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Thermal decomposition and isomerization of *cis*-permethrin and β-cypermethrin in the solid phase

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Abstract: The stability to heat of *cis*-permethrin and β-cypermethrin in the solid phase was studied and the decomposition products identified. Samples heated at 210 °C in an oven in the dark showed that, in the absence of potassium chlorate (the salt present in smoke-generating formulations of these pyrethroids), *cis*-permethrin was not isomerized, although in the presence of that salt, decomposition was greater and thermal isomerization occured. Other salts of the type KXO_3 or $NaXO_3$, with X being halogen or nitrogen, also led to a considerable thermal isomerization. Heating the insecticides in solution in the presence of potassium chlorate did not produce isomerization in any of the solvents assayed. Salt-catalysed thermal *cis–trans* isomerization was also found for other pyrethroids derived from permethrinic or deltamethrinic acid but not for those derived from chrysanthemic acid. The main thermal degradation processes of *cis*-permethrin and β-cypermethrin decomposition when potassium chlorate was present were cyclopropane isomerization, ester cleavage and subsequent oxidation of the resulting products. Permethrinic acid, 3-phenoxybenzyl chloride, alcohol, aldehyde and acid were identified in both cases, as well as 3-phenoxybenzyl cyanide from β-cypermethrin. A similar decomposition pattern occurred after combustion of pyrethroid fumigant formulations.

Keywords: *cis*-permethrin; β-cypermethrin; thermal stability; thermal decomposition

1 INTRODUCTION

Pyrethroids such as allethrin and furamethrin are widely used as domestic insecticides in the form of mosquito coils, sprays or vaporization units involving heating a mat or a liquid. Thus, their pyrolysis products have been well studied.[1–4] The use of more stable pyrethroids such as *cis*-permethrin and β-cypermethrin in smoke-generating formulations has been studied.[5] These smoke-generating formulations are nowadays important tools for the control of the conenose bugs (*Triatoma infestans* Klug), which are vectors of Chagas' disease.[6–10] They are being successfully tested by now in Argentina in spatial treatments for *Aedes aegypti* L, the mosquito vector of Dengue fever.[11]

The recoveries of different pyrethroids from the smoke of smoke-generating formulations has been measured[5] as well as the influence on their thermal decomposition of foaming agents and antioxidants incorporated with the mixture. In addition, the isomerization process during pyrethroid delivery has been evaluated.

Once conditions for the best recovery and least isomerization of pyrethroids in the smoke had been established, it was necessary to know the decomposition and isomerization products to ensure efficient and safe use of these formulations, as not all the isomers of a pyrethroid are bioactive.[5]

In this work, the stability to heat of *cis*-permethrin and β-cypermethrin in the solid phase has been studied and the decomposition products have been identified. Both pyrethroids are established as active principles with high insecticidal activity against *T infestans*, the principal vector of Chagas' disease in Latin America, and they were studied as components of smoke-generating formulations for vector control. The factors favouring isomerization during combustion of the smoke-generating mixtures was studied, particularly the catalytic effect of the inorganic salt (potassium chlorate) in the mixture.

2 MATERIALS AND METHODS

2.1 Insecticides

cis-Permethrin, [3-phenoxybenzyl (1*RS*)-*cis*-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate] (*cis:trans* 99:1), was crystallized in our laboratory according to a patented procedure,[12] starting from commercial permethrin (97.4%; *cis:trans* 60:40; Chemotecnica (Argentine). β-Cypermethrin [(*RS*)-α-

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cyano-3-phenoxybenzyl (1*RS*)-*cis*-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate] (95.1%) was also provided by Chemotecnica (Argentine). Tetramethrin [(cyclohex-1-ene-1,2-dicarboxymidomethyl (1*RS*)-*cis-trans*-2,2-dimethyl-3-(2-methylprop-1-enyl)-cyclopropanecarboxylate] (94%), phenothrin [3-phenoxybenzyl (1*RS*)-*cis-trans*-2,2-dimethyl-3-(2-methylprop-1-enyl)cyclopropanecarboxylate] (*cis:trans* 15:85) and allethrin [(*RS*)-3-allyl-2-methyl-4-oxocyclopent-2-enyl (1*RS*)-*cis-trans*-2,2-dimethyl-3-(2-methylprop-1-enyl)cyclopropanecarboxylate] (92.2%) were all provided by Sumitomo (Japan). Deltamethrin [(*S*)-α-cyano-3-phenoxybenzyl (1*R*, 3*R*)-*cis*-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate] (98%), λ-cyhalothrin [(*S*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*R*)-*cis*-3-(2-chloro-3,3,3-trifluoropropenyl)-2,2-dimethylcyclopropanecarboxylate + (*R*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*S*)-*cis*-3-(2-chloro-3,3,3-trifluoro-propenyl)-2,2-dimethylcyclopropanecarboxylate, 1 + 1] (99.7%) and β-cyfluthrin [(*SR*)-α-cyano-4-fluoro-3-phenoxybenzyl (1*RS*)-*cis*-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate and (*SR*)-α-cyano-4-fluoro-3-phenoxybenzyl (1*RS*)-*trans*-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate, 1 + 2] (94.5%) were from AgrEvo, ICI and Bayer, respectively.

2.2 Chemicals

All solvents were analytical grade from Sintorgan (Argentina) and distilled when necessary. The inorganic salts were analytical reagent grade (Aldrich, USA). Permethrinic acid, 3-phenoxybenzaldehyde and 3-phenoxybenzyl alcohol were provided by Chemotécnica (Argentina) and 3-phenoxybenzoic acid was from Aldrich (USA).

2.3 Smoke-generating mixtures

These mixtures where prepared with potassium chlorate, kaolin, dextrin and a foaming agent as previously described.[5] Dextrin is an important component of smoke-generating mixtures.

2.4 Chromatographic analysis

Decomposition and isomerization products were analyzed by GC against standards in a Varian Chromatograph 3400 CX, using a 0.53-mm DB-1 column, with temperature programming starting from 70 °C for 1 min, then for 10 °C min^{-1} up to 280 °C and finally 5 min at 280 °C, and by GC–MS using a Finnigan Mat GCQ Trio-2 VG Mass Lab apparatus (USA) with a 50-m DB-5 column having a 0.25 mm film. The column temperature was as before, the injector temperature 240 °C and the detector temperature 280 °C. Carrier gas was helium, and electronic impact (EI) of positive ions at 70 eV and chemical ionization (CI) with methane at 0.08 mm Hg.

2.5 Preparation of authentic samples

3-Phenoxybenzyl chloride was prepared according to a published procedure[13] by treating 3-phenoxybenzyl alcohol (5 g; 0.025 mol) in dichloromethane with thionyl chloride (0.025 mol) added dropwise with vigorous agitation over 4 h. The organic layer was separated and dried over calcium chloride, evaporated and characterized by GC–MS (see Table 4).

3-Phenoxybenzyl cyanide was prepared[14] by treating 3-phenoxybenzyl chloride (0.02 mol) obtained previously with a solution of potassium cyanide (0.04 mol) in *N*-*N*-dimethylformamide + acetonitrile (1 + 1 by volume) and refluxing overnight. The salt was filtered, the precipitate washed and solvent evaporated. The resulting oil was distilled (bp 137 °C/13 mm Hg) and characterized by GC–MS (see Table 4).

2.6 Thermal decomposition in solid phase-potassium chlorate catalysis

Amber glass ampoules (2 ml) containing insecticide (*cis*-permethrin or β-cypermethrin; 20 mg) alone, or insecticide + potassium chlorate (80 + 20), or insecticide + potassium chlorate + dextrin (80 + 10 + 10), or other inorganic salts replacing potassium chlorate were heated in a furnace tube (Thermolyne Type F21100, USA), held at 210 (±0.5) °C. At intervals, samples were removed, dissolved in dichloromethane and analyzed by GC–MS.

2.7 Potassium chlorate-catalyzed isomerization of other pyrethroids

Other pyrethroids (Fig 1) and related compounds were heated under the conditions of Section 2.6 in the presence of potassium chlorate and analyzed by GLC to study whether these were also isomerized.

2.8 Isomerization in solution

Solutions of *cis*-permethrin (1.0 g $litre^{-1}$) in solvents of different polarities (xylene, toluene, acetone, methanol, *N*,*N*-dimethylformamide, hexane + water, chloroform + water, ethanol + water and dioxan + water (all 1 + 1 by volume) containing potassium chlorate (0.2 g $litre^{-1}$) were refluxed for 20 min and then analyzed by GLC as described. A phase-transfer catalyzer (Aliquat® 336) was also added in two-phase systems.

3 RESULTS AND DISCUSSION

3.1 Decomposition and isomerization of *cis*-permethrin

To avoid photochemical reactions, the sample was heated at 210 °C in the dark. This temperature was chosen as the combustion temperature of smoke-generating mixtures as measured by differential scanning calorimetry.[5] The percentage thermal decomposition and isomerization of *cis*-permethrin in the presence and absence of potassium chlorate was plotted against the reaction time. Without potassium chlorate, *cis*-permethrin was the major compound recovered (see Section 3.5). When 200 g kg^{-1} potassium chlorate was added, other important peaks

appeared in the GC trace and the decomposition increased.

No *trans*-permethrin was formed (by isomerization) in the absence of the salt at 210 °C, and *cis*-permethrin was decomposed to other products (Fig 2A). However, in the presence of potassium chlorate there was significant isomerization and the amount of *trans*-permethrin rose to a maximum and then fell (Fig 2B), probably due to other decomposition processes. From the curves of Fig 2A–C, the initial reaction rates for loss of *cis*-permethrin and appearance of *trans*-permethrin were calculated for *cis*-permethrin alone and in the presence of potassium chlorate and of potassium chlorate plus dextrin. The highest rate of *cis*-permethrin decomposition was observed for *cis*-permethrin plus potassium chlorate ($1.18\,min^{-1}$), followed by *cis*-permethrin plus potassium chlorate and dextrin and by *cis*-permethrin alone (0.70 and $0.56\,min^{-1}$ respectively). The rates for appearance of *trans*-permethrin followed the same relative order (0.4, 0.3 and $0\,min^{-1}$).

The isomerization process only occurred in the presence of potassium chlorate and higher amounts of the latter increasing the percentage of isomerization (Paola González Audino, unpublished results); thus, it is not exclusively of thermal origin. In the presence of dextrin, the percentage of isomerization and decomposition were lower, probably because dextrin reacted with potassium chlorate during the thermolysis process at 210 °C.

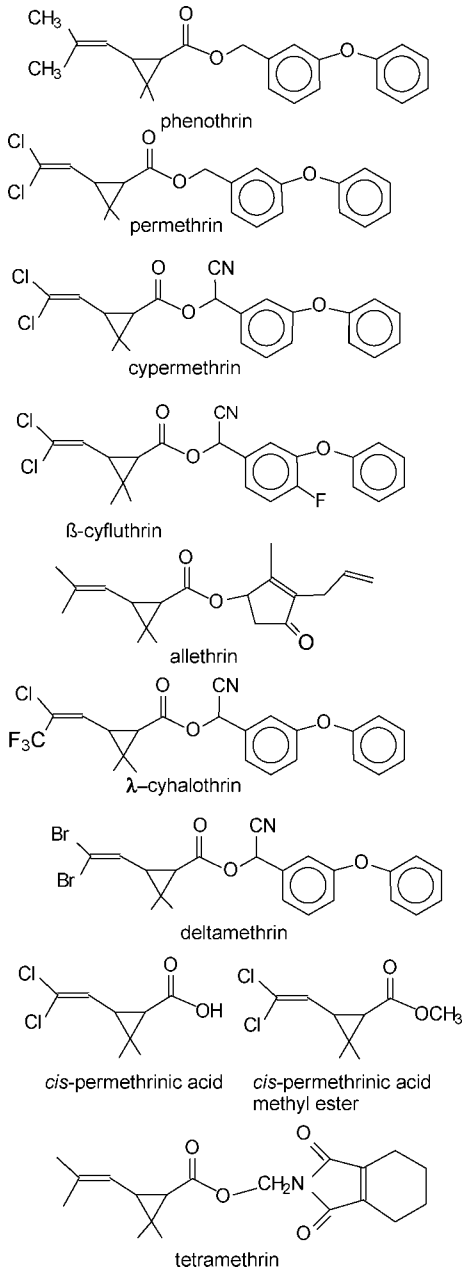


Figure 1. Pyrethroids and precursors submitted to thermal isomerization with potassium chlorate. Stereochemistry is given in Section 2.1.

3.2 Solid phase reaction: catalysis by inorganic salts

Of the other inorganic salts tested for their influence on the thermal isomerization of *cis*-permethrin, only structures of the type KXO_3 or $NaXO_3$, where X is halogen or nitrogen, gave substantial *cis*- to *trans*-permethrin isomerization (Table 1).

3.3 Potassium chlorate-catalyzed isomerization of other pyrethroids

Pyrethroids whose acid moiety is permethrinic or deltamethrinic acid (chlorine and bromine respectively as substituents on vinyl carbons) and whose alcohol moiety is 3-phenoxybenzyl alcohol or its cyano derivative were susceptible to thermal isomerization by potassium chlorate. This susceptibility was retained when fluorine is introduced as a substituent on the alcohol moiety (*β*-cyfluthrin) (Table 2). Chrysanthemic acid derivatives (methyl substituent on vinyl carbons) were not susceptible to isomerization. Neither free permethrinic acid nor its methyl ester were isomerized.

3.4 Isomerization catalyzed by potassium chlorate in solution

Xylene, toluene, acetone, methanol, *N, N*-dimethylformamide and two-phase systems such as hexane–water, chloroform–water, ethanol–water and dioxan–water were used to determine whether isomerization occurred only in the solid state or in solution as well. In none of the cases studied, including those including a phase-transfer catalyzer, was any degree of isomerization obtained.

3.5 Identification of decomposition products

3.5.1 cis-*Permethrin*

An oily brown liquid was obtained as a product of heating. Analysis showed that decomposition was greater in the presence of potassium chlorate and the degree of oxidation of products formed was also increased.

The main reactions observed were isomerization, ester cleavage and, in the presence of the salt, oxidation of the products formed.

Permethrinic acid, 3-phenoxybenzyl chloride, 3-

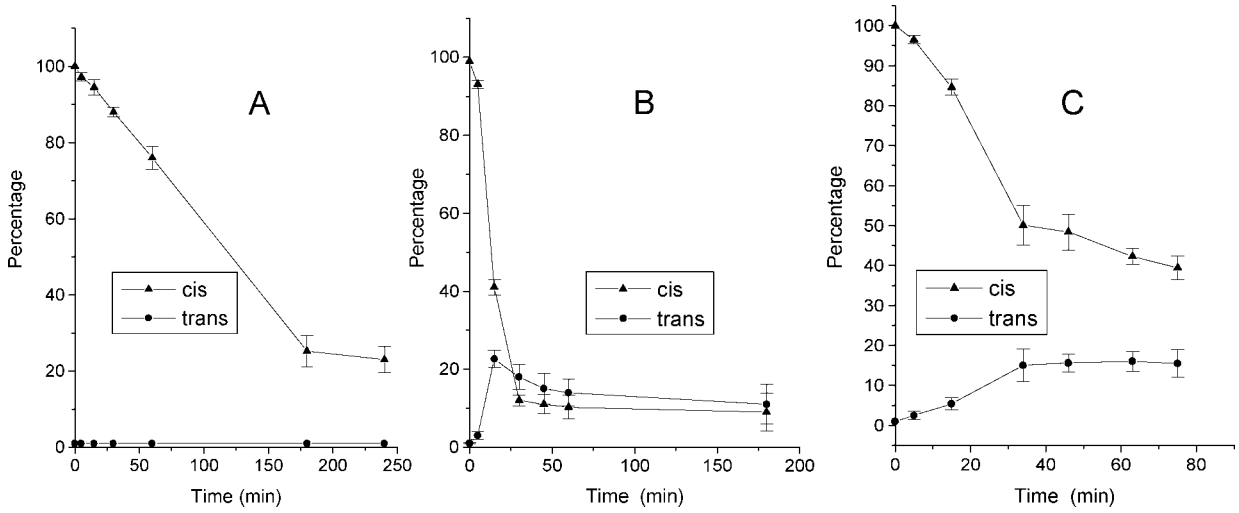


Figure 2. Thermal decomposition and isomerization of *cis*-permethrin vs time: (A) *Cis*-permethrin alone; (B) *Cis*-permethrin + potassium chlorate (80 + 20); (C) *cis*-permethrin + potassium chlorate + dextrin (80 + 10 + 10).

phenoxybenzyl alcohol, 3-phenoxybenzoic acid, 3-phenoxybenzaldehyde and *trans*-permethrin were identified by EI and CI GC–MS (Tables 3 and 4) and co-chromatography with authentic samples.

A peak present in very small amounts, at $R_t = 20.09$, presented an M^+ ion of 318 and a mass spectrum ($m/z = 119$, $m/z = 91$) that could be compatible with the *o*-toluic derivative shown in Fig 3. This compound might be formed in an aromatization process previously described for the thermolysis of some esters of permethrinic acid.[15]

Figure 4 shows a possible thermal-decomposition scheme for *cis*-permethrin in the presence and absence of potassium chlorate. The formation of permethrinic acid, 3-phenoxybenzaldehyde and 3-phenoxybenzyl alcohol has also been described for the photochemical decomposition of permethrin.[16] The loss of hydrogen chloride from permethrin could be the origin of the chloride derivative formed during thermolysis. 3-Phenoxybenzyl chloride must be formed prior to rupture, since 3-phenoxybenzyl alcohol heated to 210 °C in the presence of hydrochloric acid did not produce 3-phenoxybenzyl chloride.

Table 1. Thermal isomerization of *cis*-permethrin at 210 °C for 30 min in the presence of inorganic salts

Salt (200 g kg^{-1})	*Isomerization (%)*
$KClO_3$	47 (±5)
KNO_3[a]	23 (±4)
$NaIO_3$	15 (±3)
$KBrO_3$	11 (±4)
Na NO_2	0
KCl	0
$NaCr_2O_7$	0
$AlCl_3$	0
$NaOCH_3$	0
$KMnO_4$	0
NH_4ClO_4	0

[a] Heating was interrupted at 5 min owing to the high rate of decomposition of the pyrethroid.

Table 2. Thermal isomerization of pyrethroids at 210 °C in the presence of potassium chlorate

Compound	*Isomerization (%)*
Deltamethrin	30 (±4)[a]
λ-Cyhalothrin	0
Allethrin	0
β-Cyfluthrin	7 (±3)[b]
Fenothrin	0
Tetramethrin	0
Furamethrin	0
Permethrinic acid	0
Permethrinic acid methyl ester	0

[a] Measured as percentage of *cis* 1 + *trans* 1 + *trans* 2 (Reference 17).
[b] Measured as percentage of *cis* 1 + *trans* 1.

Product	*m/z (relative abundance)*
Permethrinic acid	209 (M^+ 1,35), 173 (M^- 35, 32), 113 (100), 112 (62), 93 (61)
3-Phenoxybenzyl aldehyde	199 (100, M^+ 1), 171 (M-CO, 84), 227 (M^+ 29)
3-Phenoxybenzoic acid	215 (100, M^+ 1), 243 (M^+ 29), 197 (M^- 17, 12), 153 (18)
3-Phenoxybenzyl alcohol	183 (100, M^- 17), 201 (22, M^+ 1)
β-Cypermethrin	181 (100), 183 (68), 208 (52), 210 (82), 191 (95)[a]

[a] Relative abundance of the ions for the major isomer.

Table 3. CI-MS of identified products

Table 4. EI-MS of identified products

Product	*m/z (Relative abundance)*
Permethrinic acid	173 (100, M-Cl), 163 (M-COOH, 35), 127 (163-HCl, 95), 91 (127-HCl, 78)
3-Phenoxybenzyl alcohol	200 (M^+, 100), 171 (24)
3-Phenoxybenzyl chloride	218 (M^+, 69), 183 (M-Cl, 100)
3-Phenoxybenzyl cyanide	209 (100), 210 (12), 181 (15.34), 141 (21.21), 77 (21.59)
3-Phenoxybenzyl aldehyde	197 (100), 198 (11.6, M^+), 169 (5.74) 141 (21.21), 77 (21.59)
3-Phenoxybenzoic acid	214 (100, M^+), 196 (68 M-H_2O), 169 (84 M-COOH)
Permethrin	183 (100), 184 (12), 163 (25), 390 (<1, IM), 77 (9)
β-Cypermethrin	163 (100), 165 (70), 167 (13), 181 (61.), 209 (21), 208 (12.35), 415 (2 M^+)[a]

[a] Relative abundance of the ions for the major isomer.

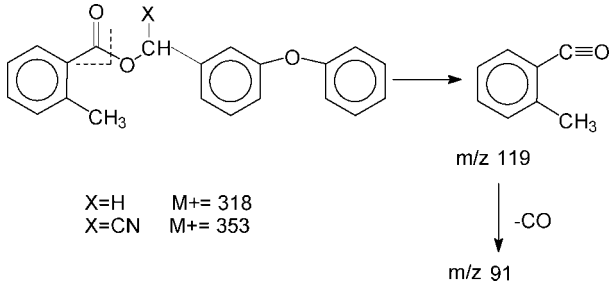


Figure 3. Postulated structure for the unknown decomposition product arising from *cis*-permethrin or β-cypermethrin.

The solutions obtained after combustion of the smoke-generating formulations were analyzed in the same way.[5] In Table 5 the retention times and percentage areas of thermal decomposition products formed in the presence and absence of potassium chlorate are given and compared with the values obtained in the combustion of smoke-generating mixtures for *cis*-permethrin. As can be seen, in smoke-generating mixtures a similar decomposition pattern is obtained.

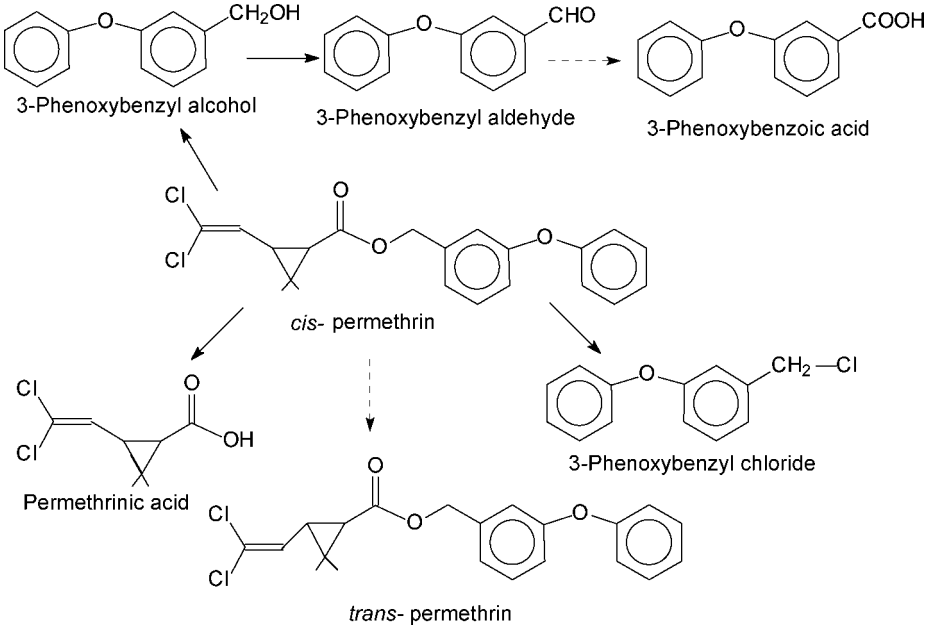


Figure 4. Proposed thermal decomposition pathways for *cis*-permethrin at 210°C in the presence of potassium chlorate.

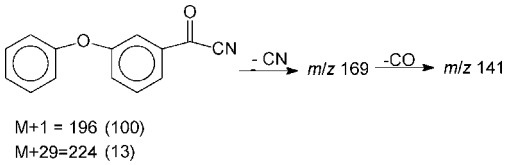
		Areas (%)[a]		
Product	R *(min)*	*Without $KClO_3$*	*With $KClO_3$*	*After combustion*[b]
Permethrinic acid	10.09	0.3	0.9	1.1
3-Phenoxybenzyl aldehyde	13.19	0.3	0.7	0.9
3-Phenoxybenzyl chloride	13.82	0.7	3.7	0.3
3-Phenoxybenzyl alcohol	14.09	0.7	3.3	6.6
3-Phenoxybenzoic acid	15.12	0	2.4	0
Permethrin[c]	21.92	98.0	88.5	91.1

Table 5. Percentage distribution of decomposition products formed from *cis*-permethrin

[a] From *cis*-permethrin at 210°C during 15 min.
[b] In the smoke generating formulation prepared according to Reference 5.
[c] *cis* + *trans*.

Compound	R *(min)*	Areas (%)[a]		
		Without $KClO_3$	*With $KClO_3$*	*After combustion*[b]
Permethrinic acid	10.09	0.4	3.1	0.1
3-Phenoxybenzyl aldehyde	13.19	0.5	22.1	1.9
3-Phenoxybenzoyl cyanide[c]	13.29	—	2.5	0.09
3-Phenoxybenzyl alcohol	14.09	0.3	—	0.5
3-Phenoxybenzyl cyanide	14.88	0.1	4.8	0.2
Unknown[d]	20.96	—	2.9	—
Total cypermethrin	22.76/22.88	98.7	57.0	97.2

[a] From β-cypermethrin at 210°C during 15 min.
[b] From the smoke generating formulation.
[c] Structure assigned according to MS:



[d] Based on CI and EI-MS, an aromatic structure is postulated (see Fig 3).

Table 6. Percentage distribution of decomposition products formed from β-cypermethrin

3.5.2 β-Cypermethrin

An oily brown liquid was obtained on heating, analysis showing that decomposition patterns of β-cypermethrin were similar for those of *cis*-permethrin.

Permethrinic acid, 3-phenoxybenzyl alcohol, 3-phenoxybenzoic acid, 3-phenoxybenzaldehyde, 3-phenoxybenzyl cyanide and cypermethrin isomers were identified by EI and CI GC–MS (Tables 3 and 4) and co-chromatography with authentic samples. 3-Phenoxybenzoyl cyanide as characterized by EI and CI MS (Table 6).

As for permethrin, a possible aromatization process was observed (Table 6). Comparison of thermal decomposition products with those following combustion of the fumigant formulation revealed a similar decomposition pattern (Table 6). Figure 5 shows a possible thermal-decomposition scheme for β-cypermethrin in the presence and absence of potassium chlorate.

This improved understanding of the thermal behaviour of pyrethroid compounds aids the development of smoke-generating formulations. These formulations are part of a programme whose objective is to reduce the financial costs of vector-control programmes and so protect communities at risk from Chagas' disease.

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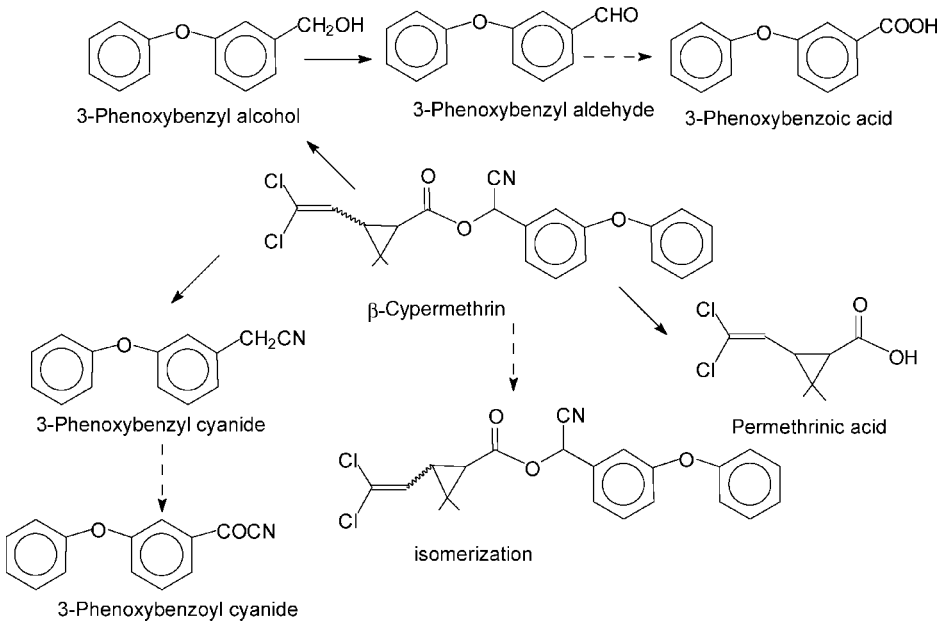

Figure 5. Proposed thermal decomposition pathways for β-cypermethrin at 210°C in the presence of potassium chlorate.

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