Section A1 Applicant

Annex Point IIA1

1.1	**Applicant**	1) **Name:** BASF SE [redacted] **Address:** [redacted] **Telephone:** [redacted] **Fax number:** [redacted] **E-mail:** [redacted]
1.2	**Manufacturer of Active Substance** (if different)	The contact information of the manufacturer of the active substance is given in Appendix 1 to Document IIIA (confidential information)
1.3	**Manufacturer of Products** (if different)	The contact information of the manufacturer of products is given in Appendix 1 to Document IIIA (confidential information)

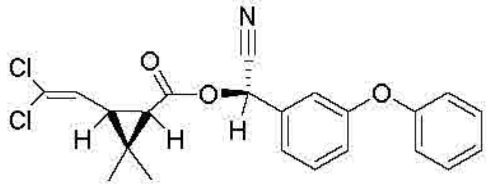
Section A2 Identity of Active Substance

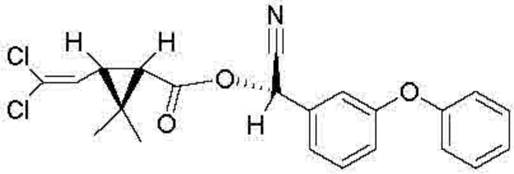
Annex Point IIA2

Official use only

2.1	**Common name (IIA2.1)**	Alphacypermethrin
2.2	**Chemical name (IIA2.2)**	1:1 mixture of the pair of enantiomers (S)-α-cyano-3-phenoxybenzyl-(1R,3R)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate and (R)-α-cyano-3-phenoxybenzyl-(1S,3S)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
2.3	**Manufacturer's development code number(s) (IIA2.3)**	BAS 310 I (current BASF code) *Former code numbers:* NRDC-160 (Cyanamid) AC 900,049 (Cyanamid) CL 900,049 (Cyanamid) WL85871 (Shell) WL085871 (Shell)
2.4	**CAS No and EC numbers (IIA2.4)**	
2.4.1	CAS-No	67375-30-8
2.4.2	EC-No	EINECS: Not allocated Biocide notification no.: [N039]A
2.4.3	Other	CIPAC: 454
2.5	**Molecular and structural formula, molecular mass (IIA2.5)**	
2.5.1	Molecular formula	$C_{22}H_{19}Cl_2NO_3$

2.5.2 Structural formula





2.5.3 Molecular mass 416.3 g/mol

Section A2 Identity of Active Substance

Annex Point IIA2

			Official use only
2.6	**Method of manufacture of the active substance (IIA2.6)**	The information on the method of manufacture is considered to be a trade secret of BASF and therefore claimed to be CONFIDENTIAL. Thus, the manufacturing process is summarised in Appendix 1 to Document III-A (confidential information).	
2.7	**Specification of the purity of the active substance, as appropriate (IIA2.7)**	**Reference A2.7/01:** Fotiou F (1995) Specification, impurities, and analytical profiles for technical grade CL 900,049 (Alphacypermethrin, FASTAC) and CL 900,051 (Cypermethrin, RIPCORD). American Cyanamid Company, Agricultural Products Division, Princenton, NJ, USA, Report No. APBR 457, April 27, 1995 (unpublished), BASF RDI no: AL-380-007. Active ingredient content: > 930 g/kg (93.0 % w/w) ≥ 93.0 % w/w Since the original document allocated to this data requirement is considered to contain commercially sensitive information, thus being a trade secret of BASF, further information is provided in Appendix 1 to Document III-A (confidential information).	
2.8	**Identity of impurities and additives, as appropriate (IIA2.8)**	The information on the identity of impurities is considered to be a trade secret of BASF and therefore claimed to be CONFIDENTIAL. Thus, the corresponding data, summarised in the separate standard format for Section A2.8, are given in Appendix 1 to Document III-A (confidential information). Information on the isomeric composition is given under 2.8.1 below.	
2.8.1	Isomeric composition	1:1 mixture of the pair of enantiomers (S)-α-cyano-3-phenoxybenzyl-(1R,3R)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate and (R)-α-cyano-3-phenoxybenzyl-(1S,3S)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate	
2.9	**The origin of the natural active substance or the precursor(s) of the active substance (IIA2.9)**	Not of natural origin.	

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)	
Date	19/6	
Materials and Methods		
Results and discussion	OK	
Conclusion		
Reliability	1	
Acceptability	Acceptable	
Remarks		
	COMMENTS FROM ...	
Date		
Materials and Methods		
Results and discussion		
Conclusion		
Reliability		
Acceptability		
Remarks		

Section A2.10 — Exposure data in conformity with Annex VIIA to Council Directive 92/32/EEC (OJ No L, 05.06.1992, p. 1) amending Council Directive 67/548/EEC

Annex Point IIA2.10

Subsection

Official use only

2.10.1 Human exposure towards active substance

2.10.1.1 Production

The active substance is manufactured outside the EU. Therefore, no exposure data with respect to this production step are required.

With respect to the formulation of the biocidal example product, exposure-relevant information is given as follows:

i) Description of process	Business Confidential Information, see confidential folder.
ii) Workplace description	Personal protective equipment in handling of raw materials for Tenopa formulations: Safety boots Protective suit made of Tyvec Rubber gloves (EN 374) Protective mask with filter EN 143 P2 Safety glasses (EN 166) Only trained and qualified personnel perform operations. Loading Equipment: Loading of α-Cypermethrin and Flufenoxuron is performed in a properly designed stainless steel hood connected to feed hopper and to an exhaust system. The flow velocity of the exhaust system is checked periodically to guarantee efficiency.
iii) Inhalation exposure	Inhalation exposure is effectively limited by the RPE described above.
iv) Dermal exposure	Dermal exposure is effectively limited by the PPE described above.

2.10.1.2 Intended use(s)

1. Professional Users

i) Description of application process	Tenopa is applied by professional PCOs using low-pressure spraying (< 2 bar).
ii) Workplace description	Any infested area (domestic and public), which typically constitutes, in view of the way of living of the target insects, cracks, crevices, spaces behind furnishings etc.
iii) Inhalation exposure	See document IIB of the dossier

	iv) Dermal exposure	See document IIB of the dossier
	2. Non-professional users including the general public	The biocidal product is not for sale to or use by non-professional users.
	3. Secondary exposure	Acute and chronic phases:
	Inhalation exposure	Adult, children and infant inhaling volatilised residues from treated carpet: See document IIB
	i) Dermal and ingestion	Infant playing on treated carpet and mouthing hands See document IIB
2.10.2	Environmental exposure towards active substance	
2.10.2.1 Production		The active substance is manufactured outside the EU. Therefore, no exposure data with respect to this production step are required. With respect to the formulation of the biocidal example product, exposure-relevant information is given as follows:
	(i) Releases into water	Tenopa formulation and filling areas are segregated by a dedicated drainage. Every leaking of liquid or washing up is collected to two pits; waste water in pits is analysed and treated by activated carbon and filtration on precoat and send to another pit for characterization. If total pesticide is < 1 ppm, the waste water is transferred to an equalisation basin to feed the biological treatment plant. The effluent after treatment is checked periodically by a third party laboratory selected by the manufacturing plant and by a public laboratory under direct control of the environmental protection authority.
	(ii) Releases into air	Exhaust air from loading points is filtered by a multiple bag filter with final HEPA filter. Filter efficiency is checked periodically by a third party company. Results of measurements are stored for authorities.
	(iii) Waste disposal	All waste is submitted to controlled incineration.
2.10.2.2 Intended use(s)		
	Affected compartments:	Percentage distribution (equilibrium processes) between the different environmental compartments as estimated from Mackay level I model: **Reference A.2.10.2/01:** Sendor T (2005) Estimation of distribution in the environment of Alphacypermethrin. EBRC Consulting GmbH, Hannover, Germany, Report No. BAS-051213-01, December 13, 2005 (unpublished), BASF Doc-ID: 2005/1034076.
	Water	0.348%

Section A2.10 Annex Point IIA2.10	**Exposure data in conformity with Annex VIIA to Council Directive 92/32/EEC (OJ No L, 05.06.1992, p. 1) amending Council Directive 67/548/EEC**

Sediment	2.16%
Air	0.0049%
Soil	97.4%

Predicted concentration in the affected compartments	According to the EUSES report (Appendix 1 to Document II-B), the PECs in environmental compartments following biocidal use were estimated as follows (worst-case assumptions):
Water	0.0108 µg/l
Sediment	17.9 µg/kg wwt
Air	Negligible
Soil	1.82×10^{-3} mg/kg wwt

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	Septembre 2008
Materials and Methods	Applicants version is acceptable
Results and discussion	
Conclusion	Adopt applicant´s version including revised version for some parts
Reliability	
Acceptability	Acceptable
Remarks	
	COMMENTS FROM ...
Date	
Materials and Methods	
Results and discussion	
Conclusion	
Reliability	
Acceptability	
Remarks	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)		Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.1	**Melting point, boiling point, relative density (IIA3.1)**								
3.1.1	Melting point	EC method A.1 (84/449/EEC) OECD guideline 102 (1981) Capillary method: Liquid bath method and heat block method were used	Specification as given in Section A2 Batch no.: ST91/118 Expiry date: April 01, 1993 Purity: 97.3 %	*Liquid bath method:* $T_m = 81.4–83.7$ °C *Heat block method:* $T_m = 81.5$ °C	Supportive data	Y	1	**A3.1.1/01:** van Helvoirt JAMW (1992) Alphacypermethrin (FASTAC): Determination of the melting point / melting range. RCC NOTOX B.V., 's-Hertogenbosch, Netherlands, Report no. 057239, January 20, 1992 (unpublished), BASF RDI No.: AL-303-001.	
		OECD guideline 102 (1981) Differential scanning calorimetry (DSC)	Specification as given in Section A2 Batch no.: COD-000166 Purity: 99.3 %	$T_m = 82.3$ °C	Key study	Y	1	**A3.1.1/02:** Kaestel R (2005) Melting point and vapour pressure of alpha-Cypermethrin (TGAI, source: Gujarat Agrochem). BASF Agricultural Center Limburgerhof, Limburgerhof, Germany, Report no. 208906_1, January 07, 2005 (unpublished), BASF Doc ID: 2005/1004903.	

Section A3	Physical and Chemical Properties of Active Substance							
Subsection (Annex point)	**Method/ Guideline**	**Purity/ Specification**	**Results**	**Remarks/ Justification**	**GLP (Y/N)**	**Reliability**	**Reference**	Official use only
3.1.2 Boiling point	Non-guideline study. A "boiling point apparatus" was used, without further description. In addition, thermogravimetric analysis was performed.	Specification as given in Section A2 Batch no.: B2A Expiry date: not stated Purity: 99%	*In the vacuum* (0.07 mm Hg = 0.093 hPa): $T_b = 200°C$ *At atmospheric pressure (thermogravimetric analysis):* Exothermic reaction (decomposition) between 234 and 315°C; Thus, it may be concluded that alphacypermethrin does not have a boiling point at atmospheric pressure.	Supportive data	N	3	**A3.1.2/01:** Salisbury K, Langnere E, Whitehead S (1982) Physical and chemical properties of WL85871 and some compounds important in its synthesis. Shell Research Ltd., Sittingbourne, UK, Report no. SBRN.82.087, March 1982 (unpublished), BASF RDI No.: AL-301-002.	
	OECD 113 Differential scanning calorimetry/ thermogravimetry (DSC/TG)	Specification as given in Section A2 Batch no.: COD-000166 Purity: 99.3%	Onset of decomposition: 220 °C No endothermic reactions other than melting at ca. 82 °C and exothermic decomposition were observed. Thus, it may be concluded that alphacypermethrin does not have a boiling point at atmospheric pressure.	Although the study was formally performed following OECD guideline 113 instead of 102, the method (DSC/TG) is covered by both guidelines. The conduct of the study can therefore be considered as acceptable.	Y	1	**A3.10/01** (cross-reference)	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)		**Method/ Guideline**	**Purity/ Specification**	**Results**	**Remarks/ Justification**	**GLP (Y/N)**	**Reliability**	**Reference**	Official use only
3.1.3	Bulk density/ relative density	EC method A.3 (84/449/EEC) OECD guideline 109 (1981) Gas comparison pycnometer	Specification as given in Section A2 Batch no.: ST91/118 Expiry date: April 01, 1993 Purity: 97.3%	$D_4^{20} = 1.33$	Supportive data	Y	1	**A3.1.3/01:** van Helvoirt JAMW (1992) Alphacypermethrin (FASTAC): Determination of the density. RCC NOTOX B.V., ‘s-Hertogenbosch, Netherlands, Report no. 057241, January 20, 1992 (unpublished), BASF RDI No.: AL-308-001.	
		EC method A.3, chapter 1.4.3 (92/69/EEC) OECD guideline 109 (1995) Gas comparison pycnometer	Specification as given in Section A2 Batch no.: COD-000595 Expiry date: September 01, 2009 Purity: 99.7 %	$D_4^{20} = 1.33$	Although not performed under GLP, the study is considered as valid without restrictions. Key study	N	1	**A3.1.3/02:** Petersen-Thiery M (2009) Alpha-cypermethrin – determination of density. BASF Crop Protection, Limburgerhof, Germany, unnumbered report dated April 24, 2009 (unpublished), BASF Doc ID: 2009/1065032.	
3.2	**Vapour pressure (IIA3.2)**	EC method A.4 (84/449/EEC) Gas saturation method	Specification as given in Section A2 Batch no.: ST91/118 Expiry date: April 01, 1993 Purity: 97.3%	Measurements carried out at 25 and 51°C. p (51°C) = 1.9×10^{-5} Pa p (25°C) = 3.4×10^{-7} Pa (by extrapolation)	Supportive data	Y	1	**A3.2/01:** Harteveld J (1992) Determination of the vapour pressure of Alphacypermethrin. TNO, Rijswijk, Netherlands, Report no. PML 1992C39, April 24, 1992 (unpublished), BASF RDI No.: AL-306-002.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)		Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
		OECD guideline 104 (1981) Thermogravimetry	Specification as given in Section A2 Batch no.: COD-000166 Purity: 99.3 %	p (20 °C) = 2.5×10^{-7} Pa p (25 °C) = 5.6×10^{-7} Pa Both figures were obtained by extrapolation from measurement at 130–170 °C.	In the original report, the figures are given as 2.5×10^{-9} and 5.6×10^{-9} mbar, respectively. Key study	Y	1	**A3.1.1/02** (cross-reference)	
3.2.1	Henry's Law Constant	Calculation based on the vapour pressure at 25°C (3.2 boven~~3.2 above~~) and the water solubility (3.5 onder~~3.5 below~~).	Not applicable (calculation)	H = 0.069 Pa × m³/mol	Supportive data	n.a.	0 (not applicable since not an experimental study)	**A3.2.1/01:** Martin C (1999) Alphacypermethrin (AC 900049): Calculation of Henry's Law Constant. American Cyanamid Co., Cyanamid Agricultural Research Center Ewing, NJ, USA, Report no. ENV 99-004, June 25, 1999, (unpublished), BASF RDI No.: AL-390-025	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
	Calculation based on the vapour pressure at 20 °C (3.2 boven~~3.2 above~~, ref. A3.1.1/02) and the water solubility (3.5 onder~~3.5 below~~).	Not applicable (calculation)	H = 5.687 × 10^{-3} Pa × m³/mol	In the original report, the figure is given as 5.687 × 10^{-6} kPa × m³/mol. Key study	n.a.	0 (not applicable since not an experimental study)	**A3.2.1/02:** Brem G (2005) Henry's Law Constant for alpha-cypermethrin. BASF Product Safety and Registration, unnumbered report dated March 23, 2005, (unpublished), BASF Doc ID: 2005/1038458.	
3.3 Appearance (IIA3.3)								
3.3.1 Physical state	US EPA OPPTS 830.6303 (visual inspection)	Technical grade active substance Specification as given in Section A2 Batch no.: 162 Purity: 96.3%	Solid (crystalline powder)		N	1	**A3.3.1/01:** Cevasco A (1999) Determination of the physical state, color, and odor for alpha-cypermethrin (AC 900049) purified active substance (PAS) and technical active substance (TAS). American Cyanamid Co., Agricultural Products Research Division Princeton, NJ, USA, Report no. P 285, May 20, 1999 (unpublished), BASF RDI No.: AL-123-056	

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
	US EPA OPPTS 830.6303 (visual inspection)	Purified active substance Specification as given in Section A2 Batch no.: AC9575-006 Purity: 99.8%	Solid (fine powder)		N	1	**A3.3.1/01**	
3.3.2 Colour	US EPA OPPTS 830.6302 (visual inspection)	Technical grade active substance Specification as given in Section A2 Batch no.: 162 Purity: 96.3%	Creamy white		N	1	**A3.3.1/01**	
	US EPA OPPTS 830.6302 (visual inspection)	Purified active substance Specification as given in Section A2 Batch no.: AC9575-006 Purity: 99.8%	White		N	1	**A3.3.1/01**	

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.3.3 Odour	US EPA OPPTS 830.6304 (olfactory assessment)	Technical grade active substance Specification as given in Section A2 Batch no.: 162 Purity: 96.3%	Slight chemical odour		N	1	**A3.3.1/01**	
	US EPA OPPTS 830.6304 (olfactory assessment)	Purified active substance Specification as given in Section A2 Batch no.: AC9575-006 Purity: 99.8%	No odour detectable		N	1	**A3.3.1/01**	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.4 Absorption spectra (IIA3.4)							**A3.4/01:** Jones M (1995) Spectral database for Alphacypermethrin (CL 900,049) pure active substance. Cyanamid Forschung GmbH, Schwabenheim, Germany, Report no. APBR 466, June 15, 1995 (unpublished), BASF RDI No.: AL-360-006. **A3.4/02:** Ferri J, Zhang Y (1994) Spectral database for Fastac Technical (CL 900,049). American Cyanamid Co., Agricultural Research Division, Princeton, NJ, USA, Report no. APBR #342, June 23, 1994 (unpublished), BASF RDI No.: AL-360-003.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
UV/VIS	Non-guideline study UV/VIS spectrometry, λ = 200–400 nm	Purified active substance (analytical standard) Batch no.: AC 9575-006 Expiry date: January 03, 2000 Purity: 99.8%	Molar absorption coefficients were determined at the maxima at 240 and 278 nm: $\varepsilon_{240} = 9220\ M^{-1} \times cm^{-1}$ $\varepsilon_{278} = 2037\ M^{-1} \times cm^{-1}$ The spectrum is presented in Figure A3-1.	The spectrum for technical grade active substance (ref. A3.4/02) is basically identical to that of pure a.i.; see original report.	N	2	**A3.4/01** **A3.4/02**	
IR	Guideline: not applicable Method: Infrared spectroscopy, KBr disc	Purified active substance (analytical standard) Batch no.: AC 9575-006 Expiry date: January 03, 2000 Purity: 99.8%	The spectrum is displayed in Figure A3-2. The assignment of absorption bands is presented in Table A3- 1.	The spectrum for technical grade active substance (ref. A3.4.1/02) is basically identical to that of pure a.i.; see original report.	N	2	**A3.4/01** **A3.4/02**	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
^{1}H-NMR	Guideline: not applicable Method: ^{1}H-NMR spectroscopy	Purified active substance (analytical standard) Batch no.: AC 9575-006 Expiry date: January 03, 2000 Purity: 99.8%	The spectrum is displayed in Figure A3- 2 and Figure A3- 4.	The spectrum for technical grade active substance (ref. A3.4.1/02) is basically identical to that of pure a.i.; see original report.	N	2	**A3.4/01** **A3.4/02**	
^{13}C-NMR	Guideline: not applicable Method: ^{13}C-NMR spectroscopy	Purified active substance (analytical standard) Batch no.: AC 9575-006 Expiry date: January 03, 2000 Purity: 99.8%	The spectrum is displayed in Figure A3- 5.	The spectrum for technical grade active substance (ref. A3.4.1/02) is basically identical to that of pure a.i.; see original report.	N	2	**A3.4/01** **A3.4/02**	

Subsection (Annex point)	**Method/ Guideline**	**Purity/ Specification**	**Results**	**Remarks/ Justification**	**GLP (Y/N)**	**Reliability**	**Reference**	Official use only
MS	Guideline: not applicable Method: Electron ionisation (EI) and methane chemical ionisation (CI) mass spectroscopy	Purified active substance (analytical standard) Batch no.: AC 9575-006 Expiry date: January 03, 2000 Purity: 99.8%	EI and CI spectra are displayed in Figure A3-6.	The spectra for technical grade active substance (ref. A3.4.1/02) are basically identical to those of pure a.i.; see original report.	N	2	**A3.4/01** **A3.4/02**	
3.5 Solubility in water (IIA3.5)	EC method A.6 (84/449/EEC) OECD guideline 105 (1981) Column elution method	Specification as given in Section A2 Batch no.: F890380 Expiry date: not stated Purity: 98%	Test temperature: 293 K (20°C) pH Solub. [µg/l] 4 4.59 7 5.80 9 7.87 Distilled water 2.06	Full saturation was achieved.	Y	1	**A3.5/01:** Baldwin M (1990) Alphacypermethrin (FASTAC) water solubility at various pH values. SRC, Sittingbourne, UK, Report no. SBGR.90.158, November 30, 1990 (unpublished), BASF RDI No.: AL-311-002.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.6 Dissociation constant **(-)**				*Justification for non-submission:* Investigation of the molecular structure reveals that Alphacypermethrin does not contain dissociating groups. Due to this lack of potential for dissociation, the experimental testing of the dissociation constant is scientifically not justified.				

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.7 Solubility in organic solvents, including the effect of temperature on solubility (IIIA3.1)	OECD 105, flask method	Specification as given in Section A2 Batch no.: ST91/118 Expiry date: April 01, 1993 Purity: 97.3 %	Test temperature: 21°C Solvent Solub. [g/l] Toluene 596 Methanol 21.3 Ethyl acetate 584 n-hexane 6.5 2-propanol 9.6 Acetone Miscible Dichloromethane Miscible	Supportive data	Y	1	**A3.7/01:** Bohle J (1991) Alphacypermethrin (FASTAC): Determination of the solubility in different solvents. RCC NOTOX, 's-Hertogenbosch, Netherlands, Report no. 057263, December 05, 1991 (unpublished), BASF RDI No.: AL-312-002. **A3.7/02:** Bixler T, Kukel C (2001) CL 900049 (α-cypermethrin): Solubility in acetone:hexane, stability in hexane and freezer storage stability of CL 900049 residues in cattle tissues (muscle, liver, kidney and fat) and milk. BASF Agro Research, Princeton, NJ, USA. Report no. RES 01-002, May 16, 2001 (unpublished), BASF RDI No.: AL-326-035.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
	Non-guideline study primarily focussed on stability of the a.s. in cattle tissues and in the extraction solvent acetone:hexane. Solubility of Alphacypermethrin in acetone:hexane was determined by mixing a defined amount with the solvent followed by visual observation.	Specification as given in Section A2 Batch no.: AC 10194-61 Expiry date: July 17, 2002 Purity: 96 %	Solubility in acetone:hexane: > 500 µg/mL	Supportive data	Y	2	**A3.7/02:** Bixler T, Kukel C (2001) CL 900049 (α-cypermethrin): Solubility in acetone:hexane, stability in hexane and freezer storage stability of CL 900049 residues in cattle tissues (muscle, liver, kidney and fat) and milk. BASF Agro Research, Princeton, NJ, USA. Report no. RES 01-002, May 16, 2001 (unpublished), BASF RDI No.: AL-326-035.	
	EU method A.6 OECD 105 (1995) Shake flask method	Specification as given in Section A2 Batch no.: COD-000166 Purity: 99.3 %	Test temperature: 20 °C Solv. Solub. [g/100 ml] Methanol 2.7 Toluene > 37.4 n-heptane 0.53 Ethyl acetate > 37.6 Dichloromethane > 37.6 Acetone > 37.9	In the original report, solubility is additionally given as g/100 ml saturated solution. However, since the TNsG specify that the results should be given as "g/l solvent", reference is specifically made to pure solvent volume. Key study	Y	1	**A3.7/03:** Daum A (2005) Determination of the solubility in organic solvents at 20 °C of alpha-Cypermethrin (BAS 310I, RegNo 4078193) TGAI. BASF Agricultural Center Limburgerhof, Limburgerhof, Germany, Report no. 208816_1, March 21, 2005 (unpublished), BASF Doc ID: 2005/1007401.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.8 Stability in organic solvents used in b.p. and identity of relevant breakdown products (IIIA3.2)				*Justification for non-submission:* According to the TNsG, such data are only required if organic solvents are included in the biocidal product. Since the biocidal products do not contain organic solvents, data on stability in organic solvents are not considered to be required.				
3.9 Partition coefficient n-octanol/water (IIA3.6)	EC method A.8 (84/449/EEC) OECD 117 (1989) – HPLC method –	Specification as given in Section A2 Batch no.: 02156 Expiry date: July 01, 1994 Purity: 95.4%	log $P_{ow} = 5.5 \pm 0.4$	Since the molecule does not contain dissociating groups, pH dependence of the log P_{ow} is not of relevance. Supportive data	Y	2	**A3.9/01:** Langner EJ, Fisk P (1993) Alphacypermethrin (Fastac): Estimation of the octanol-water partition coefficient. SRC, Sittingbourne, UK, Report no. SBTR.92.030, March 12, 1993 (unpublished), BASF RDI No.: AL-315-001.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
	OECD 117 (1989) – HPLC method –	Specification as given in Section A2 Batch no.: COD-000166 Purity: 99.3 %	log P_{ow} = 5.67	Key study	Y	1	**A3.9/02:** Daum A (2005) of the Determination of the octanol/water partition coefficient of alpha-Cypermethrin (BAS 310I, RegNo 4078193) TGAI at 20 °C. BASF Agricultural Center Limburgerhof, Limburgerhof, Germany, Report no. 208828_1, February 09, 2005 (unpublished), BASF Doc ID: 2005/1004705.	
3.10 Thermal stability, identity of relevant breakdown products (IIA3.7)	OECD 113 Differential scanning calorimetry/ thermogravimetry (DSC/TG)	Specification as given in Section A2 Batch no.: COD-000166 Purity: 99.3 %	Onset of decomposition: 220 °C Thus, according to the criteria of OECD 113, the test substance is stable at room temperature.		Y	1	**A3.10/01:** Kroehl T (2006) Thermal stability of alpha-cypermethrin (Reg. No. 4078193, BAS 310 I) according to OECD guideline 113. BASF AG, Limburgerhof, Germany, Report no. 168571_2, February 09, 2006 (unpublished), BASF DocID: 2006/1008229.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.11 Flammability, including auto-flammability and identity of combustion products (IIA3.8)	Flammability: EC method A.10 (84/449/EEC) Auto-flammability: EC method A.16 (84/449/EEC)	Specification as given in Section A2 Batch no.: ST91/118 Expiry date: April 01, 1993 Purity: 97.3%	*Flammability:* The test substance could not be ignited. Alphacypermethrin is not "highly flammable". *Auto-flammability:* No self-ignition was observed. Alphacypermethrin is not auto-flammable.		Y	1	**A3.11/01:** van Helvoirt JAMW (1991) Determination of the flammability of Alphacypermethrin. RCC NOTOX, 's-Hertogenbosch, Netherlands, Report no. 057296, November 13, 1991 (unpublished), BASF RDI No.: AL-330-001. **A3.11/02:** van Helvoirt JAMW (1991) Determination of the auto-flammability of Alphacypermethrin. RCC NOTOX, 's-Hertogenbosch, Netherlands, Report no. 057318, November 13, 1991 (unpublished), BASF RDI No.: AL-330-002.	

Section A3 **Physical and Chemical Properties of Active Substance**

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
Flammability in contact with water				*Justification for non-submission:* Experimental data are not submitted since these are considered to be scientifically unjustified: From the structural formula and the composition of the substance it can be safely concluded that the substance does not evolve any flammable gases in contact with water or humid air. In addition, experience in use (alphacypermethrin formulated as an aqueous dispersion) clearly indicates the absence of this property.				

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
Pyrophoric properties				*Justification for non-submission:* Experimental data are not submitted since these are considered to be scientifically unjustified: From the structural formula and the composition of the substance it can be safely concluded that the substance is stable in air at room temperature and is not pyrophoric.				
3.12 Flash-point (IIA3.9)				*Justification for non-submission:* A test on this endpoint must be provided for liquids whose vapours can be ignited; this does not apply to Alphacypermethrin, which is a solid with low vapour pressure.				

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.13 Surface tension (IIA3.10)				*Justification for non-submission:* EC method A.5 expressly states that the surface tension of a compound need not to be determined if the water solubility is lower than 1 mg/l. In view of the results reported under 3.5 boven~~3.5 above~~, experimental determination of the surface tension is not considered to be required.				
3.14 Viscosity (-)				*Justification for non-submission:* The substance is solid, which is why viscosity cannot be determined. Thus, this data requirement is not applicable.				

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.15 Explosive properties (IIA3.11)	EC method A.14 (84/449/EEC)	Specification as given in Section A2 Batch no.: ST91/118 Expiry date: April 01, 1993 Purity: 97.3%	Alphacypermethrin was not sensitive to thermal and mechanical stimuli. Thus, the substance is not explosive.		Y	1	**A3.15/01:** van Helvoirt JAMW (1991) Determination of the explosive properties of Alphacypermethrin. RCC NOTOX, 's-Hertogenbosch, Netherlands, Report no. 057307, November 13, 1991 (unpublished), BASF RDI No.: AL-334-001.	
3.16 Oxidizing properties (IIA3.12)	EC method A.17 (84/449/EEC) - Full test - Test with inert material - Test under inert atmosphere	Specification as given in Section A2 Batch no.: ST91/118 Expiry date: April 01, 1993 Purity: 97.3%	In the first test, at a cellulose/ test substance ratio of 80/20, the burning rate was close to the highest burning rate of the reference. However, the burning behaviour was different (combustion only at the surface), indicating a false positive result. The tests both with inert material (celite) and under inert atmosphere were clearly negative. Thus, Alphacypermethrin is not oxidising.		Y	1	**A3.16/01:** van Helvoirt JAMW (1991) Alphacypermethrin (FASTAC): Determination of the oxidizing properties. RCC NOTOX, 's-Hertogenbosch, Netherlands, Report no. 057329, December 1013, 1991 (unpublished), BASF RDI No.: AL-356-001.	

Section A3 Physical and Chemical Properties of Active Substance

Subsection (Annex point)	Method/ Guideline	Purity/ Specification	Results	Remarks/ Justification	GLP (Y/N)	Reliability	Reference	Official use only
3.17 Reactivity towards container material (IIA3.13)			Suitable container material, based on experience in use and the chemical structure, is specified as follows: Polyethylene is a suitable material for packaging of the active substance. Any potential for reaction and/or corrosion can be excluded based on the chemical structure of the active substance. Materials to avoid for direct contact: plain mild steel, tinplate or other metal packaging.					

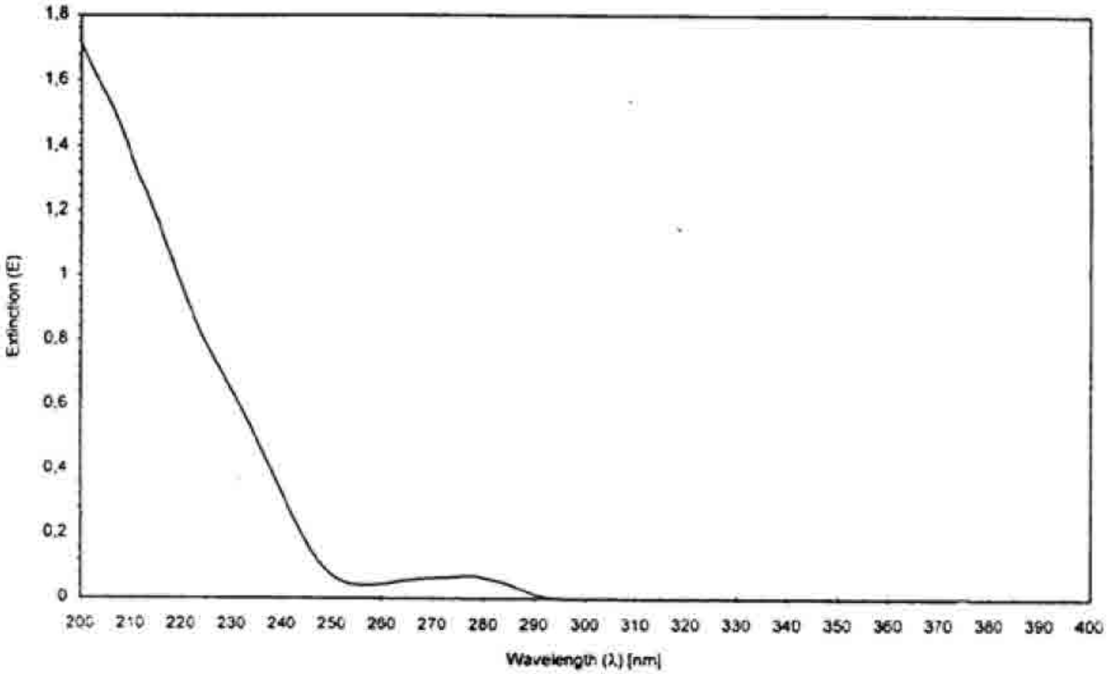


Figure A3- 1: UV/VIS spectrum of Alphacypermethrin.

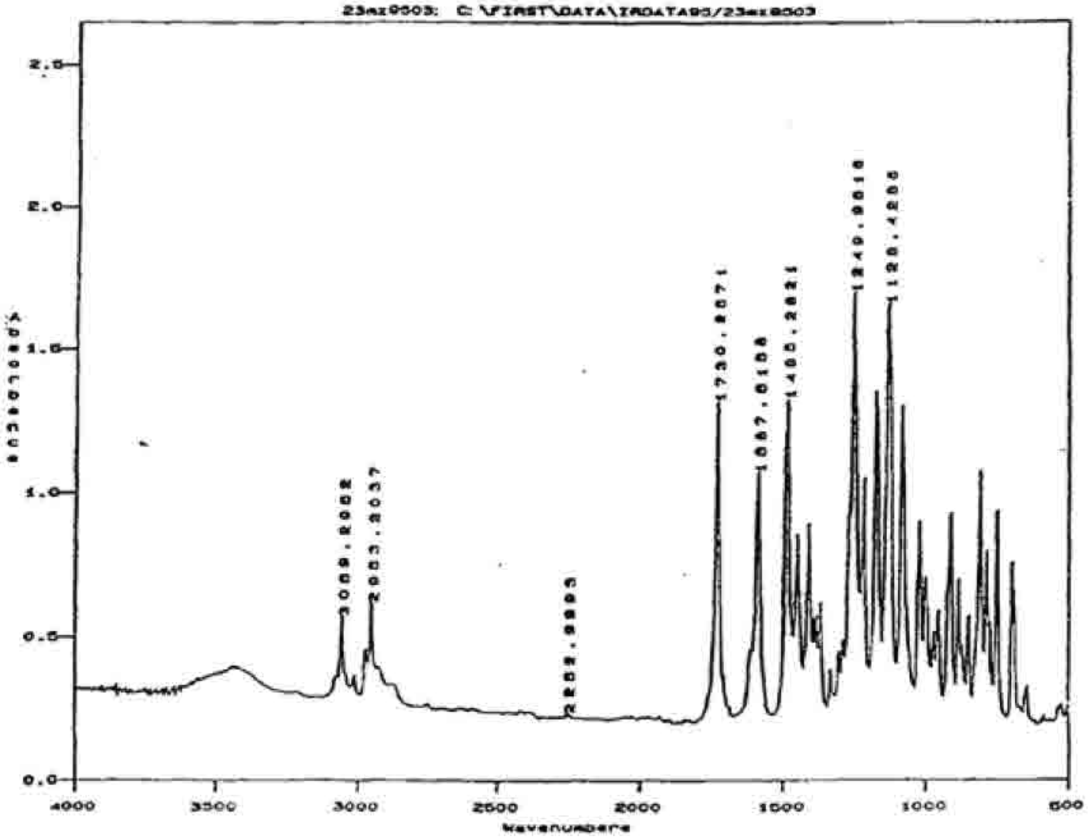


Figure A3- 2: Diffuse reflectance IR spectrum of Alphacypermethrin.

Table A3- 1: Assignments for infrared absorption bands of Alphacypermethrin.

IR band wavenumber [cm^{-1}]	**Assignment**
2953	C-H stretch
2253	-C ≡N stretch
1730	Ester, C=O stretch

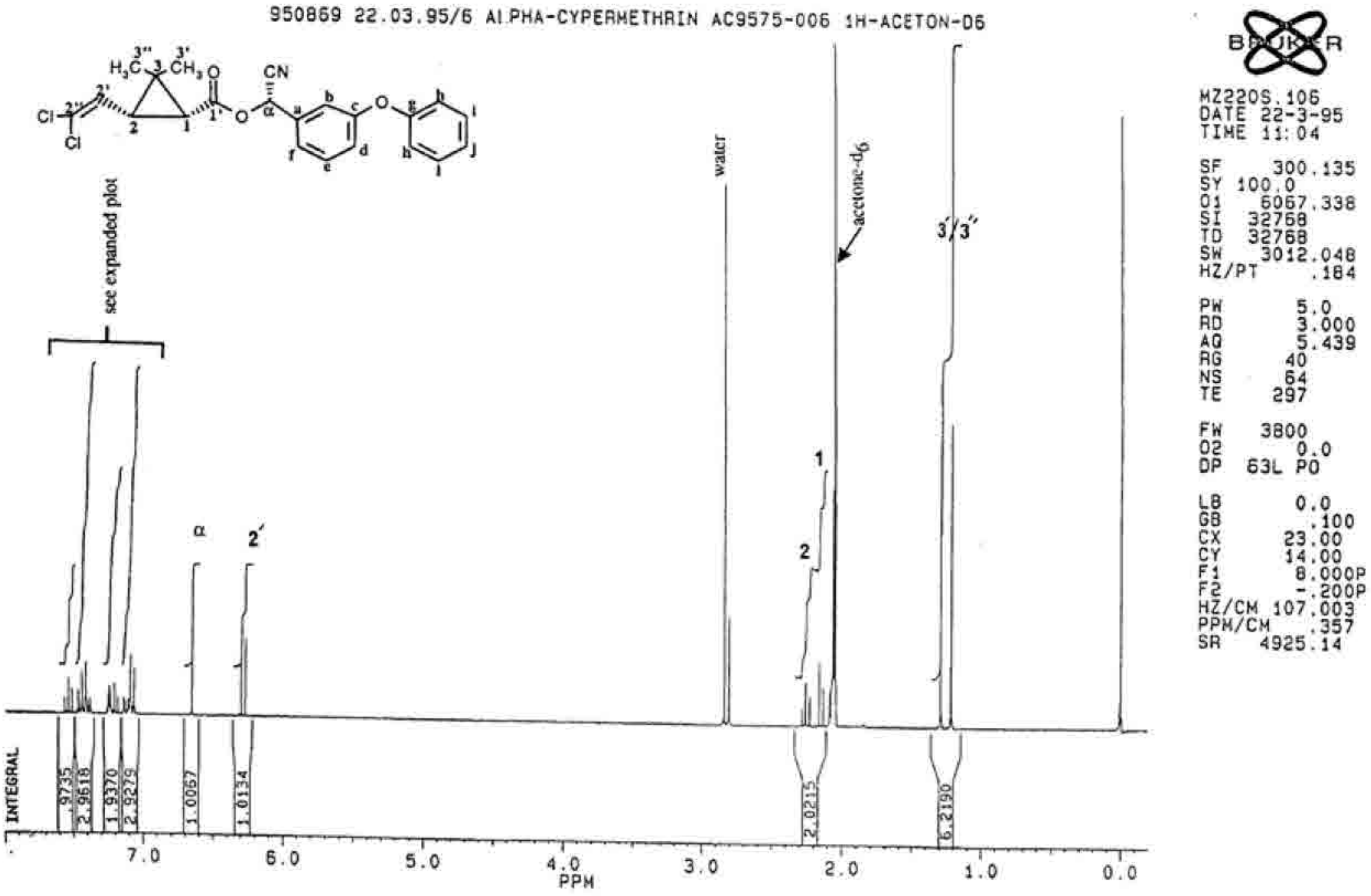
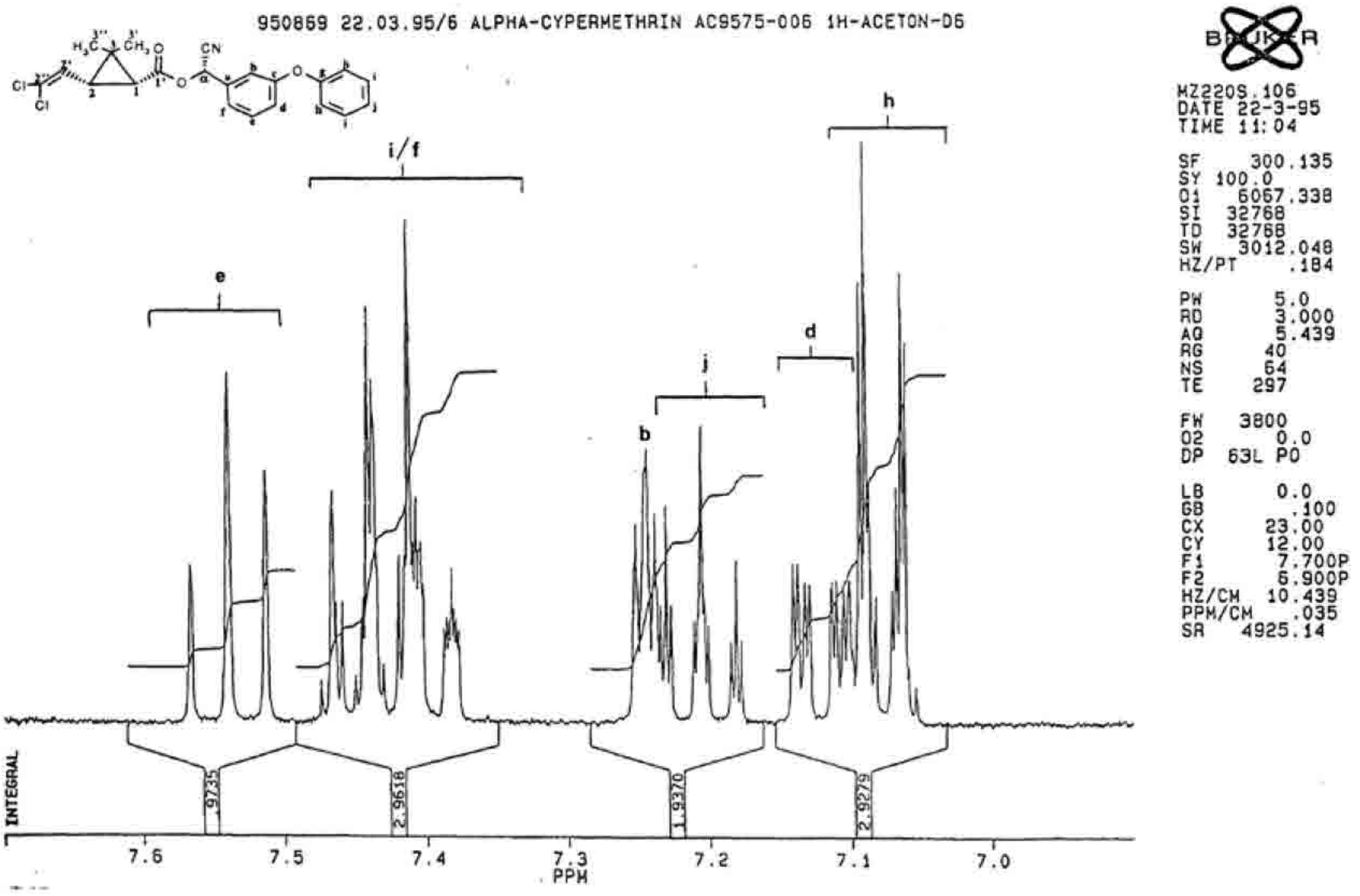


Figure A3- 3: Proton NMR spectrum of Alphacypermethrin.



950869 22.03.95/6 ALPHA-CYPERMETHRIN AC9575-006 1H-ACETON-D6

```
MZ220S.106
DATE 22-3-95
TIME 11:04

SF      300.135
SY  100.0
O1     6067.338
SI   32768
TD   32768
SW     3012.048
HZ/PT      .184

PW         5.0
RD         3.000
AQ         5.439
RG        40
NS        64
TE       297

FW    3800
O2         0.0
DP   63L PO

LB         0.0
GB          .100
CX        23.00
CY        12.00
F1         7.700P
F2         6.900P
HZ/CM   10.439
PPM/CM     .035
SR    4925.14
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Figure A3- 4: Proton NMR spectrum of Alphacypermethrin, expanded plot from Figure A3- 3.

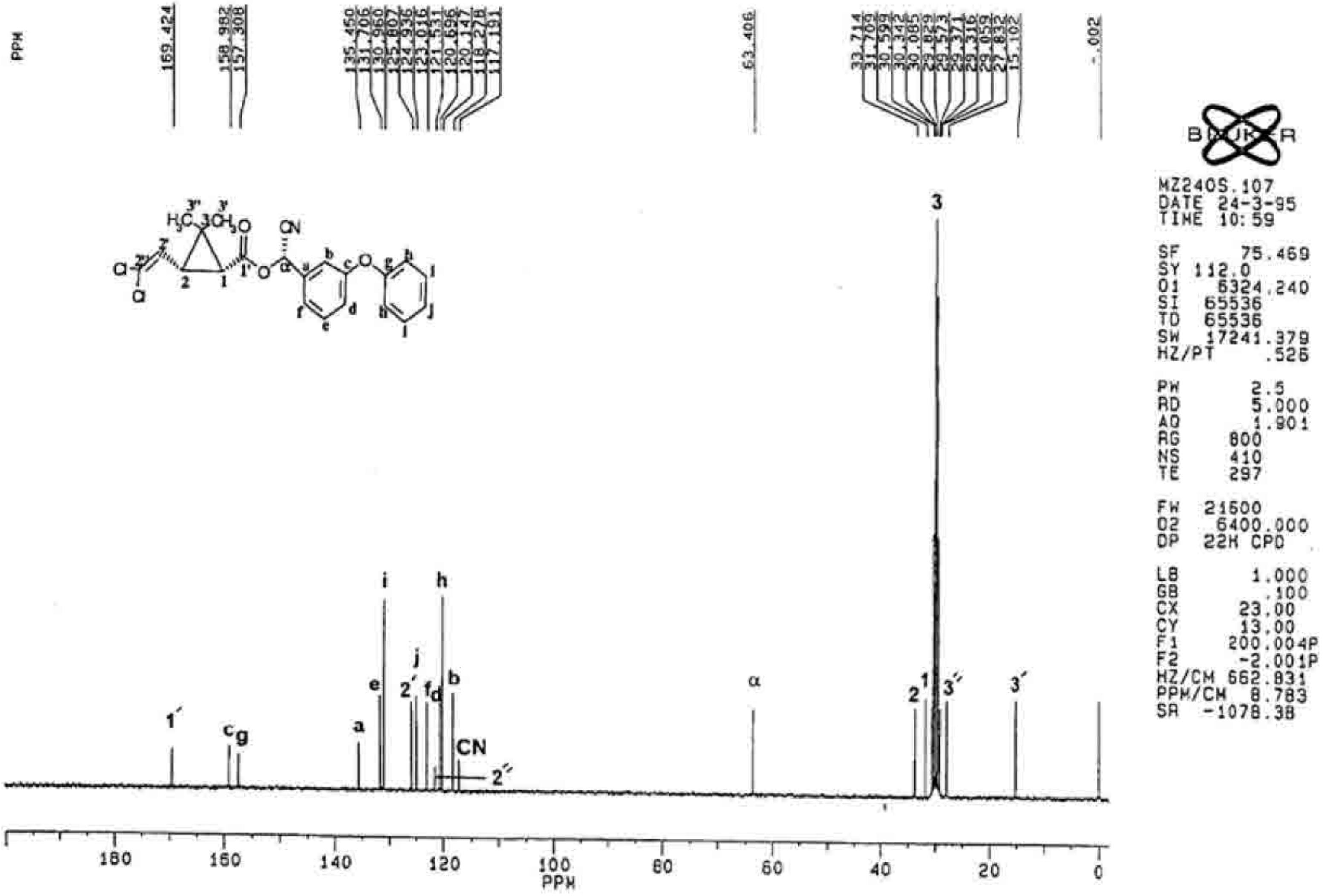

Figure A3- 5: ^{13}C-NMR spectrum of Alphacypermethrin.

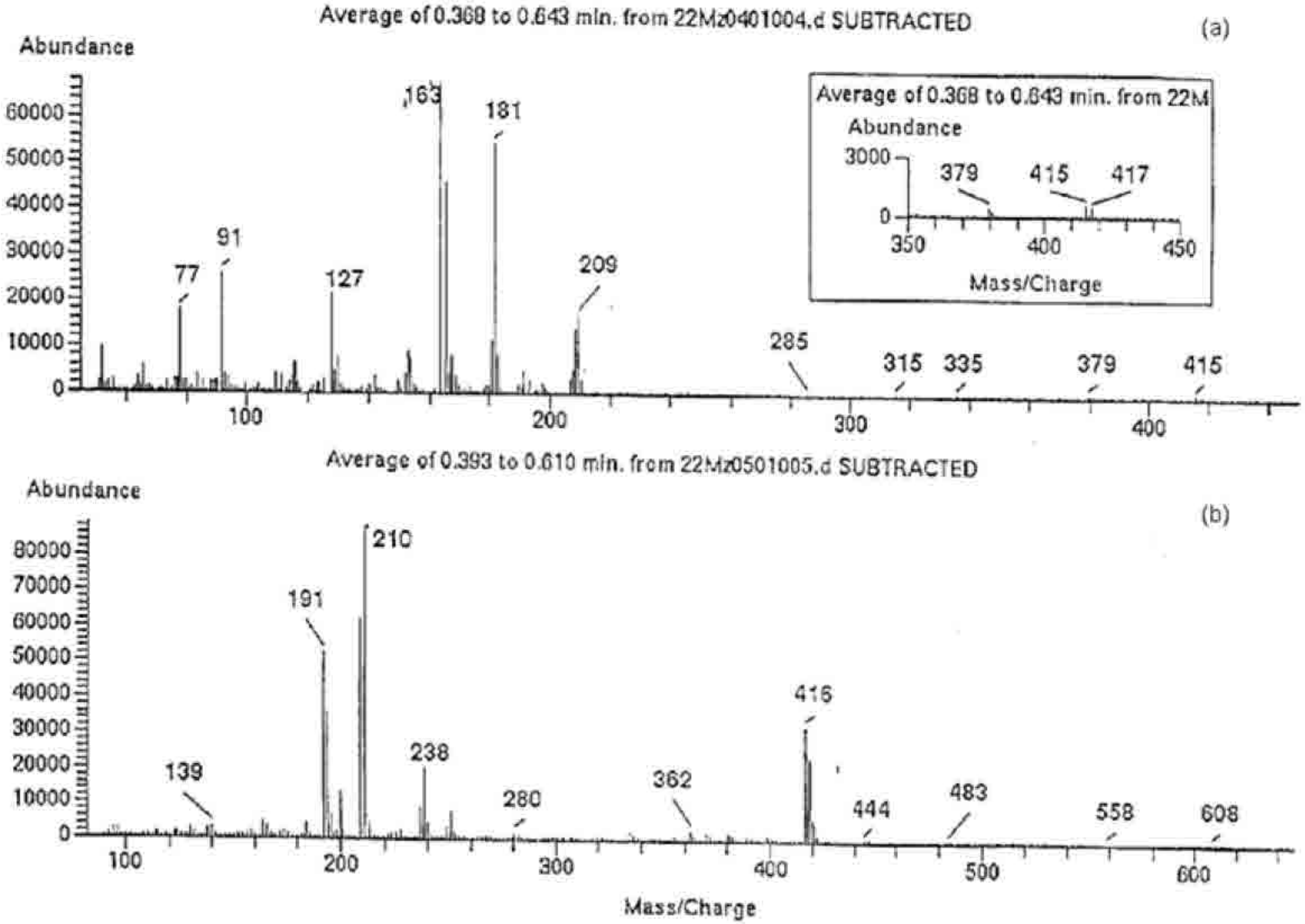

Figure A3- 6: Mass spectra of Alphacypermethrin; (a) electron ionisation (EI) spectrum, (b) methane chemical ionisation (CI) spectrum.

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)	
Date	May 2009	
Materials and Methods	acceptable	
Results and discussion	acceptable	
Conclusion	accepted	
Reliability		
Acceptability		
Remarks		
	COMMENTS FROM ...	
Date		
Materials and Methods		
Results and discussion		
Conclusion		
Reliability		
Acceptability		
Remarks		

Evaluation by Competent Authorities	
3.9 Partition coefficient n-octanol/water	

	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	Aug 2012
Materials and Methods	acceptable
Results and discussion	acceptable
Conclusion	Reliability has been set on 2, because temperature at which the test was conducted is missing in the test report. It is proposed to mention that 'room temperature' is the appropriate way to define in the LoEP the temperature at which the Log Pow was determined here.
Reliability	2
Acceptability	yes
Remarks	
	COMMENTS FROM ...
Date	
Materials and Methods	
Results and discussion	
Conclusion	
Reliability	
Acceptability	
Remarks	

Section A4.1 **Annex Point IIA4.1**	**Analytical methods for detection and identification** **– Purity of technical grade active substance –** **– Impurities in technical grade active substance –** **– Isomeric composition of technical grade active substance –**
	Remark: The analytical methods as specified in the section header contain commercially sensitive information. Thus, they are considered as trade secrets of BASF and are therefore allocated to the confidential file (for the study summaries, see Appendix 1 to Document III-A – Confidential data).

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	
Materials and Methods	
Results and discussion	OK
Conclusion	
Reliability	1
Acceptability	Acceptable
Remarks	
	COMMENTS FROM ...
Date	
Materials and Methods	
Results and discussion	
Conclusion	
Reliability	
Acceptability	
Remarks	

Section A4.2
Annex Point IIA4.2

Analytical Methods for Detection and Identification in (a) soil

Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/01:** Werle H (1999) Alphacypermethrin (CL 900049): Validation of method SAMS 354-2 for the determination of residues in soils. BioChem GmbH, Karlsruhe, Germany, Report No. 98 50 40 421, April 16, 1999 (unpublished). (BASF-Ref.: AL-242-006)
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes SANCO/8064/VI/97 rev. 4; and BBA guideline: Residue analytical methods for post-registration control purposes (July 21, 1998)
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	None

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	Samples of a German standard soil (LUFA Speyer 2.3) previously fortified with Alphacypermethrin at levels of 0.05 and 0.50 mg/kg were mixed with anhydrous sodium sulphate. Thereafter the mixture was extracted with acetone and n-hexane (1+1 v/v).
3.1.2	Cleanup	The extracts were washed with water to remove the acetone and cleaned up by liquid-solid chromatography using Florisil.
3.2	**Detection**	
3.2.1	Separation method	Capillary gas chromatography (GC). Minor adaptations of the original method (e.g. column, injection, column temperature program) were made.
3.2.2	Detector	Electron capture detector (ECD) for all samples. Mass selective detector (MSD) for one control sample and one representative fortified sample at 0.5 mg/kg.

Section A4.2 Analytical Methods for Detection and Identification in (a) soil

Annex Point IIA4.2

<table>
<tr><td>3.2.3</td><td>Standard(s)</td><td>Alphacypermethrin (CL 900,049), purity: 96.1%</td></tr>
<tr><td>3.2.4</td><td>Interfering substance(s)</td><td>None</td></tr>
<tr><td>3.3</td><td>Linearity</td><td></td></tr>
<tr><td>3.3.1</td><td>Calibration range</td><td>0.0126, 0.0251, 0.0502, and 0.251 µg/mL</td></tr>
<tr><td>3.3.2</td><td>Number of measurements</td><td>1 (highest concentration was analysed twice)</td></tr>
<tr><td>3.3.3</td><td>Linearity</td><td>The method was determined to be linear over the calibration range.
$y = 117418.557\,x + 478.68$, $r^2 = 0.9984$</td></tr>
<tr><td>3.4</td><td>Specificity: interfering substances</td><td>For confirmatory reasons, one control sample, as well as one representative fortified sample (0.5 mg/kg) were analysed by GC/MS. These investigations proved unequivocally the peak identity by comparing the retention time and the EI mass spectra of the analyte in the sample with the reference substance.</td></tr>
<tr><td>3.5</td><td>Recovery rates and relative standard deviations</td><td>
<table>
<tr><th>Fortification</th><th>Recovery</th><th>RSD</th><th>n</th></tr>
<tr><td>0.05 mg/kg</td><td>90 %</td><td>6.1 %</td><td>4</td></tr>
<tr><td>0.5 mg/kg</td><td>88 %</td><td>4.7 %</td><td>5</td></tr>
</table>
Individual recovery values obtained per fortification level ranged between 83 and 99%. One outlier was identified by Q-test at 0.05 mg/kg (123%).</td></tr>
<tr><td>3.6</td><td>Limit of determination</td><td>LOQ: 0.05 mg/kg
LOD: 0.01 mg/kg</td></tr>
<tr><td>3.7</td><td>Precision</td><td></td></tr>
<tr><td>3.7.1</td><td>Repeatability</td><td>The method was successfully validated with four to five values at both fortification levels, with recoveries in the range from 70% to 110% and relative standard deviations below 20%.</td></tr>
<tr><td>3.7.2</td><td>Independent laboratory validation</td><td>Not applicable</td></tr>
</table>

Section A4.2 Annex Point IIA4.2	**Analytical Methods for Detection and Identification in (a) soil**

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	For the validation of the analytical method SAMS 354-2 for the determination of Alphacypermethrin residues in soils, aliquots of a German standard soil (LUFA Speyer 2.3) were fortified with Alphacypermethrin at levels of 0.05 and 0.50 mg/kg. Five replicates were used per fortification level, as well as two control samples, and analysed by GC/ECD. In addition, one control sample and one representative fortified sample (0.5 mg/kg) were analysed by GC/MS for confirmatory reasons.
4.2	**Conclusion**	The results for linearity, precision (repeatability) and accuracy demonstrate that the analytical method SAMS 354-2 is suitable for the determination of Alphacypermethrin in soil.
4.2.1	Reliability	1
4.2.2	Deficiencies	None

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)	
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	COMMENTS FROM ...	

Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/02:** Müller-Kallert, HM (1992) Development of a method for the determination of Alphacypermethrin in air. RCC Umweltchemie AG, Itingen, Switzerland, Report No. 249120, April 22, 1992 (unpublished). (BASF-Ref.: AL-241-002) **Remark:** In principle, analytical methods for the determination and identification of Alphacypermethrin in air are not required due to the limited volatility of the test substance (p (25°C) = 3.4×10^{-7} Pa) and since the application technique does not involve high-pressure spraying. However, an available study is submitted for the sake of completeness.
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes BBA guideline: part I (1-2)
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	Not applicable

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Air sampling	Air of known volume was spiked with the test item at 4 different levels (0.02, 0.1, 0.2, 0.5 $\mu g/m^3$) and was drawn through a tube trap packed with Chromosorb 102 as trapping adsorbent. The temperature, sampling time and relative humidity were varied from 21°C to 30°C, 4 hours to 8 hours and 40 to 90%, respectively.
3.1.2	Desorption	The trapping material was desorbed by solvent extraction with acetone, followed by micro-concentration.

Section A4.2 Analytical Methods for Detection and Identification in (b) air

Annex Point IIA4.2

3.2 Detection

3.2.1 Separation method: The test substance is analysed by capillary gas chromatography (GC).

3.2.2 Detector: Nitrogen phosphorus detector (NPD)

3.2.3 Standard(s): Alphacypermethrin, batch no.: 003/88, purity: 99.8%

3.3 Desorption efficiency

Sampling time: ca. 1 min

Sampling flow-rate: approx. 250 mL/min

Concentration	Recoveries	Mean recovery
0.02 $\mu g/m^3$	109%	100%
0.02 $\mu g/m^3$	102%	
0.1 $\mu g/m^3$	90%	
0.1 $\mu g/m^3$	99%	

3.4 Trapping efficiency

The trapping efficiency was examined at temperatures of 21 ± 2°C, and 30 ± 2°C, a relative humidity of 40 ± 10%, and 90 ± 10%, a concentration level of 0.5 $\mu g/m^3$ (50-fold of the nominal determination limit of 0.01 $\mu g/m^3$) and sampling times of 4 or 8 hours. The air flow rate varied between 250 and 500 mL/min and the sample volume was 120 or 240 l. The trapping efficiency was not significantly influenced by temperature, sampling time and relative humidity and was in a range of 80–106% (average of all trapping efficiencies: 92.5%).

3.5 Stability

Alphacypermethrin which was trapped on Chromosorb 102 at a concentration of 0.2 $\mu g/m^3$ is stable for at least eight weeks at 4°C and also at room temperature.

3.6 Linearity

3.6.1 Calibration range: 0.01, 0.02, 0.0.5, 0.1, 0.2, and 0.5 $\mu g/mL$

3.6.2 Number of measurements: 1 per concentration

3.6.3 Linearity: The method was found to be linear over the calibration range.

$y = \exp(1.07 \ln x + 0.0000402)$, $r = 0.9984$

3.7 Recovery rates and relative standard deviations

Average recovery rates at 0.02–0.5 $\mu g/m^3$ were in a range of 83.0–104.6% calculated as averages of 5 and 4 values.

Concentration	Temp.	Humidity	Recovery	RSD	n
0.02 $\mu g/m^3$	21°C	65%	104.6%	7.84%	5
0.1 $\mu g/m^3$	21°C	65%	92.6%	8.14%	5
0.5 $\mu g/m^3$	21°C	65%	83.0%	9.21%	5
0.2 $\mu g/m^3$	30°C	90%	92.5%	1.40%	4

3.8 Limit of determination

LOD: 0.01 $\mu g/m^3$
LOQ: 0.02 $\mu g/m^3$

Section A4.2 Annex Point IIA4.2		**Analytical Methods for Detection and Identification in (b) air**
		4 APPLICANT'S SUMMARY AND CONCLUSION
4.1	**Materials and methods**	Air of known volume was spiked with the test item at 4 different levels (0.02, 0.1, 0.2, 0.5 µg/m³) and was drawn through a tube trap packed with Chromosorb 102 as trapping adsorbent. The temperature, sampling time and relative humidity were varied from 21°C to 30°C, 4 hours to 8 hours and 40 to 90%, respectively. The trapping material was desorbed by solvent extraction with acetone, followed by micro-concentration and analysis by GC/NPD.
4.2	**Conclusion**	The method is suitable for Alphacypermethrin analysis in air with a LOQ of 0.02 µg/m³.
4.2.1	Reliability	1
4.2.2	Deficiencies	None

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	COMMENTS FROM ...

Section A4.2
Annex Point IIA4.2

Analytical Methods for Detection and Identification in (c) water

Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/03:** Werle H (1999) Alphacypermethrin (CL 900049): Validation of method SAMS 469-2 for the determination of residues in surface water. BioChem GmbH, Karlsruhe, Germany, Report No. 98 50 40 420, March 08, 1999 (unpublished). (BASF-Ref.: AL-243-006)
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes SANCO/8064/VI/97 rev. 4; and BBA guideline: Residue analytical methods for post-registration control purposes (July 21, 1998)
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	None

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	Samples of surface water from the river Rhine were extracted by means of liquid/liquid partitioning with n-hexane.
3.1.2	Cleanup	The extracts were cleaned up using a Florisil disposable cartridge.
3.2	**Detection**	
3.2.1	Separation method	Capillary gas chromatography Minor adaptations of the original method SAMS 469-2 (e.g. column, injection, column temperature program) were made.
3.2.2	Detector	Electron capture detection (ECD) Mass selective detector (MSD) for one control sample as well as one sample per fortification level (0.05, 0.5 μg/l)
3.2.3	Standard(s)	Alphacypermethrin (CL 900,049), purity: 96.1%

Section A4.2 **Annex Point IIA4.2**		**Analytical Methods for Detection and Identification in (c) water**
3.2.4	Interfering substance(s)	None
3.3	**Linearity**	
3.3.1	Calibration range	0.502, 0.251, 0.0502, 0.0251, 0.0126 µg/mL
3.3.2	Number of measurements	2
3.3.3	Linearity	The method was determined to be linear over the calibration range. $y = 90114.01\,x + 504.08$, $r^2 = 0.9926$
3.4	**Specificity: interfering substances**	For confirmatory reasons, one control sample, as well as one sample per fortification level (0.05, 0.5 µg/l) were analysed with GC/MS. These investigations proved unequivocally the peak identity by comparing the retention time and the EI mass spectra of the analyte in the sample with the reference substance.
3.5	**Recovery rates and relative standard deviations**	see table below

Fortification	Recovery	RSD	n
0.05 µg/l	88 %	8.9 %	5
0.5 µg/l	89 %	12 %	5

Individual recovery values obtained per fortification level ranged between 78 and 107%. No outlier was identified.

3.6	**Limit of determination**	LOQ: 0.05 µg/l LOD: 0.01 µg/l
3.7	**Precision**	
3.7.1	Repeatability	The method was successfully validated using five replicates at both fortification levels, with recoveries in the range from 70% to 110% and relative standard deviations below 20%.
3.7.2	Independent laboratory validation	Not applicable.

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	For the validation of the analytical method SAMS 469-2 for the determination of Alphacypermethrin residues in surface water, samples from the river Rhine were fortified with Alphacypermethrin at levels of 0.05 and 0.50 µg/l. Five replicates were used per fortification level, as well as two control samples and were analysed by GC/ECD. In addition, one control sample as well as one sample per fortification level (0.05, 0.5 µg/l) were analysed by GC/MS for confirmatory reasons.
4.2	**Conclusion**	The results for linearity, precision (repeatability) and accuracy demonstrate that the analytical method SAMS 469-2 is suitable for the determination of Alphacypermethrin in surface water.
4.2.1	Reliability	1
4.2.2	Deficiencies	None

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)	
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	COMMENTS FROM ...	

Section A4.2 **Annex Point IIA4.2**	**Analytical Methods for Detection and Identification in (c) water**

Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/04:** Kwasniok A, Pelz S, Christensen GW (1997) Validation of method SAMS 469-2 for the determination of AC 900049 (Alphacypermethrin) applied as a formulation (FASTAC^{TM} 100 g/L OESC insecticide) in pond water. Dr. Specht & Partner, Hamburg, Germany, Report No. ECO 97-138, August 21, 1997 (unpublished). (BASF-Ref.: AL-243-003)
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	No Guideline compliance is not stated in the report, but the method is consistent in all important aspects to SANCO/825/00 rev. 6. Two instead of five values were determined at each fortification level. However, this deviation does not affect the integrity and quality of the study.
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	Not applicable

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	Samples of distilled water and surface water (pond) were extracted by solvent partition with hexane.
3.1.2	Cleanup	The extracts were cleaned up using a Florisil disposable cartridge.
3.2	**Detection**	
3.2.1	Separation method	Gas-liquid chromatography Major modifications (capillary column) and minor modifications (sample extraction, clean up) to the original method SAMS 469-2 were made. Peak heights instead of peak areas were measured.
3.2.2	Detector	Electron capture detection (ECD)

Section A4.2 **Annex Point IIA4.2**	**Analytical Methods for Detection and Identification in (c) water**

3.2.3	Test substance	Formulation FASTAC 100 g/l OESC insecticide, batch no.: FAS 400001, active ingredient content: 101 g/l Alphacypermethrin
3.2.4	Reference substance	Alphacypermethrin (CL 900,049), batch no.: AC 10194-61, purity: 96.1%
3.2.5	Interfering substance(s)	None
3.3	**Linearity**	
3.3.1	Calibration range	0.503, 0.0503, 0.0252, 0.00503 µg/ml
3.3.2	Number of measurements	2 (except for 0.0252, 0.0503 µg/ml only single measurements)
3.3.3	Linearity	The method was determined to be linear over the calibration range. $y = 2626x + -20.092$, $r = 0.9997$
3.4	**Specificity: interfering substances**	No significant interferences from the sample matrix were detected in any of the control samples. Representative chromatograms are presented in the original report.
3.5	**Recovery rates and relative standard deviations**	

Sample matrix	Fortification	Recovery	n
Distilled water	0.01 µg a.i./l	88.6 %	2
	0.05 µg a.i./l	95.6 %	2
	0.1 µg a.i./l	84.6 %	2
	1.0 µg a.i./l	96.8 %	2
	10 µg a.i./l	91.1 %	2
Pond water	0.01 µg a.i./l	74.4 %	1
	0.02 µg a.i./l	105.5 %	1
	0.05 µg a.i./l	107.6 %	2
	0.1 µg a.i./l	90.5 %	2
	1.0 µg a.i./l	88.5 %	2
	10 µg a.i./l	84.8 %	2

The overall average recovery for fortified samples of distilled water was 91.3% ($n = 10$) with a coefficient of variation of 8.3%. The overall average recovery for fortified samples of pond water was 92.2% ($n = 10$) with a coefficient of variation of 13.8%.

3.6	**Limit of determination**	LOQ: 0.01 µg/l (Alphacypermethrin) LOD: 0.002 µg/l
3.7	**Precision**	
3.7.1	Repeatability	Not determined
3.7.2	Independent laboratory validation	Not applicable

Section A4.2 Annex Point IIA4.2	**Analytical Methods for Detection and Identification in (c) water**

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	For the validation of the analytical method SAMS 469-2 for the determination of Alphacypermethrin residues in surface water, samples of pond water were fortified with Alphacypermethrin formulated as FASTAC at levels of 0.01 and 10 µg a.i./l. After extraction by solvent partitioning with hexane and clean up using a Florisil disposable cartridge, analysis by GC/ECD was performed.
4.2	**Conclusion**	The results for linearity and accuracy confirm that the analytical method SAMS 469-2 is suitable for the determination of Alphacypermethrin in surface water in the concentration range of 0.01–10 ppb.
4.2.1	Reliability	2
4.2.2	Deficiencies	The precision (repeatability) of the method was not determined. However, the results obtained for accuracy confirmed the suitability of the method.

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	COMMENTS FROM ...

Section A4.2 Annex Point IIA4.2 — Analytical Methods for Detection and Identification in (d) animal and human body fluids and tissues

Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/05:** Walker B, Linkerhägner M (2000) Alphycypermethrin (AC 900049): Validation of the DFG method S 19 (extended revision) for the determination of residues of Alphacypermethrin in blood and urine of animal origin (Swine). Dr. Specht & Partner, Hamburg, Germany, Report no. CYA-0001V, September 14, 2000 (unpublished). (BASF-Ref.: AL-245-008)
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes SANCO/825/00 rev. 6, BBA guideline: Residue analytical methods for post-registration control purposes (July 21, 1998), US EPA OPPTS 860.1340
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	None

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	Samples of blood and urine (swine) were extracted with acetone with a acetone:water ratio constant of 2:1 (v/v). Liquid-liquid partitioning was performed with ethyl acetate / cyclohexane (1+1) and sodium chloride (extraction module E 1 of DFG method S 19).
3.1.2	Cleanup	The extracts were cleaned up by gel permeation chromatography using a mixture of ethyl acetate / cyclohexane (1+1) as eluent (DFG method S 19 module GPC). The residue-containing fraction was concentrated and subjected to supplemental silica gel mini column chromatography (DFG method S 19 module C 1).
3.2	**Detection**	
3.2.1	Separation method	Capillary gas chromatography

Section A4.2 Annex Point IIA4.2		**Analytical Methods for Detection and Identification in (d) animal and human body fluids and tissues**
3.2.2	Detector	Electron capture detection (ECD)
3.2.3	Standard(s)	Alphacypermethrin (CL 900,049), batch no.: AC 10194-61, purity: 96.1%
3.2.4	Interfering substance(s)	None
3.3	**Linearity**	
3.3.1	Calibration range	0.00250, 0.00499, 0.00998, 0.0250, 0.0499, 0.0998 µg/ml
3.3.2	Number of measurements	1
3.3.3	Linearity	The method was found to be linear over the calibration range. $y = 98647\,x - 29.0$, $r = 0.9996$
3.4	**Specificity: interfering substances**	No significant interferences from the specimen matrices were detected in any of the control specimens. Representative chromatograms are given in the original report.
3.5	**Recovery rates and relative standard deviations**	The recovery rates were in a range of 70–110%. The overall mean recovery over the whole validation range (0.005 and 0.05 mg/l) was 103% (4.0% RSD) for whole blood and 95% (4.7% RSD) for urine. Details are presented in Table A4.2- 1.
3.6	**Limit of determination**	LOQ: 0.005 mg/l in both matrices tested. LOD: 0.001 mg/l in both matrices tested.
3.7	**Precision**	
3.7.1	Repeatability	The method was successfully validated with five values at both fortification levels, with recoveries in the range from 70% to 110% and relative standard deviations below 20%. The results are presented in Table A4.2- 1.
3.7.2	Independent laboratory validation	Not applicable

Section A4.2 Annex Point IIA4.2	**Analytical Methods for Detection and Identification in (d) animal and human body fluids and tissues**

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	The applicability of the extended version of the multi-residue method S 19 (DFG method) for the determination of Alphacypermethrin in blood and urine of animal origin (swine) was validated. The extraction of Alphacypermethrin from blood and urine was performed according to extraction module E 1 followed by cleanup by gel permeation chromatography (module GPC) and on a silica gel column module C 1. Analysis was performed by GC/ECD.
4.2	**Conclusion**	Based on the results for linearity, accuracy and precision (repeatability), the extended version of DFG method S 19 is suitable for the determination of Alphacypermethrin in blood and urine of animal origin with a validated LoQ of 0.005 mg/l.
4.2.1	Reliability	1
4.2.2	Deficiencies	None

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	April, 2009
Materials and Methods	Applicant's version adopted
Results and discussion	Applicant's version adopted
Conclusion	Applicant's version adopted
Reliability	1
Acceptability	acceptable
Remarks	none
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Results and discussion	
Conclusion	
Reliability	
Acceptability	
Remarks	

Table A4.2- 1: Recoveries obtained for Alphacypermethrin using GC/ECD.

Matrix	Fortification level [mg/l]	Recoveries		RSD [%]
		Single values [%]	Mean ± SD [%]	
Blood	0.005	98, 109, 107, 106, 103	105 ± 4.3	4.1
	0.05	100, 98, 101, 107, 100	101 ± 3.4	3.4
Urine	0.005	95, 90, 92, 105, 99	96 ± 6.0	6.3
	0.05	99, 92, 93, 95, 94	95 ± 2.7	2.8

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1 REFERENCE

1.1	**Reference**	**A4.2/06:** Doran AM, Mayer IH, Khunachack A (1999) RENEGADE® Alphacypermethrin (CL 900049): Validation of analytical methods SAMS 461-1 and SAMS 456-1 for the determination of Alphacypermethrin residues in cattle tissues (muscle, fat, kidney and liver) and milk. Inveresk Research, Tranent, Scotland, Report No. RES 99-014, July 28, 1999 (unpublished). (BASF-Ref.: AL-245-006)
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	No Guideline compliance is not stated in the report, but the method is consistent to SANCO/825/00 rev. 6 in all important aspects. Three instead of five replicates were employed at each fortification level. However, this deviation does not affect the integrity and quality of the study.
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	Three instead of five replicates were employed at each fortification level. However, this deviation does not affect the integrity and quality of the study.

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Cattle tissues	According to SAMS 461-1, with minor deviations: Samples of tissues were chopped or minced finely and extracted by boiling with a mixture of acetone and hexane (1 + 2 v/v). The solvent is evaporated and the residue is re-dissolved in hexane. For fat and muscle a portion of the extract is partitioned with acetonitrile by using a Chem Elut CE 1020 extraction cartridge. A normal hexane/acetonitrile partitioning is used for the liver and kidney extracts. Extracts are further cleaned up by liquid-solid chromatography on a Florisil cartridge.

Section A4.2 Annex Point IIA4.2 — Analytical Methods for Detection and Identification in (d) animal and human body fluids and tissues

3.1.2	Milk	According to SAMS 456-1, with minor deviations: Samples of milk were treated with potassium oxalate solution and ethanol and extracted with diethyl ether and hexane. The extract was evaporated to dryness and a hexane solution was passed through a Chem Elut CE 1020 extraction column. Further clean up was performed by use of a cyano Bond Elut cartridge.
3.2	**Detection**	
3.2.1	Separation method	Gas chromatography (GC) Minor deviations from the original methods SAMS 461-1 and SAMS 456-1 (e.g. column, injection, column temperature program) were made.
3.2.2	Detector	Electron capture detection (ECD) Mass selective detector (MSD) for one control sample and one representative fortified sample at the LOQ of each commodity.
3.2.3	Standard(s)	Alphacypermethrin (CL 900,049), batch no.: AC 10194-61, purity: 96.1%
3.2.4	Interfering substance(s)	None
3.3	**Linearity**	
3.3.1	Calibration range	0.002, 0.006, 0.01 and 0.02 µg/mL Corresponding to 0.01, 0.03, 0.05 and 0.1 ng per injection (5µL), as given in the report. In the report (Table 11) the amount was given as µg, which should, however, read as "ng" and is supposed to be a typing error.
3.3.2	Number of measurements	1
3.3.3	Linearity	The method was determined to be linear over the calibration range. $y = 12428237\,x + 23138.23$, $r = 0.999714$
3.4	**Specificity: interfering substances**	The specificity of the methods SAMS 461-1 and SAMS 456-1 was demonstrated by analysing one control and one representative fortified sample at the LOQ of each commodity by GC/MS. The methods were found to be specific for the analysis of Alphacypermethrin in cattle tissues and milk, respectively.
3.5	**Recovery rates and relative standard deviations**	Single recovery rates were in a range of 70–110% for all matrices with one exception (one of three milk samples fortified at 40 ppb). The overall average recoveries, calculated from triplicate measurements at three fortification levels of Alphacypermethrin in muscle, fat, liver, kidney and milk were 102%, 95.5%, 91.0%, 95.3% and 99.0% with coefficients of variation of 6.8%, 10%, 8.9%, 9.7% and 12%, respectively. The results are summarised in Table A4.2- 2. Representative chromatograms are given in the original report.

3.6	**Limit of determination**	LOQ: 50.0 ppb for all cattle tissues (muscle, fat, liver, kidney) 10.0 ppb for whole milk LOD: 3.90, 3.13, 1.40, 5.68 ppb for muscle, fat, liver and kidney, respectively 0.593 ppb for whole milk
3.7	**Precision**	
3.7.1	Repeatability	The method was successfully validated employing triplicate measurement at three fortification levels (LOQ, 2x LOQ, 4x LOQ), with recoveries in the range from 70% to 110% (with one exception) and relative standard deviations below 20%. The results are summarised in Table A4.2- 2.
3.7.2	Independent laboratory validation	Not applicable

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	The methods SAMS 461-1 for cattle tissue (muscle, fat, kidney, liver) and the method SAMS 456-1 for whole milk samples were validated. After extraction and clean up of samples of cattle tissues or milk analysis by GC/ECD was performed.
4.2	**Conclusion**	Based on the results for linearity, accuracy and precision (repeatability), the methods SAMS 461-1 and SAMS 456-1 are suitable for the determination of Alphacypermethrin in cattle tissue with a LOQ of 50 ppb and milk with a LOQ of 10 ppb, respectively.
4.2.1	Reliability	1
4.2.2	Deficiencies	None

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	

	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	April, 2009
Materials and Methods	Applicant's version adopted
Results and discussion	Applicant's version adopted
Conclusion	Applicant's version adopted
Reliability	2 (no guideline, even if the method is consistent to SANCO/825/00 rev. 6 in all important aspects. Moreover, three instead of five replicates were employed at each fortification level.)
Acceptability	acceptable
Remarks	none

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Conclusion	
Reliability	
Acceptability	
Remarks	

Table A4.2- 2: Accuracy and precision of the methods for the different matrices.

Matrix	Fortification level [ppb]	Recovery [%]	Mean [%]	CV [%]
Cattle muscle				
	50.0	94.1, 92.1, 105	97.1	7.2
	100	109, 106, 108	108	1.4
	200	92.7, 100, 107	99.9	7.2
Cattle fat				
	50.0	89.4, 85.3, 78.4	84.4	6.6
	100	106, 104, 108	106	1.9
	200	95.3, 96.0, 97.2	96.2	1.0
Cattle liver				
	50.0	78.3, 82.7, 83.7	81.6	3.5
	100	101, 99.8, 98.9	100	1.1
	200	93.0, 89.8, 91.7	91.5	1.8
Cattle kidney				
	50.0	99.0, 98.3, 106	101	4.2
	100	81.8, 83.9, 84.9	83.5	1.9
	200	101, 97.7, 105	101	3.6
Milk				
	10.0	85.8, 84.6, 83.6	84.7	1.3
	20.0	103, 101, 104	103	1.5
	40.0	108, 106, 115	110	4.3

Section A4.2
Annex Point IIA4.2

Analytical Methods for Detection and Identification in (a) soil, (c) water, (d) animal and human body fluids and tissues

Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/07:** Xu B (2001) BAS 301 I (Alpha-cypermethrin): Validation of method M 3499 for the confirmation of BAS 310 I residues in water, soil and blood by GC/MS. BASF Agro Research, Princeton, NJ, USA, Report No. RES 01-058, October 25, 2001 (unpublished). (BASF-Ref.: AL-210-012)
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes SANCO/825/00 rev. 6
2.2	**GLP**	Yes (self-certified laboratory)
2.3	**Deviations**	Only one fortification level at the LOQ with three instead of five replicates was used. One control sample instead of two control samples was included. However, these deviations do not affect the integrity and quality of the study.

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Soil	According to SAMS 354-2: Soil samples were mixed with anhydrous sodium sulphate. Thereafter the mixture was extracted with acetone and hexane (1+1 v/v). The extracts were washed with water to remove the acetone and cleaned up by liquid-solid chromatography using Florisil.
3.1.2	Water	According to SAMS 469-2: Ground water samples were extracted by solvent partitioning with hexane. The extract was cleaned up using a Florisil disposable cartridge.
3.1.3	Blood	According to M 3499: Samples of bovine blood were diluted with water and extracted with acetone, ethyl acetate/cyclohexane (1+1) and sodium chloride. This procedure is identical to the extraction process of the DFG S-19 multi-residue method for Alphacypermethrin in blood.

3.2	**Detection**	
3.2.1	Separation method	Gas chromatography (GC).
3.2.2	Detector	Mass selective detector (MSD) The negative ion chemical ionisation mode is used with methane as the reagent gas to allow the ions at m/z 207, 209 and 211 to be monitored. The presence of Alphacypermethrin is confirmed by direct comparison of the sample peak's retention time to the retention time of the bracketing standards.
3.2.3	Standard(s)	Alphacypermethrin (BAS 310 I), batch no. AC 12395-18, purity: 95.1%
3.2.4	Interfering substance(s)	None
3.3	**Linearity**	
3.3.1	Calibration range	0.0025, 0.005, 0.01, 0.02, 0.04 µg/mL
3.3.2	Number of measurements	3
3.3.3	Linearity	The method was determined to be linear over the calibration range. $r > 0.999$
3.4	**Retention times**	The ion m/z 207 was used for the retention time analysis because it is the most prevalent ion. The average retention times for water, soil and blood are 17.19 min, 17.17 min and 17.19 min, respectively. For all of the three matrices, the retention times of the Alphacypermethrin peaks in the fortified samples were less than 0.1% different from the retention times of their bracketing standards. The results are summarised in Table A4.2- 3.
3.5	**Ion ratios**	For all the matrices, the 207/209 ratios were less than 2% different when compared to the average ratios of the standards. The 207/211 ion ratios were less than 6% different. The results are summarised in Table A4.2- 4.
3.6	**Limit of quantification (LOQ)**	Soil: 50 ppb Water: 0.05 ppb Blood: 5 ppb

Section A4.2
Annex Point IIA4.2

Analytical Methods for Detection and Identification in (a) soil, (c) water, (d) animal and human body fluids and tissues

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	For the validation of the analytical method M 3499 for the determination of Alphacypermethrin residues in soils, water or blood, aliquots of respective samples were fortified with Alphacypermethrin at the limit of quantification. Three replicates were used per fortification level, as well as one control sample and were analysed by GC/MS.
4.2	**Conclusion**	The results for retention times and mass of the analyte demonstrate that the analytical method M 3499 is suitable for the determination of Alphacypermethrin in water, soil and blood at the respective LOQ levels of 0.05 ppb, 50 ppb and 5 ppb.
4.2.1	Reliability	1
4.2.2	Deficiencies	None

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	April, 2009 ! ONLY HUMAN TOXICOLOGY
Materials and Methods	Applicant's version adopted
Results and discussion	Applicant's version adopted
Conclusion	Applicant's version adopted
Reliability	1
Acceptability	Acceptable
Remarks	none
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Acceptability	
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Table A4.2- 3: Retention times for water, soil and blood samples.

Matrix	Sample	Retention time (m/z 207)	Average standard retention time [min]	% difference
Water				
	Standard (0.005 µg/mL)	17.19		
	Control	ND	17.18	–
	Fortified sample 1	17.20	17.18	0.1
	Standard (0.005 µg/mL)	17.16		
	Fortified sample 2	17.17	17.18	0.0
	Fortified sample 3	17.20	17.18	0.1
	Standard (0.005 µg/mL)	17.19		
Soil				
	Standard (0.005 µg/mL)	17.19		
	Control	ND	17.18	–
	Fortified sample 1	17.18	17.18	0.0
	Standard (0.005 µg/mL)	17.16		
	Fortified sample 2	17.17	17.16	0.1
	Fortified sample 3	17.16	17.16	0.0
	Standard (0.005 µg/mL)	17.16		
Blood				
	Standard (0.005 µg/mL)	17.19		
	Control	ND	17.20	–
	Fortified sample 1	17.17	17.20	–0.1
	Standard (0.005 µg/mL)	17.20		
	Fortified sample 2	17.20	17.20	0.0
	Fortified sample 3	17.19	17.20	0.0
	Standard (0.005 µg/mL)	17.19		

Average standard retention time = average of two standards bracketing the sample.

% difference = (sample retention time – average standard retention time) x 100/average standard retention time

Table A4.2- 4: Ion ratios determined in water, soil and blood samples.

Matrix	**Sample**	**Response (peak area)**			**Ion ratio**		**Standard average**		**% difference**	
		m/z 207	**m/z 209**	**m/z 211**	**207/209**	**207/211**	**207/209**	**207/211**	**207/209**	**207/211**
Water										
	Standard (0.005 µg/mL)	46271	29839	4509	1.551	10.262				
	Control	ND	ND	ND	–	–	1.547	10.123	–	–
	Fortified sample 1	96060	62166	9961	1.545	9.644	1.547	10.123	–0.1	–4.7
	Standard (0.005 µg/mL)	39045	25295	3911	1.544	9.983				
	Fortified sample 2	93965	60948	9617	1.542	9.771	1.554	10.071	–0.8	–3.0
	Fortified sample 3	85788	55340	8963	1.550	9.571	1.554	10.071	–0.2	–5.0
	Standard (0.005 µg/mL)	35345	22606	3479	1.564	10.160				
Soil										
	Standard (0.005 µg/mL)	33379	21350	3333	1.563	10.015				
	Control	ND	ND	ND	–	–	1.563	10.137	–	–
	Fortified sample 1	67540	43469	6981	1.554	9.675	1.563	10.137	–0.6	–4.6
	Standard (0.005 µg/mL)	34371	22003	3350	1.562	10.260				
	Fortified sample 2	67456	43631	6836	1.546	9.868	1.562	10.010	–1.0	–1.4
	Fortified sample 3	63712	40800	6564	1.562	9.706	1.562	10.010	0.0	–3.0
	Standard (0.005 µg/mL)	31965	20457	3275	1.563	9.760				
Blood										
	Standard (0.005 µg/mL)	31402	20022	3010	1.568	10.433				
	Control	ND	ND	ND	–	–	1.555	10.047	–	–
	Fortified sample 1	20930	13421	2204	1.559	9.496	1.555	10.047	0.3	–5.5
	Standard (0.005 µg/mL)	32202	20886	3333	1.542	9.662				
	Fortified sample 2	20584	13259	1947	1.552	10.572	1.554	10.034	–0.1	5.4
	Fortified sample 3	16476	10476	1670	1.573	9.866	1.554	10.034	1.2	–1.7
	Standard (0.005 µg/mL)	30834	19692	2963	1.566	10.406				

Section A4.2 Annex Point IIA4.2	Analytical Methods for Detection and Identification in (a) soil

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1 REFERENCE

1.1 Reference	**A4.2/08:** Carter ML, Schmitt JL (2013) Validation of BASF Analytical Method R0034/01: "Method for the Quantitation of the Diastereomeric Forms of BAS 311 I (Reg. 127266) and Its Metabolites 3-Phenoxybenzoic Acid (Reg. No. 130213) and Dichlorovinyl Alcohol (Cis and Trans Isomers, Reg. No. 180011) in Soil by LC-MS/MS" ADPEN Laboratories, Inc., Jacksonville, FL 32224, USA, Report No. 2K13-903-405215 (BASF Study No 405215).
1.2 Data protection	Yes
1.2.1 Data owner	BASF
1.2.2 Companies with letter of access	No
1.2.3 Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1 Guideline study	Yes OPPTS 850.7100, OPPTS 835.6100, PR Notice 2011-3 SANCO/3029/99 rev. 4, 2000 (pre-registration residue methods) SANCO/825/00 rev. 8, 2000 (post-registration residue methods)
2.2 GLP	Yes (certified laboratory)
2.3 Deviations	None

3 MATERIALS AND METHODS

3.1 Preliminary treatment	
3.1.1 Test system	Clay Loam, Louisiana (R1200660030) Sandy Loam, California (R1200690025) Soil, New York (R1200680025) (Extractability) Soil, New York (R1200680400) (Extractability)
3.1.2 Analytes	Cypermethrin (BAS 311 I) diastereomers (Cis I, Cis II, Trans III, Trans IV) Metabolite 3-phenoxybenzoic acid (3-PBA) Metabolite dichlorovinyl alcohol (DCVA; Cis and Trans)

3.1.3 Enrichment	The method was validated at two fortification levels (0.001 and 0.01 mg/kg) for sandy and clay loam soil samples. For each fortification level and matrix, five replicates were analysed. Additionally, a method blank and at least two replicates of unfortified samples were examined.
3.1.4 Extraction	Soil samples (5 g) are extracted for cypermethrin isomers by shaking with 50 mL of 0.1% formic acid in acetonitrile. An aliquot (20%) from the extract is evaporated to dryness and then re-dissolved with acetonitrile/water with 0.1% formic acid (50/50, v/v). Soil samples (5 g) are extracted for the metabolites of cypermethrin (cis-DCVA, trans-DCVA, and 3-PBA) by shaking twice with 25 mL acetonitrile/water (70/30, v/v). An aliquot (10%) from the combined extract is evaporated to dryness and then dissolved in methanol/water (20/80, v/v) with 0.1% formic acid.
3.2 Detection	
3.2.1 Separation method	Liquid chromatography (LC). The methods, analytes and analytical modes are summarised in Table A4.2- 1.
3.2.2 Detector	Tandem Mass Spectrometry (MS/MS) The monitoring ions used for quantitation and confirmation in the different methods are outlined in Table A4.2- 1.

3.2.3 Standard(s)

Common name	**CAS-No.**	**Batch-No.**	**Purity**
Alpha-cypermethrin (Cis II)	67375-30-8	AC9575-006	99.8%
Cis I/Cis II Isomers (45/55)	211504-93-7	AC8949-76	99.8%
Trans III/Trans IV (43.5/56.5)	211504-94-8	AC8949-77	99.8%
DCVA (Mixture of cis- and trans-isomers, 51.5/48.5)	55701-05-8	AC9966-87	99.0%
3-PBA	3739-38-6	AC12251-34	100.0%

3.2.4 Interfering substance(s)	None
3.3 Linearity	
3.3.1 Calibration range	Cypermethrin (cis I, cis II, trans III, trans IV): 0.05 – 10 ng/mL 3-PBA, DCVA (cis, trans): 0.1 – 10 ng/mL
3.3.2 Number of measurements	2
3.3.3 Linearity	Cypermethrin (cis I, cis II, trans III, trans IV): r > 0.99 for both mass transitions 3-PBA, DCVA (cis, trans): 0.1 – 10 ng/mL: r > 0.99 for both mass transitions

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3.4	**Specificity**	The method R0034/01 determines residues of cypermethrin (cis-I, cis-II, trans-III, trans-IV) and its metabolites, 3-PBA and dichlorovinyl alcohol (cis- and trans-isomers) in soil. Interferences may be observed during analysis of the metabolites of cypermethrin depending on soil type. This can be alleviated by testing the LC-MS/MS gradient before GLP analysis. Retention time shifting was observed for the diastereomeric forms of cypermethrin and is instrument dependent. However, this does not affect quantitation as long as the retention pattern is observed and compared with the reference standards. The instrument must be sufficiently conditioned with injections of sample matrix prior to analysis.
3.5	**Matrix effects**	Clay and sandy loam soil samples were tested for matrix effects for the diastereomers of BAS 311 I. Clay soil samples were tested for matrix effects for the metabolites of BAS 311 I. No significant suppression (greater than 20%) occurred for any of the analytes when analyzed by LC-MS/MS. If significant suppression occurs, matrix-matched standards may be utilized.
3.6	**Recovery rates and relative standard deviations**	The validation results (recoveries, relative standard deviations) for cypermethrin diastereomers (cis I, cis II, trans III, trans IV), 3-phenoxybenzoic acid (3-PBA) and dichlorovinyl alcohol (DCVA; cis and trans) are summarised in Tables A4.2-3 to A4.2-12.
3.7	**Limit of quantification and determination**	LOQ: 0.001 mg/kg LOD: 0.0002 mg/kg (all analytes)
3.8	**Precision**	
3.8.1	Repeatability	The method was successfully validated with five values at both fortification levels, with recoveries in the range from 70% to 110% and relative standard deviations below 20% for all analytes and ion transitions. Detailed results are presented in Tables A4.2-3 to A4.2-12.
3.8.2	Independent laboratory validation	The method was successfully validated by an independent laboratory (Yang J, Michener P, 2013; PASC Study Number: 053-0894; PASC Report Number: PASC-REP-0416; BASF Study Number: 405216).

3.9 Storage Stability of Fortification and Standard Solutions

During the method validation, it was found that the calibration standard solutions of cypermethrin were stable (less than 20% decline) for at least 64 days refrigerated and the fortification and concentrated stock solutions were stable for at least 65 days. The metabolites of cypermethrin were stable (less than 20%) for at least 38 days for calibration and fortification solutions and 69 days for the concentrated stock solution.

3.10 Storage Stability of Extracts in Final Volume

The mean stored recovery values found during the storage stability experiment for clay loam soil extracts for the diastereomers of cypermethrin was 79–108%. This demonstrates that the diastereomers of cypermethrin are stable in the extracts of the clay loam for 14 days.

The mean stored recovery values found during the storage stability experiment for clay loam soil extracts for the metabolites of BAS 311 I was 79–95%.

3.11 Extractability

The extractability of the diastereomers of cypermethrin in soil was tested using the residue and metabolism extraction procedures. An extractability set consisted of one control, two fortifications at the LOQ (0.001 mg/kg for each diastereomer), one fortification at 10× LOQ (0.01 mg/kg) and one sample with incurred residues from the terrestrial field dissipation study number 380198. The Results show that the diastereomers of BAS 311 I in soil are comparable from both residue and metabolism extraction procedures.

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1 Materials and methods

The residue analytical method R0034/01 determines residues of the diastereomeric forms of BAS 311 I (cypermethrin, cis-I, cis-II, trans-III, trans-IV) and its metabolites 3-phenoxybenzoic acid and dichlorovinyl alcohol (cis- and trans-isomers) in soil. For the validation of the method, aliquots of a sandy loam (California; R1200690025) and clay loam (Louisiana; R1200660030) soil were fortified with each analyte at levels of 0.001 and 0.01 mg/kg. Five replicates were used per fortification level. Additionally, a method blank and at least two replicates of unfortified samples were examined.

Primary and confirmatory mass transitions for the diastereomeric forms of cypermethrin (cis-I, cis-II, trans-III, trans-IV) was analyzed using UPLC and HPLC Methods B and C, respectively. Primary and confirmatory mass transitions for 3-PBA were analysed using UPLC and HPLC Methods G and E, respectively. The ions monitored for DCVA (cis and trans) were analyzed using UPLC and HPLC Methods G and E, respectively. For DCVA (cis and trans), Method E was used for primary quantitation in HPLC mode and confirmatory quantitation for Method G. Additionally, Method F was used for confirmatory quantitation both for HPLC mode and for Method E.

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4.2 Conclusion	It was demonstrated that method R0034/01 fulfils the requirements with regard to specificity, repeatability, limit of quantification, and recoveries and is, therefore, applicable to correctly determine residues of the insecticide cypermethrin (BAS 311 I) and its metabolites 3-phenoxybenzoic acid (3-PBA) and dichlorovinyl alcohol (cis- and trans-isomers) in soil. The method was successfully validated by an independent laboratory and is therefore valid for monitoring and enforcement purposes.
4.2.1 Reliability	1
4.2.2 Deficiencies	None

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	COMMENTS FROM ...

Table A4.2- 1: Validated methods, analytes and analytical modes

Method	Analyte	Analytical Mode (Column)
B	Alpha-cypermethrin: Primary and Confirmatory Quantitation	UPLC (Acquity UPLC HSS T3 1.8 µm 2.1x150 mm)
C	Alpha-cypermethrin: Primary and Confirmatory Quantitation	HPLC (XSelect HSS T3 2.5 µm 2.1x150 mm)
E	Primary and Confirmatory Quantitation of 3-PBA; Primary Quantitation in HPLC mode of DCVA (cis and trans) and Confirmatory Quantitation for Method G	HPLC (BEH Phenyl 2.5 µm 2.1x100 mm)
F	DCVA (cis and trans) Confirmatory Quantitation both for HPLC Mode and for Method E	HPLC (XSelect HSS T3 2.5 µm 2.1x150 mm)
G	Primary and Confirmatory Quantitation of 3-PBA and Primary Quantitation of DCVA (cis and trans)	UPLC (Acquity UPLC HSS T3 1.8 µm 2.1x150 mm)

Table A4.2- 2: Monitoring ions used for quantitation and confirmation in the various methods

Analyte	Method	Quantitation	Confirmation
Cypermethrin – cis I	B/C	m/z 191.0	m/z 193.0
Cypermethrin – cis II	B/C	m/z 191.0	m/z 193.0
Cypermethrin – trans III	B/C	m/z 191.0	m/z 193.0
Cypermethrin – trans IV	B/C	m/z 191.0	m/z 193.0
DCVA – cis	E/F/G	m/z 207.0	m/z 207.0
DCVA – trans	E/F/G	m/z 207.0	m/z 207.0
3-PBA	E/G	m/z 93.0	m/z 169.0

Table A4.2- 3: Recoveries for Cis I, Cis II, Trans III, and Trans IV in Sandy Loam Soil Using Method B (UPLC Mode):

Primary and Confirmatory Quantitation

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Sandy loam	Cis I: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	71	2.4	3.4	5
	0.01	72	2.9	4.0	5
	Cis I: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	74	4.1	5.5	5
	0.01	75	3.5	4.6	5
	Cis II: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	73	4.7	6.5	5
	0.01	74	3.9	5.3	5
	Cis II: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	72	3.7	5.1	5
	0.01	73	3.1	4.3	5
	Trans III: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	83	7.7	9.3	5
	0.01	78	3.7	4.7	5
	Trans III: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	82	10.3	12.6	5
	0.01	81	7.4	9.1	5
	Trans IV: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	77	6.0	7.9	5
	0.01	74	7.7	10.4	5
	Trans IV: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	76	6.1	7.9	5
	0.01	74	8.4	11.4	5

Table A4.2- 4: Recoveries for Cis I, Cis II, Trans III, and Trans IV in Clay Loam Soil Using Method B (UPLC Mode):
Primary and Confirmatory Quantitation

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Clay loam	Cis I: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	81	0.6	0.7	5
	0.01	88	2.0	2.3	5
	Cis I: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	86	0.9	1.0	5
	0.01	89	2.0	2.2	5
	Cis II: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	86	0.9	1.1	5
	0.01	91	2.2	2.4	5
	Cis II: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	88	1.6	1.8	5
	0.01	90	2.6	2.9	5
	Trans III: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	97	4.0	4.1	5
	0.01	90	3.1	3.4	5
	Trans III: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	95	3.4	3.6	5
	0.01	93	6.1	6.5	5
	Trans IV: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	91	3.2	3.5	5
	0.01	88	5.7	6.4	5
	Trans IV: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	91	3.8	4.2	5
	0.01	88	5.7	6.5	5

Table A4.2- 5: Recoveries for Cis I, Cis II, Trans III, and Trans IV in Sandy Loam Soil Using Method C (HPLC Mode):
Primary and Confirmatory Quantitation

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Sandy loam	Cis I: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	93	8.0	8.6	5
	0.01	91	3.0	3.4	5
	Cis I: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	94	7.5	7.9	5
	0.01	93	5.4	5.8	5
	Cis II: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	100	4.2	4.2	5
	0.01	98	3.0	3.1	5
	Cis II: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	98	9.0	9.1	5
	0.01	97	4.6	4.7	5
	Trans III: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	100	10.8	10.9	5
	0.01	103	9.0	8.7	5
	Trans III: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	99	9.5	9.7	5
	0.01	103	9.9	9.6	5
	Trans IV: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	101	8.1	8.1	5
	0.01	105	9.6	9.2	5
	Trans IV: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	108	3.1	2.9	5
	0.01	103	9.0	8.7	5

Table A4.2- 6: Recoveries for Cis I, Cis II, Trans III, and Trans IV in Clay Loam Soil Using Method C (HPLC Mode):
Primary and Confirmatory Quantitation

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Clay loam	Cis I: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	83	6.0	7.2	5
	0.01	92	13.6	14.9	5
	Cis I: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	104	9.9	9.5	5
	0.01	110	17.2	15.6	5
	Cis II: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	94	5.8	6.2	5
	0.01	95	12.8	13.4	5
	Cis II: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	93	8.3	8.9	5
	0.01	95	11.3	11.9	5
	Trans III: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	91	1.7	1.9	5
	0.01	97	9.2	9.5	5
	Trans III: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	84	9.5	11.3	5
	0.01	96	7.9	8.2	5
	Trans IV: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	94	7.3	7.8	5
	0.01	97	8.3	8.5	5
	Trans IV: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	97	5.7	5.9	5
	0.01	98	9.3	9.5	5

Table A4.2- 7: Recoveries for DCVA (Cis and Trans) and 3-PBA in Sandy Loam Soil Using Method G (UPLC Mode):
Primary and Confirmatory Quantitation of 3-PBA and Primary Quantitation of DCVA (Cis and Trans)

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Sandy loam	DCVA-cis: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	94	5.8	6.2	5
	0.01	96	3.7	3.9	5
	DCVA-trans: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	105	4.5	4.3	5
	0.01	99	5.3	5.4	5
	3-PBA: Primary Quantitation (m/z 213.0 → m/z 93.0)				
	0.001	99	5.1	5.2	5
	0.01	96	5.7	6.0	5
	3-PBA: Confirmatory Quantitation (m/z 213.0 → m/z 169.0)				
	0.001	96	4.2	4.4	5
	0.01	98	5.3	5.4	5

Table A4.2- 8: Recoveries for DCVA (Cis and Trans) and 3-PBA in Clay Loam Soil Using Method G (UPLC Mode):
Primary and Confirmatory Quantitation of 3-PBA and Primary Quantitation of DCVA (Cis and Trans)

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Clay loam	DCVA-cis: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	96	6.1	6.4	5
	0.01	100	4.9	4.9	5
	DCVA-trans: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	102	9.5	9.3	5
	0.01	104	4.8	4.6	5
	3-PBA: Primary Quantitation (m/z 213.0 → m/z 93.0)				
	0.001	105	3.4	3.3	5
	0.01	105	4.6	4.3	5
	3-PBA: Confirmatory Quantitation (m/z 213.0 → m/z 169.0)				
	0.001	102	8.1	8.0	5
	0.01	107	2.7	2.5	5

Table A4.2- 9: Recoveries for DCVA (Cis and Trans) and 3-PBA in Sandy Loam Soil Using Method E: Primary and Confirmatory Quantitation of 3-PBA in HPLC mode; Primary Quantitation in HPLC mode of DCVA (Cis and Trans) and Confirmatory Quantitation for Method G

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Sandy loam	DCVA-cis: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	92	11.7	12.8	5
	0.01	82	7.2	8.7	5
	DCVA-trans: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	103	6.5	6.4	5
	0.01	90	7.9	8.8	5
	3-PBA: Primary Quantitation (m/z 213.0 → m/z 93.0)				
	0.001	97	9.4	9.7	5
	0.01	98	8.2	8.4	5
	3-PBA: Confirmatory Quantitation (m/z 213.0 → m/z 169.0)				
	0.001	98	6.7	6.8	5
	0.01	98	9.8	10.0	5

Table A4.2- 10: Recoveries for DCVA (Cis and Trans) and 3-PBA in Clay Loam Soil Using Method E: Primary and Confirmatory Quantitation of 3-PBA in HPLC mode; Primary Quantitation in HPLC mode of DCVA (Cis and Trans) and Confirmatory Quantitation for Method G

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Clay loam	DCVA-cis: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	94	4.9	5.2	5
	0.01	96	5.2	5.4	5
	DCVA-trans: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	90	9.5	10.6	5
	0.01	98	6.4	6.5	5
	3-PBA: Primary Quantitation (m/z 213.0 → m/z 93.0)				
	0.001	100	13.4	13.4	5
	0.01	101	6.4	6.3	5
	3-PBA: Confirmatory Quantitation (m/z 213.0 → m/z 169.0)				
	0.001	95	10.7	11.2	5
	0.01	102	4.3	4.2	5

Table A4.2- 11: Recoveries for DCVA (Cis and Trans) in Sandy Loam Soil Using Method F: DCVA (Cis and Trans) Confirmatory Quantitation both for HPLC Mode and for Method E

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Sandy loam	DCVA-cis: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	101	16.2	16.0	5
	0.01	94	5.1	5.4	5
	DCVA-trans: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	97	4.7	4.9	5
	0.01	98	4.9	4.9	5

Table A4.2- 12: Recoveries for DCVA (Cis and Trans) in Clay Loam Soil Using Method F: DCVA (Cis and Trans) Confirmatory Quantitation both for HPLC Mode and for Method E

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Clay loam	DCVA-cis: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	87	7.2	8.3	5
	0.01	97	4.4	4.5	5
	DCVA-trans: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	100	12.3	12.3	5
	0.01	97	3.8	3.9	5

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1 REFERENCE

1.1	**Reference**	**A4.2/09:** Yang J, Michener P (2013) Independent Laboratory Validation of R0034/01: "Method for the Quantitation of the Diastereomeric Forms of BAS 311 I (Reg. 127266) and Its Metabolites 3-Phenoxybenzoic Acid (Reg. No. 130213) and Dichlorovinyl Alcohol (Cis and Trans Isomers, Reg. No. 180011) in Soil by LC-MS/MS" Primera Analytical Solutions Corp. (PASC), Princeton, NJ 08540, USA, Report No. PASC-REP-0416, Study No. 053-0894 (BASF Study No. 405216).
1.2	**Data protection**	Yes
1.2.1	Data owner	BASF
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes OPPTS 850.7100, OPPTS 835.6100, PR Notice 2011-3 SANCO/3029/99 rev. 4, 2000 (pre-registration residue methods) SANCO/825/00 rev. 8, 2000 (post-registration residue methods)
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	None

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Test system	Soil not characterised (soil matrix was provided by BASF)
3.1.2	Analytes	Cypermethrin (BAS 311 I) diastereomers (Cis I, Cis II, Trans III, Trans IV) Metabolite 3-phenoxybenzoic acid (3-PBA) Metabolite dichlorovinyl alcohol (DCVA; Cis and Trans)

Section A4.2 **Annex Point IIA4.2**		**Analytical Methods for Detection and Identification in (a) soil**
3.1.3	Enrichment	The method was validated by an independent laboratory at two fortification levels (0.001 and 0.01 mg/kg). The analytical sets each consisted of a reagent blank, two controls, five replicates fortified with analytes at the method limit of quantitation (LOQ, 0.001 mg/kg), and five replicates fortified at a higher level, corresponding to 10x LOQ (0.01 mg/kg).
3.1.4	Extraction	See analytical method report (Ref A4.2/08).
3.2	**Detection**	
3.2.1	Separation method	Liquid chromatography (LC; see analytical method report, Ref A4.2/08).
3.2.2	Detector	Tandem Mass Spectrometry (MS/MS; see analytical method report, Ref A4.2/08).
3.2.3	Standard(s)	See analytical method report (Ref A4.2/08).
3.2.4	Interfering substance(s)	None
3.3	**Linearity**	
3.3.1	Calibration range	Cypermethrin (cis I, cis II, trans III, trans IV), 3-PBA, DCVA (cis, trans): 0.1 – 10 ng/mL
3.3.2	Number of measurements	2
3.3.3	Linearity	Cypermethrin (cis I, cis II, trans III, trans IV), 3-PBA, DCVA (cis, trans): r > 0.99 for both mass transitions
3.4	**Specificity**	See analytical method report (Ref A4.2/08).
3.5	**Recovery rates and relative standard deviations**	The validation results (recoveries, relative standard deviations) for cypermethrin diastereomers (cis I, cis II, trans III, trans IV), 3-phenoxybenzoic acid (3-PBA) and dichlorovinyl alcohol (DCVA; cis and trans) are summarised in Tables A4.2-3 to A4.2-12.
3.6	**Limit of quantification and determination**	LOQ: 0.001 mg/kg LOD: 0.0002 mg/kg (all analytes)
3.7	**Precision**	
3.7.1	Repeatability	The method was successfully validated with five values at both fortification levels, with recoveries in the range from 70% to 110% and relative standard deviations below 20% for all analytes and ion transitions. Detailed results are presented in Tables A4.2-1 to A4.2-10.
3.7.2	Independent laboratory validation	Yes

Section A4.2 **Annex Point IIA4.2**	**Analytical Methods for Detection and Identification in (a) soil**

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1 Materials and methods	See analytical method report (Ref A4.2/08).
4.2 Conclusion	The independent laboratory validation was successfully completed at PASC. Recovery results and statistical data demonstrate Analytical Method R0034/01 can be performed successfully for quantitation of diastereomeric forms of BAS 311 I and its metabolites 3-PBA and Dichlorovinyl Alcohol down to a level of 0.001 mg/kg in soil. The method is well-written and contains a fair amount of comments to guide the analyst through the procedure for the first time.
4.2.1 Reliability	1
4.2.2 Deficiencies	None

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	COMMENTS FROM ...

Table A4.2- 1: Recoveries for Cis I, Cis II, Trans III, and Trans IV in Soil Using Method B (UPLC Mode): Primary and Confirmatory Quantitation

Matrix	**Fortification (mg/kg)**	**Recovery (%)**	**SD**	**RSD (%)**	**n**
Soil	Cis I: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	94.7	4.53	4.79	5
	0.01	93.3	3.75	4.02	5
	Cis I: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	94.2	5.04	5.35	5
	0.01	94.1	3.03	3.22	5
	Cis II: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	92.8	3.93	4.23	5
	0.01	88.6	2.68	3.02	5
	Cis II: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	76.4	4.53	5.92	5
	0.01	96.4	3.45	3.58	5
	Trans III: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	91.0	7.81	8.58	5
	0.01	96.6	8.65	8.96	5
	Trans III: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	90.2	5.19	5.76	5
	0.01	81.3	6.45	7.94	5
	Trans IV: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	90.7	1.51	1.66	5
	0.01	94.8	1.46	1.54	5
	Trans IV: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	96.9	3.54	3.65	5
	0.01	94.3	2.14	2.26	5

Table A4.2- 2: Recoveries for Cis I, Cis II, Trans III, and Trans IV in Soil Using Method C (HPLC Mode): Primary and Confirmatory Quantitation

Matrix	Fortification (mg/kg)	Recovery (%)	SD	RSD (%)	n
Soil	Cis I: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	90.5	7.11	7.85	5
	0.01	86.9	4.48	5.16	5
	Cis I: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	86.6	10.18	11.75	5
	0.01	87.5	5.64	6.44	5
	Cis II: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	87.8	5.00	5.70	5
	0.01	87.0	3.85	4.42	5
	Cis II: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	99.6	14.64	14.69	5
	0.01	87.0	5.00	5.74	5
	Trans III: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	86.0	7.82	9.09	5
	0.01	85.7	5.26	6.13	5
	Trans III: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	87.2	10.66	12.22	5
	0.01	87.5	4.56	5.21	5
	Trans IV: Primary Quantitation (m/z 433.0 → m/z 191.0)				
	0.001	89.2	8.38	9.39	5
	0.01	85.6	4.42	5.16	5
	Trans IV: Confirmatory Quantitation (m/z 435.0 → m/z 193.0)				
	0.001	99.8	12.02	12.04	5
	0.01	85.5	5.52	6.46	5

Table A4.2- 3: Recoveries for DCVA (Cis and Trans) and 3-PBA in Soil Using Method G modified (UPLC Mode):
Primary Quantitation of 3-PBA and DCVA (Cis and Trans)

Matrix	Fortification (mg/kg)	Recovery (%)	SD	RSD (%)	n
Soil	DCVA-cis: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	118.8	7.35	6.19	5
	0.01	98.7	6.35	6.43	5
	DCVA-trans: Primary Quantitation (m/z 207.0 → m/z 207.0)				
	0.001	98.6	18.33	18.58	5
	0.01	100.2	4.71	4.70	5
	3-PBA: Primary Quantitation (m/z 213.0 → m/z 93.0)				
	0.001	97.1	0.65	0.67	5
	0.01	99.7	2.88	2.89	5

Table A4.2- 4: Recoveries for DCVA (Cis and Trans) and 3-PBA in Soil Using Method E:
Primary Quantitation of 3-PBA in HPLC mode;
Primary Quantitation in HPLC mode of DCVA (Cis and Trans) and Confirmatory Quantitation for Method I

Matrix	Fortification (mg/kg)	Recovery (%)	SD	RSD (%)	n
Soil	DCVA-cis: Primary Quantitation (m/z 207.0)				
	0.001	95.6	7.82	8.18	5
	0.01	101.2	3.33	3.29	5
	DCVA-cis: Confirmatory Quantitation (m/z 209.0)				
	0.001	94.0	13.57	14.43	5
	0.01	101.7	3.09	3.04	5
	DCVA-trans: Primary Quantitation (m/z 207.0)				
	0.001	81.5	5.30	6.51	5
	0.01	96.0	2.48	2.59	5
	DCVA-trans: Confirmatory Quantitation (m/z 209.0)				
	0.001	90.4	7.33	8.10	5
	0.01	94.7	2.34	2.47	5
	3-PBA: Primary Quantitation (m/z 213.0 → m/z 93.0)				
	0.001	103.1	5.28	5.13	5
	0.01	103.8	4.36	4.21	5

Table A4.2- 5: Recoveries for DCVA (Cis and Trans) and 3-PBA Using Method F:
Confirmatory Quantitation for HPLC of DCVA (Cis and Trans) and 3-PBA

Matrix	Fortification (mg/kg)	Recovery (%)	SD	RSD (%)	n
Soil	DCVA-cis: Confirmatory Quantitation (m/z 207.0)				
	0.001	91.5	5.35	5.85	5
	0.01	92.5	11.87	12.83	5
	DCVA-trans: Confirmatory Quantitation (m/z 207.0)				
	0.001	93.6	4.65	4.97	5
	0.01	96.8	5.73	5.92	5
	3-PBA: Confirmatory Quantitation (m/z 213.0 → m/z 93.0)				
	0.001	90.7	7.37	8.12	5
	0.01	85.8	12.43	14.48	5

Section A4.2 **Annex Point IIA4.2**	**Analytical Methods for Detection and Identification in (b) air**

Official use only

1 REFERENCE

1.1 Reference	**A4.2/10:** Bending, P (2013) Development and validation of an analytical method for the determination of Alpha-cypermethrin (BAS 310 I) in air. PTRL Europe, 89081 Ulm, Germany, Report No. P 2989 G, August 21, 2013 (unpublished). (BASF Study No. 429012).
1.2 Data protection	Yes
1.2.1 Data owner	BASF
1.2.2 Companies with letter of access	No
1.2.3 Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1 Guideline study	Yes EC Guidance document on residue analytical methods SANCO/825/00, rev. 8.1 (16/11/2010) and SANCO/3029/99, rev. 4 (11/07/2000)
2.2 GLP	Yes (certified laboratory)
2.3 Deviations	None

3 MATERIALS AND METHODS

3.1 Preliminary treatment	
3.1.1 Air sampling	Air sampling was carried out by adsorption tubes filled with two layers of XAD porous polymer. Particles and aerosols are trapped by filtration or impact onto the adsorbent material. Air was sampled for 6 hours at about 1.0 L/min (total air sampling volume ≈ 0.36 m^3). Warm air (approx. 35°C) was passed over warm water before diverting the humidified air flow (relative humidity approx. 95%) over the sampling cartridges.
3.1.2 Fortification	Fortification was performed directly onto the XAD adsorption material. The fortification solution was dosed in one 10 µL portion directly onto the front layer XAD adsorbent material. The method was validated at fortification levels of 0.50 µg and 5.0 µg per adsorption tube, corresponding to about 1.4 µg/m^3 (LOQ) and approximately 14 µg/m^3 (10xLOQ).

Section A4.2 Annex Point IIA4.2 — Analytical Methods for Detection and Identification in (b) air

3.1.3 Desorption

The adsorption material is extracted with ethyl acetate, the internal standard lambda-cyhalothrin is added to the extract.

3.2 Detection

3.2.1 Separation method

The test substance is analysed by gas chromatography (GC).

3.2.2 Detector

Mass spectrometic detector (MS), negative chemical ionisation (NCI), three fragment ions were monitored (m/z 207 for quantitation, m/z 209 and m/z 171 for confirmation).

3.2.3 Standard(s)

Alphacypermethrin, batch no.: L80-24, purity: 99.4%.

3.2.4 Interfering substance(s)

None.

3.3 Extraction efficiency

Extraction efficiency was examined by fortifying the analyte (duplicates of each adsorbent portion fortified at 5.0 μg, n = 4) onto adsorbent portions of sampling cartridges.

The extraction efficiency was demonstrated with an average recovery of 99 % (for m/z 207).

Fortification	Recoveries	Mean recovery	RSD
5.0 μg	97-103%	99%	3%

3.4 Storage stability

Storage stability of adsorbed alpha-cypermethrin was examined by fortifying the analyte onto adsorbent layers of sampling cartridges (duplicates fortified at 5.0 μg). The cartridges were sealed and stored for a 6-day period at freezer temperature (approximately -20°C).

Fortification	Recoveries	Mean recovery	RSD
5.0 μg	93-96%	95%	n/a

3.5 Linearity

3.5.1 Calibration range

0.01, 0.02, 0.0.5, 0.1, 0.2, and 0.3 μg/mL

3.5.2 Number of measurements

1 per concentration level

3.5.3 Linearity

The method was found to be linear over the calibration range.

$y = 0.00798237+0.00248815\,x$, $r = 0.9980$

3.6 Recovery rates and relative standard deviations

Average recovery rates at 0.02-0.5 $\mu g/m^3$ were in a range of 83.0–104.6% calculated as averages of 5 and 4 values.

Fortification	Concentration in air	Recovery	RSD	n
0.5 μg	1.39 $\mu g/m^3$	102%	6%	5
5.0 μg	13.9 $\mu g/m^3$	86%	12%	5
Overall		94%	12%	10

3.7 Limit of quantification and determination

LOQ: 1.4 $\mu g/m^3$
LOD: <0.30 $\mu g/m^3$

3.8 Precision

3.8.1 Repeatability

The method was successfully validated with five values at both fortification levels, with recoveries in the range from 70% to 110% and relative standard deviations below 20% for all analytes and ion transitions.

Section A4.2 **Annex Point IIA4.2**	**Analytical Methods for Detection and Identification in (b) air**
3.8.2 Independent laboratory validation	No
	4 APPLICANT'S SUMMARY AND CONCLUSION
4.1 Materials and methods	Air is sucked through XAD adsorption tubes at about 1.0 L/min for 6 hours (total air sampling volume ≈ 0.36 m^3). Subsequently, the adsorption material is extracted with ethyl acetate, the internatandard lambda-cyhalothrin is added to the extract, and finally analysed by gas chromatography with mass spectrometric detection in the negative chemical ionisation mode (GC/MS(NCI)), monitoring three fragment ions. The method was successfully validated.
4.2 Conclusion	The method is suitable for the analytical determination of Alpha-cypermethrin in air with a LOQ of 1.4 µg/m^3. The method validation fulfils the requirements of SANCO/825/00 rev. 8.1 (16/11/2010) and SANCO/3029/99 rev. 4, (11/07/2000).
4.2.1 Reliability	1
4.2.2 Deficiencies	None

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)	
Date **Materials and Methods** **Results and discussion** **Conclusion** **Reliability** **Acceptability** **Remarks**	COMMENTS FROM ...	

Section A4.3
Annex Point IIIA4.1

Analytical methods for the active substance, and for residues thereof, in/on food or feedstuffs and other products where relevant

	JUSTIFICATION FOR NON-SUBMISSION OF DATA	Official use only
Other existing data [ ]	**Technically not feasible** [ ] **Scientifically unjustified** [ ]	
Limited exposure [X]	**Other justification** [ ]	
Detailed justification:	An analytical determination of residues in/on food or feedstuff is only required if the active substance or the material treated with it is to be used in a manner which may cause contact with food or feedstuff or intended to be placed on, in or near soils in agricultural or horticultural use. The use instructions for the biocidal example product clearly preclude any such contamination: The treated surface areas comprise cracks, crevices, spaces behind furnishings and other spaces suitable as hiding places for insect pests. These areas are characterised by limited accessibility for humans. For this reason, any contamination of food and/or feeding stuffs with Alphacypermethrin, e.g. by contact with the treated surfaces can be safely excluded. Hence, the submission of analytical methods for these commodities is not considered to be necessary.	
Undertaking of intended data submission [ ]		

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
Date **Evaluation of applicant's justification** **Conclusion** **Remarks**	EVALUATION BY RAPPORTEUR MEMBER STATE (*)	
Date **Evaluation of applicant's justification** **Conclusion** **Remarks**	COMMENTS FROM ...	

Section A5 (Annex Point) — Effectiveness against target organisms and intended uses

			Official use only
5.1	**Function (IIA5.1)**	Insecticide PT 18	
5.2	**Organism(s) to be controlled and products, organisms or objects to be protected (IIA5.2)**		
5.2.1	Organism(s) to be controlled	The organisms to be controlled by the biocidal example product are: Cockroaches (Blattodea) Fleas (Siphonaptera)	
5.2.2	Products, organisms or objects to be protected	Domestic premises, public buildings and hospitals (including occupied wards), as well as in hotels, restaurants, kitchens and in industrial premises including food-processing plants. No application on animals (e.g. against cat fleas).	