National Environmental Research Institute
Ministry of the Environment · Denmark

Control of Pesticides 2003

Chemical Substances and Chemical Preparations

NERI Technical Report No. 534

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2005

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Data sheet

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Abstract

Four different groups of products covered by the pesticide regulation were included in the 2003 analytical chemical authority control: 1) Herbicides containing clodinafop-propargyl, clomazone, fluroxypyr and glyphosate. 2) Fungicides containing bitertanol, fuberidazole, fenhexamid and pencycuron. 3) Insecticides containing cypermethrin, deltamethrin, lambda-cyhalothrin, methoprene and cyromazine. 4) Plant growth regulators containing 1-napthylacetic acid. All products were examined for the content of the respective active ingredients and for the content of OPEO and NPEO. All samples but one containing methoprene complied with the accepted tolerance limits with respect to the content of the active ingredient as specified in Danish Statutory Order on pesticides. None of the 44 examined samples contained OPEO, but 5 of the samples contained NPEO. Three of these five samples were produced before the agreement. On three products, the content of active ingredient was declared only in g/L, but not in % (w/w). One product was declared as the ester and not as the acid.

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Contents

Summary

The analytical chemical authority control of pesticide products on the Danish market performed in 2003 is reported. Samples of selected groups of pesticides have been collected from the market and analysed to verify whether the actual contents of the respective active ingredients in the products comply with the labelled content. The tolerance of deviation from the labelled content of active ingredient is set by the Danish Statutory order on pesticides. In addition to the examination of the content of active ingredients all collected samples are examined for content of octylphenol ethoxylates (OPEO) and nonylphenol ethoxylates (NPEO). The industry and the Danish authorities have agreed on removing these compounds from all Danish-sold pesticide formulations produced after June 2000.

Four different groups of products covered by the pesticide regulation were included in the 2003 analytical chemical authority control: 1) Herbicides containing clodinafop-propargyl, clomazone, fluroxypyr and glyphosate. 2) Fungicides containing bitertanol, fuberidazole, fenhexamid and pencycuron. 3) Insecticides containing cypermethrin, deltamethrin, lambda-cyhalothrin, methoprene and cyromazine. 4) Plant growth regulators containing 1-napthylacetic acid.

Satisfactory results were found for herbicides containing clodinafop-propargyl, clomazone, fluroxypyr and glyphosate, for fungicides containing bitertanol, fuberidazole, fenhexamid and pencycuron, for insecticides containing cypermethrin, deltamethrin, lambda-cyhalothrin and cyromazine and for plant growth regulators containing 1-napthylacetic acid. Thus, the 43 analysed samples of these formulations complied with the accepted tolerance limits with respect to the content of the active ingredient as specified in Danish Statutory Order on pesticides.

The product containing methoprene did not comply with the tolerance limit for content of the active ingredient. The content of the active ingredient was too low compared with the declared content. The manufacturer has not sold the formulation since 1999. Production date or expiry date did not appear from the label.

None of the 44 examined samples contain OPEO, but 5 of the samples contain NPEO. Three of these five samples were produced before the industry and the Danish authorities agreed on removing these kinds of substances from the pesticide formulations.

On three products the content of active ingredient were declared only in g/L, but not in % (w/w) as required by the Statutory Order. On one product containing fluroxypyr the contents of active ingredient was declared as the ester and not as the acid, as the Statutory Order requires.

Resumé

Den analytisk kemiske kontrol af pesticidprodukter på det danske marked udført i 2003 af de danske myndigheder er her afrapporteret. Prøver af udvalgte grupper af bekæmpelsesmidler er blevet samlet fra markedet og analyseret for at verificere om det aktuelle indhold af de respektive aktivstoffer er i overensstemmelse med det deklarerede indhold. Grænsen for en accepteret afvigelse af indholdet af aktivstof fra det deklarerede indhold er fastsat i bekendtgørelsen om bekæmpelsesmidler. Udover kontrol af indholdet af aktivstof er alle indsamlede prøver kontrolleret for indhold af octylphenolethoxylater (OPEO) og nonylphenolethoxylater (NPEO). Industrien og de danske myndigheder har indgået en frivillig aftale om at udfase disse forbindelser fra alle dansk-solgte pesticidprodukter produceret efter juni 2000.

Fire forskellige grupper produkter er inkluderet i den analytisk-kemiske kontrol udført af myndighederne i 2003: 1) Ukrudtsmidler indeholdende clodinafop-propargyl, clomazone, fluroxypyr og glyphosat. 2) Svampemidler indeholdene bitertanol, fuberidazol, fenhexamid og pencycuron. 3) Insektmidler indeholdene cypermethrin, deltamethrin, lambda-cyhalothrin, methopren og cyromazin. 4) Vækstregulerende midler indeholdende 1-napthyleddikesyre.

Der blev opnået tilfredsstillende resultater blandt ukrudtsmidler indeholdende clodinafop-propargyl, clomazone, fluroxypyr og glyphosat, blandt svampemidler indeholdene bitertanol, fuberidazol, fenhexamid og pencycuron, blandt insektmidler indeholdene cypermethrin, deltamethrin, lambda-cyhalothrin og cyromazin og blandt vækstregulerende midler indeholdende 1-napthyleddikesyre. Indholdet af aktivstof i alle de 43 analyserede prøver af disse bekæmpelsesmidler var indenfor den accepterede tolerance, der er fastsat i bekendtgørelsen om bekæmpelsesmidler.

Produktet, der indeholder methopren var ikke indenfor den accepterede tolerance for indhold af aktivstof. Indholdet var for lavt i forhold til det deklarerede indhold. Producenten har ikke solgt formuleringen siden 1999. Produktionsdato og holdbarhedsdato fremgår ikke af etiketten.

Ingen af de 44 undersøgte produkter indeholder OPEO, men 5 af prøverne indeholder NPEO. Tre af de fem prøver er produceret før industrien og de danske myndigheder indgik en frivillige aftale om at udfase denne type stoffer.

På tre produkter var indholdet af aktivstof kun deklareret i g/l og ikke i % som det ellers er krævet i bekendtgørelsen. På et produkt indeholdende fluroxypyr er indholdet af aktivstof deklareret som esteren og ikke som syren som krævet i bekendtgørelsen.

1 Introduction

In Denmark, the Danish Environmental Protection Agency (DEPA) is responsible for the evaluation and the authorisation of all pesticide formulations before introduction on the Danish market. The requirements for the formulations are given in a Statutory Order from the Ministry of the Environment *(Miljøministeriet, 2003)*, which also states that DEPA is responsible for control of pesticides.

In practice authority control activities of pesticides on the market are organised in the following way: the Chemicals Inspection Service at DEPA conducts non-laboratory control and the National Environmental Research Institute conducts the laboratory control of pesticides as an assistance to DEPA. The present report describes only the part of the authority control of pesticides involving laboratory control.

Laboratory control of pesticides covers analytical chemical examination of technical pesticides or pesticide formulations in order to verify that the products comply with the legal requirements of pesticides as well as with the specification of contents stated in the application for the pesticide product.

Analytical chemical control of pesticides may involve verification of the content of active ingredients as well as content of auxiliary substances or levels of impurities.

Laboratory control work is carried out by means of two types of projects: 1) Ordinary control by way of planned campaigns, where all products with a common characteristic, e.g. the same active ingredient, are collected from the market and examined, and 2) *ad hoc* projects, which consist of laboratory control in connection with administrative work at the regulatory authorities, e.g. complaints from users concerning a specific product, suspicion of a product not complying with regulations, specifications, etc.

Only the first type of laboratory control i.e. campaigns are covered by this report, which describes the laboratory control performed in 2003.

2 Control Campaigns in 2003

Control campaigns conducted in 2003 have covered active ingredients and auxiliary substances belonging to four different groups of pesticides: herbicides, fungicides, insecticides and grow regulators. All analytical chemical control has aimed at examining the content of active ingredient compared with the declared content on the label. Statutory Order in Denmark *(Miljøministeriet, 2003)* specifies general tolerance of deviation from declared content. These are given in Table 2.1. In addition to the examination of the content of active ingredients all samples are examined for content of octylphenol ethoxylates and nonylphenol ethoxylates.

Samples of the various pesticide formulations covered in the 2003 control campaigns have been collected by the Chemical Inspection Service at DEPA during the months March – July 2003 from either whole sale dealers/importers or at retailer out-lets. One sample of each product has been collected.

Samples were stored at NERI in unopened containers until the time of analysis. The samples were stored at ambient temperature (approx. 20°C) protected from light.

Table 2.1 Tolerance of deviations from declared content of active ingredients (a.i.) in pesticides.

Declared content of a.i., %, w/w	**Tolerance, %**	
conc. ≥ 50	± 2.5%	(abs.)
25 < conc. ≤ 50	± 5%	(rel.)
10 < conc. ≤ 25	± 6%	(rel.)
2.5 < conc. ≤ 10	± 10%	(rel.)
conc. ≤ 2.5	± 15%	(rel.)

2.1 Herbicides

2.1.1 Introduction

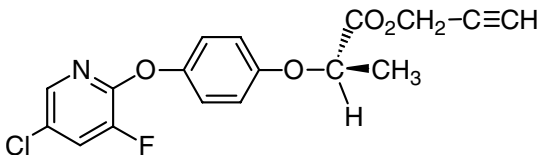
Among the about 44 different active ingredients in herbicide formulations available on the Danish market (*Miljøstyrelsen, 2002*), products containing clodinafop-propargyl, clomazone, fluroxypyr and glyphosate as active ingredients were selected for control in 2003. All products were examined for the content of active ingredient and for the content of octylphenol and nonylphenol.

Clodinafop-propargyl (Figure 1,a) belongs to the group of aryloxyphenoxypropionate herbicides. It is used only to control weeds in winter wheat in Denmark. Clodinafop-propargyl is a post-emergence, systemic herbicide, which inhibits the synthesis of fatty acids by inhibition of acetyl CoA carboxylase. Clodinafop-propargyl is a new active ingredient on the Danish market. It was included in the Danish register of approved pesticides in 2001 and has therefore not previously been selected for authority control.

Clomazone (Figure 1,b) is an isoxazolidinone herbicide, which in Denmark is used to control a broad spectrum of broad-leaved weeds and grass in potatoes and winter rape. It is a pre-emergence selective systemic herbicide that is absorbed through the roots and shoots and inhibits the biosynthesis of carotene. Clomazone is a new active ingredient on the Danish market. It was included in the Danish register of approved pesticides in 2001 and has therefore not previously been selected for authority control.

Fluroxypyr (Figure 1,c) belongs to the group of pyridinecarboxylic acid herbicides. It is used to control a range of broad-leaved weeds in cereals, grass areas and maize used for feed in Denmark. It is a systemic post-emergence herbicide, which is absorbed through the leaves. Herbicide formulations containing fluroxypyr have not previously been selected for authority control.

Glyphosate (Figure 1,d) is a glycine derivative, which in Denmark is used as a defoliant and as a broad-spectrum herbicide used to control weeds in cereals, cruciferous crops, beans, peas, and grass and used to control weeds in orchards and forestry. Glyphosate is a non-selective systemic herbicide that is absorbed by the leaves, with rapid translocation throughout the plant. It prevents the synthesis of essential aromatic amino acids needed for protein biosynthesis. Herbicide formulations containing glyphosate were selected for authority control in 1992, where all five examined products complied with the declared content with respect to the active ingredient.



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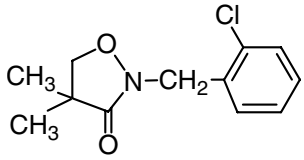

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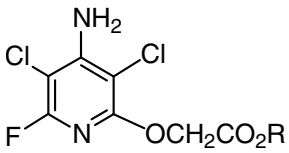
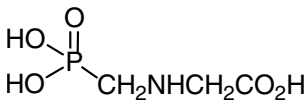


2-butoxy-1-methylethyl R = $CH_3(CH_2)_3OCH_2CH(CH_3)$-

meptyl (1-methylheptyl) R = $CH_3(CH_2)_5CH(CH_3)$-

(c)



(d)

Figure 1
Chemical structures of the herbicide active ingredients: clodinafop-propargyl (a), clomazone (b), fluroxypyr (c), and glyphosate (d).

2.1.2 Samples

At the time of sampling for the control campaign, one product containing clodinafop-propargyl, one product containing clomazone, and five products containing fluroxypyr were approved for use in Denmark. All these products were available on the market. 27 products containing glyphosate were approved for use in Denmark, 18 of these products were available on the market during the period of the sample collection. One sample of each herbicide product was collected. The samples are listed in Appendix I.

The sample containing clomazone was analysed in September-October 2003. The sample containing clodinafop-propargyl was analysed in December 2003 - January 2004, fluroxypyr-containing products were analysed during the period January-February 2004 and products containing glyphosate were analysed in February-March 2004.

2.1.3 Results and Discussion

The contents of clodinafop-propargyl were determined using gas chromatography and flame ionisation detection (GC-FID) *(Krongaard, 2004a)*. As no CIPAC-method on clodinafop-propargyl exists, the method is developed on the basis of information from the manufacturer.

The content of clomazone was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2004b)*. As no CIPAC-method on clomazone exists, the method is developed on the basis of information from the manufacturer.

The contents of fluroxypyr were determined using gas chromatography and flame ionisation detection (GC-FID) *(Krongaard, 2003a)*. As no CIPAC-method on fluroxypyr exists, the method is developed on the basis of information from the manufacturer.

The content of glyphosate was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV with a strong anion exchange column *(Krongaard, 2004c)*. The method is developed on the basis of the existing CIPAC method.

Table 2.2 shows an agreement between declared and determined content for all the samples containing clodinafop-propargyl, clomazone, fluroxypyr and glyphosate. On two products containing glyphosate the contents of active ingredient was only declared in g/L not in % (w/w) as the Statutory Order requires. On one product containing fluroxypyr the contents of active ingredient was declared as the ester and not as the acid, as the Statutory Order requires. The manufacturer of the latter has informed that the label will be corrected.

Table 2.2 Content of active ingredient in samples of herbicides.

Active ingredient	Content				NERI sample no.
	Label claim		Analysis[1]	Tolerance[2]	
Clodinafop-propargyl	9.6 %	(100 g/L)	9.26 ± 0.15 %	8.64 – 10.56 %	03-0177
Clomazone	31.4 %	(360 g/L)	31.53 ± 0.22 %	29.83 – 32.97 %	02-0179
Fluroxypyr	18 %	(180 g/L)	17.98 ± 0.07 %	16.92 – 19.08 %	03-0174
Fluroxypyr	18 %	(180 g/L)	18.31 ± 0.07 %	16.92 – 19.08 %	03-0175
Fluroxypyr	3.64 %	(40 g/L)	3.65 ± 0.01 %	3.28 – 4.00 %	03-0222
Fluroxypyr	9.52[5]	(100 g/L)	8.72 ± 0.03 %	8.57 – 10.47 %	03-0223
Fluroxypyr	3.63 %	(40 g/L)	3.70 ± 0.01 %	3.27 – 3.99 %	03-0400
Glyphosate	40 %	(480 g/L)	39.83 ± 0.0 9%	38 – 42 %	03-0162
Glyphosate	30 %	(360 g/L)	29.66 ± 0.07 %	28.5 – 31.5 %	03-0164
Glyphosate	30 %	(360 g/L)	31.10 ± 0.07 %	28.5 – 31.5 %	03-0165
Glyphosate	68 %	(680 g/kg)	68.14 ± 0.31 %	66.3 – 69.7 %	03-0166
Glyphosate	30 %	(360 g/L)	30.18 ± 0.07 %	28.5 – 31.5 %	03-0167
Glyphosate	-[3]	(360 g/L)	30.37 ± 0.07 %[4]	28.5 – 31.5 %	03-0168
Glyphosate	30 %	(360 g/L)	29.92 ± 0.07 %	28.5 – 31.5 %	03-0169
Glyphosate	30 %	(360 g/L)	30.02 ± 0.07 %	28.5 – 31.5 %	03-0170
Glyphosate	28.3 %	(360 g/L)	27.47 ± 0.06 %	26.9 – 29.7 %	03-0171
Glyphosate	30 %	(360 g/L)	31.31 ± 0.07 %	28.5 – 31.5 %	03-0221
Glyphosate	11.3 %	(120 g/L)	11.70 ± 0.03 %	10.6 – 12.0 %	03-0232
Glyphosate	30 %	(360 g/L)	30.21 ± 0.07 %	28.5 – 31.5 %	03-0233
Glyphosate	0.72 %	(7.2 g/L)	0.69 ± 0.01 %	0.61 – 0.83 %	03-0234
Glyphosate	5.8 %	(60 g/L)	5.41 ± 0.01 %	5.22 – 6.38 %	03-0235
Glyphosate	30 %	(360 g/L)	30.09 ± 0.07 %	28.5 – 31.5 %	03-0236
Glyphosate	-[3]	(7.2 g/L)	0.70 ± 0.01 %[4]	0.61 – 0.83 %	03-0399
Glyphosate	30 %	(360 g/L)	30.06 ± 0.07 %	28.5 – 31.5 %	03-0402
Glyphosate	30 %	(360 g/L)	30.89 ± 0.07 %	28.5 – 31.5 %	03-0682

1) Mean ± 95% confidence limits.
2) Tolerance limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 2003)*.
3) Content (expressed as %) not declared.
4) Calculated on the basis of the declared content in g/l and on measured density.
5) The content is declared as the ester on the label, but the content is here expressed as the acid.

2.2 Fungicides

2.2.1 Introduction

About 36 active ingredients in fungicide formulations are approved in Denmark (*Miljøstyrelsen, 2002*). The products containing bitertanol, fuberidazole, fenhexamid and pencycuron were selected for control in 2003 and examined for content of active ingredient.

Bitertanol (Figure 2a) belongs to the large group of triazole fungicides. It is used as seed treatment in winter cereals and to control fungal diseases on apples, pears and cherries in Denmark. Bitertanol is a foliar fungicide, which inhibits steroid demethylation. Bitertanol was included in the Danish register of approved pesticides in 1988 but has not previously been selected for authority control.

Fuberidazole (Figure 2b) belongs to the group of benzimidazoles. It is a selective fungicide used only as seed treatment in winter cereals to control Fusarium in Denmark and only in combination with bitertanol. Fuberidazole is a systemic fungicide, which inhibits mitosis. Fuberidazole has not previously been selected for authority control.

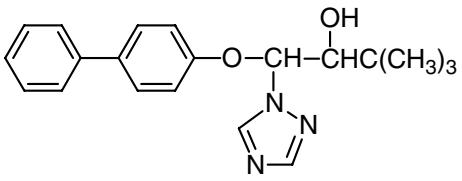
Fenhexamid (Figure 2c) is a hydroyanilide fungicides, which is used only to control fungal diseases in strawberries, cherries, and in black- and red currants in Denmark. Fenhexamid is a foliar fungicide with unknown biochemical target, but it inhibits germ tube elongation and mycelium growth. Fenhexamid was included in the Danish register of approved pesticides in 1999 but has not previously been selected for authority control.

Pencycuron (Figure 2d) is a phenylurea fungicide, which is used only as seed treatment in potatoes to control fungal diseases in Denmark. Pencycuron is a non-systemic fungicide, which was included in the Danish register of approved pesticides in 1993 but has not previously been selected for authority control.

2.2.2 Samples

At the time of sampling for the control campaign, two products containing bitertanol, one product containing fenhexamid, one product containing fuberidazole and two products containing pencycuron were approved for use in Denmark. All these products were available on the market. One sample of each fungicide product was collected. The samples are listed in Appendix I.

The samples containing pencycuron were analysed in March 2003. The samples containing bitertanol and fuberidazole were analysed in August - September 2003 and the fenhexamid-containing product was analysed during the period October-November 2003.



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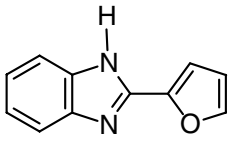

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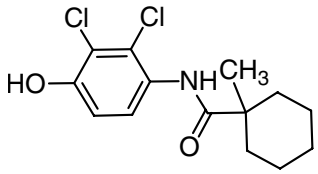

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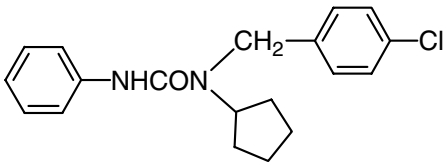


(d)

Figure 2
Chemical structures of the fungicide active ingredients: bitertanol (a), fuberidazole (b), fenhexamid (c), and pencycuron (d).

2.2.3 Results and Discussion

The contents of bitertanol and fuberidazole were determined using gas chromatography and flame ionisation detection (GC-FID) *(Krongaard, 2003b)*. As no CIPAC-method on fuberidazole exists, the method is developed on the basis of information from the manufacturer. The method allows simultaneous determination of both components

The content of fenhexamid was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2003c)*. As no CIPAC-method on fenhexamid exists, the method is developed on the basis of information from the manufacturer.

The content of pencycuron was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2003c)*. A CIPAC-method on clomazone exists, but it is based on normal phase HPLC. A new method based on reversed phase HPLC is developed.

Table 2.3 shows an agreement between declared and determined content in all six samples containing bitertanol, fenhexamid, fuberidazole and pencycuron as active ingredients.

Table 2.3 Content of active ingredient in samples of fungicides.

Active ingredient	Content				NERI sample no.
	Label claim		Analysis[1)]	Tolerance[2)]	
Bitertanol	27 %	280 g/L	26.0 ± 0.2%	25.7 – 28.4%	03-0172
Bitertanol	25 %	-	25.8 ± 0.2%	23.5 – 26.5 %	03-0173
Fuberidazole	1.74 %	18 g/L	1.75 ± 0.01%	1.48 – 2.00 %	03-0172
Fenhexamid	50 %	-	48.5 ± 0.2%	47.5 – 52.5 %	03-0178
Pencycuron	22.8 %	250 g/L	21.9 ± 0.2%	21.4 – 24.2 %	03-0224
Pencycuron	12.5 %	-	11.8 ± 0.1%	11.8 – 13.3 %	03-0225

1) Mean ± 95% confidence limits.
2) Tolerated limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 2003)*.

2.3 Insecticides

2.3.1 Introduction

Among the different insecticide formulations available on the Danish market (*Miljøstyrelsen, 2002*) the products containing cypermethrin, deltamethrin, lambda-cyhalothrin, methoprene and cyromazine as active ingredients were selected for control in 2003. All products were examined for content of active ingredient.

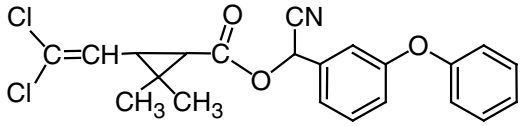
Cypermethrin (Figure 3a) belongs to the group of pyrethroids. It is used for control of creeping and crawling insects in and around houses in Denmark. Cypermethrin is a non-systemic insecticide with contact and stomach action. It prevents the sodium channels from functioning so that transmission of nerve impulses can take place. Insecticide formulations containing cypermethrin have been included in the Danish register of approved pesticides for many years. Cypermethrin has not been selected for authority control in the last decade.

Deltamethrin (Figure 3b) belongs to the group of pyrethroids too. It is used, as cypermethrin, for control of creeping and crawling insects in and around houses in Denmark. Like cypermethrin it is a non-systemic insecticide with contact and stomach action. Insecticide formulations containing deltamethrin have been included in the Danish register of approved pesticides for many years. Deltamethrin has not been selected for authority control in the last decade.

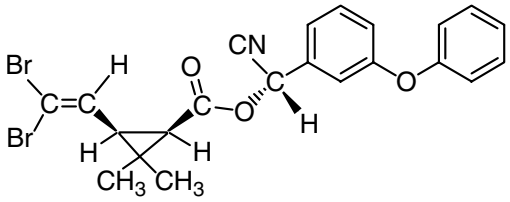
Lambda-cyhalothrin (Figure 3c) belongs to the group of pyrethroids as the two previous insecticides. Lambda-cyhalothrin is used for control of insects in a broad range of agricultural crops, forestry and nurseries in Denmark. Like the other pyrethroids it is a non-systemic insecticide with contact and stomach action. Insecticide formulations containing lambda-cyhalothrin were included in the Danish register in 1991 has not previously been selected for authority control.

Methoprene (Figure 3d) is a juvenile hormone mimic, which is used for control of flea eggs and larvae on cats and dogs in Denmark. It is a growth regulator, which prevent metamorphosis to viable adults when applied to larvae stages. Insecticide formulations containing methoprene have been included in the Danish register of approved pesticides for many years. Methoprene has not been selected for authority control in the last decade.

Cyromazine (Figure 3e) is an insect growth regulator, which is used for control of fly maggots in stables, cowsheds, piggeries, poultry and mink farms and on dunghills in Denmark. Cyromazine has contact action. It interferes with moulting and pupation. Insecticide formulations containing cyromazine have been included in the Danish register of approved pesticides for many years. Cyromazine has not been selected for authority control in the last decade.

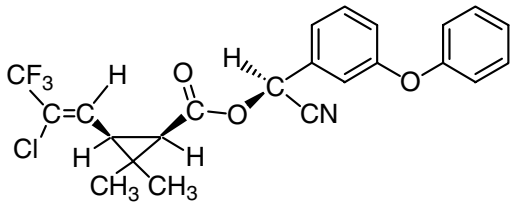


(a)



(b)

(*S*) (*Z*)-(1*R*)-*cis*-



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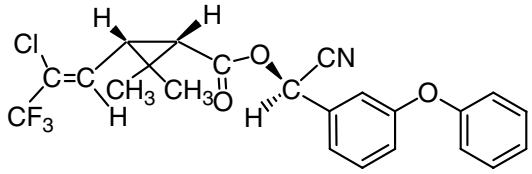
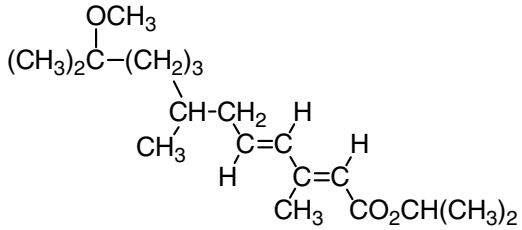
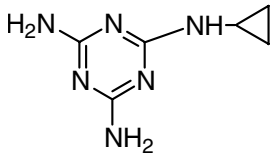


(*R*) (*Z*)-(1*S*)-*cis*-

(c)



(d)



(e)

Figure 3
Chemical structure of the insecticide active ingredients: cypermethrin (a), deltamethrin (b), lambda-cyhalothrin (c), methoprene (d), and cyromazine (e).

2.3.2 Samples

At the time of sampling, one product containing deltamethrin and one product containing lambda-cyhalothrin were approved for use in Denmark. Both products were available on the market during the period of the sample collection. One out of two products containing methoprene, three out of four products containing cyromazine and three out of five products containing cypermethrin as active ingredient were available. One sample of each insecticide product was collected. The samples are listed in Appendix I.

The samples containing cyromazine were analysed in August – September 2003. The sample containing lambda-cyhalothrin was analysed in October, the product containing deltamethrin was analysed in November, the products containing cypermethrin were analysed in December 2003 - February 2004 and the product containing methoprene was analysed in February 2004.

2.3.3 Results and Discussion

The content of cypermethrin was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2004d).* A CIPAC-method on clomazone exists, but it is based on packed GC-column. A reversed phase HPLC method is developed based on information from the manufacturer.

The content of deltamethrin was determined using normal phase high performance liquid chromatography and UV-detector, NP-HPLC-UV *(Krongaard, 2003d).* The method is developed on the basis of a method proposed as a new CIPAC method.

The content of lambda-cyhalothrin was determined using gas chromatography and flame ionisation detection (GC-FID) *(Krongaard, 2003e)*. The method is developed on the basis of the existing CIPAC method.

The content of methoprene was determined using gas chromatography and flame ionisation detection (GC-FID) *(Krongaard, 2004e)*. As no CIPAC-method on methoprene exists, the method is developed on the basis of information from the manufacturer.

The content of cyromazine was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2003f).* As no CIPAC-method on cyromazine exists, the method is developed on the basis of information from the manufacturer.

Table 2.4 shows an agreement between declared and determined content made for the sample containing cypermethrin, deltamethrin, lambda-cyhalothrin and cyromazine whereas the content of active ingredient in the product containing methoprene was found to be outside the tolerance limit. The content of the active ingredient in the sample was too low compared with the declared content. On one product containing cypermethrin the content of active ingredient was only declared in g/L not in % (w/w) as the Statutory Order requires.

Subsequent contact to the manufacturer of the methoprene product revealed that the product controlled has not been sold since 1999 in Denmark. There is no production date or expiry date on the package.

Table 2.4 Content of active ingredient in samples of insecticides.

Active ingredient	Content				NERI sample no.
	Label claim		Analysis[1]	Tolerance[2]	
Cypermethrin	-[3]	(100 g/L)	93.7 ± 0.3 g/L	90 – 110 %[4]	03-0229
Cypermethrin	11 %	(100 g/L)	11.31 ± 0.04 %	10.3 – 11.7 %	03-0230
Cypermethrin	11 %	(100 g/L)	11.81 ± 0.04 %	10.3 – 11.7 %	03-0231
Deltamethrin	1 %	(10 g/L)	1.048 ± 0.003 %	0.85 – 1.15 %	03-0398
Lambda-cyhalothrin	2.5 %	(25 g/kg)	2.63 ± 0.02 %	2.25 – 2.75 %	03-0176
Methoprene	0.5 %	(0.4 g/L)	0.367 ± 0.003 %	0.425 – 0.575 %	03-0228*)
Cyromazine	2 %	-	1.91 ± 0.04 %	1.7 – 2.3 %	03-0180
Cyromazine	2 %	-	2.00 ± 0.05 %	1.7 – 2.3 %	03-0226
Cyromazine	2 %	-	1.94 ± 0.04 %	1.7 – 2.3 %	03-0403

1) Mean ± 95% confidence limits.
2) Tolerance limits for content of active ingredients according to Danish regulation *(Miljø- og Energiministeriet, 2003)*.
3) Content (expressed as %) not declared.
4) Calculated on the basis of the declared content in g/L.
*) Found content is outside the accepted tolerance.

2.4 Plant growth regulators

2.4.1 Introduction

Among the nine plant growth regulators available on the Danish market (*Miljøstyrelsen, 2002*) the plant growth regulators formulations containing 1-naphtylacetic acid as active ingredient was selected for control in 2003, and examined for the content of active ingredients.

1-naphtylacetic acid (Figure 4) is a plant growth regulator with auxin-like activity. It is used to prevent fruit drop, used as a fruit-thinning agent and used to promote the formation of roots in cuttings in Denmark. Formulations containing 1-naphtylacetic acid have been included in the Danish register of approved pesticides for many years. 1-naphtylacetic acid has not been selected for authority control in the last decade.

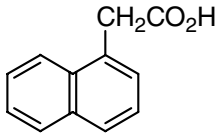


Figure 4
Chemical structure of the plant growth regulator 1-naphtylacetic acid

2.4.2 Samples

At the time of sampling for the control campaign, five products containing 1-naphtylacetic acid as active ingredient were approved for use in Denmark. Four of these five products were available on the market during the period of the sample collection. One sample of each product was collected. The samples are listed in Appendix I

The samples were analysed in October 2003 and due to an inadequate analytical method again in the period February - March 2004.

2.4.3 Results and Discussion

The content of 1-naphtylacetic acid was determined by using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2004b).* As no CIPAC-method on methoprene exists, the method is developed on the basis of information from the manufacturer.

Table 2.5 shows an agreement between declared and determined content in all four samples containing 1-naphtylacetic acid as active ingredient.

Table 2.5 Content of active ingredient in samples of plant growth regulators.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis[1)]	Tolerance[2)]	
1-naphtyl acetic acid	1.5 % -	1.49 ± 0.01 %	1.275 – 1.725 %	03-0227
1-naphtyl acetic acid	0.1 % -	0.109 ± 0.003 %	0.085 – 0.115 %	03-0237
1-naphtyl acetic acid	0.2 % -	0.197 ± 0.005 %	0.17 – 0.23 %	03-0238
1-naphtyl acetic acid	0.4 % -	0.413 ± 0.011 %	0.34 – 0.46 %	03-0239

1) Mean ± 95% confidence limits.
2) Tolerated limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 2003)*.

2.5 Additives

2.5.1 Introduction

Among the many additives used in pesticide formulations nonylphenol ethoxylates (NPEO) and octylphenol ethoxylates (OPEO) were selected for control in 2003. All formulations are examined for the content of NPEO and OPEO.

NPEO and OPEO belong to the group of alkylphenol ethoxylates (APEO), a group of surface-active compounds, which is widely used in formulation of plant protection products. They are added to the formulation to change the physical properties e.g. to facilitate the transport of the active ingredient into the plants or into the insects. In the 1990's APEO was recognised to have estrogenic effects. This kind of substances were/are are suspected to be the contributory reason to the decrease in the male reproduction ability, and to the increase in the cases of abnormality in the male sexual organs and the cases of testicle cancer. The same effects are also seen in wild living male animals. OPEO is not used as widely as NPEO in pesticide formulations, but the estrogenic effect is several times higher. The industry and the Danish authorities have agreed on removing these compounds from all Danish-sold pesticide formulations produced after June 2000 except for few exemptions given by the Danish authorities. Dealers are allowed to sell stocks after this date.

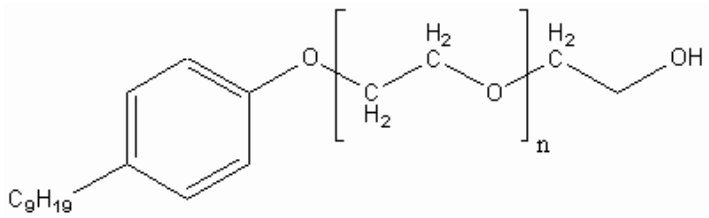


Figure 5
Chemical structure of the addtive nonylphenol ethoxylate. The structure of octylphenol ethoxylate is similar, C_9H_{19} is replaced with C_8H_{17}.

2.5.2 Samples

Beside the examination of the content of active ingredient all pesticide formulations sampled in 2003 are examined for content of NPEO and OPEO. One formulated additive for manual addition is examined too. The samples are listed in Appendix I

The samples were analysed in the period October 2003 - March 2004.

2.5.3 Results and Discussion

The content of NPEO and OPEO was determined by using reversed phase high performance liquid chromatography and MS-detector, RP-HPLC-MS *(Krongaard, 2004g).* As no CIPAC-method on NPEO and OPEO exists, the method is developed in the laboratory. The analytical method is capable to analyse NPEO and OPEO simultaneously.

Table 2.6 Content of NPEO and OPEO in samples of pesticide formulations.

No. of samples	No. of samples with NPEO/OPEO	No. of samples without NPEO or OPEO
44	5/0	39

Table 2.6 shows that none of the 44 examined samples contain OPEO, but 5 of the samples contain NPEO. Three of the five samples were produced before the industry and the Danish authorities agreed on removing these kinds of substances from the pesticide formulations. One of the manufacturers of the other two samples has repeated the analysis with same result as NERI. The other manufacturer has not yet repeated the analysis.

3 Conclusions

Four different groups of products covered by the pesticide regulation were included in the 2003 analytical chemical authority control: 1) Herbicides containing clodinafop-propargyl, clomazone, fluroxypyr and glyphosate. 2) Fungicides containing bitertanol, fuberidazole, fenhexamid and pencycuron. 3) Insecticides containing cypermethrin, deltamethrin, lambda-cyhalothrin, methoprene and cyromazine. 4) Plant growth regulators containing 1-napthylacetic acid. All products were examined for the content of the active ingredients. In addition to the examination of the content of active ingredients all collected samples were examined for content of octylphenol ethoxylates and nonylphenol ethoxylates.

Satisfactory results were found for herbicides containing clodinafop-propargyl, clomazone, fluroxypyr and glyphosate, for fungicides containing bitertanol, fuberidazole, fenhexamid and pencycuron, for insecticides containing cypermethrin, deltamethrin, lambda-cyhalothrin and cyromazine and for plant growth regulators containing 1-napthylacetic acid. Thus, the 43 analysed samples of these formulations complied with the accepted tolerance limits with respect to the content of the active ingredient as specified in Danish Statutory Order on pesticides.

The product containing methoprene did not comply with the tolerance limit for content of this active ingredient. The content of the active ingredient was too low compared with the declared content. Subsequent contact to the manufacturer revealed that the manufacturer has not sold the formulation since 1999. Production date or expiry date did not appear from the label.

None of the 44 examined samples contain OPEO, but 5 of the samples contain NPEO. Three of the five samples were produced before the industry and the Danish authorities agreed on removing these kinds of substances from the pesticide formulations. One of the manufacturers of the other two samples has repeated the analysis with same result as NERI. The other manufacturer has not yet repeated the analysis.

On three products the content of active ingredient were declared only in g/L, but not in % (w/w) as required by the Statutory Order. On one product containing fluroxypyr the contents of active ingredient was declared as the ester and not as the acid, as the Statutory Order requires. The manufacturer of the latter has informed that the label will be corrected.

4 References

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Appendix I

Samples of pesticide formulations collected from the Danish market for authority control in 2003.

Table 1 Herbicides

Active ingredient	Product	Formulation type[1)]	Company	NERI sample no.
clodinafop-propargyl	Topik 100 EC	EC	Syngenta Crop Protection	03-0177
Clomazone	Commands	CS	BASF	03-0179
Fluroxypyr	Starane 18 S	EC	Dow Agro Sciences	03-0174
Fluroxypyr	Ethosan Tomahawk 180EC	EC	Korn- og Forderstofkompagniet	03-0175
Fluroxypyr	Ariane FG	EC	Dow Agro Sciences	03-0222
Fluroxypyr	Ariane Super	EC	Dow Agro Sciences	03-0223
Fluroxypyr	Greenor	EC	Dow Agro Sciences	03-0400
Glyphosate	Roundup 3000	SL	Monsanto Crop Sciences	03-0162
	Additive to Roundup 3000		Monsanto Crop Sciences	03-0163
Glyphosate	Roundup Bio	SL	Monsanto Crop Sciences	03-0164
Glyphosate	Glyfonova Plus	SL	Cheminova Agro	03-0165
Glyphosate	Roundup Max	SG	Monsanto Crop Sciences	03-0166
Glyphosate	Jablo Glyphosat	SL	Jablo Plant Protection	03-0167
Glyphosate	ND Glyphosat 360	SL	NEDAB	03-0168
Glyphosate	OK 500	SL	Danagri	03-0169
Glyphosate	Roundup	SL	Monsanto Crop Sciences	03-0170
Glyphosate	Touchdown premium	SL	Syngenta Crop Protection	03-0171
Glyphosate	Glyfonova 360	SL	Cheminova Agro	03-0221
Glyphosate	Roundup Garden	SL	Monsanto Crop Sciences	03-0232
Glyphosate	Bonus ukrudtsmiddel	SL	Aako	03-0233
Glyphosate	Lotus ukrudtsmiddel	AL	Tanaco	03-0234
Glyphosate	Ukrudtsmiddel Lotus	SL	Tanaco	03-0235
Glyphosate	LFS Gyphosat	SL	LFS Kemi	03-0236
Glyphosate	Mod ukrudt overalt	AL	Agrodan	03-0399
Glyphosate	LFS Glyphosat Ultra	SL	LFS Kemi	03-0402
Glyphosate	Dan-Kvik	SL	Cheminova Agro	03-0682

AL: Any other liquid; CS: Capsule suspension; EC: Emusifiable concentrate; SG: water soluble granule; SL: Soluble concentrate;

Table 2 Fungicides

Active ingredient	Product	Formulation type[1)]	Company	NERI sample no.
Bitertanol	Sibutol LS 280	LS	Bayer	03-0172
Bitertanol	Baycor WP25	WP	Bayer	03-0173
Fenhexamid	Teldor WG 50	WG	Bayer	03-0178
Fuberidazol	Sibutol LS 280	LS	Bayer	03-0172
Pencycuron	Monceren FS 250	FS	Bayer	03-0224
Pencycuron	Monceren DS 12,5	DS	Bayer	03-0225

DS: Powder for dry seed treatment; FS: Flowable concentrate for seed treatment; LS: Solution for seed treatment; WG Water dispersible granule; WP: Wettable powder.

Table 3 Insecticides

Active ingredient	Product	Formulation type[1)]	Company	NERI sample no.
Deltamethrin	Coopersect Spot On	SA	Schering-plough Animal Health	03-0398
Cypermethrin	IT-Cypermethrin	EC	Inter-Trade	03-0229
Cypermethrin	CYMPA-TI Extra	EC	DK-Petrokemi	03-0230
Cypermethrin	Cyperb	EC	KemiAgro	03-0231
Cyromazine	NEP REX WSG 2	SG	Novartis	03-0180
Cyromazine	Mortalin Cyromazin mod fluelarver	SG	Mortalin	03-0226
Cyromazine	LFS Cyromazin	SG	LFS Kemi	03-0403
Lambda-cyhalothrin	Karate 2.5 WG	WG	Syngenta Crop Protection	03-0176
Methopren	Kattespray med methopren	SA	Matas	03-0228

EC: emulsifiable concentrate; WG Water dispersible granule; SA: Spot-On; SG Water soluble granule

Table 4 Plant growth regulator

Active ingredient	Product	Formulation type[1)]	Company	NERI sample no.
1-naphtylacetic acid	Pomoxon	SL	Novotrade	03-0227
1-naphtylacetic acid	Floramon A pudder	AP	Novotrade	03-0237
1-naphtylacetic acid	Floramon B pudder	AP	Novotrade	03-0238
1-naphtylacetic acid	Floramon C pudder	AP	Novotrade	03-0239

AP: Any other powder; SL: Soluble concentrate;

National Environmental Research Institute

The National Environmental Research Institute, NERI, is a research institute of the Ministry of the Environment. In Danish, NERI is called *Danmarks Miljøundersøgelser (DMU)*.
NERI's tasks are primarily to conduct research, collect data, and give advice on problems related to the environment and nature.

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Publications:
NERI publishes professional reports, technical instructions, and the annual report. A R&D projects' catalogue is available in an electronic version on the World Wide Web.
Included in the annual report is a list of the publications from the current year.

Faglige rapporter fra DMU/NERI Technical Reports

2004

Nr. 490: Reservatnetværk for trækkende vandfugle. En gennemgang af udvalgte arters antal og fordeling i Danmark 1994-2001. Af Clausen, P. et al. 142 s. , 150,00 kr.
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Nr. 498: Analyse af højt NO2 niveau i København og prognose for 2010. Af Berkowicz, R. et al. 30 s. (elektronisk)
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Nr. 500: Aquatic Environment 2003. State and Trends - technical summary. By Andersen, J.M. et al. 50 pp. , 100,00 DDK
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Nr. 502: Samfundsøkonomiske analyser af ammoniakbufferzoner. Udredning for Skov- og Naturstyrelsen. Af Schou, J.S., Gyldenkærne, S. & Bak, J.L. 36 s. (elektronisk)
Nr. 503: Luftforurening fra trafik, industri og landbrug i Frederiksborg Amt. Af Hertel, O. et al. 88 s. (elektronisk)
Nr. 504: Vingeindsamling fra jagtsæsonen 2003/04 i Danmark. Af Clausager, I. 70 s. (elektronisk)
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Four different groups of products covered by the pesticide regulation were included in the 2003 analytical chemical authority control: 1) Herbicides containing clodina-fop-propargyl, clomazone, fluroxypyr and glyphosate. 2) Fungicides containing bitertanol, fuberidazole, fenhexamid and pencycuron. 3) Insecticides containing cypermethrin, deltamethrin, lambda-cyhalothrin, methoprene and cyromazine. 4) Plant growth regulators containing 1-napthylacetic acid. All products were examined for the content of the respective active ingredients and for the content of OPEO and NPEO. All samples but one containing methoprene complied with the accepted tole-rance limits with respect to the content of the active ingredient as specified in Danish Statutory Order on pesticides. None of the 44 examined samples contained OPEO, but 5 of the samples contained NPEO. Three of these five samples were produced be-fore the agreement. On three products, the content of active ingredient was declared only in g/L, but not in % (w/w). One product was declared as the ester and not as the acid.

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