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Chemical Substances and Chemical Preparations

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2002

Teddy Krongaard
Kitty K. Petersen
Christel Christoffersen

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Abstract: Three different groups of products covered by the pesticide regulation have been included in the 2001 analytical chemical authority control: 1) herbicides containing pendimethalin and methabenzthiazuron 2) Fungicides containing azaconazole, tolylfluanid, propamocarb and cyprodinil 3) Insecticides containing amitraz, diflubenzuron, azamethiphos, malathion, phosalone and phoxim. All products were examined for content of active ingredients. Satisfactory results were found among herbicides containing pendimethalin and methabenzthiazuron, among fungicides containing azaconazole, tolylfluanid, propamocarb and cyprodinil, and among insecticides containing amitraz, phosalone and diflubenzuron. Thus, all analysed samples of these pesticides complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides. Two of six samples containing malathion, nine of twelve samples containing azamethiphos and two of five samples containing phoxim did not comply with the accepted limits of content of active ingredient. All in all only one out of eleven samples of ant-boxes and fly strips/sheets comply with the accepted tolerance with respect to content of active ingredient.

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Contents

Summary

The analytical chemical authority control on pesticide products on the Danish market performed in 2001 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 label-claimed content. The tolerance of deviation from the label-claimed content of active ingredient is set by the Danish pesticide regulation.

Three different groups of products covered by the pesticide regulation have been included in the 2001 analytical chemical authority control: 1) herbicides containing pendimethalin and methabenzthiazuron. 2) Fungicides containing azaconazole, tolylfluanid, propamocarb and cyprodinil. 3) Insecticides containing amitraz, diflubenzuron, azamethiphos, malathion, phosalone and phoxim. All products were examined for content of active ingredient.

Satisfactory results were found among herbicides containing pendimethalin and methabenzthiazuron, among fungicides containing azaconazole, tolylfluanid, propamocarb and cyprodinil, and among insecticides containing amitraz, phosalone and diflubenzuron. Thus, the twelve analysed samples of these pesticides complied with the accepted tolerances with respect to the content of active ingredients set by the Danish regulation of pesticides.

Two of the six samples containing malathion did not comply with the accepted limits of content of active ingredient. The three EC/EW-formulations did comply with the accepted limits, but two out of three powder-formulations did not. In one of the powder-formulations the content was found to be too high and in the other formulation no malathion was found at all. The manufacturer of the latter products explains that the collected batch is 10 years old and that the malathion is probably evaporated through the paper bag.

Nine of the twelve samples containing azamethiphos did not comply with the accepted limits of content of active ingredient. The three powder-formulations did comply with the accepted limits, but all nine ant-boxes and fly strips did not. In all these samples the content was found to be too low.

Two of five samples containing phoxim did not comply with the accepted limits of content of active ingredient. One powder-formulation had too high content and one ant-box had too low content.

All in all only one out of eleven samples of ant-boxes and fly strips/sheets comply with the accepted tolerance with respect to content of active ingredient.

Resumé

Den analytisk kemiske kontrol af pesticidprodukter på det danske marked udført i 2001 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.

Tre forskellige grupper af produkter er inkluderet i den analytiskkemiske kontrol udført af myndighederne i 2000: 1) herbicider indeholdende pendimethalin og methabenzthiazuron 2) fungicider indeholdende azaconazol, tolylfluanid, propamocarb og cyprodinil 3) insekticider indeholdende amitraz, diflubenzuron, azamethiphos, malathion, phosalon og phoxim.

Der blev opnået tilfredsstillende resultater blandt herbicider indeholdende pendimethalin og methabenzthiazuron, blandt fungicider indeholdende azaconazol, tolylfluanid, propamocarb og cyprodinil og blandt insekticider indeholdende amitraz, phosalon og diflubenzuron. Indholdet af aktivstof i alle de tolv analyserede prøver af disse bekæmpelsesmidler var indenfor den accepterede tolerance, der er fastsat i bekendtgørelsen om bekæmpelsesmidler.

To ud af seks prøver indeholdende malathion var ikke indenfor den accepterede tolerance for indhold af aktivstof. De tre EC/EW-formuleringer var alle indenfor den accepterede tolerance, men to ud af tre pulverformuleringer var udenfor tolerancen. I en af pulverformuleringerne var indholdet for højt, mens der i en anden pulverformulering slet ikke blev fundet malathion. Efterfølgende har producenten af den sidstnævnte formulering forklaret at den analyserede batch var 10 år gammel og at malathion formentlig var fordampet ud gennem papirsækken.

Ni ud af tolv prøver indeholdende azamethiphos var ikke indenfor den accepterede tolerance for indhold af aktivstof. De tre pulverformuleringer var indenfor den accepterede tolerance, mens alle ni myrelokkedåser og fluestrips/plader var udenfor. I alle disse prøver var indholdet for lavt.

To ud af fem prøver indeholdende phoxim var ikke inden for den accepterede tolerance for indhold af aktivstof. Én pulverformulering havde for højt indhold af aktivstof og én myrelokkedåse havde for lavt indhold.

Alt i alt var kun én ud af elleve prøver af myrelokkedåser og fluestrips/plader inden for den accepterede tolerance for indhold af aktivstof.

1 Introduction

In Denmark the Danish Environmental Protection Agency (DEPA) is responsible for the evaluation and the authorisation of all pesticides before an introduction on the Danish market. Legal regulations of pesticides are given in a Statutory Order from the Ministry of the Environment and Energy *(Miljø- og Energiministeriet, 2000),* 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 regulations 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 matters 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 2001.

2 Control Campaigns in 2001

Control campaigns conducted in 2001 have covered pesticides belonging to three different groups of pesticides: Herbicides, fungicides and insecticides. All analytical chemical control has aimed at examining the content of active ingredient compared with the declared content on the label. Regulation in Denmark *(Miljø- og Energiministeriet, 2000)* specifies general tolerance of deviation from declared content. These are given in Table 2.1.

Samples of the various pesticide formulations covered in the 2001 control campaigns have been collected by the Chemical Inspection Service at DEPA during the months April - December 2001 from either whole sale dealers/-importers or at retailers. One sample of each product has been collected. NERI has collected some samples from retailers in November 2001 and January 2002 to verify some analytical results obtained during the campaigns.

Samples have been stored at NERI in unopened containers until the time of analysis. The samples have been stored at ambient temperature (app. 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.)

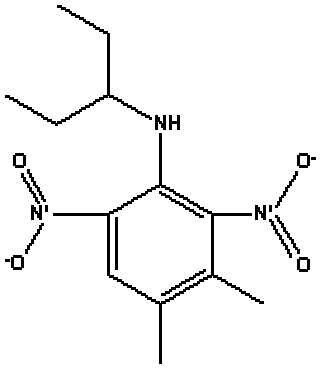
2.1 Herbicides

2.1.1 Introduction

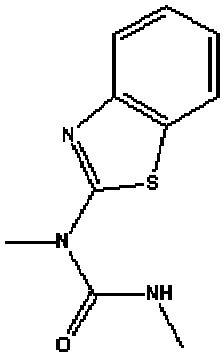
Among the 48 different active ingredient in herbicides available on the Danish market, products containing pendimethalin and methabenzthiazuron as active ingredients were selected for control in 2001. All products were examined for content of active ingredient.

Pendimethalin (Figure 1,a) is a 2,6-dinitroaniline herbicide, which in Denmark is used for control of grass and broad-leaved weeds in winter grain and to ripen peas. Pendimethalin inhibits cell division and cell elongation. It is a selective herbicide that is absorbed by the roots and leaves. Herbicide formulations containing pendimethalin have not previously been selected for authority control.

Methabenzthiazuron (Figure 1,b) belongs to the group of urea herbicide, which is used to control a broad spectrum of broad-leaved weeds and grasses in winter corn, spring wheat, grass seed and in nurseries. Methabenzthiazuron inhibits the photosynthetic electron transport. It is a selective herbicide that is primarily absorbed through the roots. Herbicide formulations containing methabenzthiazuron have not previously been selected for authority control.



(a)



(b)

Figure 1
Chemical structures of the herbicide active ingredients: pendimethalin (a) and methabenzthiazuron (b).

2.1.2 Samples

At the time of sampling for the control campaign only one product containing methabenzthiazuron and two products containing pendimethalin were approved for use in Denmark. All three products were available on the market during the period of the sample collection. One sample of each pesticide product was collected. The samples are listed in Appendix I.

The sample containing methabenzthiazuron was analysed at NERI in February 2002. The samples containing pendimethalin were analysed in February-March 2002.

2.1.3 Results and Discussion

The content of methabenzthiazuron was determined using normal phase high performance liquid chromatography and UV-detector, NP-HPLC-UV *(Krongaard, 2002a)*. The method is developed based on the CIPAC method and on information from the manufacturer.

The content of pendimethalin was determined using gas chromatography and flame ionisation detection (GC-FID) *(Krongaard, 2002b)*. We developed a new method in the laboratory as no CIPAC method on pendimethalin exists, and as the method provided from the manufacturer was based on packed GC-columns, which we do not use in the laboratory anymore.

Table 2.2 shows an agreement between declared and determined content for all three samples containing methabenzthiazuron and pendimethalin. Hence, all the samples complied with the tolerated limits for content of active ingredient.

Table 2.2 Content of active ingredient in samples of herbicides.

Active ingredient	Content				NERI sample no.
	Label claim		Analysis[1)]	Tolerance[2)]	
Methabenzthiazuron	70 %	-	68,8 ± 0,2%	67,5 - 72,5%	01-1568
Pendimethalin	36 %	(400 g / l)	35,77 ± 0,27%	34,2 - 37,8%	01-1566
Pendimethalin	34 %	(330 g / l)	33,81 ± 0,04%	32,3 - 35,7%	01-1567

1) Mean ± 95% confidence limits.
2) Tolerated limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 2000)*.

2.2 Fungicides

2.2.1 Introduction

About 35 active ingredients in fungicides were approved in Denmark in 2001. Products containing azaconazole, tolylfluanid, propamocarb and cyprodinil were selected for control in 2001 and examined for content of active ingredient.

Azaconazole (Figure 2,a) is an azole type of fungicide, which is used only in combination with imazalil for wound heeling on trees in Denmark. Azaconazole inhibits steroid demethylation. Azaconazole is a rather new active ingredient (included in the Danish register of approved pesticides in 1998) and has not previously been selected for authority control.

Tolylfluanid (Figure 2,b) is an N-trihalomethylthio compound, which is approved to be used only on tomatoes and ornamentals in green houses for controlling different fungal diseases (e.g. mildew, rust and leaf spot diseases) in Denmark. Tolylfluanid is a multi-site inhibitor. Formulations containing tolylfluanid have not previously been selected for authority control.

Propamocarb (Figure 2,c) is a carbamate compound, which is approved only to be used on cucumber and tomatoes in green houses and outdoors on ornamentals, salad and potatoes to control different fungal diseases in Denmark. Propamocarb is a multi-site inhibitor. Formulations containing propamocarb have not previously been selected for authority control.

Cyprodinil (Figure 2,d) is an anilinopyrimidine compound, which is approved only to be used for fungal disease control on wheat, barley and rye in Denmark. Cyprodinil inhibits the methionine biosynthesis. The formulation containing cyprodinil is new on the Danish market (included in the Danish register of approved pesticides in 2000) and the formulations therefore have not previously been selected for authority control.

2.2.2 Samples

At the time of sampling for the control campaign only one product containing azaconazole and one product containing tolylfluanid were approved for use in Denmark. Two products containing cyprodinil and three products containing propamocarb as active ingredients were approved for use. Only one product of each active ingredient was available on the market during the sampling period. One sample of each pesticide product was collected. The samples are listed in Appendix I.

The samples containing propamocarb were analysed at NERI in September-October 2001 and July 2002, the sample containing tolylfluanid was analysed in January 2002, the sample containing cyprodinil was analysed in February 2002 and the sample containing azaconazole as active ingredient was analysed in March 2002.

2.2.3 Results and Discussion

The content of propamocarb was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2001a)*. The method is based on a CIPAC method.

The content of tolylfluanid was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2002c)*. As no CIPAC method on tolylfluanid exists, the method is developed on the basis of information from the manufacturer.

The contents of cyprodinil were determined using gas chromatography with flame ionisation detector (GC-FID) *(Krongaard, 2002d)*. As no CIPAC method on kresoxim-methyl exists, the method is developed on the basis of information from the manufacturer.

The content of azaconazole was determined using gas chromatography and flame ionisation detection (GC-FID) *(Krongaard, 2002e)*. We developed a new laboratory method because no CIPAC method for azaconazole exists. The method provided from the manufacturer was based on packed GC-columns, which we do not use in the laboratory anymore.

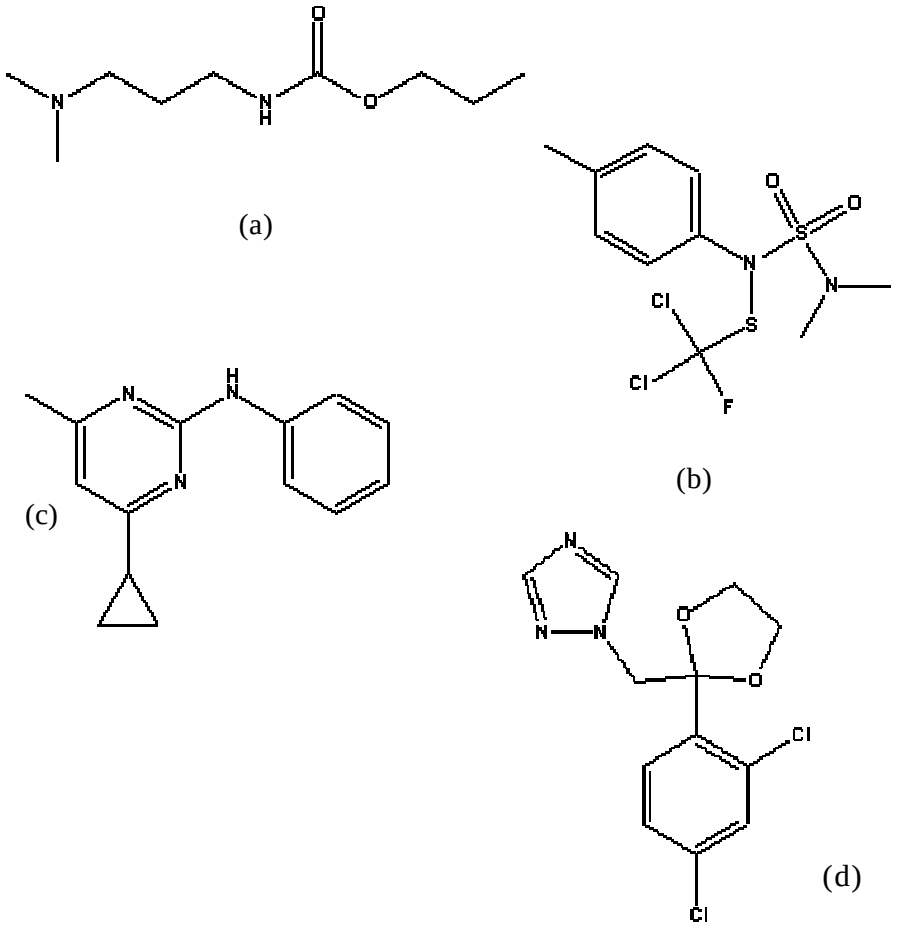


Figure 2
Chemical structure of the fungicide active ingredients propamocarb (a), tolylfluanid (b), cyprodinil (c) and azaconazole (d).

Table 2.3 Content of active ingredient in samples of fungicides.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis[1)]	Tolerance[2)]	
Tolylfluanid	50 % -	50,7 ± 0,3%	47,5 - 52,5%	01-1569
Cyprodinil	24 % (250 g / l)	24,08 ± 0,07%	22,6 - 25,4%	01-1565
Azaconazole	1 % (10 g / l)	0,99 ± 0,01%	0,85 - 1,15%	02-0035
Propamocarb	58,8 % (640 g / l)	58,4 ± 0,3%	56,3 - 61,3%	01-0564
Propamocarb	20 % (248 g / l)	20,6 ± 0,1%	18 - 22%	01-0152

1) Mean ± 95% confidence limits.
2) Tolerated limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 2000).*

Table 2.3 shows an agreement between declared and determined content found in all the samples containing azaconazole, tolylfluanid, propamocarb and cyprodinil Hence, all the samples complied with the tolerated limits.

2.3 Insecticides

2.3.1 Introduction

Among the different insecticides available on the Danish market the insecticides containing amitraz and diflubenzuron, and the organophosphorus compounds: azamethiphos, malathion, phosalone and phoxim as active ingredients were selected for control in 2001.

Amitraz (Figure 3a) is an amidine insecticide, which is used only for control of insects on ornamentals in greenhouses in Denmark. Amitraz increases the nervous activity. Formulations containing amitraz as active ingredient have not previously been selected for authority control.

Diflubenzuron (Figure 3b) is a benzoylurea insecticide, which is used for control of larvae of flies and mosquitoes in mushrooms, control of larvae of flies in manure pits, stalls and poultry- and mink farms in Denmark. It is used also to control a wide range of leaf-eating insects in fruit trees and spruces. Diflubenzuron inhibits synthesis of chitin and so interferes with the formation of the insect cuticle. Formulations containing diflubenzuron as active ingredient have not previously been selected for authority control.

Azamethiphos (Figure 3c) is a organophosphorus insecticide that is used only for control of flies and ants both indoor and outdoor in Denmark. Azamethiphos inhibits the cholinesterase. Formulations containing azamethiphos as active ingredient have not previously been selected for authority control.

Phoxim (Figure 3d) is an organophosphorus insecticide that is used only for control of ants indoor and outdoor and for control of larvae of a mosquito in soil to cutting and seed cultivation in Denmark. Phoxim inhibits the cholinesterase. Formulations containing phoxim as active ingredient have not previously been selected for authority control.

Phosalone (Figure 3e) is an organophosphorus insecticide that is used only for control of certain insects in pomes and ornamentals in Denmark. Phoxim inhibits the cholinesterase. Formulations containing phosalone as active ingredient have not previously been selected for authority control.

Malathion (Figure 3f) is an organophosphorus insecticide that is used for protection of stored grain and to control certain insects in certain crops in agriculture, fruit growing and forestry. Malathion inhibits the cholinesterase in Denmark. Formulations containing malathion as active ingredient have not previously been selected for authority control.

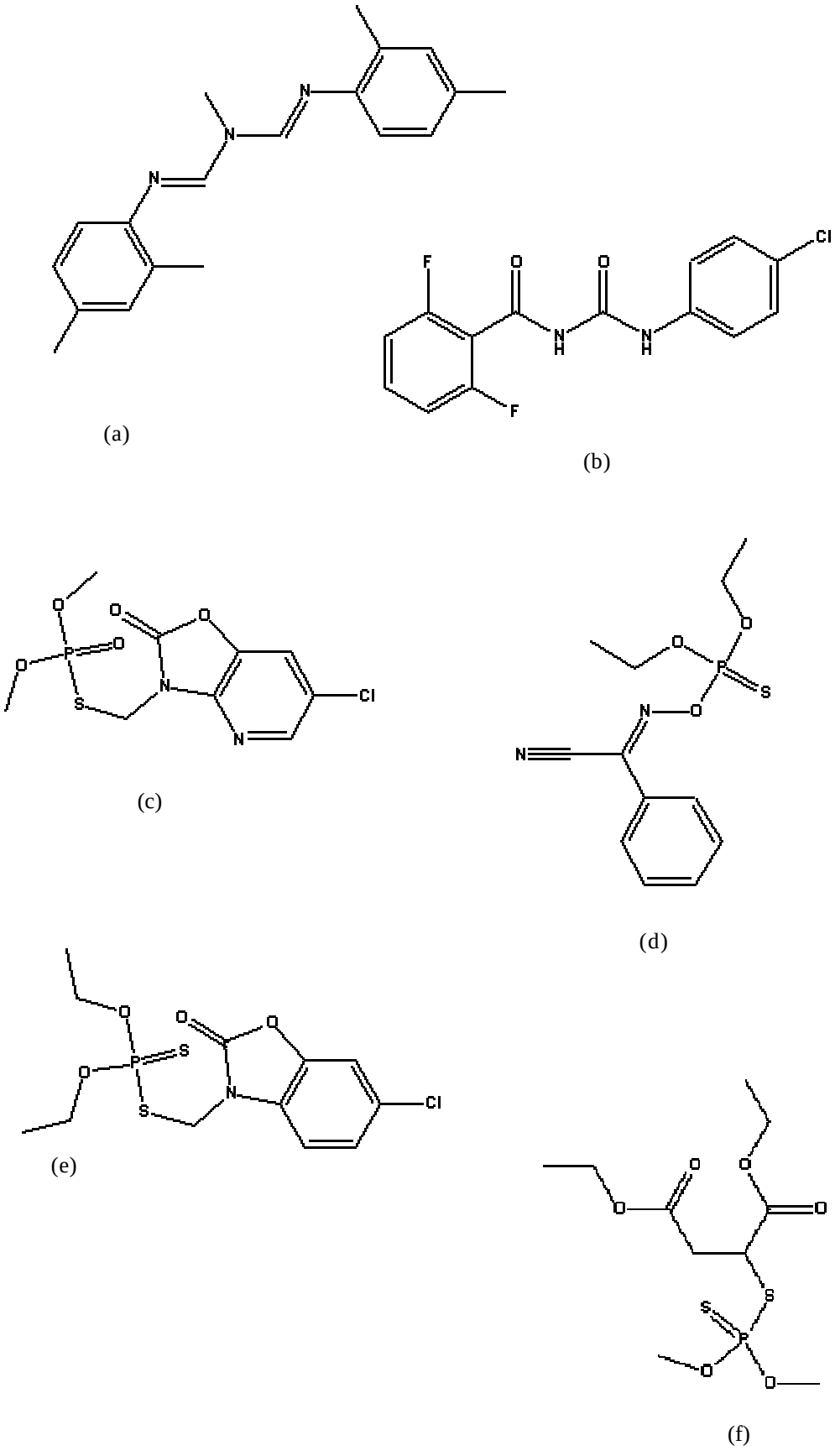


Figure 3
Chemical structure of the insecticide active ingredient amitraz (a), diflubenzuron (b), azamethiphos (c), phoxim (d), phosalone (e) and malathion (f).

2.3.2 Samples

At the time of sampling for the control campaign only one product containing amitraz and product containing phosalone as active ingredient were approved for use in Denmark. Both products were available on the market during the period of the sample collection. Two out of three products containing diflubenzuron were available, four out of seven products containing phoxim were available, twelve out of fourteen products containing azamethiphos were available and all six products containing malathion were available during the sampling period. Generally one sample of each pesticide product was collected. Due to the small amount of active ingredient in the ant boxes more than one box were collected. The samples are listed in Appendix I.

The sample containing phosalone was analysed at NERI in October 2001, phoxim during the period October 2001 - January 2002, malathion in November 2001 - January 2002, diflubenzuron in January 2002, amitraz in February 2002, and the products containing azamethiphos were analysed in January - March 2002.

2.3.3 Results and Discussion

The content of amitraz was determined using gas chromatography with flame ionisation detector (GC-FID) *(Krongaard, 2002f)*. The method is developed based on information from the manufacturer. The CIPAC method is based on packed GC-columns, which we do not use in the laboratory anymore.

The content of diflubenzuron was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2002g)*. The method is developed based on information from the manufacturer.

The content of azamethiphos was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2002h; Krongaard, 2002i)*. Two methods are developed - one method for powder formulations and one method for ant boxes and fly strips. The methods are developed based on information from the manufacturer.

The content of phoxim was determined using reversed phase high performance liquid chromatography and UV-detector, RP-HPLC-UV *(Krongaard, 2002j)*. The method is developed based on information from the manufacturer. The CIPAC method is a rather complex method based on normal phase HPLC.

The content of phosalone was determined using gas chromatography with flame ionisation detector (GC-FID) *(Krongaard, 2002k)*. The method is developed based on the CIPAC method which uses packed GC-columns, which we do not use in the laboratory anymore.

The content of malathion was determined using gas chromatography with flame ionisation detector (GC-FID) *(Krongaard, 2002l)*. The method is developed based on the CIPAC method.

Table 2.4 shows an agreement between declared and determined content made for the sample containing amitraz, phosalone and diflubenzuron, whereas the content of active ingredient in many of the products containing malathion, azamethiphos and phoxim were found to be outside the tolerance.

The products containing malathion can be divided into two categories: 4% powder formulations and app. 45 % EC and EW formulations. The content of active ingredient in the EW and EC formulations is satisfactory, whereas only one out of three powder formulations comply with the accepted tolerance. One of the three products had too high content of malathion and one of the other two products did not contain malathion at all. Subsequent contact to the manufacturer of the former product which had a too high content of malathion did not lead to an explanation. The manufacturer of the product, which does not contain malathion at all, explained that the collected batch was 10 years old and that the malathion is probably evaporated through the paper bag. There is no date of expiry valid on the package.

The products containing azamethiphos may be divided into three categories: 10 % powder formulations, 1% and 7,75 % fly strips/-sheets and 1 % ant boxes. The powder formulations comply with the accepted tolerance with respect to content of azamethiphos. The content corresponded with the declaration , whereas no samples of fly strips/sheets or ant boxes comply with the accepted tolerance with respect to the content of active ingredient. In all these samples the content was found to be too low.

The products containing phoxim can be divided into two categories: 3 % powder formulations and 0,08 % ant boxes. The two samples of ant boxes are samples of the same product, but from different packages. One sample in each category did not comply with the accepted tolerance. In one sample the content of phoxim was found to be too high and in another sample it was found to be too low.

All in all only one out of eleven samples of ant-boxes and fly strips/sheets comply with the accepted tolerance with respect to content of active ingredient.

Table 2.4 Content of active ingredient in samples of insecticides.

Active ingredient	Content				NERI sample no.
	Label claim		Analysis[1)]	Tolerance[2)]	
Amitraz	21,2 %	(200 g/l)	21,80 ± 0,15%	19,9 - 22,5%	2-0036
Phosalone	-	(500 g/l)[3)]	497,8 ± 6,4 g/l	475 - 525 g/l[4)]	01-0563
Diflubenzuron	25 %	-	26,48 ± 0,13 %	23,5 - 26,5 %	01-0156
Diflubenzuron	25 %	-	24,49 ± 0,12 %	23,5 - 26,5 %	01-0207
Malathion	4 %	-	5,75 ± 0,12 %	3,6 - 4,4 %	01-0151*)
Malathion	4 %	-	3,52 ± 0,07 %	3,6 - 4,4%	01-0332
Malathion	4 %	-	< 0,1 %	3,6 - 4,4 %	01-0565*)
Malathion	42 %	(440 g/l)	40,4 ± 1,1 %	39,9 - 44,1%	01-0155
Malathion	45 %	(440 g/l)	46,1 ± 0,9 %	42,75 - 47,25%	01-0154
Malathion	45 %	(450 g/l)	44,0 ± 0,9 %	42,75 - 47,25%	01-0333
Azamethiphos	10 %	-	10,01 ± 0,05 %	9 - 11 %	01-0153
Azamethiphos	10 %	-	10,01 ± 0,05 %	9 - 11 %	01-0567
Azamethiphos	10 %	-	9,96 ± 0, 05 %	9 - 11 %	01-0580
Azamethiphos	7,75 %	10 mg/strip	0,57 ± 0,02 mg/strip	9 - 11 mg/strip	01-0212*)
Azamethiphos	7,75 %	10 mg/strip	3,85 ± 0,12 mg/strip	9 - 11 mg/strip	01-0335*)
Azamethiphos	7,75 %	10 mg/strip	0,59 ± 0,02 mg/strip	9 - 11 mg/strip	01-0566*)
Azamethiphos	7,75 %	10 mg/strip	1,91 ± 0,06 mg/strip	9 - 11 mg/strip	01-0910*)
Azamethiphos	1 %	100 mg/sheet	61,9± 0,9 mg/sheet	85 - 115 mg/sheet	01-0157*)
Azamethiphos	1 %	10 mg /box	1,5 ± 0,1 mg/box	9 - 11 mg/box	01-0211*)
Azamethiphos	1 %	10 mg /box	0,07 ± 0,01 mg/box	9 - 11 mg/box	01-0336*)
Azamethiphos	1 %	10 mg /box	2,64 ± 0,2 mg/box	9 - 11 mg/box	01-0337*)
Azamethiphos	1 %	10 mg /box	0,38 ± 0,03 mg/box	9 - 11 mg/box	01-0909*)
Phoxim	3 %	-	3,11 ± 0,03 %	2,7 - 3,3 %	01-0159
Phoxim	3 %	-	3,43 ± 0,03 %	2,7 - 3,3 %	01-0208
Phoxim	3 %	-	3,89 ± 0,03 %	2,7 - 3,3 %	01-0334*)
Phoxim	0,08 %	-	0,061 ± 0,004 %	0,068 - 0,092 %	01-1666*)
Phoxim	0,08 %	-	0,073 ± 0,004 %	0,068 - 0,092 %	01-1667

1) Mean ± 95% confidence limits.
2) Tolerated limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 2000).*
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.

3 Conclusions

Three different groups of products covered by the pesticide regulation have been included in the 2001 analytical chemical authority control: 1) herbicides containing pendimethalin and methabenzthiazuron 2) Fungicides containing azaconazole, tolylfluanid, propamocarb and cyprodinil 3) Insecticides containing amitraz, diflubenzuron, azamethiphos, malathion, phosalone and phoxim. All products were examined for content of the active ingredients.

Satisfactory results were found among herbicides containing pendimethalin and methabenzthiazuron, among fungicides containing azaconazole, tolylfluanid, propamocarb and cyprodinil, and among insecticides containing amitraz, phosalone and diflubenzuron. Thus, the twelve analysed samples of these pesticides complied with the accepted tolerances with respect to the content of active ingredients as specified per Danish regulation of pesticides.

Two of the six samples containing malathion did not comply with the accepted limits of content of active ingredient. The three EC/EW-formulations did comply with the accepted limits, but two out of three powder-formulations did not. In one of the powder-formulations the content was found to be too high and in the other formulation no malathion was found at all. The manufacturer of the latter product explains that the collected batch is 10 years old and that the malathion is probably evaporated through the paper bag. There is no date of expiry on the package.

Nine of the twelve samples containing azamethiphos did not comply with the accepted limits of content as regards active ingredient. The three powder-formulations did comply with the accepted limits, but all nine ant-boxes and fly strips did not. In all these samples the content was found to be too low.

Two of the five samples containing phoxim did not comply with the accepted limits of active ingredient. One powder-formulation had a too high content and one ant-box had a too low content.

All in all only one out of eleven samples of ant-boxes and fly strips/sheets complied with the accepted tolerance with respect to the content of the active ingredient.

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Appendix I

Samples of pesticides collected from the Danish market in 2001 for authority control

Table 1 Herbicides

Active ingredient	Product	Formulation type[1)]	Company	NERI sample no.
Methabenz-thiazuron	Tribunil	WP	Bayer A/S	01-1568
Pendimethalin	Stomp	SC	BASF A/S	01-1566
Pendimethalin	Inter-Pendimethalin	EC	Inter-Trade A/S	01-1567

1) WP: Wettable powder; SC: suspension concentrate; EC: emulsifiable concentrate.

Table 2 Fungicides

Active ingredient	Product	Formulation type[1)]	Company	NERI sample no.
Azaconazole	Nectec Pasta	PA	Cillus A/S	02-0035
Cyprodinil	Stereo	EC	Novartis Agri A/S	01-1565
Propamocarb	Tattoo	SC	Hoechst Schering AgrEvo A/S	01-0152
Propamocarb	Previcur N	SL	Hoechst Schering AgrEvo A/S	01-0564
Tolylfluanid	Euparen Multi	SG	Bayer A/S	01-1569

1) PA: Paste; EC: emulsifiable concentrate; SC: suspension concentrate; SL: soluble concentrate; SG: water soluble granule.

Table 3 Insecticides

Active ingredient	Product	Formulation type[1)]	Company	NERI sample no.
Amitraz	Mitac 20 EC Dist.: Grøn Plantebeskyttelse	EC	Aventis Crop Science A/S	2-0036
Azamethiphos	Alficron Plus	WP	Novartis Agri A/S	01-0153
Azamethiphos	LFS Azamethiphos	WP	LFS Kemi	01-0567
Azamethiphos	AZA Fluesmøremiddel Dist.: Mortalin A/S	WP	Novartis Agri A/S	01-0580
Azamethiphos	Trinol Strips Dist.: Trinol A/S	CL	Novartis Agri A/S	01-0212
Azamethiphos	Kvit Fluestrips Dist.: Bayer A/S	CL	Novartis Agri A/S	01-0335
Azamethiphos	Bonus Fluestrips Dist.: F.S. Import	CL	Novartis Agri A/S	01-0566
Azamethiphos	Linds Fluetape Dist.: Linds Handelsselskab	CL	Novartis Agri A/S	01-0910
Azamethiphos	Snip Flueplader Dist.: Ciba Geigy A/S	CL	Novartis Agri A/S	01-0157
Azamethiphos	Myrelokkedåse Dist.: Matas	CL	Novartis Agri A/S	01-0211
Azamethiphos	Myrelokkedåse Dist.: Tanaco Danmark A/S	CL	Novartis Agri A/S	01-0336
Azamethiphos	Bonus Myrelokkedåse Dist.: F.S.Import	CL	Novartis Agri A/S	01-0337
Azamethiphos	Myrelokkedåse Dist.: Linds Handelsselskab	CL	Novartis Agri A/S	01-0909
Diflubenzuron	Dimilin	WP	KVK Agro A/S	01-0156
Diflubenzuron	Trinol	WP	KVK Agro A/S	01-0207
Malathion	Maladan	CP	Bayer A/S	01-0151
Malathion	Malathion insektpudder	CP	Tanaco Danmark A/S	01-0332
Malathion	Malathion pudder	CP	G.F.Agro A/S	01-0565
Malathion	Maladan 44 EW	EW	Bayer A/S	01-0155
Malathion	DLG Malathion 45	EC	AgroDan A/S	01-0154
Malathion	Tanaco Malathion	EC	Tanaco Danmark A/S	01-0333
Phosalone	Zolone Flo	SC	Rhone Poulenc	01-0563
Phoxim	Baythion	WP	Bayer A/S	01-0159
Phoxim	Myremiddel til vanding Dist.: Matas	WP	Bayer A/S	01-0208
Phoxim	Bonus Myrepulver	WP	LFS Kemi	01-0334
Phoxim	Baygon	CL	Bayer A/S	01-1666
Phoxim	Baygon	CL	Bayer A/S	01-1667

1) EC: emulsifiable concentrate; WP: Wettable powder; CL: contact liquid or gel; CP: contact powder; EW: emulsion, oil in water, SC: suspension concentrate

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Addresses:

URL: http://www.dmu.dk

National Environmental Research Institute
Frederiksborgvej 399
PO Box 358
DK-4000 Roskilde
Denmark
Tel: +45 46 30 12 00
Fax: +45 46 30 11 14

Management
Personnel and Economy Secretariat
Research and Development Section
Department of Policy Analysis
Department of Atmospheric Environment
Department of Marine Ecology
Department of Environmental Chemistry and Microbiology
Department of Arctic Environment
Project Manager for Quality Management and Analyses

National Environmental Research Institute
Vejlsøvej 25
PO Box 314
DK-8600 Silkeborg
Denmark
Tel: +45 89 20 14 00
Fax: +45 89 20 14 14

Environmental Monitoring Co-ordination Section
Department of Terrestrial Ecology
Department of Freshwater Ecology
Project Manager for Surface Waters

National Environmental Research Institute
Grenåvej 12-14, Kalø
DK-8410 Rønde
Denmark
Tel: +45 89 20 17 00
Fax: +45 89 20 15 15

Department of Landscape Ecology
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