

**Ministry of Environment and Food of Denmark** Environmental Protection Agency

Control of Pesticides and Biocides 2016 The Analytical Chemical Control of Pesticides and Biocides on the Danish Market

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## Preface

35 different types of products, covered by the pesticide and biocide regulation, were included in the Danish authority control in 2016. 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.

One parallel product and the related original product were screened by GC-MS and FTIR to investigate if there were any differences.

For the 2016 programme, 1 tested product (1 pesticide) 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>

# Sammenfatning og konklusion

Denne rapport beskriver den analytisk kemiske kontrol af biocid- og pesticidprodukter på det danske marked, der er udført af Miljøstyrelsens Kemikalieinspektion i 2016.<sup>4,5,6</sup>

35 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.

35 forskellige produkter er inkluderet i den analytisk kemiske kontrol, der blev udført af myndighederne i 2016:

- 5 insekticider (biocider) indeholdende diflubenzuron og cypermethrin
- 5 insekticider (pesticider) indeholdende alpha-cypermethrin, cypermethrin, lambdacyhalothrin og tau-fluvalinate
- 21 herbicider indeholdende glyphosate, diflufenican, fluroxypyr, clopyralid, MCPA, florasulam, bromoxynil, aclonifen, clomazon og prosulfocarb
- 1 fungicid indeholdende dimethomorph ٠
- 3 vækstreguleringsmidler indeholdende chlormequat-chlorid. ٠

Aktivstof	Antal analyserede produkter	Antal indenfor tolerance	Antal udenfor tolerance
Diflubenzuron	1	1	0
Cypermethrin	5	4	1
Alpha-cypermethrin	1	1	0
Lambda-cyhalothrin	2	2	0
Tau-fluvalinat	1	1	0
Glyphosat	10	10	0
Glyphosat & Diflufenican	1	1	0
Fluroxypyr	1	1	0
Fluroxypyr, MCPA & Clopyaralid	1	1	0
Fluroxypyr & Florasulam	2	2	0
Bromoxynil	1	*	0
Aclonifen	1	1	0
Aclonifen & Clomazon	1	1	0
Prosulfocarb	3	3	0
Dimethomorph	1	1	0
Chlormequat-chlorid	3	3	0
Total	35	33	1

#### Tabel 1

\* Udløbsdatoen på produktet var overskredet ved afslutningen af analyserne.

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. 1750 af 14. december 2015 og i forordning 1107/2009 om markedsføring af plantebeskyttelsesmidler.<sup>1,2,3</sup>

Det målte indhold af aktivstoffer er ikke i overensstemmelse med det deklarerede indhold i 1 produkt, og overholder således ikke gældende dansk lovgivning, idet der ikke er overlap mellem interval for analyseresultat og tolerance.

Det målte indhold af aktivstoffer er i overensstemmelse med det deklarerede indhold i 34 produkter, og overholder således gældende dansk lovgivning, idet der er overlap mellem interval for analyseresultat og tolerance.

Densitet blev målt på alle flydende produkter. Vandindhold og pH blev målt på produkter, hvor der forligger en specificeret værdi til sammenligning.

Der blev foretaget screeninger af to parallelprodukter og de tilhørende originalprodukter for at undersøge, om der var forskel på indhold af urenheder eller andre formuleringsingredienser. Desuden blev der foretaget en sammenlignende screening af fire parallelprodukter, hvor originalproduktet ikke var tilgængeligt.

# Summary and Conclusion

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 2016.<sup>4,5,6</sup>

35 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 complies with the product specification and the declared content.

35 different products are covered by the analytical chemical control that was carried out by the authorities in 2016:

- 5 insecticides (biocides) containing diflubenzuron and cypermethrin
- 5 insecticides (pesticides) containing alpha-cypermethrin, cypermethrin, lambdacyhalothrin and tau-fluvalinate
- 21 herbicides containing glyphosate, diflufenican, fluroxypyr, clopyralid, MCPA, florasulam, bromoxynil, aclonifen, clomazone and prosulfocarb
- 1 fungicide containing dimethomorph
- 3 growth regulators containing chlormequat-chloride

#### Table 2

Outline of total number of analysed products and conclusion.

Active substance	Number of analysed products	Number of products comply	Number of products non-comply
Diflubenzuron	1	1	0
Cypermethrin	5	4	1
Alpha-cypermethrin	1	1	0
Lambda-cyhalothrin	2	2	0
Tau-fluvalinate	1	1	0
Glyphosate	10	10	0
Glyphosate & Diflufenican	1	1	0
Fluroxypyr	1	1	0
Fluroxypyr, MCPA & Clopyaralid	1	1	0
Fluroxypyr & Florasulam	2	2	0
Bromoxynil	1	*	0
Aclonifen	1	1	0
Aclonifen & Clomazone	1	1	0
Prosulfocarb	3	3	0
Dimethomorph	1	1	0
Chlormequat-chloride	3	3	0
Total	35	33	1

\* On the conclusion of the analyses, the expiration date of the product had been exceeded.

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. 1750 of 14 December 2015, and in Regulation 1107/2009 concerning Marketing of Plant Protection Products.<sup>1,2,3</sup>

The measured content of active substance did not comply with the declared content for 1 product, and therefore it is not in compliance with applicable Danish law, as there is no overlap between the range of analysis results and tolerance.

The measured content of active substances complies with the declared content for 34 products and is in compliance with applicable Danish law, as there is an overlap between the range of analysis results and tolerance.

The density of all liquid products was measured. The water content and pH were determined for products where a specified value was available for comparison.

Screenings were carried out on two parallel products and the corresponding original products to investigate if there was a difference in the content of impurities or other formulation ingredients. A comparative screening was carried out for four parallel products where the original product was not available for comparison.

## 1. Control campaigns in 2016

## 1.1 Selected active substances

The control campaign conducted in 2016 covered 16 active substances in pesticides. Overall, pesticides are divided into two types: plant protection products (PPP) and biocides (non-agricultural pesticides).

A summary of the selected active substances is given in following table:

Туре	Active substance	CAS no.	Area of application	
Disside	Diflubenzuron	35367-38-5	Insecticide	
Biocide	Cypermethrin	52315-07-8	Insecticide	
	Alpha-cypermethrin	67375-30-8	Insecticide	
	Cypermethrin	52315-07-8	Insecticide	
	Lambda-cyhalothrin	91465-08-6	Insecticide	
	Tau-fluvalinate	102851-06-9	Insecticide	
	Glyphosate	1071-83-6	Herbicide	
	Diflufenican	83164-33-4	Herbicide	
	Fluroxypyr	69377-81-7	Herbicide	
	Clopyralid	1702-17-6	Herbicide	
Pesticides	MCPA	94-74-6	Herbicide	
	Florasulam	145701-23-1	Herbicide	
	Bromoxynil	1689-84-5	Herbicide	
	Aclonifen	74070-46-5	Herbicide	
	Clomazon	81777-89-1	Herbicide	
	Prosulfocarb	52888-80-9	Herbicide	
	Dimethomorph	110488-70-5	Fungicide	
	Chlormequat-chloride	999-81-5	Growth regulator	

## 1.2 Collecting products

Product samples of the various pesticide and biocide formulations from different manufacturers, covered in the 2016 control campaigns, were collected by the Chemical Inspection Service of the Danish Environmental Protection Agency during the period from February to April 2016. 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)<sup>7</sup> 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.

## 1.3 Tolerance for active substances

The objective of the Danish EPA was to examine the content of active substances in the products, and to 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. 1750 of 14 December 2015, 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 Table 4.

Table 4

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

16.

#### 1.4 Chemical analysis 2016

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.7

The Laboratory for Chemistry and Microbiology is accredited by DANAK (Danish Accreditation and Metrology Fund), registration no. 90, according DS/EN ISO/IEC 17025.8 The laboratory is accredited for a flexible scope regarding parameters, performance of the method and test methods within parts of the scope.

#### 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. Recovery was determined by adding a known amount of the relevant active substance to each product.

As far as possible, the expanded uncertainty U<sub>E</sub> (k=2) of each product was calculated on the basis of the spread of the results of the analyses and the spread of the three density measurements (only included if the product was liquid). The bias was calculated on the basis of the recovery determination. The expanded analysis uncertainty is used to determine a 95% confidence interval for the analysis result.

The results ± 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 density meter (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 Analysing water content The water content of the formulations was determined by Karl Fisher titration. The water content was determined for products with a specified content of water and analysis was performed in triplicate.

### 1.4.4 Analysing pH

The pH for undiluted samples and for 1% solutions of the formulation was determined by potentiometric titration according to the Institute's analysis method UA-310 *Determination of pH in pesticides*. The method is based on CIPAC MT 75.3. The pH was determined for products with specified pH values and the analysis was performed in triplicate.

### 1.4.5 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 objective of the active substances, contained in the biocidal products, is to protect humans, animals, materials or articles against harmful organisms such as pests or bacteria.

For the Danish 2016 Pesticide Control Campaign, 5 biocide insecticides were selected. The selected active substances were diflubenzuron and cypermethrin.

## 2.1 Insecticides (Biocides)

One insecticide containing diflubenzuron as active substance was selected for the 2016 control campaign. Four insecticides containing cypermethrin were selected.

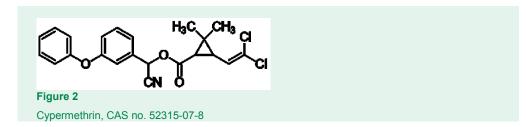
### 2.1.1 Diflubenzuron

Diflubenzuron is an insecticide used in Denmark for pest control, e.g., of fleas on farm mink. Chemically, it belongs to the group of benzoylureas and is a non-systemic insect growth regulator with contact and stomach action. Diflubenzuron acts as a chitin synthesis inhibitor, and interferes with the formation of the insect cuticle.<sup>9,10</sup> Products with diflubenzuron were previously selected in 2001 and 2005 by the Danish EPA for control of content of active substances.

Figure 1 Diflubenzuron, CAS no. 35367-38-5

### 2.1.2 Cypermethrin

Cypermethrin is an insecticide used in Denmark for control of insects in and around residential areas. Chemically, it belongs to the group of pyrethroids and is a non-systemic insecticide with contact and stomach action. Cypermethrin acts on the nervous system of the insect, and disturbs the function of neurons by interaction with the sodium channel.<sup>9,10</sup> Products with cypermethrin were previously selected in 2003 and 2013 by the Danish EPA for control of content of active substances.



#### 2.1.3 Analysis

Diflubenzuron was analysed by reversed phase high-performance liquid chromatography combined with diode array detection (HPLC-DAD). All samples were dissolved in dioxane by ultrasonification before the analysis. The method is a modified HPLC method according to CIPAC diflubenzuron 339.<sup>11</sup> Cypermethrin was analysed by gas chromatography coupled with mass selective detection (GC-MS). All samples were dissolved in a mixture of tetrahydrofuran and methanol with citric acid. The method is a modified GC-FID method according to CIPAC alpha-cypermethrin 454.<sup>11</sup>

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

The method for determination of Diflubenzuron is accredited.

### 2.1.4 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 the determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

#### Table 5

Analysis results of insecticides (biocides), g/kg.

DTI sample no.	Active sub- stance	Label claim g/kg	Toler- ance interval	Analysis result g/kg	RSD %	U <sub>e</sub> g	Analysis interval	Comply/ Non- comply
686013-5	Diflubenzuron	50	40-60	51	2	2.5	49-54	Comply

#### Table 6

Analysis results of insecticides (biocides), g/L.

DTI sample no.	Active sub- stance	Label claim g/L	Tolerance interval	Analysis result g/L	RSD %	U <sub>E</sub> g/L	Analysis interval	Comply/ Non-comply
686013-13	Cypermethrin	0.5	0.43-0.58	0.45	2.5	0.06	0.39-0.51	Comply
686013-16	Cypermethrin	0.5	0.43-0.58	0.46	1.7	0.06	0.40-0.52	Comply
686013-23	Cypermethrin	0.5	0.43-0.58	0.46	5.8	0.12	0.34-0.58	Comply
686013-33	Cypermethrin	0.5	0.43-0.58	0.52	5.2	0.09	0.44-0.61	Comply

### 2.1.5 Conclusion

The measured content of diflubenzuron and cypermethrin complies with the declared content for the 5 products and with applicable Danish law, as there is an overlap between the range of analysis results and tolerance.

## 3. Pesticides

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

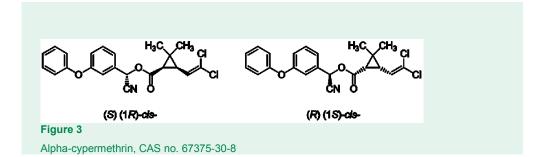
In the 2016 Danish Pesticide Control Campaign, 30 pesticide products were selected: insecticides, herbicides, fungicides and growth regulators. The active substances in the selected pesticide products were alpha-cypermethrin, cypermethrin, lambda-cyhalothrin, tau-fluvalinate, glyphosate, diflufenican, fluroxypyr, clopyralid, MCPA, florasulam, bromoxynil, aclonifen, clomazone, prosulfocarb, dimethomorph and chlormequat-chloride.

#### 3.1 Insecticides (Pesticides)

Five insecticides, one containing alpha-cypermethrin, one containing cypermethrin, two with lambda-cyhalothrin and one with tau-fluvalinate as active substances were selected for the 2016 control campaign

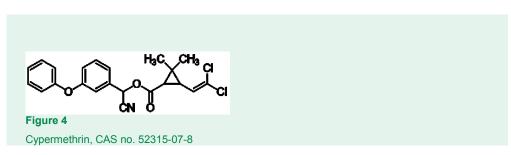
#### 3.1.1 Alpha-cypermethrin

Alpha-cypermethrin is an insecticide used in Denmark for pest control in, e.g., cereal, rapeseed, cabbage, strawberries and tomatoes. Chemically, it belongs to the group of pyrethroids and is a non-systemic insecticide with contact and stomach action. Alpha-cypermethrin acts by preventing transmission of impulses along nerves, brought about by blocking the passage of sodium ions through sodium channels in nerve membranes.<sup>9,10</sup> Products with alpha-cypermethrin were previously selected in 2008 by the Danish EPA for control of content of active substances.



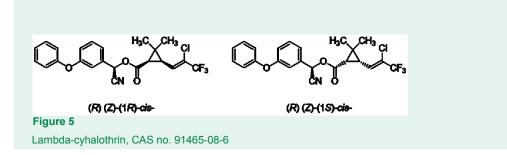
#### 3.1.2 Cypermethrin

Cypermethrin is an insecticide used in Denmark for pest control in, e.g., ornamental plants, potatoes, parsley, cabbage and apples. Chemically, it belongs to the group of pyrethroids and is a non-systemic insecticide with contact and stomach action. Cypermethrin acts on the nervous system of the insect, and disturbs the function of neurons by interaction with the sodium channel.<sup>9,10</sup> Products with cypermethrin were previously selected in 2003 and 2013 by the Danish EPA for control of content of active substances.



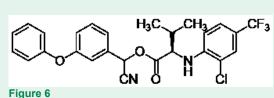
### 3.1.3 Lambda-cyhalothrin

Lambda-cyhalothrin is an insecticide used in Denmark for pest control in, e.g., rapeseed, cereals and potatoes. Chemically, it belongs to the group of pyrethroids and is a non-systemic insecticide with contact and stomach action, and repellent properties. Lambda-cyhalothrin acts on the nervous system of the insect, and disturbs the function of neurons by interaction with the sodium channel.<sup>9,10</sup> Products with lambda-cyhalothrin were previously selected in 2003 by the Danish EPA for control of content of active substances.



### 3.1.4 Tau-fluvalinate

Tau-fluvalinate is an insecticide used in Denmark for pest control in, e.g., cereal, rapeseed, potatoes and peas. Chemically, it belongs to the group of pyrethroids and is an insecticide and acaricide with contact and stomach action. Tau-fluvalinate 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 tau-fluvalinate were previously selected in 1998 by the Danish EPA for control of content of active substances.



Tau-fluvalinate, CAS no. 102851-06-9

## 3.1.5 Analysis

Alpha-cypermethrin was analysed by gas chromatography coupled with mass selective detection (GC-MS). Cypermethrin was analysed by gas chromatography coupled with tandem mass spectrometry (GC-MS/MS). All alpha-cypermethrin and cypermethrin samples were dissolved in a mixture of tetrahydrofuran and methanol with citric acid. Both the alpha-cypermethrin and cypermethrin methods are a modified GC-FID method according to CIPAC alpha-cypermethrin 454.<sup>11</sup>

Lambda-cyhalothrin was analysed by GC-MS. All samples were dissolved in dichlormethane before analysis. The method is a modified GC-FID method according to CIPAC lambda-cyhalothrin 463.<sup>11</sup>

Tau-fluvalinate was analysed by reversed phase high-performance liquid chromatography combined with diode array detection (HPLC-DAD). All samples were dissolved in acetonitrile by ultra-sonification before the analysis.

Details concerning the implementation of the analyses and calculation of the result and expanded analysis uncertainty are described in section 1.4. The method for determination of tau-fluvalinate is accredited.

#### 3.1.6 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 the determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

#### Table 7

Analysis results of insecticides (biocides), g/kg.

DTI sample no.	Active substance	Label claim g/kg	Tolerance interval	Analysis result g/kg	RSD %	U <sub>E</sub> g	Analysis interval	Comply/ Non- comply
686013-11	Lambda-cyhalothrin	25.0	21.3-28.8	24.4	3.4	3.7	20.7-28.1	Comply
686013-21	Lambda-cyhalothrin	50.0	45.0-55.0	49.4	3.2	6.7	42.7-56.1	Comply

#### Table 8

Analysis results of insecticides (biocides), g/L.

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
686013-17	Tau-fluvalinate	240	226-254	227	0.6	31	196-258	Comply
686013-25	Alpha-Cypermethrin	50	45-55	50	3.6	10	40-60	Comply
686013-29	Cypermethrin	0.020	0.017-0.023	0.0007	15	0.0002	0.0004-0.0009	Non- Comply

### 3.1.7 Conclusion

The measured content of cypermethrin does not comply with applicable Danish law as there is no overlap between the range of analysis results and tolerance.

The measured content of lambda-cyhalothrin, tau-fluvalinate, and alpha-cypermethrin complies with the declared content for the 4 products and with applicable Danish law, as there is an overlap between the range of analysis results and tolerance.

### 3.2 Herbicides (Pesticides)

Twenty-one herbicides containing glyphosate, diflufenican, fluroxypyr, clopyralid, MCPA, florasulam, bromoxynil, aclonifen, clomazone and prosulfocarb as active substances were selected for the 2016 control campaign.

#### 3.2.1 Glyphosate

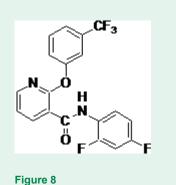
Glyphosate is a herbicide used in Denmark for crop desiccation and weed control. Chemically, it is a glycine derivative and it is a non-selective systemic herbicide, absorbed by the foliage. Glyphosate inhibits 5-enolpyruvylshikimate-3-phosphate synthase (EPSPS), an enzyme of the aromatic acid biosynthetic pathway, and hereby prevents synthesis of essential aromatic amino acids needed for protein biosynthesis.<sup>9,10</sup> Products with glyphosate were previously selected in 2003 and 2011 by the Danish EPA for control of content of active substances.

NHCH-CO-H

Figure 7 Glyphosate, CAS no. 1071-83-6

#### 3.2.2 Diflufenican

Diflufenican is a herbicide used in Denmark for weed control in, e.g., winter wheat, winter rye and triticale. Chemically, it belongs to the group of pyridinecarboxamides and is a selective contact and residual herbicide, absorbed principally by the shoots of germinating seedlings. Diflufenican acts by blocking the carotenoid biosynthesis, by inhibition of phytoene desaturase.<sup>9,10</sup> Products with diflufenican were previously selected in 2007 and 2012 by the Danish EPA for control of content of active substances.



Diflufenican, CAS no. 83164-33-4

### 3.2.3 Fluroxypyr

Fluroxypyr is a herbicide used in Denmark for weed control in cereals, on grass areas and in maize. Chemically, it belongs to the group of pyridinecarboxylic acids and is a synthetic auxin acting like indolylacetic acid. Fluroxypyr acts by inducing auxin-type responses, e.g., leaf curling. Fluroxypyr is applied as an ester. After foliar uptake, the ester is hydrolysed to the parent acid, which is the herbicidally active form, and it is translocated rapidly to other parts of the plants.<sup>9,10</sup> In the 2016 control campaign four products with fluroxypyr formulated as fluroxypyr-meptyl (CAS no. 81406-37-3) were analysed. Products with fluroxypyr were previously selected in 2003 and 2009 by the Danish EPA for control of content of active substances.



### 3.2.4 Clopyralid

Clopyralid is a herbicide used in Denmark for weed control in beets, beet root, winter rapeseed and on pastures. Chemically, it belongs to the group of pyredinecarboxylic acids and is a synthetic auxin acting like indolylacetic acid. Clopyralid is a selective systemic herbicide that accumulates in meristematic tissue and acts on cell elongation and respiration.<sup>9,10</sup> In the 2016 control campaign one product with clopyralid formulated as clopyralid-olamine (CAS no. 57754-85-5) was analysed. Products with clopyralid were previously selected in 2000, 2009 and 2014 by the Danish EPA for control of content of active substances.

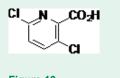


Figure 10 Clopyralid, CAS no. 1702-17-6

#### 3.2.5 MCPA

MCPA, (4-chloro-2-methylphenoxy)acetic acid, is a herbicide used in Denmark for weed control in pastures and lawns. Chemically, it belongs to the group of phenoxycarboxylic acids and is a synthetic auxin acting like indolylacetic acid. MCPA concentrates in the meristematic regions where it inhibits growth.<sup>9,10</sup> Products with MCPA were previously selected in 2004 and 2012 by the Danish EPA for control of content of active substances.

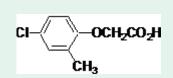


Figure 11 MCPA, CAS no. 94-74-6

#### 3.2.6 Florasulam

Florasulam is a herbicide used in Denmark for weed control in, e.g., wheat, barley and rye and in meadows. Chemically, it belongs to the group of triazolopyrimidines and is a selective herbicide for post-emergence control of broad-leaved weeds. Florasulam inhibits branched chain amino acid (leucine, isoleucine and valine) synthesis.<sup>9,10</sup> Products with florasulam were previously selected in 2005 by the Danish EPA for control of content of active substances.

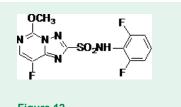


Figure 12 Florasulam, CAS no. 145701-23-1

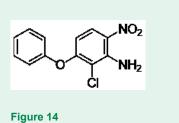
#### 3.2.7 Bromoxynil

Bromoxynil is a herbicide used in Denmark for weed control in, e.g., winter and spring cereal. Chemically, it belongs to the hydroxybenzonitrils and is a selective contact herbicide with some systemic activity. Bromoxynil is a photosynthetic electron transport inhibitor at the photosystem II receptor site and also uncouples oxidative phosphorylation.<sup>9,10</sup> Products with bromoxynil were previously selected in 2000 and 2008 by the Danish EPA for control of content of active substances.



### 3.2.8 Aclonifen

Aclonifen is a herbicide used in Denmark for weed control in, e.g., potatoes, carrots, peas and beans. Chemically, it belongs to the group of diphenyl ethers and is a systemic, selective herbicide. Aclonifen inhibits carotenoid biosynthesis, however, the target enzyme is not known.<sup>9,10</sup> Products with aclonifen were previously selected in 2000 and 2008 by the Danish EPA for control of content of active substances.



Aclonifen, CAS no. 74070-46-5

#### 3.2.9 Clomazone

Clomazone is a herbicide used in Denmark for weed control in, e.g., potatoes, carrots, peas and carrots. Chemically, it belongs to the group of isoxazolidinones and is a selective herbicide, absorbed by the roots and shoots and translocated upward. Clomazone inhibits carotenoid biosynthesis, however, the target enzyme is not known.<sup>9,10</sup> Products with clomazone were pre-viously selected in 2003 by the Danish EPA for control of content of active substances.

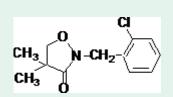


Figure 15 Clomazone, CAS no. 81777-89-1

### 3.2.10 Prosulfocarb

Prosulfocarb is a herbicide used in Denmark for weed control in, e.g., winter cereals and potatoes. Chemically, it belongs to the group of thiocarbamates and is a selective herbicide, absorbed by the leaves and roots. Prosulfocarb acts by inhibiting growth in the meristematic region by inhibition of lipid synthesis.<sup>9,10</sup> Products with prosulfocarb were previously selected in 2009 by the Danish EPA for control of content of active substances.



Prosulfocarb, CAS no. 52888-80-9

### 3.2.11 Analysis

Glyphosate was analysed by reversed phase HPLC-DAD. All samples were dissolved in an aqueous phosphate buffer by ultra-sonification before the analysis. The method is a modified HPLC method according to CIPAC Glyphosate 284.<sup>11</sup>

Fluroxypyr and clopyralid were formulated as fluroxypyr-meptyl and clopyralid-olamine, respectively, in the analysed products. Therefore, the method was adapted to the formulated compounds. Fluroxypyr-meptyl, clopyralid-olamine, MCPA and florasulam were analysed by reversed phase HPLC-DAD. All samples were dissolved in a mixture of water and acetonitrile by ultra-sonification before analyses.

Diflufenican, bromoxynil, aclonifen and clomazone were analysed by reverse-phase HPLC-DAD. All samples were dissolved in acetonitrile by ultra-sonication before analysis. The diflufenican method is a modified HPLC method according to CIPAC diflufenican 462.

Prosulfocarb was analysed by GC-MS. Depending on the formulation, samples were dissolved in either dichloromethane or acetone before analysis.

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

The methods for determination of glyphosate, diflufenican, fluroxypyr-meptyl, MCPA, clopyralidolamine and aclonifen are accredited.

### 3.2.12 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 the determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

Table 9	
Analysis results of herbicides (pesticides), g/L	

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
686013-2	Glyphosate	450	428-473	465	2.6	63	402-528	Comply
686013-4	Glyphosate	360	342-378	373	0.7	35	338-406	Comply
	Fluroxypyr-meptyl	57.6	51.9-63.4	56.6	4.3	6.0	50.7-62.6	Comply
686013-6	MCPA	200	188-212	192	4	23	168-215	Comply
	Clopyralid-olamine	26.4	23.7-29.0	26.7	1.0	1.4	25.3-28.0	Comply
686013-7	Glyphosate	7.2	6.1-8.3	7.7	0.7	1.2	6.5-8.9	Comply
686013-8	Bromoxynil	402	382-422	366	0.9	17	349-383	*
686013-9	Aclonifen	600	575-625	599	0.7	16.5	583-616	Comply
686013-10	Prosulfocarb	800	775-825	803	2.4	119	684-922	Comply
686013-14	Glyphosate	360	342-378	371	4.8	57	314-427	Comply
	Glyphosate	250	235-265	264	0.6	14	250-278	Comply
686013-15	Diflufenican	40	36-44	41	0.4	3	38-44	Comply
686013-19	Glyphosate	120	113-127	126	4.9	17	109-143	Comply
000010.00	Fluroxypyr-meptyl	144	136-153	142	2.8	13	129-155	Comply
686013-22	Florasulam	2.5	2.1-2.9	2.3	2.8	0.16	2.1-2.4	Comply
686013-24	Glyphosate	7.2	6.1-8.3	6.9	1.7	1.4	5.5-8.3	Comply
686013-26	Glyphosate	360	342-378	375	2.4	55	320-431	Comply
686013-27	Glyphosate	7.2	6.1-8.3	7.7	9	1.7	6.1-9.4	Comply
686013-28	Glyphosate	120	113-127	125	0.6	14	111-140	Comply
686013-30	Glyphosate	360	342-378	365	1.5	56	309-421	Comply
686013-31	Prosulfocarb	800	775-825	782	1.4	78	704-860	Comply
000010.00	Fluroxypyr-meptyl	144	136-153	139	0.6	6	133-146	Comply
686013-32	Florasulam	5.0	4.3-5.8	4.7	0.9	0.2	4.5-4.9	Comply
686013-35	Fluroxypyr-meptyl	480	456-504	493	1.6	67	426-560	Comply
686013-36	Prosulfocarb	800	775-825	829	1.4	58	771-887	Comply

\* On the conclusion of the analyses, the expiration date of the product had been exceeded.

DTI sample no.	Active substanc- es	Label claim g/kg	Tolerance interval	Analysis result g/kg	RSD %	U <sub>E</sub> g/L	Analysis interval	Comply/ Non- comply
606012.20	Aclonifen	500	475-525	507	1.1	20	488-527	Comply
686013-20	Clomazone	30	27-33	30	9	7.9	22-38	Comply

 Table 10

 Analysis results of herbicides (pesticides), g/kg

### 3.2.13 Conclusion

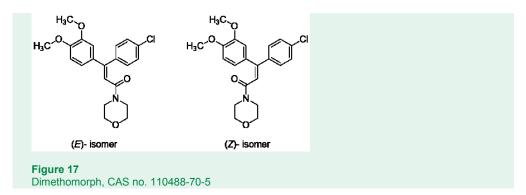
The measured content of glyphosate, fluroxypyr, MCPA, clopyralid, aclonifen, prosulfocarb, diflufenican and florasulam for the 21 products complies with the declared content and with applicable Danish law, as there is an overlap between the range of analysis results and tolerance.

## 3.3 Fungicides (Pesticides)

One fungicide containing dimethomorph as active substance was selected for the 2016 campaign.

### 3.3.1 Dimethomorph

Dimethomorph is a fungicide used in Denmark for control of fungal diseases in potatoes and onions. Chemically, it belongs to the group of cinnamamides and is a local systemic fungicide with protectant and antisporulant activity. Dimethomorph inhibits the formation of the oomycete fungal cell wall.<sup>9,10</sup> Products with dimethomorph were previously selected in 2007 by the Danish EPA for control of content of active substances.



## 3.3.2 Analysis

Dimethomorph was analysed by reversed phase high-performance liquid chromatography combined with diode array detection, HPLC-DAD. The samples were dissolved in water and acetonitrile by ultra-sonication before the analysis. The method is a modified HPLC method according to CIPAC method dimethomorph 483.<sup>11</sup>

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

The method for dimethomorph is accredited.

### 3.3.3 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 the determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

#### Table 11

Analysis results of fungicides (pesticides), g/kg

DTI sample no.	Active substances	Label claim g/kg	Tolerance interval	Analysis result g/kg	RSD %	U <sub>E</sub> g/L	Analysis interval	Comply/ Non- comply
686013-18	Dimethomorph	75	67.5-82.5	73	2	4.6	68.0-77.2	Comply

### 3.3.4 Conclusion

The measured content of dimethomorph complies with the declared content and with applicable Danish law, as there is an overlap between the range of analysis results and tolerance.

## 3.4 Growth regulators (Pesticides)

Three growth regulators containing chlormequat-chloride as active substance were selected for the 2016 control campaign.

### 3.4.1 Chlormequat-chloride

Chlormequat-chloride is a plant growth regulator used in Denmark for growth regulation in, e.g., winter wheat, spring wheat, rye and oat. Chemically, it is a quaternary ammonium compound and it is a plant growth regulator that inhibits cell elongation. Chlormequat-chloride acts as gibberellin biosynthesis inhibitor.<sup>9,10</sup> Products with chlormequat-chloride were previously selected in 2006 by the Danish EPA for control of content of active substances.

## CICH<sub>2</sub>CH<sub>2</sub>N(CH<sub>3</sub>)<sub>3</sub> CI

Figure 18 Chlormequat-chloride, CAS no. 999-81-5

## 3.4.2 Analysis

Chlormequat-chloride was analysed by hydrophilic interaction chromatography combined with mass spectrometry, LC-MS. The samples were dissolved in water and acetonitrile before the analysis.

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

## 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 the determinations of the product.  $U_E$  is the calculated expanded measurement uncertainty described in section 1.4.1.

 Table 12

 Analysis results of growth regulators (pesticides), g/L.

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
686013-1	Chlormequat-chloride	750	725-775	773	2.5	50	723-823	Comply
686013-3	Chlormequat-chloride	750	725-775	791	1.9	58	733-849	Comply
686013-34	Chlormequat-chloride	750	725-775	778	2.7	77	725-775	Comply

#### 3.4.4 Conclusion

The measured content of chlormequat-chloride complies with the declared content and with applicable Danish law, as there is an overlap between the range of analysis results and tolerance. Product 686013-3 had expired upon analysis.

## 4. Physical-chemical testing

The density of all liquid products was measured. The water content and pH were determined for products where a specified value was available for comparison. The measured parameters were compared to the values specified for the product and evaluated as comply/non-comply. As the physical-chemical parameters are subject to confidentiality, only the general conclusions have been included in this report.

### 4.1 Density

The density was measured for a total of 31 liquid products. Table 13 lists the 19 products for which a specified value was available for comparison.

#### Table 13

Products for which density was measured.

DTI sample no.	Active substance	Product name
686013-1	Chlormequat-chlorid	Trece 750
686013-2	Glyphosate	Glyfonova Accelerator
686013-3	Chlormequat-chlorid	Cycocel 750
686013-6	Fluroxypyr, MCPA & Clopyralid	Ariane FGS S
686013-8	Bromoxynil	Xinca
686013-9	Aclonifen	Fenix
686013-10	Prosulfocarb	Fidox EC
686013-14	Glyphosate	Agro-Glyfo Toupar
686013-15	Glyphosate & Diflufenican	Pistol
686013-17	Tau-Fluvalinate	Maverik 2F
686013-22	Fluroxypyr & Florasulam	Starane XL
686013-24	Glyphosate	Tanaco SPOT ON ukrudtsmiddel
686013-25	Alpha-cypermethrin	Fastac 50
686013-26	Glyphosate	Glyfonova plus
686013-30	Glyphosate	Glypper
686013-31	Prosulfocarb	LFS Prosulfocarb
686013-32	Fluroxypyr & Florasulam	Primus XL
686013-35	Fluroxypyr	Starane 333HL
686013-36	Prosulfocarb	Boxer

### 4.1.1 Conclusion

The measured values deviated by less than 0.01 kg/L from the specified value for all products and the specified densities are comparable with no major discrepancies.

#### 4.2 Water content

The water content was measured for a total of 27 products. Table 14 lists the products.

 Table 14

 Products for which water content was measured.

DTI sample no.	Active substance	Product name
686013-1	Chlormequat-chlorid	Trece 750
686013-2	Glyphosate	Glyfonova Accelerator
686013-3	Chlormequat-chlorid	Cycocel 750
686013-4	Glyphosate	ROUNDUP BIO
686013-6	Fluroxypyr, MCPA & Clopyralid	Ariane FGS S
686013-7	Glyphosate	ROUNDUP terrasse
686013-8	Bromoxynil	Xinca
686013-9	Aclonifen	Fenix
686013-13	Cypermethrin	Tanaco RTU Universal Insekt
686013-14	Glyphosate	Agro-Glyfo Toupar
686013-15	Glyphosate & Diflufenican	Pistol
686013-16	Cypermethrin	Tanaco Myrespray
686013-17	Tau-Fluvalinate	Maverik 2F
686013-18	Mancozeb & Dimethomorph	Acrobat NEW
686013-20	Aclonifen & Clomazone	Novitron DAMTec
686013-22	Fluroxypyr & Florasulam	Starane XL
686013-23	Cypermethrin	Gardol Myrespray
686013-24	Glyphosate	Tanaco SPOT ON ukrudtsmidde
686013-26	Glyphosate	Glyfonova plus
686013-27	Glyphosate	ROUNDUP spray
686013-28	Glyphosate	ROUNDUP garden
686013-30	Glyphosate	Glypper
686013-32	Fluroxypyr & Florasulam	Primus XL
686013-33	Cypermethrin	Garden Myre og Insekt
686013-34	Chlormequat-chlorid	Stabilan Extra
686013-35	Fluroxypyr	Starane 333HL
686013-36	Prosulfocarb	Boxer

### 4.2.1 Conclusion

Of the 27 products, 25 deviated by less than 5% from the specified contents. The water content in one product deviated by approximately 10% from the specified value.

A deviation larger than 10% between the measured and specified values for water content was observed in another product.

### 4.3 pH

The pH was determined for 1% solutions of the formulation in 20 products and for undiluted formulations in 5 products. Table 15 lists these products.

#### Table 15

Products for which pH in 1% solution was measured.						
DTI sample no.	Active substance	Product name				
686013-1	Chlormequat-chlorid	Trece 750				
686013-2	Glyphosate	Glyfonova Accelerator				
686013-3	Chlormequat-chlorid	Cycocel 750				
686013-6	Fluroxypyr, MCPA & Clopyralid	Ariane FGS S				
686013-8	Bromoxynil	Xinca				
686013-9	Aclonifen	Fenix				
686013-11	Lambda-cyhalothrin	Karate 2.5 WG				
686013-14	Glyphosate	Agro-Glyfo Toupar				
686013-15	Glyphosate & Diflufenican	Pistol				
686013-17	Tau-Fluvalinate	Maverik 2F				
686013-18	Mancozeb & Dimethomorph	Acrobat NEW				
686013-20	Aclonifen & Clomazone	Novitron DAMTec				
686013-21	Lambda-cyhalothrin	Kaiso Sorbie				
686013-22	Fluroxypyr & Florasulam	Starane XL				
686013-25	Alpha-cypermethrin	Fastac 50				
686013-26	Glyphosate	Glyfonova plus				
686013-30	Glyphosate	Glypper				
686013-32	Fluroxypyr & Florasulam	Primus XL				
686013-34	Chlormequat-chlorid	Stabilan Extra				
686013-35	Fluroxypyr	Starane 333HL				

#### Table 16

DTI sample no.	Active substance	Product name	
686013-1	Chlormequat-chlorid	Trece 750	
686013-3	Chlormequat-chlorid	Cycocel 750	
686013-6	Fluroxypyr, MCPA & Clopyralid	Ariane FGS S	
686013-8	Bromoxynil	Xinca	
686013-34	Chlormequat-chlorid	Stabilan Extra	
4.2.4	Conclusion		

4.3.1 Conclusion

The measured pH of the 1% solutions was comparable with the specified values for 17 of the 20 products. For three products, the measured pH deviated about one pH-unit.

For the pH in the undiluted samples, the measured pH was comparable with the specified values.

## 5. Screenings

Products under a parallel trade permit were 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.

## 5.1 Regulations in Denmark

The requirements for parallel trade permits are described in detail in Article 52 of Regulation No. 1107/2009.<sup>15</sup>

### 5.1.1 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. For each country, an application must be made 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, the parallel trade permit will be similarly affected.

### 5.1.2 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.

## 5.2 Analysed products

The analysed product appears in Table 17. For cypermethrin products, only parallel products were available and a comparison between the parallel products rather than between the original and the parallel was therefore made.

#### Table 17

Comparison of parallel products containing cypermethrin and comparison of original product with parallel product containing glyphosate or prosulfocarb.

Active substance	Original product DTI sam- ple no.	Product name	Reg. no.	Parallel product DTI sample no.	Product name	Reg. no.
		Cythrin		686013-13	Tanaco RTU universal Insekt	361-34
Cypermehtrin	-		361-14	686013-16	Tanaco Myrespray	361-35
				686013-23	Gradol Myrespray	361-44
				686013-33	Garden Myre og Insekt	361-32
Glyphosate	686013-27	ROUNDUP Spray	48-30	686013-7	ROUNDUP Terrasse	48-37
Prosulfocarb	686013-12	Boxer	1-211	686013-31	LFS Prosulfocarb	318-69

#### 5.2.1 Analysis

#### 5.2.1.1 GC-MS

A subsample (app. 5 g) was weighed in a 50 mL calibrated flask, and dichloromethane was added up to the label. Another subsample was added methanol. 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), scan m/z 31 - 450.

#### 5.2.1.2 FTIR

A drop of the product was placed on an ATR crystal. Fourier transform infrared spectroscopy (FTIR) was carried out on a spectrometer of the type Agilent Technologies 4500. The spectra were taken with ATR (*attenuated total reflectance*) with 64 repetitions and a solution of 8 cm-1. The cypermethrin parallel products, 686013-23, 686013-16, 686013-13 and 686013-33, contained too much water to perform the FTIR analysis. It was attempted to dry the samples for two weeks and to analyse the sample by FTIR-ARO (*all reflecting objective*), however, it was not possible to obtain meaningful IR spectra for these samples.

#### 5.2.2 Results and conclusion

The difference between the determined concentrations and densities is within the analysis uncertainties of the methods; refer to Table 18.

#### Table 18

Quantitative determination of the concentration of cypermethrin, glyphosate and prosulfocarb and density determination. Comparison of parallel products containing cypermethrin and original products with parallel products containing glyphosate or prosulfocarb.

Active sub- stance	DTI sample no.	Product name	Analysis result g/L	RSD %	Density kg/L
	686013-13	Tanaco RTU Universal Insekt	0.45	2.5	1.00
Cypermehtrin	686013-16	Tanaco Myrespray	0.46	1.7	1.00
	686013-23	Gradol Myrespray	0.46	5.8	1.00
	686013-33	Garden Myre og Insekt	0.52	5.2	1.00
Dresulfacerh	686013-12	Boxer	770	1.1	1.02
Prosulfocarb	686013-31	LFS Prosulfocarb	782	1.4	1.02
Glyphosate	686013-7	ROUNDUP terrasse	7.7	0.7	1.02
	686013-27	ROUNDUP spray	7.7	9	1.02

The comparison of FTIR spectra demonstrated differences in intensity, but all peaks appear in both spectra for original and parallel products.

The colour and appearance of the dichloromethane and methanol sample extracts were assessed to be identical for comparable samples.

When comparing the GC-MS chromatograms, some differences in the peak intensity were observed, however, the chromatographic profile was identical for comparable samples.

In conclusion, there was no difference between the cypermethrin parallels, or the glyphosate and prosulfocarb originals and parallel products based on the applied parameters.

## References

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- 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.
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- 13. Danish Standard DS 235:1978
- 14. http://www.fao.org/docrep/x5042e/x5042e0a.htm
- 15. The requirements for parallel trade permits, Article 52 of Regulation No 1107/2009, http://mst.dk/82449

## Appendix 1.

## Pesticide & biocide samples collected on the Danish market for authority control 2016

#### Table 19

Collected insecticides (Biocides).

DTI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
686013-5	Diflubenzuron	558-9	Dimilin 5	Arysta LifeScience
686013-13	Cypermethrin	361-34	Tanaco RTU universal insekt	Arysta LifeScience
686013-16	Cypermethrin	361-35	Tanaco Myrespray	Arysta LifeScience
686013-23	Cypermethrin	361-44	Gardol Myrespray	Arysta LifeScience
686013-33	Cypermethrin	361-32	Garden Myre og insekt	Arysta LifeScience

#### Table 20

Collected insecticides (Pesticides).

DTI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
686013-11	Lambda-cyhalothrin	1-163	Karate 2.5 WG	Syngenta
686013-17	Tau-fluvalinate	396-13	Mavrik 2F	ADAMA Registrations B.V.
686013-21	Lambda-cyhalothrin	347-25	Kaiso Sorbie	Nufarm
686013-25	Alpha-cypermethrin	19-139	Fastac 50	BASF
686013-29	Cypermethrin	579-4	Maladan insektspray W	SBM Développement

#### Table 21

Collected herbicides (Pesticides).

DTI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
686013-2	Glyphosate	11-44	Glyfonova Accelerator	Cheminova
686013-4	Glyphosate	48-16	Roundup Bio	Monsanto Crop Sciences
686013-6	Fluroxypyr, MCPA & Clopyralid	64-39	Ariane FG S	DOW
686013-7	Glyphosate	48-37	Roundup Terrasse	Monsanto Crop Sciences
686013-8	Bromoxynil	347-21	Xinca	Nufarm
686013-9	Aclonifen	18-417	Fenix	Bayer
686013-10	Prosulfocarb	613-3	Fidox EC	Globachem NV
686013-12	Prosulfocarb	1-211	Boxer	Syngenta
686013-14	Glyphosate	600-1	Agro-Glyfo Toupar	Saphire bvda
686013-15	Glyphosate & Diflufenican	18-497	Pistol	Bayer
686013-19	Glyphosate	49-106	Trinol ukrudtsmiddel	Klarsø A/S
686013-20	Aclonifen & Clomazone	421-7	Novitron DAMTec	FMC Chemical
686013-22	Fluroxypyr & Florasulam	64-68	Starane XL	DOW
686013-24	Glyphosate	11-46	Tanaco SPOT ON ukrudtsmiddel	Cheminova
686013-26	Glyphosate	11-12	Glyfonova Plus	Cheminova
686013-27	Glyphosate	48-30	Roundup spray	Monsanto Crop Sciences
686013-28	Glyphosate	48-26	Roundup Garden	Monsanto Crop Sciences
686013-30	Glyphosate	396-53	Glypper	ADAMA Registrations B.V
686013-31	Prosulfocarb	318-69	LFS Prosulfocarb	LFS Kemi
686013-32	Fluroxypyr & Florasulam	64-78	Primus XL	DOW
686013-35	Fluroxypyr	64-82	Starane 333 HL	DOW
686013-36	Prosulfocarb	1-211	Boxer	Syngenta

#### Table 22

Collected fungicides (Pesticides).

DTI sample no	. Active substances	Reg. no.	Name of product	Authorisation holder
686013-18	Mancozeb* & Dimethomorph	19-222	Acrobat New	BASF
+==				

\*The concentration of mancozeb has not been determined

#### Table 23

Collected growth regulators (Pesticides).

DTI sample no.	Active substances	Reg. no.	Name of product	Authorisation holder
686013-1	Chlormequat-chlorid	49-42	Trece 750	Klarsø A/S
686013-3	Chlormequat-chlorid	19-22	Cycocel 750	BASF
686013-34	Chlormequat-chlorid	347-16	Stabilan Extra	Nufarm

#### **Control of Pesticides and Biocides 2016**

#### English

The analytical chemical authority control of pesticide and biocide products on the Danish market that was carried out in 2016 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 complies with the product specification and the labelled content. A total of 35 different pesticide products were analysed. The content of active substance in 1 of the analysed products was not within the accepted tolerance that is determined in the Danish Statutory Order on pesticides.

#### Danish

Den analytisk kemiske kontrol af biocid- og pesticidprodukter på det danske marked, der er udført i 2016 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 35 bekæmpelsesmidler. Indholdet af aktivstoffet i 1 af de analyserede produkter var ikke inden for den accepterede tolerance, der er fastsat i bekendtgørelsen om bekæmpelsesmid-ler[Bagside Tekst]



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