



Miljø- og Fødevareministeriet

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# Control of Pesticides and Biocides 2014

The Analytical Chemical Control of  
Pesticides and Biocides on the Danish  
Market

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Appendix 1:

Pesticide and biocide samples collected on the Danish market for authority control in 2014.

# Foreword

32 different types of products, covered by the pesticide and biocide regulation, were included in the Danish authority control in 2014. In this report pesticides are defined as plant protection products (PPP).

All collected products were examined for content of one or more active substances according to the declared content and the specification of the product given by the authorisation holder. The density of all liquid products was investigated.

All parallel products were screened by GC-MS and FTIR to investigate if there was a difference compared to the original products.

For the 2014 programme, 8 tested products (7 biocides and 1 pesticides) did not comply with the tolerance limits defined by the Danish Statutory Order on pesticides No. 151 of 18. February 2014 incl. revisions, and in Regulation 1107/2009 concerning marketing of plant protection products<sup>1+2+3</sup>.

# Konklusion og sammenfatning

Denne rapport beskriver den analytisk kemiske kontrol af biocid- og pesticidprodukter på det danske marked, der er udført af Miljøstyrelsens Kemikalieinspektion i 2014.<sup>4</sup>

32 produkter fra udvalgte typer af bekæmpelsesmidler er blevet indsamlet og analyseret for at verificere, om indholdet af de respektive aktivstoffer er i overensstemmelse med produktspecifikationen og det deklarerede indhold.

32 forskellige produkter er inkluderet i den analytisk kemiske kontrol, der blev udført af myndighederne i 2014:

- 1 insekticid (pesticid) indeholdende bifenazat
- 12 insekticider (biocider) indeholdende cyfluthrin, esbiothrin og permethrin
- 11 herbicider (pesticider) indeholdende 2,4-D, 2,4-D ethylhexyl, fenoxaprop-P-ethyl, maleinhydrazid, clopyralid og picloram.
- 3 sneglegifte (pesticider) indeholdende jern(III)fosfat
- 5 fungicider (pesticider) indeholdende svovl, metconazol, pyrimethanil, difenoconazol og epoxiconazol.

Aktivstof	Antal analyserede produkter	Antal inden for tolerance	Antal uden for tolerance
2,4-D	1	1	0
2,4-D ethylhexyl	2	2	0
Bifenazat	1	1	0
Clopyralid & picloram	1	1	0
Difenoconazol	1	1	0
Esbiothrin	1	0	1
Esbiothrin & permethrin	10	4	6
Fenoxaprop-P-ethyl	3	3	0
Jern(III)fosfat	4	4	0
Maleinhydrazid	3	2	1
Metconazol	1	1	0
Metconazol & epoxiconazol	1	1	0
Pyrimethanil	2	2	0
Svovl	1	1	0
Total	32	24	8

TABLE 1  
OVERSIGT OVER ANTAL ANALYSEREDE PRODUKTER OG KONKLUSION

Grænsen for en accepteret afvigelse i indholdet af et aktivstof i forhold til det deklarerede indhold og produktspecifikationen er fastsat i bekendtgørelsen om bekæmpelsesmidler Nr. 151 af 18. februar 2014 inkl. revisioner og i forordning 1107/2009 om markedsføring af plantebeskyttelsesmidler<sup>1+2+3+5+6</sup>.

Det målte indhold af aktivstoffer er ikke i overensstemmelse med det deklarerede indhold i 9 produkter, og overholder således ikke gældende dansk lovgivning, idet der mangler overlap mellem interval for analyseresultat og tolerance.

Det målte indhold af aktivstoffer er i overensstemmelse med det deklarerede indhold i 24 produkter, og overholder således gældende dansk lovgivning, idet der er overlap mellem interval for analyseresultat og tolerance.

Densitet blev målt på alle flydende produkter. Der blev foretaget screeninger af alle parallelprodukter og det tilhørende originalprodukt for at undersøge, om der var forskel på indhold af urenheder eller andre formuleringsingredienser.

# Conclusion and summary

This report describes the analytical chemical control of pesticide and biocide products on the Danish market that was carried out by the Danish Environmental Protection Agency (Danish EPA), Chemical Inspection Service, in 2014.<sup>4</sup>

32 products from selected types of pesticides and biocides were collected and analysed to verify whether or not the content of the active substances in the products in question complied with the product specification and the declared content.

32 different products are covered by the analytical chemical control that was carried out by the authorities in 2014:

- 1 insecticide (Pesticide) containing Bifenazate
- 12 insecticides (Biocides) containing Cyfluthrin, Esbiothrin and Permethrin
- 11 herbicides containing 2,4-D, 2,4-D ethylhexyl, Fenoxaprop-P-ethyl, Maleic hydrazide, Clopyralid and Picloram
- 3 molluscicides (pesticides) containing Iron(III)phosphate.
- 5 fungicides (pesticides) containing Sulphur, Pyrimethanil, Difenoconazole, Metconazole and Epoxiconazole

Active substances	Number of analysed products	Number of products comply	Number of products non-comply
2,4-D	1	1	0
2,4-D ethylhexyl	2	2	0
Bifenazate	1	1	0
Clopyralid & Picloram	1	1	0
Difenoconazole	1	1	0
Esbiothrin	1	0	1
Esbiothrin & Permethrin	10	4	6
Fenoxaprop-P-ethyl	3	3	0
Iron(III)Phosphate	4	4	0
Maleic hydrazide	3	2	1
Metconazole	1	1	0
Metconazole & Epoxiconazole	1	1	0
Pyrimethanil	2	2	0
Sulphur	1	1	0
Total	32	24	8

**TABLE 1**  
OUTLINE OF TOTAL OF ANALYSED PRODUCTS AND CONCLUSION



The tolerance of an accepted deviation in the content of an active substance compared to the declared content and product specification is determined in the Danish Statutory Order on pesticides No. 151 of 18. February 2014 incl. revisions, and in Regulation 1107/2009 concerning Marketing of Plant Protection Products<sup>1+2+3+5+6</sup>.

The measured content of active component did not comply with the declared content for 9 products and is not in compliance with applicable Danish law, because of the lack of overlap between the range of analysis results and tolerance.

The measured content of active compounds complies with the declared content for 24 products and is in compliance with applicable Danish law, because of the overlap between the range of analysis results and tolerance.

The density of all liquid products was measured. Screenings were carried out of all parallel products including the original product to investigate if there was a difference in the content of impurities or other formulation ingredients.

# 1. Control campaigns in 2014

## 1.1 Selected active substances

The control campaigns conducted in 2014 covered 16 active substances in pesticides. Overall, pesticides are distinguished in two types: plant protection products and biocides (non-agricultural pesticides).

An outline of the selected active substances is given in following table:

Type	Active substance	CAS no.	Area of application
<b>Biocides</b>	Cyfluthrin	68359-37-5	Insecticide
	Esbiothrin	584-79-2	Insecticide
	Permethrin	5645-53-1	Insecticide
<b>Pesticides</b>	2,4-D	94-75-7	Herbicide
	2,4-D ethylhexyl	1928-43-4	Herbicide
	Bifenazate	149877-41-8	Insecticide
	Clopyralid	1702-17-6	Herbicide
	Picloram	1918-02-1	Herbicide
	Difenoconazole	119446-68-3	Fungicide
	Fenoxaprop-P-ethyl	71283-80-2	Herbicide
	Iron(III)phosphate	10045-96-0	Molluscicide
	Maleic hydrazide	123-33-1	Herbicide
	Metconazole	125116-23-6	Fungicide
	Epoxiconazole	133855-98-8	Fungicide
	Pyrimethanil	53112-28-0	Fungicide
	Sulphur	7704-34-9	Fungicide

**TABLE 2**  
OUTLINE OF SELECTED ACTIVE SUBSTANCES IN CONTROL CAMPAIGNS IN 2014

## 1.2 Collecting products

Product samples of the various pesticide and biocide formulations from different manufacturers, covered in the 2014 control campaigns, were collected by the Chemical Inspection Service from the Danish Environmental Protection Agency, Chemical Inspection Service, during the period from April to August 2014. The product samples were collected either from wholesale dealers/importers or at retailer outlets.

The collected product samples were stored at Danish Technological Institute (DTI) in the original packaging until the chemical analyses were initiated. The product samples were stored at ambient temperature and protected from light during the entire storage period. One product was stored in a refrigerator.

## 1.3 Tolerance for active substances

The Danish EPA aims to examine the content of active substances in the products and will compare the result of the chemical analysis with the content according to the specification of the products and the declared content on the label supplied by the authorisation holder.

The Danish Statutory Order on pesticides No. 151 of 18. February 2014 incl. revisions, and the Regulation 1107/2009 concerning Marketing of Plant Protection Products specify the general tolerance of deviation from the declared content<sup>1+2+3</sup>. These tolerances are listed in the following table:

Content of active substances in g/kg or g/L at 20°C	Tolerance of deviation
Up to 25	± 15% homogeneous formulation
	± 25% non-homogeneous formulation
More than 25 up to 100	± 10%
More than 100 up to 250	± 6%
More than 250 up to 500	± 5%
More than 500	± 25 g/kg or ± 25 g/L

TABLE 3  
THE TOLERANCE OF DEVIATION FROM THE DECLARED CONTENT OF ACTIVE SUBSTANCES 2014

## 1.4 Chemical analysis 2014

The analyses of the products for the active substances were performed by Danish Technological Institute, Laboratory for Chemistry and Microbiology. DTI is a self-owned and not-for-profit institute<sup>7</sup>.

The Laboratory for Chemistry and Microbiology is accredited by DANAK (Danish Accreditation and Metrology Fund), registration no. 90, according DS/EN ISO/IEC 17025<sup>8</sup>. The Laboratory also complies with the GMP regulation of the Danish Medicines Agency regarding analysis of medicinal products and intermediate products (§ 39).

#### **1.4.1 Analysing active substances**

If the sample amount was sufficient, the chemical analyses were as far as possible performed as eight freshly prepared samples of each product. The analyses were distributed over two days (four samples each day), typically within the same week. The mean value of the eight analyses and the SD (standard deviation) were calculated for each day and for all eight results.

Validation of the applied analytical method was carried out. The method was validated with regard to linearity, specificity, accuracy and control tests at two levels spread over 2 days. Eight samples were analysed from each product to which a known amount of the relevant active substance had been added in order to calculate the recovery.

For each product, the expanded uncertainty ( $k=2$ ) was calculated on the basis of the spread of the results of the eight analyses, the spread of the three density measurements (only included if the product is liquid), and bias was calculated on the basis of the eight recovery investigations. The expanded analysis uncertainty is used to determine a 95% confidence interval concerning the analysis result.

The results  $\pm$  the expanded uncertainty were compared with the tolerance of deviation calculated from the declared content of active substances.

#### **1.4.2 Analysing density**

The density of liquid products was determined in triplicate by measuring with a densimeter (Densito 30 PX). The mean value and the SD were calculated.

The result in % (w/w) has been converted to g/L by using the measured density: Result (g/L) = Result (% (w/w)) \* density (kg/L)\*10.

#### **1.4.3 Screenings**

Screenings were carried out of selected products by gas chromatography with mass spectrometric detection (GC-MS) and Fourier transform infrared spectroscopy (FTIR).

## 2. Biocides

The action of the active substances, contained in the biocidal products, protects humans, animals, materials or articles against harmful organisms such as pests or bacteria.

For the Danish 2014 Pesticide Control Campaign, 12 biocides were selected, all of them insecticides. The selected active substances were Cyfluthrin, Esbiothrin and Permethrin.

### 2.1 Insecticides (Biocides)

Insecticides containing Cyfluthrin, Esbiothrin, and Permethrin as active substances were selected for the 2014 control campaign. All products were examined for their content of active substance

#### 2.1.1 Cyfluthrin

Cyfluthrin is an insecticide used in Denmark for control of flies, moth, ants and crop pests. Chemically it belongs to the pyrethroids and is a neurotoxin. Cyfluthrin acts on the nervous system of insects, and disturbs the function of neurons by interaction with the sodium channel.<sup>9+10</sup> Products with Cyfluthrin have not previously been selected by the Danish EPA for control of the content of active substances.

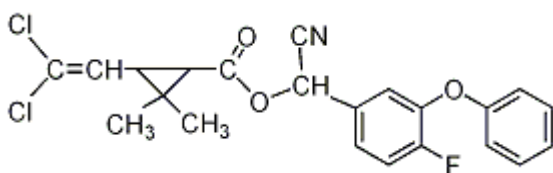


FIGURE 1 MOLECULAR STRUCTURE OF CYFLUTHRIN<sup>10</sup>, CAS NO. 68359-37-5

#### 2.1.2 Esbiothrin

Esbiothrin is an insecticide used in Denmark for control of flies, moth, ants and crop pests etc. Esbiothrin consists of a mixture of two (1R)-stereoisomers of allethrin and is also called by this name.<sup>12</sup> Chemically it belongs to the pyrethroids and is a neurotoxin. Esbiothrin acts on the nervous system of insects, and disturbs the function of neurons by interaction with the sodium channel.<sup>9+10</sup> Products with Esbiothrin have not previously been selected by the Danish EPA for control of the content of active substances.

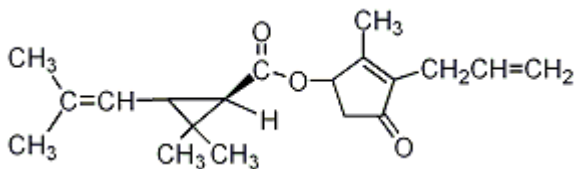


FIGURE 2 MOLECULAR STRUCTURE OF ESBIOTHRIN (ALLETHRIN)<sup>10</sup>, CAS NO. 584-79-2

#### 2.1.3 Permethrin

Permethrin is an insecticide used in Denmark for control of flies, moth, ants, and crop pests etc. Chemically it belongs to the pyrethroids and is a neurotoxin. Permethrin acts on the nervous system of insects, and disturbs the function of neurons by interaction with the sodium channel.<sup>9+10</sup>

Products with Permethrin were previously selected in 2008 by the Danish EPA for control of content of active substances.

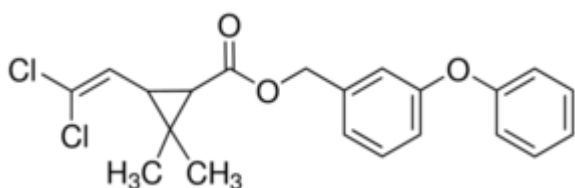


FIGURE 3 MOLECULAR STRUCTURE OF PERMETHRIN<sup>®</sup>, CAS NO. 52645-53-1

#### 2.1.4 Analysis methods

Cyfluthrin was analysed by reverse phase liquid chromatography combined with diode array detection (HPLC-DAD). Samples were dissolved and diluted in acetonitril by ultrasonication before analysis. The method is a modified HPLC method according to AOAC-CIPAC-method no. 385.

Esbiothrin and Permethrin were analysed by reverse phase liquid chromatography combined with diode array detection (HPLC-DAD). Before analysis, samples were dissolved and diluted in acetone by ultrasonication. The method is a modified HPLC method according to CIPAC-method no. 751 for Esbiothrin

Details concerning the implementation of the analyses and the calculation of the result and expanded analysis uncertainty are described in chapter 1.4.

The methods are accredited.

#### 2.1.5 Results

The results from the performed measurements and the corresponding tolerance intervals are listed in the following tables. % RSD is the percentage relative reproducibility of 8 determinations of the product. U<sub>E</sub> is the calculated expanded measurement uncertainty described in section 1.4.1.

DTI sample no.	Active substance	Label claim g/L	Tolerance interval	Analysis result g/L	RSD %	U <sub>E</sub> g/L	Analysis interval	Comply/ Non-Comply
38247-28	Cyfluthrin	50	45-55	54.4	0.3	1.7	52.7-56.1	Comply
38247-12	Esbiothrin	0.4	0.34-0.46	0.33	1.22	0.05	0.27-0.38	Comply
	Permethrin	2.4	2.04-2.76	1.40	1.27	0.06	1.34-1.46	Non-comply
38247-13	Esbiothrin	0.4	0.34-0.46	0.36	0.97	0.03	0.33-0.38	Comply
	Permethrin	2.4	2.04-2.76	1.48	1.07	0.08	1.40-1.56	Non-comply
38247-20	Esbiothrin	0.4	0.34-0.46	0.41	5.82	0.06	0.35-0.47	Comply
	Permethrin	2.4	2.04-2.76	2.03	5.82	0.25	1.77-2.28	Comply
38247-22	Esbiothrin	2.0	1.7-2.3	2.37	0.47	0.09	2.28-2.46	Comply
	Permethrin	2.5	2.125-2.875	2.79	0.99	0.10	2.69-2.89	Comply
38247-26	Esbiothrin	0.4	0.34-0.46	0.33	0.47	0.02	0.31-0.35	Comply
	Permethrin	2.4	2.04-2.76	1.41	0.41	0.06	1.34-1.47	Non-comply
38247-27	Esbiothrin	0.4	0.34-0.46	0.51	0.33	0.03	0.47-0.54	Non-comply
	Permethrin	2.4	2.04-2.76	2.26	0.33	0.10	2.16-2.37	Comply

DTI sample no.	Active substance	Label claim g/L	Tolerance interval	Analysis result g/L	RSD %	U <sub>E</sub> g/L	Analysis interval	Comply/ Non-Comply
38247-30	Esbiothrin	0.4	0.34-0.46	0.30	1.19	0.02	0.28-0.32	Non-comply
	Permethrin	2.4	2.04-2.76	1.33	0.96	0.06	1.27-1.39	Non-comply
38247-32	Esbiothrin	2	1.7-2.3	1.94	0.92	0.08	1.86-2.02	Comply
	Permethrin	2.5	2.13-2.88	2.41	0.92	0.08	2.33-2.49	Comply
38247-34	Esbiothrin	3	2.55-3.45	<0.1	-	-	-	Non-comply
38247-35	Esbiothrin	0.4	0.34-0.46	0.31	0.93	0.03	0.28-0.34	Comply
	Permethrin	2.4	2.04-2.76	1.55	2.04	0.12	1.43-1.67	Non-comply
38247-36	Esbiothrin	2	1.7-2.3	1.85	1.15	0.09	1.77-1.94	Comply
	Permethrin	2.5	2.13-2.88	2.35	0.55	0.08	2.28-2.43	Comply

**TABLE 4**  
ANALYSIS RESULTS OF INSECTICIDES (BIOCIDES)

### 2.1.6 Conclusion

The measured content of Esbiothrin and Permethrin does not comply with the declared content for 7 products and is not in compliance with applicable Danish law, because of the lack of overlap between the range of analysis results and tolerance.

The measured content of Esbiothrin and Permethrin complies with the declared content for 4 products with applicable Danish law, because of the overlap between the range of analysis results and tolerance.

The measured content of Cyfluthrin complies with the declared content for 1 product with applicable Danish law, because of the overlap between the range of analysis results and tolerance.

# 3. Pesticides

In this report pesticides are defined as plant protection products (PPP).

In the 2014 Danish Pesticide Control Campaign, 20 pesticide products were selected: herbicides, molluscicides and fungicides. The active substances in the selected pesticide products were 2,4-D, 2,4-D ethyl hexyl, Fenoxaprop-P-ethyl, Maleic hydrazide, Clopyralid, Picloram and Ferric phosphate.

## 3.1 Herbicides (Pesticides)

Herbicides containing 2,4-D ethylhexyl, Fenoxaprop-P-ethyl, Maleic hydrazide, Clopyralid and Picloram as active substances were selected for the control campaign for 2014.

### 3.1.1 2,4-D and 2,4-D ethylhexyl

2,4-D and 2,4-D ethylhexyl are herbicides used in Denmark for weed control in certain types of cereal. Chemically, they belong to the phenoxy acetic acids and are growth inhibitors. 2,4-D and 2,4-D ethylhexyl are synthetic auxins, i.e., they mimic plant hormones that regulate growth. Products with 2,4-D and 2,4-D ethylhexyl have not previously been selected by the Danish EPA for control of the content of active substances.

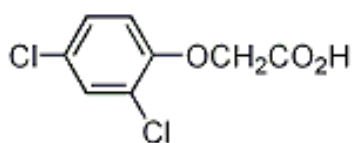


FIGURE 4 MOLECULAR STRUCTURE OF 2,4-D<sup>13</sup>, CAS NO. 94-75-7

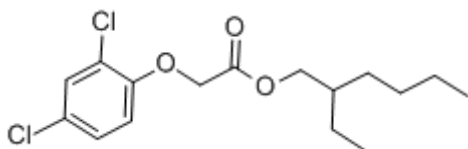


FIGURE 5 MOLECULAR STRUCTURE OF 2,4-D EHTYL HEXYL<sup>13</sup>, CAS NO. 1928-43-4

### 3.1.2 Fenoxaprop-P-ethyl

Fenoxaprop-P-ethyl is a herbicide used in Denmark for control of common wild oat, black-grass and loose silky-bent grass in crop. Chemically it belongs to the group of aryloxyphenoxypropionates and is a fatty acid synthesis inhibitor. Fenoxaprop-P-ethyl inhibits fatty acid synthesis by inhibition of acetyl CoA carboxylase (ACCase).<sup>9+10</sup> Products with Fenoxaprop-P-ethyl were previously selected in 2008 by the Danish EPA for control of content of active substances.



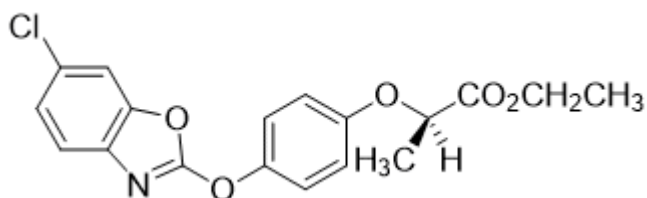


FIGURE 6 MOLECULAR STRUCTURE OF FENOXAPROP-P-ETHYL<sup>10</sup>, CAS NO. 71283-80-2

### 3.1.3 Maleic hydrazide

Maleic hydrazide is a plant growth regulator used in Denmark for weed control and sprout suppression. Chemically it belongs to the pyridazines and is a plant growth regulator. Maleic hydrazide inhibits plant growth by inhibiting cell division in the meristematic regions of the plant.<sup>9+10</sup> Products with maleic hydrazide have not previously been selected by the Danish EPA for control of the content of active substances.

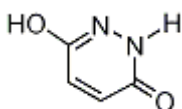


FIGURE 7 MOLECULAR STRUCTURE OF MALEIC HYDRAZIDE<sup>10</sup>, CAS NO. 123-33-1

### 3.1.4 Clopyralid

Clopyralid is a herbicide used in Denmark for weed control in crop. Chemically it belongs to the pyridinecarboxylic acids and is a synthetic auxin (acting like indolylacetic acid). Clopyralid exhibits an auxin-type reaction and regulates growth by acting on cell elongation and respiration.<sup>9+10</sup> Products with Clopyralid were previously selected in 2009 by the Danish EPA for control of content of active substances.

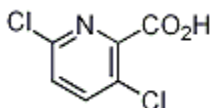


FIGURE 8 MOLECULAR STRUCTURE OF CLOPYRALID<sup>10</sup>, CAS NO. 1702-17-6

### 3.1.5 Picloram

Picloram is a herbicide used in Denmark for weed control in crop. Chemically it belongs to the pyridinecarboxylic acids and is a synthetic auxin (acting like indolylacetic acid). Picloram exhibits an auxin-type reaction and regulates plant growth by accumulating and acting on new growth.<sup>9+10</sup> Products with Picloram have not previously been selected by the Danish EPA for control of the content of active substances.

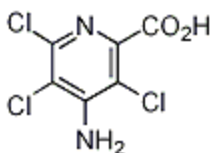


FIGURE 9 MOLECULAR STRUCTURE OF PICLORAM<sup>10</sup>, CAS NO. 1918-02-1

### 3.1.6 Analysis

2,4-D and 2,4-D ethylhexyl was analysed by reversed phase high-performance liquid chromatography combined with UV detection, HPLC-UV. All samples were dissolved in an acetonitrile and ultrasonicated before the analysis. The method is a modified HPLC method according to AOAC-CIPAC 1983, 2,4-D.

Fenoxaprop-P-ethyl was analysed by normal phase liquid chromatography combined with diode array detection (HPLC-DAD). Before analysis, samples were dissolved by ultrasonication in tetrahydrofuran containing trimethylamine. The method is confidential.

Maleic hydrazide was analysed by reverse phase liquid chromatography combined with diode array detection (HPLC-DAD). Samples were dissolved by ultrasonication in a 0.1M NaOH-solution before analysis. The method is a modified HPLC method according to HPLC application ID no. 20035, Phenomenex and confidential method.<sup>14</sup>

Clopyralid and Picloram were analysed by reverse phase liquid chromatography combined with diode array detection (HPLC-DAD). Before analysis, samples were dissolved and diluted in acetonitrile and water and treated with ultrasonication. The method is a modified HPLC method according to CIPAC-method no. 174.<sup>11</sup>

Details concerning the implementation of the analyses and calculation of the result and expanded analysis uncertainty are described in chapter 1.4.

The methods for Maleic hydrazide, Clopyralid and Picloram are accredited.

### 3.1.7 Results

The results from the performed measurements and the corresponding tolerance intervals are listed in the following tables. % RSD is the percentage relative reproducibility of 8 determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

DTI sample no.	Active substance	Label claim g/kg	Tolerance interval	Analysis result g/kg	RSD %	$U_E$ g/kg	Analysis interval	Comply / Non-Comply
38247-17	Maleic hydrazide	600	575-625	618	0.62	2.6	602-634	Comply

TABLE 5  
ANALYSIS RESULTS OF HERBICIDES (PESTICIDES)

DTI sample no.	Active substance	Label claim g/L	Tolerance interval	Analysis result g/L	RSD %	$U_E$ g/L	Analysis interval	Comply / Non-Comply
38247-6	2,4-D ethylhexyl	271.5	258-285	251	3.7	48	203-299	Comply
38247-18	2,4-D ethylhexyl	452.49	430-475	442	14	150	292-592	Comply
38247-24	2,4-D	0.7	0.59-0.81	0.57	5.4	0.14	0.43-0.71	Comply
38247-1	Fenoxaprop-P-ethyl	69	62.1-75.9	69.9	1.7	2.5	67.4-72.4	Comply

DTI sample no.	Active substance	Label claim g/L	Tolerance interval	Analysis result g/L	RSD %	U <sub>E</sub> g/L	Analysis interval	Comply / Non-Comply
38247-7	Fenoxaprop-P-ethyl	69	62.1-75.9	67.7	0.7	1.8	65.9-69.5	Comply
38247-15	Fenoxaprop-P-ethyl	69	62.1-75.9	71.9	0.8	1.7	70.2-72.6	Comply
38247-19	Maleic hydrazide	5	4.25-5.75	6.17	0.83	0.39	5.78-6.56	Non-comply
38247-21	Maleic hydrazide	30	27-33	29.0	0.76	0.64	28.4-29.6	Comply
38247-5	Clopyralid	267	240-294	268	1.44	11.0	257-279	Comply
	Picloram	67	63.7-70.4	68.4	2.07	3.42	65.0-71.8	Comply

TABLE 6  
ANALYSIS RESULTS OF HERBICIDES (PESTICIDES)

### 3.1.8 Conclusion

The measured content of 2,4-D, 2,4-D ethylhexyl, Fenoxaprop P-ethyl, Clopyralid and Picloram complies with the declared content with applicable Danish law, because of the overlap between the range of analysis results and tolerance.

The measured content of Maleic hydrazide complies with the declared content for 2 products with applicable Danish law, because of the overlap between the range of analysis results and tolerance.

The measured content of Maleic hydrazide does not comply with the declared content for 1 product with applicable Danish law, because of the lack of the overlap between the range of analysis results and tolerance.

### 3.2 Molluscicides (Pesticides)

Molluscicides containing Iron(III) phosphate as active substances were selected for the control campaign for 2014.

#### 3.2.1 Iron (III) phosphate (Ferric phosphate)

Iron (III) phosphate is a molluscicide used in Denmark to control slugs and snails. It is an inorganic salt and inhibits feeding. Iron (III) phosphate interferes with the calcium metabolism in the gut of the slug causing it to stop feeding.<sup>9+10</sup> Products containing Iron (III) phosphate have not previously been selected by the Danish EPA for control of the content of active substances.

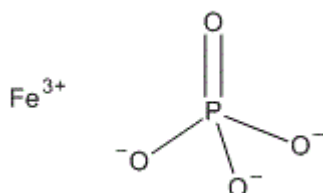


FIGURE 10 MOLECULAR STRUCTURE OF IRON (III) PHOSPHATE<sup>10</sup>, CAS NO. 10045-86-0

#### 3.2.2 Analysis

Iron(III)phosphate was analysed by inductively coupled plasma atomic emission spectrometry (ICP-AES). Before analysis, samples were dissolved with diluted in 2.8 M nitric acid and digested in microwave oven. The method is a modified EPA Method 3050.<sup>15</sup>

### 3.2.3 Results

The results from the performed measurements and the corresponding tolerance intervals are listed in the following tables. % RSD is the percentage relative reproducibility of 8 determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

DTI sample no.	Active substance	Label claim g/kg	Tolerance interval	Analysis result g/kg	RSD %	$U_E$ g/kg	Analysis interval	Comply / Non-Comply
38247-4	Iron(III) phosphate	10	8.5-11.5	11	0.005	1	10-12	Comply
38247-9	Iron(III) phosphate	30	27-33	26.8	1.2	0.88	25.9-27.7	Comply
38247-11	Iron(III) phosphate	16.2	12.2-20.3	18	0.02	1.8	16.2-19.8	Comply
38247-31	Iron(III) phosphate	9.9	8.4-11.4	10	2.5	0.1	9.9-10.1	Comply
38247-37	Iron(III) phosphate	9.9	8.4-11.4	9.0	4.1	1.1	7.9-10.1	Comply

TABLE 7  
ANALYSIS RESULTS OF MOLLUSCICIDES (PESTICIDES)

### 3.2.4 Conclusion

The measured content of Iron(III)phosphate complies with the declared content for all 5 products with applicable Danish law, because of the overlap between the range of analysis results and tolerance.

## 3.3 Fungicides (Pesticides)

Fungicides containing Sulphur, Metconazole, Pyrimethanil, Difeconazole and Epoxiconazole as active substances were selected for the Danish Pesticide Control Campaign in 2014.

### 3.3.1 Sulphur

Sulphur (CAS no. 7704-34-9) is a fungicide used in Denmark to control fungal diseases in pome fruit, beets and ornamental plants. Chemically, it is an inorganic compound and is a non-specific thiol reactant that inhibits respiration. Sulphur is a non-systemic protective fungicide with contact and vapour action when reduced to hydrogen sulphide, which is the active form. Additionally, it has acaricidal activity.<sup>9+10</sup> Products with sulphur have not previously been selected by the Danish EPA for control of content of active substances.

### 3.3.2 Metconazole

Metconazole is a fungicide used in Denmark to control fungal diseases in crop, e.g. wheat, barley and rye. Chemically, it belongs to the group of triazoles. Metconazole is a systemic fungicide that acts by inhibiting ergosterol biosynthesis. Metconazole is applied post emergence and exhibits penetrant, local and acropetal systemicity and inhibits ergosterol synthesis by preventing sterole demethylation.<sup>9+10</sup> Products containing Metconazole have not previously been selected by the Danish EPA for control of content of active substances.

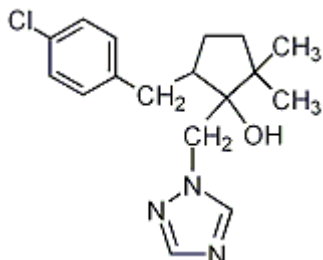


FIGURE 11 MOLECULAR STRUCTURE OF METCONAZOLE<sup>10</sup>, CAS. NO. 125116-23-6

### 3.3.3 Pyrimethanil

Pyrimethanil is a fungicide used in Denmark to control fungal diseases in apples, pears and strawberries. Chemically it belongs to the anilinopyrimidines. Pyrimethanil has been proposed to be an inhibitor of methionine biosynthesis, thus leading to inhibition of the secretion of enzymes necessary for infection. Pyrimethanil is a contact fungicide with translaminar activity and provides both protective and curative effect.<sup>9+10</sup> Products containing Pyrimethanil have not previously been selected by the Danish EPA for control of content of active substances.

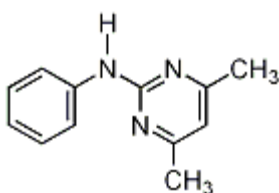


FIGURE 12 MOLECULAR STRUCTURE OF PYRIMETHANIL<sup>10</sup>, CAS. NO. 53112-28-0

### 3.3.4 Difenoconazole

Difenoconazole is a fungicide used in Denmark for control of fungal diseases in cereals and vegetables, e.g. asparagus, broccoli, carrots, potatoes and lettuce. Chemically it belongs to the triazoles and is a sterol demethylation inhibitor. Difenoconazole is a systemic fungicide with preventive and curative actions. It acts by inhibiting cell membrane ergosterol biosynthesis, thereby stopping development of the fungus.<sup>9+10</sup> Products containing Difenoconazole have not previously been selected by the Danish EPA for control of content of active substances.

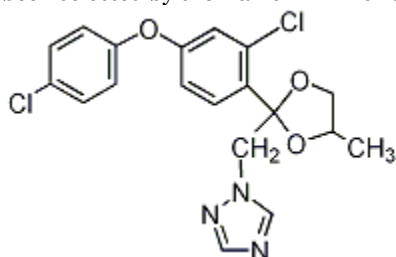


FIGURE 13 MOLECULAR STRUCTURE OF DIFENOCONAZOLE<sup>10</sup>, CAS. NO. 119446-68-3

### 3.3.5 Epoxiconazole

Epoxiconazole is a fungicide used in Denmark for control of fungal diseases in wheat, barley, rye and beets. Chemically it belongs to the triazoles. Epoxiconazole has preventive and curative properties and acts by inhibiting C-14-demethylase in sterol biosynthesis.<sup>9+10</sup> Products with Epoxiconazole were previously selected in 2007 by the Danish EPA for control of content of active substances.

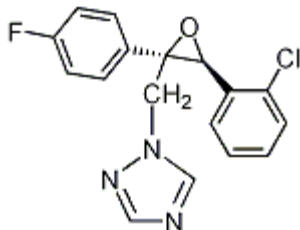


FIGURE 14 MOLECULAR STRUCTURE OF EPOXICONAZOLE<sup>10</sup>, CAS. NO. 133855-98-8

### 3.3.6 Analysis

Sulphur was analysed by iodometry. The sulfur was converted to sodium thiosulfate by reflux with sodium sulfite. The amount to thiosulfate was determined by titration with a standardized iodide-solution. The results were corrected for the presence of thiosulfate and other sulphur compounds because only free sulphur should be determined. The method is according to CIPAC method Sulphur 18, 1967.<sup>11</sup>

Metaconazole, Epoxiconazole and Difenoconazole were analysed by reversed phase high-performance liquid chromatography combined with diode array detection, HPLC-DAD. All samples were dissolved in acetonitrile and ultrasonicated before the analysis. The method is confidential.

Pyrimethanil was analysed by reversed phase high-performance liquid chromatography combined with diode array detection, HPLC-DAD. All samples were dissolved in acetonitrile and ultrasonicated before the analysis. The method is a modified HPLC method according to CIPAC-method pendimethalin 357.<sup>11</sup>

Details concerning the implementation of the analyses and calculation of the result and expanded analysis uncertainty are described in chapter 1.4.

The methods for Sulphur, Metaconazole, Epoxiconazole, Difenoconazole and Pyrimethalin are accredited.

### 3.3.7 Results

The results from the performed measurements and the corresponding tolerance intervals are listed in the following tables. % RSD is the percentage relative reproducibility of 8 determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

DTI sample no.	Active substance	Label claim g/kg	Tolerance interval	Analysis result g/kg	RSD %	$U_E$ g/kg	Analysis interval	Comply / Non-Comply
38247-10	Sulphur	800	775-825	808	0.71	14	794-822	Comply

TABLE 8 ANALYSIS RESULTS OF FUNGICIDES (PESTICIDES)

DTI sample no.	Active substance	Label claim g/L	Tolerance interval	Analysis result g/L	RSD %	U <sub>E</sub> g/L	Analysis interval	Comply / Non-Comply
38247-8	Metconazole	90	81-99	90.7	1.36	3.00	87.7-93.7	Comply
38247-14	Pyrimethanil	400	380-420	399	3.29	39.8	359-439	Comply
38247-16	Difenoconazole	125	117.5-132.5	126	3.1	9.5	116.5-135.5	Comply
38247-23	Metconazole	27.5	24.8-30.3	28.9	3.24	2.41	26.5-31.3	Comply
	Epoxiconazole	37.5	33.8-41.3	39.2	1.78	1.67	37.6-40.9	Comply

**TABLE 9**  
ANALYSIS RESULTS OF FUNGICIDES (PESTICIDES)

### 3.3.8 Conclusion

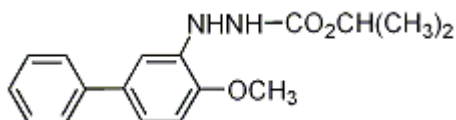
The measured content of Sulphur, Metconazole, Pyrimethanil, Difeoconazole, and Epoxiconazole complies with the declared content with applicable Danish law, because of the overlap between the range of analysis results and tolerance.

## 3.4 Insecticides (Pesticides)

An insecticides containing Bifenazate as active substance was selected for the 2014 control campaign.

### 3.4.1 Bifenazate

Bifenazate is an insecticide used in Denmark to control insect pests in, e.g. ornamental plants tomatoes, cucumbers and strawberries. Bifenazate is a non-systemic acaricide with predominantly contact action and long residual action.<sup>9+10</sup> Products containing Bifenazate have not previously been selected by the Danish EPA for control of content of active substances.



**FIGURE 15** MOLECULAR STRUCTURE OF BIFENAZATE<sup>10</sup>, CAS NO. 149877-41-8

### 3.4.2 Analysis methods

Bifenazate was analysed by reversed phase high-performance liquid chromatography combined with diode array detection, HPLC-DAD. All samples were dissolved in acetonitrile and ultrasonicated before the analysis. The method is a modified HPLC method according to CIPAC-method pendimethalin 357.<sup>11</sup>

Details concerning the implementation of the analyses and calculation of the result and expanded analysis uncertainty are described in chapter 1.4.

The method for Bifenazate is accredited.

### 3.4.3 Results

The results from the performed measurements and the corresponding tolerance intervals are listed in the following tables. % RSD is the percentage relative reproducibility of 8 determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

DTI sample no.	Active substance	Label claim g/L	Tolerance interval	Analysis result g/L	RSD %	$U_E$ g/L	Analysis interval	Comply / Non-Comply
38247-29	Bifenazate	240	226-254	240	2.92	15.8	224-256	Comply

**TABLE 10**  
ANALYSIS RESULTS OF INSECTICIDES (PESTICIDES), CONTENT IN G/L

### 3.4.4 Conclusion

The measured content of Bifenazate complies with the declared content with applicable Danish law, because of the overlap between the range of analysis results and tolerance.



# 4. Screenings

Products under a parallel trade permit are compared by gas chromatography with mass spectrometric detection (GC-MS) and Fourier transform infrared spectroscopy (FTIR) to investigate if they contain the same solvents and additives as the original product.

## 4.1 Regulations in Denmark

The requirements for parallel trade permits are described in detail in Article 52 of Regulation No. 1107/2009.<sup>16, 17</sup>

### 4.1.1 Parallel trade permits

A parallel trade permit is an authorisation for the import of a plant protection product that is identical to a product already authorised in Denmark. A parallel trade permit is valid only for Denmark. It is not valid in the rest of the North zone. An application must be made in each country for which you wish to obtain the permit.

A plant protection product under a parallel trade permit may only be placed on the market and used in accordance with the authorisation of the original product. The parallel trade permit will expire at the same time as the authorisation of the original product. If the authorisation of the original product is withdrawn for reasons other than safety reasons, the parallel trade permit will be similarly affected.

### 4.1.2 Identical products

A plant protection product is identical with a product already authorised in Denmark only if:

- a) It is produced by the same company, or an associated company or under licence according to the same method of manufacture as that of the authorised product;
- b) It has the same specification, that is, contains the same active substance, safeners and synergists. The plant protection product is of the same formulation type; and the contents of the co-formulants and the packaging are the same or equivalent.

The packaging and co-formulants may not have more negative effects on health or the environment than the original product.

## 4.2 Analysed products

The analysed products are stated in the following tables.

Parallel products laboratory no.	Product name	Reg. no.
38247-22	Universal Insekt spray	49-112
38247-32	Cur EraFormica myremiddel	49-114
38247-38	Cur-Era Bugs Insektmiddel	49-113

**TABLE 11**  
COMPARISON OF PARALLEL PRODUCTS CONTAINING PERMETHRIN AND ESBIOTHRIN. ORIGINAL PRODUCT REG. NO. 49-98 NOT FOUND ON THE DANISH MARKET.

Parallel products laboratory no.	Product name	Reg. no.	Original product laboratory no.	Product name	Reg. no.
38247-1	Fenoxaprop	650-49	38247-7	Foxtrot	11-31

**TABLE 12**  
COMPARISON OF ORIGINAL PRODUCTS WITH PARALLEL PRODUCTS CONTAINING FENOXAPROP-P-ETHYL.

Parallel products laboratory no.	Product name	Reg. no.	Original product laboratory no.	Product name	Reg. no.
38247-12	Lotus Insekt Xtra against ants and other vermin	14-273			
38247-13	Lotus ant spray Xtra	14-274	38247-35	Bonus Myremiddel	14-268
38247-26	Ant spray Xtra	14-272			
38247-27	RTU Xtra	14-271			
38247-30	Material Shop Ant spary Xtra	14-277			
38247-20	Trinol no. 810 myremiddel	14-283	38247-27	RTU Xtra	14-271

**TABLE 13**  
COMPARISON OF ORIGINAL PRODUCT WITH PARALLEL PRODUCTS CONTAINING PERMETHRIN AND ESBIOTHRIN.

#### 4.2.1 Analysis Methods

##### 4.2.1.1 GC-MS

A subsample (app. 5 g) was weighed in a 50 ml calibrated flask and acetone was added up to the label. Then 1 ml was drawn and 1 ml dichloromethane was added. The analysis was carried out by gas chromatography with mass spectrometric detection (GC-MS).

##### 4.2.1.2 FTIR

A drop of the product was placed on an ARO object glass. The drop dried up at room temperature before the measurement by FTIR. Fourier transform infrared spectroscopy (FTIR) was carried out on a Renishaw InVia Streamline microscope attached to FTIR, IlluminatIR II from Smiths Detection. The spectra were taken with ARO (All Reflective Objective) with 64 repetitions and a resolution of 8 cm<sup>-1</sup>.

#### 4.2.2 Conclusion

No discrepancies were proven between the content in the original products and the parallel products with a starting point in a comparison between the FTIR spectra and the GC-MS chromatogrammes.

In connection with the products that contained Esbiothrin and Permethrin, the GC-MS screening of the parallel products reg. no. 14-271 and reg. no. 14-277 detected three components that seem to have a different concentration than the original product reg. no. 14-268. A quantitative analysis was not carried out and the identification of the components was only carried out by comparison with the NIST GC-MS library. Further analyses have to be carried out if the components are to be verified and compared further.

# References

1. Statutory Order on Pesticides. Statutory Order from the Ministry of the Environment, No. 151 of 18. February 2014 incl. revisions, Statutory Order is in Danish.
2. REGULATION (EC) No. 1107/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC.
3. The regulation of the European Commission (EU) No. 546/2011 of 10 June 2011 concerning the implementation of the regulation of the European Parliament and the European Council (EF) No. 1107/2009 concerning uniform principles for evaluation and approval of crop protection agents.
4. Danish Ministry of the Environment, Environmental Protection Agency (Danish EPA) site; <http://www.mst.dk/English/Pesticides/>.
5. Danish EPA, 2009. List of approved Pesticides 2009, the Danish Environmental Protection Agency. The list is in Danish; [http://www.mst.dk/Virksomhed\\_og\\_myndighed/Bekaempelsesmidler/Find+godkendt+bek%C3%A6mpelsesmiddel/](http://www.mst.dk/Virksomhed_og_myndighed/Bekaempelsesmidler/Find+godkendt+bek%C3%A6mpelsesmiddel/).
6. Danish EPA, legislation relating to pesticides, document is in Danish; [http://www.mst.dk/Virksomhed\\_og\\_myndighed/Bekaempelsesmidler/Pesticider/Lovgivning\\_bekaempelsesmidler.htm](http://www.mst.dk/Virksomhed_og_myndighed/Bekaempelsesmidler/Pesticider/Lovgivning_bekaempelsesmidler.htm).
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8. The Danish Accreditation and Metrology Fund – DANAK, <http://english.danak.dk/>.
9. Tomlin, C.D.S (2009): The Pesticide Manual, fifteenth Edition. British Crop Protection Council, United Kingdom.
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13. [www.chemicalbook.com](http://www.chemicalbook.com)
14. HPLC application ID no. 20035, Phenomenex
15. EPA Method 3050
16. The requirements for parallel trade permits, Article 52 of Regulation No 1107/2009
17. <http://mst.dk/82449>

**Pesticide and biocide samples collected on the Danish market for authority control in 2014**

DTI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
38247-28	Cyfluthrin	18-333	Solfac 50EW	Bayer A/S, Bayer CropScience
38247-12	Esbiothrin & Permethrin	14-273	Lotus Insekt Xtra mod myrer og andre kryb	AgroDan ApS, c/o United Phosphorus
38247-13	Esbiothrin & Permethrin	14-274	Lotus Myrespray Xtra	AgroDan ApS, c/o United Phosphorus
38247-20	Esbiothrin & Permethrin	14-283	Trinol nr. 810 myremiddel	AgroDan ApS, c/o United Phosphorus
38247-22	Esbiothrin & Permethrin	49-112	Universal Insekt spray, Bayer Garden	Klarsø A/S
38247-26	Esbiothrin & Permethrin	14-272	Myrespray Xtra, Tanaco	AgroDan ApS, c/o United Phosphorus
38247-27	Esbiothrin & Permethrin	14-271	RTU Xtra, Tanaco	AgroDan ApS, c/o United Phosphorus
38247-30	Esbiothrin & Permethrin	14-277	Material Shop Myrespray Xtra	AgroDan ApS, c/o United Phosphorus
38247-32	Esbiothrin & Permethrin	49-114	Cur EraFormica myremiddel, NelsonGarden	Klarsø A/S
38247-34	Esbiothrin	49-101	nr.810 Myrespray, Trinol	Klarsø A/S
38247-35	Esbiothrin & Permethrin	14-268	Bonus Myremiddel, Fausol	AgroDan
38247-36	Esbiothrin & Permethrin	49-113	Cur+Era Bugs Insektmiddel, NelsonGarden	Klarsø A/S

**TABLE 14**  
COLLECTED INSECTICIDES (BIOCIDES)

DTI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
38247-6	2,4-D ethylhexyl	64-70	MUSTANG Forte	Dow AgroSciences Danmark A/S
38247-18	2,4-D ethylhexyl	64-67	Catch	Dow AgroSciences Danmark A/S
38247-24	2,4-D	361-23	Tanaco Plænerens Klar til brug	Agriphar S.A.
38247-1	Fenoxaprop-P-ethyl	650-94 P-11-31	Fenoxaprop	Agros Aps
38247-7	Fenoxaprop-P-ethyl	11-31	Foxtrot	Cheminova A/S
38247-15	Fenoxaprop-P-ethyl	18-432	Primera Super	Bayer CorpScience
38247-17	Maleic hydrazide	558-4	Antergon MH	Chemtura Europe Ltd
38247-19	Maleic hydrazide	364-21	Finalsan Ultima Ukrudtsbekæmp else klar-til-brug	W. Neudorff GmbH KG
38247-21	Maleic hydrazide	364-20	Finalsan Ultima Ukrudtsbekæmp else Koncentrat	W. Neudorff GmbH KG
38247-5	Clopyralid and Picloram	64-71	Galera, Bayer Garden	Dow AgroSciences Danmark A/S

**TABLE 15**  
COLLECTED HERBICIDES (PESTICIDES)

DTI	Active substances	Reg. no.	Name of product	Authorisation holder
38247-4	Iron(III) phosphate	364-8	SnegleFri Ferramol	W. Neudorff GmbH KG
38247-9	Iron(III) phosphate	364-59	Sluxx HP	W. Neudorff GmbH KG
38247-11	Iron(III) phosphate	18-518	SmartBayt	Bayer CropScience
38247-31	Iron(III) phosphate	386-15	Bonus Sneglefri	Fausol A/S
38247-37	Iron(III) phosphate	650-18	Agros Sneglegift	Agros Aps

**TABLE 16**  
COLLECTED MOLLUSCICIDES (PESTICIDES)

TI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
38247-8	Metconazole	19-137	Juventus 90, Juventus	BASF A/S
38247-10	Sulphur	19-34	Kumulus S, ECOstyle	BASF
38247-14	Pyrimethanil	19-159	Scala	BASF A/S
38247-16	Difenoconazole and Azoxystrobin	1-203	Ortiva Top	Syngenta Nordics A/S
38247-23	Metconazole and Epoxiconazole	19-182	Osiris	BASF A/S

**TABLE 17**  
COLLECTED FUNGICIDES (PESTICIDES)

TI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
38247-29	Bifenazate	558-5	Floramite 240 SC	Chemtura Europe Ltd

**TABLE 88**  
COLLECTED INSECTICIDES (PESTICIDES)



## **Control of Pesticides and Biocides 2014**

The analytical chemical authority control of pesticide and biocide products on the Danish market that was carried out in 2014 by the Danish Environmental Protection Agency (Danish EPA), Chemical Inspection Service, is described in this report. Samples of selected types of pesticides and biocides were collected on the Danish market and analysed to verify whether or not the content of the active substances in the products in question complied with the product specification and the labelled content. A total of 32 different pesticide products were analysed. The content of active substance in 8 of the analysed products was not within the accepted tolerance that is determined in the Danish Statutory Order on pesticides.

Den analytisk kemiske kontrol af biocid- og pesticidprodukter på det danske marked, der er udført i 2014 af den danske Miljøstyrelses Kemikalieinspektion, er beskrevet i denne rapport. Prøver fra udvalgte typer af bekæmpelsesmidler er blevet indsamlet og analyseret for at verificere, om indholdet af de respektive aktivstoffer er i overensstemmelse med det deklarerede indhold. Der blev undersøgt i alt 32 bekæmpelsesmidler. Indholdet af aktivstoffet i 8 af de analyserede produkter var ikke inden for den accepterede tolerance, der er fastsat i bekendtgørelsen om bekæmpelsesmidler



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