

Chemical Substances and
Chemical Preparations

Control of Pesticides 1995

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Summary

The analytical chemical authority control performed in 1995 on pesticide products on the Danish market is reported. Samples of selected groups of pesticides have been collected from the market and analysed to check whether the actual content of active ingredient agreed with the label claimed content. The tolerated limits for deviation of active ingredient content from label claim content are set by Danish pesticide regulations.

Two different groups of products covered by the pesticide regulations have been included in the authority control for 1995: 1) herbicides containing either prosulfocarb or triasulfuron as active ingredients and 2) insecticides containing either pyriproxifen or pyrethrin I and II as active ingredients.

Satisfactory results were found for products containing either prosulfocarb, triasulfuron or pyriproxifen as active ingredients. All samples examined complied with the tolerated limits for active ingredient content.

Results from the analysis of products containing pyrethrin I and II were less satisfactory. Thus, 22 of 30 examined samples had contents of either pyrethrin I and II or the synergist piperonyl butoxide that did not comply with the tolerated limits for content of active ingredients.

1 Introduction

In Denmark the Danish Environmental Protection Agency (DEPA) is responsible for evaluation and approval 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 (*Miljøministeriet, 1994*), 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 1995.

2 Control Campaigns in 1995

Control campaigns conducted in 1995 have covered pesticides belonging to two different groups of pesticides: herbicides and insecticides. All analytical chemical control has aimed at examining the content of active ingredient compared to the stated content on the label. Regulation in Denmark (*Miljøministeriet, 1994*) specifies generally tolerated limits of deviation from declared content. These are given in Table 1.

Samples of the various pesticides covered in the 1995 control campaigns have been collected by the Chemical Inspection Service at DEPA during the months March - May 1995 either at wholesale dealers/importers or at retailers. Usually only one sample of each product has been collected, but in a few cases, depending on availability on the market, more samples representing different production batches of the product have 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 1. Tolerated limits of deviations from declared content of active ingredients (a.i.) in pesticides.

Content of a.i., %, w/w	Tolerated limits
> 50	± 2.5% (abs.)
50 - 25	± 5% (rel.)
25 - 10	± 6% (rel.)
10 - 2.5	± 10% (rel.)
< 2.5	± 15% (rel.)

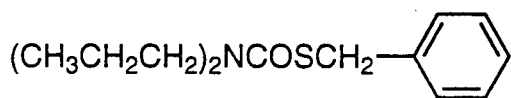
2.1 Herbicides

2.1.1 Introduction

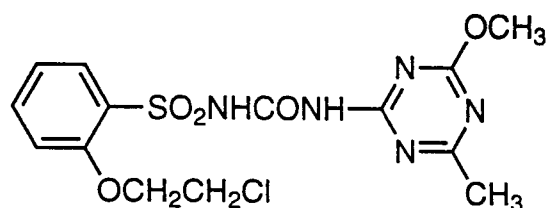
Among the many different herbicides available on the Danish market herbicides containing either prosulfocarb or triasulfuron as active ingredients were selected for control in 1995.

Prosulfocarb (Figure 1, I) is a thiocarbamate herbicide, which is used for control of a wide range of grass and broad-leaved weeds. Prosulfocarb is a rather new herbicide (introduced on the Danish market in 1994), and formulations containing prosulfocarb have not been selected for authority control earlier.

Triasulfuron (Figure 1, II) is an active ingredient, which belongs to the sulfonylurea group of herbicides. Triasulfuron is the newest sulfonylurea compound on the Danish market (introduced in 1995), and is used for control of broad-leaved weeds in both winter and spring cereals. Neither formulations containing triasulfuron have been selected for authority control earlier.



I



II

Figure 1. Chemical structure of the herbicide active ingredients prosulfocarb (I) and triasulfuron (II).

2.1.2 Samples

At the time of sample collection for the control campaign (March - May 1995) only one product containing each of the two active ingredients were approved for use in Denmark (*Miljøstyrelsen, 1995*). One sample of each pesticide product was collected. A list of the samples is given Appendix I.

Both samples were analysed at NERI in the period November - December 1995.

2.1.3 Results and Discussion

The content of prosulfocarb was determined using capillary column gas chromatography with a method (*Køppen, 1995a*) based on information from the manufacturer company on method of analysis.

Similarly, content of triasulfuron was analysed using reversed phase high pressure liquid chromatography (RP-HPLC) with a method (*Køppen, 1994*) also based on information from the manufacturer company on method of analysis. Results from the analyses are shown in Table 2.

As apparent from the table there were good agreement between declared and found contents. Hence, both samples complied with the tolerated limits for content of active ingredient.

Table 2. Content of active ingredient in samples of herbicides.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
prosulfocarb	800 g/l	818 ± 10 g/l, (80.5%)	780 - 820 g/l	5-0345
triasulfuron	20%	19.9 ± 0.2%	18.8 - 21.2%	5-1273

1) Mean (minimum triplicate determinations) ± 95% confidence limits.

2) Tolerated limits for content of active ingredients according to Danish regulations (*Miljøministeriet, 1994*).

2.2 Insecticides

2.2.1 Introduction

In 1995 52 different insecticide active ingredients were on the market in Denmark (*Miljøstyrelsen, 1995*). Products containing two of these active ingredients, pyrethrin I and II and pyriproxifen were selected for control in 1995.

Pyrethrin I and pyrethrin II constitutes the major part of the insecticidal compounds known as pyrethrins, which in fact is a mixture of six closely related compounds (Figure 2, III). The four other compounds are known as cinerin I and II and jasmolin I and II. The ratio pyrethrin:cinerin:jasmolin can vary, but is generally 71:21:7 (*Tomlin, 1994*).

Pyrethrins are naturally occurring insecticides in the flower *Pyrethrum cinerariaefolium*. A concentrated extract from the flowers containing the pyrethrins is used in the production of the commercial insecticide formulations. The insecticidal effect of pyrethrins have been known for more than hundred years (*Tomlin, 1994*), and pyrethrin containing pesticides have been on the market even before modern procedures concerning approval and regulation by the authorities were introduced. For many applications, especially in the agriculture, the use of pyrethrins has been replaced by other newer developed synthetic insecticides, but for some limited applications pyrethrins are still being used.

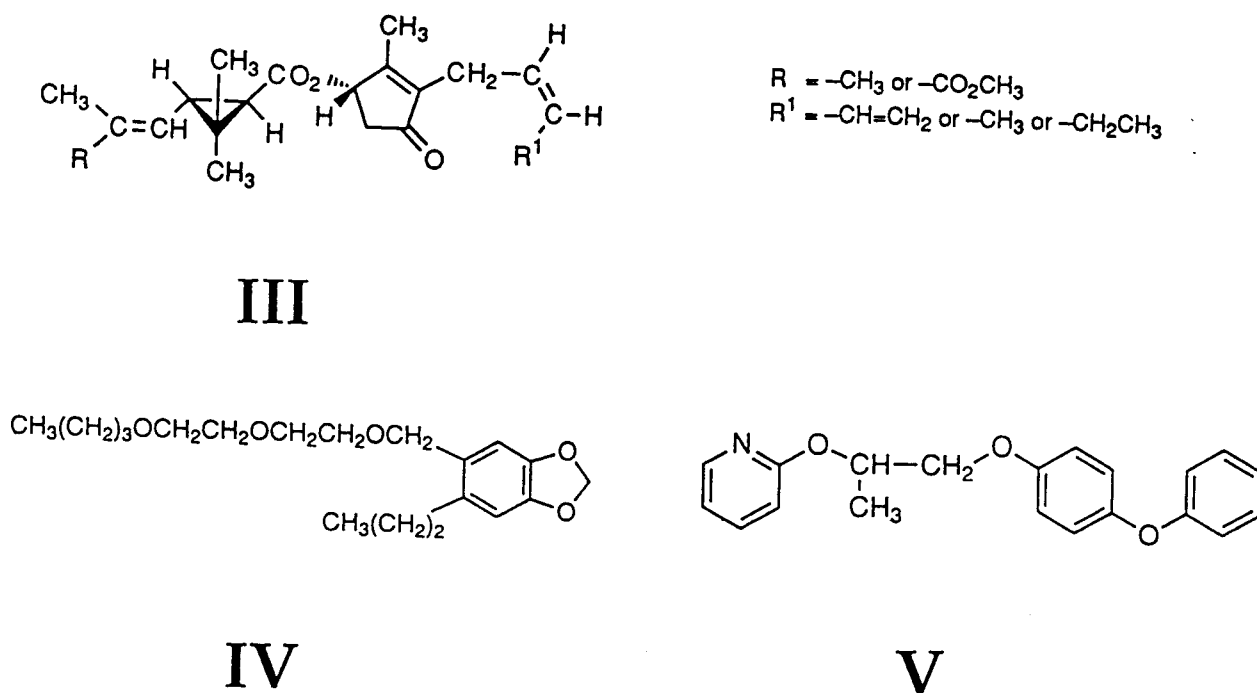


Figure 2. Chemical structures of the insecticide active ingredients pyrethrins (III), piperonyl butoxide (IV) and pyriproxifen (V). (pyrethrin I: R = -CH₃, R¹ = -CH=CH₂; pyrethrin II: R = -CO₂CH₃, R¹ = -CH=CH₂).

Pyrethrin insecticides are used against a range of insects on vegetable crops, plants in horticulture and in animal houses, but are also widely used as household pesticide against insects on house plants and pets (e.g. fleas on cats and dogs).

Nearly all pyrethrin insecticide formulations also contain the compound piperonyl butoxide (Figure 2, IV). Piperonyl butoxide has no insecticidal effect itself, but increases the effect of pyrethrins by inhibiting detoxification (Tomlin, 1994).

Before 1995 insecticide formulations containing pyrethrins (and piperonyl butoxide) had not been selected for authority control for the last more than ten years.

Pyriproxifen (Figure 2, V) is an insecticidal compound, which is used only commercially against a limited number of insects (certain aphids) on plants grown in greenhouses. Pyriproxifen is a rather new insecticide (introduced on the Danish market in 1995), and formulations containing pyriproxifen have not been selected for authority control earlier.

2.2.2 Samples

At the time of sample collection for the control campaign (March - May 1995) 50 different insecticide products containing pyrethrin I and II as active ingredient and one single product containing pyriproxifen were approved for use in Denmark (Miljøstyrelsen, 1995). 30 of these 51 products were available on the market during the period of sample collection. In total 31 samples were collected representing these 30 products. A list of the samples is given in Appendix I.

All samples were analysed at NERI in the period June - October 1995.

2.2.3 Results and Discussion

The contents of pyrethrin I and II and piperonyl butoxide were determined using normal phase HPLC with a method (Køppen, 1995b), which is a slight modification of the method described by Bushway (Bushway, 1985). The method allow simultaneous determination of the pyrethrins and piperonyl butoxide and is capable of separating all six pyrethrin compounds.

Table 3. Content of active ingredient(s) in samples of insecticides containing either pyrethrin I and II or pyriproxifen.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
pyrethrin I and II piperonyl butoxide	0.3%	0.22 ± 0.01% *)	0.25 - 0.35%	5-0233
	1.5%	1.72 ± 0.04%	1.27 - 1.73%	
pyrethrin I and II piperonyl butoxide	0.4%	0.25 ± 0.02% *)	0.34 - 0.46%	5-0234
	2.4%	2.56 ± 0.06%	2.04 - 2.76%	
pyrethrin I and II piperonyl butoxide	0.4%	0.35 ± 0.02%	0.34 - 0.46%	5-0235
	2.4%	2.10 ± 0.07%	2.04 - 2.76%	
pyrethrin I and II piperonyl butoxide	0.55%	0.48 ± 0.02%	0.47 - 0.63%	5-0236
	2.24%	2.15 ± 0.07%	1.90 - 2.58%	
pyrethrin I and II piperonyl butoxide	0.36%	0.32 ± 0.02%	0.31 - 0.41%	5-0238
	2.16%	2.41 ± 0.08%	1.83 - 2.49%	
pyrethrin I and II piperonyl butoxide	0.55%	0.43 ± 0.03% *)	0.47 - 0.63%	5-0239
	2.24%	2.05 ± 0.07%	1.90 - 2.58%	
pyrethrin I and II piperonyl butoxide	0.52%	0.42 ± 0.03%	0.44 - 0.60%	5-0240
	3.3%	4.2 ± 0.1% *)	2.97 - 3.63%	
pyrethrin I and II piperonyl butoxide	1.51%	1.23 ± 0.08%	1.28 - 1.74%	5-0241
	15.1%	17.1 ± 0.5% *)	14.2 - 16.0%	
pyrethrin I and II	1.25%	1.03 ± 0.10%	1.06 - 1.44%	5-0242
pyrethrin I and II piperonyl butoxide	0.12%	0.073 ± 0.005% *)	0.10 - 0.14%	5-0243
	0.4%	0.45 ± 0.02%	0.34 - 0.46%	
pyrethrin I and II piperonyl butoxide	0.2%	0.13 ± 0.01% *)	0.17 - 0.23%	5-0244
	1.3%	0.15 ± 0.01% *)	1.10 - 1.50%	
pyrethrin I and II piperonyl butoxide	0.1%	0.074 ± 0.005% *)	0.085 - 0.115%	5-0245
	0.4%	0.48 ± 0.02%	0.34 - 0.46%	
pyrethrin I and II piperonyl butoxide	0.9%	0.68 ± 0.04% *)	0.77 - 1.03%	5-0337
	8.87%	7.8 ± 0.3%	7.98 - 9.76%	
pyrethrin I and II piperonyl butoxide	0.3%	0.23 ± 0.01% *)	0.25 - 0.35%	5-0338
	1.8%	1.96 ± 0.06%	1.53 - 2.07%	
pyrethrin I and II piperonyl butoxide	0.1%	0.078 ± 0.005% *)	0.085 - 0.115%	5-0340
	0.4%	0.48 ± 0.02%	0.34 - 0.46%	
pyrethrin I and II piperonyl butoxide	0.4%	0.32 ± 0.02%	0.34 - 0.46%	5-0342
	2.4%	2.55 ± 0.07%	2.04 - 2.76%	

1) Mean (minimum duplicate determinations) ± 95% confidence limits.

2) Tolerated limits for content of active ingredients according to Danish regulations (*Miljøministeriet, 1994*).

*) Found content is outside the accepted tolerance.

Table 3. (continued). Content of active ingredient(s) in samples of insecticides containing either pyrethrin I and II or pyriproxifen.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
pyrethrin I and II piperonyl butoxide	0.25% 1.0%	0.22 ± 0.01% 1.14 ± 0.04%	0.21 - 0.29% 0.85 - 1.15%	5-0634
pyrethrin I and II piperonyl butoxide	0.52% 3.3%	0.41 ± 0.02% *) 3.8 ± 0.1% *)	0.44 - 0.60% 2.97 - 3.63%	5-0635
pyrethrin I and II piperonyl butoxide	0.2% 1.3%	0.14 ± 0.01% *) 0.43 ± 0.02% *)	0.17 - 0.23% 1.10 - 1.50%	5-0637
pyrethrin I and II piperonyl butoxide	0.4% 2.4%	0.30 ± 0.01% *) 3.41 ± 0.08% *)	0.34 - 0.46% 2.04 - 2.76%	5-0638
pyrethrin I and II piperonyl butoxide	0.2% 0.5%	0.17 ± 0.01% 0.56 ± 0.02%	0.17 - 0.23% 0.42 - 0.58%	5-0959
pyrethrin I and II piperonyl butoxide	0.2% 1.2%	0.18 ± 0.01% 1.38 ± 0.04%	0.17 - 0.23% 1.02 - 1.38%	5-0960
pyrethrin I and II piperonyl butoxide	0.3% 1.25%	0.22 ± 0.02% *) 1.20 ± 0.04%	0.25 - 0.35% 1.06 - 1.44%	5-0961
pyrethrin I and II piperonyl butoxide	0.47% 2.99%	0.35 ± 0.02% *) 2.78 ± 0.09%	0.40 - 0.54% 2.69 - 3.29%	5-0963
pyrethrin I and II piperonyl butoxide	0.075% 0.15%	0.058 ± 0.003% *) 0.167 ± 0.04%	0.063 - 0.087% 0.127 - 0.173%	5-1042
pyrethrin I and II piperonyl butoxide	0.1% 0.4%	0.076 ± 0.005% *) 0.50 ± 0.02% *)	0.085 - 0.115% 0.34 - 0.46%	5-1043
pyrethrin I and II piperonyl butoxide	0.1% 0.4%	0.075 ± 0.005% *) 0.50 ± 0.02% *)	0.085 - 0.115% 0.34 - 0.46%	5-1044
pyrethrin I and II piperonyl butoxide	3.3% 14.7%	2.58 ± 0.10% *) 15.5 ± 0.4%	2.97 - 3.63% 13.8 - 15.6%	5-1275
pyrethrin I and II piperonyl butoxide	1.0% 4.0%	0.64 ± 0.03% *) 3.8 ± 0.1%	0.85 - 1.15% 3.6 - 4.4%	5-1276
pyrethrin I and II piperonyl butoxide	0.4% 2.4%	0.33 ± 0.02% 3.6 ± 0.1% *)	0.34 - 0.46% 2.04 - 2.76%	5-1277
pyriproxifen	100 g/l	102 ± 2 g/l (11.2%)	94 - 106 g/l	5-1274

1) Mean (minimum duplicate determinations) ± 95% confidence limits.

2) Tolerated limits for content of active ingredients according to Danish regulations (*Miljøministeriet, 1994*).

*) Found content is outside the accepted tolerance.

The content of pyriproxifen was analysed using reversed phase HPLC with a method (Køppen, 1995c) based on information from the manufacturer company on method of analysis. Results from the analyses are shown in Table 3.

The results demonstrate that only nine of the 31 analysed samples complied with the tolerated limits for content of active ingredient. This includes the single analysed sample containing pyriproxifen and eight of the analysed samples containing pyrethrin I and II as active ingredient. 19 samples had a content of pyrethrin I and II deviating more from the declared content than what is acceptable. Further, nine samples had a content of piperonyl butoxide deviating unacceptably much from the declared content.

The fact that more than two thirds of the samples containing pyrethrins did not comply with the tolerances, is an unusually high frequency compared to what is normally found in pesticide control. The 19 samples deviating with respect to content of pyrethrin I and II had all too low contents, whereas among the nine samples deviating with respect to content of piperonyl butoxide both samples with too low and too high contents were found.

With respect to the samples of formulations exhibiting too low content of pyrethrin I and II the most likely explanation is an unawareness by several companies of the possible discrepancy between the terms "total pyrethrins content" and "pyrethrin I and II content". The total pyrethrins content is typically stated on the pyrethrum extract used by the companies in the manufacture of the formulations. This content usually corresponds to the total content of all six pyrethrins, whereas the content stated on the label is only the sum of pyrethrin I and pyrethrin II contents. According to subsequent information from several of the manufacturing companies producing these pyrethrin containing formulations, the companies have not been aware of this discrepancy, and no correction has therefore been made when producing the formulations. This had led to formulations containing typically 20 - 30% less pyrethrin I and II than the what was intended.

Another possible explanation could be a limited stability of the pyrethrins. It is well known (Tomlin, 1994) that pyrethrins are susceptible to degradation when exposed to air and light. Thus, precautions must be taken by the manufacturers to avoid such exposure both in case of the formulated products and of the pyrethrum extract used in the manufacture of the formulated products.

Such action has been taken by one of the manufacturing companies producing formulations that were found to have too low content of pyrethrin I and II. According to subsequent information from the company (Kristensen, 1996) a different type of container, which give the formulation a better protection against light, will be used in the future.

Concerning the deviations of piperonyl butoxide from the declared content, no single explanation could be deduced from manufacturer company informations regarding the history of the actual samples. The most likely explanations are technical matters with relation to the manufacturing process of the formulations. Thus, possible explanations were given covering accidental faults during weighings, mixing etc. or deviation from the declared content of the technical material used in the manufacture of the formulations. (Several companies explained, that they did not control the actual content of technical material piperonyl butoxide used in the manufacturing process).

3 Conclusions

Two different groups of products covered by the pesticide regulations have been included in the 1995 analytical chemical authority control: 1) herbicides containing triasulfuron or prosulfocarb as active ingredients and 2) insecticides containing pyrethrin I and II or pyriproxifen as active ingredients.

Satisfactory results were found among products containing the active ingredients triasulfuron, prosulfocarb and pyroproxifen. Thus all three examined samples of these pesticides complied with the accepted tolerances with respect to content of active ingredient set by the Danish regulation of pesticides.

Unsatisfactory results were found among products containing pyrethrin I and II as active ingredient. 22 of the 30 examined samples were found to have a content of either pyrethrin I and II or of the synergist piperonyl butoxide deviating more from the declared content than accepted by the regulations. All deviations of pyrethrin I and II from the declared content were samples with too low contents. The reason for this could in most cases be explained by a systematic too low addition of pyrethrin extract during manufacture of the formulations, because of a discrepancy between the meaning of the stated content on the extract and on the labelled formulation.

Another possible explanation of the low content of pyrethrin I and II in the samples could be a limited stability of the pyrethrins in some formulations.

The deviations of piperonyl butoxide from the declared content covered both positive and negative deviations, which could indicate that they are most likely caused by various technical matters related to the manufacture of the formulations. One general explanation could not be deduced from manufacturer companies information concerning the analysed samples.

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Appendix I

Samples of pesticides collected from the Danish market in 1995 for authority control

1 Herbicides

Product	Active ingredient	Formulation type ¹⁾	Company	NERI sample no.
Boxer	prosulfocarb	EC	Zeneca Agro	5-0345
Logran 20 WG	triasulfuron	WG	Ciba-Geigy	5-1273

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

2 Insecticides

Product	Active ingredient(s)	Formulation type ¹⁾	Company	NERI sample no.
Trinol Bio	pyrethrin I and II piperonyl butoxide	SL	Trinol a/s	5-0233
Special 86	pyrethrin I and II piperonyl butoxide	SL	Mortalin Prod. Aps.	5-0234
Special Flueaerosol	pyrethrin I and II piperonyl butoxide	AE	Mortalin Prod. Aps.	5-0235
Kill-it Fluespray S	pyrethrin I and II piperonyl butoxide	AE	Bayer Danmark a/s	5-0236
Kvit Fluespray	pyrethrin I and II piperonyl butoxide	AE	Bayer Danmark a/s	5-0238
Kill-it Fluespray S	pyrethrin I and II piperonyl butoxide	AE	Bayer Danmark a/s	5-0239
Absolut spray	pyrethrin I and II piperonyl butoxide	AE	Bayer Danmark a/s	5-0240
Coopermatic insektdræber aerosol 88	pyrethrin I and II piperonyl butoxide	AE	Mortalin Prod. Aps.	5-0241
Gesal natur sprøjtemiddel	pyrethrin I and II	SL	Bayer Danmark a/s	5-0242
Hundeshampoo mod utøj	pyrethrin I and II piperonyl butoxide	SH	Kvik Dyreartikler a/s	5-0243
Absolut loppepulver	pyrethrin I and II piperonyl butoxide	PW	Bayer Danmark a/s	5-0244
Shampoo til hunde	pyrethrin I and II piperonyl butoxide	SH	Bayer Danmark a/s	5-0245
Swak Natural	pyrethrin I and II piperonyl butoxide	AE	Scan Fresh Airsystem	5-0337
EK insektspray	pyrethrin I and II piperonyl butoxide	AE	Esbjerg Kemi	5-0338
KW Utøjsshampoo	pyrethrin I and II piperonyl butoxide	SH	KW Hunde- og katteartikler	5-0340
Pytoxan fluemiddel	pyrethrin I and II piperonyl butoxide	SL	Bayer Danmark a/s	5-0342

1) SL = solution concentrate; AE = aerosol dispenser; SH = shampoo; PW = powder.

2 Insecticides (continued)

Product	Active ingredient(s)	Formulation type ¹⁾	Company	NERI sample no.
Myre spray	pyrethrin I and II piperonyl butoxide	AE	Bayer Danmark a/s	5-0634
Utøjsspray til hunde	pyrethrin I and II piperonyl butoxide	AE	Bayer Danmark a/s	5-0635
Loppepulver til hunde	pyrethrin I and II piperonyl butoxide	PW	Bayer Danmark a/s	5-0637
PLK Pyrethrum mod fluer	pyrethrin I and II piperonyl butoxide	SL	G.F. Agro a/s	5-0638
Kibrill spray	pyrethrin I and II piperonyl butoxide	AE	Præstrud & Kjeldsmark Aps	5-0959
Becovett utøjsspray	pyrethrin I and II piperonyl butoxide	AE	Cederroth a/s	5-0960
Substral bladluspray	pyrethrin I and II piperonyl butoxide	AE	Henkel Barnängen a/s	5-0961
Radar Universal	pyrethrin I and II piperonyl butoxide	AE	Henkel Barnängen a/s	5-0963
Meku Insektskræk	pyrethrin I and II piperonyl butoxide	SL	Medicinkemi Uldum a/s	5-1042
Utøjsshampoo	pyrethrin I and II piperonyl butoxide	SH	Medicinkemi Uldum a/s	5-1043
Dyrlægens sortiment utøjs- shampoo	pyrethrin I and II piperonyl butoxide	SH	Medicinkemi Uldum a/s	5-1044
Admiral 10 EC	pyriproxifen	EC	Du Pont de Nemeours a/s	5-1274
Pyrsol	pyrethrin I and II piperonyl butoxide	SL	Du Pont de Nemeours a/s	5-1275
EK Bladlusmiddel	pyrethrin I and II piperonyl butoxide	SL	Esbjerg Kemi a/s	5-1276
Trinol Fluemiddel	pyrethrin I and II piperonyl butoxide	SL	Trinol a/s	5-1277

- 1) SL = solution concentrate; AE = aerosol dispenser; SH = shampoo; PW = powder; EC = emulsifiable concentrate.

National Environmental Research Institute

The National Environmental Research Institute - NERI - is a research institute of the Ministry of the Environment and Energy. NERI's tasks are primarily to do research, collect data and give advice on problems related to the environment and Nature.

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NERI publishes professional reports, technical instructions, reprints of scientific and professional articles and an annual report.

Included in the annual report is a review of the publications from the year in question. The annual report and an up-to-date review of the year's publications are available upon request.