identity of the samples. Enforcement actions included issuing of warning letters and investigations. Enforcement sampling was not performed due to financial limitations.

## 3. Quality assurance

Samples were sent for multi-residue Analysis to CEFIT Laboratory (Sicily, Italy) which is accredited by Sinal (Sistema Nazionale Accredimento Laboratori - National System of Laboratory Accreditation), with accreditation No. 0194. This lab is in compliance with the criteria of European standard UNI-EN 45001

(ISO/ICE 17025). The laboratory participates in the Ring-Test Arpa-Ferrara, Ministero Agricoltura (Patologia Vegetale), Roma.

The described methods are based on EU legislation and are based on procedures that are carried out in four stages as described below:

- Extraction - pesticide residues are extracted from the sample matrix by employing appropriate solvents in such a way to obtain maximum extraction efficiency and minimum co-extraction of the sample matrix which could interfere with the analysis.
- Purification - the purification has the aim of removing interfering materials in such a way to obtain a solvent devoid of interfering substances.
- Analysis - the analysis is performed using gas-chromatography technique and using selective detectors: electronic capture device (ECD) for halogenated organic substances, NPD for organophosphates, and organonitrates.
- Confirmation of results - in order to confirm residues qualitatively and quantitatively in cases where MRL infringements are observed. In these cases liquid gas chromatography (LC-MS) is employed.

The method used by the sub-contracted laboratory is Method M, a brief description of which follows. Extraction with acetone and liquid partitioning/dichloromethane/petroleum ether and if necessary purification with Florisil. A portion of chopped sample is homogenised with acetone and filtered. An aliquot of the filtrate is extracted with a mixture of petroleum ether / dicloromethane. The organic phase can be injected directly in the gas chromatography equipment with an appropriate detector or purified through a Florisil column. The eluant is concentrated for chromatographic analysis as described the sections above. The uncertainty of results is communicated on the certificate of analysis.

## NETHERLANDS

### 1. SUMMARY OF RESULTS

In 2005 about the same percentage of non-compliances was found for non-domestic samples as in previous years (Figure 1). For the third year EU product show a decrease in percentage non-compliances, whereas non-EU product violations increase. Probably progressing EU-harmonisation contributes to this effect. In 2005 the contribution of EU-MRLs to the number of MRL-violations has risen to 67 %, compared to 53 % in 2004. About 63 % of the residue findings is governed by EU-MRLs (Table 3). Table 1 gives the most frequently non-complying pesticide/crop combinations with the main countries of origin. In 2005 the Netherlands issued seven rapid or information alerts on pesticide residues. Table 2 gives an overview. Both at the non-compliances and the RASFF-notifications occurrences of highly toxic obsolete pesticides from third world countries are predominant. In about 3500 samples about 6000 residues of 189 different analytes were found. The scope of the coordinated program comprised 70 % of the residues found. For a majority of the results an Acute Reference Dose (ARfD) has been established (table 3). For a number of citrus (3), pear (2) and cucumber (1) samples, products in the co-ordinated program, a homogeneity exercise has been performed. Table 4 summarises the results. The homogeneity factor varied considerably, being quite high for most of the pesticides in the cucumber sample and low for post-harvest treatment pesticides on citrus fruits.

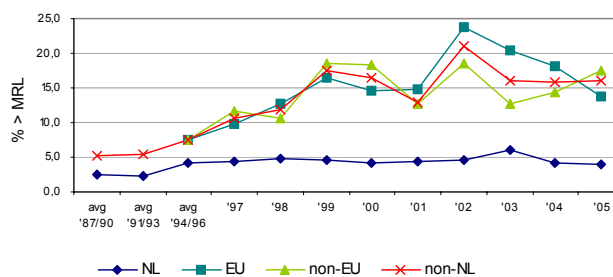


Figure 1. Percentage of MRL violations not including incidents

Table 1. Main products with high percentages of non-compliances, with corresponding pesticides and countries of origin.

| Product                            | Pesticides                              | % >MRL | Countries                |
|------------------------------------|---|--------|--------------------------|
| Tropical legume vegetables (fresh) | dimethoate, omethoate, epn              | 50,0   | Thailand                 |
| Pepper                             | carbendzim, dimethoate, methamidofos, d | 32,7   | Thailand                 |
| Tangerines                         | fenthion                                | 24,4   | Spain, Turkey            |
| Peach/nectarine                    | etofenprox, fenthion                    | 21,7   | Spain                    |
| Beans with pod (fresh)             | dimethoate, dicofol                     | 20,9   | Kenya, Senegal, Thailand |
| Orange                             | dimethoate                              | 18,2   | Egypt                    |
| Grape                              | tebufenpyrad, imazalil                  | 16,3   | Italy, Turkey            |
| Sweet pepper                       | methiocarb, pyrimethanil                | 14,9   | Spain                    |

Table 2. Alerts to the RASFF system issued by the Netherlands.

| Product         | Pesticide                                     | Country         |
|-----------------|---|-----------------|
| lettuce         | oxydemeton-methyl (0.94 mg/kg)                | Belgium         |
| edible orchids  | cypermethrin (14 mg/kg) and omethoate (12 mg) | The Netherlands |
| yard long beans | dicrotophos (0.9 mg/kg)                       | Thailand        |
| yard long bean  | triazophos (0.45 mg/kg) and EPN (0.22 mg/kg)  | Thailand        |
| kiwi            | methidathion (0,36 mg/kg)                     | Greece          |
| grapes          | methomyl (0.36 mg/kg)                         | South Africa    |
| yard long beans | dicrotophos (0,94 mg/kg)                      | Thailand        |

Table 3. Pesticide residues found in the EU-coordinated and Dutch monitoring program.

|                           | active substances | number of residues of pesticides in samples |                |              | with EU-MRL | total |
|---------------------------|-------------------|---|----------------|--------------|-------------|-------|
|                           |                   | with ARfD                                   | no ARfD needed | ARfD unknown |             |       |
| EU-coordinated monitoring | 55                | 2765  | 1354           | 4            | 3240        | 4123  |
| Dutch national program    | 124               | 1116  | 651            | 104          | 557         | 1871  |
| Total                     | 179               | 3881  | 2005           | 108          | 3797        | 5994  |



Table 4. Summary of homogeneity factors (maximum unit concentration divided by sample mean) from exercise on citrus, pear and cucumber.

| Pesticides in citrus | samples | mean | minimum | maximum | Product      | Pesticides         | homogeneity factor |
|----------------------|---------|------|---------|---------|--------------|--------------------|--------------------|
| chloorpyriphos       | 2       | 3,6  | 2,4     | 4,7 *   | Pear         | carbendazim        | 1,6                |
| imazalil             | 3       | 1,3  | 1,1     | 1,4     |              | dmst               | 1,5                |
| malathion            | 1       | 5,5  |         |         |              | tolyfluanide       | 1,5                |
| thiabendazole        | 1       | 1,7  |         |         |              | acetamiprid        | 2,0                |
| o-phnylphenol        | 1       | 2,4  |         |         |              | difenoconazool     | 2,7                |
| pyriproxifen         | 1       | 3,2  |         |         |              | diflubenzuron      | 1,8                |
| fenthion (sum)       | 1       | 2,6  |         |         |              | lambda-cyhalothrin | 1,7                |
|                      |         |      |         |         |              | spirodiclofen      | 4,1                |
|                      |         |      |         |         |              | Cucumber           | carbendazim        |
|                      |         |      |         |         | dimethomorph |                    | 4,2                |
|                      |         |      |         |         | famoxadone * |                    | 3,2                |
|                      |         |      |         |         | imidacloprid |                    | 6,2                |
|                      |         |      |         |         | metalaxyl *  |                    | 3,6                |

\* concentration in composite sample below reporting limit

## 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

The Food and Consumer Product Safety Authority performs the official monitoring in the Netherlands. The samples are taken without prior information about the presence of pesticides in the sample. Therefore, they represent the situation on 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. Therefore, high violation rates can indicate both an efficient sampling strategy and problems in the agricultural practice.

The Dutch Food and Commodity Law regulates the sampling procedure, i.e. the number of subsamples taken from a lot. This regulation is the implementation of the EC-directive 2002/63/EC. Inspectors of the five regional inspectorates are taking samples.

The main sampling points are the premises of the auction system for Dutch products and importers, warehouses and distribution centres of retail chains for both domestic and non-domestic products. At those inspection points it is clear who is responsible for the product, so that appropriate legal action can be taken in case of non-compliance.

## 3. ANALYSIS AND QUALITY ASSURANCE

One regional laboratory (Northwest, in Amsterdam) performs the analyses of the samples. The general strategy is detecting as many pesticides as possible in one analysis by using Multi-Residue-Methods (MRMs). The Dutch method consists of an acetone extraction, followed by a partition step of the residues into dichloromethane/petroleum ether. The extracts are analysed by a chromatographic separation and selective detection of residues. The main detection methods are Gas Chromatography (GC) - Ion-Trap Mass Spectrometric Detection (GC-ITD) and Liquid Chromatography – tandem Mass Spectrometry (LC-MS/MS). Only for some analytes that are not detectable sensitively enough by ITD, additionally GC with Electron Capture Detection (ECD) is used.

For some pesticides not amenable to the MRM, Single Residue Methods based on LC-MS/MS detection are used. In the 2005 program this was only the case for chlormequat.

Dithiocarbamates are analysed as CS<sub>2</sub> using GC-FPD and GC-ITD after decomposing with acidic tin-chloride solution and extraction into iso-octane.

Together the scope of the methods is about 400 analytes.

The validity of the analytical results is governed by a quality assurance system under ISO17025 accreditation. The multi-residue methods are within the scope of the accreditation of the laboratory. The centralised laboratory has implemented the EU Guideline on Quality Control Procedures. It takes part in FAPAS and EU proficiency tests.

The average inter-laboratory relative standard deviation (RSDR) is estimated at 25 % based on EU-proficiency tests. Only in case an MRL is exceeded by more than the estimated expanded average measurement uncertainty of 50 %, legal action is taken.



## AUSTRIA

### 1. SUMMARY OF RESULTS

In 2005 a total of 1775 samples of fresh fruits and vegetables were analysed under the co-ordinated program, the national pesticide monitoring program and as routine samples. Beside that other products like cereals (29 samples), processed products (125 samples) and baby food (183 samples) were analyzed.

46,7 % of all samples of fruits and vegetables were from Austria, 32,8 % from the European market and 20,5 % from third countries. For cereals this rates were 45 %, 24 % and 31 % respectively. The rates for processed food were 61,6 %, 31,2 % and 7,2 %. Baby food was predominantly from the European market including Switzerland (100 %).

In 51,5 % of the samples of fruits and vegetables no pesticide-residues could be detected. 41,5 % of the samples had residues under the harmonized and/or national Maximum Residue Limits (MRL). In sum 93 % of these samples were in compliance with the regulations. 7 % of the samples of fruits and vegetables contained one or more pesticide(s) above the MRL.

In all analysed samples including processed food and baby food the percentages were 57,4 %, 36,6 % and 6 % respectively.

In 490 samples (27,6 %) more than one pesticide was analysed. Up to 17 pesticides were found in some samples. The samples with more than 9 pesticides were in all cases grapes or peppers (12 samples).

In the samples were analysed up to 262 different pesticides. Totally 271 different pesticides were sought, of which 121 (44,6 %) were found. The most frequently found residues in fruits and vegetables were in the co-ordinated and national programme and routine samples (percentage): Maneb-group, Procymidone, Chlorpyrifos, Imidacloprid, Cyprodinil, Azoxystrobin, Iprodione, Fludioxinil, Endosulfane, and Fenhexamid.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

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 Health and Women. The plan was based on data concerning dietary consumption, production and import of fruits and vegetables and results of former measurements. Furthermore the results of earlier monitoring-programs, the analytical possibilities and the budgetary situation were taken into account, too. The co-ordinated programme of the European Commission was of course also done. Samples of leek haven't been analysed under this program due to a misunderstanding

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

### 3. QUALITY ASSURANCE

The analysis of the co-ordinated programme, the national monitoring programme and routine samples also were made by two laboratories for food control (Austrian Agency for Health and Food Safety, Institute for Food Control, Vienna and Institute for Food Control, Innsbruck). One Laboratory in Vienna (Regional Institute for Food Control in Vienna) analysed routine samples.

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 262 pesticides. The methods used were a GC multimethod with ECD-, NPD- and FPD-detection, a HPLC-method and a spectrophotometric method for the sum of dithiocarbamates. GC/MS-methods are primarily applied for confirmation purposes of the other GC methods.

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

#### **4. OTHER INFORMATION**

Due to the fact, that there were some commodities for the national programme isolated, of which higher risk for residues was identified in the last years, these specific data are representative for the Austrian market, but the monitoring has to be seen partially as „targeted monitoring“. It was the aim, to reflect to the results of the last years and to choose special commodities of interest for further examination. This type of monitoring is foreseen for the next years.

Furthermore the routine sampling includes special samples, of which European alerts were given, too and thus the number of exceedance is higher than by doing statistical based sampling over all commodities and time of the year.

Additionally there were studies on the homogeneity of residues exercised on 3 pesticides: pears: 2 x Chlorpyrifos (homogeneity factor 1,67; 2,05), Phosmet (2,18).

## 1. SUMMARY OF RESULTS

In 2005 in Poland in the range of official control and monitoring total of 1251 samples of food of plant origin were analysed for pesticide residues; the samples were taken from market (retail and wholesale) and in some cases directly from producers or in the range of border control. 71% of samples were fresh and frozen fruit and vegetables. 10% of samples were food for infants and babies including cereal products, milk-cereal products and vegetable-meet preserves. Samples analysed for coordinated EU monitoring composed 33% of a total number of examined samples. In 2005 in the range of coordinated monitoring also 7 samples of organic products were analysed (1 sample of fruits and 6 of cereals).

73% of samples were of domestic origin; remaining samples originated from other EU Member States (18%) and from the states outside the EU (9%). Among these samples 60% were without any residues and in 38% of samples the residues were found below or at MLRs. In this group were also samples of organic products - 1 sample of strawberries and 2 of cereals.

In 2005 the national, as well as EU MRLs exceedances were found in 25 (2%) samples of fruit and vegetables: spinach (10 samples), lettuce (3 samples), carrot (4 samples), tomato (1 sample), strawberry (2 samples), black currants (1 sample), grapes (1 sample), banana (1 sample), peach and nectarine (2 sample). The majority of samples with exceeding MRLs were of domestic origin.

The following pesticides were found above MRLs: maneb group, benomyl group, endosulfan, thiabendazole, chlorpyrifos, and captan.

Administration action was undertaken for 19 samples of food commodities where the MRLs were exceeded. For 15 samples of the above mentioned, risk assessment was provided and they have not been qualified for RASFF since they did not pose concern for human health. The remaining 6 samples were not prohibited after taking into account uncertainty of results.

Independently, for the purpose of the follow-up official control of food commodities of plant origin, 24 samples were checked including 52% domestic samples. In 86% of these samples no residues were found. The residues at or below of MRLs were recorded in 5 samples of fruit and vegetables and in 1 sample of cereals. No MRLs exceedances were noted in the follow-up surveillance samples.

## 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

Monitoring of pesticides in food commodities of food of plant origin is organised and supervised by the State Sanitary Inspection (SSI), which belongs to the Ministry of Health. However, monitoring plans are developed by experts from the National Institute of Hygiene (Państwowy Zakład Higieny PZH), also belonging to the Ministry of Health, and forwarded to the SSI, which authorises and distributes these plans to the Voivodship Sanitary Epidemiological Stations (VSES).

Monitoring plans in 2005 took into account the co-ordinated EU monitoring according to the Commission recommendation and national monitoring as well as the official control of food for compliance with MRLs. In this last case the specific conditions of Polish agriculture and consumption data as well as product for infants and babies as a group of special susceptibility for pesticide residues were also considered.

The integrated laboratories in 16 VSES (with branches) participated in the monitoring and official control. Specially trained staff of Poviats Sanitary Epidemiological Stations sampled food

commodities according to the guidelines published in the Regulation of the Minister of Health (*Regulation of the Minister of Health of 20 April 2004 on sampling of food for the analysis of the residues of plant protection products (Polish O.J. 2004, No. 86, item 810)*), which implemented the provisions of *Commission Directive 2002/63/EC*.

### 3. QUALITY ASSURANCE

16 integrated regional laboratories (with branches) of SSI participate in monitoring and official food control. 15 laboratories were accredited according to *PN-EN ISO/IEC 17025:2001+Ap.1:2003 (data updated on the 31-st December 2005)*; the certificates were issued by the Polish Accreditation Centre. The scope of accreditation covers different number of pesticides residues that laboratories are able to analyse in food.

For the determination of residues of pesticides belonging to different chemical groups the analytical methods published in standards *PN-EN 12393:2000*, *PN-EN 12396:2002*, *PN-EN 13191:2002* and in house methods for benomyl and thiabendazole groups published in Polish language in the *Wydawnictwa Metodyczne PZH: 2002* were used. In the case of products for infants and babies the following methods were used: methods published in the above mentioned standards, standards *PN-EN 15285:2000* and in house methods (published in Polish language in the *Wydawnictwa Metodyczne PZH:1996*).

All laboratories participating in monitoring and official food control of pesticide residues in food apply internal control of results and some of them participate in proficiency trials. In 2005 14 laboratories participated in proficiency trials organized by the National Institute of Hygiene in Warsaw, which was appointed by the Ministry of Health as the reference laboratory for VSES.

All methods used by VSES laboratories have been validated. Validation parameters include also uncertainty. Validation process underwent according to guidelines published in a handbook: "*Validation of analytical methods and assessment of uncertainty of results in analysis of chemical contaminants in food*" (in Polish) developed by the experts from the National Institute of Hygiene on the basis of document DG SANCO "Quality control Procedures for Pesticide Residues Analysis", 2000 and published in the *Wydawnictwa Metodyczne PZH: 2003*.

### 4. OTHER INFORMATION

The monitoring of pesticides residues and official control of food in this scope are performed in Poland in order to check compliance with the MRLs as set in the Regulation of the Minister of Health of 16 April 2004 on maximum residue levels of chemical plant protection products that may be present in food commodities or on their surface (*Polish O.J. 2004, No 85, item 801, as amended*). This regulation implements all EU MRLs and additionally includes national MRLs for some of those active substances for which no EU MRLs have been set. For the sake of reporting of the monitoring results the 'reporting levels' have not been established, therefore all results were based on the quantification limit of the analytical method.

## PORTUGAL

### 1. SUMMARY OF RESULTS

In 2005, a total of 909 samples were analysed for residues of up to 137 pesticides and two groups (benomyl and maneb), amounting to 160 analytes. This number of samples comprised 779 fruits and vegetables, 73 cereals, 20 baby foods and 37 other processed products, specially wines and tomato products. The numbers above include three fruit and vegetables and two cereal follow up enforcement samples. The majority of the samples of fruits and vegetables were analysed in the framework of the EU co-ordinated monitoring programme. Residues of at least one of the pesticides sought were found in about 30% of the fresh products sampled (fruits, vegetables and cereals) and multiple residues occurred in 94 samples of the same products (about 10 % of the sampling). In considering these figures, it should be noted, though, that in about 30% of these samples the scope of the control was limited to 15 to 25 compounds and one or two groups, respectively benomyl and maneb. In total, 54 different pesticide residues have been found in fruits and vegetables. The most frequent residue detected was, by far, the maneb group (12,6% occurrences), which was followed by imazalil and benomyl group. The most frequent insecticide detected, fenthion, appeared at the 8<sup>th</sup> position, with 2,1% occurrences. Infringements to EC MRLs and national MRLs (including EC MRLs) were reported for 2,4% and 2,7%, respectively, of the fruits and vegetables sampled. The most frequent pesticides detected in cereals were, by decreasing order, malathion, deltamethrin and pirimiphos-methyl. Opposite to fruits and vegetables, where the rate of infringements was significantly lower than in previous years, violations of EC and national MRL in cereals amounted to 7% of the sampling. These violations concerned deltamethrin in rice and diazinon in wheat. No residues of any of the pesticides sought occurred in the 20 samples of baby food analysed. Other processed foods analysed, total of 37 samples, showed no residues or very low residues compared to the MRLs set for the raw commodities.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

Until December 2005, the responsibility of monitoring programmes and sampling continued to lie in the Ministry of Agriculture, except for baby foods, for which the Ministry of Health is responsible. For products of vegetable origin the responsible bodies were Directorate General of Food Quality Control and Inspection (DGFCQA) and Directorate General for Crop Protection (DGPC), with the cooperation of regional agricultural services. Sampling was carried out by DGFCQA and regional trained inspectors over the country, which acted under DGFCQA coordination. In the autonomic region of Madeira sampling was carried out by the Regional Inspectorate of Economical Activities (IRAE). The autonomic region of Açores also participated in the programme, with sampling carried out by Institute for Food and Agricultural Markets (IAMA) and shipment of samples to DGPC laboratory. Results obtained for products sampled in the mainland were reported by the laboratories to DGFCQA and, in case of violations, risk assessment was carried out by DGPC. DGFCQA is also the body responsible for enforcement actions, such as official warnings, levying of fines or preparation of prosecutions by the court, according to the severity of infringements. In the autonomic regions, Madeira IRAE and Açores IRAE are responsible for these actions.

Monitoring programmes are elaborated in a meeting with the participation of representatives of the intervening bodies (sampling inspectors and analysts). The national programme for 2005 was based on the EU coordinated monitoring programme, which was extended to other pesticides according to the capabilities of the laboratories and to other commodities, mainly strawberries and lettuce, as a result of infringements found in previous years. Sampling was carried out by trained officers,

according to the procedures laid down in directive 2002/63/EC. In the mainland samples were taken both at wholesale commerce, wholesaler's warehouses and retail. In Madeira and Açores most samples were taken at retail.

### **3. QUALITY ASSURANCE**

The following pesticide residue laboratories contributed to the national monitoring programme: DGPC (lab 1) Regional Agricultural Directorate of Madeira (lab 2), Regional Agricultural Directorate of Entre Douro e Minho (lab 3), and Regional Agricultural Directorate of Algarve (lab 4). Following official audit in December 2004, Lab 1 was accredited for the majority of compounds analysed by GC-multi-residue method (GC-MRM) and for N-methylcarbamates. By the end of 2005, work for accreditation of the dithiocarbamates method was in the final stage in lab 4. Implementation of EU quality control procedures is ongoing in the three regional laboratories, as shown in Table G.

The GC multi-residue methods used are method P (labs 1 and 3) and method M (labs 2 and 4) in European Standard 12393. The determination of maneb group is based in European Standards 12396-1 (labs 1, 3 and 4) and 12396-2 (lab 2). The benomyl group is determined by HPLC after ethyl acetate extraction and acid-base partition (labs 1 and 4). N-methylcarbamates are determined by HPLC-FLD with on-line OPA derivatization, after extraction and clean-up identical to method P above (lab 1). Organophosphorus insecticides precursors of sulphoxides and sulphones are analysed by oxidation of the cleaned extract obtained according to method P (lab 1). In 2005 labs 1 and 2 have both participated in the 7th EU proficiency test. Lab 1 also participated in one FAPAS exercise (MRM) and lab 4 participated in TESTQUAL exercises for dithiocarbamates and MRM.

All values detected above MRL (mean of at least 2 separate analyses) are reported as infringements. However, administrative actions are taken only in those cases where the levels found exceed generally 1,4 x MRL. For values up to 1,4 x MRL action taken consists of official warnings. This figure is based on the estimation of a maximum standard uncertainty of 20% for a number of pesticides and application of a coverage factor of 2 for a confidence level of 95%, and it is subject to generally small variations derived from routine quality control data.

### **4. OTHER INFORMATION**

In 2005 the homogeneity exercise was not carried out.

For all samples where infringements found related to pesticides for which an acute reference dose, ARfD, has been set or with relevant acute toxicity, risk assessment was carried out both for adults and children. In three samples of rice with violative residues of deltamethrin the respective acute reference dose was exceeded for children when the UK model was used.

## 1. SUMMARY OF RESULTS

In the Republic of Slovenia in 2005 totally 1018 samples of foodstuffs were analysed out of it 990 surveillance samples and 28 follow-up enforcement samples.

Surveillance samples included: 762 samples of vegetables and fruit, 32 samples of cereals, 135 samples of processed products of plant and fruit base and 61 samples of baby food. Totally, 538 samples were without detectable residue, 422 samples with residues at or below MRL (national or EC) or for which no MRL is set, 30 samples with residues exceeding the MRL (national or EC) out of it 14 samples with residues exceeding EC-MRLs. Follow up was carried out always when feasible.

Follow-up enforcement samples include 19 samples of vegetables and fruit, 1 sample of cereals and 8 samples of processed food. Totally, 21 samples were without detectable residues, 6 samples were with residues at or below MRL (national or EC) and 1 sample of vegetables and fruit was with residues exceeding the MRL (national).

Samples of fresh and frozen fruit and vegetables were analysed on presence of up to 105 pesticides, residues of 71 pesticides were detectable (67,6%). Most frequently detectable residues were Maneb-grup > Procymidone > Iprodione > Cyprodinil > Benomyl-group > Thiametoxam > Carbaryl > Fludioxonil > Thiabendazol. Samples of cereals were analysed on presence of up to 106 pesticides and where residues of 8 pesticides were detectable (7.5%). Most frequently detectable residues were Carbaryl > Bitertanol > Iprodion > Pirimiphos - methyl > Azoksytrobin > Malathion > Pyrethrins. Samples of baby food were analysed on presence of up to 124 pesticides (or a group of some of them) and no residues were not detectable.

Samples of processed food were analysed on presence of up to 114 pesticides. 89 samples were without detectable residues, 39 samples with residue at or below MRL (national or EC) or for which no MRL is set and 10 samples with residues exceeding the MRL (EC).

In sample of apples, cherries, pears, oranges, mandarins, nectarines, plums, lettuce carrots, spinach, cucumbers, cereals, strawberries, tomatoes, pepper, lemon grapes, bananas and grapefruit more than one residue of analysed pesticides was detectable.

Samples of pears, oranges, carrots and potatoes were taken for homogeneity test. Factor for the homogeneity of the sample were between 1.00 and 5.15.

Calculated value for PSTI was in one sample of pears exceeding the ARfD for toddlers, therefore RASFF notification was issued.

## 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

### Responsibilities

Organization of pesticide residues monitoring is responsibility of the Ministry of Health (MH). Its Sector for Food Safety within the Directorate for Public Health is responsible for the preparation of pesticide residues monitoring programme for foodstuffs. Health Inspectorate of the Republic of Slovenia (HIRS), which operates as a constituent body of the MH, is responsible for the implementation of the monitoring programme.

### Designing of Programmes (priorities, targeting)

Various reference sources were used to determine which commodities are due to be surveyed: information on commodities surveyed during last 5 years; results of national programme carried out in the previous year; data of Statistical Office of the Republic of Slovenia on average annual quantity of purchased food and beverages per household member; GEMS/food regional diets; European Commission Rapid Alert System for Food and Feed (RASFF); Commission

Recommendations concerning a coordinated Community monitoring programme; a summary of the European Monitoring programme for pesticides residues.

The number of samples per survey in general varied between 5-30 samples, a special survey of 300 samples on was made on lettuce. The current programme is designed to generate information on typical residue profiles of particular types of produce and on major trends in the incidence and levels of pesticides.

The choice of pesticides to be sought is primarily influenced by pesticide use; potential for residues based on use pattern and the physical-chemical properties of the pesticide; toxicological profile of the pesticide; analytical capabilities.

#### Sampling: personnel, procedures, sampling points

Monitoring of PR was designed with participation of stakeholders with interest in PR: official control bodies, administrative bodies, risk assessors, consumers, analytics. Pesticide residue monitoring in foodstuffs was developing during past five years with the aim of sampling in the following areas: dietary staples, rolling programme, EU surveys, follow-up surveys, additional surveys.

#### Enforcement action

According to the provisions of the Inspection Act, Health Inspection Act and Act Regulating the Sanitary Suitability of Foodstuffs, Products and Materials Coming in Contact with Foodstuffs HIRS have the following possible administrative measures to be taken in the cases of detected infringements: warning, temporarily prohibiting the production and trade, ordering the removal of deficiencies in the production and trade, prohibiting the production and trade, ordering the foodstuffs, which endanger public health to be seized and safely destroyed at the expense of the legal or natural person at whose premises such product were discovered, sealing establishments, work areas, equipment and items, or prohibiting the use of an establishment, working areas, equipment and items in case the determined deficiencies representing a threat to sanitary suitability/safety of foodstuffs, thus endangering human health. According to the Offence Act the health inspectors are since the beginning of the year 2005 also pronouncing offences. Possible options, related to the level of offence are: verbal warning, order to pay; written order.

### **3. QUALITY ASSURANCE**

#### Status of accreditation of laboratories; number of laboratories

There are two institutes nominated for performing the analyses of samples of foodstuffs taken within monitoring programs - National Public Health Institute, Trubarjeva 2, 1000 Ljubljana, and Regional Public Health Institute of Maribor, Prvomajska ulica 1, 2000 Maribor.

Both laboratories are accredited: National Public Health Institute, achieved accreditation 22<sup>nd</sup> of August 2003, Public Health Institute of Maribor, 14<sup>th</sup> of November 2001.

#### Analytical method used

Samples are analysed using analytical methods, mainly multiresidue methods based on gas and liquid chromatographic techniques employing element-selective and mass-spectrometric detection systems (with increasing share of LC/MS and LC/LS/MS/MS), which have been validated according to the procedures described in the Guidelines for Residues Monitoring in the European Union and comply with the relevant minimum required performance criteria of the EU Recommendations and Quality Control documents.

#### Participation in proficiency test

Both laboratories participate in few proficiency testing scheme, basic ones are FAPAS and EUPT-6 (European Commission's Proficiency Test on Pesticide Residues in Fruit Vegetables), within both of them, 5 – 8 different testings per year.

#### Implementation of EU quality control procedure

With respect to quality of data generated in the frame of the EU residue coordinated programme, the results of the analysis of foodstuffs are obtained by analytical laboratories which are in compliance with the requirements of Directive 1993/99/EEC, its Article 3, applying Quality Control

Procedures for Pesticide Residue Analysis and accredited according to the ISO17025. The pesticide analyses of foodstuffs are performed following entirely the guidelines of Quality control procedures for pesticide residues analysis, Document N° SANCO/10476/2003, 5/February/2004 (and updating issues). Others documents are taking in to account, for example, EURACHEM/CITAC Guide Quantifying Uncertainty in Analytical Measurement, QUAM: 2000.P1, Second Edition: QUAM: 2000.P1.

#### Analytical uncertainty

Residue analytical results have a relatively large inherent uncertainty depending on the concentration of the analyte (the relative uncertainty of analysis, realistically expected, is to be around 25% in the residue range of 0.001-10 mg/kg). These results are in agreement with Horwitz's findings indicating a relative uncertainty of about 17% for average residues of 0.1 mg/kg (Horwitz *et al.*, 2001). According to the Commission Directive 2002/63/EC analytical *the result x is stated together with the expanded uncertainty U calculated using a coverage factor Quantifying Uncertainty Reporting Uncertainty QUAM:2000.P1 (which gives a level of confidence of approximately 95%)*.

## **4. OTHER INFORMATION**

#### Details on reporting level

In the table A2 Part II - Fruits and vegetables in the 5th column two reporting levels are indicated for certain pesticide residues due to different reporting level which were used for matrix concerned.

#### Details of homogeneity exercise

Homogeneity exercise was performed following entirely the guidelines of Quality control procedures for pesticide residues analysis, Document N° SANCO/10476/2003, 5/February/2004 (and updating issues) and Commission Directive 2002/63/EC establishing Community methods of sampling for the official control of pesticide residues in and on products of plant and animal origin and repealing Directive 79/700/EEC.

#### Details of risk assessment

To assess acute risk, we use the UK Consumer Exposure model for determination of short time exposure using high portion consumption (97,5 percentile of food consumption) for toddlers and adult persons, the observed residue in the sample and variability factor (depending of the weight of the commodity unit). The pesticide intake/exposure is compared to the ARfD or ADI when ARfD is not determined (DG SANCO/3346/2001 recommended methodology for the evaluation of the risk to the consumers).

For chronic risk assessment we use the data of average food consumption /day/person (data from Slovene Statistic Office – Yearly Household Budget Survey or data from WHO European Diet when there is a lack of Slovene data for a specific commodity) and average pesticide residue (sometimes 90,0 percentile), compared to the ADI (%ADI).

## 1. RESULTS SUMMARY

In 2005, pesticide residue control was performed under the National Pesticide Residue Control Plan in samples of plant origin, implementing Commission Recommendation concerning the EU monitoring for 2005. The total number of samples was 1,074; thereof 915 fruits and vegetables, 55 cereals, 90 selected processed foodstuffs and 14 baby foods (Table 1). The national or EU harmonized Maximum Residue Limit (national or EC-MRL) was exceeded in 60 fruit and vegetable samples (6.5%) and 1 cereal sample (1.8%).

Table 1: Results of Pesticide Residue Control in the Slovak Republic

| Samples              | Analyzed | Without residues | With residues <MRL | With residues >MRL | With residues >MRL (%) | Country of origin *   |      |       |      |                 |      |
|----------------------|----------|------------------|--------------------|--------------------|------------------------|---|------|-------|------|-----------------|------|
|                      |          |                  |                    |                    |                        | SK  |      | EU    |      | Third countries |      |
|                      |          |                  |                    |                    |                        | Total   | >MRL | Total | >MRL | Total           | >MRL |
| Fruit and vegetable  | 915      | 510              | 345                | 60                 | 6.5                    | 346   | 9    | 409   | 42   | 129             | 9    |
| Cereal               | 55       | 46               | 8                  | 1                  | 1.8                    | 32  | 1    | 18    | 0    | 1               | 0    |
| Processed foodstuffs | 90       | 75               | 15                 | 0                  | 0                      | The country of origin was not known in 35 samples of fruits, vegetables and cereal. |      |       |      |                 |      |
| Baby foods           | 14       | 14               | 0                  | 0                  | 0                      |   |      |       |      |                 |      |
| <b>Total</b>         | 1,074    | 645              | 368                | 61                 | 5.6                    |   |      |       |      |                 |      |

97 samples of pears, green beans, potatoes, carrots, oranges or tangerines, spinach, rice and cucumbers were analyzed in the frame of EU coordinated program, by exceeding of MRL in 3 samples (3.0%).

The testing laboratory also performed additional unit analysis in case of two samples, which results are documented in Table F (in Annex).

## 2. ORGANIZATION OF MONITORING PROGRAM AND SAMPLING

The State Veterinary and Food Administration is responsible for the organization and performance of pesticide residue monitoring program in the SR. For selection of commodity and number of samples, the criteria as consumption of specific commodity in Slovakia, observations from sample analyses in previous year, information from the RASFF were under consideration. Concerning commodity selection, we focussed on fresh fruit and vegetable. The number of taken samples was also limited by workplace analytical capacity. The coordinated EU program was a part of national program. In terms of its requirements, 16 samples of organic agricultural products were taken and analyzed. They applied to the samples of domestic origin that were taken directly at grower or processor sites.

The sampling procedure was in compliance with Commission Directive 2002/63/EC that has been implemented into the Slovak food legislation. The sampling was performed by trained inspectors from 40 District Veterinary and Food Administrations, mainly at grower sites (samples from domestic production), packing stations (nuts, dry fruits), wholesale warehouses, retail, as well as in control of food import from third countries at the point of entry. The sampling plan was elaborated for individual District Veterinary and Food Administrations, specifying the number of samples, commodities and sampling week. In case of exceeding MRL (after consideration of the measurement

uncertainty), the inspectors acted in compliance with the legislation of the SR. The samples of domestic origin exceeding MRL and taken from retails or wholesale warehouses were solved within the grower or processor concerned. Regarding samples from import (Intra-Community exchange or products from third countries), the investigation was conducted within the importer.

### 3. QUALITY ASSURANCE

The Reference Laboratory for Pesticide Residues in Foods, the State Veterinary and Food Institute in Bratislava was authorized to perform the analyses of samples. This laboratory analyzed the samples from all 40 districts. The general strategy for determination of the widest possible spectrum of pesticides by multiresidual method (MRM) was adopted. The principal of this method is extraction into ethylacetate followed by purification using gel permeation chromatography in ethylacetate/cyclohexane system. The detection is performed using GC/ECD, FPD and NPD. The positive findings are confirmed with MSD. For three fungicides (benomyl group, thiabendazol and imidacloprid), a modified method for measurement by HPLC/DAD/FLD system has been used. For determination of maneb group, a separate method of derivatization to CS<sub>2</sub>, followed by GC/ECD detection has been used. LC/MS/MS has been used for determination of aldicarb and methomyl. The quality assurance of analytical methods is in compliance with the requirements of SANCO/10476/2003 Document. The laboratory is accredited according to the standard STN EN ISO/IEC 17 025 by the Slovak National Accreditation Service (SNAS). The laboratory took part in FAPAS and EU proficiency tests.

## 1. Summary of results

In 2005, a total of 2055 samples of fruit, vegetables, cereals, processed products and baby foods were analysed for residues of 213 pesticides. 356 samples were of domestic origin, 843 from other EU countries and 856 from third countries. The total number of samples includes 85 enforcement samples collected as follow up of violations.

Pesticide residues above the reporting limits were found in 40 % of the surveillance samples. The frequency of samples with residues was 42 % for fruit and vegetables, 36 % for processed products and 32 % for cereals. Pesticide residues were found in 31 % of domestic samples, in 45 % of samples from other member states, and in 52 % of third country samples.

The maximum residue levels (MRLs) were exceeded in 84 surveillance samples (4.3 %), of which 44 were from third countries, 40 from other member states. No violations were found in domestic samples. The number of samples taken of organic products was 84, and three of them had detectable residues but they were below the MRLs. The enforcement samples targeted to suspected lots had a violation rate of 29 %.

## 2. Organisation of the monitoring programmes and sampling

### Responsibilities

The Finnish Food Safety Authority Evira (formerly National Food Agency) is the central competent authority for the planning and control of pesticide residues in food. The control of non-domestic food products has been assigned to Customs administration while municipalities and Evira are responsible for control of products on domestic markets.

### Design of the programmes (priorities, targeting)

The annual monitoring program is worked out in co-ordination under Evira providing a sampling plan for residue control of fruit and vegetables, cereals, processed products of plant origin, baby-food and organic products including the commodities and pesticides required in the EU co-ordinated programme. Control is designed to cover all important dietary commodities. The sampling frequency of different commodities is determined by the results of previous monitoring programs and priorities are also set according to known residue problems. A special survey of residues in breakfast cereals was carried out as part of Nordic co-operation.

### Sampling: personnel, procedures, sampling points

Domestic samples are collected by local health inspectors from wholesalers, packing companies, retail shops or farms according to the annual sampling plan and guide prepared by Evira. The samples of non-domestic food products are collected by customs inspectors from wholesalers. All inspectors have theoretical and practical training in sampling organised by Evira or the Customs. The sampling directive 2002/63/EEC is followed.

### Enforcement

Each confirmed exceeding of MRL resulted in either legal action or voluntary action by the operator. For surveillance samples exceeding the MRL, the holder of the product is requested to prevent further distribution and selling of the lot. On subsequent lots of the same origin, follow-up samples are taken (enforcement samples). In case of enforcement sampling, the lots are detained for the duration of the investigation, and lots confirmed to exceed the MRLs are to be destroyed. Under

certain conditions and by the permission of authorities, a non-complying lot may be returned to the seller or to a third country or rendered compliant to regulations (e.g. aeration to decrease the level of fumigant residues).

### **3. Quality assurance**

#### **Status of accreditation of laboratories; number of laboratories**

The analyses were carried out in two accredited (FINAS) laboratories: Finnish Customs Laboratory (87 % of samples) and Environmental Laboratory of the City of Helsinki (13 % of samples). Both laboratories have accreditation according to ISO17025 for the main analytical methods but some single residue methods are not accredited.

#### **Analytical methods used**

Virtually all samples were analysed by the multiresidue method. This method is based on acetone extraction of samples, partitioning with petroleum ether and dichloromethane and detection by GCEC, GCNP, GCMS and LCMSMS. The number of pesticides monitored for by the multiresidue method is 178 in the Customs laboratory and 72 in the Helsinki City laboratory. In addition, chlormequat, diquat, glyphosate, maleic hydrazide, inorganic bromides and hydrogen phosphide were analysed from selected samples in the Customs laboratory.

#### **Participation in proficiency tests**

Both laboratories participated in the proficiency test organised by EU (PT7). Customs laboratory participated also in five proficiency tests of pesticide residues organised by FAPAS and one organised by the NFA Sweden.

#### **Implementation of EU quality control procedures**

The EU quality control guidelines (SANCO/10476/2003) have been implemented in both laboratories, albeit only partly for some elements.

#### **Analytical uncertainty**

The analytical uncertainty is determined from the daily quality control samples. Two times relative standard deviation is used as the measure of uncertainty. All results even slightly above the MRLs are defined in this report as exceedances, but enforcement action is taken only if the result, subtracted by the uncertainty value, still exceeds the MRL.

### **4. Other information**

#### **Details of homogeneity exercise**

Twelve samples with residues of pesticides posing an acute risk, were subjected to analysis of individual units of the second laboratory sample. The samples were taken from lots where the grower could not be identified and they may have been mixed lots.

#### **RASFF notifications**

No RASFF notifications were sent in 2005 following the results of pesticide residue monitoring. The exceedances of MRLs posed no acute health risk for consumers.

## SWEDEN

### 1. SUMMARY OF RESULTS

In 2005, a total of 2 096 surveillance samples of fruits, vegetables, juices, fruit drinks, cereal grains, cereal products and vegetable oils were analysed for residues of 251 pesticides (296 analytes). National or EU harmonised Maximum Residue Limits (EC-MRLs) were exceeded by 89 samples (4.2 %). Only one of the samples of domestic origin contained residues above the MRLs.

Pears, beans, potatoes, carrots, oranges, mandarins, spinach, rice and cucumber, in all 529 samples, were analysed in the 2005 EU co-ordinated programme. Twenty of these samples exceeded the EC-MRLs for the pesticides that were included in the co-ordinated programme.

A total of 247 samples of cereal grains were analysed. Most of the samples contained no residues and none of the samples exceeded any MRLs.

No residues were found in the 95 samples of foods for infants and young children.

In the enforcement sampling 68 samples of fruits and vegetables were collected and 13 lots (28.2 tons) were prohibited from being sold.

The short-term intake was estimated for all pesticides with an acute reference dose (ARfD) set by EU, WHO or US-EPA. The calculation was based on the residue found in a surveillance (composite) sample and UK consumption data. The ARfD was exceeded for toddlers in six cases.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

#### RESPONSIBILITIES

The National Food Administration (NFA) is the responsible authority for the monitoring of pesticide residues in foods.

#### DESIGN OF THE PROGRAMMES (PRIORITIES, TARGETING, CRITERIA FOR THE PERCENTAGE OF SAMPLES TO BE TAKEN FROM THE ORGANIC SECTOR)

The number of samples to be collected of each food is risk related and partly linked to the food's consumption rate and takes into account both the amount of domestic production and the amount of imports from EU-countries and third countries. However, the number is also based on the importance of the foodstuff in the diets of infants and young children as well as residues found in prior samples. The number of samples from the organic sector was roughly dependent on its share of the market and availability on the market.

#### SAMPLING: PERSONNEL, PROCEDURES, SAMPLING POINTS

Samples collected in accordance with the monitoring programme were defined as surveillance samples i.e. there were no suspicions about excessive amounts of pesticide residues in the lots prior to sampling

*Personnel:* Plant inspectors from the National Board of Agriculture collected most of the samples in accordance with instructions from NFA.

*Procedures:* The sampling was done according to Commission Directive 2002/63/EEC. Each sample was sealed and labelled with a unique sample identity.

*Sampling points:* Fresh fruit and vegetables were sampled at wholesalers' warehouses in the first trade channel. The imported cereal grains were sampled at the port where the shipment was discharged. Samples of domestic produced cereal grains were collected at the milling plants. Most of the samples of processed or frozen fruit and vegetables, juices, fruit drinks, rice, cereal products and vegetable oils were collected in retail shops or department stores.

## ENFORCEMENT ACTION

When a surveillance sample contained a pesticide residue above the national or EC-MRL, the National Food Administration prescribed a condition for the offering for sale or other handling of the food or lot to which the food belonged. The remaining part of the lot, if any, was prohibited for being put on the market. As a follow-up, next lots of the commodity from the grower/exporter were detained and enforcement samples were collected.

### 3. QUALITY ASSURANCE

#### STATUS OF ACCREDITATION OF LABORATORIES; NUMBER OF LABORATORIES

All analyses were carried out at AnalyCen Nordic AB, Lidköping. The laboratory changed name to Lantmännen Analycen AB 4 April, 2006. The laboratory is accredited by the Swedish accreditation authority SWEDAC for all analytical methods used for the NFA's official control of pesticide residues in food of plant origin.

#### ANALYTICAL METHODS USED

Practically all samples of fruit and vegetables were analysed by the multi-residue method. By this method, the samples were extracted with ethyl acetate after addition of sodium hydrogen carbonate. GPC clean up was only used for extracts prior to GC determination. The uncleaned extracts were determined by LC-MS/MS.

In all, by using both multi-residue methods and single residue methods it was possible to determine 251 pesticides corresponding to 296 analytes. About 368 000 residues (pesticide/commodity combinations) were sought (Table A2-Part I-II).

#### PARTICIPATION IN PROFICIENCY TESTS

THE LABORATORY HAS PARTICIPATED IN ONE PROFICIENCY TEST ORGANISED BY EU AND IN 23 TESTS ORGANISED BY FAPAS, UK, NFA, SWEDEN, DFVF, DENMARK AND MUVA, GERMANY (TABLE G).

#### IMPLEMENTATION OF EU QUALITY CONTROL PROCEDURES

The EC guidelines SANCO/10476/2003 "Quality Control Procedures for Pesticide Residue Analysis", third edition, 2003 have been implemented as far as practicable (Table G).

#### ANALYTICAL UNCERTAINTY

THE RESIDUE FIGURES FOUND ARE COMPARED WITH THE MRLS. IF THE FIGURES, WITHOUT ANY CORRECTION, ARE MATHEMATICALLY ABOVE THE MRL, THE SAMPLE IS DEFINED AS AN EXCEEDING. HOWEVER, BEFORE ANY ENFORCEMENT ACTIONS ARE TAKEN THE ANALYTICAL UNCERTAINTY IS SUBTRACTED FROM THE MEASURED VALUE (95 PERCENT CONFIDENCE INTERVAL). IF THE CORRECTED FIGURE STILL EXCEEDS THE MRL ENFORCEMENT ACTIONS COULD BE TAKEN.

### 4. OTHER INFORMATION

#### DETAILS OF HOMOGENEITY EXERCISE

In the EU co-ordinated programme the homogeneity factor should be determined for at least certain pesticide/commodity combinations. None of these combinations were found or could be reanalysed. However, the homogeneity factor was calculated for dimethoate and omethoate in lettuce (Table F).

#### BACKGROUND ON LEGISLATION

Sweden has implemented all EC-MRLs. For some other pesticide/commodity combinations National limits are in force.

#### DETAILS OF RISK ASSESSMENT

The short-term intake has been calculated for those pesticides that have an ARfD assigned by EU, WHO or US-EPA. In six cases the ARfD was exceed. Furthermore, the chronic intake was estimated using Swedish monitoring data from 2001-2005. Of the 22 pesticides included in the

calculations dicofol reached the highest intake, 11.9 % of the ADI when the most conservative estimate was applied.

## UNITED KINGDOM

### 1. SUMMARY OF RESULTS

In 2005 a total of 3787 samples were analysed for up to 159 pesticide residues. This was composed of 1956 fruit and vegetables, 897 animal products, 622 miscellaneous, 240 baby food, and 72 cereal samples.

This includes the samples analysed under the co-ordinated EU programme and the national monitoring programme. Samples of oranges, pears, spinach, cucumber, carrots, potatoes, green beans and rice were analysed for a range of pesticides indicated in the recommendation.

In the overall programme 65 (1.7%) of the samples contained residues exceeding the MRL. All of these exceedances were in the fruit and vegetable category with the greatest proportion in the green beans and exotic fruit surveys. In all cases where MRLs were exceeded, or where there were potential intake issues, a separate consumer risk assessment was carried out to establish whether the level of residues present would represent a risk to human health. The risk assessments indicated that none of the residues found were of concern for human health.

51.7% of the samples were UK origin, 21.5% were from other member states, and 24.8% from third countries.

14.5% of samples contained residues of more than one pesticide.

Residues of conventional pesticides below the MRL were found in 2 samples of vegetables labelled as organic.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

#### Responsibilities

The UK monitoring programme is overseen by the independent Pesticide Residues Committee (PRC). The purpose of the UK monitoring is to:

- back up the statutory approvals process for pesticides by checking that no unexpected residues are occurring;
  - check that residues do not exceed statutory EU and UK MRLs and
  - check that human dietary intakes of residues are at acceptable levels.
- 
- Design of Programmes

The food chosen to include in our surveys takes account of:

- foods covered by the EU programme of surveys;
- the importance of the food within UK diet;
- evidence of residues in earlier surveys or other information;
- information on use of pesticides;
- the time that has passed since they were last tested;
- the balance of food types;
- cost.

6% of samples were labelled as organic.

86 % of samples were obtained at retail level from two towns/cities in each of twelve regions of the UK. The places selected are changed each year. Official inspectors from Department for Environment, Food and Rural Affairs (Defra) also collected samples at non-retail sources; for carrots, grapes, potatoes and pears.

Samples were taken, prepared and analysed according to Commission Directive 2002/63/EC.

#### **Follow-up action**

When the level of pesticide residues found indicated use of a non-approved pesticide, or the use of a pesticide not in accordance with the conditions of its approval, the source of the sample was investigated to establish the cause of the residues. Further targeted surveillance is carried out to check whether the result represented an isolated incident.

Where there are persistent or re-occurring problems, then a separate enforcement programme can be undertaken. Samples are collected by enforcement officers and follow-up action is taken where any offences have occurred. This may involve legal action and a directed information campaign to assist growers in correct agricultural practice.

If problems are identified in retail samples of produce of EU or third country origin, the details are reported to the Member State or exporting country concerned.

### **3. QUALITY ASSURANCE**

Analytical methods comply with the 3rd edition (SANCO/10476/2003) of the EU Quality Control Procedures for Pesticide Residues Analysis (Guidelines for Residues Monitoring in the European Union).

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.

All laboratories carrying out work have taken part in proficiency testing exercises, including European Union Proficiency Testing (EU PT). Defra undertakes its own proficiency testing programme known as the Food Analysis Performance Assessment Scheme (FAPAS). All of the laboratories used by the PRC participate in FAPAS. 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). Methods are validated in accordance with ISO 5725:1995 or IUPAC harmonised guidelines, as appropriate.

### **4. OTHER INFORMATION**

In 2005, the UK did not carry out the homogeneity exercise.

The results for grapes were reported to PSD monthly and published on the PRC website.

## NORWAY

### 1. SUMMARY OF RESULTS

A total of 1788 samples of fresh and frozen fruit, vegetables and cereals, both imported (67 %) and domestically produced (33 %) were analysed in the monitoring programme for 2005. In addition, the monitoring programme included 200 samples of processed products (148) and baby food (52). The processed products were mainly tomato based like ketchup, pasta-/salsa sauces and tomato purée. Eighteen follow-up samples were taken in 2005, sixteen samples from Thailand and one sample each from Egypt and Kenya. Fourteen of the follow-up samples had pesticide residues. Of these, seven samples had pesticide residues exceeding EC-MRLs. All of these samples were from Thailand. A total of 116 different commodities of fresh and frozen fruit, vegetables and cereals were analysed. The monitoring programme covered 233 pesticides including some isomers and breakdown products. Of these, 93 compounds were found.

Analysis of fresh fruit and vegetables, including potatoes, showed that 58 % were without detectable pesticide residues. The EC-MRLs were exceeded in 3.2 % of the samples. Among the domestic products four samples had pesticide residues exceeding the EC-MRLs, two samples of celery and one sample each of oat and carrot. Among the imported products (50 samples) beans with pod, lettuce, celery and peas with pod had the highest number of samples exceeding the EC-MRLs.

Analysis of 52 samples of baby food showed that non sample had detectable pesticide residues.

A total of 110 samples of cereals were analysed. Of these, 78 (72 %) had no detectable pesticide residues. One sample of domestic oat exceeded the MRL for the growthregulator chlormequat. A total of 32 samples were marked and sold as organic grown products. Non of the samples had detectable pesticide residues.

Three samples, all beans with pod from Thailand, contained residues of pesticides (omethoate/dimethoate and EPN) in amounts that were considered to represent health risk. The findings were reported via the Rapid Alert System for Food and Feed.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

The samples were mainly taken at wholesaler's warehouse in different parts of Norway. Some samples were taken at retailers, farms or at marked places. The number of samples of each commodity does not reflect their share of the market as more samples were taken of commodities suspected to contain residues. Trained inspectors from the food control authorities were responsible for taking samples in accordance with a national sample plan and official guidelines for sampling.

Eighteen follow-up samples were taken in 2005. The consignments were suspended until the results of the analysis were available. Seven of the samples were not released.

### 3. QUALITY ASSURANCE

The Norwegian Crop Research Institute, Pesticide Laboratory, was responsible for the analyses. The Pesticide Laboratory was accredited on April 1st 1997. The accreditation is according to NS-EN ISO/IEC 17025. The analyses were carried out in accordance with the Quality Assurance Manual of the Pesticide Laboratory and the EU Guidelines for Pesticide Residues Analysis. As a part of the quality assurance programme the laboratory regularly participates in international intercalibrations / proficiency tests.

All samples of fruit and vegetables were analysed using a GC multi-residue method based on extraction with acetone (Luke method). The method covers 166 pesticides including isomers and breakdown products. A new LC-MS/MS multi-residue method was developed and used from 28<sup>th</sup> of October. The new method covers 73 pesticides using the same extraction method. LC-MS/MS was also used for analysis of diquat. Chlormequat/mepiquat and propamocarb were determined using LC-MS. Biphenyl, orto-phenylphenol, progargite and isoproturon were analysed using GC-MS. Phenoxyacid herbicides and glyphosate/AMPA were analysed by GC-MS after derivatization. The dithiocarbamates were determined with a spectrophotometric method after distillation of CS<sub>2</sub>. The reporting levels are the limit of quantification for all methods.

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 or LC-MS/(MS).

The pesticide residues figures found are compared with the MRLs. If the results are above the MRL, the sample is defined as an exceeding. However, before any enforcement action is taken, the analytical uncertainty (95 % confidence interval) is subtracted from the measured value. If this figure still exceeds the MRL, enforcement action could be taken.

## ICELAND

### 1. SUMMARY OF RESULTS

This report summarizes the results of the monitoring of pesticide residues in fruits, vegetables and cereals in Iceland. A total of 315 samples were taken in the year 2005, approximately 25% of domestic commodities, 75% of imported commodities, there of 28% imported commodities from third countries. 290 samples were taken of fresh fruits and vegetables, 10 samples of processed foods and 15 samples of cereals.

54% of the samples of fruit and vegetables were without detectable residues, 44% were with residues at or below MRL and 2% had residues, which exceeded the MRL. No residues were detected in the samples of processed foods. 87% of the samples of cereals were without detectable residues and 13% were with residues at or below MRL and 0% had residues, which exceeded the MRL. Commodities originated from 24 countries, most of them came from The Netherlands and Spain.

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.

Iceland only participated partly in the co-ordinated program.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

- Responsibilities and design of programmes: Sampling plan is made every year based on information on import volumes and domestic production. Experience is also taken into account, as to what pesticide residues are most often analysed in a particular product.
- Sampling: Samples were collected according to national regulation no 736/2003 on sampling methods for contaminants in foodstuffs which is based on EC directives. The samples are taken at wholesaler's warehouses in Reykjavik. The Environmental health and protection office in Reykjavik collects the samples. Chemical analysis of fruit and vegetables was done at the laboratory of the Environment and food agency of Iceland. Analysis of cereals was done at **AnalyCen Nordic** laboratory in Sweden. The most frequently found residues were: imazalil, thiabendazol, orthophenylphenol, chlorpyrifos, malathion, biphenyl, tolyfluanid, prochloraz, chlorotalonil, chlorfenvinphos, iprodione and bromopropylate.
- Enforcement action: Actions are taken if the pesticide residues are over MRL plus deviation.

### 3. QUALITY ASSURANCE

- Status of accreditation of laboratories and number of laboratories: Two laboratories were used. The Laboratory of the Environment and food agency of Iceland, which was not accredited in 2005 and **AnalyCen Nordic**, which is accredited.
- Analytical methods used: Extraction with organic solvents followed by GC-MS analysis.
- Participation in proficiency tests: The laboratory of the Environmental and Food Agency takes part in FAPAS.
- Implementation of EU quality control procedures: The following parts of EU quality control procedures were followed: Sampling, transport, processing and storage of samples, pesticide standards, calibration, solution, extraction and concentration, contamination and inference, analytical calibration and chromatographic integration, analytical methods and analytical

performance and proficiency testing and analysis of reference material. Analytical uncertainty estimation is applied on results.

### **1. Summary of Results**

This report summarizes the results of the co-ordinated and the national pesticide monitoring programme in fruits, vegetables and cereals on the Liechtenstein market in 2005. This report has been prepared according to the recommendation of the ESA as far as technically possible.

A total of 33 samples were analysed out of which 6 fruits, 22 vegetables and 5 cereals.

In 33 samples no pesticide residues were detected. 10 samples showed pesticide contamination below national and EC MRLs.

The total number of residues analyzed was 55, sought for in 8 samples, 54 residues in 5 samples and 53 residues in 25 samples. Residues of 6 different pesticides were found, 1 belonging to the group of growth regulators in cereals and residues of 5 different pesticides in vegetables and fruits, belonging to the categories of fungicides (2), insecticides (2) and herbicides (1).

Summary of results:

|   | 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 and national programme, vegetables and cereals | 33                            | 23                              | 10   | -                            | -                           |

### **2. Organisation of Monitoring Programme and Sampling**

The samples were collected according to the annual sampling plan prepared by the Office of Food Inspection and Veterinary Affairs. The sampling plan is based on the domestic production situation in the agricultural sector and on the ESA co-ordinated monitoring programme. The programme started in spring 2005.

Samples of fresh fruits, vegetables and cereals were collected mostly from retailers (20), but also from farm gates (8) and food processing plants (5). Samples were taken by trained staff-members of the OFV, mostly according to directive 2002/63/EC. The size of a laboratory sample was at least 1 kg.

### **3. Quality Assurance**

Analyses have been carried out to 100 % in the Labor Dr. Matt AG, Im alten Riet 36, FL-9494 Schaan, a private laboratory, with which the OFV has a contract defining the co-operation, the tasks and the obligations. The laboratory is accredited in accordance with the provisions of article 3 of directive 93/99 (according to DIN EN ISO/IEC 17025 [see copy of accreditation certificate in the annex]).

Most samples were analysed by a gas chromatographic multiresidue method (modified German official method DFG S19). This method consists of an acetone/cyclohexane/ethylacetate extraction and a clean-up on a bio beads S-X3 gel permeation column. The extract was analysed by GC-

MS. Polar pesticides, after a QuEChERS sample extraction according to Chemisches- und Veterinäruntersuchungsamt Stuttgart, were analysed by LC-MS-MS.

Dithiocarbamates were also determined on each sample by using a CS<sub>2</sub> evolution method. The CS<sub>2</sub> is determined by headspace analysing using GC with ECD (DIN 12396-2).

The laboratory carrying out the analyses participates as a part of its quality assurance programme in proficiency tests.

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 Residues Analysis"- as set out in Document N° SANCO/10476/2003, 5/February/2004.

The reporting levels are the same as the limits of quantification (generally in the range of 0,005 - 0,05 mg/kg). The limits of quantification are determined by recovery tests.

Analytical uncertainty applied on results varies depending to substance and method between 10 - 15 %.