

Chemical Substances and
Chemical Preparations

Control of Pesticides 1996

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Summary

The analytical chemical authority control performed in 1996 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 composition of the products are in accordance with the specification of product composition that has been given to the Danish authorities in connection with application for approval of the single products. The control has primarily covered comparing actual contents of active ingredients with label claimed content, but for one type of product an additional control of levels of a selected possible impurity was included.

Four different groups of products covered by the pesticide regulations have been included in the authority control for 1996: 1) herbicides containing haloxyfop-ethoxyethyl as active ingredient, 2) insecticides containing chlorpyrifos as active ingredient, 3) fungicides containing prochloraz as active ingredient and 4) plant growth regulators containing ethephon as active ingredient.

Satisfactory results were found for the herbicide and fungicide products, where all samples examined complied with the tolerated limits for active ingredient content. Furthermore, examination of content of an unwanted impurity 2,4,6-trichlorophenol, potentially present in the fungicide active ingredient prochloraz, showed residues at levels below a specified maximum limit in all samples.

The control of insecticides and plant growth regulators displayed some samples, where the found content of active ingredient did not agree with the label claimed content. Three out of five analysed samples of insecticides containing chlorpyrifos as active ingredient deviated unacceptably from label claimed content. Similarly, one out of ten analysed samples of plant growth regulator products containing ethephon as active ingredient was found to deviate unacceptably from the label claimed content.

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 and Energy (*Miljø- og Energiministeriet, 1996a*), 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 Chemical 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 1996.

2 Control Campaigns in 1996

Control campaigns conducted in 1996 have covered pesticides belonging to four different groups of pesticides: herbicides, insecticides, fungicides and plant growth regulators. The analytical chemical control has aimed at examining the content of active ingredient compared to the stated content on the label. In one of the campaigns a control of the level of a certain impurity in the pesticide active ingredient has been included too.

Regulation in Denmark (*Miljø- og Energiministeriet, 1996a*) specifies generally tolerated limits of deviation from declared content. These are given in Table 1.

Samples of the various pesticides covered in the 1996 control campaigns have been collected by the Chemical Inspection Service at DEPA during the months March - May 1996 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 only one type of product was selected for control in 1996. It concerns herbicides containing haloxyfop-ethoxyethyl as active ingredient.

Haloxyfop-ethoxyethyl (Figure 1, I) is a phenoxypropionic acid ester type of herbicide, which is used for control of grasses in a variety of different crops. Haloxyfop-ethoxyethyl is a rather new herbicide (introduced on the Danish market in 1994), and formulations containing haloxyfop-ethoxyethyl have not been selected for authority control earlier.

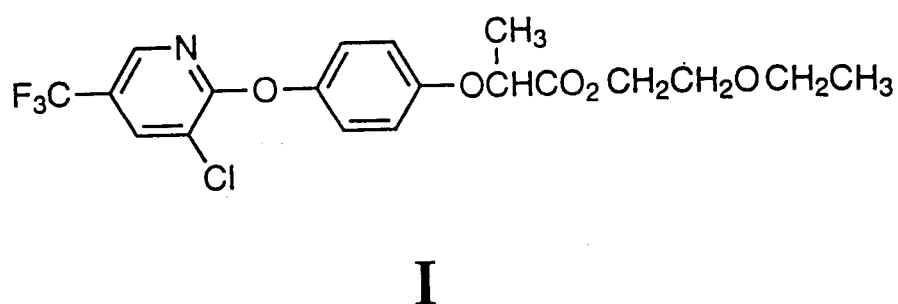


Figure 1. Chemical structure of the herbicide active ingredient haloxyfop-ethoxyethyl (I).

2.1.2 Samples

At the time of sample collection for the control campaign (March - May 1996) only one product containing haloxyfop-ethoxyethyl as active ingredient was approved for use in Denmark (*Miljøstyrelsen, 1996a*). Only one sample of the pesticide product was collected. Information about the sample is given in the list of the collected samples in Appendix I.

The sample was analysed at NERI in July 1996.

2.1.3 Results and Discussion

The content of haloxyfop-ethoxyethyl was determined using reversed phase high performance liquid chromatography (RP-HPLC) with a method (*Køppen, 1996a*) based on information from the manufacturer company on method of analysis. The result from the analysis is shown in Table 2.

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

Table 2. Content of active ingredient in one sample of herbicide containing haloxyfop-ethoxyethyl.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
haloxyfop-ethoxyethyl	12,93 %, (125 g/l)	12,4 ± 0,1 %, (120 g/l)	12,1 - 13,7 %	6-0191

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

2) Tolerated limits for content of active ingredients according to Danish regulations (*Miljø- og Energi-ministeriet, 1996a*).

2.2 Insecticides

2.2.1 Introduction

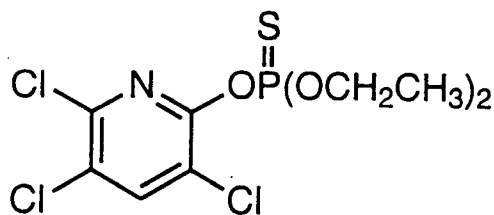
In 1996 48 different insecticide active ingredients were on the market in Denmark (*Miljøstyrelsen, 1996a*). Only products containing one of these active ingredients, chlorpyrifos, were selected for control in 1996.

Chlorpyrifos (Figure 2, II) is an organophosphorous type of insecticide, which is used against certain insects on plants grown in greenhouses. Products containing chlorpyrifos as active ingredient are also widely used as household pesticides.

2.2.2 Samples

At the time of sample collection for the control campaign (March - May 1996) seven different insecticide products containing chlorpyrifos were approved for use in Denmark (*Miljøstyrelsen, 1996a*). Five of these seven products were available on the market during the period of sample collection. One sample of each of the five products was collected for control. A list of the samples is given in Appendix I.

All samples were analysed at NERI in the period September - October 1996.



II

Figure 2. Chemical structure of the insecticide active ingredient chlorpyrifos (II).

2.2.3 Results and Discussion

The contents of chlorpyrifos were determined using RP-HPLC with a method (Køppen, 1996b), which is a slight modification of the official CIPAC method (CIPAC, 1985).

Results from the analyses are shown in Table 3.

As it is indicated in the table two out of the five samples analysed were found to have too high contents of active ingredient, as well as one sample was found to have too low content of active ingredient. For one of these samples (NERI sample no. 6-0283) a discrepancy between the label claimed contents of chlorpyrifos expressed as either % or g/l was found. This meant that the found content (expressed as %) was too high compared to the label claimed content, whereas the same found content (expressed as g/l) was too low.

A similar discrepancy between label claimed contents, expressed as either % or g/l, was also found for the other sample (NERI sample no. 6-0225) for which the content of chlorpyrifos was too high. In this case, however, the found content (expressed as g/l) was in good agreement with the label claimed content.

Table 3. Content of active ingredient in samples of insecticides containing chlorpyrifos.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
chlorpyrifos	2,5 %, (25 g/l)	2,91 ± 0,02 % ^{*)} , (25,6 g/l)	2,12 - 2,88 %	6-0225
chlorpyrifos	20,0 %, (208 g/l)	20,7 ± 0,1 %, (215 g/l)	18,8 - 21,2 %	6-0226
chlorpyrifos	0.625 g	0.62 ± 0.04 g	0.56 - 0.69 g	6-0282
chlorpyrifos	2,5 %, (36 g/l)	3,05 ± 0.02 % ^{*)} , (24,6 g/l)	2,12 - 2,88 %	6-0283
chlorpyrifos	0,8 %, (8,0 g/l)	0,59 ± 0,05 % ^{*)} , (5,9 g/l)	0,68 - 0,92 %	6-0284

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

2) Tolerated limits for content of active ingredients according to Danish regulations (Miljø- og Energi-ministeriet, 1996a).

*) Found content is outside the accepted tolerance.

The three samples found to have contents of chlorpyrifos deviating unacceptably from the declared content were of two different types of formulations, an organic solvent based solution (lacquer) and microencapsulated aqueous suspension concentrate. Subsequent contact to the manufacturer companies of the products revealed, that in case of one of the lacquer formulations (NERI sample no. 6-0283) the high content in the sample was a result of an incorrect prescription used in the manufacturing process leading systematically to formulations with too high contents. This fault has now been corrected by the company. In case of the other two samples, (NERI sample no. 6-0225 and 6-0284) no specific explanation was found for the deviating contents in the analysed samples.

2.3 Fungicides

2.3.1 Introduction

In 1996 28 different fungicide active ingredients were on the market in Denmark (*Miljøstyrelsen, 1996a*). Among the many formulations containing these active ingredients, only products containing prochloraz were selected for control in 1996.

Prochloraz (Figure 3, III) is an imidazole type of fungicide, which is used for seed treatment as well as for spraying against a wide range of diseases affecting cereals, fruits and other crops.

Prochloraz is an imidazole derivative type of pesticide containing a 2,4,6-trichlorophenoxy moiety in the molecule and the compound 2,4,6-trichlorophenol (Figure 3, IV) is used in the synthesis of prochloraz. Thus, 2,4,6-trichlorophenol is a possible impurity in the prochloraz technical material used in the manufacture of prochloraz formulations. 2,4,6-Trichlorophenol is a suspected carcinogen (*Miljø- og Energiministeriet, 1996b*) and because of this unwanted risk to human health, a maximum allowed limit of this potential impurity has been specified by the manufacturer of the active ingredient prochloraz in relation to application for approval of these formulations in Denmark. In connection with the conducted control of content of active ingredient in prochloraz formulations, an investigation of the level of 2,4,6-trichlorophenol as impurity in formulations as well as in technical grade prochloraz has been done too.

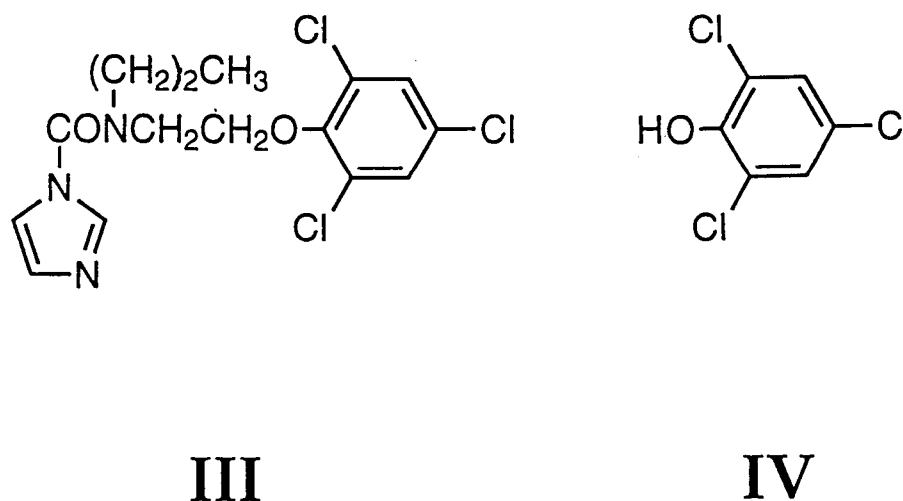


Figure 3. Chemical structures of the fungicide active ingredient prochloraz (III) and a possible impurity in prochloraz, 2,4,6-trichlorophenol (IV).

2.3.2 Samples

At the time of sample collection for the control campaign (March - May 1996) five different fungicide products containing prochloraz were approved for use in Denmark (*Miljøstyrelsen, 1996a*). Three of these five products were available on the market during the period of sample collection. In total four formulated samples were collected from the market and one sample of prochloraz technical material was received for control from the only company having prochloraz containing products approved for sale on the Danish market. A list of the samples is given in Appendix I.

All samples were analysed at NERI in the period May - July 1996.

2.3.3 Results and Discussion

The contents of prochloraz were determined using capillary column gas chromatography with a method (*Køppen, 1996c*) based on information from the manufacturer company on method of analysis.

The contents of the impurity 2,4,6-trichlorophenol were determined using RP-HPLC with a method (*Køppen, 1996d*) developed in our laboratory.

Results from the analyses of contents of active ingredient and the impurity are shown in Tables 4 and 5, respectively.

Table 4. Content of active ingredient in samples of fungicides containing prochloraz.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
prochloraz	39,8 %, (450 g/l)	38,6 ± 0,4 %, (438 g/l)	37,8 - 41,8 %	6-0055
prochloraz	21,6 %, (225 g/l)	20,2 ± 0,3 %, (212 g/l)	20,3 - 22,9 %	6-0056
prochloraz	39,8 %, (450 g/l)	38,6 ± 0,4 %, (437 g/l)	37,8 - 41,8 %	6-0192
prochloraz	40,0 %, (450 g/l)	38,3 ± 0,6 %, (431 g/l)	38,0 - 42,0 %	6-0327

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

2) Tolerated limits for content of active ingredients according to Danish regulations (*Miljø- og Energi-ministeriet, 1996a*).

As apparent from Table 4 all samples were found to have a content slightly lower than the label claimed content, but all found contents complied with the tolerated limits for content of active ingredient.

From the results shown in Table 5 it can be concluded that the found contents of the impurity 2,4,6-trichlorophenol in the samples were in the range 0.06 - 0.28 %.

It has not been possible to include the actual specified maximum limit of 2,4,6-trichlorophenol in technical material in this report, because such information has been given by the manufacturer company to the Danish EPA in confidentiality. However, it can be stated that the content found in the sample of technical material prochloraz was lower than the specified maximum limit. Since the contents of 2,4,6-trichlorophenol relative to the content of prochloraz found in the formulated samples were at about the same level or lower, this was taken as a good indication of compliance with the specified maximum limit for the batches of technical prochloraz used in these formulations too.

Table 5. Content of the impurity 2,4,6-trichlorophenol in various samples containing prochloraz as active ingredient.

Sample type ¹⁾	Content of 2,4,6-trichlorophenol found, %		NERI sample no.
	In sample	Rel. to a.i. ²⁾	
EW	0.070	0.177 ± 0.010	6-0055
EC	0.059	0.273 ± 0.008	6-0056
EW	0.062	0.155 ± 0.003	6-0192
TC	0.269	0.280 ± 0.007	6-0195
EC	0.068	0.170 ± 0.002	6-0327

1) EW = oil in water emulsion; EC = emulsifiable concentrate; TC = technical material.

2) Relative to content of prochloraz active ingredient. Mean (minimum duplicate determinations) ± 95% confidence limits.

2.4 Plant Growth Regulators

2.4.1 Introduction

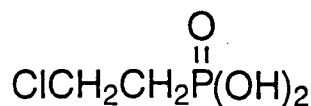
In 1996 10 different active ingredients were approved for use as plant growth regulators in Denmark (*Miljøstyrelsen, 1996a*). Only products containing one of these active ingredients, ethephon, were selected for control in 1996.

Ethephon (Figure 4, V) is an alkylphosphonic acid, which acts by liberation of the active, growth stimulating substance ethylene when taken up by the plants. It is used for several growth regulating purposes within fruit growing and gardening, such as to promote ripening in fruits and to induce flowering.

2.4.2 Samples

At the time of sample collection for the control campaign (August - September 1995 and March - May 1996) six different plant growth regulating products containing ethephon were approved for use in Denmark (*Miljøstyrelsen, 1996a, Miljøstyrelsen 1996b*). All these six products were available on the market during the two periods of sample collection. A total of ten samples representing these six products were collected for control. A list of the samples is given in Appendix I.

All samples were analysed at NERI in the period August - September 1996.



V

Figure 2. Chemical structure of the plant growth regulating active ingredient ethephon (V).

2.4.3 Results and Discussion

The contents of ethephon were determined using the official titrimetric method of analysis (CIPAC, 1996). Results from the analyses are shown in Table 6.

As it appears from the table one of the analysed samples did not comply with the tolerated limits for 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 ethephon, did not lead to any explanation for this discrepancy. Accidental errors during the manufacturing process of the formulation is considered the most likely explanation.

Table 6. Content of active ingredient in samples of plant growth regulating products containing ethephon.

Active ingredient	Content			NERI sample no.
	Label claim	Analysis ¹⁾	Tolerance ²⁾	
ethephon	40 %, (480 g/l)	41,0 ± 0,3 %, (495 g/l)	38,0 - 42,0 %	5-0246
ethephon	14,2 %, (155 g/l)	14,3 ± 0,1 %, (156 g/l)	13,3 - 15,1 %	5-0344
ethephon	39 %, (480 g/l)	40,0 ± 0,2 %, (488 g/l)	37,1 - 40,9 %	5-0639
ethephon	⁻³⁾ , (480 g/l)	40,5 ± 0,3 %, (492 g/l)	⁻ , 456 - 504 g/l ⁴⁾	5-0640
ethephon	39 %, (480 g/l)	38,4 ± 0,2 %, (467 g/l)	37,1 - 40,9 %	6-0051
ethephon	40 %, (480 g/l)	39,2 ± 0,2 %, (475 g/l)	38,0 - 42,0 %	6-0052
ethephon	⁻³⁾ , 480 g/l	39,6 ± 0,2 %, (484 g/l)	⁻ , 456 - 504 g/l ⁴⁾	6-0053
ethephon	14,2 %, (155 g/l)	14,4 ± 0,1 %, (157 g/l)	13,3 - 15,1 %	6-0054
ethephon	⁻³⁾ , 480 g/l	40,7 ± 0,3 %, (494 g/l)	⁻ , 456 - 504 g/l ⁴⁾	6-0224
ethephon	⁻³⁾ , 480 g/l	34,6 ± 0,2 %, (420 g/l) ^{*)}	⁻ , 456 - 504 g/l ⁴⁾	6-0285

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

2) Tolerated limits for content of active ingredients according to Danish regulations (*Miljø- og Energi-ministeriet, 1996a*).

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

Four different groups of products covered by the pesticide regulations have been included in the 1996 analytical chemical authority control: 1) herbicides containing haloxyfop-ethoxyethyl as active ingredient, 2) insecticides containing chlorpyrifos as active ingredient, 3) fungicides containing prochloraz as active ingredient and 4) plant growth regulators containing ethephon as active ingredient.

Satisfactory results were found among products containing the active ingredients haloxyfop-ethoxyethyl and prochloraz. Thus, all five examined samples of these pesticides complied with the accepted tolerances with respect to content of active ingredient set by the Danish regulation of pesticides. Furthermore, examination of content of an unwanted impurity 2,4,6-trichlorophenol, potentially present in the fungicide active ingredient prochloraz, showed residues levels in all samples below a specified maximum limit.

Unsatisfactory results were found among products chlorpyrifos and ethephon as active ingredients. Three of the five analysed samples of products containing chlorpyrifos did not comply with the accepted tolerances with respect to content of active ingredient. In addition, two of the five products were found to have a discrepancy between the declared contents expressed as % w/w and g/l. Subsequent contact with the manufacturer of these products revealed that, in one of the two cases, the found deviation from the declared content was related to unawareness about this discrepancy. In the other cases, no distinct explanation was found for the deviation from the declared contents.

Similarly, one out of ten analysed samples of product containing ethephon did not comply with the accepted tolerances with respect to content of active ingredient. Subsequent contact with the manufacturer of the product of which the analysed sample had too low content of ethephon did not lead to an explanation for this deviation. Most likely, the deviation is caused by an accidental error related to the manufacturing process.

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Appendix I

Samples of pesticides collected from the Danish market in 1996 for authority control

1 Herbicides

Product	Active ingredient	Formulation type ¹⁾	Company	NERI sample no.
Gallant	haloxyfop-ethoxy-ethyl	EC	DowElanco Danmark	6-0191

1) EC: emulsifiable concentrate.

2 Insecticides

Product	Active ingredient(s)	Formulation type ¹⁾	Company	NERI sample no.
Mortalin insektlak	chlorpyrifos	LA	a/s Mortalin	6-0225
Empire 20	chlorpyrifos	EW	DowElanco Danmark	6-0226
Nexa mølhænger	chlorpyrifos	IP	Lopex	6-0282
Tanaco-Partners insektlak	chlorpyrifos	LA	Tanaco Danmark a/s	6-0283
Gett	chlorpyrifos	EW	DowElanco Danmark	6-0284

1) LA = lacquer; EW = microencapsulated oil-in-water emulsion concentrate; IP = impregnated paper.

3 Fungicides

Product	Active ingredient(s)	Formulation type ¹⁾	Company	NERI sample no.
Sportak EW	prochloraz	EW	AgrEvo a/s	6-0055
Rival	prochloraz	EC	AgrEvo a/s	6-0056
Sportak EW	prochloraz	EW	AgrEvo a/s	6-0192
Prochloraz techn.	prochloraz	TC	AgrEvo a/s	6-0195
Sportak EC	prochloraz	EC	AgrEvo a/s	6-0327

1) EW = oil-in-water emulsion concentrate; EC = emulsifiable concentrate; TC = technical material.

4 Plant Growth Regulators

Product	Active ingredient	Formulation type ¹⁾	Company	NERI sample no.
Cerone	ethephon	SL	RP Agro Norden a/s	5-0246
Terpal	ethephon	SL	BASF Danmark a/s	5-0344
Regufon	ethephon	SL	KVK Agro a/s	5-0639
Inter-Ethephon 480	ethephon	SL	Inter-trade a/s	5-0640
Regufon	ethephon	SL	KVK Agro a/s	6-0051
Cerone	ethephon	SL	RP Agro Norden a/s	6-0052
Inter-Ethephon	ethephon	SL	Inter-Trade a/s	6-0053
Terpal	ethephon	SL	BASF Danmark a/s	6-0054
Inter-Ethephon 480	ethephon	SL	Inter-Trade a/s	6-0224
DLG Ethephon 480	ethephon	SL	Agrodan a/s	6-0285

1) SL = solution concentrate.

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