Section A4.1

Annex Point IIA4.1/4.2 & IIIA-IV.1

Analytical Method for Detection and Identification of Active Ingredient

Official use only

1 REFERENCE

1.1	**Reference**	[redacted]. Analytical Methods to Verify Certified Limits of [redacted] technical Grade. Environmental Health Science Laboratory, Sumitomo Chemical Co., Ltd. [redacted] 15 June 1995 The study was conducted between 30 July 1993 – 15 June 1995.
1.2	**Data protection**	Yes
1.2.1	Data owner	Sumitomo Chemical Co. (SCC) Ltd., Japan
1.2.2		
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 1.

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2.1	**Guideline study**	Yes U.S EPA-FIFRA, 40 CFR, Subdivision D. Guideline 62-3.
2.2	**GLP**	Yes
2.3	**Deviations**	No

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	Not required
3.1.2	Cleanup	Not required
3.2	**Detection**	
3.2.1	Separation method	Analytical method 1, determination of [redacted] content: Determined by gas chromatography (GC) on a column of 3% Thermon 3000 (3mm ID x 1m). FID detector. Oven temperature 220 °C, injection port and detector temperature 270 °C. Carrier gas, nitrogen. Retention time of [redacted] approx. 12 minutes. Analytical method 2, determination of Trans-isomer ratio: Determined by gas chromatography (GC) on a column of 5% Silicone DC QF-1 (3mm ID x 2m). FID detector. Oven temperature 200 °C, injection port and detector temperature 250 °C. Carrier gas, nitrogen. Retention time of *trans*-isomer approx. 30-35 minutes. Analytical method 3, (1R)- isomer ratio: Determined by normal phase high performance liquid chromatography (HPLC) on a column of SUMICHIRAL OA-2000 (5 µm, 4mm ID x 25cm). Detection: UV absorption photometer, wavelength 230 nm. Mobile phase is hexane/dichloromethane/acetonitrile, 70/29/1. Column temperature is ambient. Retention time of (1R)-*trans*-isomer approx. 25-35 minutes.

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3.2.2	Detector	For GC - flame ionisation detector (FID) - temperatures given in 3.2.1 above. For HPLC – an ultraviolet absorption photometer, wavelength 230 nm.
3.2.3	Standard(s)	An internal standard is used in the analysis of active ingredient content. The internal standard is di-2-ethylhexyl phthalate (180 mg dissolved in 100 mL acetone).
3.2.4	Interfering substance(s)	None
3.3	**Linearity**	
3.3.1	Calibration range	[redacted]: 2.5 to 10 mg/mL
3.3.2	Number of measurements	[redacted]: 5
3.3.3	Linearity	[redacted]: $r^2 = 1.0000$
3.4	**Specificity: interfering substances**	[redacted]: there were no interfering substances. The geometrical isomers and optical isomers were separated and no other ingredients interfered with the determination.
3.5	**Recovery rates at different levels**	As a number of impurities were underestimated, correction factors were introduced such that the final recovery range was 99 to 110%.
3.5.1	Relative standard deviation	Not stated
3.6	**Limit of determination**	
3.7	**Precision**	
3.7.1	Repeatability	[redacted]:

Analytical data (%)	Mean (%)	RSD (%)
91.8, 92.0, 91.9, 92.2, 92.0, 92.0	92.0	0.1

3.7.2	Independent laboratory validation	Not required as the method is not for use to determine residues levels in foodstuffs. Within the method validation, "ruggedness" was determined by conducting the analysis using two analysts with different equipment on different days.

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4 APPLICANT'S SUMMARY AND CONCLUSION

4.1 Materials and methods

Analytical methods were developed and validated to measure active ingredient content, optical and geometric isomer ratio and impurity content of imiprothrin.

The methods use standard laboratory equipment, either gas chromatography (GC) or normal phase high performance liquid chromatography (HPLC).

4.2 Conclusion

Method validation for the active substance:

Linearity – The analytical calibration was conducted over 2.5 to 10 mg/mL. The concentration used in the method was 5 mg/mL (ie the calibration extended more than ± 20% of the concentration of the analyte used in the method). The correlation coefficient was acceptable (1.0000) and was determined using single determinations at 5 concentrations.

Specificity – there were no interferences.

Precision (repeatability) – Six replicate sample determinations were conducted for [redacted] content, *trans*-isomer ratio and (1R)-isomer ratio. The mean value was reported for each. The RSD was acceptable for all analyses (0.0 - 0.1%).

4.2.1 Reliability

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4.2.2 Deficiencies

Yes

The method included some deficiencies; there was no definitive characterisation of the impurities (e.g. using MS) and there was no linearity measurement for the impurity standards. Nevertheless the method is sufficiently robust to determine the active substance and impurity content at ≥ 1g/kg.

Evaluation by Competent Authorities

Use separate "evaluation boxes" to provide transparency as to the comments and views submitted

EVALUATION BY RAPPORTEUR MEMBER STATE

Date

24/05/2010

Materials and methods

Conclusion

Adopt applicant's version however it is noted that discussion surrounding the deficiencies of the method in relation to determination of impurities should be removed.

Reliability

[redacted]

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Acceptability	As the use of GC-FID/HPLC-UV is not considered a sufficiently specific method, further data are required to confirm specificity of the method for the determination of imiprothrin in the technical material.
Remarks	
	COMMENTS FROM ...
Date	*Give date of comments submitted*
Results and discussion	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant´s summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	