

THE SWEDISH CONTROL OF PESTICIDE RESIDUES IN FRUITS AND VEGETABLES

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INTRODUCTION

The National Food Administration (NFA), a governmental authority which falls under the Ministry of Agriculture, has the responsibility for the food control in Sweden. The NFA both issues food standards, for instance lays down MRLs for pesticides, carries out control and decides about appropriate actions to be taken. Another authority, the National Chemicals Inspectorate, has the responsibility for the registration of pesticides to be used in Sweden.

Since the mid-1960s fresh fruits and vegetables have been checked for pesticide residues. In the beginning only a few pesticides were covered by the methods used, and maximum residue limits (MRLs) were set for only about ten pesticides. During the last decade an average of about 5.000 of samples have been analysed each year. The number of pesticides, including isomers and break down products, have been increased to around 180. Most of the samples are analysed at four, by NFA authorized and contracted laboratories. The cost for the control is about 15 million Swedish crowns per year.

SAMPLING

Sampling program

Surveillance sampling programs for imported and domestic grown fruits and vegetables are laid down on an annual basis (July-June). In these programs

about 4,000 samples of imports and 1,000 of domestic origin are analysed for pesticide residues. The number of samples taken of each commodity from a given country is roughly proportional to the share it has of the Swedish market. However, and this is important, the number of samples is also based on prior (or arising) residue problems with a particular food/country, and therefore the surveillance sampling is more or less biased.

Compliance sampling

If a surveillance sample contains a pesticide residue above the Swedish Maximum Residue Limit (MRL), the NFA will probably prescribe a condition for further import or sale of the commodity. Compliance samples are then collected, and the following lots are detained pending analyses and clearance. The bill for the compliance samples has to be paid by the importer or domestic grower.

Most of the samples are taken by sampling officers, who belong to the Plant Inspection Service, at the Swedish Board of Agriculture. A small part of the domestic sampling is carried out by municipal Environmental and Health Protection Committees.

Most of the samples in the surveillance program are analysed by the multiresidue method. The table shows the total number of samples analysed, grouped by analytical methods. The sampling was carried out during July 1990 - June 1991.

| Method | Fruits | Vegetables | Total |
|--------------------------|--------|------------|-------|
| Multi-method | 1673 | 2971 | 4644 |
| Biphenyl/2-phenylphenol | 157 | 0 | 157 |
| Bromide (inorganic) | 0 | 43 | 43 |
| Carbendazim incl benomyl | 477 | 197 | 674 |
| Daminozide | 47 | 0 | 47 |
| Diquat | 0 | 37 | 37 |
| Dithianon | 45 | 0 | 45 |
| Dithiocarbamates | 675 | 473 | 1148 |
| Imazalil | 0 | 6 | 6 |
| Thiabendazole | 42 | 58 | 100 |

Sampling procedure

Usually, a "sample" consists of about 3-5 kilos of the commodity taken from three different places (boxes) in the lot. The sub-samples are packed and labelled individually. A delivery note with detailed information about the sample follows the sample to the laboratory. A lot is an identifiable quantity of goods having characteristics presumed to be uniform. The Swedish sampling procedure is very close to those in the EEC and Codex Alimentarius. At the NFA, a great deal of efforts have been laid down on comparing different sampling techniques. We have found that the mean values of different composite samples will be about the same, either small sub-samples are taken from 10-15 boxes or larger sub-samples are taken from three boxes in the same lot. This is important, as one should not open (distroy) more boxes at the time of sampling than necessary to achive a representative sample.

"Mixed lots"

If there are suspicions that a "lot" represents more than one grower or for post-harvest applied pesticides, more than one packing-house, the sub-samples may be analysed separately. In such a case, actions may be taken on the residue contents in one of the sub-samples.

ANALYSIS

Analytical methods

During January 1981 - June 1989 the Swedish control of pesticide residues was based on a multi-residue method, in which the extraction was done by acetone followed by partitioning steps with dichloromethane/n-hexane. The cleanup was performed on a SX-3 gel permeation column (GPC) and the determination of the residues was done by capillary gas chromatography, using SE-30 and OV-1701 columns and EC- and NP-detectors. More than 35,000 samples were analysed with the method during those years.

Since July 1989, the "acetone-method" has been replaced by an ethyl acetate multi-residue method. Both clean-up and the GC-determination are the same as in the previous "acetone-method". Comparison of the efficiency of the two multi-residue methods on crops containing pesticides was shown as a poster at the IUPAC congress in Hamburg in 1990. Some advantages

with the "ethyl acetate-method" are: simplicity, reduction of analysis time and solvents, resulting in cheaper analyses. A major disadvantage is that there are somewhat more coextractives in the GC-extract for some commodities.

By adding sodium hydroxide to the sample homogenate before the extraction step, thiabendazole, imazalil, benomyl, and carbendazim can be analysed. For these pesticides, NFA uses both GC and HPLC for quantification of the residues.

Individual methods are used for the determination of dithiocarbamates, dithianon, daminozide, diquat and inorganic bromide.

Quality control

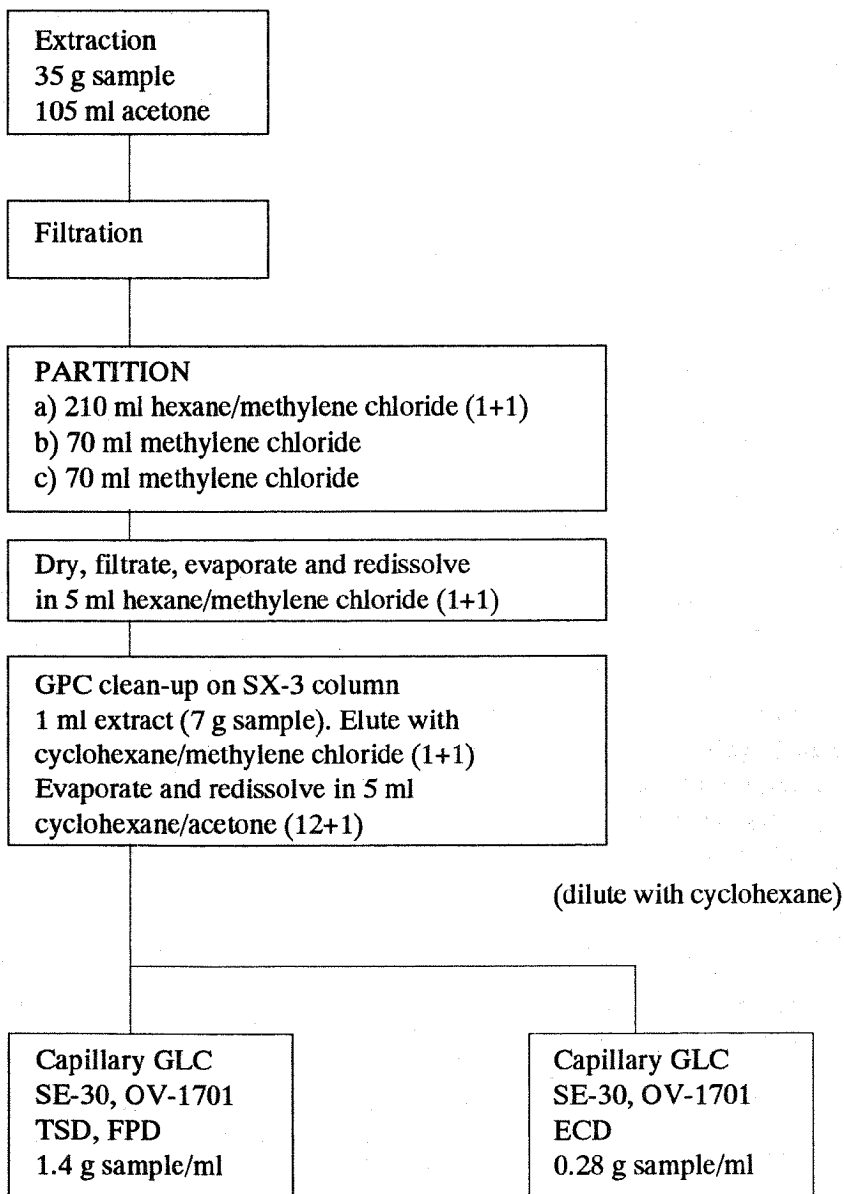
The analytical methods used have to be approved or developed by the National Food Administration.

The NFA buys all the primary standards for the control and makes up the "stock" solutions. These are also quality checked by GC-MS, and new solutions are compared with the previous ones for checking concentration and stability. The contracted laboratories are supplied by NFA with standard solutions, both single standards and mixtures. Furthermore, the laboratories have to participate in a number of intercalibrations.

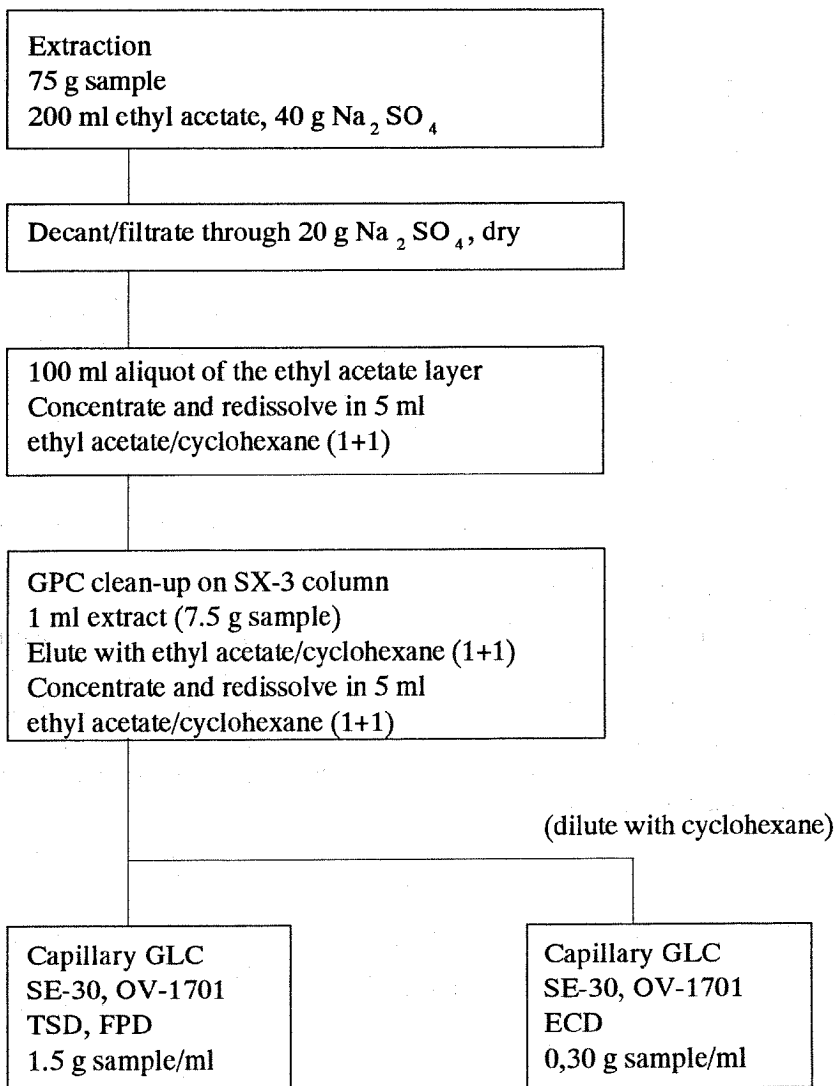
In general, when a pesticide residue exceeds a maximum residue limit, a second analyses is carried out. At the same time, when ever possible, the first result is checked by GC-MS, both for identity and quantity. All the GC-MS work has up to now been done at the NFA, but within a few weeks the GC-MS confirmation will be carried out at two of our contracted laboratories.

From 1990, all residues found by the laboratories and reported to our central computer system have to be determined on two different capillary columns, and if possible, also with two different detectors. As a rule, only pesticide residues above the tolerances are confirmed by GC-MS. This means that some errors, false positives, could have been reported as findings, but only at a level which didn't effect the decision making. To minimize these possible errors, the first pesticide residue found in a commodity/country combination will be confirmed by GC-MS. The extension of the confirmatory work will start this year.

ACETONE METHOD



ETHYL ACETATE METHOD



Reporting results

The contracted laboratories have to inform the NFA within 48 hours if a pesticide residue in a surveillance sample may exceed a tolerance (screening result). Final results, including the second analyses, have to be transferred, via telephone modem, to the central computer at the NFA within three days. For a compliance sample the corresponding space of time is 24 hours and 48 hours, respectively.

MAXIMUM RESIDUE LEVELS

The National Food Administration's Ordinance on Foreign Substances in Food issued in 1983 (SLV FS 1983:1) and the amendment (SLV FS 1989:29) contain maximum residue levels for about 100 pesticides. The commodities are grouped in two main groups, fruits and vegetables and potatoes, respectively. For a great number of pesticides there are exceptions for one or a few commodities, for instance citrus fruits, which in many cases have a higher MRL than the one applied for fruits and vegetables in general.

If there is no regulation concerning a certain pesticide in the above-mentioned Ordinance the NFA decides on a case by case basis if it is judged necessary the maximum level to be applied. At present, this is the case for carbendazim including benomyl, chlorpropham, chlorpyrifos-methyl and daminozide, "paragraph" 4 of the above mentioned Ordinance reads as follows: "If regulations concerning a certain foreign substance or a certain group of food products are not given in this Ordinance, the Food Administration decides the maximum level to be applied".

In a near future, the MRLs will probably be converted to the system of food grouping, which is laid down by the EEC.

The maximum residue limits are valid equal for imported and domestic products. There is no obstacle to prescribe a Swedish maximum residue limit for a pesticide, though the pesticide is not registered for use in Sweden.

FIXING MRLs IN SWEDEN

A number of different factors has input in the administrative procedure of fixing MRLs in Sweden. Of main importance are: toxicological evaluation

of the pesticide; Good Agriculture Practise (GAP) and residue data from supervised trials; analytical method including limit of determination for the pesticide/commodity combination concerned; residues found in the surveillance sampling; harmonization efforts (Codex, EEC, Nordic countries).

REPORTING LIMIT

In most of the residue reports from the National Food Administration's regulatory monitoring of pesticide residues in fruits and vegetables, the level for reporting findings has been set at 20% of the corresponding maximum level, provided that it is possible to determine the pesticide at this level. It means that figures below this "reporting limit" are not shown and are often called "no residues".

Reasons not to analyse all samples from the limit of determination are: lack of time and costs for the analytical work. The main purpose for the present control is to prevent, that lots with excessive residues reach the consumer.

RESULTS AND DISCUSSION

The results from the Swedish regulatory monitoring of pesticide residues in fruits and vegetables, during July 1990 - June 1991, are summarized in figure 1. A total number of 4693 samples were analysed. Of these, 4.2% (197 samples) contained excessive residues, and 75% contained "no residues". Most of the samples with excessive residues have been imported from EEC-countries. These countries are also the main suppliers of Sweden with many different commodities.

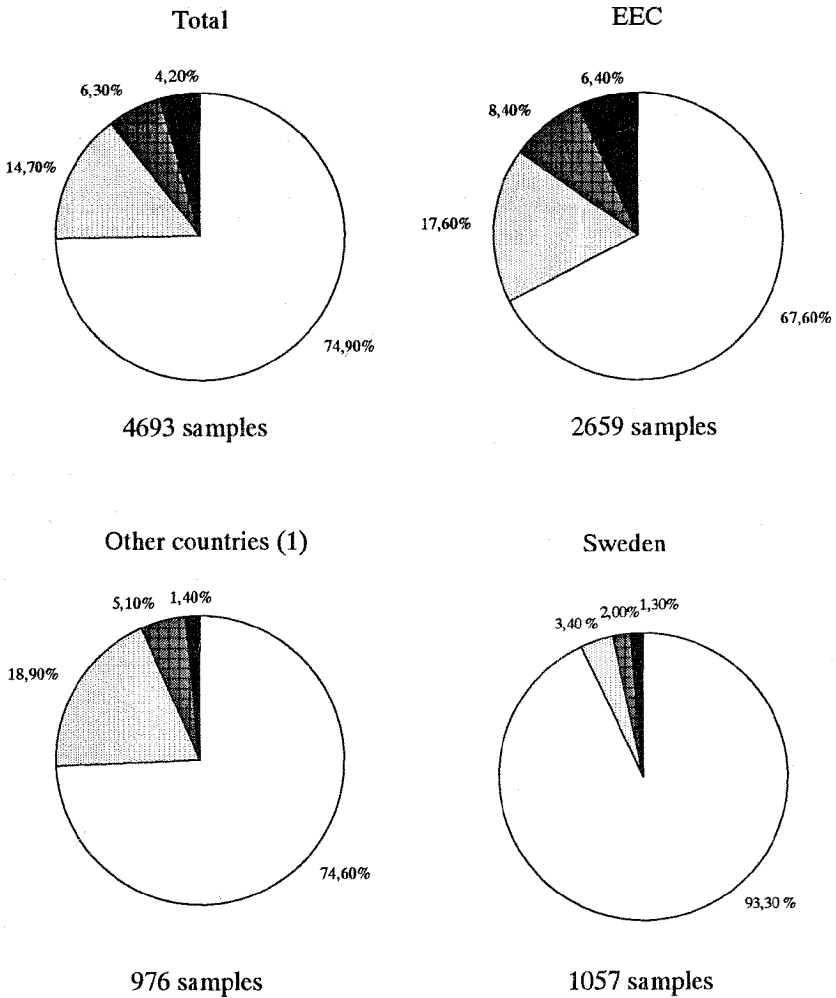
Figure 1 shows also a comparison between samples originated from Sweden, Spain and the Netherlands, respectively. The great number of exceedings of products imported from the Netherlands were caused by excessive residues of chlorothalonil in leeks. The violations of imported products from Spain, 31 samples, were mainly caused by excessive amounts of endosulfan and methamidophos in sweet peppers (21 samples). The results of the Swedish control of pesticide residues in commodities imported from Spain, during July 1990 - June 1991, are given in detail as an appendix.

If a violation occurs in the compliance sampling, and in some cases in the surveillance sampling, the lot will be rejected or prohibited for sale. The table shows all lots, that were rejected or prohibited for sale during July 1990 - June 1991.

| Food | Country | Number of lots | Weight, ton |
|----------------|----------------|-----------------------|--------------------|
| Apple | Argentina | 1 | 1.9 |
| Apple | Italy | 1 | 16.4 |
| Apple | USA | 1 | 57.5 |
| Dill | Italy | 1 | 0.6 |
| Grapes | Cyprus | 1 | 15.4 |
| Grapes | Turkey | 3 | 5.8 |
| Leeks | France | 1 | 9.6 |
| Leeks | Netherlands | 25 | 18.5 |
| Lettuce | Netherlands | 1 | 0.2 |
| Lettuce | Sweden | 2 | 0.4 |
| Pear | Italy | 3 | 21.9 |
| Pepper (sweet) | Spain | 8 | 11.7 |
| Potatoes | Ireland | 2 | 35.8 |
| Potatoes | Netherlands | 2 | 45.7 |
| Potatoes | United Kingdom | 1 | 8.4 |
| Potatoes | Sweden | 1 | 8.5 |
| Strawberries | Italy | 2 | 13.2 |
| Total | | 56 | 271.5 |

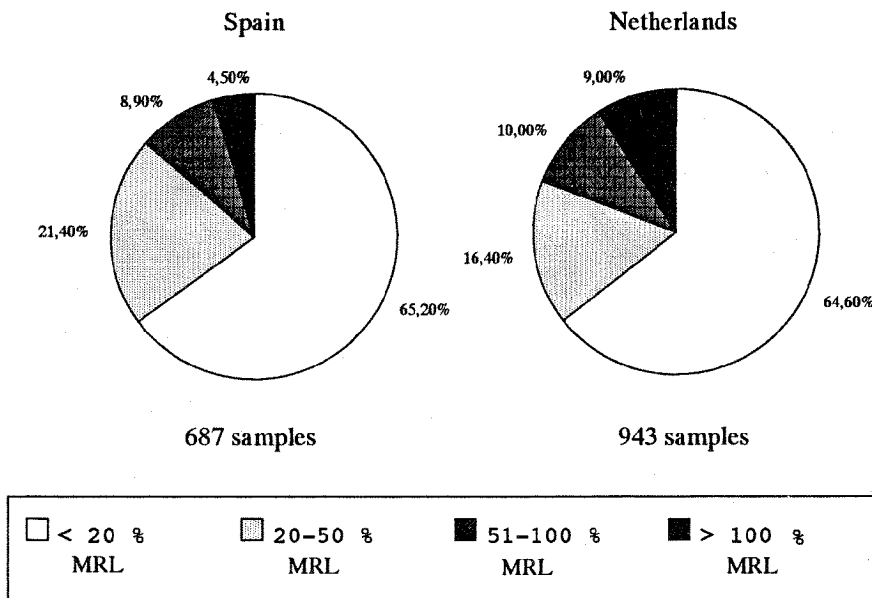
Figure 1. Summary of the Swedish regulatory monitoring of pesticide residues in fruits and vegetables, July 1990 - June 1991.

Figura 1. Resumen del programa de control sueco de residuos de plaguicidas en frutas y hortalizas, Julio 1990 - Junio 1991.



(1) Excl. EEC and Sweden

Figure 1 / Figura 1



APPENDIX: Regulatory monitoring (all samples): Pesticide residues in fruits and vegetables imported from Spain, July 1990 - June 1991. The ranges are given in percent of the Swedish Maximum Residues Limit (MRL) of Guideline Level (GL). K= Swedish MRL, R= Swedish GL.

APENDICE: Programa de control (todas las muestras): residuos de plaguicidas en frutas y hortalizas importadas desde España. Junio 1990 - Julio 1991. Los intervalos están dados en % del límite máximo del residuo sueco (MRL) o Nivel Guía (GL). K= MRL Sueco, R=GL Sueco

| Food | Country of origin | Number of samples | | Pesticide | Number of findings within intervals (%) | | | MRL mg / kg | |
|-----------------|-------------------|-------------------|----------------------|--------------------|---|--------|------|-------------|-------|
| | | Total | Within intervals (%) | | 20-50 | 51-100 | >100 | | |
| APPLE | SPAIN | 11 | 2 | CHLORFENVINPHOS | 1 | | | 0,4 K | |
| | | | | DIAZINON | 1 | | | 0,3 K | |
| | | | | DIPHENYLAMINE | 1 | | | 3,0 K | |
| APRICOT | SPAIN | 3 | 1 | CAPTAN + FOLPET | | 1 | | 2,0 K | |
| | | | | DITHIOCARBAMATES | 1 | | | 1,0 K | |
| AVOCADO | SPAIN | 1 | | | | | | | |
| BROCCOLI | SPAIN | 9 | | CAPTAN + FOLPET | | | 1 | 0,1 K | |
| CARROT | SPAIN | 1 | | | | | | | |
| CELERY | SPAIN | 3 | 1 | BROMIDE, INORGANIC | 1 | | | 30 K | |
| | | | | | CAPTAN + FOLPET | | 1 | | 0,1 K |
| | | | | | CHLOROTHALONIL | 1 | | 1 | 1,0 K |
| | | | | | TRIAZOPHOS | 1 | | | 0,1 K |
| CHINESE CABBAGE | SPAIN | 22 | 2 | BROMIDE INORGANIC | 1 | | | 30 K | |
| | | | | DITHIOCARBAMATES | 1 | | | 1,0 K | |
| | | | | METHAMIDOPHOS | | | 1 | 0,2 K | |

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| Food | Country of origin | Number of samples | | Pesticide | Number of findings within intervals (%) | | MRL mg / kg | | |
|--------------------|-------------------|-------------------|---|-----------|---|--------------------|-------------|---|--------|
| | | Total | Within intervals (%) 20-50 51-100 >100 | | 20-50 | 51-100 >100 | | | |
| CUCUMBER | SPAIN | 67 | 15 | 4 | 3 | ACEPHATE | 3 | 1 | 1,0 K |
| | | | | | | ALDRIN + DIELDRIN | 1 | 1 | 0,05 K |
| | | | | | | CAPTAN + FOLPET | | 1 | 0,1 K |
| | | | | | | CARBOFURAN | | 1 | 0,1 K |
| | | | | | | CHLOROTHALONIL | 2 | | 1,0 K |
| | | | | | | DITHIOCARBAMATES | 8 | 2 | 1,0 K |
| | | | | | | ENDOSULFAN (SUM) | 3 | | 0,5 K |
| | | | | | | METHAMIDOPHOS | 4 | | 0,2 K |
| | | | | | | EGG PLANT | SPAIN | 2 | |
| CAPTAN + FOLPET | | 1 | 3,0 K | | | | | | |
| CHLORPYRIFOS | 4 | 2 | 0,5 K | | | | | | |
| DITHIOCARBAMATES | 5 | | 1,0 K | | | | | | |
| METHAMIDOPHOS | 3 | | 0,2 K | | | | | | |
| MONOCROTOPHOS | | 2 | 0,1 K | | | | | | |
| PARATHION - METHYL | 1 | 1 | 0,1 K | | | | | | |
| CHLORFENVINPHOS | 1 | | 0,4 K | | | | | | |
| DICLORAN | 1 | | 5,0 K | | | | | | |
| LEMON | SPAIN | 36 | 6 | | | ENDOSULFAN (SUM) | 1 | | 0,5 K |
| | | | | | | METHIDATHION | 2 | | 2,0 K |
| | | | | | | PHENYLPHENOL, 2- | 1 | | 10 K |
| | | | | | | ACEPHATE | | 1 | 1,0 K |
| | | | | | | CAPTAN + FOLPET | 1 | | 2,0 K |
| GARLIC | SPAIN | 5 | | | | | | | |
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| GRAPES | SPAIN | 36 | 11 | 5 | 1 | ACEPHATE | 4 | | 1,0 K |
| | | | | | | CAPTAN + FOLPET | | 1 | 3,0 K |
| | | | | | | CHLORPYRIFOS | 4 | 2 | 0,5 K |
| | | | | | | DITHIOCARBAMATES | 5 | | 1,0 K |
| | | | | | | METHAMIDOPHOS | 3 | | 0,2 K |
| | | | | | | MONOCROTOPHOS | | 2 | 0,1 K |
| | | | | | | PARATHION - METHYL | 1 | 1 | 0,1 K |
| | | | | | | CHLORFENVINPHOS | 1 | | 0,4 K |
| | | | | | | DICLORAN | 1 | | 5,0 K |
| LETTUCE, ICEBERG | SPAIN | 82 | 6 | 1 | | ENDOSULFAN (SUM) | 1 | | 0,5 K |
| | | | | | | METHIDATHION | 2 | | 2,0 K |
| | | | | | | PHENYLPHENOL, 2- | 1 | | 10 K |
| | | | | | | ACEPHATE | | 1 | 1,0 K |
| | | | | | | CAPTAN + FOLPET | 1 | | 2,0 K |
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| Food | Country of origin | Number of samples | | Pesticide | Number of findings within intervals (%) | | MRL mg / kg | | |
|------------------|-------------------|-------------------|---|------------------|---|-------------------|-------------|----|-------|
| | | Total | Within intervals (%) 20-50 51-100 >100 | | 20-50 | 51-100 >100 | | | |
| LETTUCE, ICEBERG | | | | CHLORPYRIFOS | 1 | | 0,5 K | | |
| | | | | DITHIOCARBAMATES | 3 | | 1,0 K | | |
| | | | | ENDOSULFAN (SUM) | 1 | | 0,5 K | | |
| MANDARIN | SPAIN | 37 | 14 | 4 | 1 | AZINPHOS - METHYL | 7 | 1 | 1,0 K |
| | | | | | | CARBOPHENOITHION | 1 | | 0,5 K |
| | | | | | | DICOFOL | 2 | | 3,0 K |
| | | | | | | FENTROTHION | 1 | 1 | 0,5 K |
| | | | | | | FENTHION (SUM) | 1 | | 1,0 K |
| | | | | | | MALATHION | 1 | | 3,0 K |
| | | | | | | MECARBAM | 1 | | 2,0 K |
| | | | | | | METHIDATHION | 4 | 2 | 2,0 K |
| | | | | | | TETRADIFON | 1 | | 2,0 K |
| | | | | | | MELONS | SPAIN | 39 | 7 |
| ENDOSULFAN (SUM) | 6 | | 0,5 K | | | | | | |
| HEPTACHLOR (SUM) | | 1 | 0,05 K | | | | | | |
| NECTARINE | SPAIN | 2 | | | | | | | |
| ONION | SPAIN | 4 | | | | | | | |
| ORANGE | SPAIN | 33 | 8 | 1 | 1 | AZINPHOS - METHYL | 1 | | 1,0 K |
| | | | | | | CAPTAN + FOLPET | | 1 | 0,1 K |
| | | | | | | CHLORFENVINPHOS | 1 | | 0,4 K |
| | | | | | | DICLORAN | | 1 | 5,0 K |
| | | | | | | ENDOSULFAN (SUM) | 1 | | 0,5 K |
| | | | | | | FENTHION (SUM) | 2 | | 1,0 K |
| METHIDATHION | 3 | | 2,0 K | | | | | | |
| THIABENDAZOLE | | 1 | | | 10 | K | | | |

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| Food | Country of origin | Number of samples | | Pesticide | Number of findings within intervals (%) | | | MRL, mg / kg | | | |
|----------------|-------------------|-------------------|----------------------|------------------|---|-----------------------|------|--------------|-----|-----|---|
| | | Total | Within intervals (%) | | 20-50 | 51-100 | >100 | | | | |
| PEACH | SPAIN | 6 | | | | | | | | | |
| PEAR | SPAIN | 2 | 2 | DITHIOCARBAMATES | 2 | | 1,0 | K | | | |
| PEPPERS, SWEET | SPAIN | 166 | 58 | 38 | 21 | ACEPHATE | 3 | 2 | 1,0 | K | |
| | | | | | | CARBENDAZIM | 1 | | 1,0 | R | |
| | | | | | | CHLORPYRIFOS | 7 | 5 | 3 | 0,5 | K |
| | | | | | | CHLORPYRIFOS - METHYL | 3 | 3 | 1 | 0,1 | R |
| | | | | | | CYPERMETHRIN | 6 | 1 | | 2,0 | K |
| | | | | | | DICHLORVOS | 1 | | | 0,1 | K |
| | | | | | | DITHIOCARBAMATES | 2 | | | 1,0 | K |
| | | | | | | ENDOSULFAN (SUM) | 58 | 34 | 12 | 0,5 | K |
| | | | | | | HCH (SUM) | 5 | 2 | | 1,0 | K |
| | | | | | | METHAMIDOPHOS | 12 | 7 | 6 | 0,2 | K |
| | | | | | | OMETHOATE | 3 | 1 | | 0,2 | K |
| PLUM | SPAIN | 9 | | | | ORGANOPHOSPHOROUS | 1 | 3 | 2 | 1,0 | K |
| | | | | | | PIRIMIPHOS - METHYL | 14 | 6 | 3 | 1,0 | K |
| | | | | | | TRICHLORFON | 3 | 3 | 3 | 0,2 | K |
| | | | | | | ENDOSULFAN (SUM) | 1 | | | 0,5 | K |
| POMELO | SPAIN | 1 | | | | | | | | | |
| POTATO | SPAIN | 6 | | | | | | | | | |
| SQUASH | SPAIN | 2 | 1 | | | | | | | | |
| STRAWBERRIES | SPAIN | 18 | 4 | 1 | 2 | CAPTAN + FOLPET | 1 | | 1 | 3,0 | K |
| | | | | | | CARBENDAZIM | 2 | 1 | | 1,0 | R |
| | | | | | | CHLORPYRIFOS | 1 | | | 0,5 | K |

| Food | Country of origin | Number of samples | | Pesticide | Number of findings within intervals (%) | | MRL mg / kg | | |
|-----------------------------|-------------------|-------------------|----------------------|------------------|---|---------------------|-------------|-------|-------|
| | | Total | Within intervals (%) | | 20-50 | 51-100 >100 | | | |
| STARWBERRIES | | | | DICHOFLUANID | 1 | | 5,0 K | | |
| | | | | DICOFOL | 1 | | 3,0 K | | |
| | | | | DITHIOCARBAMATES | 2 | 2 | 1,0 K | | |
| | | | | IPRODIONE | 1 | | 10 K | | |
| | | | | TETRADIFON | 1 | | 2,0 K | | |
| | | | | VINCLOZOLIN | 1 | | 2,0 K | | |
| TOMATO | SPAIN | 79 | 8 | 2 | 1 | ACEPHATE | 2 | 1,0 K | |
| | | | | | | CARBENDAZIM | 1 | 1,0 R | |
| | | | | | | CHLOROTHALONIL | 1 | 1,0 K | |
| | | | | | | DITHIOCARBAMATES | 4 | 1,0 K | |
| | | | | | | ENDOSULFAN (SUM) | 1 | 0,5 K | |
| | | | | | | METHAMIDOPHOS | 1 | 2 | 0,2 K |
| | | | | | | PIRIMIPHOS - METHYL | 1 | 1,0 K | |
| | | | | | | ENDOSULFAN (SUM) | 1 | 0,5 K | |
| | | | | | | TRICHLORFON | 1 | 0,2 K | |
| | | | | | | WATERMELON | SPAIN | 5 | 1 |
| Total number of samples | | 687 | 147 | 61 | 31 | | | | |
| Total number of samples (%) | | | 21 | 8,9 | 4,5 | | | | |