Ministry of Environment and Energy
National Environmental Research Institute

Control of Pesticides 1998

Chemical Substances and Chemical Preparations

NERI Technical Report No. 281

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1999

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Abstract: Two different groups of products covered by the pesticide regulations have been included in the authority control for 1998: 1) insecticides/acaricides containing dimethoate or tau-fluvalinate as active ingredients, 2) fungicides containing chlorothalonil or tebuconazole as active ingredients.

Products containing either tau-fluvalinate or chlorothalonil as active ingredients complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides. Four of the six examined samples of products containing dimethoate and three of the nine examined samples of products containing tebuconazole as active ingredients did not comply with the accepted tolerance. The content of different impurities in a technical material of dimethoate comply with the specifications.

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Contents

Summary

The analytical chemical authority control on pesticide products on the Danish market performed in 1998 is reported. Samples of selected groups of pesticides have been collected from the market and analysed to verify whether the actual content of active ingredient agreed with the label-claimed content. Furthermore a technical grade active ingredient was analysed for impurities to see if they complied with the specifications. The tolerance of deviation of active ingredient content from label-claimed content are set by the Danish pesticide regulation.

Two different groups of products covered by the pesticide regulation have been included in the 1998 analytical chemical authority control: 1) insecticides/acaricides containing dimethoate and tau-fluvalinate as active ingredients, 2) fungicides containing chlorothalonil and tebuconazole as active ingredients.

Satisfactory results were found among products containing the active ingredients tau-fluvalinate and chlorothalonil. Thus, the three examined samples of these pesticides complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides.

Four of the six examined samples of products containing dimethoate as active ingredient did not comply with the accepted tolerance with respect to content of active ingredient, the content of dimethoate being too low possibly due to degradation of the active ingredient in the product. The content of different impurities in a technical material of dimethoate complied with the specifications.

Three of the nine examined samples of products containing tebuconazole as active ingredient did not comply with the accepted tolerance with respect to content of active ingredient, the content of tebuconazole being too low in one sample and too high in two samples. Difficulties with sample collection and settling of the tebuconazole could be an explanation for the high content of tebuconazole in the latter samples.

Resumé

Den analytisk kemiske kontrol af pesticidprodukter på det danske marked udført i 1998 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 aktivstof er i overensstemmelse med det deklarerede indhold. Derudover er et teknisk aktivstof analyseret for urenheder for at se om det overholdt specifikationen. Grænsen for en accepteret afvigelse af indholdet af aktivstof fra det deklarerede indhold er fastsat i bekendtgørelsen om bekæmpelsesmidler.

To forskellige grupper af produkter er inkluderet i den analytisk-kemiske kontrol udført af myndighederne i 1998: 1) insekticider/acaricider indeholdende dimethoat og tau-fluvalinat som aktivstoffer. 2) fungicider indeholdende chlorothalonil og tebuconazol som aktivstoffer.

Der blev opnået tilfredsstillende resultater blandt produkter indeholdende tau-fluvalinat og chlorothalonil. Indholdet af aktivstof for de tre undersøgte prøver af disse bekæmpelsesmidler var indenfor den accepterede tolerance, der er fastsat i bekendtgørelsen om bekæmpelsesmidler.

I fire ud af seks undersøgte prøver af produkter indeholdende dimethoat som aktivstof var indholdet af dimethoat lavere end den accepterede tolerance, hvilket kan skyldes nedbrydning af aktivstoffet i produktet. Indholdet af forskellige urenheder i et teknisk materiale af dimethoat var alle indenfor specifikationerne.

I tre ud af ni undersøgte prøver af produkter indeholdende tebuconazol som aktivstof var indholdet af tebuconazol udenfor den accepterede tolerance. Indholdet var for lavt i én prøve og for højt i to andre. Da tebuconazol danner en overfladefilm og der samtidig var vanskeligheder med prøveudtagningen kan det være en forklaring på det høje indhold af tebuconazol i de to sidstnævnte prøver.

1 Introduction

In Denmark the Danish Environmental Protection Agency (DEPA) is responsible for evaluation and authorisation of all pesticides before introduction on the Danish market. Legal regulations for pesticides are given in a Statutory Order from the Ministry of the Environment and Energy *(Miljø- og Energiministeriet, 1998)*, which also states that DEPA is responsible for control in relation to pesticides.

In practice authority control activities of pesticides on the market are organised in a way that 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 control whether the products comply with regulation as well as with the specification of contents stated in connection with application for approval of the pesticide product.

Analytical chemical control can involve verification of content of active ingredient as well as content of auxiliary matters or levels of impurities.

Laboratory control work covers two types of projects: 1) Ordinary control in the form 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 needed 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 control i.e., campaigns, is covered by this report, which describes the laboratory control performed in 1998.

2 Control Campaigns in 1998

Control campaigns conducted in 1998 have covered pesticides belonging to two different groups of pesticides: insecticides/acaricides and fungicides. The analytical chemical control has aimed at examining the content of active ingredient compared to that stated on the label. In one of the campaigns a control of the levels of different impurities in the pesticide active ingredient has been included too. Regulation in Denmark *(Miljø- og Energiministeriet, 1998)* specifies general tolerance of deviations from declared content. These are given in Table 2.1.

Samples of the various pesticides covered in the 1998 control campaigns have been collected by the Chemical Inspection Service at DEPA during the months January - November 1998 either at whole sale dealers/-importers or at retailers. One sample of each product has been collected.

Samples have been stored at NERI in the 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**
• 50	± 2.5% (abs.)
25 < X • 50	± 5% (rel.)
10 < X • 25	± 6% (rel.)
2.5 < X • 10	± 10% (rel.)
• 2.5	± 15% (rel.)

2.1 Insecticides/acaricides

2.1.1 Introduction

Among the different insecticides/acaricides available on the Danish market insecticides/acaricides containing either tau-fluvalinate or dimethoate as active ingredients were selected for control in 1998.

Tau-fluvalinate (Figure 1, top) is a pyrethroid insecticide which in Denmark is used only for control of insects in cereal crops and in seed-growing of crucifera. Tau-fluvalinate is a new insecticide (introduced on the Danish market in 1996). Formulations containing tau-fluvalinate have not previously been selected for authority control.

Dimethoate (Figure 1, bottom) is an organophosphorus type of insecticide and acaricide, which is used for control of certain insects on plants and for control of flies in animal houses. Insecticide/acaricide formulations containing dimethoate have not previously been selected for authority control.

In connection with the conducted control of content of active ingredient in dimethoate formulations, an investigation of the level of six different impurities in technical grade dimethoate has further been carried out.

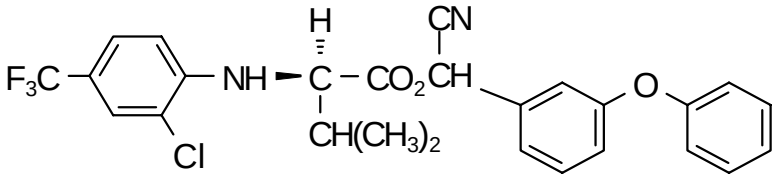


Figure 1
Chemical structure of the insecticide/acaricide active ingredients tau-fluvalinate (top) and dimethoate (bottom).

2.1.2 Samples

At the time of sampling for the control campaign (January - July 1998) only one product containing tau-fluvalinate was approved for use in Denmark. It was available on the market during the period of the sample collection. nine different products containing dimethoate were approved for use in Denmark. six of these products were approved as insecticides and six were approved as acaricides (*Miljøstyrelsen, 1998*). six of these products were available on the market during the period of the sampling. Technical grade dimethoate to impurity control was collected from one manufacturer. One sample of each pesticide product was collected. A list of the samples is given Appendix I.

The sample containing tau-fluvalinate was analysed at NERI during May-June 1998. The samples containing dimethoate were analysed at NERI in the period October 1998-January 1999.

2.1.3 Results and Discussion

The contents of tau-fluvalinate were determined using reversed phase high performance liquid chromatography (RP-HPLC) with methods *(Krongaard, 1998a)* based on information from the manufacturer on method of analysis.

Similarly, contents of dimethoate, were determined using reversed phase high performance liquid chromatography (RP-HPLC) with a method *(Krongaard, 1999a)* based on information from the manufacturer on method of analysis. Results from the analyses are shown in Table 2.2.

Table 2.2. Content of active ingredient in samples of insecticides/acaricides.

Active ingredient	**Content**				**NERI sample no.**
	Label claim		**Analysis[1)]**	**Tolerance[2)]**	
tau-fluvalinate		240 g/l[3)]	238,9 ± 0,8 g/l (22,1%)	226 - 254 g/l[4)]	8-0092
dimethoate	37,6%	(400 g/l)	31,1 ± 0,2%[*)]	35,7 - 39,5%	8-0053
dimethoate	45,7%	(500 g/l)	44,7 ± 0,3%	43,4 - 48,0%	8-0054
dimethoate	27,5%	(280 g/l)	24,7 ± 0,2%[*)]	26,1 - 28,9%	8-0095
dimethoate	38%	(400 g/l)	35,7 ± 0,2%[*)]	36,1 - 39,9%	8-0096
dimethoate	38%		33,4 ± 0,2%[*)]	36,1 - 39,9%	8-0097
dimethoate	38%	(400 g/l)	38,1 ± 0,3%	36,1 - 39,9%	8-0345

1) Mean (minimum duplicate determinations) ± 95% confidence limits.
2) Tolerated limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 1998).*
3) Content (expressed as %) not declared.
4) Calculated on basis of declared content in g/l.
*) Found content is outside the accepted tolerance.

As apparent from Table 2.2 good agreement between declared and determined content was found for the sample containing tau-fluvalinate. Hence, the sample complied with the tolerated limits for content of active ingredient.

There was not a good agreement between declared and determined content for samples containing dimethoate. Four out of six of the analysed samples containing dimethoate did not comply with the accepted tolerance of content of active ingredient. With respect to the samples of formulations exhibiting too low content of dimethoate a possible explanation could be a limited stability of the dimethoate. The samples which did not comply with tolerated limits of contents were all produced 4-6 years ago.

The contents of different impurities in technical grade dimethoate were also determined using reversed phase high performance liquid chromatography (RP-HPLC) with methods *(Krongaard, 1998b; Krongaard, 1998c)* based on information from the manufacturer on method of analysis.

All investigated impurities were below the specification. The impurities and the contents are confidential information

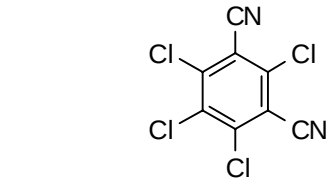
2.2 Fungicides

2.2.1 Introduction

In 1998 29 different fungicide active ingredients were approved in Denmark *(Miljøstyrelsen, 1998)*. Among these active ingredients products containing chlorothalonil and tebuconazole were selected for control in 1998.

Chlorothalonil (Figure 2, top) is used for control of many fungal diseases on wheat, potatoes, peas, onions, strawberries, cucumbers, and ornamentals. Formulations containing chlorothalonil have not been selected for authority control earlier.

Tebuconazole (Figure 2, bottom) is an azole type of fungicide which in Denmark is used only as seed dressing and wood preservatives controlling many fungal diseases. Tebuconazole is a new fungicide (introduced on the Danish market in 1998) and formulations containing tebuconazole have not previously been selected for authority control.



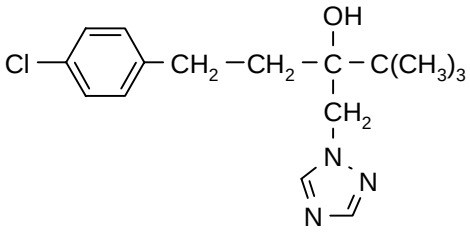


Figure 2
Chemical structure of the fungicide active ingredient chlorothalonil (top) and tebuconazole (bottom)

2.2.2 Samples

At the time of sampling for the control campaign (January - November 1998) two products containing chlorothalonil as active ingredient were approved for use in Denmark. Both of them were available on the market during the sampling period. Ten products containing tebuconazole as active ingredient were approved for use in Denmark, two of them as seed dressing and eight of them as wood preservatives. Nine of ten products containing tebuconazole as active ingredient were available on the market during the period of the sampling. One sample of each fungicide product was collected. Samples of five products (8-0347, 8-0348, 8-0349, 8-0564 and 8-0565) were collected by the Chemicals Inspections Service at the manufacturing company from containers.

Samples of two products (8-0610 and 8-0611) were collected by the user, a compregnating plant, as the products are delivered directly from road tankers to a closed process plant. Information about the samples are given in Appendix I.

The samples containing chlorothalonil were analysed at NERI in the period December 1998 - January 1999. The samples containing tebuconazole were analysed at NERI in the period October 1998 - January 1999.

2.2.3 Results and Discussion

The content of chlorothalonil was determined using gas chromatography with flame ionisation detection (GC-FID) with a method *(Krongaard, 1999b)* based on information from the manufacturer on method of analysis.

Similarly, the content of tebuconazole was determined using gas chromatography with flame ionisation detection (GC-FID) with a method *(Krongaard, 1999c)* based on information from the manufacturer on method of analysis. Results from the analyses are shown in Table 2.3.

As apparent from Table 2.3 good agreement between declared and determined content was found for samples containing chlorothalonil. Hence, the samples complied with the tolerated limits for content of active ingredient.

Three of nine analysed samples containing tebuconazole did not comply with the accepted limits of content of active ingredient. One sample was too low in content and two samples were too high in content. The latter two are the samples collected by the user at a compregnating plant. The manufacturer had informed that: "…if (the products are) left to stand for some time there can be some slight settlement of the tebuconazole emulsion component on the surface of the product. This is easily and rapidly redispersed during any mechanical movement and is therefore not an issue during normal treatment plant operation...". Because of the closed process plant it is difficult to collect a representative sample. The difficulties with the sample collection and the settling of the tebuconazole could be plausible explanations for the high content of tebuconazole in the samples.

Table 2.3. Content of active ingredient in samples of fungicides.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis[1)]	Tolerance[2)]	
chlorothalonil	500 g/l[3)]	494,9 ± 0,6 g/l (39,8%)	475 - 525 g/l[4)]	8-0055
chlorothalonil	500 g/l[3)]	492,7 ± 0,6 g/l (39,4%)	475 - 525 g/l[4)]	8-0275
tebuconazole	25% (250 g/l)	25,94 ± 0,05%	23,5 - 26,5%	8-0270
tebuconazole	0,2% (1,60 g/l)	0,204 ± 0,001%	0,17 - 0,23%	8-0347
tebuconazole	1,0% (10,10 g/l)	1,037 ± 0,002%	0,85 - 1,15%	8-0348
tebuconazole	0,3% (3,03 g/l)	0,317 ± 0,001%	0,26 - 0,35%	8-0349
tebuconazole	1,52% (15 g/l)	1,407 ± 0,003%	1,29 - 1,75%	8-0564
tebuconazole	0,37% (2,9 g/l)	0,373 ± 0,001%	0,31 - 0,43%	8-0565
tebuconazole	0,93% (7,4 g/l)	*) 0,563 ± 0,001%	0,79 - 1,07%	8-0566
tebuconazole	0,45%	*) 0,558 ± 0,015%	0,38 - 0,52%	8-0610
tebuconazole	0,24%	*) 0,300 ± 0,008%	0,20 - 0,28%	8-0611

1) Mean (minimum duplicate determination) ± 95% confidence limits.
2) Tolerated limits for content of active ingredients according to Danish regulations *(Miljø- og Energiministeriet, 1998).*
3) Content (expressed as %) not declared.
4) Calculated on basis of declared content in g/l.
*) Found content is outside the accepted tolerance.

3 Conclusions

Two different groups of products covered by the pesticide regulation have been included in the 1998 analytical chemical authority control: 1) insecticides/acaricides containing dimethoate and tau-fluvalinate as active ingredients, 2) fungicides containing chlorothalonil and tebuconazole as active ingredients. Furthermore a sample of technical grade dimethoate was analysed for impurities to see if they complied with the specifications.

Satisfactory results were found among products containing the active ingredients tau-fluvalinate and chlorothalonil. Thus, the three examined samples of these pesticides complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides.

Four of the six examined samples of products containing dimethoate as active ingredient did not comply with the accepted tolerance with respect to content of active ingredient, the content of dimethoate being too low. Subsequent contact with the manufacturers of the products revealed that these products were 4-6 years old. The content of different impurities in a technical material of dimethoate comply with the specifications.

Three of the nine examined samples of products containing tebuconazole as active ingredient did not comply with the accepted tolerance with respect to content of active ingredient, the content of tebuconazole being too low in one sample and too high in two samples. Difficulties with sample collection and settling of the tebuconazole could be plausible explanations for the high content of tebuconazole in the latter samples. According to the manufacturer it is not an issue during normal treatment plant operations due to the easy and rapid redispersion during any mechanical movement.

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Appendix I

Samples of pesticides collected from the Danish market in 1998 for authority control

1 Insecticides

Product	Active ingredient(s)	Formulation type[1)]	Company	NERI sample no.
	dimethoate	TC	Cheminova A/S	41-36
Roxion 40 EC	dimethoate	EC	Shell Chemicals A/S	8-0053
Perfekthion 500 S	dimethoate	EC	BASF A/S	8-0054
Mavrik 2 F	tau-fluvalinate	SC	Sandoz A/S	8-0092
DLG Dimethoate 28	dimethoate	EC	Esbjerg Kemi A/S	8-0095
KVK Dimethoate	dimethoate	EC	KVK Agro A/S	8-0096
Perfekthion EC	dimethoate	EC	BASF A/S	8-0097
Danadim TM	dimethoate	EC	Cheminova A/S	8-0345

1) EC: emulsifiable concentrate; SC: suspension concentrate; TC: technical material.

2 Fungicides

Product	Active ingredient	Formulation type[1)]	Company	NERI sample no.
Daconil 500 F	chlorothalonil	SC	BASF A/S	8-0055
Folicur EW	tebuconazole	EW	Bayer A/S	8-0270
Bravo 500 F	chlorothalonil	SC	Ciba-Geigy A/S	8-0275
Gori Vac TH 92	tebuconazole	EC	S. Dyrup & Co. A/S	8-0347
Pres 10 Gori	tebuconazole	EC	S. Dyrup & Co. A/S	8-0348
Gori 356	tebuconazole	EC	S. Dyrup & Co. A/S	8-0349
Raxil IM 035 ES	tebuconazole	ES	Bayer A/S	8-0564
Resistol	tebuconazole	EC	A/S Hygæa	8-0565
Secu type 010	tebuconazole	AL	Larco Farve- & lakfabrik	8-0566
Tanalith E 3491	tebuconazole	EC	Hickson Timber Products Ltd.	8-0610
Tanalith E 3492	tebuconazole	EC	Hickson Timber Products Ltd.	8-0611

1) AL: any other liquid; EC: emulsifiable concentrate; ES: emulsion for seed treatment; SC: suspension concentrate.

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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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Two different groups of products covered by the pesticide regulations have been included in the authority control for 1998: 1) insecticides/ acaricides containing dimethoate or tau-fluvalinate as active ingredients, 2) fungicides containing chlorothalonil or tebuconazole as active ingredients.

Products containing either tau-fluvalinate or chlorothalonil as active ingredients complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides. Four of the six examined samples of products containing dimethoate and three of the nine examined samples of products containing tebuconazole as active ingredients did not comply with the accepted tolerance. The content of different impurities in a technical material of dimethoate comply with the specifications.

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