

CRL for Cereals and Feeding Stuff

National Food Institute

Danish Technical University



Cereals and feeding stuff
– production, consumption and pesticides
(Version 4)

Karen Hjorth
Susan Strange Herrmann,
Hanne Bjerre Christensen
and Mette Erecius Poulsen

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1. Introduction

One of the most important tasks of the Community Reference Laboratory (CRL) for Pesticides in Cereals and Feeding stuff (CRL-CF) is to give advice to the Commission concerning the prioritisation of the work on method development. To enable the prioritization, various information has been collected and evaluated. The information concerns the following subjects within the Member States (MS):

- Production
- Consumption
- Pesticides authorised for use in cereal production
- MRLs for pesticide in cereal and toxicological data on the authorised pesticides, e.g. ADI and ARfD
- Intake of pesticides from cereals
- Feasibility to include the pesticide in multi residue method

This document (version 4) is an update of the document prepared in 2007 (version 1).

The analysis of undesirable contaminants in various food and feed samples is nowadays a problem of primary concern for quality control laboratories due to human and animal health risks associated with the accumulation of these substances. Contaminants of animal feed can cause harmful health effects in the animals and may be harmful to people through secondary exposure of consumers to contaminants deriving from these animals. In the European Union (EU), feeding stuffs are subject to legislation covering their composition, manufacture, storage, transport and usage¹. Because of the above listed the 3rd version was extended with a chapter on feeding stuff including sections on consumption of feeding stuff and import of feeding stuff in regard to a future monitoring program on feeding stuff.

This year's update of our report "Cereals and feeding stuff– production, consumption and pesticides" will primarily focus on with pesticide residue data for animal feeding stuff. However, published data on feed are very scattered and not easy to find and a compilation of feed monitoring data are still in the early stages. Pesticide residue data of animal feed would however be very useful for example when designing pesticide monitoring in products of animal origin. It would give a better idea of which pesticides to search for in the animal matrices.

2. Cereals

The MS with the largest production of cereals in 2006 were France, Germany, Poland, United Kingdom and Italy. The total cereal production in these countries amounted to 62, 44, 22, 21 and 19 million ton, respectively and the main producers of wheat were France, Germany, UK, Poland and Romania. The production figures for cereals in 2006 are presented in more detail in Table 2.1 (the figures are from “Agriculture in the European Union – Statistical and economic information 2007”²). The production figures are presented for the five largest producers of cereal in total, wheat, barley, oat, rye, maize and rice.

Table 2.1. Production figures for EU-25^a and the six largest producers of cereals in total, wheat, barley, oat, rye, rice, and maize in the European Union in 2006².

	Cereal in total ^b	Common Wheat	Durum wheat	Barley	Oat ^d	Rye ^c	Maize	Rice
EU-25^a	245.2	108.6	8.9	54.8	11.1	6.7	44.8	2.6
Austria			0.8					
Czech Rep.								
Denmark				3.3		0.1		
Finland					1.1			
France	61.6	33.3	2.1	10.4		0.1	12.9	0.1
Germany	43.5	22.4		12.0	0.9	2.7	4.1	
Greece			1.1					0.2
Hungary							8.3	
Italy	18.7		3.9				9.7	1.4
Poland	21.8	7.1			4.2	2.8		
Portugal								0.2
Romania		5.5						
Spain			1.6	8.3	0.9	0.2	3.5	0.8
Sweden								
UK	20.9	14.7		5.3	0.7			

^a: EU-25: Including 25 member states. ^b: Total cereal production not including rice. ^c: including meslin. ^d: including mixed cereals.

2.1. Consumption of cereals in the EU-member states

Various consumption data are available from different countries consumption surveys but unfortunately it is not all countries that have consumption data available. In 2006 EFSA initiated “The temporary MRL exercise”. The goal of this exercise was to identify for each combination of active substance/commodity the highest reported national MRL (Maximum Residue Limits), and to set harmonized temporary EU MRLs to these highest figures. Before this was possible the temporary EU MRLs had to be risk assessed.

To be able to perform a risk assessment covering all EU citizens’ consumption data from all EU MS was collected and reported³. The consumption data includes consumption rates reported by different MS and the consumption rates according to different WHO diets. In Table 2.2 are the highest reported chronic consumption rates for the different types of cereals presented, which model/MS it originates from and the average consumption of all the reported data for the different types of cereals.

Table 2.2. The highest chronic consumption of the different types of cereals reported for an EU-citizen and the average consumption of all reported consumptions.

Commodity	Highest reported consumption (g/kg bw/day)	Model/Reporting MS	Average of all reported consumptions (g/kg bw/day)
Cereals (total)	11.89	WHO Cluster diet B	4.71
Barley	1.24	IE adult	0.17
Buckwheat	0.28	IE adult	0.02
Maize	2.47	WHO Cluster diet B	0.31
Millet	0.09	WHO Cluster diet D	0.01
Oats	0.40	DK child	0.07
Rice	0.79	PT general population	0.30
Rye	4.42	DK child	0.38
Sorghum	0.02	DE child	0.001
Wheat	8.54	WHO Cluster diet B	3.33
Other cereal	1.50	IT kids/toddler	0.08

In Table 2.3 is the chronic dietary pesticide intake of cereals in g/person/day for a few MS presented. The EU citizens represented here both in Table 2.2 and Table 2.3 consumes more wheat than any of the other types of cereals. The average of all reported consumption of wheat (3.3 g/kg bw/day in table 2.2) corresponds with the average of

the chronic intakes reported by the different MS (table 2.3). According to the Danish and German consumption data, rye accounts for the second largest fraction of the total daily cereal consumption. Rice accounts for the second largest fraction of the total daily cereal consumption when referring to the data covering France and UK.

Table 2.3. The chronic dietary intake of cereals in g/person/day according to models often used for risk assessment of the dietary intake.

	Consumption (g/kg bw/day)							
	Cereals in total	Barley	Buck-wheat	Maize	Oat	Rice	Rye	Wheat
Denmark⁴								
Adult (74 kg bw)	2.89				0.11	0.086	0.68	2.0
Children (22 kg bw)	10.41				0.40	0.10	4.42	5.51
France⁵								
General population (60 kg bw)	3.52	0.01				0.11		3.28
Toddler (10.6 kg bw)	2.98					0.36		2.62 ^a
Germany⁶								
Children Age 2-5 (16.5 kg bw)	5.45	0.01	0.01	0.15	0.2	0.26	0.78	4.04
UK⁷								
Adults (76 kg bw)	2.09	0.003	L/C	0.003	0.02	0.37	0.01	1.67
Young children/Toddler (14.5 kg bw)	4.55	0.01	L/C	0.01	0.05	0.58	0.01	3.94

^a: The type of commodity stated in the reference is "bread" and it is assumed that all the bread is wheat bread.

Thus, since the cereal consumed in largest amounts is wheat, wheat could also be the type of cereal contributing most to the human exposure to pesticide residue. This is though depending on the frequency and amounts with which residues are found in wheat compared to the other types.

2.2. Pesticides authorised for use in cereals

When prioritising the work on method development it is relevant to consider which pesticides are authorised for use on cereal commodities since residues of these pesticides could be expected. A complete list of all authorised uses in the different MS would be a helpful tool. However, such a list is not available. Lists of which pesticides are authorised for use in the MS are available on the EU CIRCA Database. Unfortunately, this list does not include information on which crops the pesticides are authorised for and it is therefore not possible to evaluate which pesticides are authorised for which types of cereals in which MS. Additionally, no information is given on how frequently the pesticides in fact are used on cereals.

Information on authorisations are however available but these are non-exclusive since they only include information about some MS or for a part of the pesticides available on the European market.

The Faculty of Agricultural Sciences, Aarhus University, Denmark has as part of a project financed by the Danish EPA compiled lists of pesticides authorised for use in wheat and four other commodities in the different northern and central EU MS. The report in Danish was published by the Danish EPA in 2007⁸. In Table 2.4 a list of pesticides authorised for use in wheat in ¹⁾ more than 14 MS and ²⁾ more than 9 but less than 14 of the 17 MS evaluated is shown. Data on the frequency or which amounts of the pesticides have been used is not available.

From Table 2.4 it can be seen that the triazoles are an often authorised type of fungicide in the northern and central European MS. In the group of herbicides the sulfonyl urea type are widely represented. The list of authorised insecticides vary from MS to MS which results in only one insecticide authorised in 14 or more of the 17 MS. The most frequently authorised type of insecticide was the pyrethroids. In the 17 MS were only seven different plant growth regulator authorised for use on wheat. The plant growth regulators authorised in most MS were chlormequat, trinexapac and ethephon. These plant growth regulators were authorised for use on wheat in 15, 14 and 12 of the 17 MS, respectively. Only two types of insecticides are authorised for use in wheat in France. These are not represented by the active substances authorised for use in more than 9 other MS.

Table 2.4. A summary of data⁸ on which fungicides, herbicides, insecticides and plant growth regulators are authorised for use in wheat in ¹⁾ 14 or more and ²⁾ more than 9 but less than 14 of 17 northern and central European member states + France.

	Pesticide authorised for use in wheat	
	≥14 MS	14 > MS > 9
Fungicides	Azoxystrobin Epoxiconazole Fenpropidin Fenpropimorph Kresoxim-methyl Prochloraz Propioconazole Spiroxamine Tebuconazole Trifloxystrobin	Carbendazim Carboxin Chlorothalonil Cyproconazole Difenoconazole Fludioxonyl Fluoxastrobin Flutriafol Guazatine Mancozeb Metconazole Picozystrobin Prothioconazole Pyraclostrobin Triadimenol Triticonazole
Herbicides	2,4-D Amidosulfuron Dichlorprop-P Fenoxaprop-P Florasulam Fluroxypyr Glyphosate Iodosulfuron-methyl-sodium MCPA Mecoprop-p Pendimethalin Sulfosulfuron	Bentazone Carfentrazone-ethyl Dicamba Diflufenican Flupyr-sulfuron methyl Isoproturon Metsulfuron Propoxycarbazone Thifensulfuron Triasulfuron Tribenuron
Insecticides	Lambda-Cyhalothrin	Alpha-cypermethrin Deltamethrin Esfenvalerate Pirimicarb
Plant growth regulators	Chlormequat Trinexapac	Ethephon

In connection with the work performed by EFSA with setting TMRLs, information on authorised uses of 77 pending substances from 20 MS have been collected. Authorised uses have been collected for e.g. wheat (including spelt and triticale), oat, rye, rice, maize and barley. The collected data include information on authorised uses from 13 northern European MS and 7 southern European MS. The complete lists are available on the CIRCA database⁹. In Table 2.5 and table 2.6 are listed the top three most often authorised pesticides for each of the cereals types; wheat, oat, rye, rice, maize and

barley. In many cases more than one pesticide is authorised in equal number of MS. More than one pesticide can therefore in Table 2.5 occur as e.g. the second most often authorised pesticide. The number of MS in which the top three pesticides are authorised is presented in brackets in the tables.

Table 2.5. The group of most often authorised pesticides for use in cereals in 13 northern European member states. In brackets is the number of MS the pesticide(s) are authorised for use in cereals in presented^a.

List of authorised pesticides in NORTHERN^a European MS.			
	1	2	3
Wheat	Fenpropidin (11)	Metconazole Tebuconazole (10)	Cyproconazole Epoxiconazole Fludioxonyl (9)
Oat	Tebuconazole (7)	Carboxin Fludioxonyl Pirimicarb (6)	Epoxiconazole Zeta-cypermethrin (5)
Rye	Fludioxonyl (9)	Epoxiconazole Tebuconazole (8)	Cyproconazole Flusilazole (7)
Rice^b			
Maize	Fludioxonyl Terbuthylazine (5)	Carboxin (4)	Glufosinate (3)
Barley	Fenpropidin Tebuconazole (10)	Cyproconazole Epoxiconazole Fludioxonyl Metconazole (9)	Flusilazole Flutriafol (8)

^a Including Austria, Belgium, Czech republic, Denmark, Estonia, Germany, Latvia, Luxembourg, Poland, Slovak Republic, Sweden, The Netherlands and United Kingdom.

^b Only few pesticides authorised for use in UK. No pesticides are authorised for use in the other northern member states.

Table 2.6. The group of most often authorised pesticides for use in cereals in 7 southern European member states. In brackets is the number of MS the pesticide(s) are authorised for use in cereals in presented⁹.

List of authorised pesticides in SOUTHERN^a European MS.			
	1	2	3
Wheat	Tebuconazole (6)	Diclofop Epoxiconazole Flusilazole Pirimicarb (4)	Cyproconazole Diniconazole Fenbuconazole (3)
Oat	Pirimicarb (4)	Cyproconazole Epoxiconazole Tebuconazole (3)	Carboxin Metconazole (2)
Rye	Cyproconazole Epoxiconazole Pirimicarb Tebuconazole (3)	Metconazole (2)	
Rice	Oxadiazon (5)	Propanil (4)	Tebufoenozide (2)
Maize	Tefluthrin Terbuthylazine (5)	Ethoprophos Fludioxonyl Zeta-cypermethrin (3)	Carboxin Malathion Trichlorfon (2)
Barley	Tebuconazole (5)	Diclofop Epoxiconazole Flusilazole Pirimicarb (4)	Cyproconazole Diniconazole Tetraconazole (3)

^a Including Greece, France, Hungary, Italy, Portugal, Slovenia and Spain.

^b Other authorised pesticides only authorised in one MS.

Table 2.5 and table 2.6 show that the triazole type pesticides are often authorised for use in cereals. This is especially true for wheat, oat, rye and barley. Many of the pesticides are authorised for use both in the northern and southern EU. Even though a pesticide is authorised for use it is not given that residues will be found.

The participating states in the EU monitoring program have reported the ten most frequently found pesticides¹⁰. The most frequently found pesticides in wheat were mainly insecticides in 2006, and this is in line with the findings of previous years. More specifically the most often found compounds using multi residue method were: Primiphos-methyl, chlorpyrifos-methyl, deltamethrin, malathion, dichlorvos, chlormequat, piperonyl butoxide, chlorpyrifos and permethrin. Most often found compounds for single residue methods were: Chlormequat, hydrogen phosphide,

mepiquat, glyphosate, bromide, benomyl group, spiroxamine, maneb group, trinexapac ethyl, and phosphine.

The majority of these pesticides often found in the EU monitoring program are also represented in the table of often authorised pesticides (Table 2.4 –table 2.6). 3 compounds are identical concerning most frequently findings and authorised for use: spiroxamin, glyphosate, and chlormequat. A reason for the lack of connection could be that not all authorised pesticides are included in the monitoring programs. Another reason could be that monitoring programs often are made up based on the fruit and vegetables programs and in this way important pesticides are left out in the monitoring program for cereals. Finally the list of authorised pesticides may include pesticides that are induced early in the season and therefore are not found at the time of harvesting.

Each year the EU monitoring program contains one kind of crop which is renewed each year. In 2004 it was rye/oat and in 2006 it was wheat which represented the cereals¹¹. Table 2.7 presents in percent the results obtained for these two commodities in the EU monitoring program, in regard to samples without, with residues below or at MRL and with residues exceeding the MRL.

Table 2.7. Residues found in wheat and rye/oat analysed by the EU co-ordinated monitoring program¹¹.

	Without detectable residue %	With residues below or at MRL	With residues above MRL
Wheat (2006)	73	27	0.1
Rye/oat (2004)	81	19	0.6

More wheat samples are found to contain residues below or at MRL compared to rye/oat. However on the other hand exceeding of the MRLs were more often found for rye/oat compared to wheat. Since rye and oat also often is used as feed material and residues are found in quantity similar to wheat a monitoring of this crops would be interesting for a future monitoring program for feed.

2.3. Intake of pesticides from cereals

Intake calculations are not available for many European Countries and if they are available they are often not easily digested down to the intake of pesticides from cereals. The focus is often on the intake from fruit and vegetables since the primary intake is from these food items.

The intake of pesticide residues from cereals has been evaluated on the basis of Danish consumption data and on the results of the Danish monitoring program¹². The total

intake of pesticide residues from cereals was estimated to 18 $\mu\text{g/day/person}$ in 2007. The intake from wheat alone was estimated to 15.4 $\mu\text{g/day/person}$. The estimated intake from cereals accounted for 21% of the estimated total intake of pesticide residues from food and beverages which was 83 $\mu\text{g/day}$.

Intake calculations from cereals based on cereal consumption data and monitoring data from European countries besides Denmark is a challenge and have been lacking in previous versions of the paper. The reason for this is that results from the EU coordinated monitoring program and the MS national programs are reported by the individual MS in intervals and intake calculations can not be calculated on such intervals.

Earlier in this document it has been concluded that EU citizens in general consume more wheat than any of the other types of cereals. It has also been concluded that the consumption of wheat for an average EU citizen is in the same order as for a Danish citizen. So if it also could be documented that the residues found in Denmark and other EU member states are similar (quantitatively and qualitatively) the intake of pesticides from cereals by EU citizens could be estimated to be similar to the estimated Danish intake.

The studies available in the literature are cases studies reporting the intake of a certain group of pesticide¹³, e.g. organochlorine pesticides or dithiocarbonates. We have therefore looked at the results in the EU monitoring report from 2006¹⁴ and will compare with the monitoring data used for estimating of the Danish intake. In 2006 were 1531 samples of wheat analysed, 1112 samples were without detectable residues (73%), 417 samples were with residues below or at MRL (40.8%), and 2 were with residues above MRL (0.1%). The main pesticide-commodity where detectable residues were found most frequently in wheat was chlormequat and pirimiphos-methyl, in 36.41% and 10.27% of wheat samples, respectively (all other pesticides occurred in $\leq 5\%$ of the samples).

In comparison, data from the Danish monitoring program revealed that chlormequat and pirimiphos-methyl were found in 33% and 17.5% of the wheat samples. This should be hold up against the average consumption of wheat for an EU-citizen, 3.3 g/kg bw/day and a Danish citizen, 2.0 g/kg bw/day Table 2.3. Danish people have in many years eaten less wheat than an average EU-citizen instead they eat more rye and oat. But in 2007 wheat was the second most important crop concerning the residues of pesticide in Denmark and rye bread was only number 20 on that list which means that in this perspective the Danish diet are more similar to the European diet than ever before. And because the most frequently found pesticides are the same and found with same frequency and because the consumption of wheat is also in the same order (Danish

versus EU) it would be expected that the estimated intake for Danish citizens is similar to the average intake of the EU citizens.

3. Feeding stuff

Within the EU-25 about 450 million tons of feeding stuffs are consumed by livestock each year. Of this quantity 215 million tons mostly are roughages grown and used on the farm of origin. The balance, i.e. 235 million tons of feed, includes cereals or other feeding stuff grown and used on the farm of origin (51 million tons) and feed purchased by livestock producers to supplement their own feed resources (either feed materials or compound feed)¹⁵.

The market for feeding stuffs depends on the market for livestock products and vice versa. In 2006, the EU-25 livestock population produced 45 million tons of meat (thereof 8 million tons of beef, 21 million tons of pork and 11 million tons of poultry meat), 131 million tons of milk and 6 million tons of eggs. Average per capita consumption of meat in 2006 was 93.4 kg, compared to only 50 kg in the EC-6 during the late 1950s. The value of livestock production - amounting to €130 billion - accounts for 42% of the overall EU-25 agricultural output amounting to €309 billion in 2006.

Germany is the leading cattle meat producer, Spain is now the leader for pig meat and France clearly breaks away from the other countries for poultry meat production¹⁶. The primary producers of milk in the EU are Germany and France. The countries having a large production of meat, milk or other animal products must also have a high production/import of fodder.

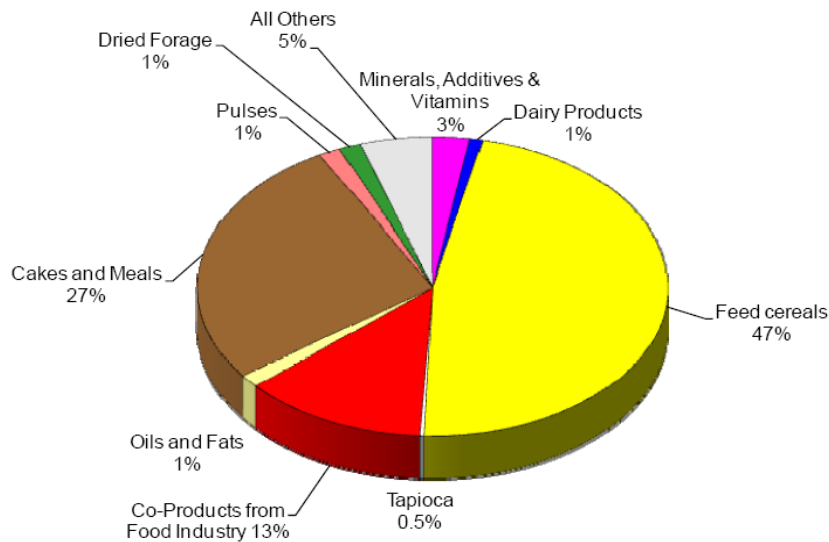
3.1. Consumption of feeding stuff in EU

Feeding stuff may be comprised of a wide range of ingredients. In council directive 92/25/EC of 29 April 1996 is presented a non-exclusive list of the main feed materials. This list include cereal grains, a wide range of by products from different cereal processing's, e.g. cereal bran and middling, a wide range of different by-products of the manufacturing of oil (e.g. rapeseed, palm kernel, soybeans, cotton seed, sunflower seeds, linseeds, cocoa bean), sugar (e.g. sugar beet pulp and molasses), different potato products (e.g. pulp, starch, protein, flakes), fruit juice (e.g. citrus pulp, tomato pulp). The list also includes legume seeds, alfalfa, clover, grass and cereal straw¹⁷.

Some of the information and illustrations in the following are from a presentation by Finn Vestergaard from DLG given at CRL workshop in Copenhagen in 2008. The presentation is available at <http://www.crl-pesticides.eu>.

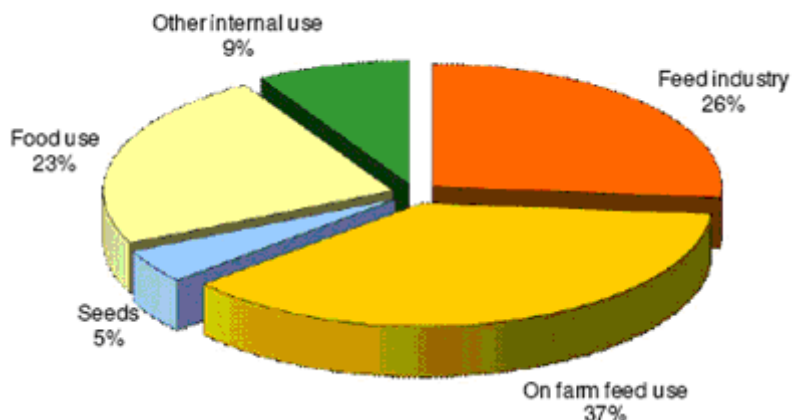
Figure 3.1 shows the feed material consumption by the EU compound feed industry. The proportion of feed materials per category has remained relatively stable (47% for cereals, 27% for oilseed meals) compared to previous years¹⁸, and feed cereals account for almost half of the raw materials in the production of compound feed.

Figure 3.1. Feed material consumption by the compound feed industry in 2007 in the EU-27¹⁹.



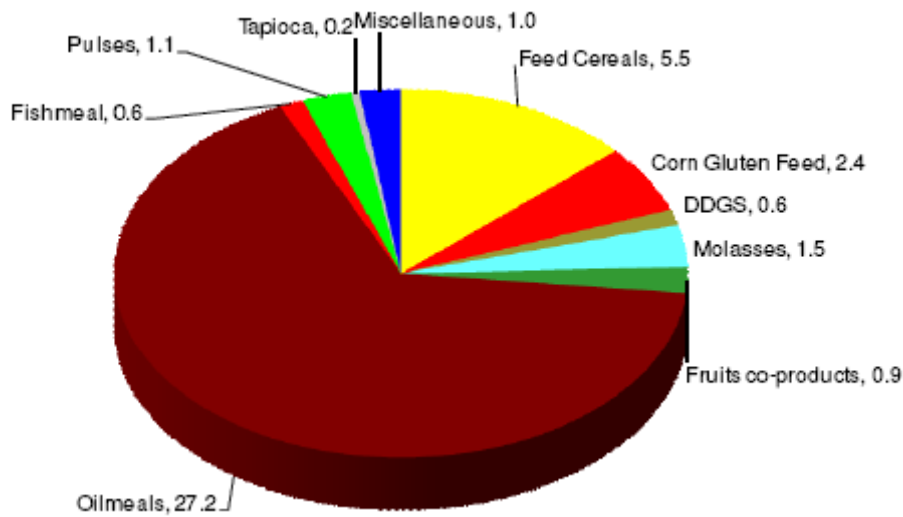
The production of feed accounts for a large part of the agricultural activity in the EU. Approximately 120 million tons of feeding stuffs are produced in the EU each year²⁰. The production of cereals was in 2007 spilt up between food uses and feed as described in Figure 3.2. The term 'on farm feed use' means that the farmer uses his own crop to feed his animals, which means that the feed never leaves the farm. Thus by far the largest function of the cereal use in EU is for feed.

Figure 3.2: Usage of cereals in the EU27 in 2007-08¹⁹.



Some important ingredients which are not feasible to grow in the EU are imported from third countries. These diverse sources of raw material supplies are an important factor in the industry's ability to manufacture feeds of both high quality and at competitive prices for livestock farmers. Figure 3.3 shows import of feed materials in the EU from 2000-2005. As it is seen from the figure it is primarily oil meals which include soy meals which are imported into EU.

Figure 3.3. Imports of feed materials in the EU-25 in 2006¹⁹.



EU is to a large extent self sufficient in production of cereals (Table 3.1) which means that a monitoring program for cereals should focus on cereals produced within the EU. In contradiction does the self sufficiency of soy within EU almost not exist (between 6 and 18 %), so for this crop the focus should be outside EU.

Table 3.1. Self sufficiency in production of cereals²¹

	Total cereals	Common wheat	Durum wheat	Barley	Rye	Maize	Oats ^a
Human consumption (%)	23	40	87	0.7	42	6	17
Animal feed (%)	63	49	4	75	30	83	75
EU Self sufficiency (%)	98	101	88	106	89	92	89

^a: Including mixed corn summer cereals

As the fourth most produced grain in the world, after maize, wheat and rice, soybean has the highest content of proteins. Besides its potential as a nutrient for both humans and animals, this legume also enriches the soil with nitrogen through biological processes. Only four countries, the USA, Brazil, Argentina and China, are responsible for approximately 90% of the world production and these countries are also nearly EU's exclusive trading partners for soybeans and soy meal imports. Over recent years, there has been an increase in worldwide soybean production, as well as in the import/export and processing of soybeans. Brazil is the second largest producer of soybeans in the world. Much of the soybean production in Brazil, around 19 million tons of the 2004/2005 crops, is exported to several countries on different continents²². The degree of self-sufficiency in EU varies between 6% (soy meal) and 18% (soy oil) in 1998/99²³.

3.2. Composition of feed

When establishing a pesticide control for feed an evaluation of the division between human consumption/animal feed versus the daily feed ration for animal is needed to estimate which feeding stuff to attach importance in the control. A starting point could be to focus on the crop/products which make up the largest fraction of an average feed ration for a given type of livestock. An important crop for animal feed is maize. Table 3.1 show Europe is almost self sufficient and that animals consume 83 % of the production of maize. An example is given in Table 3.2 where almost half of the daily feed ration for a Danish dairy cow is maize silage. Besides maize half of the wheat production in EU is consumed by animals and 75 % of barley and oat production. But the production of barley and oat is smaller than the production of maize, wheat, and rice.

Table 3.2. Daily ration for a Danish dairy cow¹⁹.

	Kg Dry matter
Clover grass silage	3.5
Maize silage	9
Straw	0.5
Minerals	0.2
Compound feed	7
Total	20.2

According to Table 3.3 the proportion of soy in animal feed can be 10-30% of the dry matter depending on the livestock species which is in the same order of magnitude as what can be estimated from the information in Table 3.2. Since about 30% of the 7 kg drymatter in a daily portion for a dairy cow is accounted for by cakes and meals.

Exceedings found in soy products from the UK monitoring are listed in an Annual Report of the Pesticide residues committee 2006 of samples collected throughout 2006. The main findings were:

- Out of 60 samples of soy milk, five samples contained endosulfan.
- Out of 60 samples of soy pieces and tofu, 11 contained residues of glyphosate, five contained residues of endosulfan and one contained residues of diazinon²⁴.

In Guidance document for Directive 91/414, Appendix G a table give examples of how a feed for pig, beef cattle, dairy cattle and hens can be comprised. The table is included in this document as Table 3.3 According to this table beef and dairy cattle can be feed on a 100% grass, silage or hay. A major constituent in feed for pigs can be grains (up to 80%) and root and tuber vegetables (e.g. sugar beet)(up to 60%).

Table 3.3. Maximum feed intakes expressed in percentages terms for certain feeding stuffs frequently used in the nutrition of the four indicator livestock species. The table is from the Guidance document for Directive 91/414, Appendix G: Livestock feeding studies.

	% Dry Matter (DM)	Chicken	Dairy Cattle	Beef Cattle	Pig
Body weight		1.9 kg	550 kg	350 kg	75 kg
Daily Maximum Feed (Dry Matter) DM		120 g	20 kg	15 kg	3 kg
Maximum Percentage		% DM	% DM	% DM	% DM
Group Crop/Commodity					
I Green Forage (include. Hay)					
Grasses	20	-	100	100	-
Alfalfa/Clover	20	-	40	40	15
Forage Rape	14	-	-	35	15
Kale/Cabbage	14	5	35	35	15
Sugar Beet leaves and tops	16	-	30	30	25
Silage (Clover, Grasses (...))	20	-	100	100	15
Fruit Pomace (Apples, Citrus)	23	-	10	30	-
Hay	85	-	100	100	15
II Grains					
Grains except Maize	86	70	40	80	80
Maize	86	70	30	30	40
Bran (Wheat and Rye)	89	15	20	20	20
III Straws (cereals)	86	-	20	50	-
IV Pulses	86	30	20	20	40
V Root and Tubers (e.g. Potatoes, Swede/Turnip/ Sugar and Fodder Beet)					
(e.g. Potatoes)	15	20	30	60	60
Swede/Turnip/	10	20	30	60	60
Sugar and Fodder Beet	20	20	30	60	60
VI Oil Seed (Meal, Cake) (eg Soya bean, Peanuts, Rape seed, Sunflower seed, Linseed)	86	10	30	30	20

3.3. Pesticides authorised for use in feeding stuff

The difficulties with pesticide residue analysis of animal feed samples are caused by the fact that these matrices are burdened with large quantities of interfering matrixes after extraction. Animal feeds can be complex mixtures that include constituents such as grains, milling by-products, added vitamins, minerals, fats, and other nutritional and energy sources. Even simpler cereal matrices contain much more co-extractants than typical matrices of high water content such as fruits and vegetables. Additionally, the exact composition of the sample is often unknown to the testing laboratory.

The report published by the Danish EPA in 2007 mentioned in section 2.2 also includes information on pesticides authorised for use in rape and maize²⁵. Rapeseed and maize are potential feeding stuff constituents. Meal cake of rapeseed and other by products from the rapeseed oil production is also used as fodder. The information on pesticides authorised for use on rapeseed and maize is summarised in Table 3.4 and Table 3.5.

Barley is also a potential feeding stuff constituent and from Table 2.5 it is indicated that the analysis of e.g. several triazoles could be of relevance when analyzing barley.

The pesticides included in the top 3 list of pesticides authorised for use in maize (Table 3.5) is not all included in the list of most often authorised pesticides in Table 2.5. This indicates that several of the most often authorised pesticides for use in maize are not included in the list of the 77 substances pending in the EU authorisations system.

Table 3.4. Pesticides authorised for use in rapeseed in ten or more of 17 member states and in less than 10 but more than 5 member states.

	Pesticide authorised for use in rapeseed	
	≥10 MS	10 > MS > 5
Fungicides	Iprodione	Azoxystrobin
	Metconazole	Carbendazim
	Prochloraz	Fludioxonyl
	Tebuconazole	Metalaxyl-M
		Procymidone
		Thiram
		Vinclozolin
Herbicides	Clomazone	Dimethachlor
	Clopyralid	
	Cycloxydim	
	Diquat (dibromide)	

	Fluazifop-P	
	Glufosinate	
	Glyphosate (incl trimesium aka sulfosate)	
	Haloxypop-R	
	Metazachlor	
	Napropamide	
	Propaquizafop	
	Propyzamide	
	Quinmerac	
	Quizalofop-P	
	Trifluralin	
Insecticides	alpha-Cypermethrin (aka alphamethrin)	Esfenvalerate
	beta-Cyfluthrin	Methiocarb (aka mercaptodimethur)
	Cypermethrin	Phosalone
	Deltamethrin	Pirimicarb
	Imidacloprid	tau-Fluvalinate
	lambda-Cyhalothrin	Thiacloprid
	zeta-Cypermethrin	Thiamethoxam
Plant growth regulators	Chlorpropham (only 3 MS)	
	Trinexapac (only 3 MS)	

Table 3.5. Pesticides authorised for use in maize in ten or more of 17 member states and in less than 10 but more than 5 member states.

Pesticide authorised for use in Maize		
	≥10 MS	10 > MS > 5
Fungicides		Fludioxonyl Metalaxyl-M Thiram
Herbicides	Bentazone Bromoxynil Clopyralid Dicamba Fluroxypyr	2,4-D Glyphosate (incl trimesium aka sulfosate) Dimethenamide Linuron Isoxaflutole

Foramsulfuron	S-Metholachlor
Mesotrione	Iodosulfuron-methyl-sodium
Nicosulfuron	
Pendimethalin	
Rimsulfuron (aka renriduron)	
Terbuthylazine	
Thifensulfuron (aka thiameturon)	

Insecticides

Carbofuran
Deltamethrin
lambda-Cyhalothrin
alpha-Cypermethrin (aka alphasmethrin)
Imidacloprid
Chlorpyrifos

Plant growth regulators Dimethipin (only 1 MS)
2,4-D (only 1 MS)

The use of pesticides may involve risks and hazards for humans, animals and the environment, especially if placed on the market without having been officially tested and authorized and if incorrectly used. Imported crops from third countries can be problematic in this regard. For example if pesticides which are not aloud in the EU (annex 1, 91/414) are not stated in the application for export and if the same pesticides are not included in the monitoring programs then the respective pesticides are not kept under surveillance. Finn Vestergaard from Danish Cooperative Farm Supply listed a provisional list of problematic pesticide/crop combination for Argentina under EU Regulation 396/2005. All of these pesticide has not been included in Annex I directive 91/414 and are therefore not allowed to use in the EU but they could be relevant to monitor in regard to illegal uses and imported feed.

Table 3.6. List of problematic pesticide/crop combination for Argentina¹⁹.

Soybean	Sunflower	Maize
Acifluorfen	benazolin	atrazina
Benazolin	fenoxaprop	fentoato
Fenpropatine	fenpropatina	fenvalerato
Fentoato	fentoato	imazetapyr

Fenvalerato	fenvalorato	metolacoloro
Fluazifop	Fluazifop	permetrina
Fluoroglicofen	haloxyfop	primisulfuron
Haloxifop	metolacoloro	setoxydim
Imazetapyr	permetrina	dalapon
Metolacoloro	profenofos	simazina
Naptalan	prometrina	endosulfan
Permetrina	setoxydim	
Profenofos	endosulfan	
Prometrina		
Setoxydim		
Endosulfan		

3.4. Pesticide residues in animal feeding stuff

Plant protection products may be ingested or absorbed by livestock in three ways:

- following direct application of the product to the animal
- through residues in feedstuffs
- as a result of treatment of their accommodation.

The usual source of residues is through the legitimate use of pesticides (herbicides, insecticides and fungicides) in the production of crops used in preparation of feeds. The need for information relevant to the conduct of risk profiles or for management of residues will always remain.

Published data on feed are very scattered and not easy to find. The results are not necessarily published and a compilation of feed monitoring data is still in the early stages. The following section contains pesticide residue in animal feed from three different countries, USA, Denmark, and the Netherlands. USA and the Netherlands have collected feed samples from crop/products which include feed rations for a most types of livestock. The data from Denmark consist of cereal samples for feed uses.

Feed samples analysed by the Danish Plant Directory

The Danish Plant Directory controls Danish produced cereals for feeding for pesticide residues. During the last 3 years 165 samples (104 samples in 2007, 30 in 2008, and 31 in 2009) have been analysed for 25 pesticides of which 15 were the most applied pesticides in Danish cereals. In 2008 and 2009 chlormequat, mepiquat and glyphosat were added to the list. The samples were collected at farmers and companies. The cereals analysed were wheat, barley, oat, rye, and triticale. The pesticides analysed are shown in Table 3.7.

Table 3.7. The Pesticide list of the the Danish Plant Directory.

Pesticides		
2-4-5-T	Dimethoat	MCPB
2-4-5-TP-Fenoprop	Epoxiconazole	MCPB-Mecoprop
2-4-D	Fenoxaprop-p-ethyl	Mepiquat
2-4-DB	Fenpropidin	Pendimethalin
2-4-DP-Dichlorprop	Fenpropimorph	Prochloraz
Azoxystrobin	Flamprop-M-isopropyl	Propiconazole
Bentazone	Fluroxypyr	Prosulfocarb
Bromoxynil	Glyphosate	Tebuconazole
Chlormequat	Ioxynil	
Dicamba	MCPA	

No exceedings of MRL for the cereal samples were found in any of the samples in the 3 year period. Out of the 31 samples analysed in 2009, 10 samples contained residues (32 %). Table 3.8 shows the cereal samples from 2009 that contained pesticide residues all below MRL.

Table 3.8. Cereal samples from 2009 with pesticide residues.

Food	Pesticide Residue	Result mg/kg	MRL mg/kg
Barley	Epoxiconazole	0.014	1
Barley	Glyphosate	1.44	20
Barley	Fenpropidin	0.02	0.5
Barley	Propiconazole	0.01	0.2
Wheat	Chlormequat	0.197	2
Wheat	Chlormequat	0.205	2
Wheat	Chlormequat	0.561	2
Wheat	Chlormequat	0.649	2
Wheat	Chlormequat	1.66	2
Wheat	Glyphosate	0.71	10
Wheat	Glyphosate	0.28	10
Wheat	Glyphosate	0.18	10
Wheat	Glyphosate	0.3	10
Wheat	Glyphosate	0.05	10

In 2008 15 out of 30 samples (50%) contained pesticide residues. The pesticides were primarily chlormequat (9 samples) and glyphosate (6 samples) found in triticale, barley

and wheat. In addition to this, one oat sample contained residues of tebuconazole and fluroxpyr was found in a barley sample.

Feed samples analysed by the Institute of Food Safety, Netherlands

In the Netherlands the Food and Consumer Product Safety Authority (VWA) is the competent authority for the official control of feed. As part of this control feed samples are taken at feed producers and as border control. The animal feed samples were collected from 2006-2009, the samples were analysed by RIKILT, Institute of Food Safety, by a multi-residue method for pesticide residues.

In the four year period the RIKILT institute analysed 840 samples. The type of feed samples analysed covered a large variety of animal feed ranging from cereals, grains and seeds, to more complex mixture of compound feed along with by-products from the food industry. The type of feeding stuff analysed also covers the diary of the four most important livestock species, pig, beef cattle, dairy cattle and hens (see table 3.3 in section 3.2 Consumption of feed). The type and number of feed samples are listed in Table 3.9.

Table 3.9. Samples with detectable residues in feed samples from Netherlands.

Type of feed	Number of samples analysed	Samples with detectable residues	%
COMPOUND FEEDINGSTUFFS	95	76	80
BYPRODUCT OF FOODINDUSTRY	62	38	61
CEREAL GRAINS, THEIR PRODUCTS AND BY-PRODUCTS	96	41	43
FAT/OIL VEGETABLE AND ANIMAL MIXED	6	1	17
FISH, OTHER MARINE ANIMALS, AND BY-PRODUCTS (FAT/OIL)	10	5	50
FORAGES AND ROUGHAGE	89	2	2
LAND ANIMAL PRODUCTS (FAT/OIL)	72	1	1
MILK PRODUCTS	2	2	100
OIL SEEDS, OIL FRUITS, THEIR PRODUCTS AND BY-PRODUCTS	154	18	12
OIL SEEDS, OILS FRUITS, (VEGETABLE FATTY ACIDS/FAT/OIL)	139	38	27
OTHER PLANTS, THEIR PRODUCTS AND BY-PRODUCTS	7	2	29
OTHER SEEDS AND FRUITS THEIR PRODUCTS AND BY-PRODUCTS	3	1	33
TUBER, ROOTS, THEIR PRODUCTS AND BY-PRODUCTS	53	13	25
MILK REPLACER	52	34	65
TOTAL	840	272	32

The number of samples with detectable residues were 272 (32 %) of the 840 samples analysed. The percentage of samples with detectable residues varied a great deal depending on the type of food. The highest percentage of residues was found in milk products (100%), compound feeding stuffs (80%), milk replacers (65%), and by-products of the food industry (61%). Exceedings of the MRL have not been possible to list. So far no MRL for animal feed has not been defined, except for feed that is also used as food, e.g. cereals.

The frequency of the eight most often found pesticides is shown in Figure 3.4. A total of 53 different pesticides were found. Pirimiphos-methyl and ethoxyquin were the pesticide most frequently found and were detected in 124 (15 %) and 46 (6 %) samples, respectively, out of the 840 samples analysed. The highest concentration of a pesticide residue was 211 mg/kg of ethoxyquin in salmon oil analysed in 2006.

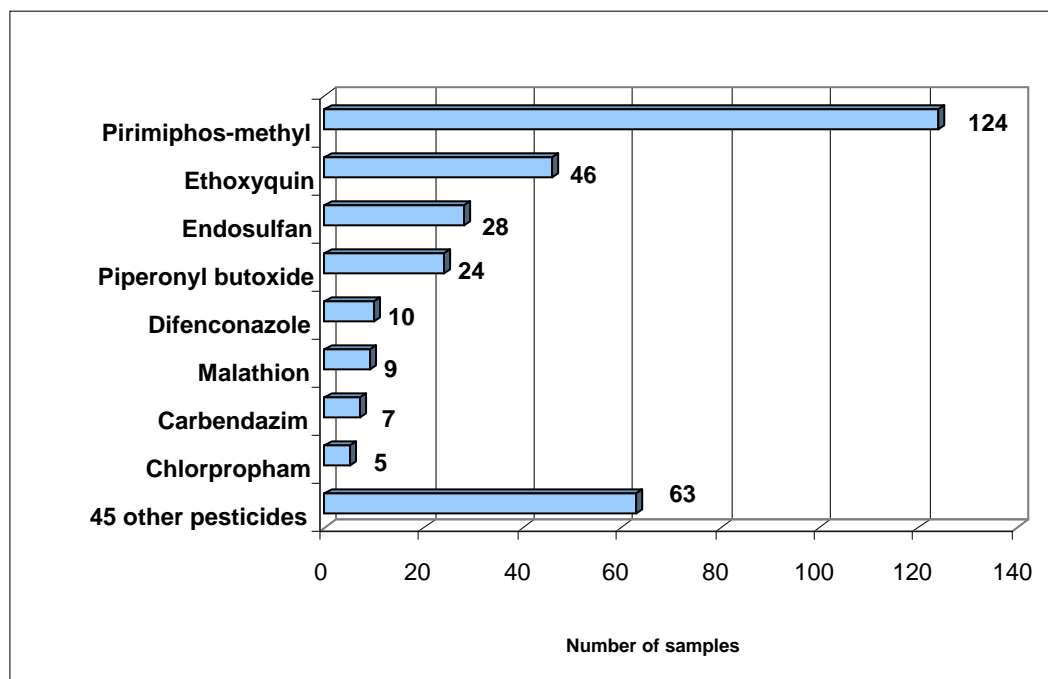
A large part of the most frequently found pesticides are only found in very few commodities.

Endosulfan is one of the most widely-used cotton and soya pesticides. Out of the 28 samples containing endosulfan, 24 of them were found in soya products, mostly soya oil. The content ranged from 0.01-0.76 mg/kg. There are no MRL for soya oil but the MRL for soya bean is 0.5 mg/kg, this would correspond to 4 exceedings of MRL in soya products. Endosulfan has been proposed as a chemical to be listed under the Stockholm Convention on Persistent Organic Pollutants.

Eight out of ten samples containing difenoconazole were from samples of beet pulp. This corresponds to the application of difenoconazole. Difenoconazole is used to control foliar fungi which have spread all over the Netherlands and causes sugar beet yield reductions up to 40%.

Ethoxyquin have been found in 46 samples. Ethoxyquin is an antioxidant used as a food preservative and a pesticide. As a pesticide it is used as prevention of common scald (browning) in apples and pears by post-harvest treatment. As a food preservative ethoxyquin is used as an antioxidant in feeding stuff, to prevent the oxidative decomposition of N3 fatty acid during long-term storage. Ethoxyquin has also been reported to have been added to the diets of dairy cattle to reduce the oxidized flavour of milk. This may explain why ethoxyquin have been found some oils in very high amounts (211 mg/kg in a salmon oil, and 141 mg/kg in a linseed oil). About one third of the milk replacer samples contain ethoxyquin in a concentration from 0.11 – 3.8 mg/kg.

Figure 3.4. Frequency of the most often found pesticides.



Feed samples analysed by U.S. Food and Drug Administration

U.S. Food and Drug Administration, FDA, samples and analyses domestic and imported animal feeds for pesticide residues. The monitoring focuses on feeds for livestock and poultry - animals that ultimately become or produce foods for human consumption. The data presented here are from 2006 and 2007.

The Environmental Protection Agency, EPA, determines the safety and effectiveness of the chemicals and establishes tolerance levels and regulatory guidance for residues on feed crops, as well as for raw and processed foods. These tolerance levels (the amount of pesticide allowed to be present in a food product) are normally set 100 times below the level that might cause harm to people or the environment. In the following the exceedings of regulatory guidance found by the FDA have been cross-checked for exceedings of MRLs in the EU pesticide database.

In 2007, 292 feed samples (196 domestic surveillance and 96 imported) were analysed for pesticides by the FDA (Table 3.10). Of the 196 domestic surveillance samples, 145 samples (74 %) contained no detectable pesticide residues, 44 samples (22 %) contained one or more detectable residues that did not exceed regulatory guidance, and 7 (3.6 %) contained a residue which exceeded regulatory guidance. Of the 96 import samples, 78 (81 %) contained no detectable pesticide residues, 18 (19 %) contained one or more

detectable residues that did not exceed regulatory guidance, and none samples contained a residue which exceeded regulatory guidance.

The 7 domestic surveillance samples of animal feed with residues that exceeded regulatory guidance were 3 grains and 4 plant by-products. Two corn samples from Ohio contained 0.110 and 0.143 mg/kg of chlorpyrifos, respectively. These levels exceed the 0.05 mg/kg tolerance for chlorpyrifos on field corn (the EU-MRL for chlorpyrifos in corn is also 0.05 mg/kg). One corn sample from Missouri contained 0.030 mg/kg of chlorpyrifos-methyl. No tolerances have been established by the EPA for chlorpyrifos-methyl on corn. (This sample would not have exceeded the EU MRL for chlorpyrifos-methyl in corn which is 3 mg/kg).

Two samples, a soy hull pellet and a canola meal, contained 0.037 mg/kg and 0.066 mg/kg of tris (chloropropyl) phosphate, respectively. No tolerances, action levels, or guidance has been established by the EPA or FDA (or by the EU) for this fire retardant in animal feed so any quantifiable level is considered to have exceeded regulatory guidance. One sample of cotton burrs from Texas contained 14.8 mg/kg of malathion. This level exceeds the 2 mg/kg tolerance for malathion on delinted cotton seed. The EU MRL for malathion in cotton seed is 0.02 mg/kg. One sample of peanut hulls from Texas contained 0.058 mg/kg of DEF (tribuphos). No tolerances have been established by the EPA for DEF on peanuts hulls (or by the EU).

Table 3.10. Summary of the 196 domestic surveillance and 96 import samples of animal feed that were analyzed for Pesticides by FDA in 2007.

Type of Feed	Number of Samples analysed	Samples with No Pesticide Residues	%	Samples Exceeding Regulatory Guidance	%
Whole/Ground Grains	115	99	86	3	2.6
Plant By-products	80	57	71	4	5
Mixed Feed Rations	59	34	58	0	0
Supplements/Misc.	19	17	90	0	0
Hay & Hay Products	13	10	77	0	0
Animal By-products	6	6	100	0	0
TOTALS	292	223	76	7	2.4

In the 51 domestic surveillance and 18 import samples of animal feed in which one or more pesticides were detected, there were 90 quantifiable residues. Malathion and ethoxyquin were the most frequently found and accounted for 55 % of all residues

detected. Table 3.11 shows the number of quantifiable levels for each pesticide and the concentration range.

Table 3.11. Summary of the pesticides in 51 domestic surveillance and 18 import samples of animal feed, that contained one or more detectable residues in 2007.

Pesticide	Quantifiable Levels	Range (mg/kg)	Median (mg/kg)
Malathion	32	0.011 - 14.8	0.111
Ethoxyquin	20	0.068 - 29.8	0.571
Chlorpyrifos-methyl	6	0.011 - 0.175	0.028
DDE + TDE + DDT	6	0.002 - 0.046	0.014
DEF	6	0.056 - 1.82	0.253
Chlorpyrifos	3	0.018 - 0.143	0.11
Chlorpropham	2	0.070 - 0.073	
Fenpropathrin	2	0.210 - 0.600	
Myclobutanil	2	0.047 - 1.20	
Quinoxifen	2	0.077 - 0.330	
Tris (chloropropyl) phosphate	2	0.037 - 0.066	
Azoxystrobin	1	0.08	
All others	6	0.025 - 2.10	0.134

In 2006, 335 feed samples (264 domestic surveillance and 71 imports) were analyzed for pesticides by the FDA. Of the 264 domestic surveillance samples, 196 (74 %) contained no detectable pesticide residues, 66 (25 %) contained residues at levels not exceeding regulatory guidance, and 2 (0.8 %) contained residues which exceeded regulatory guidance. Of the 71 import samples none contained a residue which exceeded regulatory guidance. The 2 residues that exceeded regulatory guidance was a vitamin premix sample from Canada that contained 82.070 mg/kg of ethoxyquin, and a sample of tallow collected by the Chicago District Office contained 0.069 mg/kg of *o*-phenylphenol.

In the 68 domestic surveillance and 3 import samples of animal feed in which one or more pesticides were detected, there were 99 pesticide residues. Malathion and ethoxyquin were the most frequently found and accounted for 60.6% of all residues detected.

4. MRLs and toxicological data

In the EU, as from 1 September 2008, a new legislative framework (Regulation (EC) No 396/2005 of the European Parliament and of the Council) on pesticide residues is applicable. This Regulation completes the harmonisation and simplification of pesticide MRLs, whilst ensuring better consumer protection throughout the EU.

All national MRLs will thereby no longer apply. The regulation will include all “old” EU-MRLs as well as many new EU-MRLs which have been agreed on based on all of the member states national MRLs. If there is a combination of a commodity and pesticide for which there is no MRL specified in the regulation then a default MRL of 0.01 mg/kg will apply. The MRL in force from the 1st of September are listed in the regulation no. 149/2008 of 29²⁶.

So far the focus of the work in EU in regard to safety evaluations of pesticide residues has been focused on residues in food. However the Regulation (EC) NO 396/2005 of 23 February 2005 on maximum levels also relate to MRLs in feed. So far however the group of “Products intended for animal feed” has not been defined further (Directive 202/32/EC of 7 May 2002). Feeding stuff is defined by crops solely intended for feeding stuff, grass, straw, green forage for ensilage, fodder peas etc.

Residues should not be present at levels presenting an unacceptable risk to humans and, where relevant, to animals (Regulation (EC) NO 396/2005). Maximum contents for some persistent pesticides have been laid down in Council Directive 1999/29/EC of 22 April 1999 on the undesirable substances and products in animal nutrition²⁷. These pesticides include aldrin, dieldrin, camphechlor, DDT, endosulfan, endrin, heptachlor, hexachlorbenzene and hexachlorocyclo-hexane (HCH). For these pesticides a maximum content in mg/kg relative to a feedingstuff with a moisture content of 12% have been defined.

No MRLs have so far been set for feedingstuff.

Toxicological data as ADI and ARfD could also be taken into account when considering which pesticide to include in monitoring programs. It could be argued that pesticides for which low ADI and/or ARfD has been set are more relevant to monitor for than pesticides with higher values. A combination of high MRLs and low ADI and/or ARfD could increase the importance of monitoring for residues of this particular pesticide²⁸.

5. Feasibility to include the pesticide in multiresidue methods

Multiresidue methods are cost effective and are therefore when ever possible preferable over single residue methods. The majority of the pesticides authorised for use on wheat in more than 9 of 16 northern and central European MS (listed in Table 2.4) are possible to include in a multi method. The exceptions are glyphosate and perhaps chlormequat.

Both LC- and GC-compounds are represented in Table 2.4. The sulfonylurea type is in general possible to include in LC-methods. Though, these types of pesticides can be difficult to detect, because the sulfonylurea compounds are very potent and only spread in very low amounts per hectare, resulting in low residue levels.

The triazole and pyrethroid types are possible to include in GC-methods, whereas it varies for the strobilurin type whether GC- or LC-methods are most applicable.

It is difficult to give general recommendations on which pesticides are of greater or less relevance when analysing feeding stuff, since feed can be composed of a wide range of products and by-products.

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