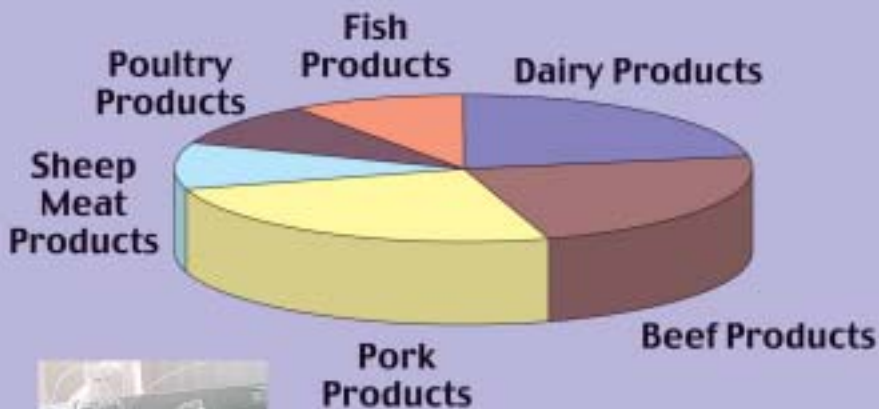


# Food Residue Database

## Food Residue Database Studies



**The National  
Food Centre**

RESEARCH & TRAINING FOR THE FOOD INDUSTRY  
RESEARCH REPORT NO 46



## FOOD RESIDUE DATABASE

### Authors:

---

Michael O'Keeffe B.Sc. Ph.D.  
Orla Kennedy B.Sc. Ph.D.  
Frank Farrell B.Sc. M.Sc. Ph.D.  
Marie-Louise Nolan B.Sc. Ph.D.  
Martin Dooley B.Sc.  
Patrick Byrne Grad RSC M.Sc.  
Audrey Nugent B.Sc. M.Sc. Grad Dip. Sci. (Toxicology)  
Helen Cantwell B.Sc. Ph.D.  
Elizabeth Horne B.Sc. Ph.D.  
Victor Nelson B.Sc. Ph.D.  
**The National Food Centre, Dunsinea,  
Castleknoock, Dublin 15**

and

David McGrath B.Sc. Ph.D.  
**Johnstown Castle Research Centre, Wexford**

ISBN 1 84170 264 1  
November 2001





## CONTENTS

Summary	1
Introduction	1
Methodology	3
Results	5
Veterinary drugs and pesticides	5
Antimicrobials	5
Antiparasitics and Coccidiostats	7
Beta-agonists	9
Pesticides	9
Contaminants	10
Dioxins	10
Mycotoxins	12
Heavy metals	14
Polycyclic Aromatic Hydrocarbons	16
Nitrate and Nitrite	17
Recommendations to industry	19
Publications from this project	20



## SUMMARY

The *Food Residue Database* contains a broad range of residue studies in foods of animal origin for the period 1995 to 2000, covering veterinary drugs, pesticides and contaminants. In most cases, such as antiparasitic drugs, beta-agonists, pesticides, dioxins, mycotoxins, heavy metals and polycyclic aromatic hydrocarbons, the picture for Irish dairy, meat and fish products is good with residue levels being low or non-measurable. In a few cases, such as ivermectin in farmed salmon and tetracycline residues in pork, improvements in the situation were observed with subsequent studies. Antimicrobial residues, in general, are not a problem but levels above MRL values have been found indicating the need for good practice in use of veterinary medicines. A problem with elevated nitrate levels in dairy powders may be resolved by the industry through observance of good manufacturing practices. Summary Reports on all the studies carried out for the *Food Residue Database* are available to food companies and other interested parties.

## INTRODUCTION

Production of a database on the residue status of Irish foods - the *Food Residue Database* - was aimed at developing residue information to be used by the Irish food industry to assist production, processing and marketing of food products.

Irish food has a green and wholesome image. However, trading based on an image cannot continue indefinitely. A need exists for comprehensive data to support this image, especially for customers of Irish products on the export market. The database has a number of potential uses. Primarily it may be used as a marketing tool to promote Irish food from a food safety point of view. The database may be used also to demonstrate compliance of Irish products with regulations and with customer specifications. Finally, the database may be used to identify potential or actual problems with production and processing practices, enabling early corrective action to be taken.

The database is comprised of a series of discrete studies, usually one year in duration, mainly on foods of animal origin because of the importance of these



products on the export market. Examples are meat products, dairy products and farmed fish. The residues of interest fall into the general categories of natural contaminants, agrochemicals, veterinary drugs and industrial contaminants.

Within each study, strict criteria were met to ensure the integrity of the database. The samples were randomly selected, were characteristic of the food on the export market and were seasonally and geographically representative of the production. In practice, samples were obtained on a regular basis directly from companies producing the products of interest. The samples were analysed by validated laboratory methods. The results were consolidated into a computerised database where the data may be manipulated and accessed for specific users.

Apart from studies carried out at The National Food Centre and co-operating laboratories, the database also contains the results of relevant studies carried out by other organisations with the permission of those organisations.

Results of studies carried out at The National Food Centre were made available in a series of confidential reports to the companies which provided samples for analysis. Summary Reports of studies on the database are available to persons with a commercial interest in the marketing or production of Irish food. Access to the database is at the discretion of The National Food Centre and Teagasc.



## METHODOLOGY

### Samples

The samples of food products used for the various studies were representative of food in the export market. In total, over 40 food companies provided samples for analysis. Sampling was in accordance with schedules provided by The National Food Centre to ensure random geographical and seasonal distribution of samples. Samples were stored frozen or refrigerated at the laboratory prior to analysis.

### Methods of analysis

The methods of analysis used were validated, through recovery studies, to ensure the accuracy of the results obtained. Modern techniques were used for residue extraction and extract clean-up prior to residue determination by immunoassay, chromatography, spectrophotometry or mass spectrometry. The methods used had adequate sensitivity to measure residues comfortably below the levels of concern. Analysis of all residues were undertaken at The National Food Centre with the exception of heavy metals and PAHs (Johnstown Castle Research Centre, Teagasc) and dioxins (RIKILT, Wageningen, The Netherlands)

### Reports

The results of each study were entered on a computerised database and final reports were prepared for the food industry participants; these reports were specific to each participating company, indicating the results for their samples. A Booklet of Summary Reports (Table 1) was also issued to food companies and agencies. A number of newsletters on the *Food Residue Database* have been distributed widely.



**Table 1: Summary Reports on studies carried out for the *Food Residue Database*.**

<b>Dairy products</b>
Heavy Metals in Dairy Powders, 1995/96
Aflatoxin M1 in Dairy Powders, 1996
Nitrates and Nitrites in Dairy Powders, 1996/97
Polycyclic Aromatic Hydrocarbons (PAHs) in Dairy Powders, 1996/97
Pesticide and PCB Residues in Butter and Cheese, 1997
Nitrates and Nitrites in Dairy Powders (Continuation Study), 1998/99
Dioxins and coplanar PCBs in Cheese, 1998/99
<b>Beef products</b>
β-Agonists in Bovine Liver, 1997/98
Pesticide and PCB Residues in Carcass Fat, 1997/98
Heavy Metals in Meat, 1997/98
Antibiotic Residues in Kidney, 1998/99
Polycyclic Aromatic Hydrocarbons (PAHs) in Carcass Fat, 1998/99
Pyrethroid Residues in Carcass Fat, 1999
Dioxins and coplanar PCBs in Carcass Fat, 2000
<b>Pork products</b>
Tetracycline Antibiotics in Meat and Kidney, 1996
Pesticide and PCB Residues in Carcass Fat, 1996/97
Tetracycline Antibiotics in Meat (Continuation Study), 1997/98
Heavy Metals in Meat, 1997/98
Ochratoxin A in Kidney and Meat, 1996/97 and 1999
Antibiotic Residues in Kidney, 1998/99
Polycyclic Aromatic Hydrocarbons (PAHs) in Carcass Fat, 1998/99
Dioxins and coplanar PCBs in Carcass Fat, 2000
<b>Sheep Meat products</b>
Pyrethroid Residues in Carcass Fat, 1999
Antibiotic Residues in Kidney, 1999/2000
Dioxins and coplanar PCBs in Carcass Fat, 2000
<b>Poultry products</b>
Antibiotic Residues in Kidney and Meat, 1999/2000
Coccidiostat Residues in Meat, 1999/2000
Dioxins and coplanar PCBs in Carcass Fat, 2000
<b>Fish products</b>
Ivermectin in Farmed Salmon, 1995/96
Ivermectin in Farmed Salmon (Continuation Study), 1997/98
Dichlorvos and Ivermectin in Farmed Salmon, 1999/2000



## RESULTS

### Veterinary drugs and Pesticides

#### *Antimicrobials*

Antimicrobial substances, commonly referred to as antibiotics, are widely used in conventional animal production to prevent and treat bacterial infection. The use of antibiotics may be therapeutic, in treatment of current infections in animals, prophylactic, to prevent the occurrence of infections, or as feed additives (limited, specific compounds) to improve performance. A broad range of antibiotics are used in animal production including beta-lactams (penicillins, cephalosporins), tetracyclines (chlortetracycline, oxytetracycline), sulphonamides (sulphamethazine, sulphadiazine), aminoglycosides (streptomycin, neomycin), macrolides (tylosin, erythromycin) and there are some prohibited substances, such as chloramphenicol and the nitrofurans.

There are two areas of particular concern with regard to use of antibiotics in animal production. Firstly, residues of these substances may occur at unacceptable levels in edible tissues at slaughter giving rise to the potential for toxic effects in susceptible individuals. Secondly, their widespread use in agriculture may contribute to the development of resistant strains of bacteria.

Two studies were undertaken for the *Food Residue Database*, during the period February 1996 to February 1998, specifically on the occurrence of residues of tetracycline antibiotics in pork. Six pig processing plants provided samples of meat and kidney for testing. During the first year (1996/97), 140 samples of meat and 57 samples of kidney were tested and during the second year (1997/98) a further 42 samples of meat were tested. The results for these studies (Table 2) show that 30% of meat samples taken during 1996/97 were residue-positive for chlortetracycline, with 5% above the permitted maximum residue limit (MRL) of 100 ppb. All meat samples taken during 1997/98 were free of measurable residues or had levels much lower than the MRL, indicating that the problem with chlortetracycline in pork during 1996/97 had been reduced or eliminated.

Following on these specific studies on the incidence of tetracycline residues in





**Table 2:** Chlortetracycline antibiotic residues in pork samples obtained from the Irish pork industry.

Period of sampling	Number (%) of samples at each residue level		
	ND <sup>a</sup>	Less than the MRL <sup>b</sup>	Greater than the MRL
1996/97	98 (70%)	35 (25%)	7 (5%)
1997/98	37 (88%)	5 (12%)	0 (0%)

<sup>a</sup>ND - no residue detected, limit of determination 15 ppb

<sup>b</sup>MRL - maximum residue limit, 100 ppb

pork, a more comprehensive study on the occurrence of different classes of antimicrobial residues in pork, beef, poultry and sheep meat was undertaken during the period September 1998 to May 2000. Five pig, eight beef, four poultry and five sheep processing plants supplied samples of kidney over periods of approximately 16 months for testing. In total, 109 pork, 112 beef, 93 poultry and 104 sheep kidney samples were analysed for antibiotic residues using a three-stage screening (Inhibitory Substances Test), post-screening (Charm II Tests) and confirmatory (Chromatography) process.

The results for the testing are summarised in Table 3. While a number of samples of pork kidney contained measurable residues of chlortetracycline at 20-80 ppb, these levels are much lower than the maximum residue limit (MRL) of 600 ppb for chlortetracycline in kidney. No samples of beef or poultry kidney contained measurable residues of antibiotics. One sample of sheep kidney contained chlortetracycline residues at 650 ppb, which level is above the MRL.

While these more recent studies on antibiotic residues in meat indicated that, in general, residue levels do not occur above MRL values, the occurrence of residue-positive pork samples and a sheep sample with a violative level of chlortetracycline indicates the need for correct usage of these veterinary drugs in animal production.



**Table 3:** Antibiotic residues in commercial pork, beef, poultry and sheep kidney during the period 1998 to 2000.

Species	Number of samples tested	Number of samples positive in Charm II test	Number of samples and antibiotic residues identified by chromatography
Pork	109	11 (tetracyclines) 6 (sulphonamides) 2 (beta-lactams)	7 (20-80 ppb chlortetracycline)
Beef	112	0	0
Poultry	93	10 (sulphonamides)	0
Sheep	104	1 (tetracyclines) 4 (sulphonamides)	1 (650 ppb chlortetracycline)

### *Antiparasitics and Coccidiostats*

Three areas of antiparasitic and coccidiostat residues in foods of animal origin were studied: ivermectin and dichlorvos residues in farmed salmon, pyrethroid residues in beef and sheep meat and coccidiostat residues in poultry. While not approved for use on fish, there had been a history of use of ivermectin to control sea-lice in farmed salmon, together with the approved organophosphorus compound, dichlorvos. Pyrethroids are widely used to control ectoparasites in cattle and sheep. Coccidiosis is a particular problem in poultry production and coccidiostats are used to control this disease.

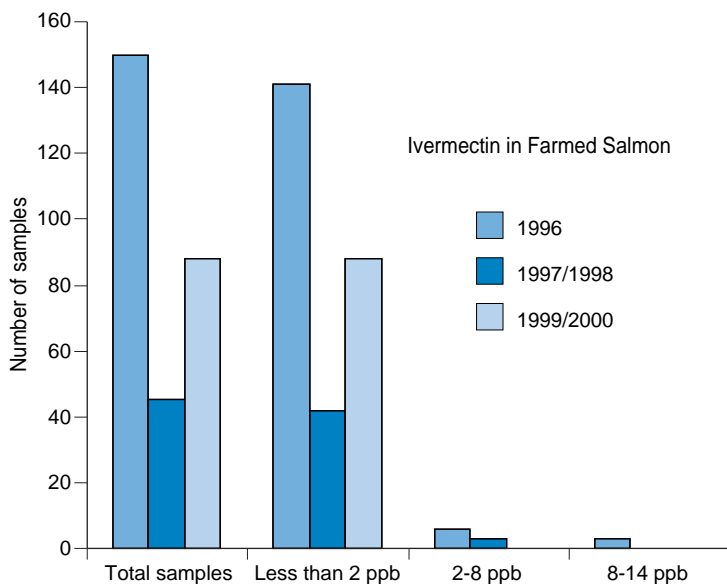
A series of three studies on ivermectin residues in farmed salmon was undertaken in 1996, 1997/98 and 1999/2000, with the latter study also including dichlorvos. For the 1996 study, 150 samples were analysed and 9 samples (6%) contained ivermectin residues ranging from 2 to 14 ppb; all of the residue-positive samples were taken in the period March to June. The follow-up study in 1997/98 on 45 samples found 3 samples to contain ivermectin in the range of 2-4 ppb, indicating lower residue levels but continued use of the drug in fish farming. The most recent study, on 88



samples, found no samples containing measurable residues of ivermectin or of the approved drug, dichlorvos. These results indicate an improving situation for these residues in farmed salmon (Figure 1).

For the study on pyrethroid residues in beef and sheep meat, carcass fat samples were analysed as fat is the edible tissue in which the highest concentration of pyrethroids, if present, are likely to occur. Eight beef and five sheep meat slaughtering plants provided samples for analysis during the period January to December 1999. A total of 155 samples of fat were analysed for residues of tetramethrin, permethrin, deltamethrin, cypermethrin and fenvalerate by gas chromatography. No measurable residues (i.e. less than 25 ppb) of these antiparasitic chemicals were determined in any of the fat samples tested.

The study on coccidiostat residues in poultry meat was undertaken during the period November 1999 to June 2000. 35 samples from three poultry



**Figure 1:** Results for ivermectin residues in farmed salmon for studies in 1996, 1997/1998 and 1999/2000.



processing plants were analysed for residues of monensin, salinomycin and narasin. No measurable residues (i.e. less than 20 ppb) of these drugs were found in any of the samples.

### *Beta-agonists*

Beta-agonists, such as clenbuterol and salbutamol, may be used to affect the growth of beef animals and produce a leaner carcass. Clenbuterol is the only beta-agonist licensed for veterinary use, for the treatment of respiration disorders, but all beta-agonists including clenbuterol are prohibited within the EU for use as growth enhancing agents. In some European countries there was a history of illicit use of beta-agonists; a European study in 1994 on retail liver found over 20% of samples from some countries contained clenbuterol.

Over the period February 1997 to January 1998, 137 samples of liver from eight beef slaughtering plants were tested and no positive samples were found. These results suggest that the extensive efforts made by the Department of Agriculture, Food and Rural Development against the illicit use of growth enhancing substances in beef production have been successful. The results for the national residue testing plan over the period 1998 to 2000 confirm the improving situation; in 1998, 15 (of 40,000) samples were positive for beta-agonists while in 1999 and 2000, all samples (of 39,000 and 28,000 respectively) were negative.

### *Pesticides*

Foods of animal origin, such as meat and dairy products, are open to two main sources of potential contamination from pesticide residues, either from direct application of a pesticide to the animal, for example spraying/dipping with insecticides, or from consumption of feedstuffs which have been contaminated with pesticides. The two main classes of pesticides are organochlorine (OC) and organophosphorus (OP) pesticides. Three studies were undertaken during 1997 on the occurrence of OC and OP pesticides in beef and pork fat and in dairy products (cheese and butter). For these twelve-month studies, 140 samples of beef fat, 132 samples of pork fat and 130 samples of cheese/butter were analysed for pesticide residues. In the case of beef fat, no measurable residues of OC pesticides were determined in any



sample. In the case of pork fat, 128 samples (97%) showed no measurable residues of OC pesticides. Two samples contained Dieldrin at approximately 0.01 mg/kg, ppm, which is 20 times lower than the MRL for that pesticide (0.2 mg/kg fat). Two further samples contained Lindane at 0.025 mg/kg, ppm, which is 40 times lower than the MRL for that pesticide (1.0 mg/kg fat). In the case of dairy products, only one sample contained Lindane, at a level close to the limit of detection, 0.01mg/kg, ppm. No sample in any of the studies contained measurable residues of OP pesticides (less than 0.1 mg/kg, ppm).

These results indicate that residues of the common OC and OP pesticides are not a problem in these foods of animal origin. However, studies by the Pesticide Control Service of the Department of Agriculture, Food and Rural Development found 3 samples of pork fat in 1998 to contain dicofol residues above the MRL for that pesticide and one sample of milk in 1997 to contain  $\beta$ -HCH residues above the MRL for that pesticide.

## Contaminants

The other major area of residues in food covered by the *Food Residue Database* is that of contaminants. Contaminants include chemical substances occurring naturally in food, such as mycotoxins, chemical substances occurring as environmental and industrial contaminants, such as heavy metals and dioxins, and chemical substances occurring due to food processing, such as nitrates/nitrites and polycyclic aromatic hydrocarbons.

### *Dioxins*

Dioxins is a general name applied to a group of related compounds which share common chemical structure and biological properties. Certain polychlorinated biphenyls (PCBs), those with a planar structure, also have properties similar to dioxins. Dioxins and planar PCBs are carcinogenic and may have adverse effects on reproduction. These substances occur as by-products of chemical processes, from combustion and may be found in oil waste. Dioxins and planar PCBs are fat soluble chemicals and exposure of humans to these contaminants is largely from fat-containing foods of animal



origin. In recent years, there have been a number of incidences of dioxin contamination of food, through dioxin-contaminated citrus pulp from Brazil in 1998 which was used in feed for dairy animals in France and resulted in contaminated milk, and through gross contamination of waste edible oil with machine oil in Belgium in 1999, resulting in contaminated animal feed and contaminated food products such as poultry, eggs, red meat and milk.

The only previous substantial study on dioxins in Ireland had been by the EPA (Environmental Protection Agency) in 1995 on milk, so it was decided to undertake studies on dioxins and planar PCBs in dairy products (cheese) and in meat (beef, pork, poultry and sheep). The study on cheese examined 90 samples from seven dairy companies during the period September 1998 to August 1999. Because of the specialised equipment and expertise required, these analyses were undertaken by the RIKILT laboratory in Wageningen, The Netherlands. The range of values in cheese samples for dioxins was from less than 0.1 to 0.8 pg TEQ/g fat, for planar PCBs was from less than 0.1 to 0.5 pg TEQ/g fat and for dioxins and planar PCBs combined was from 0.1 to 1.2 pg TEQ/g fat; TEQ is a system for expressing all the different dioxins and PCBs in a common way related to their "Toxic Equivalents". A maximum value of 1.2 pg TEQ/g fat may be regarded as satisfactory, with values of 0.5 - 3.5 pg TEQ/g fat being reported for milk fat from other European countries and maximum permissible limits of 5 - 6 pg TEQ/g fat being applied by some countries.

A further study on carcass fat from the four main meat species was undertaken between January and July 2000. 15 samples each of beef, pork, poultry and sheep fat, from 13 meat processing companies, were tested for dioxin and planar PCB residues. All of the samples contained less than 5 pg TEQ/g fat for dioxins and planar PCBs combined. All samples of beef, pork and poultry fat contained dioxin and planar PCB residues less than or equal to 1 pg TEQ/g fat. In the case of sheep fat, some samples contained dioxins and planar PCBs at levels up to 3 pg TEQ/g fat.

Within the European Commission, a committee on dioxins in food has suggested a maximum permissible level for dioxins of 2 pg TEQ/g fat for dairy products and for meat (1 pg TEQ/g fat for pork). The *Food Residue Database*



studies (Figure 2) indicate that Irish food products, with the exception of some sheep meat, meet these standards.

### *Mycotoxins*

Mycotoxins are chemical toxins which occur naturally in a range of plant products as a by-product of fungal growth. In foods of animal origin, mycotoxin residues may occur due to ingestion of mycotoxins by animals from feed. Mycotoxins are highly toxic contaminants and low maximum residue limits have been specified or recommended. For the *Food Residue Database*, two studies on mycotoxins were undertaken, one on aflatoxin M1 in dairy products and one on ochratoxin A in pork.

Aflatoxin M1 residues may occur in dairy products as a metabolite of aflatoxin B1 ingested in feedstuffs by the dairy cow. 11 dairy companies supplied samples of skim milk and casein powders for testing during the period January to December 1996. 65 samples of skim milk and 72 samples of casein powders were tested and approximately 60% of samples contained no measurable residues of aflatoxin M1 (0.025 ppb). The levels determined in skim milk and casein

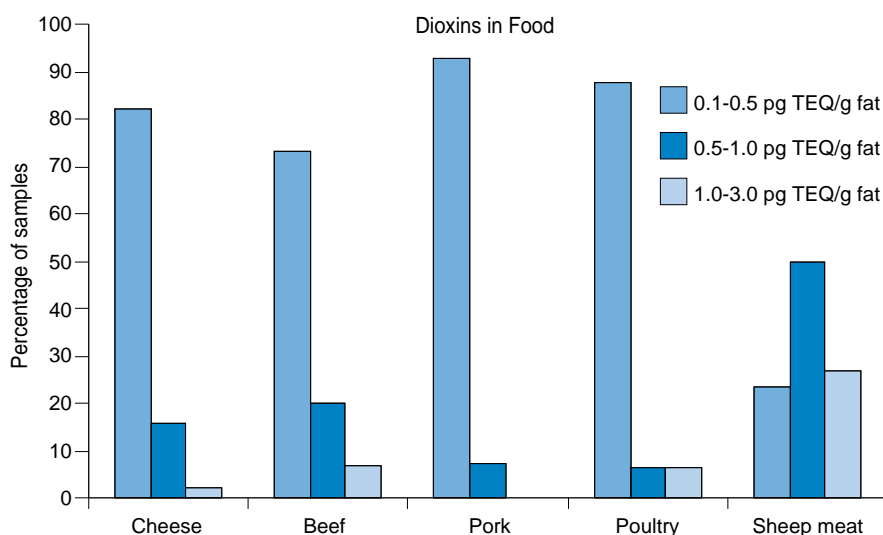
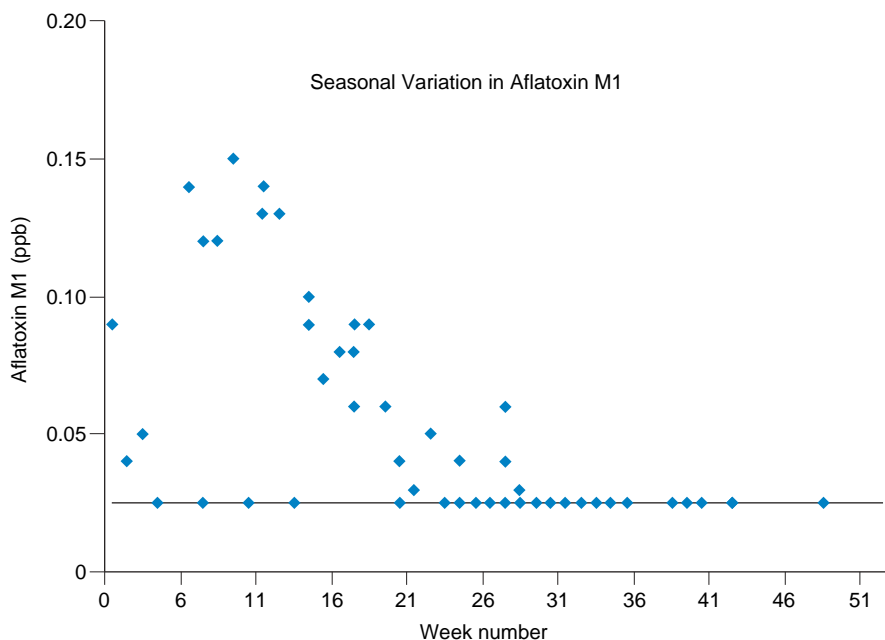


Figure 2: Dioxin residues in foods of animal origin.



powders were 0.03 - 0.15 ppb and 0.03 - 0.28 ppb, respectively. The maximum levels determined were much lower than the maximum residue limits of 0.5 ppb (for skim milk powder) and 1.75 ppb (for casein powder), derived from the recommended MRL of 0.05 ppb in milk. An interesting seasonal trend was observed for aflatoxin M1 in dairy powders (Figure 3), with the highest residue levels being associated with powders produced in the first quarter of the year, indicating that the occurrence of aflatoxin M1 may be related to supplementary feeding of compound feed to dairy cows.

Ochratoxin A may occur as a contaminant of cereals used in animal feedstuffs and has been found in pig kidneys in the Scandinavian countries, Hungary and Poland. Six pork processing companies supplied samples of pork kidney and meat for a two-part study during the periods October 1996 - March 1997 and April 1999 - September 1999. A total of 130 samples of kidney were tested for ochratoxin A and nearly half the samples (46%) contained no measurable



**Figure 3:** Seasonal variation for Aflatoxin M1 in skim milk powder.





residues (less than 0.1 ppb). A further 44% of samples contained ochratoxin A residues below 0.5 ppb. Twelve samples (10%) contained residues of ochratoxin A between 0.5 and 2.2 ppb. 20 paired meat samples, chosen to reflect relatively high (greater than 0.5 ppb), low (0.2 - 0.5 ppb) and non-measurable (less than 0.1 ppb) levels of ochratoxin A in the corresponding kidney, were analysed (Table 4). Only three of the meat samples contained ochratoxin A, at levels of less than 1 ppb. The results indicated that pork products may be contaminated with ochratoxin A, although the levels found were lower, generally, than in other countries, where ochratoxin A levels in excess of 5 ppb were found.

### *Heavy metals*

Heavy metals occur as natural constituents of milk and meat (for example, zinc, copper and selenium) or as contaminants from environmental or industrial sources (for example, lead, tin, cadmium, mercury and arsenic). While MRLs have not been established, generally, for heavy metals in food products, many customers for Irish food products have specifications for heavy metals. Three studies were undertaken on heavy metals in dairy products (powders), in pork and in beef for the *Food Residue Database*.

**Table 4:** Ochratoxin A residues in samples of pig kidney and lean meat.

Total Study, Kidney (n = 130):	Number of samples at each residue level		
	ND <sup>a</sup>	0.1 - 0.5 ppb	0.5 - 2.5 ppb
	60	57	13
Kidney/Lean meat intercomparison (n = 20):	Number of lean meat samples at each residue level		
	ND <sup>a</sup>	0.1 - 0.5 ppb	0.5 - 2.5 ppb
Kidney at ND <sup>a</sup> (n = 8)	8		
Kidney at 0.1-0.5 ppb (n = 5)	5		
Kidney at 0.5 - 2.2 ppb (n = 7)	4	2	1

<sup>a</sup>ND - no residue detected, limit of determination 0.1 ppb



Over the 12-month period from May 1995 to May 1996, 11 dairy companies provided samples of skim milk powder (71 samples), acid casein (43 samples) and rennet casein (33 samples). Analysis of these samples for heavy metals was undertaken, at Johnstown Castle Research Centre, using the appropriate atomic absorption and atomic fluorescence techniques. The levels of zinc, copper and selenium determined were similar to levels reported for these food products in the scientific literature. No cadmium (less than 0.005 ppm), mercury (less than 0.01 ppm) or arsenic (less than 0.04 ppm) were

**Table 5:** Heavy metal content in beef and pork.

Heavy metal	Beef (n=126)		Pork (n=101)	
	Content (range, ppm)	Other studies <sup>a</sup> (range, ppm)	Content (range, ppm)	Other studies <sup>a</sup> (range, ppm)
Zinc	22 - 82	18 - 75	10 - 48	14 - 42
Copper	0.6 - 3.1	0.4 - 2.2	0.6 - 3.4	0.4 - 1.3
Selenium	0.03 - 0.4	<0.03 - 0.24	0.08 - 0.30	0.04 - 0.82
Tin	ND <sup>b</sup>	- <sup>c</sup>	ND - 0.05 <sup>e</sup>	-
Arsenic	ND	-	ND	-
Lead	ND - 0.05	(0.05) <sup>d</sup>	ND - 0.02	ND
Mercury	ND	-	ND	-
Cadmium	<0.001 - 0.003	(0.006) <sup>d</sup>	NA <sup>f</sup>	

<sup>a</sup> Jorhem, L., Sundstrom, B., Engman, J., Astrand-Yates, C. and Olsson, I. 1996. *Food Additives and Contaminants* **13**: 737-745.

<sup>b</sup> Not detected

<sup>c</sup> No data available

<sup>d</sup> Typical levels for meat (WHO data)

<sup>e</sup> One sample contained an untypically high level of tin (0.8 ppm)

<sup>f</sup> Not analysed



determined in any of the dairy powder samples. Tin was not determined (less than 0.05 ppm) in any skim milk powders nor in casein powders with the exception of two acid casein (0.08 and 0.09 ppm) and one rennet casein (0.14 ppm) samples. Lead was not determined (less than 0.02 ppm) in skim milk powders with the exception of two samples containing lead between 0.02 and 0.04 ppm. Lead levels in casein powders were in the range of 0.03 to 0.11 ppm; the Codex Alimentarius draft standard specifies a limit for lead in casein powders of 1 ppm. The results of the study indicate that heavy metals in dairy powders are at expected levels, for natural constituents, and are very low or non-measurable, for contaminants.

Eight beef and six pork processing plants provided 125 and 100 samples, respectively, of meat for heavy metals analysis during the period January 1997 to February 1998. The levels of zinc, copper and selenium determined (Table 5) were similar to the levels reported in the scientific literature. No tin (less than 0.05 ppm), mercury (less than 0.01 ppm) or arsenic (less than 0.02 ppm) were determined in any of the samples tested. Lead was not determined (less than 0.01 ppm) in pork samples with the exception of one sample containing 0.07 ppm. Lead in beef ranged between 0.01 and 0.2 ppm; a draft European Commission regulation (III/5125/95.Rev.2, September 1996) specifies a limit of 0.2 ppm for lead in meat. The results of these studies indicate that heavy metals in meat are at expected levels, for natural constituents, and are low or non-measurable, for contaminants.

### *Polycyclic Aromatic Hydrocarbons (PAHs)*

Polycyclic aromatic hydrocarbons (PAHs) are industrial contaminants which may occur as products of incomplete combustion, for example from incinerators or from food processing operations involving heating or smoking. These compounds may be carcinogenic and studies on residues in food often concentrate on measuring levels of the most common carcinogenic PAH, benzo[ $\alpha$ ]pyrene, using it as an indicator for contamination of food products with PAHs. Published reports on benzo[ $\alpha$ ]pyrene in meat products indicate levels ranging from less than 0.1 to 4 ppb; data for dairy products are not available.



Two studies were undertaken for the *Food Residue Database* on PAHs in dairy powders and in meat products. 50 samples of dairy powders from nine dairy companies were tested for PAHs, during the period October 1996 to March 1997. The PAH contents of the samples, expressed as benzo[ $\alpha$ ]pyrene equivalents, were within the range of 0 to 0.6  $\mu\text{g/kg}$ , ppb. For the second study, pork and beef fat samples were obtained from 13 meat processing companies during the period September 1998 to June 1999 and samples of cured/smoked meat products from three companies were obtained during May 2000. No samples of meat fat or of cured/smoked products contained benzo[ $\alpha$ ]pyrene at levels above 0.1 ppb. In some European countries, a permissible level for benzo[ $\alpha$ ]pyrene in food has been set at 1 ppb; the results for this study show that Irish food products are well below such a limit.

### *Nitrate and Nitrite*

Nitrate and nitrite occur as contaminants in plant foods and as additives in cured meats and cheese. There is concern to maintain levels of nitrate and nitrite as low as possible because of suspected adverse effects on oxygenation of the blood and/or indirect carcinogenic effects, through formation of nitrosamines. Specifications for maximum nitrate levels in dairy powders are as low as 30 ppm. Because Irish dairy powder manufacturers may have difficulty in matching these specifications, studies on nitrate and nitrite in dairy powders were included in the *Food Residue Database*.

A first study, during the period October 1996 to September 1997, was undertaken to obtain a profile for nitrate and nitrite contents in dairy powders. A total of 132 samples of skim milk, acid casein and rennet casein powders from 11 dairy companies were analysed. Nitrate levels were at 5 - 120 ppm for skim milk, at less than 5 to 88 ppm for acid casein and at less than 5 to 56 ppm for rennet casein. Some 22% of dairy powder samples contained nitrate above the specified limit of 30 ppm. The second study, during the period September 1998 to August 1999, on 144 dairy powder samples from 10 dairy companies, gave similar results; approximately 35% of samples contained nitrate in excess of 30 ppm.



An investigation of the cause of these elevated nitrate levels in dairy powder samples was undertaken by measuring the nitrate level in product at the various stages of the manufacturing process. The nitrate level in the starting whole milk was low (less than 2 ppm) but levels in excess of 30 ppm were found in intermediate products and in the final dairy powder product. These elevated nitrate levels were associated with the first production runs immediately following equipment cleaning and were not found in later production runs, when nitrate was at less than 30 ppm in the dairy powder product. These results identify factors associated with equipment cleaning and start-up as contributing to high nitrate levels in dairy powders.



## RECOMMENDATIONS TO INDUSTRY

- In the case of veterinary drugs and pesticides, residue levels in Irish food products are generally low and, where problems have occurred, such as with chlortetracycline residues in pork or sheep meat or with ivermectin in farmed salmon, these may be avoided through use of permitted substances only, under the approved conditions of treatment, dosage and withholding periods.
- Irish dairy and meat products contain relatively low levels of contaminants such as mycotoxins, dioxins, heavy metals and PAHs. These low levels reflect good quality of animal feed and of the environment. It is important that the high quality of these conditions for animal production be maintained so as to ensure a continued high standard of quality and safety for Irish food products. Where manufacturing practices are contributing to a problem, such as nitrate levels in dairy powders, it is important that corrective actions are taken by the manufacturers.
- Continued monitoring of the status of food is required to provide current data to support the claims for Irish food products as green and wholesome. Irish food processors may obtain Summary Reports on studies of interest to them from The National Food Centre.



## PUBLICATIONS FROM THIS PROJECT

**O'Keeffe, M. and Kennedy, O.** 1998. Residues - a food safety problem? *Journal of Food Safety*, 18, 297-319.

**O'Keeffe, M. and Farrell, F.** 2000. The importance of chemical residues as a food safety issue. *Irish Journal of Agricultural and Food Research* 39, 257-264.

**Kennedy, O. and O'Keeffe, M.** 1997. Food safety - the residue database project. *The Irish Scientist*, 5, 44.

**O'Keeffe, M. and Kennedy, O.** 1998. Database on residues of veterinary drugs and other contaminants in food. In: *Third International Symposium on Hormone and Veterinary Drug Residue Analysis*. Bruges, Belgium, 2 June, p10.

**O'Keeffe, M., Kennedy, O., Byrne, P., Farrell, F. and Horne, E.** 1999. Database on residues of veterinary drugs and other contaminants in food. *Irish Journal of Agricultural and Food Research* 38 (1) 158.

**Nolan, M-L., Dooley, M. and O'Keeffe, M.** 2000. Antibiotic residue testing in kidney tissue: comparison of a modified One - Plate Test with the EC Four - Plate Method and Charm II Tests. *Irish Journal of Agricultural and Food Research* 39 (1) 144-145.

**Nolan, M-L., Dooley, M., Nugent, A. and O'Keeffe, M.** 2000. Antibiotic residue testing in kidney tissue: comparison of a modified One-Plate test with the EC Four-plate test and the Charm II test. In: *Proceedings of EuroResidue IV Conference on Veterinary Drugs in Food*, Veldhoven, The Netherlands. ISBN 91-804925-1-5, 781-785.

**Farrell, F., O'Keeffe, M. and McGrath, D.** 2001. The Food Residue Database. *Irish Journal of Agricultural and Food Research* 40 (1) 117.

**O'Keeffe, M.** 1999. *Food Residue Database Newsletter*, December. The National Food Centre, Teagasc. 8 pp.

**O'Keeffe, M.** 2001. *Food Residue Database Newsletter*, June. The National Food Centre, Teagasc, 4 pp.

# The National Food Centre

RESEARCH & TRAINING FOR THE FOOD INDUSTRY

Dunsinea, Castleknock, Dublin 15, Ireland.

Telephone: (+353 1) 805 9500

Fax: (+353 1) 805 9550