

Chemical Substances and
Chemical Preparations

Control of Pesticides 1997

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Abstract: Three different groups of products covered by the pesticide regulations have been included in the authority control for 1997: 1) herbicides containing phenmedipham, desmedipham, ethofumesate, triflurosulfuron-methyl and/or propaquizafop as active ingredients, 2) fungicides containing penconazole as active ingredient and 3) slimicides containing 2,2-dibrom-2-cyanoacetamide as active ingredient. Products containing either phenmedipham, desmedipham, triflurosulfuron-methyl and/or propaquizafop as active ingredients complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides. One of the 11 examined samples of products containing ethofumesate and the examined samples of the slimicide product containing 2,2-dibrom-2-cyanoacetamide as active ingredient did not comply with the accepted tolerance.

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Summary

The analytical chemical authority control performed in 1997 on pesticide products on the Danish market 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. The tolerated limits for deviation of active ingredient content from label-claimed content are set by the Danish pesticide regulations.

Three different groups of products covered by the pesticide regulations have been included in the authority control for 1997: 1) herbicides containing phenmedipham, desmedipham, ethofumesate, triflurosulfuron-methyl and/or propaquizafop as active ingredients, 2) fungicides containing penconazole as active ingredient and 3) slimicides containing 2,2-dibrom-2-cyanoacetamide as active ingredient.

Satisfactory results were found among products containing either phenmedipham, desmedipham, triflurosulfuron-methyl and/or propaquizafop as active ingredient. All 17 examined samples of these pesticides complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides.

One of the 11 examined samples of products containing ethofumesate as active ingredient did not comply with the accepted tolerance, the content of ethofumesate being too low.

Only one product containing 2,2-dibrom-2-cyanoacetamide was approved to the Danish market. The analysed samples did not comply with the accepted tolerance. In one case possible due to an inappropriate storage.

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 (*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 organized 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 regulations as well as with the specification of contents supplied 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, campaigns, is covered by this report, which describes the laboratory control performed in 1997.

2 Control Campaigns in 1997

Control campaigns conducted in 1997 have covered pesticides belonging to three different groups of pesticides: herbicides, fungicides and slimicides. All analytical chemical control has aimed at examining the content of active ingredient compared to the stated content on the label. Regulation in Denmark (*Miljø- og Energiministeriet, 1998*) specifies generally tolerated limits of deviation from declared content. These are given in Table 2.1.

Samples of the various pesticides covered in the 1997 control campaigns have been collected by the Chemical Inspection Service at DEPA during the months February - May 1997 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 room temperature and protected from light.

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

Content of a.i., %, w/w	Tolerated limits
≥ 50	$\pm 2.5\%$ (abs.)
$25 < X \leq 50$	$\pm 5\%$ (rel.)
$10 < X \leq 25$	$\pm 6\%$ (rel.)
$2.5 < X \leq 10$	$\pm 10\%$ (rel.)
≤ 2.5	$\pm 15\%$ (rel.)

2.1 Herbicides

2.1.1 Introduction

Among the many different herbicides available on the Danish market herbicides containing either phenmedipham, desmedipham, ethofumesate, triflusaluron-methyl or propaquizafop as active ingredients were selected for control in 1997.

Phenmedipham (Figure 1, I) is a bis-carbamate herbicide, which is used for control of most broad-leaved weeds in beet crops, especially sugar beet. It is also used on strawberries. Before 1997 herbicide formulations containing phenmedipham have not been selected for authority control since 1985.

Desmedipham (Figure 1, II) is like phenmedipham a bis-carbamate herbicide, which is used for control of most broad-leaved weeds in beet crops, especially sugar beet. It is also used on strawberries. Desmedipham is usually sprayed in combination with phenmedipham and ethofumesate. Desmedipham was introduced on the Danish market in 1991. Formulations containing desmedipham have not previously been selected for authority control.

Ethofumesate (Figure 1, III) is a benzofuranyl alkanesulfonate type of herbicide, which is used for control of a wide range of broad-leaved weeds in beet crops. It is also used on strawberries. Formulations containing ethofumesate have not previously been selected for authority control.

Triflusaluron-methyl (Figure 1, IV) is a sulfonylurea herbicide, which is used for control of many annual and perennial broad-leaved weeds in sugar beet. Triflusaluron-methyl is a new herbicide (introduced on the Danish market in 1996). Formulations containing triflusaluron-methyl have not previously been selected for authority control.

Propaquizafop (Figure 1, V) is a phenoxypropionic acid herbicide, which is used for control of many annual and perennial grasses in sugar beet, rape, peas and potatoes. Propaquizafop is a new herbicide (introduced on the Danish market in 1995). Formulations containing propaquizafop have not previously been selected for authority control.

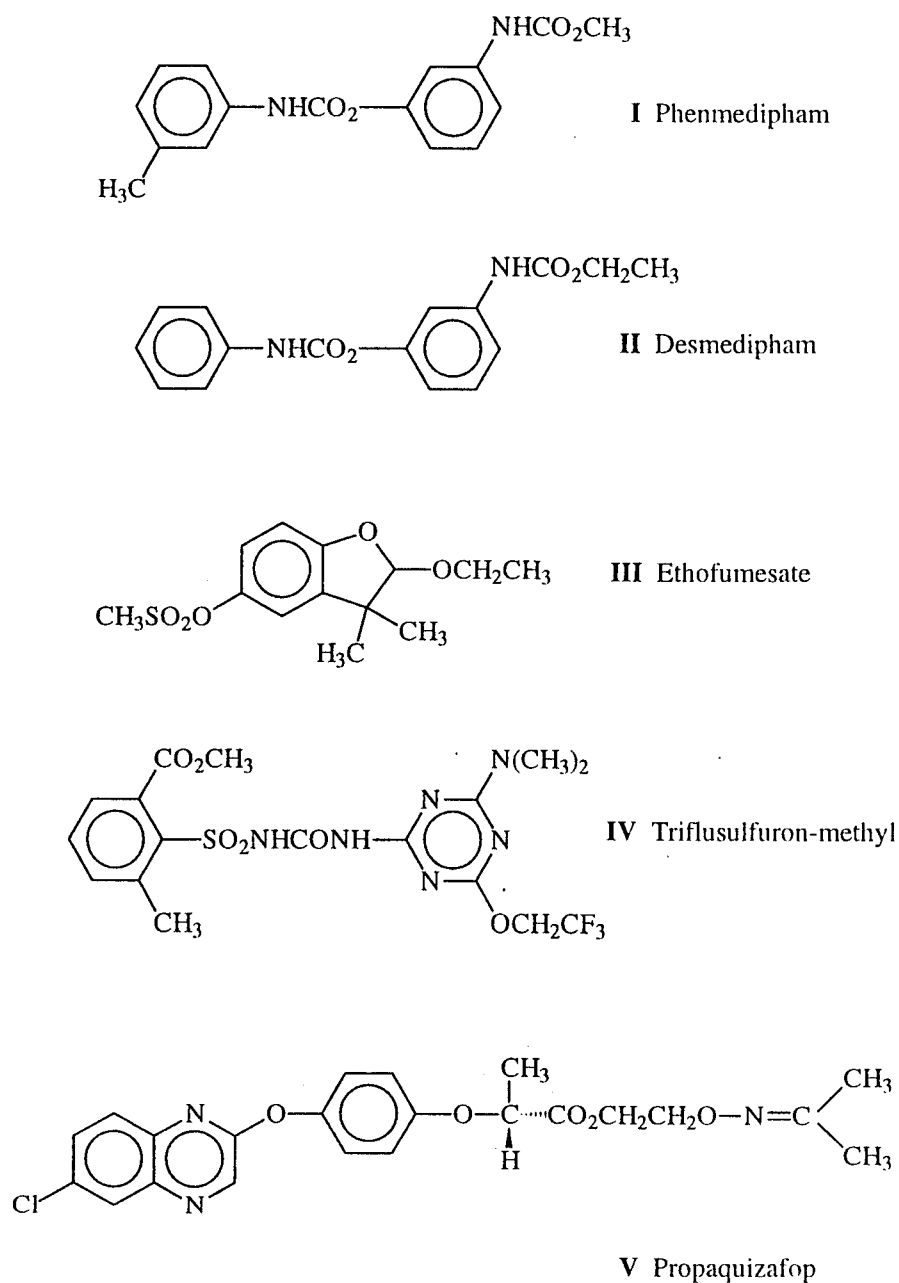


Figure 1. Chemical structure of the herbicide active ingredients phenmedipham (I), desmedipham (II), ethofumesate (III), triflurosulfuron-methyl (IV) and propaquizafop (V).

2.1.2 Samples

At the time of sample collection for the control campaign (February - May 1997) 18 different products containing phenmedipham, ethofumesat and/or desmedipham (6 of the 18 products were mixtures of two or three of the herbicides) were approved for use in Denmark. 16 of these products and 3 products withdrawn from the market, but still available in retail, were available on the market during the period of the sample collection. Two products containing propaquizafop and one product containing triflurosulfuron-methyl were approved for use in Denmark. They were all available on the market during the period of the sample collection. One sample of each pesticide product was collected. A list of the samples is given Appendix I.

The samples containing phenmedipham, desmedipham and/or ethofumesat were analysed at NERI in the period August-October 1997. The sample containing propaquizafop was analysed at NERI in September 1997. The sample containing triflurosulfuron-methyl was analysed at NERI in the period February-March 1998.

2.1.3 Results and Discussion

The contents of phenmedipham, desmedipham and ethofumesate, were determined using reversed phase high performance liquid chromatography (RP-HPLC) with a method (*Petersen, 1997*) based on information from the manufacturer company on method of analysis. The method allows simultaneous determination of all three compounds.

Similarly, contents of propaquizafop and triflurosulfuron-methyl were determined using reversed phase high performance liquid chromatography (RP-HPLC) with methods (*Køppen, 1997; Petersen, 1998a*) based on information from the manufacturer company on method of analysis. Results from the analyses are shown in Table 2.2.

As it appears from Table 2.2 one of the analysed samples (no. 7-0125 - a product containing ethofumesate) did not comply with the tolerated limits of content of active ingredient. For all the other samples a good agreement was found between declared and actual contents.

Subsequent contact to the manufacturer company of the product of which the sample was found to have too low content of ethofumesate revealed an incorrect label. The declared content was 46%, whereas it correctly should have been 45%. The label has accordingly been corrected by the company.

Table 2.2. Content of active ingredient in samples of herbicides.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
ethofumesate	500 g/l (44,3%)	500±5 g/l (44,6%)	42,1 - 46,5 %	7-0124
ethofumesate	500 g/l (46%)	455±5 g/l ¹⁾ (42,8%)	43,7 - 48,3 %	7-0125
ethofumesate	500 g/l ⁻³⁾	491±5 g/l (43,3%)	475 - 525 g/l ⁴⁾	7-0126
ethofumesate	200 g/l ⁻³⁾	205±2 g/l (21,4%)	188 - 212 g/l ⁴⁾	7-0127
ethofumesate	500 g/l (44,3%)	489±5 g/l (43,4%)	42,1 - 46,5 %	7-0520
ethofumesate phenmedipham	100 g/l (10,3%) 80 g/l (8,2%)	102±1 g/l (10,4%) 81,7±0,8 g/l (8,31%)	9,7 - 10,9 % 7,4 - 9,0 %	7-0128
ethofumesate phenmedipham	100 g/l (10,3%) 80 g/l (8,2%)	98,4±1 g/l (9,92%) 79,8±0,8 g/l (8,04%)	9,7 - 10,9 % 7,4 - 9,0%	7-0129
ethofumesate phenmedipham	200 g/l (17,5%) 320 g/l (28%)	194±2 g/l (17,4%) 306±3 g/l (27,5%)	16,4 - 18,6 % 26,6 - 29,4 %	7-0131
ethofumesate phenmedipham desmedipham	115 g/l ⁻³⁾ 75 g/l ⁻³⁾ 15 g/l ⁻³⁾	114±1 g/l (11,2%) 72,4±0,7 g/l (7,14%) 12,9±0,1 g/l (1,27%)	108 - 122 g/l ⁴⁾ 67,5 - 82,5 g/l ⁴⁾ 12,8 - 17,2 g/l ⁴⁾	7-0133
ethofumesate phenmedipham desmedipham	128 g/l (12,2%) 62 g/l (5,9%) 16 g/l (1,5%)	129±1 g/l (12,3%) 62,0±0,6 g/l (5,93%) 16,1±0,2 g/l (1,54%)	11,5 - 12,9 % 5,3 - 6,5 % 1,3 - 1,7 %	7-0134
ethofumesate phenmedipham desmedipham	115 g/l (11,3%) 75 g/l (7,4%) 15 g/l (1,5%)	114±1 g/l (11,3%) 76,0±0,8 g/l (7,51%) 14,9±0,2 g/l (1,47%)	10,6 - 12,0 % 6,7 - 8,1 % 1,3 - 1,7 %	7-0135
phenmedipham	157 g/l ⁻³⁾	167±2 g/l (16,8%)	147 - 167 g/l ⁴⁾	7-0136
phenmedipham	160 g/l (16%)	163±2 g/l (16,0%)	15,0 - 17,0 %	7-0137
phenmedipham	167 g/l (16,7%)	171±2 g/l (17,2%)	15,7 - 17,7 %	7-0138
phenmedipham	160 g/l (16%)	160±2 g/l (15,8%)	15,0 - 17,0 %	7-0139
phenmedipham	160 g/l (16%)	159±2 g/l (16,0%)	15,0 - 17,0 %	7-0184
phenmedipham	160g/l (15,8%)	159±2 g/l (15,8%)	14,9 - 16,7 %	7-0185
phenmedipham	160 g/l ⁻³⁾	158±2 g/l (16,0%)	150 - 170 g/l ⁴⁾	7-0186
propaquizafop	100 g/l ⁻³⁾	103,4 ± 0,6 g/l	90 - 110 g/l ⁴⁾	7-0140
propaquizafop	100 g/l ⁻³⁾	103,6 ± 0,6 g/l	90 - 110 g/l ⁴⁾	7-0521
triflusulfuron-methyl	50 %	49,37 ± 0,31 %	47,5 - 52,5 %	7-0900

- 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.

2.2 Fungicides

2.2.1 Introduction

In 1997 28 different fungicide active ingredients were approved to the market in Denmark (*Miljøstyrelsen, 1997*). Among the many formulations containing these active ingredients, only products containing penconazole were selected for control in 1997.

Penconazole (Figure 2, VI) is an azole type of fungicide, which is used for control of powdery mildew, pome fruit scab etc. on ornamentals. Penconazole was introduced on the Danish market in 1993, and formulations containing penconazole have not been selected for authority control earlier.

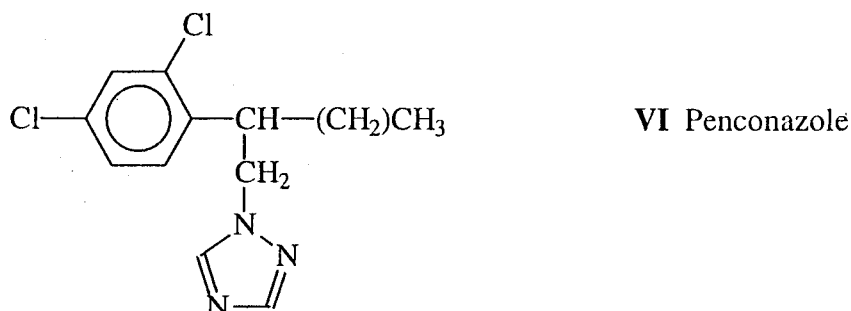


Figure 2. Chemical structure of the fungicide active ingredient penconazole (VI)

2.2.2 Samples

At the time of sample collection for the control campaign (February - May 1997) only one single product containing penconazole as active ingredient was approved for use in Denmark (*Miljøstyrelsen, 1997*). Only one sample of the fungicide product was collected. Information about the sample is given in Appendix I. The sample was analysed at NERI in January 1998.

2.2.3 Results and Discussion

The content of penconazole was determined using reversed phase high performance liquid chromatography (RP-HPLC) with a method (*Petersen, 1998b*) based on information from the manufacturer company 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. Hence, the sample complied with the tolerated limits for content of active ingredient.

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

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
penconazole	100 g/l ³⁾	101,8±0,2%	90-110 g/l ⁴⁾	7-0901

- 1) Mean (6-double 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.

2.3 Slimicides

2.3.1 Introduction

In 1997 only 3 different slimicide active ingredients used in paper manufacture were approved to the market in Denmark (*Miljøstyrelsen, 1997*). Products containing 2,2-dibrom-2-cyanoacetamide were selected for control in 1997.

2,2-dibrom-2-cyanoacetamide (Figure 3, VII) is an acetamide type of slimicide, which was introduced on the Danish market in 1993. Formulations containing 2,2-dibrom-2-cyanoacetamide have not been selected for authority control earlier.

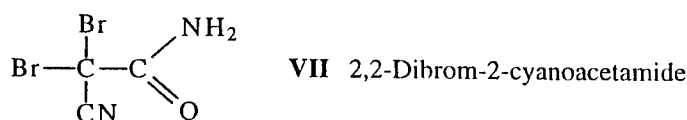


Figure 3. Chemical structure of the slimicide active ingredient 2,2-dibrom-2-cyanoacetamide (VII)

2.3.2 Samples

At the time of sample collection for the control campaign one single product containing 2,2-dibrom-2-cyanoacetamide as active ingredient was approved for use in Denmark (*Miljøstyrelsen, 1997*). Samples from two different batches of the slimicide product was collected. The samples were collected at the user (a paper mill) from sealed containers (1000L) in September 1996 and January 1997. The former was collected by the user and the latter was collected by NERI. Information about the samples is given in Appendix I.

The samples were analysed at NERI in the period January-March 1997.

2.3.3 Results and Discussion

The content of 2,2-dibrom-2-cyanoacetamide was determined using reversed phase high performance liquid chromatography (RP-HPLC) (*Køppen, 1996*). Results from the analyses are shown in Table 2.4. The results were further confirmed using iodometric titration with a method from the manufacturer company (*GRACE Deaborn, 1995*).

As apparent from Table 2.4 the analysed samples did not comply with the tolerated limits for content of active ingredient. The sample collected by the user was too low in content and the sample collected by NERI was too high in content. The result of a subsequent analysis of the two batches performed by the manufacturer company showed contents inside the accepted tolerance. These samples were stored at the manufacturer company. A possible explanation of the discrepancy may be that the product at the paper mill was inhomogeneous at the time of the sample collection. The manufacturer company had informed that the active ingredient can precipitate if it is stored below 0°C. At the paper mill the slimicide was stored in an unheated storehouse. As a consequence the manufacturer company of the product will take precautions against repetitions.

Table 2.4. Content of active ingredient in samples of slimicides containing 2,2-dibrom-2-cyanoacetamide.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
2,2-dibrom-2-cyanoacetamide (Batchno. 623014)	12 %	10,3 ± 0,1 % ^{*)}	11,2 - 12,8 %	6-0895
2,2-dibrom-2-cyanoacetamide (Batchno. 97/01762)	12 %	13,1 ± 0,1 % ^{*)}	11,2 - 12,8 %	7-0038

1) Mean (4-double determination) " 95% confidence limits.

2) Tolerated limits for content of active ingredients according to Danish regulations (*Miljø- og Energiministeriet, 1998*).

*) Found content is outside the accepted tolerance.

3 Conclusions

Three different groups of products covered by the pesticide regulations have been included in the 1997 analytical chemical authority control: 1) herbicides containing phenmedipham, desmedipham, ethofumesate, triflurosulfuron-methyl or propaquizafop as active ingredients, 2) fungicides containing penconazole as active ingredient and 3) slimicides containing 2,2-dibrom-2-cyanoacetamide as active ingredient.

Satisfactory results were found among products containing the active ingredients phenmedipham, desmedipham, triflurosulfuron-methyl and propaquizafop. Thus all 17 examined samples of these pesticides complied with the accepted tolerances with respect to content of active ingredients set by the Danish regulation of pesticides.

One of the 11 examined samples of products containing ethofumesate as active ingredient did not comply with the accepted tolerance with respect to content of active ingredient, the content of ethofumesate being too low. Subsequent contact with the manufacturer of the product of the latter sample revealed an incorrect label. The label has subsequently been corrected by the company.

Only one product containing 2,2-dibrom-2-cyanoacetamide was on the Danish market. The analysed sample did not comply with the accepted tolerance. The active ingredient can precipitate if the product is stored below 0°C. At the paper mill the slimicide was stored in an unheated storehouse, which may account for the apparent discrepancy. As a consequence the manufacturer company of the product will take precautions against repetitions.

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Appendix I

Samples of pesticides collected from the Danish market in 1997 for authority control

1 Herbicides

Product	Active ingredient(s)	Formulation type ¹⁾	Company	NERI sample no.
Nortron SC	ethofumesate	SC	Hoechst Schering AgrEvo A/S	7-0124
Ethosan	ethofumesate	SC	KVK Agro A/S	7-0125
DLG Ethuron 500 Flow	ethofumesate	SC	Esbjerg Kemi A/S	7-0126
JBC Ethofumesat	ethofumesate	EC	JBC Agro Aps	7-0127
Betaron	ethofumesate phenmedipham	EC	Hoechst Schering AgrEvo A/S	7-0128
Kemifam Duo	ethofumesate phenmedipham	EC	KVK Agro A/S	7-0129
Beta Super	ethofumesate phenmedipham	EC	Esbjerg Kemi A/S	7-0130
Spar 2	ethofumesate phenmedipham	SC	KVK Agro A/S	7-0131
Kemifam pro FL	ethofumesate phenmedipham desmedipham	SC	Kemira Agro oy Finland	7-0133
Bentanal Progress	ethofumesate phenmedipham desmedipham	EC	Hoechst Schering AgrEvo A/S	7-0134
Bentanal Optima	ethofumesate phenmedipham desmedipham	SC	Hoechst Schering AgrEvo A/S	7-0135
Inter Phenmedipham	phenmedipham	EC	Inter-trade Aalborg A/S	7-0136
Betasana Flow	phenmedipham	SC	Esbjerg Kemi A/S	7-0137
JBC Betafam E	phenmedipham	EC	JBC Agro Aps	7-0138
Herbasan	phenmedipham	SC	KVK Agro A/S	7-0139
Agil	propaquizafop	EC	Ciba-Geigy A/S	7-0140

1) SC: suspension concentrate; EC: emulsifiable concentrate; WG: water dispersible granule.

1 Herbicides(continued)

Product	Active ingredient(s)	Formulation type ¹⁾	Company	NERI sample no.
Herbaphen 86	phenmedipham	EC	Kemisk Værk Køge A/S	7-0184
Bentanal SC	phenmedipham	SC	Hoechst Schering AgrEvo A/S	7-0185
Beetup	phenmedipham	EC	DK Petrokemi A/S	7-0186
IT-Ethofumesat 50	ethofumesate	SC	Inter-trade Aalborg A/S	7-0520
LFS Propaquizafop	propaquizafop	EC	LFS Kemi	7-0521
Safari	triflurosulfuron-methyl	WG	Dupont	7-0900

1) SC: suspension concentrate; EC: emulsifiable concentrate; WG: water dispersible granule.

2 Fungicides

Product	Active ingredient	Formulation type ¹⁾	Company	NERI sample no.
Topas 100 EC	penconazole	EC	Grøn Plantebeskyttelse Aps	7-0901

1) EC emulsifiable concentrate

3 Slimicides

Product	Active ingredient	Formulation type	Company	NERI sample no.
Daracid 7819 Batchno.623014	2,2-dibrom-2-cyanoacetamide	liquid	Dearborn	6-0895
Daracid 7819 Batchno.97/01762	2,2-dibrom-2-cyanoacetamide	liquid	Dearborn	7-0038

National Environmental Research Institute

The National Environmental Research Institute, NERI, is a research institute of the Ministry of Environment and Energy. 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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