# Occurrence and trend analysis of organochlorine in animal feed

Organochlorine pesticides and non-dioxin-like PCBs

P. Adamse, R. Peters, H.J. van Egmond and J. de Jong

RIKILT
WAGENINGEN UR

# Occurrence and trend analysis of organochlorine in animal feed

Organochlorine pesticides and non-dioxin-like PCBs

P. Adamse, R. Peters, H.J. van Egmond and J. de Jong

This research was funded by the Dutch Ministry of Economic Affairs, WOT programme Food Safety, theme Animal feed.

RIKILT Wageningen UR
Wageningen, January 2014

RIKILT report 2013.009

RIKILT

WAGENINGEN UR

Adamse, P., R. Peters, H.J. van Egmond and J. de Jong, 2014. *Occurrence and trend analysis of organochlorine in animal feed; Organochlorine pesticides and non-dioxin-like PCBs.* Wageningen, RIKILT Wageningen UR (University & Research centre), RIKILT report 2013.009. 42 pp.; 7 fig.; 17 tab.; 20 ref.

Project number: 122.7225001
BAS-code: WOT-02-004-012
Project title: Statistische onderbouwing van een risicogebaseerd Nationaal Plan Diervoeders (incl. trendanalyses analyseresultaten diervoeders) (WOT-02-004-012)
Project leader: M. de Nijs



P.O. Box 230, 6700 AA Wageningen, The Netherlands, T +31 (0)317 48 02 56,
E info.RIKILT@wur.nl, www.wageningenUR.nl/en/rikilt. RIKILT is part of Wageningen UR (University & Research centre).



RIKILT report 2013.009

Distribution list:

- Netherlands Food and Consumer Product Safety Authority, Utrecht, the Netherlands (NVWA); ); R.G. Herbes, H.A. van der Schee, E. Olde Heuvel, R.C.M. Theelen, J.A. van Rhijn, H.J. Hagen-Lenselink, S. van der Meijs
- Netherlands Ministry of Economic Affairs, Department of Agro Plant Supply Chain and Food Quality, The Hague, the Netherlands (EZ-PAV); F. Leijdekkers
- Netherlands Ministry of Economic Affairs, Department of Agro Knowledge, The Hague, the Netherlands (EZ-AK); G.J. Greutink
- Netherlands National Institute for Public Health and the Environment, Bilthoven, the Netherlands (RIVM); J. van Klaveren
- European Commission, Directorate General for Health and Consumers (EC, DG SANCO E-1); F. Verstraete
- European Food Safety Authority, Contaminations in the Food Chain Panel (EFSA-CONTAM); M. Eskola
- Commodity Board Animal Feed (PDV); W.J.C. Swinkels
- Trust Feed: F. van Rossem, Y. Yntema
- GMP+ International: J. den Hartog

# Contents

# Summary

More than 5000 feed samples were analysed for organochlorine compounds in the period 2001-2011. The materials in these samples originate from all over the world, but were sampled in the Netherlands. Samples were taken by the Netherlands Food and Consumer Products Safety Authority (NVWA) for the Netherlands National Feed Monitoring program (NP samples) and by the feed industry (IND samples).

Only a limited number of samples contain levels of organochlorine; 174 out of 3972 samples from NP and 291 out of 1932 samples from IND. Even fewer (18 in total) contain levels equal to or above the regulatory limit for the particular feed material. Trend analysis therefore is difficult because of the limited number of positive samples. Endosulfan is the exception, occurring mainly in soybean oil.

In the NP and IND dataset it is difficult to decide whether a concentration of endosulfan in vegetable oil samples exceeds the limit since it is often unclear from the description of the samples whether it concerns crude oil (limit 1.0 mg/kg) or processed oil (limit 0.5 mg/kg). Using the most strict limit (0.5 mg/kg) 6 samples of soya bean oil exceed the regulatory limit. When the limit of 1.0 mg/kg is used no samples exceed the regulatory limit. There is no significant in- or decrease in the average endosulfan concentration in soya bean oil in the period 2001 till 2011.

Only five samples contain DDT concentrations above the limit of quantification, one of them (niger seeds) is at the regulatory limit. Four samples from NP contain aldin_dieldrin concentrations equal to or above the limit of quantification (LOQ), two of those values slightly exceed the regulatory limit for feed materials and compound feed (complementary pig feed and unspecified complementary compound feed, containing 50% fish oil). The other two samples (fish oil) do not exceed the regulatory limit. No samples have been reported with an endrin or methoxychlor concentration above the LOQ. A few samples exceed the LOQ but none exceed the regulatory limits for heptachlor, HCH-alpha, -beta and -gamma, HCB, toxaphene, chlordane.

Three samples (two sorghum and one millet) from NP and IND contain dichlorvos concentration above the LOQ, all three above the MRL for food. There is no regulatory limit for feed, but when it is a single commodity the limit for food also applies to feed.

In the combined NP (incl. additional GC-HRMS dataset) and IND dataset 316 of 5904 samples have NDL-PCB concentrations that exceed the LOQ. Most of the positive samples are either fat and oil (vegetable or animal), fish meal or minerals. In total 15 samples exceed the regulatory limit that has been set in 2012, all from NP. The average values for NDL-PCBs content over the period 2001-2011 are below the LOQ of the methods and consequently it is not meaningful to perform a trend analysis. This applies to all feeds combined and to most of the separate product(group)s. The exception is fish oil where the average NDL-PCB concentration remains below the regulatory limit but increases significantly in the period 2001-2011.

It is important to continue monitoring products where the regulatory limits have been exceeded or where levels close to the regulatory limit are found frequently. This is especially the case for endosulfan. Monitoring for NDL-PCBs should continue as well because maximum levels have only been introduced recently and the LOQs of the methods in many cases were too high compared to those regulatory limits. Efforts should be made to harmonize (LOQs of) the methods applied by industry and the National Plan to enable combining results in one dataset.

# 1 Introduction

Organochlorine compounds in feeds are present as residue of herbicides, insecticides, and fungicides (i.e. pesticides) as well as environmental contamination with industrial chemicals (such as non-dioxin-like PCBs). Organochlorine compounds are potential endocrine disrupting compounds, which means that they might interfere with the endocrine systems of animals. Depending on their physico-chemical characteristics, some substances are metabolized into naturally occurring and generally harmless constituents. Other substances are persistent and remain in the animal and in animal products intended for human consumption (Kan and Meijer, 2007). For example, lindane is a compound with detectable accumulation in the animal and DDT is a compound with high accumulation in the animal. Accumulation of endosulfan and methoxychlor in animals is low (Kan and Meijer, 2007). Organochlorine compounds are more soluble in fat than in water. This allows accumulation in fatty tissues.

Officially most organochlorine compounds are banned from use. Endosulfan is one of the few organochlorine pesticides that is still in use although the UN Stockholm Convention has agreed in 2011 to add endosulfan to the United Nations' list of persistent organic pollutants to be eliminated world-wide (UN, 2011).

In this report historical data are used to give insight into the trends in levels of organochlorine compounds in compound feeds and feeding materials for animals in the Netherlands. The main focus is on pesticides, but non-dioxin-like PCBs have been studied as well. The latter will be included in the trend analysis reports about dioxins and dioxin-like PCBs in the future. This analysis was performed on request of the NVWA (Netherlands Food and Consumer Product Safety Authority). The results of these analyses will enable the NVWA to develop a risk-directed sampling strategy for the National Feed Monitoring program.

From 2007 on reports have been published on the trends in levels of contaminants or residues in compound feeds and feed materials: aflatoxin B1 and dioxins en dioxin-like PCB's (Adamse *et al.*, 2007), heavy metals (Adamse *et al*., 2009a), animal proteins (Adamse *et al*., 2009b), copper and zinc (Adamse *et al*., 2011a) and mycotoxins (Adamse *et al*., 2011b). These trend analyses have been carried out in order to use acquired knowledge to reach a more risk-driven sampling plan in the National Feed Monitoring program. For these reports data have been submitted by the National Feed Monitoring program (NVWA, NP) and by Trust Feed (a co-operative alliance regarding food safety formed by seventy Dutch and Belgian animal feed producers). The data relate only to monitoring research.

The NP dataset is also stored in the KAP database. The Quality Program Agricultural Products (KAP) is a collaboration between agricultural businesses and the Dutch Government. KAP has been designed to focus on continuous monitoring the level of contaminants and residues in agricultural products such as vegetables, fruit, milk, meat, fish and feed. KAP processes the results of monitoring programmes and reports on more than 200,000 measurement results per year, from 1989 to date (van Klaveren *et al.,* 1997).

The feed materials from the Netherlands National Feed monitoring program and from the industry were analysed for endosulfan, DDT, aldrin-dieldrin, endrin, heptachlor, HCH-alpha, -beta and -gamma, HCB, toxaphene, chlordane, methoxychlor, dichlorvos and non-dioxin-like PCBs (dichlorvos is in fact not an organochlorine but an organophosphate compound). In this report occurrence data, trends in the average content and (trends in) percentages of samples above the regulatory limits are reported for these organochlorine compounds. If applicable, the relative contribution of individual congeners or metabolites to the sum for this compound is shown as well. Results are compared to notifications and alerts from RASFF (Rapid Alert System for Food and Feed) and, in case of endosulfan, compared to the results of samples obtained from targeted sampling by the former Netherlands AID (Algemene Inspectie Dienst; General Inspection Service, now part of the NVWA).

# 2 Material and methods

## 2.1 Definition of feed groups

- Feed:
  - Feed may take the form of feed materials, feed additives, medicated feeding stuffs, compound feed or pre-mixtures (from REGULATION (EC) No 767/2009)
- Feed materials:
  - Products of vegetable or animal origin, whose principal purpose is to meet animals' nutritional needs, in their natural state, fresh or preserved, and products derived from the industrial processing thereof, and organic or inorganic substances, whether or not containing feed additives, which are intended for use in oral animal-feeding either directly as such, or after processing, or in the preparation of compound feed, or as carrier of pre-mixtures;
- Compound feed:
  - A mixture of at least two feed materials, whether or not containing feed additives, for oral animal-feeding in the form of complete or complementary feed;
- Complete feed:
  - Compound feed which, by reason of its composition, is sufficient for a daily ration;
- Complementary feed:
  - Compound feed which has a high content of certain substances but which, by reason of its composition, is sufficient for a daily ration only if used in combination with other feed;
- Pre-mixtures:
  - Mixtures of feed additives or mixtures of one or more feed additives with feed materials or water used as carriers, not intended for direct feeding to animals (from Regulation (EC) No 1831/2003);
- Mineral feed[1]:
  - Complementary feed containing at least 40% crude ash;
- Milk replacer[2]:
  - Compound feed administered in dry form or after dilution in a given quantity of liquid for feeding young animals as a complement to, or substitute for, post-colostral milk or for feeding young animals such as calves, lambs or kids intended for slaughter;
- Feed additives:
  - Substances, micro-organisms or preparations, other than feed material and pre-mixtures, which are intentionally added to feed or water in order to perform, in particular, one or more of the functions mentioned in Article 5(3) in Regulation (EC) No 1831/2003;
- Medicated feeding stuffs:
  - The rules for the marketing of medicated feeding stuffs are set out in Council Directive 90/167/EEC of 26 March 1990 laying down the conditions governing the preparation, placing on the market and use of medicated feeding stuffs in the Community.

## 2.2 Material

Data have been submitted by the Netherlands National Feed Monitoring program (dataset NP) and by the Dutch feed industry (dataset IND). The data from the industry are submitted by Trust Feed, a co-operative alliance regarding feed safety formed by seventy Dutch and Belgian animal feed producers. The data relate only to monitoring research and do not contain targeted samples taken as a follow-up on incidents. In most samples all organochlorine compounds were analysed.

[1] Not mentioned in this report.
[2] Not mentioned in this report.

Table 2.1

*Number of monitoring samples per year.*

| Source \ Year | 2001 | 2002 | 2003 | 2004 | 2005 | 2006 | 2007 | 2008 | 2009 | 2010 | 2011 | Total |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NP | 462 | 434 | 352 | 243 | 391 | 178 | 272 | 418 | 491 | 422 | 309 | 3972 |
| IND | | | | | 6 | 266 | 319 | 348 | 337 | 410 | 246 | 1932 |
| all | 462 | 434 | 352 | 243 | 397 | 444 | 591 | 766 | 828 | 832 | 555 | 5904 |

NP = National Feed monitoring program samples; IND = Industry monitoring samples.

## 2.3 Methods

### 2.3.1 Selection of products for trend analysis

The products studied are feed materials such as cereals, roughages and forages, oil-containing plants as well as compound feeds. Regulatory limits for endosulfan, aldrin-dieldrin and other organochlorine pesticides (OCPs) are laid down in Directive 2002/32/EC (+ amendments) (Table 6.1 in Annex). Regulatory limits for non-dioxin-like polychlorinated biphenyls (NDL-PCBs) have recently been introduced. These regulatory limits have been included in Directive 2002/32/EC by means of Regulation (EU) No 277/2012 (Table 6.2 in Annex). Products analysed in this study are initially grouped similar to the groups in Directive 2002/32/EC and Regulation (EU) No 277/2012 . Where applicable (i.e. enough samples and some values above the limit of quantification) subgroups are analysed separately as well. For most samples the country of origin is not known so no analysis on the basis of country of origin can be performed.

The product (sub)groups analysed are:

- All feeds.
- Compound feeds:
  - Complete feeds (per animal).
  - Complementary feeds (per animal).
- Feed materials:
  - Maize and maize products derived from the processing thereof.
  - Oilseeds and products derived from the processing thereof, except crude vegetable oil.
  - Crude vegetable oil.
  - Fish, other aquatic animals, their products and by-products with the exception of fish oil.
  - Fish oil.

The product groups not analysed are:

- Premixtures -> no data available.
- Feed additives -> only 37 samples in the dataset, all of them negative.

### 2.3.2 Organochlorine compounds

The compounds analysed are shown in Table 2.2. Most compounds are a sum of individual components, such as endosulfan (sum of alpha- and beta- isomers and of endosulfansulphate). Because non-dioxin-like PCBs (NDL-PCBs) where analysed in the pesticide-multimethod together with organochlorine compounds the results are analysed in this report. NDL-PCBs are analysed in the scope of dioxin-analysis of the NP feed samples as well. Since NDL-PCBs do not belong to the group organochlorine compounds future results will be discussed together with dioxins and dioxin-like PCB's.

Table 2.2

*Organochlorine compounds studied in this report*

| Compounds | Sum of | Accumulation in animals* |
|---|---|---|
| Aldrin incl. dieldrin | aldrin and dieldrin | high |
| Chlordane | cis- and transisomers and of oxychlordane | detectable |
| Sum DDT | DDT-, TDE- and DDE isomers | high |
| Endosulfan | alpha- and beta- isomers and of endosulfansulphate | low |
| Endrin | endrin and of deltaketoi-endrin | high |
| HCH-alpha | alpha-isomers | detectable |
| HCH-beta | beta-isomers | high |
| HCH-gamma (lindane) | gamma-isomers | detectable |
| Heptachlorepoxide (HCE) | heptachlor and heptachlorepoxide | high |
| Hexachlorobenzene (HCB) | n.a. | high |
| Methoxychlor | n.a. | low |
| Toxaphene (camphechlor) | indicator congeners CHB 26, 50 and 62 | variable |
| Non-dioxin-like PCBs (NDL-PCBs) | sum of PCB 28, PCB 52, PCB 101, PCB 138, PCB 153 and PCB 180 (ICES – 6), (sum indicator PCB) | variable |

* According to Kan and Meijer (2007).

### 2.3.3 RASFF notifications and alerts

Before analysing the data from the National Plan (NP) and the industry (IND) the occurrence of notifications for organochlorine compounds in feed and feed materials were checked in the RASFF (Rapid Alert System for Food and Feed). In the RASFF system notifications and alerts are reported to the European Commission by the national authorities. This occurs after food or feed inspectors have inspected a product on the market or at the border and found that the product is non-compliant with EU regulations and needs to be reported inside the national system. The authority decides if the issue falls under the scope of the RASFF and reports it to the national RASFF contact point. The national contact point verifies and completes the RASFF notification where necessary and forwards it to the European Commission.

Numerous notifications and alerts have been registered in RASFF that concern organochlorine compounds. Most alerts concern DDT, but also endosulfan, dichlorvos, HCH and heptachlor have been detected. In the following paragraphs the alerts from those compounds are shown and compared with the results for those organochlorine compounds in the data from NP and IND.

### 2.3.4 Methods of analysis

For the NP feed materials a detection method has been used that is suitable for both qualitative screening and quantitative determination and targets over 100 pesticides, most recently described by van der Lee *et al.* (2008). The method consist of extraction with ethylacetate/cyclohexane (oil and fat) or ethyl acetate (feed materials), clean-up by gel permeation chromatography (GPC) and dispersive solid-phase extraction (SPE) with primary secondary amine phase (PSA), and analysis by comprehensive two-dimensional gas chromatography with full scan time-of-flight mass spectrometric detection (GC×GC–TOF-MS) or GC-ECD. For LOQs see Table 2.3.

Between 2001-2005 the results from each analysis were stored for each component (a limited set), also zero values (< LOQ). Starting in 2006 only the positive individual components are registered. Therefore zeros have been added for the other components to allow trend analysis. When only a nominal value (N for negative) has been registered the value zero (< LOQ) has been added for all individual components.

Part of the samples from the NP dataset presented in this report were analysed for NDL-PCBs in combination with the analysis for dioxins and DL-PCBs. These samples, mainly fish products, animal fat and minerals were analysed with GC-HRMS, according to the method of Traag *et al*. (2008).

The methods used by the industry for fat and oil are in accordance with NEN EN 1528 (Fatty food - Determination of pesticides and polychlorinated biphenyls) and for samples of plant origin NEN EN 12393 (Non-fatty foods - Multi-residue methods for determining pesticide residues by means of a gas chromatograph). In general the limit of quantification (LOQ) is as described in Table 2.3. Occasionally a lower or higher LOQ has been reported. In some cases it is not clear if the value in Table 2.3 refers to LOQ or LOD (limit of detection).

For a number of organochlorine compounds the LOQs of the methods applied by industry and in the National Plan differ by a factor of 2 to 10. For NDL-PCBs for the NP samples LOQs of individual congeners are available while for the IND samples the sum of NDL-PCBs is reported. The LOQ of this sum differs largely, between 0.001 and 5 mg/kg.

Table 2.3
*Limits of quantification (mg/kg).*

| Dataset | NP | | NP | | IND | |
|---|---|---|---|---|---|---|
| Years | 2001-2006 | | 2006-2011 | | 2006-2011 | |
| Product type | Low-fat crops | Oil and fat | Low-fat crops | Oil and fat | Low-fat crops | Oil and fat, pulp |
| Aldrin incl. dieldrin, chlordane | 0.005 | 0.02 | 0.005 | 0.02 | 0.005 | 0.01 |
| DDT total, toxaphene | 0.02 | 0.1 | 0.02 | 0.1 | 0.005 | 0.01 |
| Endosulfan | 0.01 | 0.05 | 0.01 | 0.05 | 0.005 | 0.01 |
| Endrin | 0.01 | **0.04** | 0.01 | **0.02** | 0.005 | 0.01 |
| HCH-alpha, gamma, HCB | 0.005 | 0.01 | 0.005 | 0.01 | 0.005 | 0.01 |
| HCH-beta, HCE | 0.005 | 0.02 | 0.005 | 0.02 | 0.005 | 0.01 |
| Methoxychlor | 0.001 | na | na | na | 0.005 | 0.01 |
| NDL-PCBs (individual congeners) - screening | 0.002 | **0.03** | 0.002 | 0.01 | n.a. | n.a. |
| NDL-PCBs (individual congeners) - GC-HRMS | 0.0001 | 0.0001 | 0.0001 | 0.0001 | n.a. | n.a. |
| Sum NDL-PCBs | n.a. | n.a. | < limit | < limit | between 0.001 and 5 | |

NP = National Feed monitoring program samples; IND = Industry monitoring samples.

### 2.3.5 Statistics used for trend analysis on monitoring data

Results are visualized with the help of histograms and a descriptive analysis is carried out. The sample year is displayed on the X axis and the concentration (in mg/kg) on the left Y axis. The number of samples tested per year (stated as N) is visible on a second Y axis at the right side of the graph. The concentrations are shown as averages, median and 90th percentile or maximum values; with median and 90th percentile absent when less than 5 and 10 samples, respectively, have been analysed. In addition, regulatory limits (valid in 2012) are indicated in the graph using a grey background.

The average (avg), defined as the arithmetic mean, is the central tendency of all sample outcomes. The larger the sample size (number of samples tested per year), the more reliable the average, as this value is greatly influenced by outliers (e.g. measurement errors or incidents). Therefore the median is calculated as well. The median is the value which separates the higher half of a sample outcome (concentrations) from the lower half, being the middle value. When compared to the average value, it is a good indicator if a distribution of measurements is skewed, as in this way outliers become less important. The 90th percentile (90per) indicates that 90% of the sample outcomes is below the given concentration respectively 10% is above the value. This statistic value may be useful as it gives a picture of the bandwidth of the concentrations.

In order to visualize trends in the graph, regression analysis was used by adding a linear trend line to the graph. The trend line is based on the average concentrations per year. Regression is used to predict future trends on the basis of tendencies found in the past and to look at the variability across time periods. Hereby the equation $y = mx + b$ describes a straight line for a set of data where x is the independent variable, y is the dependent variable, m represents the slope of the line (also known as the regression coefficient) and b represents the y-intercept (that is, the point where the line crosses the left y-axis). Furthermore, $R^2$ is the correlation factor which indicates how well the trend line fits the observations. A value of zero indicates no relationship (absolute randomness) between the x variable (year) and y variable (average concentration), whereas a value of 1 indicates perfect correlation (i.e. the values of y all lie on the trend line). If $R^2$ is below 0.3, no correlation can be found and a trend cannot be analysed as there is no significant connection between changing concentrations over the years.

Where possible a more in depth statistical analysis is carried out. The data are not normally distributed as the compounds are usually present in low concentrations and an asymmetric distribution is expected. Furthermore in respect to the dataset, not a constant number of monitoring samples is taken per given period of time (year) and the feed product is not always linked to the same production site and/or country of origin. In this case a non-parametric statistical test has been used, such as the Mann-Kendall test (to measure for the presence of consistent trends) or the Sen slope test - (to measure the magnitude of the slope).

The Mann-Kendall test measures the presence of monotonic increasing or decreasing trends, by comparing the relative magnitudes of sample data rather than the data values themselves (Gilbert, 1987). The advantage of this test is that the data need not to conform to any particular distribution and missing values are allowed. The template MAKESENS (Salmi *et al.,* 2002) was used to assess trends with the Mann-Kendall test. This template provides a significance value for the annual average concentrations per feed (sub)group per time period. It indicates whether the trend found with the descriptive analysis (graphs) really changed significantly over the period of investigation. A trend found with a significance level of e.g. $p=0.05$ means that there is a 5% probability that the values are from a random distribution and that there is no trend. Thus the existence of a monotonic trend is very likely (95%). The S value (or Z value when 10 or more time points are available) indicates an upward (positive value) or downward (negative value) trend. The higher the value the more increasing or decreasing trend can be found. The Mann-Kendall test requires at least 4 average values in a time series.

# 3 Results

## 3.1 Initial data analysis

Most organochlorine compounds (OCs) are rarely found in the feed samples (Figure 3.1A). NDL-PCB's occur more frequently. The NP dataset (Figure 3.1B) contains 3972 unique sample numbers. Since the compounds are analysed with multi-methods it is assumed that all samples have been analysed for all compounds. The dataset from IND starts in 2005 (Figure 3.1C), consists of 1932 unique sample numbers and contains the 'compound' Pesticides. This compound is the sum of all organochlorine pesticides and has not been specified further. This makes it difficult to compare the relative contribution of individual compounds. Combining both datasets (Figure 3.1A) shows that the largest contributions to the samples with values above the LOQ are from endosulfan, sum indicator PCBs (=NDL-PCBs) and aldrin/dieldrin. A limited amount of samples have concentrations of OCs exceeding the legal limit for that compound (endosulfan, aldrin-dieldrin, DDT and NDL-PCB's, see red bars in Figure 3.1A, B and C). The occurrence of OCs in feed samples and potential trends over the period 2001-2011 have been studied in more detail for individual compounds in the following paragraphs.

## 3.2 Endosulfan

For most feeds the regulatory limit for endosulfan is 0.1 mg/kg. For fish feed the limit is considerably lower (0.005 mg/kg) and for crude vegetable oil, (products from) oil seeds and (products from (maize) the limit is higher (Table 3.1).

Table 3.1
*Regulatory limits for Endosulfan (sum of alpha- and beta- isomers and of endosulfansulphate expressed as endosulfan) in feed (DIRECTIVE 2002/32/EC).*

| Product (group) | EU Limit (mg/kg) |
|---|---|
| All feed materials and compound feed with the exception of | 0.1 |
| • Maize and maize products derived from the processing thereof | 0.2 |
| • Oilseeds and products derived from the processing thereof with the exception of crude vegetable oil | 0.5 |
| • Crude vegetable oil | 1.0 |
| • Complete feed for fish | 0.005 |

In RASFF two alerts/notifications are registered specifically related to endosulfan (Table 3.2). The fat/oil sample, presumably soya oil, is reported as originating from the Netherlands. However, since the Netherlands do not produce soybeans but only process them the real country of origin would be important to know. In fish food levels are found that are two up to six times higher than the regulatory limit, the concentrations found in the fat/oil samples are between ten to twenty times the limit. The fat/oil incident is probably related to samples taken by the AID (General Inspection Service). In RIKILT report 2010.521 (Egmond, *et al.,* 2010) the results of feed samples taken by AID at farms in the period 2005-2009 in the context of monitoring or special surveys are presented. Endosulfan concentrations reported sometimes exceed the regulatory limit (see Table 3.1): four samples of vegetable oil (ranging from 0.32 to 5.3 mg/kg) and one sample of soya oil (8.8 mg/kg) in 2007; two samples of soya oil (0.21-0.73 mg/kg) and two samples of vegetable oil (3.7-6.5 mg/kg) in 2008. The AID samples with 5.3 and 8.8 mg/kg endosulfan have been found in November 2007 and are most likely related to the incidents reported in RASFF in 2008.

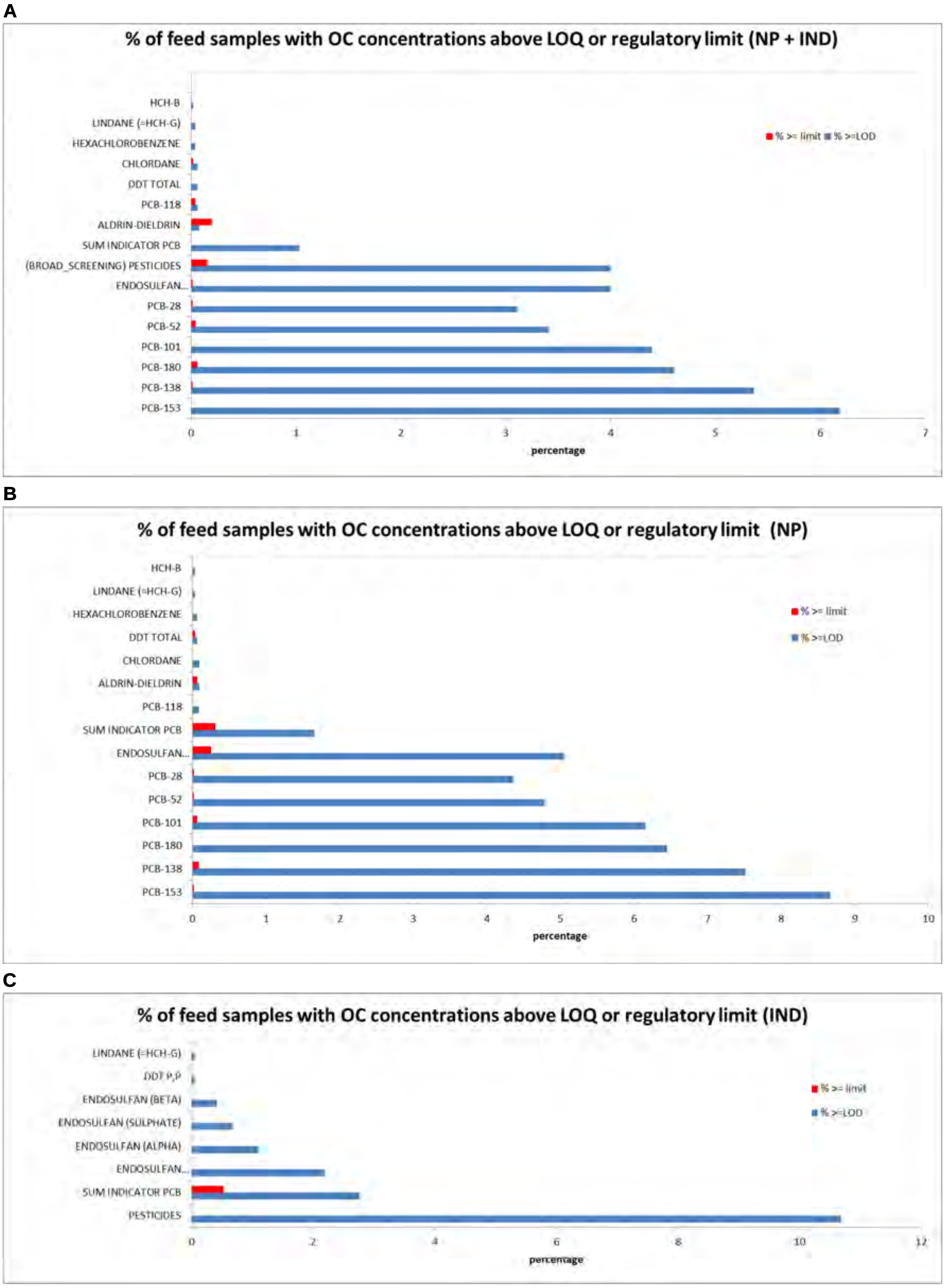



***Figure 3.1*** *Organochlorine compounds in feeds, percentages of samples with concentrations above LOQ or above the regulatory limit; A= NP + IND; B = NP; C = IND.*

Table 3.2

*RASFF alerts and notifications related to Endosulfan.*

| Product group | Reference | Notification type | Origin | Notified by | Value(s) | Unit | EU Limit | Subject |
|---|---|---|---|---|---|---|---|---|
| Fat/oil* | 2008.0314 | alert | Netherlands | Netherlands | 5.3; 8.8 | mg/kg | 0.5 | endosulfan (5.3; 8.8 mg/kg - ppm) in fats and oils from the Netherlands |
| Fish food | 2008.0334 | information | Italy | Germany | 0.013; 0.012; 0.030 | mg/kg | 0.005 | endosulfan (0.013; 0.012; 0.030 mg/kg - ppm) in single feed for trout from Italy |

* Probably soya oil (see above).

In Figure 3.2 the percentage of samples from NP and IND with endosulfan concentrations above the LOQ (variable, between 0.01 and 0.06) and above the regulatory EU limit (0.1, 0.5 or 1 mg/kg) is shown. Only in apple(pulp), fatty acids and soybean oil some samples have concentrations equal to or above the regulatory limit. From apple only eight samples were analysed in the entire period, two of them, in 2005, with values above the LOQ, one of those exceeding the regulatory limit (0.1 mg/kg).

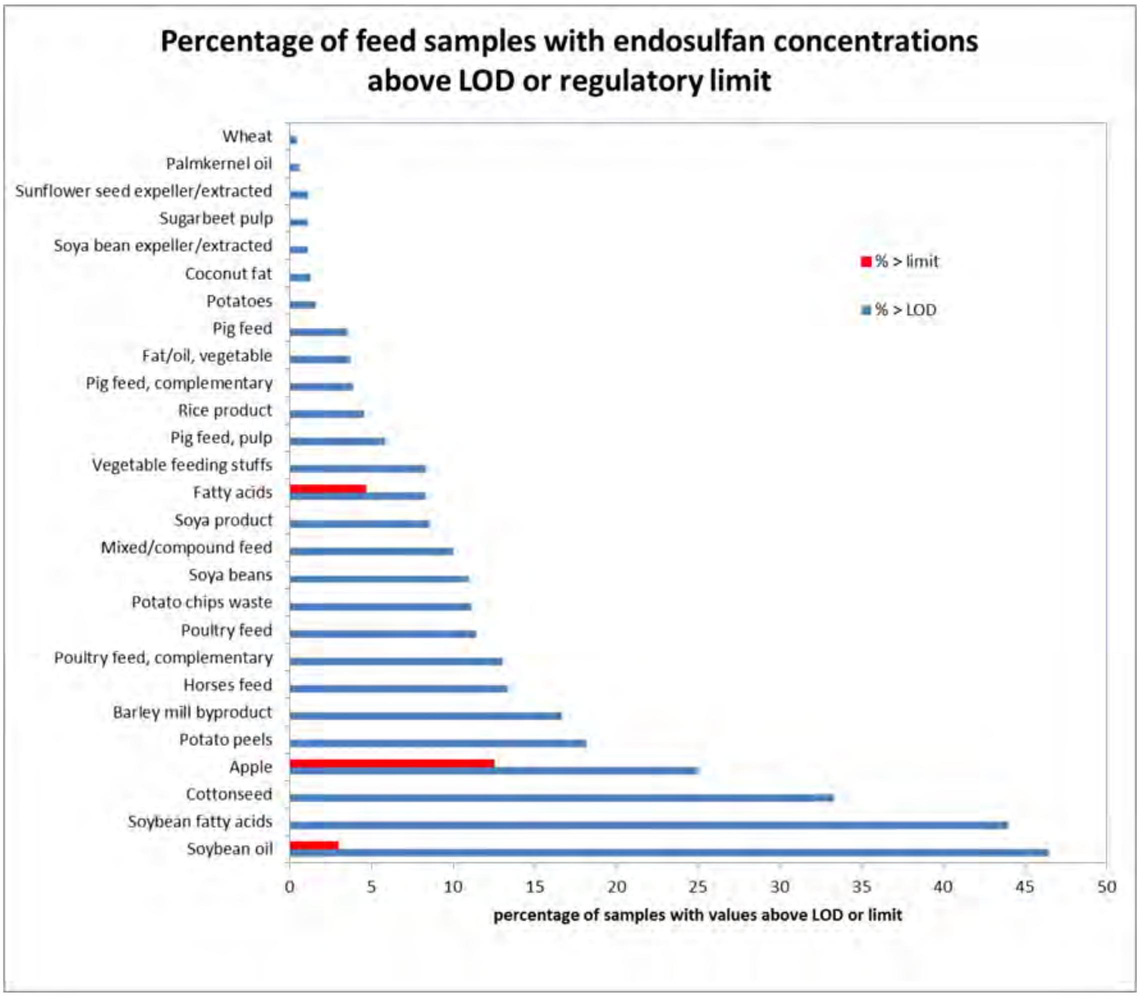



***Figure 3.2*** *Incidence of endosulfan concentrations above LOQ or regulatory limit (as percentage of the total number of samples analysed) in all feed products with 1 or more samples > LOQ;*

Neither in the NP or the IND samples levels are found that exceed the limit as dramatically as in the RASFF alerts. However, twelve of the 189 samples (Table 3.3) with endosulfan levels above the limit of quantification (of 5144 samples in total, see also Table 6.3 and Table 6.5 in Annex) contain concentrations that exceed the regulatory limit: apple pulp (NP, 2005: 0.33 mg/kg), fatty acids (NP, 2006, 2007: 0.7-2 mg/kg), soybean oil (NP and IND, 0.06-0.71 mg/kg). For the soya bean oil and palm oil samples it is not clear whether the limit for *oilseeds and products derived from the processing thereof with the exception of crude vegetable oil* (0.5 mg/kg) should be used or the limit for *crude vegetable oil* (1.0 mg/kg) since it was not registered whether it concerned crude vegetable oil or not. For soybean oil, values have been found in the range of 0.06 mg/kg - 0.71 mg/kg. When 1.0 mg/kg is used as limit for endosulfan in oil no samples exceed the limit. Only one sample of fish feed has been analysed (NP, 2003). This sample did not contain a detectable level of endosulfan.

Table 3.3

*Incidence of endosulfan in animal feed.*

| Product (group) | Year(s) with positives | N total | N pos | N ≥ regulatory limit | % N pos | % N ≥ regulatory limit | Result -range (mg/kg) (min-max) | | EU Limit (mg/kg) |
|---|---|---|---|---|---|---|---|---|---|
| Apple | 2005 | 8 | 2 | 1 | 25 | 12.5 | 0.09 | 0.33 | 0.1 |
| Barley mill byproduct | 2006 | 6 | 1 | 0 | 17 | 0 | 0 | 0 | 0.1 |
| Coconut fat | 2010 | 80 | 1 | 0 | 1 | 0 | 0.24 | 0.24 | 0.1 |
| Cottonseed | 2002 | 3 | 1 | 0 | 33 | 0 | 0.03 | 0.03 | 0.1 |
| Fat/oil, vegetable | 2004,2006 | 54 | 2 | 0 | 4 | 0 | 0.06 | 0.49 | 0.5 or 1.0[1] |
| Fatty acids | 2006,2007, 2009 | 108 | 9 | 5 | 8 | 5 | 0.12 | 2 | 0.5 |
| Horses feed | 2004 | 15 | 2 | 0 | 13 | 0 | 0.01 | 0.01 | 0.1 |
| Mixed/compound feed | 2004 | 10 | 1 | 0 | 10 | 0 | 0.01 | 0.01 | 0.1 |
| Palmkernel oil | 2010 | 169 | 1 | 0 | 1 | 0 | 0.16 | 0.16 | 0.5 or 1.0[1] |
| Pig feed | 2003,2004, 2005 | 254 | 9 | 0 | 4 | 0 | 0.01 | 0.02 | 0.1 |
| Pig feed, complementary | 2003 | 8 | 1 | 0 | 4 | 0 | 0.01 | 0.01 | 0.1 |
| Pig feed, pulp | 2005 | 51 | 3 | 0 | 6 | 0 | 0.01 | 0.02 | 0.1 |
| Potato chips waste | 2005 | 9 | 1 | 0 | 11 | 0 | 0.04 | 0.04 | 0.1 |
| Potato peels | 2005 | 11 | 2 | 0 | 18 | 0 | 0.02 | 0.02 | 0.1 |
| Potatoes | 2010 | 63 | 1 | 0 | 2 | 0 | 0.01 | 0.01 | 0.1 |
| Poultry feed | 2003, 2004, 2005 | 140 | 16 | 0 | 11 | 0 | 0.01 | 0.03 | 0.1 |
| Poultry feed, complementary | 2003, 2004 | 4 | 3 | 0 | 13 | 0 | 0.01 | 0.03 | 0.1 |
| Rice product | 2008 | 22 | 1 | 0 | 5 | 0 | 0.05 | 0.05 | 0.1 |
| Soya bean expeller/extracted | 2003, 2005 | 354 | 4 | 0 | 1 | 0 | 0.01 | 0.02 | 0.5 |
| Soya beans | 2002, 2004, 2006, 2010 | 55 | 6 | 0 | 11 | 0 | 0.02 | 0.09 | 0.5 |
| Soya product | 2002, 2003, 2004, 2005, 2010, 2011 | 152 | 13 | 0 | 9 | 0 | 0.02 | 0.16 | 0.5 |
| Soybean fatty acids | 2008, 2010 | 25 | 11 | 0 | 44 | 0 | 0.01 | 0.16 | 0.5 |
| Soybean oil | 2001-2011 | 200 | 93 | 6 | 47 | 3 | 0.06 | 0.71 | 0.5 or 1.0[1] |
| Sugarbeet pulp | 2002 | 91 | 1 | 0 | 1 | 0 | 0.07 | 0.07 | 0.1 |
| Sunflower seed expeller/extracted | 2010 | 91 | 1 | 0 | 1 | 0 | 0.03 | 0.03 | 0.5 |
| Vegetable feeding stuffs | 2003 | 24 | 2 | 0 | 8 | 0 | 0.07 | 0.07 | 0.1 |
| Wheat | 2005 | 231 | 1 | 0 | 0 | 0 | 0.02 | 0.02 | 0.1 |
| **Total** | | **2238** | **189** | **12** | 308 | | | | |

N pos = number of samples with values above LOQ (and % of N total).

N ≥ limit= number of samples with values equal to or above the legal limit (and % of N total).

1 the limit for crude oil is 1.0 and for other oils 0.5, but it is not always clear whether it is crude oil or not.

In Figures 3.3, 3.4 and 3.5 some product groups are studied in more detail. To find consistent changes over the years in the endosulfan concentrations trend lines have been calculated using simple linear regression through the averages. Linear regression through the 90$^{th}$ percentile values would have been interesting as well, but not in every year enough samples (10 or more) were available to calculate the 90th percentile.

In soybean oil the percentage of samples exceeding the limit of 0.5 mg endosulfan/kg ranges from 0 to 18% as shown in Figure 3.3. However, when 1.0 mg/kg is used as limit for endosulfan in oil (the limit for crude oil) no soybean oil samples exceed the limit. There is no significant in- or decrease in either the percentage of samples exceeding the LOQ or equal to or exceeding the regulatory limit of 0.5 mg/kg. Most samples are from NP (125 out of 131). The positive samples from IND (from 2010 and 2011, detected with LOQ of 0.01 mg/kg) are all above the LOQ from NP (0.05 mg/kg). This enables combining both datasets without causing a false trend (which would have been the result of IND samples with concentrations below the LOQ of the NP method).

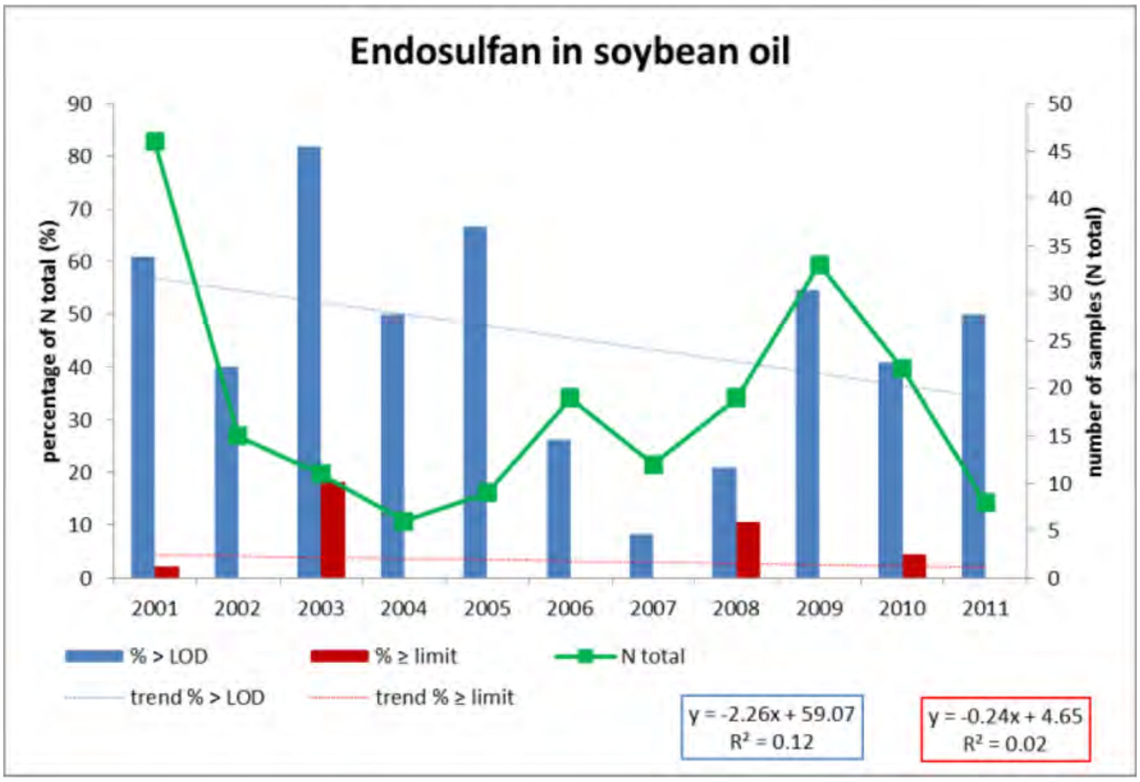



***Figure 3.3*** *Incidence of endosulfan concentrations above LOQ or regulatory limit (percentage)in soybean oil from NP and IND; regulatory limit = 0.5 mg/kg.*

In soybean oil (Figure 3.4) six samples equal or exceed the limit for *oilseeds and products derived from the processing thereof with the exception of crude vegetable oil* (0.5 mg/kg). There is no significant in- or decrease in the average endosulfan concentration between 2001 and 2011, neither in all samples combined nor in the NP samples(Figure 3.4B). The IND samples (Figure 3.4C) range from 2006 to 2011. There seems to be a significant increase in the average levels. However, the number of samples is too low for a reliable conclusion. In 2007 in RASFF a sample is reported of (presumably) soybean oil containing a level of 8.8 mg/kg endosulfan, originating from the Netherlands. In both NP and IND dataset for most samples the country of origin is not given so no conclusions with regard to the origin of the samples can be drawn.

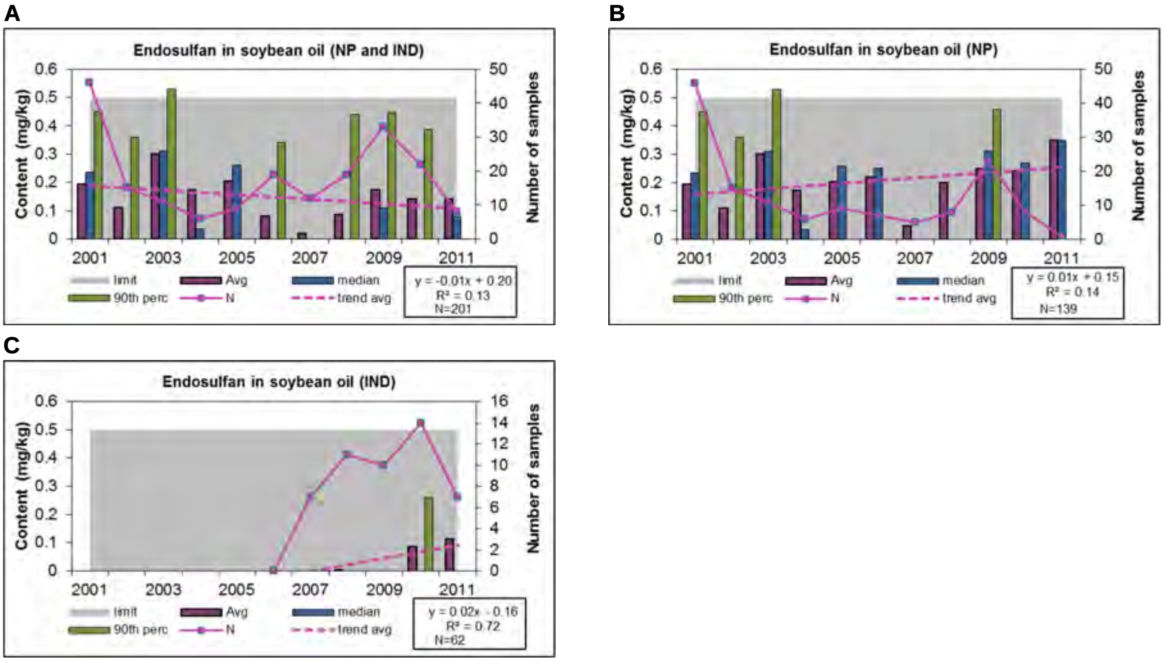



***Figure 3.4*** *Endosulfan in soybean oil; A= NP and IND combined; B= NP; C=IND; limit = 0.5 mg/kg.*

As reported before several samples of NP of fatty acids contain endosulfan concentrations above the regulatory limit (Figure 3.5A). No significant in- or decrease of the average levels between 2001 and 2011 can be shown. However, when only the period 2006-2011 is considered (with 92 of the 108 samples) there is a significant decrease ($R^2$ = 0.71, but based on only nine positive samples) of the average level (Figure 3.5B). In fatty acid samples from the industry (IND) no endosulfan concentrations above the LOQ have been detected (data not shown).

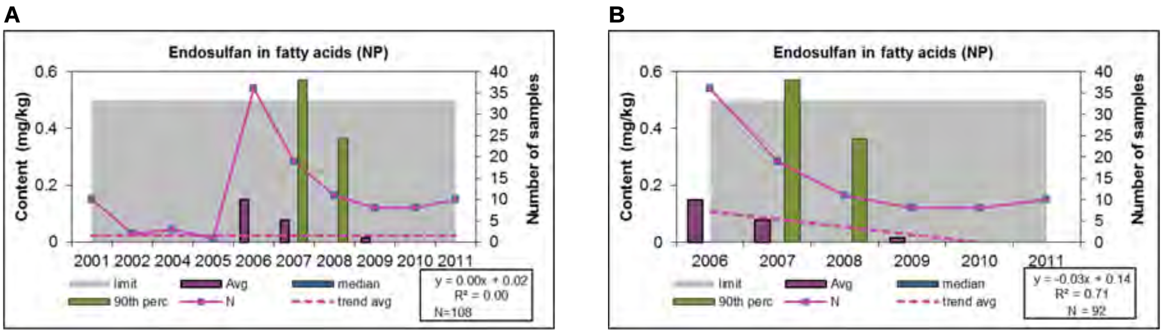



***Figure 3.5*** *Endosulfan in fatty acids; limit = 0.5 mg/kg; A = 2001-2011; B - 2006-2011.*

The number of samples taken from coconut oil is limited, both in the NP and in the IND set (63 samples, NP, 17 IND, data not shown). Only in 2008 one sample from the NP set contains an endosulfan concentration (0.24 mg/kg) above the limit of quantification (LOQ), but below the regulatory limit of 0.5 mg/kg. All other samples are below the LOQ.

## 3.3 DDT

For most feeds the regulatory limit for DDT (sum) is 0.05 mg/kg, only for fats and oil it is higher; 0.5 mg/kg (Table 3.4).

Table 3.4

*Regulatory limits for DDT (sum of DDT-, TDE- and DDE isomers, expressed as DDT) in feed (DIRECTIVE 2002/32/EC).*

| Product (group) | EU Limit (mg/kg) |
|---|---|
| All feed materials and compound feeds with the exception of | 0.05 |
| — fats and oils | 0.5 |

All of the RASFF alerts and notifications in this period related to DDT concern vegetable products or complementary mixed feeds (Table 3.5). The reported concentrations range from two up to almost eight times the legal limit.

Table 3.5

*RASFF alerts and notifications related to DDT with product type feed.*

| Product group | Reference | Notifi-cation type | Origin | Notified by | Value(s) | Unit | EU Limit | Subject |
|---|---|---|---|---|---|---|---|---|
| Wheat | 2002.425 | alert | Ukraine | France | 0.15 | mg/kg | 0.05 | DDT (0,150 mg/kg - ppm) in Wheat |
| Mixed/compound feed, complementary | 2008.0270 | alert | India | Austria | 190.28 | µg/kg | 50 | DDT (190.28 µg/kg - ppb) in complementary feed from India |
| Herbal mix | 2009.0789 | alert | India | Austria | 195.3 | µg/kg | 50 | prohibited substance DDT (195.3 µg/kg - ppb) in herbal feed supplement from India |
| Vegetable feeding stuffs | 2009.1759 | information | dispatched from UK | Austria | 141 | µg/kg | 50 | prohibited substance DDT (141 µg/kg - ppb) in devil's claw root from unknown origin, dispatched from the United Kingdom |
| Mixed/compound feed, complementary | 2010.0961 | information | India | Austria | 107.3 | µg/kg | 50 | prohibited substance DDT (107.3 µg/kg - ppb) in complementary feed for cattle, pigs, sheep, goats, poultry and game from India |
| Maize | 2011.0998 | information for attention | Ethiopia | Italy | 0.09 | mg/kg | 0.05 | prohibited substance DDT (0.09 mg/kg - ppm) in white maize from Ethiopia |
| Herbal mix | 2012.0660 | information for attention | India | Austria | 372 | µg/kg | 50 | prohibited substance DDT (372 µg/kg - ppb) in dried herbs from India |

Of the around 3000 samples from NP and almost 2000 of IND only five samples contain DDT concentrations above the limit of quantification; four from NP and one from IND (Table 3.6). One sample equals the regulatory limit: niger seeds (0.05 mg/kg). The products are from the same product group as the products in RASFF alert, but do not refer to the same incident. Due to the very limited number of samples that contain DDT concentrations above the limit of quantification, it is not possible to perform a trend analysis. The country of origin of the samples in our datasets containing DDT is not reported. Sometimes the specific ratio between different DDT metabolites (O,P'-DDT, O,P'-DDE, etc.) gives information about the origin of the DDT; DDT residues in plants caused by environmental contamination give rise to other metabolite profiles than DDT that has been applied recently on plants. Unfortunately our datasets do not contain enough information on the different metabolites to draw conclusions.

Table 3.6

*Incidence of DDT total in animal feed.*

| Product group | Year with positives | N total | N pos | N ≥ limit | % N pos | % N ≥ limit | Result-range (mg/kg) | Limit |
|---|---|---|---|---|---|---|---|---|
| Mixed/compound feed, complementary | 2004 | 3 | 1 | 0 | 33 | 0 | 0.036 | 0.05 |
| Pig feed, complementary | 2004 | 26 | 1 | 0 | 4 | 0 | 0.038 | 0.05 |
| Niger seed | 2005 | 1 | 1 | 1 | 100 | 100 | 0.05 | 0.05 |
| Grass seed | 2006 | 2 | 1 | 0 | 50 | 0 | 0.002 | 0.05 |
| Fish oil | 2008 | 35 | 1 | 0 | 3 | 0 | 0.1 | 0.5 |

N pos = number of samples with values above LOQ (and % of N total).

N ≥ limit= number of samples with values equal to or above the regulatory limit (and % of N total).

## 3.4 Aldrin (incl. dieldrin) and endrin

The regulatory limit for aldrin and dieldrin is 0.01 mg/kg for most feed material. Only fats and oils and fish feed have higher limits (0.1 and 0.02 mg/kg respectively). For most feeds the regulatory limit for endrin is 0.01 mg/kg. For fats and oils the limit is higher, 0.05 mg/kg (Table 3.7). No RASFF notification or alerts are registered concerning aldrin, dieldrin or endrin.

Table 3.7

*Regulatory limits for aldrin and dieldrin, singly or combined expressed as dieldrin and endrin (sum of endrin and of delta-keto-endrin, expressed as endrin); DIRECTIVE 2002/32/EC.*

| Compound | Product (group) | EU Limit (mg/kg) |
|---|---|---|
| Aldrin_dieldrin | All feed materials and compound feed with the exception of | 0.01 |
| | — fats and oils | 0.1 |
| | — fish feed | 0.02 |
| Endrin | All feed materials and compound feed with the exception of | 0.01 |
| | — fats and oils | 0.05 |

Four samples from NP contain aldin_dieldrin concentrations above the LOQ, two of those values slightly exceed the regulatory limit (Table 3.8). Those two samples will probably not have been reported as non-compliant since for defining non-compliant samples the measurement uncertainty is taken into account. This is 50% for the aldrin-dieldrin measurements. No samples have been reported with an endrin concentration above the LOQ (data not shown).

Table 3.8

*Incidence of aldrin, incl. dieldrin in animal feed.*

| Product group | Year(s) with positives | N total | N pos | N ≥ limit | % N pos | % N ≥ limit | Result-range (mg/kg) | EU Limit |
|---|---|---|---|---|---|---|---|---|
| Mixed/compound feed, complementary | 2004 | 3 | 1 | 1 | 33 | 33 | 0.011 | 0.01 |
| Pig feed, complementary | 2004 | 26 | 1 | 1 | 4 | 4 | 0.012 | 0.01 |
| Fish oil | 2005, 2008 | 35 | 2 | 0 | 9 | 0 | 0.03-0.04 | 0.1 |

N pos = number of samples with values above LOQ (and % of N total).

N ≥ limit = number of samples with values equal to or above the regulatory limit (and % of N total).

Due to the very limited number of samples that contain aldrin-dieldrin concentrations above the limit of quantification, it is not possible to perform a trend analysis.

## 3.5 Heptachlor, HCH-alpha, -beta and -gamma, HCB, toxaphene, chlordane and methoxychlor

Because only a limited set of samples contains values above the LOQ of these compounds they are combined into one paragraph. The regulatory limit for these compounds in most product groups is 0.01 mg/kg. The limit in fats and oils is higher, ranging from 0.05 mg/kg (chlordane) to 2 mg/kg (HCH-gamma). The limit for HCH-beta in compound feeds for dairy cattle is lowest: 0.005 mg/kg. For methoxychlor no regulatory limits is set for feed materials and compound feed.

Table 3.9

*Regulatory limits for Heptachlor, HCH-alpha, -beta and -gamma, HCB, toxaphene, chlordane and methoxychlor (DIRECTIVE 2002/32/EC).*

| Compound | Product (group) | EU Limit (mg/kg) |
|---|---|---|
| Heptachlor (sum of heptachlor and of heptachlorepoxide, expressed as heptachlor) | All feed materials and compound feed with the exception of | 0.01 |
| | — fats and oils | 0.2 |
| HCH-alpha | All feed materials and compound feed with the exception of | 0.02 |
| | — fats and oils | 0.2 |
| HCH-beta | All feed materials with the exception of | 0.01 |
| | — fats and oils | 0.1 |
| | All compounds with the exception of | 0.01 |
| | — compound feeds for dairy cattle | 0.005 |
| HCH-gamma (lindane) | All feed materials with the exception of | 0.2 |
| | — fats and oils | 2.0 |
| Hexachlorobenzene (HCB) | All feed materials with the exception of | 0.01 |
| | — fats and oils | 0.2 |
| Camphechlor (toxaphene) | Fish, other aquatic animals, their products and by-products with the exception of fish oil | 0.02 |
| | — Fish oil | 0.2 |
| | All feed materials and compound feed for fish | 0.05 |
| Chlordane (sum of cis- and transisomers and of oxychlordane, expressed as chlordane) | All feed materials and compound feed with the exception of | 0.02 |
| | — fats and oils | 0.05 |
| Methoxychlor | none found, MRL for food is 0.01 | |

In RASFF alerts three have been registered since 2001regarding heptachlor, HCH-alpha and HCH-beta, one for each component (Table 3.10). None of the positive monitoring samples from this report correspond with these alerts (Table 3.11). No RASFF notification regarding HCH-gamma, HCB, toxaphene, chlordane and methoxychlor were found.

None of the around 5000 samples analysed equal or exceed the regulatory limits for Heptachlor, HCH-alpha, -beta and -gamma, HCB, toxaphene and chlordane (Table 3.11). A few samples exceed the LOQ for one of the compounds. No samples exceed the LOQ for methoxychlor. Due to the very limited number of samples that contain concentrations above the LOQ it is not possible to perform a trend analysis.

## Table 3.10

*RASFF alerts and notifications related to Heptachlor, HCH-alpha and HCH-beta.*

| Compound | Product group | Reference | Notification type | Origin | Notified by | Value(s) | Unit | EU Limit | Subject |
|---|---|---|---|---|---|---|---|---|---|
| Heptachlor | Pig feed | 2004.BJB | information | Germany | Germany | 0.029; 0.012 | mg/kg | 0.01 | heptachlor (0.029; 0.012 mg/kg - ppm) in single feed for fattening pigs |
| HCH-alpha | Premix | 2004.536 | alert | Denmark | Denmark | 0.026; 0.039 | mg/kg | 0.02 | HCH alpha (0.026 and 0.039 mg/kg - ppm) in premix for lactating sows |
| HCH-beta | Fish food | 2011.1582 | information for follow-up | France | Italy | 0.0389 | mg/kg | 0.01 | prohibited substance beta - HCH (0.0389 mg/kg - ppm) in fish feed from France |

## Table 3.11

*Incidence of HCH-alpha, -beta and -gamma, HCB, toxaphene, chlordane and methoxychlor in animal feed.*

| Product group | Compound | Year(s) with positives | N total | N pos | N ≥ limit | % N pos | % N ≥ limit | Result-range (mg/kg) | EU regulatory limit |
|---|---|---|---|---|---|---|---|---|---|
| several products | Methoxychlor | 2001-2009 | | 0 | 0 | 0 | 0 | 0 | ? |
| Fat/oil, vegetable | HCH-gamma (lindane) | 2001 | 54 | 1 | 0 | 2 | 0 | 0.04 | 2.0 |
| Cacao waste | HCH-gamma (lindane) | 2002 | 7 | 1 | 0 | 14 | 0 | 0.01 | 0.2 |
| Groundnuts/peanuts | HCH-beta | 2002 | 3 | 1 | 0 | 33 | 0 | 0.008 | 0.01 |
| Mixed/compound feed, complementary | Chlordane | 2004 | 3 | 1 | 0 | 33 | 0 | 0.005 | 0.02 |
| Mixed/compound feed, complementary | HCB | 2004 | 3 | 1 | 0 | 33 | 0 | 0.009 | 0.01 |
| Pig feed, complementary | Chlordane | 2004 | 26 | 1 | 0 | 4 | 0 | 0.005 | 0.02 |
| Pig feed, complementary | HCB | 2004 | 26 | 1 | 0 | 4 | 0 | 0.008 | 0.01 |
| Fish oil | Chlordane | 2005 | 35 | 1 | 0 | 3 | 0 | 0.03 | 0.05 |
| Grass seed | HCH-gamma (lindane) | 2006 | 2 | 1 | 0 | 50 | 0 | 0.001 | 0.2 |
| Millet | Chlordane | 2006 | 10 | 1 | 0 | 10 | 0 | 0.005 | 0.02 |
| Millet | HCH-alpha | 2006 | 10 | 1 | 0 | 10 | 0 | 0.005 | 0.02 |

N pos = number of samples with values above LOQ (and % of N total).

N ≥ limit= number of samples with values equal to or above the regulatory limit (and % of N total).

# 3.6 Dichlorvos

Dichlorvos is an organophosphate insecticide that acts by inhibiting acetylcholinesterase (AChE), which results in a disturbance of nerve signal transmission and induces rapid respiratory failure in most insects. The same mechanism is responsible for the acute toxicity in mammals, including humans. The only permitted use of dichlorvos is against flower bulb pests during storage (EFSA, 2007). No regulatory limit is set for dichlorvos in feed, the limit for food is 0.01 mg/kg (Table 3.12). When it is a single commodity the limit for food also applies to feed.

Table 3.12

*Regulatory limits for dichlorvos.*

| Product (group) | Limit (mg/kg) |
|---|---|
| Feed | - |
| Food | 0.01 |

Three notifications relating to dichlorvos have been reported in RASFF (Table 3.13). The values are higher than the MRL for food.

Table 3.13

*RASFF alerts and notifications related to dichlorvos.*

| Product group | Reference | Notification type | Origin | Notified by | Value(s) | Unit | Limit | Subject |
|---|---|---|---|---|---|---|---|---|
| Maize | 2008.BFI | border rejection | Argentina | Spain | 0.05 | mg/kg | - | dichlorvos (0.05 mg/kg - ppm) in maize in bulk from Argentina |
| Sorghum | 2011.0165 | alert | Netherlands | Netherlands | 0.623 | mg/kg | - | unauthorised substance dichlorvos (0.623 mg/kg - ppm) in sorghum from the Netherlands, with raw material from Argentina |
| Maize | 2012.BXQ | border rejection | Argentina | Spain | 0.085 | mg/kg | - | unauthorised substance dichlorvos (0.085 mg/kg - ppm) in maize from Argentina |

Three samples (NP) out of 3206 contain dichlorvos concentrations above the LOQ, all three above the MRL for food. The two sorghum samples from 2010 could be related to the RASFF alert in 2011 since the NP samples are also from Argentina and the sample with 0.623 mg/kg has been reported by the Netherlands at the beginning of 2011. However the concentration of dichorvos in the NP samples (0.05 mg/kg) is much lower than in the RASFF sample. Due to the very limited number of samples that contain dichlorvos concentrations above the LOQ it is not possible to perform a trend analysis.

Table 3.14

*Incidence of Dichlorvos in animal feed.*

| Product group | Year with positives | N total | N pos | N ≥ limit | % N pos | % N ≥ limit | Result-range (mg/kg) | Limit |
|---|---|---|---|---|---|---|---|---|
| Millet | 2008 | 10 | 1 | 1 | 10 | 10 | 0.04 | - |
| Sorghum | 2010 | 9 | 2 | 2 | 22 | 22 | 0.05 | - |

N pos = number of samples with values above LOQ (and % of N total).

N ≥ limit= number of samples with values equal to or above the regulatory limit (and % of N total).

## 3.7 Non-dioxin-like PCBs

The regulatory limits for non-dioxin-like PCBs (NDL-PCBs) are instated only recently. Commission regulation EU No 277/2012 (amending Directive 2002/32/EC) entered into force on March 28, 2012. This means that in the period the monitoring data analysed in this report were collected no limit was in use.

Table 3.15

*Regulatory limits (Maximum content in µg/kg (ppb) relative to a feed with a moisture content of 12%) for non-dioxin-like PCBs (sum of PCB 28, PCB 52, PCB 101, PCB 138, PCB 153 and PCB 180 (ICES – 6) [1]).*

| Product (group) | Limit (µg/kg) | Limit (mg/kg) |
|---|---|---|
| Feed materials of plant origin | 10 | 0.01 |
| Feed materials of mineral origin | 10 | 0.01 |
| Feed materials of animal origin: | | |
| — Animal fat, including milk fat and egg fat | 10 | 0.01 |
| — Other land animal products including milk and milk products and eggs and egg products | 10 | 0.01 |
| — Fish oil | 175 | 0.175 |
| — Fish, other aquatic animals and products derived thereof with the exception of fish oil and fish protein, hydrolysed, containing more than 20% fat [2] | 30 | 0.03 |
| — Fish protein, hydrolysed, containing more than 20% fat | 50 | 0.05 |
| The feed additives kaolinitic clay, vermiculite, natrolite- phonolite, synthetic calcium aluminates and clinoptilolite of sedimentary origin belonging to the functional groups of binders and anti-caking agents | 10 | 0.01 |
| Feed additives belonging to the functional group of compounds of trace elements | 10 | 0.01 |
| Pre-mixtures | 10 | 0.01 |
| Compound feed with the exception of: | 10 | 0.01 |
| — compound feed for pet animals and fish | 40 | 0.04 |
| — compound feed for fur animals | — | — |

(1) Upper-bound concentrations; upper-bound concentrations are calculated on the assumption that all values of the different congeners below the limit of quantification are equal to the limit of quantification.

(2) Fresh fish and other aquatic animals directly delivered and used without intermediate processing for the production of feed for fur animals are not subject to the maximum levels, while maximum levels of 75 µg/kg product are applicable to fresh fish and 200 µg/kg product are applicable to fish liver used for the direct feeding of pet animals, zoo and circus animals or used as feed material for the production of pet food. The products or processed animal proteins produced from these animals (fur animals, pet animals, zoo and circus animals) cannot enter the food chain and cannot be fed to farmed animals which are kept, fattened or bred for the production of food.

No RASFF alerts have been reported with these non-dioxin-like PCBs. In the combined NP and IND dataset 316 of 5904 samples have NDL-PCB concentrations that exceed the LOQ (Table 3.16, N pos); 287 out of 3972 from NP (data not shown separately because it is almost identical to Table 3.16) and 29 from IND (Table 3.17). Most of the positive samples are either fat and oil (fish oil, animal fat, vegetable oils), fish meal or minerals. In total 15 samples exceed the regulatory limit (see column N > EU limit), all from NP.

Table 3.16

*Incidence of NDL-PCBs in animal feed (NP and IND).*

| Product group | Year with positives | N total | N pos | N > limit | % N pos | % N > limit | Min Result (mg/kg) | Max Result (mg/kg) | Avg (mg/kg) | Limit (mg/kg) |
|---|---|---|---|---|---|---|---|---|---|---|
| Additive | 2010 | 12 | 1 | 0 | 8 | 0 | 0.0006 | 0.00076 | 0.0006 | 0.01 |
| Alfalfa | 2007, 2009 | 36 | 3 | 0 | 8 | 0 | 0 | 0.0016 | 0.0003 | 0.01 |
| Algae/seaweed | 2009 | 2 | 1 | 0 | 50 | 0 | 0 | 0.0013 | 0.0007 | 0.01 |
| Animal fat | 2004, 2008-2011 | 131 | 8 | 1 | 6 | 1 | 0 | 0.063 | 0.0006 | 0.01 |
| Artificial milk feed | 2007 | 52 | 1 | 0 | 2 | 0 | 0 | 0.0007 | 0 | 0.01 |
| Bakery waste | 2011 | 56 | 1 | 0 | 2 | 0 | 0 | 0.007 | 0.0002 | 0.01 |
| Bovine feed, complementary | 2007 | 249 | 2 | 0 | 1 | 0 | 0 | 0.00072 | 0 | 0.01 |
| Calcium carbonate | 2008 | 9 | 1 | 0 | 11 | 0 | 0 | 0.0099 | 0.001 | 0.01 |
| Choline chloride | 2007 | 65 | 5 | 0 | 8 | 0 | 0 | 0.0007 | 0.0006 | 0.01 |
| Citrus pulp | 2005 | 84 | 1 | 0 | 1 | 0 | 0 | 0.002 | 0 | 0.01 |
| Clay minerals | 2007-2010 | 125 | 14 | 0 | 11 | 0 | 0 | 0.0089 | 0.007 | 0.01 |
| Coconut fat | 2008 | 87 | 1 | 0 | 1 | 0 | 0 | 0.0014 | 0.0001 | 0.01 |
| Duckweed | 2008 | 1 | 1 | 0 | 100 | 0 | 0.0034 | 0.0034 | 0.0034 | 0.01 |
| Fat of bovine animal | 20,092,010 | 65 | 5 | 0 | 8 | 0 | 0 | 0.0035 | 0.0002 | 0.01 |
| Fat/oil, | 2011 | 66 | 9 | 0 | 14 | 0 | 0 | 0.0065 | 0.0003 | 0.01 |

| Product group | Year with positives | N total | N pos | N > limit | % N pos | % N > limit | Min Result (mg/kg) | Max Result (mg/kg) | Avg (mg/kg) | Limit (mg/kg) |
|---|---|---|---|---|---|---|---|---|---|---|
| vegetable | | | | | | | | | | |
| Fatty acids | 2001 | 133 | 13 | 4 | 10 | 3 | 0 | 0.022 | 0.0008 | 0.01 |
| Fish food | 2008 | 2 | 1 | 0 | 50 | 0 | 0 | 0.00093 | 0.0005 | 0.04 |
| Fish meal | 2006-2011 | 130 | 93 | 1 | 72 | 1 | 0 | 0.053 | 0.003 | 0.03 |
| Fish oil | 2005, 2008-2011 | 82 | 55 | 0 | 67 | 0 | 0 | 0.09 | 0.03 | 0.175 |
| Fish protein | 2008-2011 | 12 | 11 | 0 | 92 | 0 | 0 | 0.019 | 0.0068 | 0.05 |
| Grass | 2007-2011 | 116 | 14 | 0 | 12 | 0 | 0 | 0.0011 | 0.0003 | 0.01 |
| Horse feed | 2007 | 20 | 1 | 0 | 5 | 0 | 0 | 0.0007 | 0.0002 | 0.01 |
| Iron chelate | 2007 | 1 | 1 | 0 | 100 | 0 | 0.0007 | 0.0007 | 0.0007 | 0.01 |
| Iron oxide | 2007 | 2 | 1 | 0 | 50 | 0 | 0.0006 | 0.0007 | 0.0007 | 0.01 |
| Linseed | 2007 | 42 | 1 | 0 | 2 | 0 | 0 | 0.0007 | 0 | 0.01 |
| Linseed oil | 2001 | 5 | 1 | 0 | 20 | 0 | 0 | 0.00079 | 0.0002 | 0.01 |
| Magnesium oxide | 2007, 2009, 2010 | 9 | 3 | 0 | 33 | 0 | 0 | 0.01 | 0.0037 | 0.01 |
| Maize product | 2007 | 559 | 3 | 0 | 1 | 0 | 0 | 0.001 | 0 | 0.01 |
| Milk powder | 2007, 2011 | 10 | 6 | 0 | 60 | 0 | 0 | 0.0022 | 0.0009 | 0.01 |
| Mix fat | 2011 | 27 | 3 | 3 | 11 | 11 | 0 | 0.021 | 0.0021 | 0.01 |
| Mixed/compound feed, complementary | 2004, 2008 | 4 | 2 | 2 | 50 | 50 | 0 | 0.048 | 0.0173 | 0.01 |
| Monoammonium phosphate | 2007 | 1 | 1 | 0 | 100 | 0 | 0.0007 | 0.0007 | 0.0007 | 0.01 |
| Monocalcium phosphate | 2006, 2007, 2008 | 5 | 3 | 0 | 60 | 0 | 0 | 0.01 | 0.0056 | 0.01 |
| Other commodities | 2011 | 8 | 2 | 1 | 25 | 13 | 0 | 0.019 | 0.0028 | 0.01 |
| Palmkernel oil | 2006 - 2009 | 177 | 7 | 0 | 4 | 0 | 0 | 0.005 | 0.0002 | 0.01 |
| Pig feed | 2007, 2008 | 276 | 8 | 0 | 3 | 0 | 0 | 0.00071 | 0.0001 | 0.01 |
| Pig feed, complementary | 2004 | 30 | 1 | 1 | 3 | 3 | 0 | 0.041 | 0.0014 | 0.01 |
| Pig feed, pulp | 2007 | 55 | 1 | 0 | 2 | 0 | 0 | 0.0007 | 0 | 0.01 |
| Potassium iodide | 2007 | 1 | 1 | 0 | 100 | 0 | 0.0007 | 0.0007 | 0.0007 | 0.01 |
| Potato peels | 2009, 2010 | 16 | 3 | 0 | 19 | 0 | 0 | 0.00079 | 0.0002 | 0.01 |
| Poultry feed | 2007, 2008 | 152 | 3 | 1 | 2 | 1 | 0 | 0.017 | 0.0002 | 0.01 |
| Premix | 2007 | 16 | 2 | 0 | 13 | 0 | 0.0006 | 0.0007 | 0.0006 | 0.01 |
| Rye | 2007 | 43 | 1 | 0 | 2 | 0 | 0 | 0.0007 | 0 | 0.01 |
| Shrimps | 2009, 2010 | 4 | 3 | 0 | 75 | 0 | 0.0006 | 0.0054 | 0.0029 | 0.03 |
| Sodium chloride | 2011 | 1 | 1 | 0 | 100 | 0 | 0.00086 | 0.00086 | 0.0009 | 0.01 |
| Soybean oil | 2002, 2006-2009 | 220 | 10 | 1 | 5 | 0 | 0 | 0.08 | 0.0006 | 0.01 |
| Sunflower oil | 2011 | 33 | 1 | 0 | 3 | 0 | 0 | 0.0043 | 0.0001 | 0.01 |
| Sunflower seed | 2010 | 60 | 1 | 0 | 2 | 0 | 0 | 0.00067 | 0 | 0.01 |
| Tapioca | 2007 | 29 | 1 | 0 | 3 | 0 | 0 | 0.0007 | 0 | 0.01 |
| Vitamin mix | 2009 | 7 | 1 | 0 | 14 | 0 | 0 | 0.0019 | 0.0007 | 0.01 |
| Wheat | 2007 | 236 | 1 | 0 | 0 | 0 | 0 | 0.0007 | 0 | 0.01 |
| Wheat product | 2007 | 126 | 1 | 0 | 1 | 0 | 0 | 0.0007 | 0 | 0.01 |
| Total | | | 316 | 15 | | | | | | |

In the IND dataset 29 of 1951 samples have been reported with values above the LOQ. None of those samples exceed the regulatory limit. However, 16 samples have been reported with a value of < 0.035 mg/kg. This is the LOQ for several groups, mainly fats and fatty acids, even though the regulatory limit is below that value (0.01 mg/kg). The explanation is that the limits used by IND are the limits set by GMP+. Those limits have not been exceeded. The GMP+ limit was 0.05 mg/kg for animal fat and 0.25 for fat and oil of vegetable origin (www.gmpplus.org or http://www.pdv.nl/lmbinaries/gmp14_-uk-productnormen_gmp-regeling_diervoedersector.pdf) instead of 0.01 mg/kg as enforced by Commission Regulation EU No 277/2012. The EU limit did not exist before 28-03-2012.

Table 3.17

*Incidence of NDL-PCBs in animal feed (IND only).*

| Product group | Year with positives | N total | N pos | N > limit | % N pos | % N > limit | Max Result (mg/kg) | Avg (mg/kg) | Limit (mg/kg) |
|---|---|---|---|---|---|---|---|---|---|
| Bakery waste | 2011 | 22 | 1 | 0 | 5 | 0 | 0.007 | 0.0003 | 0.01 |
| Calcium carbonate | 2008 | 8 | 1 | 0 | 13 | 0 | 0.0099 | 0.001 | 0.01 |
| Fish meal | 2006 | 35 | 1 | 0 | 3 | 0 | 0.002 | 0.0002 | 0.03 |
| Fish oil | 2008, 2009 | 19 | 3 | 0 | 16 | 0 | 0.075 | 0.007 | 0.175 |
| Magnesium oxide | 2007, 2009, 2010 | 4 | 3 | 0 | 75 | 0 | 0.01 | 0.007 | 0.01 |
| Maize product | 2007 | 390 | 1 | 0 | 0 | 0 | 0.001 | 0 | 0.01 |
| Monocalcium phosphate | 2006-2008 | 5 | 3 | 0 | 60 | 0 | 0.09 | 0.0056 | 0.01 |
| Palmkernel oil | 2006-2009 | 68 | 7 | 0 | 10 | 0 | 0.005 | 0.0005 | 0.01 |
| Soybean oil | 2006-2009 | 61 | 9 | 0 | 15 | 0 | 0.005 | 0.0007 | 0.01 |
| **Total** | | | **29** | **0** | | | | | |

N pos = number of samples with values above LOQ (and % of N total).

N ≥ limit= number of samples with values equal to or above the regulatory limit (and % of N total).

Most of the samples in both datasets have no detectable NDL-PCB levels (5588 0-values in Figure 3.6, reported in the datasets as < LOQ (upper bound), set at 0 in calculations). The highest average contents were found in fish oil (0.03 mg/kg). Fish protein (0.0068 mg/kg), fish meal (0.003 mg/kg) and shrimps (0.0029 mg/kg) are also amongst the highest average levels. This is consistent with the findings from EFSA (2012) where mean values for 26 European countries (period from 1995 - 2010) were 0.054 mg/kg for fish oil and 0.005 mg/kg for 'Fish, other aquatic animals, their product'. EFSA (2012) also reported elevated levels (0.008 mg/kg) in the category 'Feed for fur animals, pets and fish'; In the data set of this report only 2 samples of fish feed were included and the contents were low.

Often the in- or decrease over a time period is estimated using the average value per year. However, this does not take into account the variation of the number of samples taken per year. In Figure 3.6B a trend line has been calculated using all individual measurements. This figure shows the spread of the values over the years but because there are many samples with values below the LOQ (shown as 0) the trend line (the red line) is too close to the X-axis to see any difference from zero. In Figure 3.6C the 'traditional' trend line has been calculated using the average values. The average values are below the LOQ of the methods and consequently it is not meaningful to perform a trend analysis. When looking at the main product groups separately the average NDL-PCB content increases significantly in fish oil (Figure 3.6D). Most samples are taken between 2008-2011. The increase in this period is still significant (Figure 3.6E). This trend should be interpreted with caution because the contamination level largely depends on the country / region of origin, e.g. fish from the Baltic region is more contaminated than from other regions (EFSA, 2012), and this information is not known. Moreover, the number of samples per year is quite small.

Most values above LOQ are found in fish meal (Figure 3.6F). Between 2007 and 2010 (samples analysed with the GC-HRMS method, Figure 3.6G) the average NDL-PCB concentration in fish meal decreases (in 2011 only one (IND) fish meal sample has been analysed, with no detectable NDL-PCB). Again, this trend should be interpreted with caution because no information on the country of origin was available. For comparison, EFSA (2012) did not observe a significant trend for the food category 'Muscle meat from fishes other than eels' in the period from 1998 - 2010.

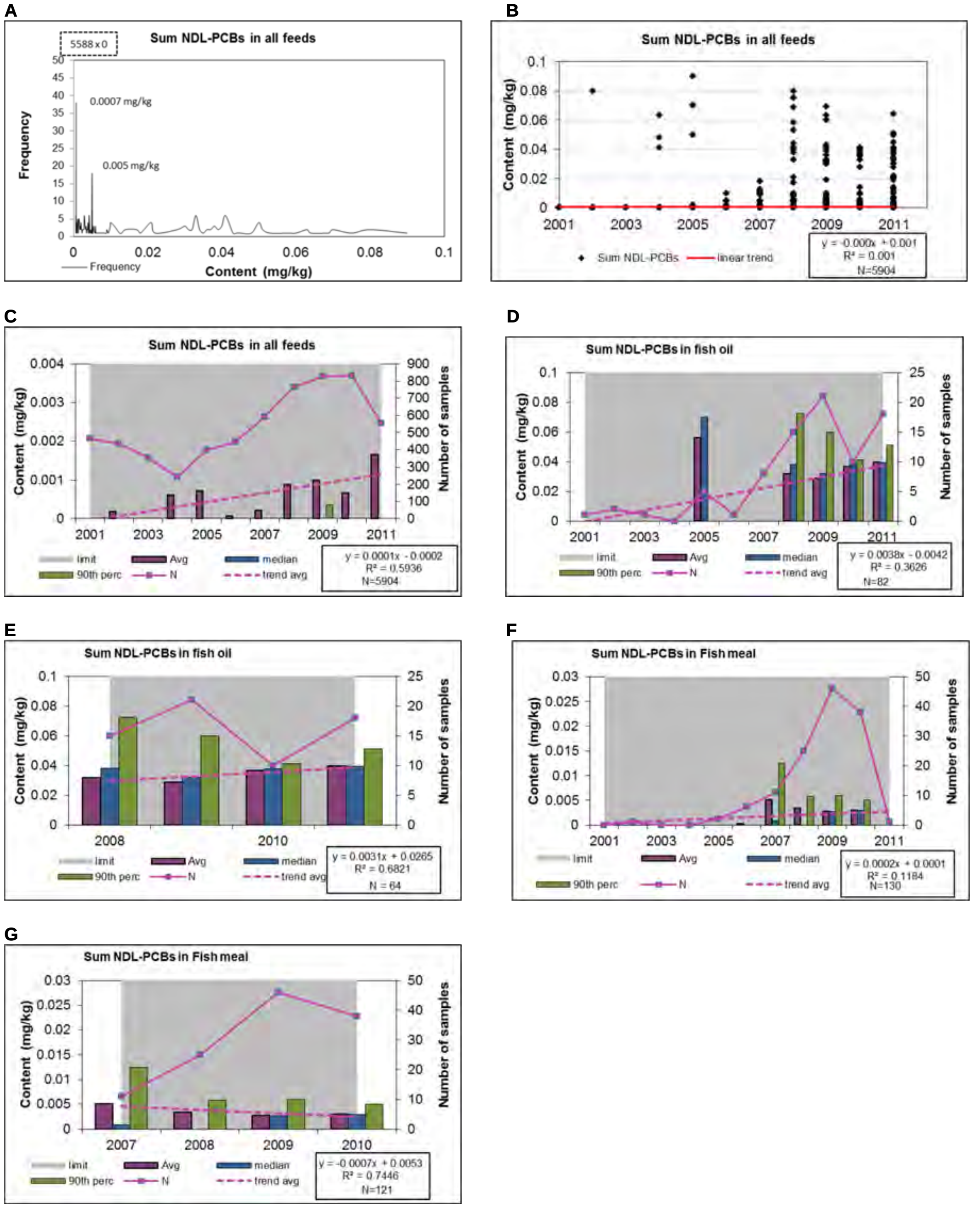
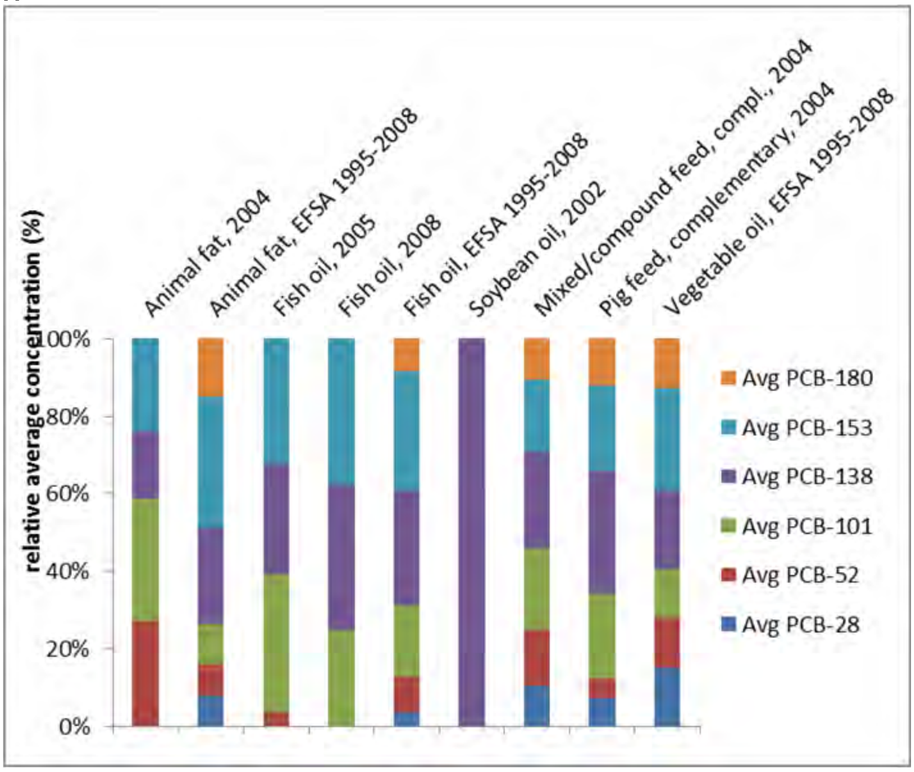



***Figure 3.6*** *NDL-PCBs (upper-bound) in all feeds from all sources ; A = frequency; B = scatter plot; C = trend all feeds; limit is between 0.01 and 0.175 mg/kg; D= fish oil (2001-2001); E=fish oil (2008-2011) limit = 0.175 mg/kg; F= fish meal (2001-2011); G = fish meal (2007-2010) limit = 0.03 mg/kg.*

In a scientific report of the EFSA (EFSA, 2010) on results of the monitoring of non-dioxin-like PCBs in food and feed, the largest contribution to the overall mean contribution of the individual indicator NDL-PCBs (upper-bound) to the sum of the six comes from PCB-153. PCB-138 and PCB-101 contribute as well but to a lower extent . According to EFSA this is in line with findings reported in other studies which demonstrated that PCB-153 has an average contribution of roughly one third of the sum of the six indicator PCBs. In the current report only data from NP are used to compare the different congeners since in the dataset from IND no individual congeners are reported. PCB-153 has a large contribution (see Figure 3.7) as well as PCB-138 and PCB-101. High contamination was reported by

EFSA in feed containing fish derived products (especially fish oil) and comparatively very low levels in feed of plant or mineral origin. This coincides with the data from NP and IND shown in the current report. However, some of the results reported by EFSA might have originated from targeted testing during specific dioxin contamination incidences.

A




B

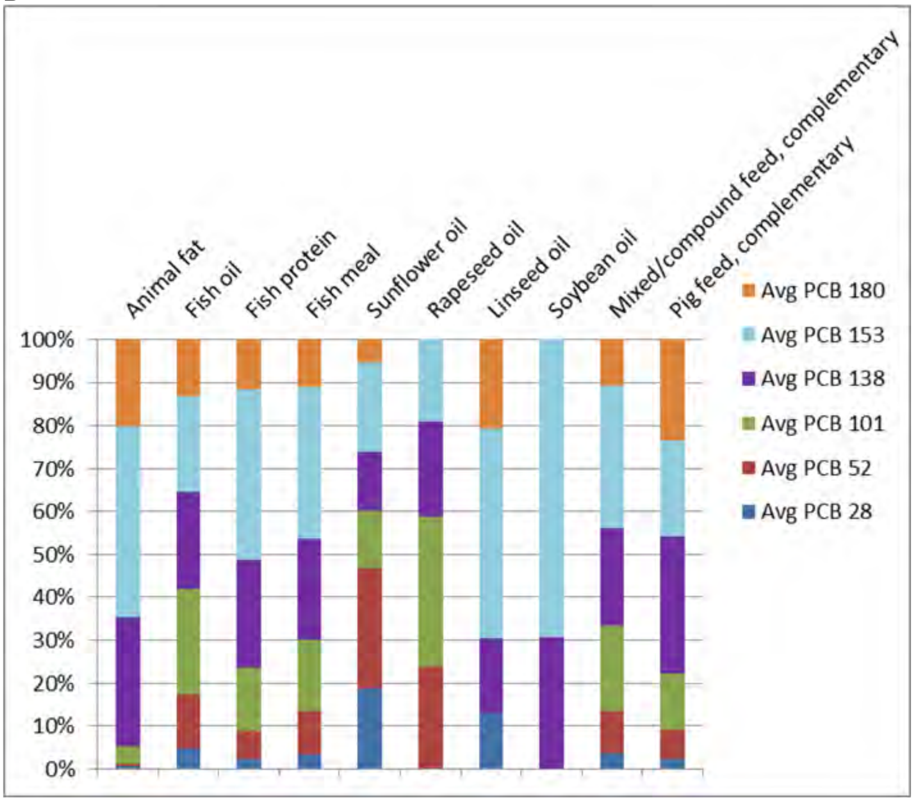



***Figure 3.7*** *Relative contribution of NDL-PCB congeners to the average NDL-PCB concentration of one or more samples from one product in a specific year; monitoring data from this report compared to data from EFSA (EFSA, 2010) A= screening method+ EFSA; B= GC-HRMS method.*

# 4 Discussion and conclusions

Only a limited number of samples contain levels of organochlorine pesticides above the LOQ; 174 out of 3972 samples from NP and 291 out of 1932 samples from IND. Even fewer (18 in total) contain levels above the regulatory limit for the particular feed/contaminant combination. Most positive samples consist of feed materials with high fat or oil content. This is to be expected since organochlorine compounds are more soluble in fat than in water.

Trend analysis often is very difficult because of the limited number of positive samples. Endosulfan is the exception, especially in soybean oil. Endosulfan is one of the few organochlorine pesticides that is still in use although the UN Stockholm Convention has agreed in 2011 to add endosulfan to the United Nations' list of persistent organic pollutants to be eliminated worldwide (UN, 2011).

Not only in the datasets described in this report endosulfan levels above the regulatory limits have been found, also in RASFF an alert has been reported. This concerned two occurrences of endosulfan concentrations above the limit in fats and oils originating from the Netherlands. Those samples could not be found in the NP dataset but could be traced back to incidents in vegetable oil reported by the former AID (currently NVWA) in 2010 (Egmond, *et al.,* 2010).

In the NP and IND dataset it is difficult to decide whether a concentration of endosulfan in vegetable oil samples exceeds the regulatory limit since it is often unclear whether it concerns crude oil (regulatory limit = 1.0 mg/kg) or processed oil (regulatory limit = 0.5 mg/kg). Using the last limit six samples of soya bean oil exceed the limit. There is no significant in- or decrease in the average endosulfan concentration in soya bean oil between 2001 and 2011.

Only five samples of 5904 in total contain DDT concentrations above the limit of quantification, one of them (niger seeds) equals the regulatory limit. Sometimes the specific ratio between different DDT metabolites (O,P'-DDT, O,P'-DDE, etc.) gives information about the origin of the DDT; old DDT residues have other metabolite profiles than recently applied DDT. The current datasets do not contain enough information about the different metabolites to draw conclusions.

Four samples of 3972 from NP contain aldin_dieldrin concentrations above the LOQ, two of those values slightly exceed the regulatory limit (complementary pig feed and unspecified complementary compound feed). The other two samples (fish oil) do not exceed the regulatory limit. No samples have been reported with an endrin concentration above the LOQ.

None of the 5904 samples equals or exceeds the regulatory limits for heptachlor, HCH-alpha, -beta and -gamma, HCB, toxaphene and chlordane. A few samples exceed the LOQ for one of the compounds. No samples exceed the LOQ for methoxychlor. In RASFF alerts have been registered regarding heptachlor (pig feed), HCH-alpha (pig feed) and HCH-beta (fish food). None of the positive (> LOQ) monitoring samples from NP or IND report correspond with these alerts.

Three samples (of 5904) from NP and IND contain dichlorvos concentration above the LOQ, all three above the MRL for food. There is no regulatory limit for feed. Two sorghum samples from 2010 might be related to an RASFF alert in 2011. However the concentrations of dichorvos in the NP samples are much lower than the RASFF sample.

In the combined NP (incl. additional GC-HRMS dataset) and IND dataset 316 of 5904 samples have NDL-PCB concentrations that exceed the LOQ. Most of the positive samples are either fat and oil (fish oil, animal fat, vegetable oils ), fish meal or minerals. In total 15 samples exceed the regulatory limit that has been set in 2012, all from NP.

The IND method often had an LOQ of 0.035 mg/kg for the sum of NDL-PCBs. This is a value above most of the current regulatory limits (between 0.01 and 0.04 mg/kg for most feed materials and 0.175 mg/kg for fish oil). However, those limits have only be instated in 2012 and were not in place during the period studied here (2001-2011). During that period the industry used the limits recommended by GMP$^{+}$. None of the IND samples exceed those limits.

The highest average contents were found in fish oil (0.03 mg/kg). Fish protein (0.0068 mg/kg), fish meal (0.003 mg/kg) and shrimps (0.0029 mg/kg) are also amongst the highest average levels. This is consistent with the findings from EFSA (2012) where mean values for 26 European countries (period from 1995 - 2010) were 0.054 mg/kg for fish oil and 0.005 mg/kg for 'Fish, other aquatic animals, their product'.

EFSA also reports that the largest contribution to the overall mean contribution of the individual indicator NDL-PCBs (upper-bound) to the sum of the six relates to PCB-153 and to a lesser extent to PCB-138 and PCB-101. In the monitoring results from the current report, with data from NP, PCB-153 has a large contribution as well, but PCB-138 and PCB-101 have a large contribution as well. In future trend analysis reports NDL-PCBs will be studied together with dioxins and dioxin-like PCBs.

# 5 Recommendations

It was not possible to relate samples from the NP and IND datasets to alerts from RASFF because for many samples the real country of origin is unknown. This has been noted in previous trend analysis reports and this time it is mainly relevant for endosulfan in soybean oil. It is recommended to improve the registration of country of origin.

Since limits for crude vegetable oil and products derived from oil seeds differ for some compounds, viz. for endosulfan, it is important to register the product names in more detail. Now it was often not possible to distinguish between the two types of oil.

Since organochlorine compounds readily dissolve in fat, feed materials with high fat or oil content (like soya oil and fatty acids) are especially at risk but, as can been seen with for example endosulfan, non-fat products like apple and potatoes can contain organochlorine compounds as well.

It is important to continue monitoring products where the regulatory limits have been exceeded or where levels close to the regulatory limit are found frequently. This is especially the case for endosulfan. Monitoring for NDL-PCBs should continue as well because maximum levels have only been introduced recently and the LOQs of the methods in many cases were too high compared to those regulatory limits.

For NDL-PCBs it is also important to explore other sources than feed that may lead to contamination of food of animal origin, e.g. eggs, through paints in stables or construction debris for free-ranging chicken (Hoogenboom and Traag, 2013).

Efforts should be made to harmonize (LOQs of) the methods applied by industry and the National Plan to enable combing results in one dataset.

# References

Adamse P, Driessen JJM, Jong J de, Polanen A van, Egmond HJ van, Jongbloed AW. 2009. Trendanalyse zware metalen in diervoeder (grondstoffen) RIKILT report 2009.019

Adamse P, Egmond HJ van, Driessen J, Rijk T de, Jong J de, Nijs M de. 2011a. Trend analysis of mycotoxins in animal feed. RIKILT report 2011.017

Adamse P, Egmond HJ van, Polanen A van, Bikker P and Jong J de. 2011b. Trend analysis of copper and zinc in animal feed. RIKILT report 2011.012

Adamse P, Jong J de, Jongbloed AW, Raamsdonk LWD van, Egmond HJ van. 2007. Trendanalyse van gehalten aan aflatoxine B1 en dioxinen/dioxine-achtige PCB's in diervoeders P. RIKILT report 2007.001

Adamse P, Raamsdonk LWD van, Egmond HJ van, Jong J de. 2009. Trendanalyse dierlijke eiwitten in diervoeder (grondstoffen) RIKILT report 2009.016

Directive 2002/32/EC of the European parliament and of the council of 7 May 2002 on undesirable substances in animal feed (OJ L 140, 30.5.2002, p. 10) Available from [http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=CONSLEG:2002L0032:20061020:EN:PDF]

EFSA. 2007. EFSA Scientific Report (2006) 77, 1-43, Conclusion on the peer review of dichlorvos http://www.efsa.europa.eu/en/efsajournal/doc/77r.pdf

EFSA. 2010. Monitoring of Non Dioxin-Like PCBs in Food and Feed. EFSA Journal 8(7):1701

Gilbert RO. 1987. Statistical methods for environmental pollution monitoring. Van Nostrand Rienhold Co., New York.

Egmond HJ van, Kuijk K van, Jong J de, Hooglugt JH, Arts FWJT, Hagen-Lenselink HJ. 2010. Resultaten van het RIKILT diervoeder onderzoek voor de AID (2005 t/m 2009). RIKILT report 2010.521

GMP+ Product standards. Available from [ https://www.gmpplus.org/pagina/9/standards.aspx]

Hoogenboom, LAP and Traag, W. 2013. Grond gerelateerde voedselincidenten met dioxines en PCB's. RIKILT report 2013.xxx, in press (in Dutch)

Kan CA, Meijer GAL. 2007. The risk of contamination of food with toxic substances present in animal feed. Animal Feed Science and Technology 133: 84–108

Klaveren JD van, Donkersgoed G van, Dooren MMH van. 1997. Results residue monitoring in the Netherlands – Quality Programme for Agricultural Products, KAP-report, number 5 , RIKILT-Institute of Food Safety, Wageningen.

Lee MK van der, Weg G van der, Traag Mol HGJ. 2008. Qualitative screening and quantitative determination of pesticides and contaminants in animal feed using comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometry. Journal of Chromatography A, 1186: 325–339

Regulation (EU) No 277/2012 amending Annexes I and II to Directive 2002/32/EC of the European Parliament and of the Council as regards maximum levels and action thresholds for dioxins and polychlorinated biphenyls. (OJ L 91, 29.3.2012) Available from [http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2012:091:0001:0007:EN:PDF]

Regulation (EC) No 767/2009 of the European Parliament and of the Council of 13 July 2009 on the placing on the market and use of feed, amending European Parliament and Council Regulation (EC) No 1831/2003 and repealing Council Directive 79/373/EEC, Commission Directive 80/511/EEC, Council Directives 82/471/EEC, 83/228/EEC, 93/74/EEC, 93/113/EC and 96/25/EC and Commission Decision 2004/217/EC. (OJ L 229, 1.9.2009)

Regulation (EC) No 1831/2003 of the European Parliament and of the Council of 22 September 2003 on additives for use in animal nutrition. (OJ L 268, 18.10.2003)

Salmi T, Määttä A, Anttila P, Ruoho-Airola T, Amnell T. 2002. Detecting trends of annual values of atmospheric pollutants by the Mann-Kendall test and Sen's slope estimates - the Excel template application MAKESENS. In: Publications on air quality, Vol. 31, Finnish Meteorological Institute, Helsinki.

Traag WA, Immerzeel J, Onstenk C, Kraats C, Lee MK, Van derWeg G, Mol H, Hoogenboom LAP. Automation of chemical analysis of PCDD/Fs, Dioxin-like PCBs , indicator PCBs and Polybrominated

Diphenyl Ethers in food and feed; Organohalogen Compounds, Volume 70 (2008), 54-57 UN 2011. United Nations targets widely-used pesticide endosulfan for phase out. Press release Stockholm Convention, Available from
[http://chm.pops.int/Convention/Media/PressReleases/COP5Geneva,3May2011Endosulfanphaseout/tabid/2216/Default.aspx]

# Annex 1

## Table 6.1

*Undesirable substances in Products intended for animal feed (DIRECTIVE 2002/32/EC).*

| Undesirable substances | Products intended for animal feed | Maximum content in mg/kg (ppm) [(1)] |
|---|---|---|
| Aldrin [(2)] | All feed materials and compound feed with the exception of | 0,01[(3)] |
| Dieldrin [(2)] | — fats and oils | 0,1[(3)] |
| | — fish feed 0,02 [(3)] | |
| Camphechlor (toxaphene) — sum of indicator congeners CHB 26, 50 and 62 [(4)] | Fish, other aquatic animals, their products and by-products with the exception of fish oil | 0,02 |
| | — Fish oil [(5)] | 0,2 |
| | All feed materials and compound feed for fish [(5)] | 0,05 |
| Chlordane (sum of cis- and transisomers and of oxychlordane, expressed as chlordane) | All feed materials and compound feed with the exception of | 0,02 |
| | — fats and oils | 0,05 |
| DDT (sum of DDT-, TDE- and DDE isomers, expressed as DDT) | All feed materials and compound feed with the exception of | 0,05 |
| | — fats and oils | 0,5 |
| Endosulfan (sum of alpha- and beta isomers and of endosulfansulphate expressed as endosulfan) | All feed materials and compound feed with the exception of | 0,1 |
| | — maize and maize products derived from the processing thereof | 0,2 |
| | — oilseeds and products derived from the processing thereof with the exception of crude vegetable oil | 0,5 |
| | — crude vegetable oil | 1,0 |
| | — complete feed for fish | 0,005 |
| Endrin (sum of endrin and of delta-keto-endrin, expressed as endrin) | All feed materials and compound feed with the exception of | 0,01 |
| | — fats and oils | 0,05 |
| Heptachlor (sum of heptachlor and of heptachlorepoxide, expressed as heptachlor) | All feed materials and compound feed with the exception of | 0,01 |
| | — fats and oils | 0,2 |
| Hexachlorobenzene (HCB) | All feed materials and compound feed with the exception of | 0,01 |
| | — fats and oils | 0,2 |
| Hexachlorocyclohexane (HCH) | | |
| alpha-isomers | All feed materials and compound feed with the exception of | 0,02 |
| | — fats and oils | 0,2 |
| beta-isomers | All feed materials with the exception of | 0,01 |
| | — fats and oils | 0,1 |
| | All compound feed with the exception of | 0,01 |
| | — compound feed for dairy cattle | 0,005 |
| gamma-isomers | All feed materials and compound feed with the exception of | 0,2 |
| | — fats and oils | 2,0 |

[(1)] relative to a feeding stuff with a moisture content of 12%.

[(2)] Singly or combined expressed as dieldrin.

[(3)] Maximum level for aldrin and dieldrin, singly or combined, expressed as dieldrin.

[(4)] Numbering system according to Parlar, prefixed by either 'CHB' or 'Parlar':

— CHB 26: 2-endo,3-exo,5-endo, 6-exo, 8,8,10,10-octochlorobornane,

— CHB 50: 2-endo,3-exo,5-endo, 6-exo, 8,8,9,10,10-nonachlorobornane,

— CHB 62: 2,2,5,5,8,9,9,10,10-nonachlorobornane.

[(5)] The levels shall be reviewed by 31 December 2007 with the aim of reducing the maximum levels.

## Table 6.2

*Maximum levels and action thresholds for dioxins and polychlorinated biphenyls Regulation (EU) No 277/2012 - Non-dioxin-like PCBs (sum of PCB 28, PCB 52, PCB 101, PCB 138, PCB 153 and PCB 180 (ICES – 6)* [(1)]*)*

| Product (group) | Limit (µg/kg) | Limit (mg/kg) |
|---|---|---|
| Feed materials of plant origin | 10 | 0.01 |
| Feed materials of mineral origin | 10 | 0.01 |
| Feed materials of animal origin: | | |
| — Animal fat, including milk fat and egg fat | 10 | 0.01 |
| — Other land animal products including milk and milk products and eggs and egg products | 10 | 0.01 |
| — Fish oil | 175 | 0.175 |
| — Fish, other aquatic animals and products derived thereof with the exception of fish oil and fish protein, hydrolysed, containing more than 20% fat [(2)] | 30 | 0.03 |
| — Fish protein, hydrolysed, containing more than 20% fat | 50 | 0.05 |
| The feed additives kaolinitic clay, vermiculite, natrolite- phonolite, synthetic calcium aluminates and clinoptilolite of sedimentary origin belonging to the functional groups of binders and anti-caking agents | 10 | 0.01 |
| Feed additives belonging to the functional group of compounds of trace elements | 10 | 0.01 |
| Pre-mixtures | 10 | 0.01 |
| Compound feed with the exception of: | 10 | 0.01 |
| — compound feed for pet animals and fish | 40 | 0.04 |
| — compound feed for fur animals | | |

(1) Upper-bound concentrations; upper-bound concentrations are calculated on the assumption that all values of the different congeners below the limit of quantification are equal to the limit of quantification.

(2) Table of TEF (= toxic equivalency factors) for dioxins, furans and dioxin-like PCBs: WHO-TEFs for human risk assessment based on the conclusions of the World Health Organisation (WHO) – International Programme on Chemical Safety (IPCS) expert meeting which was held in Geneva in June 2005 (Martin van den Berg *et al*., The 2005 World Health Organisation Re-evaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin- like Compounds. Toxicological Sciences 93(2), 223–241 (2006)).

## Table 6.3

*Number of monitoring samples per year per product - NP (additional GC HRMS samples for NDL-PCBs not included).*

| Product(group) NP | 2001 | 2002 | 2003 | 2004 | 2005 | 2006 | 2007 | 2008 | 2009 | 2010 | 2011 | Grand Total |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Alfalfa | | | | | 1 | | | 3 | 3 | | | 7 |
| Algae/seaweed | | | | | 1 | | | | | | | 1 |
| Amino acids | | | | | | | | | | 1 | 4 | 5 |
| Animal fat | 8 | 16 | 7 | 9 | 15 | 5 | 5 | 2 | | | | 67 |
| Animal feed, general | | | | | | | | 3 | | | | 3 |
| Animal meal | | | | | 2 | | | | | | | 2 |
| Animal products, other | | | | | | 1 | | | | | | 1 |
| Apple | | | | | 2 | 5 | | 1 | | | | 8 |
| Artificial milk feed | 4 | | | | 1 | | 41 | 4 | | | | 50 |
| Bakery waste | | | | | 4 | 16 | 10 | 1 | | | | 31 |
| Barley | 7 | 7 | 11 | 8 | 1 | 1 | | 6 | 2 | 3 | 15 | 61 |
| Beans | | | | | 2 | 1 | | | 1 | | 3 | 7 |
| Bird food | | | | 2 | 1 | | | | | | | 3 |
| Biscuit meal | | | | | 2 | | | | | | | 2 |
| Bovine feed | 19 | 12 | 3 | 2 | 6 | 1 | 6 | | | | | 49 |
| Bovine feed, complementary | 30 | 70 | 43 | 16 | 23 | 9 | 24 | | | | | 215 |
| Breadmeal | 2 | | | | 6 | 2 | | | | | | 10 |
| Brewers grains | | | | | 1 | | | | | 2 | | 3 |
| Cacao waste | | 1 | 2 | | | 1 | 1 | 1 | | | | 6 |
| Calciumcarbonate | | | | | 1 | | | | | | | 1 |
| Carrot product | | | | | | 3 | | | | 1 | | 4 |
| Choline chloride | | | | | | | | 2 | | | | 2 |
| Citrus pulp | 15 | 11 | 9 | 4 | 4 | | | | 2 | 1 | 1 | 47 |
| Coconut | 2 | 1 | | 1 | | | | | | | | 4 |

| Product(group) NP | 2001 | 2002 | 2003 | 2004 | 2005 | 2006 | 2007 | 2008 | 2009 | 2010 | 2011 | Grand Total |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Coconut expeller/extracted | 3 | 1 | | | | | | | | | | 4 |
| Coconut fat | 9 | 6 | 4 | 1 | 2 | 3 | 3 | 3 | 14 | 6 | 12 | 63 |
| Cole-/rapeseed | 1 | 1 | | | 2 | | | | 4 | 3 | 8 | 19 |
| Cole-/rapeseed expeller/extracted | 1 | 4 | 3 | 1 | 1 | | 1 | 6 | 6 | 2 | 2 | 27 |
| Cottonseed | | 1 | 2 | | | | | | | | | 3 |
| Distillers maize | | | | | 1 | | | | | | | 1 |
| Fat of bovine animal | 14 | 3 | 2 | | 16 | 10 | 10 | 5 | | | | 60 |
| Fat/oil, vegetable | 15 | 12 | 1 | 1 | 5 | 1 | 3 | 1 | | 3 | | 42 |
| Fatty acids | 10 | 2 | | 3 | 1 | 6 | 3 | 3 | 3 | 3 | 3 | 37 |
| Fish food | | | 1 | | | | | | | | | 1 |
| Fish meal | | 1 | | | 1 | | | | | | | 2 |
| Fish oil | 1 | 2 | 1 | | 5 | 1 | 3 | 3 | | | | 16 |
| Fish protein | | | | | | | 1 | | | | | 1 |
| Frying fat | 1 | 1 | | | | | | | | | | 2 |
| Goat feed | 1 | | | | | 1 | | | | | | 2 |
| Goat feed, complementary | | 1 | 1 | 2 | 1 | | | | | | | 5 |
| Grass | | 1 | | 1 | 4 | | | 22 | 19 | 19 | | 66 |
| Grass seed | | | | | 1 | | | | | | | 1 |
| Green maize | | | | | | | | 25 | 30 | 25 | | 80 |
| Groundnut expeller/extracted | 2 | | | | | | | | | | | 2 |
| Groundnut oil | | | | 1 | | | 1 | | 1 | 2 | 1 | 6 |
| Groundnuts, peeled | 1 | | | | | | | | | | | 1 |
| Groundnuts/peanuts | | 1 | | | 2 | | | | | | | 3 |
| Herbal mix | | | | | 1 | | | | | | | 1 |
| Horses feed | 2 | | | 4 | 7 | 1 | | | | 1 | | 15 |
| Linseed | 1 | | 1 | | | | | 1 | 4 | 6 | 6 | 19 |
| Linseed expeller/extracted | 1 | | 2 | | | | | | 1 | | | 4 |
| Linseed oil | | | 1 | 1 | | 1 | | | | 1 | | 4 |
| Lupines | 1 | | | | 1 | | | | | 1 | 4 | 7 |
| Maize kernel | | | | | 2 | | | 3 | 3 | 1 | | 9 |
| Maize product | 27 | 26 | 12 | 13 | 24 | 3 | | 17 | 15 | 11 | 11 | 159 |
| Mari seed | | 2 | | | | | | | | | | 2 |
| Melasse, sugarcane- | | | | | | | | | 1 | | | 1 |
| Milk powder | | | | | | | 2 | 1 | | | | 3 |
| Millet | 1 | | | | 1 | 1 | | 1 | 1 | | 5 | 10 |
| Mineral mix | 1 | 8 | 1 | 1 | | | | | | | | 11 |
| Mix fat | 4 | 2 | 7 | | 1 | 2 | 2 | 5 | | | | 23 |
| Mixed/compound feed | 5 | | 1 | 3 | 1 | | | | | | | 10 |
| Mixed/compound feed, complementary | | 1 | | 1 | | | | 1 | | | | 3 |
| Nigerseed | | | | | 1 | | | | | | | 1 |
| nvt | | | | | | | | 1 | | 1 | | 2 |
| Oats grain | 3 | | 1 | | | | | 1 | 1 | 1 | 7 | 14 |
| Other commodities | | | | | | | | 2 | | | | 2 |
| Palmkernel expeller/extracted | 11 | 6 | 6 | 5 | 6 | 1 | 2 | | 4 | 4 | 1 | 46 |
| Palmkernel oil | 6 | 7 | 9 | 7 | 6 | 4 | 3 | 4 | 19 | 14 | 22 | 101 |
| Palmkernels | | | | | | | | | | 1 | | 1 |
| Peas | 1 | | | 3 | 2 | 1 | | | 1 | 1 | 19 | 28 |
| Petfood | | | | 1 | 6 | | | | | | | 7 |
| Phosphate combination | | | | | | | | | 1 | | | 1 |
| Pig feed | 43 | 44 | 65 | 27 | 31 | 21 | 22 | 1 | | | | 254 |
| Pig feed, complementary | 4 | 3 | 8 | 4 | 4 | 1 | 2 | | | | | 26 |
| Pig feed, pulp | 2 | 1 | 11 | 19 | 18 | | | | | | | 51 |
| Pine Nuts | | | | | | | | | 1 | | | 1 |
| Porcine fat | 4 | 5 | 5 | 4 | 13 | 9 | 8 | 4 | | | | 52 |
| Potato chips waste | | | | | 1 | 1 | | | | 7 | | 9 |
| Potato peels | | | | | 2 | 2 | | | | 7 | | 11 |
| Potato starch | | 2 | | | | 1 | | | | 1 | | 4 |
| Potatoes | | 1 | | 1 | 3 | 6 | 5 | 1 | 1 | 29 | | 47 |
| Poultry fat | 4 | 1 | 5 | 1 | 10 | 7 | 2 | 5 | | | | 35 |
| Poultry feed | 37 | 24 | 35 | 20 | 23 | 1 | | | | | | 140 |
| Poultry feed, complementary | 3 | 7 | 7 | 3 | 3 | | | | | | | 23 |
| Rabbit food | | | | 1 | 3 | | | | | | | 4 |

| Product(group) NP | 2001 | 2002 | 2003 | 2004 | 2005 | 2006 | 2007 | 2008 | 2009 | 2010 | 2011 | Grand Total |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Rapeseed oil | | | | | 2 | 1 | 1 | 1 | 1 | 5 | 5 | 16 |
| Residues of food production | | | | | 6 | 5 | 3 | 3 | | 4 | | 21 |
| Rice | | | | | | | | 2 | 1 | 1 | | 4 |
| Rice product | | 4 | | | 2 | | | 1 | 1 | | 1 | 9 |
| Ruminant feed | | | | | | | | | | 1 | | 1 |
| Ruminant feed, complementary | 1 | 1 | 3 | 1 | 4 | 1 | | 2 | | | 1 | 14 |
| Rye | | | 1 | 1 | | | 1 | | | | 4 | 7 |
| Safflower seed | | | | | | | | | | | 1 | 1 |
| Safflowerseed expeller/extracted | | 2 | | 1 | | | | | | | | 3 |
| Sesame seed | | | | | | | 1 | | | | | 1 |
| Sheep feed | 3 | 1 | | | | 1 | | | | | | 5 |
| Sheep feed complementary | 2 | 8 | 1 | 1 | 2 | 6 | 1 | | | | | 21 |
| Sorghum | | | | | | | | 1 | | 2 | | 3 |
| Soya bean expeller/extracted | 44 | 52 | 28 | 21 | 34 | 5 | 25 | 9 | 35 | | 1 | 254 |
| Soya beans | 1 | 3 | 3 | 2 | 2 | 2 | 7 | 9 | 2 | 3 | 1 | 35 |
| Soya product | 9 | 13 | 8 | 8 | 12 | 1 | 11 | 7 | 14 | | 3 | 86 |
| Soybean fatty acids | 1 | | | | | | | | 2 | | | 3 |
| Soybean oil | 46 | 15 | 11 | 6 | 9 | 7 | 5 | 8 | 23 | 8 | 1 | 139 |
| Spelt | | 1 | | | | | | | | 1 | | 2 |
| Sugar | | | | | | 1 | | | | | | 1 |
| Sugarbeet pulp | 1 | 4 | 5 | 6 | 7 | 11 | | 19 | | 13 | 3 | 69 |
| Sunflower oil | | | | | 4 | 1 | 2 | | 4 | 11 | 9 | 31 |
| Sunflower seed | 15 | 20 | 2 | 3 | 4 | 1 | | 1 | 1 | | 1 | 48 |
| Sunflower seed expeller/extracted | 3 | 1 | 4 | 2 | 2 | | 1 | 5 | 3 | 3 | 5 | 29 |
| Table salt | | | | | 1 | | | | | | | 1 |
| Tapioca | 12 | 3 | 1 | 9 | | | | 1 | | | | 26 |
| Triticale | 1 | 1 | 4 | 1 | 1 | 1 | | | | | | 9 |
| Vegetable feeding stuffs | | 1 | 5 | | 3 | 2 | 3 | 1 | 3 | 2 | | 20 |
| Vitamin mix | | | | | | | | | | | 1 | 1 |
| Wheat | 12 | 8 | 8 | 8 | 7 | 1 | | 12 | 5 | 7 | 17 | 85 |
| Wheat product | 4 | 1 | 1 | 2 | 4 | 1 | | 2 | 2 | 3 | 2 | 22 |
| Wheat yeast concentrate | | | | | | | | | | 14 | 51 | 65 |
| Grand Total | 462 | 434 | 352 | 243 | 391 | 178 | 221 | 223 | 235 | 236 | 231 | 3206 |

Table 6.4

*Number of monitoring samples (only the additional GC HRMS samples for NDL-PCBs) per year per product – NP.*

| Product(group) NP | 2007 | 2008 | 2009 | 2010 | 2011 | Grand Total |
|---|---|---|---|---|---|---|
| Additive | | 1 | 8 | 1 | 2 | 12 |
| Alfalfa | 1 | 2 | 15 | | | 18 |
| Algae/seaweed | | | 1 | | | 1 |
| Amino acids | | | 1 | 2 | | 3 |
| Animal fat | | 1 | 3 | 2 | 1 | 7 |
| Animal feed, general | | 1 | | | | 1 |
| Artificial milk feed | 1 | 1 | | | | 2 |
| Ascorbinic acid | | | 1 | | | 1 |
| Bakery waste | | 1 | 2 | | | 3 |
| Barley | | 1 | | | | 1 |
| Bird food | | 1 | | | | 1 |
| Bovine feed | | 1 | | | | 1 |
| Bovine feed, complementary | 2 | 7 | 14 | 11 | | 34 |
| Breadmeal | | | 5 | | | 5 |
| Calciumcarbonate | | 1 | | | | 1 |
| Calciumformiate | | | 2 | | | 2 |
| Choline chloride | 6 | 14 | 21 | 22 | | 63 |
| Citrus pulp | | 1 | | | | 1 |
| Clay minerals | 9 | 25 | 47 | 43 | | 124 |
| Clover | | | 2 | | | 2 |
| Coconut fat | | 1 | | | 6 | 7 |
| Cole-/rapeseed expeller/extracted | | 1 | 2 | | | 3 |

| Product(group) NP | 2007 | 2008 | 2009 | 2010 | 2011 | Grand Total |
|---|---|---|---|---|---|---|
| Copper chelate | | 1 | 2 | 1 | | 4 |
| Copper sulphate | | 2 | 2 | | | 4 |
| Dicalciumphosphate | | 2 | | | | 2 |
| Duckweed | | 1 | | | | 1 |
| Fat of bovine animal | | | 1 | 4 | | 5 |
| Fat/oil, vegetable | | | | 2 | 10 | 12 |
| Fatty acids | | 2 | 2 | 2 | 19 | 25 |
| Fish food | | 1 | | | | 1 |
| Fish meal | 6 | 9 | 41 | 37 | | 93 |
| Fish oil | | 6 | 14 | 10 | 17 | 47 |
| Fish protein | | 1 | 1 | 9 | | 11 |
| Goat feed, complementary | | 1 | | | | 1 |
| Grass | 1 | 23 | 25 | | | 49 |
| Green maize | | 1 | | | | 1 |
| Groundnut oil | | | | | 1 | 1 |
| Herbal mix | | | 1 | | | 1 |
| Horses feed | 1 | 4 | | | | 5 |
| Iron chelate | 1 | | | | | 1 |
| Iron oxide | 1 | | | 1 | | 2 |
| Iron sulphate | | 1 | | | | 1 |
| Linseed | 1 | 1 | | | | 2 |
| Linseed oil | | | | | 1 | 1 |
| Magnesium oxide | | 4 | | 1 | | 5 |
| Maize kernel | | 1 | | | | 1 |
| Maize product | 2 | 2 | 6 | | | 10 |
| Milk powder | 1 | 1 | | | 5 | 7 |
| Mineral mix | | 3 | 4 | 6 | | 13 |
| Mix fat | | | | | 4 | 4 |
| Mixed/compound feed | | | | 1 | | 1 |
| Mixed/compound feed, complementary l | | 1 | | | | 1 |
| Monoammoniumphosphate | 1 | | | | | 1 |
| Other commodities | | | | | 6 | 6 |
| Palmkernel oil | | 2 | 4 | 1 | 1 | 8 |
| Petfood | | 2 | | | | 2 |
| Pig feed | 7 | 13 | | 2 | | 22 |
| Pig feed, complementary | | 3 | | 1 | | 4 |
| Pig feed, pulp | 1 | 3 | | | | 4 |
| Potassium iodide | 1 | | | | | 1 |
| Potato peels | | | 1 | 4 | | 5 |
| Potatoes | | | 2 | 1 | | 3 |
| Poultry feed | 2 | 10 | | | | 12 |
| Premix | 2 | 9 | 3 | 2 | | 16 |
| Rabbit food | | 4 | | | | 4 |
| Residues of food production | | 1 | | | | 1 |
| Ruminant feed, complementary | | 7 | 2 | 7 | | 16 |
| Rye | 1 | | | | | 1 |
| Shrimps | | | 2 | 2 | | 4 |
| Sodium chloride | | | | | 1 | 1 |
| Soya product | | 3 | 4 | 1 | | 8 |
| Soybean oil | | 4 | 7 | 6 | 3 | 20 |
| Sunflower oil | | | 1 | | 1 | 2 |
| Sunflower seed | | 1 | | 2 | | 3 |
| Sunflower seed expeller/extracted | | | 1 | | | 1 |
| Tapioca | 1 | | | | | 1 |
| Vegetable feeding stuffs | | | | 1 | | 1 |
| Vitamin mix | | | 5 | 1 | | 6 |
| Wheat | 1 | 4 | | | | 5 |
| Wheat product | 1 | | | | | 1 |
| Zincsulphate | | 1 | 1 | | | 2 |
| Grand Total | 51 | 195 | 256 | 186 | 78 | 766 |

## Table 6.5

*Number of monitoring samples per year per product – IND.*

| Product(group) IND | 2005 | 2006 | 2007 | 2008 | 2009 | 2010 | 2011 | Grand Total |
|---|---|---|---|---|---|---|---|---|
| Alfalfa | | | 1 | 4 | 3 | 3 | | 11 |
| Animal fat | | 12 | 13 | 13 | 12 | 5 | 2 | 57 |
| Bakery waste | | 3 | 3 | 7 | 3 | 2 | 4 | 22 |
| Barley | | 12 | 12 | 16 | 17 | 21 | 9 | 87 |
| Barley mill byproduct | | 4 | | | | | 2 | 6 |
| Beans | | | | | | 2 | 1 | 3 |
| Brewers grains | | | 1 | | 1 | 3 | | 5 |
| Cacao waste | | | | | | 1 | | 1 |
| Calciumcarbonate | | | | 2 | 2 | 1 | 2 | 7 |
| Citrus pulp | 1 | 5 | 6 | 6 | 6 | 7 | 5 | 36 |
| Clay minerals | | | | 1 | | | | 1 |
| Coconut expeller/extracted | | 3 | | | | | | 3 |
| Coconut fat | | | 3 | 7 | 3 | 2 | 2 | 17 |
| Cole-/rapeseed | | | 1 | 2 | 2 | | | 5 |
| Cole-/rapeseed expeller/extracted | 1 | 8 | 10 | 11 | 15 | 26 | 12 | 83 |
| Fat/oil, vegetable | | 4 | 3 | 2 | 1 | 1 | 1 | 12 |
| Fatty acids | | 30 | 16 | 8 | 5 | 5 | 7 | 71 |
| Fish meal | 1 | 6 | 5 | 16 | 5 | 1 | 1 | 35 |
| Fish oil | | | 5 | 6 | 7 | | 1 | 19 |
| Grass | | | | | | 1 | | 1 |
| Grass seed | | 1 | | | | | | 1 |
| Lecithin | | 4 | 4 | 2 | | | | 10 |
| Linseed | | 4 | 1 | 2 | 3 | 5 | 6 | 21 |
| Linseed expeller/extracted | | | | 2 | 2 | 3 | 1 | 8 |
| Lupines | | 2 | 1 | 1 | 2 | 2 | | 8 |
| Magnesium oxide | | | 1 | 1 | 1 | 1 | | 4 |
| Maize kernel | | 1 | 1 | | | 7 | 6 | 15 |
| Maize product | | 29 | 135 | 64 | 62 | 64 | 36 | 390 |
| Melasse, beet- | | 11 | | 1 | 2 | 1 | 1 | 16 |
| Melasse, sugarcane- | | 2 | 3 | 3 | 6 | 3 | 2 | 19 |
| Monocalciumphosphate | | 2 | 1 | 1 | 1 | | | 5 |
| Oats grain | | 3 | 5 | 7 | 7 | 9 | 10 | 41 |
| Palmkernel expeller/extracted | 1 | 3 | 3 | 2 | 6 | 15 | 6 | 36 |
| Palmkernel oil | 1 | 27 | 9 | 11 | 7 | 7 | 6 | 68 |
| Peas | | 3 | 4 | 3 | 3 | 10 | 6 | 29 |
| Potatoes | | 2 | | 2 | 1 | 5 | 6 | 16 |
| Poultry fat | | | 5 | | 3 | | | 8 |
| Rice product | | | | 1 | 2 | 5 | 5 | 13 |
| Rye | | 3 | 1 | 6 | 16 | 5 | 4 | 35 |
| Sorghum | | 1 | 1 | 4 | | | | 6 |
| Soya bean expeller/extracted | | 10 | 18 | 19 | 13 | 23 | 17 | 100 |
| Soya beans | | 4 | 3 | 5 | 3 | 4 | 1 | 20 |
| Soya product | | 6 | 6 | 7 | 6 | 27 | 14 | 66 |
| Soybean fatty acids | | 3 | 5 | 10 | 2 | 2 | | 22 |
| Soybean oil | | 12 | 7 | 11 | 10 | 14 | 7 | 61 |
| Spelt | | | | 2 | | | | 2 |
| Sugarbeet pulp | | 3 | 4 | 7 | 5 | 3 | | 22 |
| Sunflower seed | | 3 | | | 2 | 1 | 3 | 9 |
| Sunflower seed expeller/extracted | 1 | 3 | 8 | 10 | 13 | 17 | 10 | 62 |
| Tapioca | | | | 2 | | | | 2 |
| Triticale | | 2 | | 2 | 17 | 12 | 3 | 36 |
| Vegetable feeding stuffs | | | | 1 | 1 | | 1 | 3 |
| Vinasse | | 7 | 1 | 3 | 2 | 2 | 2 | 17 |
| Water | | 1 | | 2 | 3 | 7 | 1 | 14 |
| Wheat | | 19 | 8 | 37 | 38 | 29 | 15 | 146 |
| Wheat product | | 8 | 5 | 16 | 15 | 40 | 19 | 103 |
| Wheat yeast concentrate | | | | | | | 1 | 1 |
| Yeast | | | | | 1 | 6 | 8 | 15 |
| Grand Total | 6 | 266 | 319 | 348 | 337 | 410 | 246 | 1919 |

## Table 6.6

*Incidence of endosulfan in animal feed - grouped by product(group).*

| Product (group) | Year | N total* | N pos | N ≥ limit | Result -range (mg/kg) (min-max) | | Limit (mg/kg) |
|---|---|---|---|---|---|---|---|
| Apple | 2005 | 2 | 2 | 1 | 0.09 | 0.33 | 0.1 |
| Barley mill by product | 2006 | 4 | 1 | 0 | 0 | 0 | 0.1 |
| Cacao waste | 2003 | 2 | 2 | 0 | 0.03 | 0.04 | 0.1 |
| Coconut fat | 2010 | 8 | 1 | 0 | 0.24 | 0.24 | 0.1 |
| Cottonseed | 2002 | 1 | 1 | 0 | 0.03 | 0.03 | 0.1 |
| Fat/oil, vegetable | 2004 | 1 | 1 | 0 | 0.49 | 0.49 | 0.5 or 1.0 |
| Fat/oil, vegetable | 2006 | 5 | 1 | 0 | 0.06 | 0.06 | 0.5 or 1.0 |
| Fatty acids | 2006 | 36 | 5 | 4 | 0.4 | 2 | 0.5 |
| Fatty acids | 2007 | 19 | 3 | 1 | 0.35 | 0.7 | 0.5 |
| Fatty acids | 2009 | 8 | 1 | 0 | 0.12 | 0.12 | 0.5 |
| Horses feed | 2004 | 4 | 2 | 0 | 0.01 | 0.01 | 0.1 |
| Mixed/compound feed | 2004 | 3 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Palmkernel oil | 2010 | 21 | 1 | 0 | 0.16 | 0.16 | 0.5 or 1.0 |
| Pig feed | 2003 | 65 | 3 | 0 | 0.01 | 0.02 | 0.1 |
| Pig feed | 2004 | 27 | 4 | 0 | 0.01 | 0.02 | 0.1 |
| Pig feed | 2005 | 31 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Pig feed | 2006 | 21 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Pig feed, complementary | 2003 | 8 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Pig feed, pulp | 2005 | 18 | 3 | 0 | 0.01 | 0.02 | 0.1 |
| Potato chips waste | 2005 | 1 | 1 | 0 | 0.04 | 0.04 | 0.1 |
| Potato peels | 2005 | 2 | 2 | 0 | 0.02 | 0.02 | 0.1 |
| Potatoes | 2010 | 34 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Poultry feed | 2003 | 35 | 7 | 0 | 0.01 | 0.03 | 0.1 |
| Poultry feed | 2004 | 20 | 8 | 0 | 0.01 | 0.02 | 0.1 |
| Poultry feed | 2005 | 23 | 1 | 0 | 0.03 | 0.03 | 0.1 |
| Poultry feed, complementary | 2003 | 7 | 1 | 0 | 0.03 | 0.03 | 0.1 |
| Poultry feed, complementary | 2004 | 3 | 2 | 0 | 0.01 | 0.03 | 0.1 |
| Rice product | 2008 | 2 | 1 | 0 | 0.05 | 0.05 | 0.1 |
| Soya bean expeller/extracted | 2003 | 28 | 1 | 0 | 0.01 | 0.01 | 0.5 |
| Soya bean expeller/extracted | 2005 | 34 | 3 | 0 | 0.01 | 0.02 | 0.5 |
| Soya beans | 2002 | 3 | 1 | 0 | 0.06 | 0.06 | 0.5 |
| Soya beans | 2004 | 2 | 1 | 0 | 0.02 | 0.02 | 0.5 |
| Soya beans | 2006 | 6 | 2 | 0 | 0.02 | 0.09 | 0.5 |
| Soya beans | 2010 | 7 | 2 | 0 | 0.03 | 0.05 | 0.5 |
| Soya product | 2002 | 13 | 1 | 0 | 0.16 | 0.16 | 0.5 |
| Soya product | 2003 | 8 | 3 | 0 | 0.02 | 0.03 | 0.5 |
| Soya product | 2004 | 8 | 2 | 0 | 0.03 | 0.03 | 0.5 |
| Soya product | 2005 | 12 | 2 | 0 | 0.02 | 0.09 | 0.5 |
| Soya product | 2010 | 27 | 3 | 0 | 0.02 | 0.05 | 0.5 |
| Soya product | 2011 | 17 | 2 | 0 | 0.02 | 0.02 | 0.5 |
| Soybean fatty acids | 2008 | 10 | 10 | 0 | 0.01 | 0.14 | 0.5 |
| Soybean fatty acids | 2010 | 2 | 1 | 0 | 0.16 | 0.16 | 0.5 |
| Soybean oil | 2001 | 46 | 28 | 1 | 0.08 | 0.56 | 0.5 or 1.0 |
| Soybean oil | 2002 | 15 | 6 | 0 | 0.08 | 0.39 | 0.5 or 1.0 |
| Soybean oil | 2003 | 11 | 9 | 2 | 0.09 | 0.58 | 0.5 or 1.0 |
| Soybean oil | 2004 | 6 | 3 | 0 | 0.07 | 0.49 | 0.5 or 1.0 |
| Soybean oil | 2005 | 9 | 6 | 0 | 0.08 | 0.48 | 0.5 or 1.0 |
| Soybean oil | 2006 | 19 | 5 | 0 | 0.22 | 0.37 | 0.5 or 1.0 |
| Soybean oil | 2007 | 12 | 1 | 0 | 0.24 | 0.24 | 0.5 or 1.0 |
| Soybean oil | 2008 | 19 | 4 | 2 | 0.06 | 0.66 | 0.5 or 1.0 |
| Soybean oil | 2009 | 33 | 18 | 0 | 0.08 | 0.48 | 0.5 or 1.0 |
| Soybean oil | 2010 | 22 | 9 | 1 | 0.19 | 0.71 | 0.5 or 1.0 |
| Soybean oil | 2011 | 8 | 4 | 0 | 0.16 | 0.41 | 0.5 or 1.0 |
| Sugarbeet pulp | 2002 | 4 | 1 | 0 | 0.07 | 0.07 | 0.1 |
| Sunflower seed expeller/extracted | 2010 | 20 | 1 | 0 | 0.03 | 0.03 | 0.5 |
| Vegetable feeding stuffs | 2003 | 5 | 2 | 0 | 0.07 | 0.07 | 0.1 |
| Wheat | 2005 | 7 | 1 | 0 | 0.02 | 0.02 | 0.1 |

* N total = number of samples from this product group analysed for this compound in the specific year.

N pos = number of samples with values above LOQ.

N ≥ limit= number of samples with values equal to or above the legal limit.

## Table 6.7

*Incidence of endosulfan in animal feed - grouped by year.*

| Product (group) | Year | N total* | N pos | N > limit | Result -range (mg/kg) (min-max) | | Limit (mg/kg) |
|---|---|---|---|---|---|---|---|
| Soybean oil | 2001 | 46 | 28 | 1 | 0.08 | 0.56 | 0.5 |
| Cottonseed | 2002 | 1 | 1 | 0 | 0.03 | 0.03 | 0.1 |
| Soya beans | 2002 | 3 | 1 | 0 | 0.06 | 0.06 | 0.5 |
| Soya product | 2002 | 13 | 1 | 0 | 0.16 | 0.16 | 0.5 |
| Soybean oil | 2002 | 15 | 6 | 0 | 0.08 | 0.39 | 0.5 |
| Sugarbeet pulp | 2002 | 4 | 1 | 0 | 0.07 | 0.07 | 0.1 |
| Cacao waste | 2003 | 2 | 2 | 0 | 0.03 | 0.04 | 0.1 |
| Pig feed | 2003 | 65 | 3 | 0 | 0.01 | 0.02 | 0.1 |
| Pig feed, complementary | 2003 | 8 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Poultry feed | 2003 | 35 | 7 | 0 | 0.01 | 0.03 | 0.1 |
| Poultry feed, complementary | 2003 | 7 | 1 | 0 | 0.03 | 0.03 | 0.1 |
| Soya bean expeller/extracted | 2003 | 28 | 1 | 0 | 0.01 | 0.01 | 0.5 |
| Soya product | 2003 | 8 | 3 | 0 | 0.02 | 0.03 | 0.5 |
| Soybean oil | 2003 | 11 | 9 | 2 | 0.09 | 0.58 | 0.5 |
| Vegetable feeding stuffs | 2003 | 5 | 2 | 0 | 0.07 | 0.07 | 0.1 |
| Fat/oil, vegetable | 2004 | 1 | 1 | 0 | 0.49 | 0.49 | 0.5 |
| Horses feed | 2004 | 4 | 2 | 0 | 0.01 | 0.01 | 0.1 |
| Mixed/compound feed | 2004 | 3 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Pig feed | 2004 | 27 | 4 | 0 | 0.01 | 0.02 | 0.1 |
| Poultry feed | 2004 | 20 | 8 | 0 | 0.01 | 0.02 | 0.1 |
| Poultry feed, complementary | 2004 | 3 | 2 | 0 | 0.01 | 0.03 | 0.1 |
| Soya beans | 2004 | 2 | 1 | 0 | 0.02 | 0.02 | 0.5 |
| Soya product | 2004 | 8 | 2 | 0 | 0.03 | 0.03 | 0.5 |
| Soybean oil | 2004 | 6 | 3 | 0 | 0.07 | 0.49 | 0.5 |
| Apple | 2005 | 2 | 2 | 1 | 0.09 | 0.33 | 0.1 |
| Pig feed | 2005 | 31 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Pig feed, pulp | 2005 | 18 | 3 | 0 | 0.01 | 0.02 | 0.1 |
| Potato chips waste | 2005 | 1 | 1 | 0 | 0.04 | 0.04 | 0.1 |
| Potato peels | 2005 | 2 | 2 | 0 | 0.02 | 0.02 | 0.1 |
| Poultry feed | 2005 | 23 | 1 | 0 | 0.03 | 0.03 | 0.1 |
| Soya bean expeller/extracted | 2005 | 34 | 3 | 0 | 0.01 | 0.02 | 0.5 |
| Soya product | 2005 | 12 | 2 | 0 | 0.02 | 0.09 | 0.5 |
| Soybean oil | 2005 | 9 | 6 | 0 | 0.08 | 0.48 | 0.5 |
| Wheat | 2005 | 7 | 1 | 0 | 0.02 | 0.02 | 0.1 |
| Barley mill byproduct | 2006 | 4 | 1 | 0 | 0 | 0 | 0.1 |
| Fat/oil, vegetable | 2006 | 5 | 1 | 0 | 0.06 | 0.06 | 0.5 |
| Fatty acids | 2006 | 36 | 5 | 4 | 0.4 | 2 | 0.5 |
| Pig feed | 2006 | 21 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Soya beans | 2006 | 6 | 2 | 0 | 0.02 | 0.09 | 0.5 |
| Soybean oil | 2006 | 19 | 5 | 0 | 0.22 | 0.37 | 0.5 |
| Fatty acids | 2007 | 19 | 3 | 1 | 0.35 | 0.7 | 0.5 |
| Soybean oil | 2007 | 12 | 1 | 0 | 0.24 | 0.24 | 0.5 |
| Rice product | 2008 | 2 | 1 | 0 | 0.05 | 0.05 | 0.1 |
| Soybean fatty acids | 2008 | 10 | 10 | 0 | 0.01 | 0.14 | 0.5 |
| Soybean oil | 2008 | 19 | 4 | 2 | 0.06 | 0.66 | 0.5 |
| Fatty acids | 2009 | 8 | 1 | 0 | 0.12 | 0.12 | 0.5 |
| Soybean oil | 2009 | 33 | 18 | 0 | 0.08 | 0.48 | 0.5 |
| Coconut fat | 2010 | 8 | 1 | 0 | 0.24 | 0.24 | 0.1 |
| Palmkernel oil | 2010 | 21 | 1 | 0 | 0.16 | 0.16 | 0.5 |
| Potatoes | 2010 | 34 | 1 | 0 | 0.01 | 0.01 | 0.1 |
| Soya beans | 2010 | 7 | 2 | 0 | 0.03 | 0.05 | 0.5 |
| Soya product | 2010 | 27 | 3 | 0 | 0.02 | 0.05 | 0.5 |
| Soybean fatty acids | 2010 | 2 | 1 | 0 | 0.16 | 0.16 | 0.5 |
| Soybean oil | 2010 | 22 | 9 | 1 | 0.19 | 0.71 | 0.5 |
| Sunflower seed expeller/extracted | 2010 | 20 | 1 | 0 | 0.03 | 0.03 | 0.5 |
| Soya product | 2011 | 17 | 2 | 0 | 0.02 | 0.02 | 0.5 |
| Soybean oil | 2011 | 8 | 4 | 0 | 0.16 | 0.41 | 0.5 |

* N total = number of samples from this product group analysed for this compound in the specific year.

N pos = number of samples with values above LOQ.

N ≥ limit= number of samples with values equal to or above the legal limit.

RIKILT Wageningen UR
P.O. Box 230
6700 AE Wageningen
The Netherlands
T +31 (0)317 48 02 56
www.wageningenUR.nl/en/rikilt

RIKILT report 2013.009

RIKILT Wageningen UR is part of the international knowledge organisation Wageningen University & Research centre. RIKILT conducts independent research into the safety and quality of food. The institute is specialised in detecting and identifying substances in food and animal feed and determining the functionality and effect of those substances.

The mission of Wageningen UR (University & Research centre) is 'To explore the potential of nature to improve the quality of life'. Within Wageningen UR, nine specialised research institutes of the DLO Foundation have joined forces with Wageningen University to help answer the most important questions in the domain of healthy food and living environment. With approximately 30 locations, 6,000 members of staff and 9,000 students, Wageningen UR is one of the leading organisations in its domain worldwide. The integral approach to problems and the cooperation between the various disciplines are at the heart of the unique Wageningen Approach.

To explore the potential of nature to improve the quality of life

RIKILT Wageningen UR
P.O. Box 230
6700 AE Wageningen
The Netherlands
T +31 (0)317 48 02 56
www.wageningenUR.nl/en/rikilt

RIKILT report 2013.009

RIKILT Wageningen UR is part of the international knowledge organisation Wageningen University & Research centre. RIKILT conducts independent research into the safety and quality of food. The institute is specialised in detecting and identifying substances in food and animal feed and determining the functionality and effect of those substances.

The mission of Wageningen UR (University & Research centre) is 'To explore the potential of nature to improve the quality of life'. Within Wageningen UR, nine specialised research institutes of the DLO Foundation have joined forces with Wageningen University to help answer the most important questions in the domain of healthy food and living environment. With approximately 30 locations, 6,000 members of staff and 9,000 students, Wageningen UR is one of the leading organisations in its domain worldwide. The integral approach to problems and the cooperation between the various disciplines are at the heart of the unique Wageningen Approach.