Food Control 22 (2011) 1701–1706

Contents lists available at ScienceDirect

Food Control

journal homepage: www.elsevier.com/locate/foodcont

# Pesticide residues in fruits and vegetables from South America – A Nordic project

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ARTICLE INFO

*Article history:*
Received 14 December 2009
Received in revised form
27 April 2010
Accepted 4 May 2010

*Keywords:*
Pesticides
Residue analysis
South America
Fruit and vegetables

ABSTRACT

The aim of this study was to investigate the amount of pesticide residues in fruits and vegetables from South America. A total of 724 samples of 46 different fruits and vegetables from eight South American countries were collected in 2007. In 19% of the samples no residues were found, 72% of samples contained pesticide residues at or below MRL, and 8.4% of samples contained pesticide residues above MRL. Thiabendazole, imazalil and chlorpyrifos were the pesticide most frequently found. Thirty-seven pesticides were found with frequencies higher that 1% in the samples. The results emphasize the need for continuous monitoring of pesticide residues, especially in imported fruits and vegetables.



## 1. Introduction

In a cooperation project between five countries: Denmark, Estonia, Finland, Norway and Sweden, the results from each countries national monitoring programme for pesticide residues in food were compiled. It was decided that for 2007 the focus would be on samples imported from South America. This Nordic collaboration makes it possible to monitor more samples from a specific area than the individually countries are capable of doing on their own. The outcome is a better statistic foundation to investigate the frequency of residues and to evaluate if the small number of monitoring samples from each country is adequate.

The aim of this project was to gather as much information as possible on pesticide residues in crops imported from South America. Each of the five countries analysed the samples according to their own analytical scope, which means that the samples were analysed for 170 to 326 pesticides including metabolites and degradation products.

For several years the use of pesticides have been escalating in the developing countries, particularly those in the tropical regions seeking to enter the global economy by providing off-season fresh fruits and vegetables to countries in more temperate climate. Such countries are becoming important fruit baskets to the world being capable of harvesting two or even three times each year (Ecobichon, 2001). However, the ambitions to increase the exported 'cash' crops are driving the developing countries toward an increased use of pesticides.

The heavy use of pesticides may result in environmental problems like disturbance of the natural balance, widespread pest resistance, environmental pollution, hazards to non-target organisms and wildlife, and hazards to humans. Control programmes for pesticide residues in the developing countries are often limited due to lack of resources and rigorous legislation is not in place. Moreover, training programmes for technical personnel and equipment for monitoring pesticide residues are often lacking. Another reason to focus on pesticide residues in samples from South America is that the annual EU monitoring report shows that pesticide residues in these samples more often contain residues above the Maximum Residue Limits (MRLs) compared to pesticide residues in samples from EU countries (EU Commission, 2006).

The objective of this study was to investigate the amount of pesticide residues in fruits and vegetables from South America. The results will be used when designing future control programmes for this region and taking preventive actions to minimize human health risk.

## 2. Materials and methods

### 2.1. Sampling

Samples of fruits and vegetables from South America were collected as a part of the national monitoring programme for

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0956-7135/$ – see front matter © 2010 Published by Elsevier Ltd.
doi:10.1016/j.foodcont.2010.05.017

**Table 1**
Main steps in the multi-methods used.

| Name of method | Country | GC/LC | Detector | No. of pest. | Method reference | Sample weight | Extraction solvent | Clean up |
|---|---|---|---|---|---|---|---|---|
| FP017 | Denmark | GC | EC-NP-IT-MS/MS | 105 | (Poulsen & Granby, 2000) | 25 | Acetone/ethyl acetate/ cyclohexane | GPC |
| JT-1.1 | Estonia | GC | EC-NP-MS | | EN 12393-1,2,3:1998 | 20 | Acetone/dichloromethane | None |
| QuEChERS | Finland | GC | MS, EC, NP | 118 | EN 1566.2 | 10 | Acetonitrile | Dispersive SPE with PSA |
| Mini-Luke | Norway | GC | MS | 183 | (Luke, Froberg, Doose, & Masumoto, 1981) | 20 | Acetone | None |
| M200 | Sweden | GC<br>LC | MS/MS<br>MS/MS | 299 | (Pihlstrom, Blomkvist, Friman, Pagard, & Osterdahl, 2007) | 75 | Ethyl acetate | None |
| FP086 | Denmark | LC | MS/MS | 85 | (Granby, Andersen, & Christensen, 2004) | 10 | Methanol | None |
| JT-1.1 | Estonia | LC | MS/MS | | EN 12393-1,2,3:1998 | 25 | Ethyl acetate | None |
| QuEChERS | Finland | LC | MSMS | 129 | EN 15662 | 10 | Acetonitrile | Dispersive SPE with PSA |
| Mini-Luke | Norway | LC | MS/MS | 73 | Internal | 10 | Acetone | None |

pesticide residues in each of the Nordic countries including Estonia. The sampling was done according to the EU directive 2002/63/EC (EU Commission, 2002) on sampling for official control of pesticide residues. The sampling was performed by authorized personnel from the food control authorities in the countries involved. The samples were mainly taken at importers and wholesaler's warehouses in different parts of the countries. Some samples were taken at retailers or at marked places. A total of 724 samples of 46 different fruits and vegetables from eight South American countries were collected in 2007. The samples included 680 samples of fruits and 44 samples of vegetables.

### 2.2. Chemical analysis

Pesticide analyses were carried out at regional or central food control laboratories in each of the participating countries. The laboratories were accredited for all analytical methods used for the official control of pesticide residues in food of plant origin.

The analytical scope varied between the countries both in regard to pesticides included and the analytical methods used. The samples were analysed for 170 to 326 pesticides including metabolites and degradation products using (1) GC multi-methods with ECD, NPD, ITD, MS or MS/MS detection, (2) HPLC multi-methods with MS/MS detection, and (3) single residue methods for determination of dithiocarbamates and chlormequat.

Tables 1 and 2 illustrate the important steps in the analytical procedures for the multi-residue methods and single residues methods, respectively. The methods are listed including the number of pesticides covered.

### 2.3. Quality assurance

In accordance with ISO/IEC 17025:2005 (ISO, 2005), the analytical laboratories had documented quality control procedures. The EU's guidelines for Method validation and quality control procedures for pesticide residues analysis in food and feed, (EU Commission, 2007) were implemented as far as practical in each country. Each analytical batch included one to two spiked recovery samples. Acceptable limits for individual recovery results should be in the range of; mean recovery $\pm 2 \times$ RSD (%) and may be adjusted using repeatability and/or intra-laboratory data as described in Point 64, 'Acceptability of analytical performance for routine recoveries' (EU Commission, 2007).

When a pesticide residue in a sample exceeds the MRL, a second quantitative analysis is carried out to verify the first result. Recovery was checked and the identity of the pesticide was confirmed by GC–MS, GC–MS/MS or LC–MS/MS. If the residue result is above the MRL, the sample is defined as an exceedence. However, before any enforcement action was taken, the analytical uncertainty was subtracted from the measured value. If the result still exceeds the MRL, enforcement action was taken in form of stop of further distribution and selling of the lot, and follow-up sampling of subsequent lots of the same origin.

## 3. Results and discussion

The pesticides included in the analytical scope were prioritized in relation to application, toxicity and persistency. A pesticide finding in another country is also a criterion for including the pesticide in the national programme. All pesticide residues at or

**Table 2**
Main steps for each countries single residue methods.

| Name of method | Country | GC/LC | Detector | No. of pest. | Method reference | Sample weight (g) | Extraction solvent | Clean up |
|---|---|---|---|---|---|---|---|---|
| Dithiocarbamates | Finland/ Norway | UV/Vis | UV/Vis | 8 | EN 12396-1 | 200 | | Distillation |
| Dithiocarbamates | Denmark | UV/Vis | UV/Vis | | (Juhler, Lauridsen, Christensen, & Hilbert, 1999) | 100 | | Distillation |
| Dithiocarbamates | Sweden | GC | FPD(S) | 5 | Pihlström P. NFA, Sweden, Not published | 50 | Tín chloride/Hydrochloric acid isooctane | |
| Chlormequat | Denmark | LC | MS/MS | 1 | | 10 | Methanol/Water/acetic acid | |
| Chlormequat and mepiquat | Sweden | LC | MS/MS | 2 | (Alder & Startin, 2005) | 20 | IS + methanol | |