

The French system for surveillance of contamination by plant protection products in foodstuffs of animal origin

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Abstract

Every year, programmes for the surveillance and control of contamination in foodstuffs of animal origin are organised by the Directorate General for Food (DGAL). These programmes constitute an important tool in the food safety system. In animal production, eleven surveillance programmes are carried out for the detection of pesticide residues. Samples are collected in the preliminary stage in farms. Multi-residue methods are used to test for pesticide residues in foodstuffs. Programmes organised in 2014 and 2015 generated nearly 161,000 analysis results. Detected contamination levels were very low (no non-compliant samples in 2014, two in 2015) in accordance with the results obtained by other Member States. The only two non-compliant samples detected concerned lindane. This contamination was probably due to the persistence of this substance in the environment.

Keywords

Pesticides, Residues, Foodstuffs of animal origin, Surveillance programmes, Control programmes

Résumé

Le dispositif français de surveillance des produits phytosanitaires dans les denrées alimentaires d'origine animale

Les plans de surveillance et de contrôle de la contamination des denrées alimentaires d'origine animale sont mis en place chaque année par la direction générale de l'Alimentation en application de la réglementation européenne. En production animale, onze plans de surveillance sont mis en œuvre pour la recherche des résidus de pesticides. Les prélèvements sont réalisés au stade de la production primaire chez les éleveurs français. Les résidus de pesticides sont recherchés dans ces denrées alimentaires par méthodes multi-résidus. Les plans de 2014 et 2015 ont engendrés plus de 161000 résultats d'analyses. Le taux de contamination détecté est très faible (deux prélèvements non conformes en 2015 et aucun en 2014), ce qui est cohérent avec ce qui est observé dans les autres États membres. Les deux seules non-conformités détectées concernaient le lindane. Cette contamination est probablement d'origine environnementale, due à la rémanence de cette substance.

Mots-clés

Pesticides, résidus, denrées alimentaires d'origine animale, plans de surveillance, plans de contrôle

The use of pesticides (or phytosanitary products or plant protection products) developed from the end of the Second World War. Pesticides are classified into four categories depending on their use: fungicides, herbicides, insecticides and a fourth category for miscellaneous substances. In terms of production, the breakdown in tonnage in 2014 was 45%, 40%, 2% and 13%, respectively (French Plant Protection Industry Association - UIPP, 2014). The first insecticides used were synthetic products belonging to the organochlorine compound class, which, as a result of their persistence, are still found in the environment today, several decades after their use was discontinued. These chemical products accumulate all along the food chain and, due to their high lipophilic affinity, may contaminate certain foodstuffs of animal origin with high fat content. Despite the gradual phasing-out of pesticides associated with known health risks (the most problematic substances), use of plant protection products has remained common practice in conventional agriculture since the 1980s. Manufacturers have progressively replaced these pesticides by organophosphate compounds, synthetic pyrethroids, carbamates, triazoles and neonicotinoids (Table 1).

Although foodstuffs of plant origin are the main food category likely to contain pesticide residues, foodstuffs of animal origin may also lead consumers to be exposed to these contaminants. This is because once a substance is applied to a crop, residues of this substance (parent compound and/or degradation products) may be found in plant products consumed by animals, and pesticide residues are known to accumulate in animal tissues.

Objectives of the surveillance programme - Regulatory references

The European system for monitoring plant protection products in foods of animal origin addresses one of the missions of the European Food Safety Authority (EFSA), established by Regulation (EC) No 178/2002, i.e. the collection of data with the purpose of measuring consumer exposure to these residues and identifying any emerging risks.

This system is governed by the following regulations:

- Council Directive 96/23/EC, which requires that Member States of the European Union implement surveillance and control plans for chemical residues (more specifically, residues of plant protection products) in foodstuffs of animal origin. Since 1997, France has organised control plans to meet this regulatory requirement and communicates the results to the European Commission on an annual basis. Similarly, the Commission forwards compiled results from the various Member States to EFSA, mandated for this purpose within the framework of Article 31 of Regulation (EC) No 178/2002, and
- the various EU implementing regulations (788/2012 - 400/2014 - 2015/595) concerning the coordinated multiannual control programme for the years 2013 to 2018. These regulations list the active substance/foodstuff pairs to be assessed over this period. These provisions aim to evaluate compliance with maximum levels of pesticides in or on foodstuffs of plant and animal origin, and assess consumer exposure to these residues. These maximum residue levels (MRLs), applicable to pesticides found in or on food

and feed of plant or animal origin, are established in Regulation (EC) No 396/2005. These MRLs for foodstuffs, established for each substance, guarantee that the residues found in the food do not represent a risk for the consumer further to use of the active plant protection product in accordance with good agricultural practices for the treatment of crops.

Surveillance and control plans

To meet the requirements of the various regulations, the Directorate General for Food (DGAL) organises surveillance plans (SPs) and control plans (CPs). The Directorate sets up a national and then regional

sampling schedule in line with the sampling plans it determines or those stipulated in European regulations. The regional players (Regional Food, Agriculture and Forestry Directorates - DRAAF and Regional Food Authorities - SRAL) are in charge of departmental scheduling, working jointly with the DDecPPs tasked with carrying out the sampling.

The difference between SPs and CPs lies in their objectives, resulting in a different sampling strategy. In the case of SPs, the aim is to evaluate the representative level of contamination of a food category (ultimately, these data help assess consumer exposure), by random sampling within a population or sub-population, and thus without

Table 1. Groups of pesticides used

Product groups over time			
	Herbicides	Fungicides	Insecticides
Before 1900	Copper sulphate Iron sulphate	Sulphur Copper salts	Nicotine
1900-1920	Sulphuric acid		Arsenic salts
1920-1940	Nitro derivatives		
1940-1950	Plant hormones		Organochlorines Organophosphates
1950-1960	Triazines, Substituted ureas, Carbamates	Dithiocarbamates Phthalimides	Carbamates
1960-1970	Bipyridyl, Toluidines, etc.	Benzimidazoles	
1970-1980	Amino phosphonates, Propionates, etc.	Triazoles, Dicarboximides, Amides, Phosphites, Morpholines	Pyrethroids, Benzoylureas (growth regulators)
1980-1990	Sulphonylureas		
1990-2000		Phenylpyrroles, Strobilurins	

Source: French Plant Protection Industry Association (UIPP) - Brochure on research in plant protection products

Box.

Objectives

Since 1998, control plans for the detection of pesticide residues for agricultural use have been implemented in primary production to meet the requirements of Council Directive 96/23/EC of 29 April 1996 on measures to monitor certain substances and residues thereof in live animals and animal products.

The objective of these control plans is to detect any illegal treatments and/or inappropriate practices in primary production that may adversely affect the safety of foodstuffs. They help to manage the risk of foodstuff contamination by chemical substances that are considered to have probable or proven chronic toxicity. They provide surveillance data regarding this contamination in order to contribute to national and European risk assessments. The implementation of Directive 96/23/EC is aimed at guaranteeing harmonisation of national controls in each Member State to maintain the same level of safety.

Programming framework

Regulation (EC) No 78/2002, i.e. the collection of data with the purpose of measuring consumer exposure to these residues and identifying any emerging risks.

Council Directive 96/23/EC of 29 April 1996 on measures to monitor certain substances and residues thereof in live animals and animal products.

Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC.

Commission Implementing Regulations (EU) (88/2012 - 400/2014 - 2015/595) concerning the coordinated multiannual control programme for the years 2013 to 2018.

Protocol

- Type of contaminants of interest: Pesticides for agricultural use and veterinary medicinal products (acaricides).
- Targeted production sectors (populations): foodstuffs of animal origin.
- Stage of the food chain: slaughterhouses, beekeepers for honey.
- Definition of a "case": Non-compliance involves either the simple presence of pesticide residues when the substance yielding the residues is banned, or the presence of residues at concentrations above those authorised (> MRLs).
- Number of samples and sampling method: the number of samples to collect by sector and by sampling site (farm or slaughterhouse) was calculated to meet the minimum requirements of Council Directive 96/23/EC, on a pro rata basis of the number of animals slaughtered (meat and large game), slaughtered tonnages (poultry, small game and rabbits), and production volumes (farmed fish, milk, eggs and honey).
- Sampling strategy: exhaustive.
- Analytical method, type of samples: the Directorate General for Food (DGAL) organises surveillance and control plans (SCPs). The Directorate sets up a national and then regional sampling schedule in line with the chosen sampling plans or those stipulated in European regulations. The regional players (Regional Food, Agriculture and Forestry Directorates - DRAAF and Regional Food Authorities - SRAL) are in charge of departmental scheduling, working jointly with the DDecPPs tasked with carrying out the sampling.

Almost all analyses are carried out by accredited laboratories in accordance with Standard NF EN ISO/CEI 17025 and approved by the Ministry of Agriculture, Food and Forestry, and by National Reference Laboratories (NRLs).

taking into account the contamination risk level. In the case of CPs, the aim is to characterise abnormal situations and to detect suspected non-compliance or cases of fraud. Sampling in this case is targeted at a portion of the production that is assumed to have a higher risk of contamination (sampling undertaken on the basis of predetermined targeting criteria).

The planning of regional and then departmental sampling, the quality of sampling, and the precision of the collected data *versus* expected results are critical factors affecting the credibility of the generated safety information. The robustness of the system underpins satisfactory risk management and non-biased risk assessment.

The official analyses carried out on these samples are performed by laboratories approved by the Ministry of Agriculture, Food and Forestry on the basis of a clear statement of requirements, including accreditation by the French Accreditation Committee (Cofrac) in accordance with Standard NF EN ISO/CEI 17025. Only these laboratories are authorised to analyse samples taken within the framework of official controls. The network of laboratories is coordinated by the National Reference Laboratories (NRLs), which develop and validate official methods, provide technical support to laboratories, and ensure their technical proficiency to perform analyses. Some of these NRLs carry out official analyses themselves as part of the SCPs: in the event of development of a new method

Table 2. SCPs for plant protection products in foodstuffs of animal origin in 2014 and 2015 in France

	Meat (beef, pork, lamb and mutton, goat's meat, horse meat)			Poultry			Farmed fish	Rabbits	Game	Dairy products			Honey
	Muscle	Fats	Liver	Muscle	Muscle and fats	Liver	Meat	Muscle	Muscle	Milk	Butter	Eggs	
Carbamates	A			A									
Pyrethroids		A	T	A	T	T	A	A	A	A	T	A	A
Organochlorines		A	T	A	T	T	A	A	A	A	T	A	A
Organophosphates		A	T	A	T	T				A	T	A	A
Other pesticides			T		T	T	A				T	T	A
Neonicotinoids													A

A: annual; T: triennial

Table 3. Size of samples and number of analyses performed for the surveillance plans of plant protection products in foodstuffs of animal origin for 2014 and 2015 in France

	Target population annual mean	Size of the minimum annual national sample required by regulations for the testing of plant protection products		Size of the effective annual national sample		Number of results for pesticide residue concentration rates obtained 2014+2015
		N	Proportion (%)	2014	2015	
Cattle	4,775,000 (total number of cattle slaughtered over 12 months)	430	0.009	450	450	47,200
Pigs	23,933,000 (total number of pigs slaughtered over 12 months)	430	0.002	500	450	40,000
Small ruminants	4,472,000 (total number of sheep and goats slaughtered over 12 months)	90	0.002	100	60	10,000
Horses	19,000 (total number of horses slaughtered over 12 months)	Absence	Absence	10	5	1,000
Poultry	1,703,000 tonnes produced over 12 months	255 (batches)	0.01	505	445	42,000
Rabbits	46,000 tonnes produced over 12 months	10 (batches)	0.02	5	5	300
Farmed fish	50,000 tonnes produced over 12 months	Absence	Absence	30	90	3,000
Farmed game	3000 large game (red deer, roe deer, fallow deer) 9000 tonnes of small game (pigeon, quail, partridge, pheasant) slaughtered over 12 months	Absence	Absence	5	5	1,800
Milk	24,703,000 tonnes collected over 12 months	Absence	Absence	70	40	8,000
Butter		66: every 3 years		0	66	
Eggs	772,000 tonnes produced over 12 months	Absence	Absence	70	90	8,000
Honey	11,800 tonnes produced over 12 months	0.3 %	35	50	50	
TOTAL				1,795	1,756	161,300

or testing on a new matrix (e.g. detection of pesticides in butter or analyses of pesticides in honey).

Surveillance and control plans implemented in 2014 and 2015

Council Directive 96/23/EC, supplemented by Commission Decision 97/747/EC, governs the strategy, the level and the frequency of sampling for the 11 surveillance plans to be implemented in primary production each year, in the following matrices:

- beef, pork, and poultry from the farm and slaughterhouse,
- sheep/goat's meat, horse meat, rabbit, and farmed game from the slaughterhouse,
- farmed fish and milk from the farm or first processing level,
- eggs from the collection site,
- honey from the beekeeper (or elsewhere if traceability to the beekeeper is guaranteed).

The samples are taken unannounced for CPs and preferably targeted at risk criteria. However, given the difficulty in targeting, a random system of sampling was retained for pesticides. They are collected in accordance with the procedures laid down in Commission Decision No 98/179/EC.

Most of the pesticides tested for annually as part of the SCPs belong to the organochlorine, organophosphate, synthetic pyrethroid and carbamate pesticide classes, in line with the requirements of Directive 96/23/EC. However, other groups such as the neonicotinoid or benzoylurea classes may also be tested for in animal matrices known to contain this type of pesticide, or on the strength of implementing regulations regarding the multiannual pesticide control programme (Table 2).

Sampling plan for the SCPs of 2014 and 2015

The number of samples to collect by sector and by sampling site (farm or slaughterhouse) was calculated (Table 3):

- to meet the minimum requirements of Council Directive 96/23/EC, i.e. pro rata of:
 - the number of animals slaughtered for meat and large game,
 - tonnages of animals slaughtered for poultry, small game and rabbits,
 - production volumes for farmed fish, milk, eggs and honey;
- to establish prioritisation based on the number of non-compliant samples detected in previous years.

The choice of substances to be tested by class of contaminants was established jointly with the National Reference Laboratories based on expected risks of use, regulatory obligations, analytical methods used, and their performance.

The sampling strategy implemented jointly by the DGAL and the NRL for pesticides, in line with regulatory obligations, aims to define a representative pesticide residue contamination level for a food group. Although the size of the sampling may appear small compared to the target populations, the power of the methods implemented provides a large number of concentration measurements for a broad range of plant protection substances. The confidence intervals for the results obtained range from 1 to more than 3% depending on the sectors, which is not very precise and difficult to analyse as is. However, the repeatability of this plan can enable possible emerging trends to be identified.

Analytical methods

The official methods can be used to cover about 70 pesticides belonging to different classes.

Official methods

There are currently several multi-residue methods that can determine pesticide levels in foodstuffs of animal origin. These methods are generally based on an extraction protocol of pesticide residues and fats, and are therefore mostly aimed at liposoluble pesticides (Ledoux *et al.*, 2011).

Assays of pesticides are carried out by gas chromatography (GC) coupled with electron capture detectors (ECDs) and nitrogen-phosphorus detectors (NPDs). Although these detectors are still used for searching certain pesticides, mass spectrometry (MS) is now used as a detector coupled with gas chromatography (GC-MS). Laboratories now increasingly use gas and liquid chromatography coupled with tandem mass spectrometry (GC-MS/MS and LC-MS/MS). Recent developments in mass analysers and data processing mean that more accurate and specific assays can be performed for pesticides using techniques such as liquid chromatography coupled with high-resolution mass spectrometry.

Now that mass spectrometry is used routinely in laboratories, broad-spectrum multi-residue methods can be developed.

Broad-spectrum multi-residue methods

The first Quick Easy Cheap Effective Rugged and Safe (QuEChERS) type method was developed in 2003 (Anastassiades *et al.*, 2003a, 2003b). It basically includes three steps: extraction, purification, and detection. Over the past 10 years, QuEChERS methods have evolved to address the specific problems related to foodstuffs of animal origin. The European Union Reference Laboratory (EURL) for pesticides in food of animal origin and commodities with high fat content, along with the NRLs are working on these types of methods known as broad-spectrum methods because they not only have the advantage of screening a large number of pesticides of low to high polarity, but are also rapid and effective. These methods, developed and then validated according to the SANCO 12571/2013 guidance document by NRLs, can be applied to samples in the context of SCPs.

Results

A non-compliant result indicates either the simple presence of pesticide residues, when the substance of interest has been banned from use, or the presence of residues at a level higher than the MRL for authorised products.

For 2014, the overall results of the surveillance and control plans carried out in France did not reveal any non-compliance. In 2015, in 1622 samples (3034 analyses performed), two cases of non-compliance were identified: one in a muscle sample of beef (0.031 mg/kg), and the other in an egg sample (0.03 mg/kg). This involved contamination by hexachlorocyclohexane (the chemical name of lindane), for which the MRLs are 0.02 mg/kg in muscle and 0.01 mg/kg in eggs. Lindane is an organochlorine insecticide that was marketed starting from 1938. This substance has a very broad spectrum of insecticidal activity against plant-eating insects, soil-dwelling insects, and human and animal parasites. Lindane was therefore used widely in agriculture, and in pharmaceutical products for the treatment of scabies and the elimination of lice.

In France, lindane has been discontinued in agriculture since 1 July 1998, and since 2009 in the rest of the world. There are no plant protection products containing lindane authorised for sale. However, concerning the use of pesticides that are not authorised in the EU, Regulation (EC) No 396/2005 sets MRLs at a sufficiently low level to protect consumers against ingestion of pesticide residues given the persistence of some of these substances in the soil.

The investigations carried out at the farm level were not able to identify the source of contamination.

For the non-compliant beef muscle, the investigation carried out at the farm (a small-scale site with about 20 animals) was not able to identify sources of contamination via:

- environmental pollution (the production site is in a mountain environment with no nearby industry or craft trades),
- food (only hay produced on-site and mineral supplements in the form of a mineral lick),
- veterinary medicinal products (only Closamectin Pour-on). The hypothesis of treatment of surrounding trees was considered but not confirmed.

Concerning the non-compliant chicken egg sample, the investigation carried out in an outdoor holding was also unable to identify the source of contamination via food or drinking water. The hypothesis of soil pollution was retained. The producer has subsequently stopped outdoor egg production.

Comparison of data with plans in other European countries

According to the 2015 EFSA annual report that summarised, among other data, the results of analyses for regulated pesticides in foodstuffs of animal origin for 2013 for all European countries, of the 8257 samples analysed, 25 (0.3%) showed values exceeding the MRL (Table 4). The most commonly identified or detected pesticides were hexachlorobenzene, DDT, thiacloprid, lindane, endosulfan, amitraz and pirimiphos-methyl. For the most part, these products, like the organochlorine substances, are no longer used in Europe but are frequently found given their persistence in the environment.

For 2013, there were no samples with levels exceeding the MRL among the 1021 cow milk samples analysed. In contrast, a few pesticides were found in trace amounts. This involved hexachlorobenzene and DDT, both of which have been banned since 1979. A similar result was found for the 753 pork muscle samples analysed.

Discussion - Outlook

None of the results for 2014 in France showed cases of non-compliance across all the plans implemented. The results for 2015 were also satisfactory with a non-compliance rate between 0.3% (beef plan) and 1.2% (egg plan). In both these cases, the pesticide residue found was lindane. Further to the investigations carried out at the production site, it appears that the presence of this substance in the samples was not due to its use, but rather its persistence in the environment in the case of eggs.

To ensure more in-depth future investigations and to confirm or rule out the hypothesis of soil contamination, a procedure is under consideration to sample and analyse soil for this type of persistent pollutant.

The results obtained for the various national surveillance and control plans in France are comparable to those reported in other Member States, i.e. a low level of contamination by pesticides in foodstuffs of animal origin. However, some cases of non-compliance were recorded and are probably more likely related to environmental contamination than use of the pesticides themselves. Currently, the official methods cover about 70 pesticides belonging to various classes. Over the last few years, new pesticides have been produced by manufacturers and are used by agricultural producers. The list of pesticides to test has therefore evolved in Europe. National Reference Laboratories are working on developing methods that are more rapid with a broad spectrum. These will be used to extract a larger number of pesticides to better evaluate contamination of foods of animal origin.

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Table 4. Results for detection of pesticides in foodstuffs of animal origin in Europe in 2013 exceeding the MRL

Foodstuff/pesticide	Origin of foodstuff	Number of non-compliant samples (/values > MRL)	Residue levels (mg/kg) min - max	MRL (mg/kg)
Honey		6/2		
Azoxystrobin	Denmark	5/2	0.011 - 0.086	0.01
Thiacloprid	Austria	1/0	0.233	0.2
Game		4/0		
DDT	Denmark	4/0	0.057 - 0.095	0.05*
Chicken eggs		3/3		
Lindane	Austria	2/2	0.254 - 0.295	0.01*
DDT	Denmark	1/1	0.209	0.05
Pork fat, beef muscle, poultry muscle		5/4		
Permethrin	Estonia	3/3	0.077 - 0.183	0.05*
Methoxychlor	Estonia, Belgium	2/1	0.018 - 0.021	0.01*

(*) Value corresponding to the limit of quantification (LOQ) of the analytical method

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