Section A4.2 Analytical Methods for Detection and Identification

Annex Point IIA4.2 & IIIA-IV.1

			Official use only
		1 REFERENCE	
1.1	**Reference**	[redacted]. [redacted]: Analytical Methods of [redacted] for the Exposure Study. Sumitomo Chemical Co., Ltd. [redacted]. 26 May 2006	x
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 I	
		2	
2.1	**Guideline study**	No	
2.2	**GLP**	No	
2.3	**Deviation**	No	
		3 MATERIALS AND METHODS	
3.1	**Preliminary treatment**		
3.1.1	Enrichment	Extraction with acetone	
3.1.2	Cleanup	Not required	
3.2	**Detection**		
3.2.1	Separation method	Determination by gas chromatography (GC) on CBP 20-W12-100 column (12 meter). Oven temperature 220°C. Injection port 270°C. Carrier gas Helium. Carrier gas flow 40 mL/minute. Injection volume 2 µL. Retention time of S-41311 approximately 7 minutes. Internal standard (S31183) retention time approximately 9 minutes	
3.2.2	Detector	Flame thermionic detector (FTD). Detector temperature 270°C. Hydrogen gas pressure 0.6 kg/cm^2. Air gas pressure 0.5 kg/cm^2	
3.2.3	Standard(s)	[redacted] used as an internal standard.	
3.2.4	Interfering substance(s)	Not required	
3.3	**Linearity**		
3.3.1	Calibration range	0.4 to 20 µg/mL (equivalent to 0.8 to 40 ng injected onto the GC/FTD)	
3.3.2	Number of measurements	6	
3.3.3	Linearity	$r^2 = 0.9983$	

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3.4	**Specifity: interfering substances**	There were no interfering substances for [redacted]	
3.5	**Recovery rates at different levels**	Duplicate determinations of [redacted] were made at three levels. The levels were equivalent to air concentrations of 2.667 µg/m³, 13.333 µg/mL and 166.667 µg/m³. Recoveries for 2.667 µg/m³: 100 and 99.2 % mean 99.6% Recoveries for 13.333 µg/m³: 96.7 and 96.4 % mean 96.6% Recoveries for 166.667 µg/m³: 100 and 100 % mean 100%	x
3.5.1	Standard deviation	Not required	
3.6	**Limit of determination**	The limit of detection was 0.4 µg/mL (equivalent to 0.666 µg/m³).	x
3.7	**Precision**		
3.7.1	Repeatability	See section 3.5 for recoveries. Overall precision (RSD) across all levels was 1.7%	x
3.7.2	Independent laboratory validation	Not required	

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4 APPLICANT'S SUMMARY AND CONCLUSION

4.1 Materials and methods

An analytical method for the determination of [redacted] in air, was tested over the concentration range 2.667 to 166.667 µg/m^3. X

The method uses standard laboratory equipment for extraction and gas chromatography with flame thermionic detection detection (GC/FTD).

4.2 Conclusion

Linearity - The analytical procedure was conducted over the range 0.4 to 20 µg/mL. The lowest concentration was less than 30% of the lowest sample concentration analysed and the highest concentration was above the highest level to be analysed. The correlation coefficient was acceptable (0.9983) and was determined at 6 concentrations. X

Specificity - There were no interferences.

Precision and recovery - Duplicate replicate sample determinations were made at three levels (2.667, 13.333 and 166.667 µg/m^3) in air. Mean recovery of [redacted] was within the range of 96.7 to 100%.
Overall precision across all levels was 1.73%..

4.2.1 Reliability [redacted]

4.2.2 Deficiencies

Yes. x
The method contains no details of the type of air sampled (temperature and humidity ranges). The number of replicate samples at each level was insufficient to be able to calculate precision at individual levels and details of the extraction procedure are not clear (time of contact needed with acetone, shaking or sonication required?) Nevertheless the method appears sufficiently robust to extract and quantify levels of [redacted] in air over the range 2.667 to 166.667 µg/m^3.

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	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	*06/06/07*
Materials and methods	*Applicants version is acceptable with the following amendments:* ***1.1*** *delete "2006" and replace with "1995"* ***3.5*** *delete "13.333 microgram/mL" and replace with "13.333 microgram/m^3"* ***3.6*** *delete "(equivalent to 0.666 microgram/ m^3)"* ***3.7.1*** *delete text* ***4.1*** *delete first paragraph* ***4.1*** *delete "detection" – word repeated in last line of second paragraph* ***4.2*** *Delete last sentence.* ***4.2.2*** *delete last sentence* *A confirmatory method is also required as GC-FTD is not considered sufficiently specific in accordance with SANCO 825/00 rev. 8.1, and confirmatory methods are not available for either soil or water.*
Conclusion	*Adopt applicant's version with above amendments. Further information should be provided in relation to the air temperature and relative humidity used for validation of the method together with additional replicates being analysed at appropriate fortification levels to comply with the requirements of SANCO 825/00 rev. 8.1. These data may be submitted at product authorisation stage.* ***Data Gap*** *– Data to address the air temperature and relative humidity used to validate the GC-FID method.* *A confirmatory method for the determination of imiprothrin in air is required, satisfactorily validated in accordance with the requirements of SANCO 825/00 rev. 8.1.* *(NOTE: A suitable method based on GC-MS/MS has subsequently been supplied and is evaluated and summarised within the 'monitoring methods addendum'. The method is acceptably validated with a LOQ of 0.83µg/m^3.)*
Reliability	[redacted]
Acceptability	*acceptable*
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*

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Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

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	JUSTIFICATION FOR NON-SUBMISSION OF DATA	Official use only
Other existing data [ ] **Limited exposure [x]**	**Technically not feasible [ ]** **Scientifically unjustified [ ]** **Other justification [ ]**	
Detailed justification:	**Soil and Water** The biocidal products containing the active substance, Imiprothrin, are intended for use in domestic or restaurant kitchens and other areas in buildings where small infestations of cockroaches occur. The proposed area of use is restricted to indoor use and excludes use to building perimeters and should not be applied in and around drains. Treated surfaces should not be washed following application. The restricted use and exclusive pattern of the aerosol spray indicates that there will be minimal direct exposure to the aqueous or soil environment. It is not considered that any measurable quantity will reach the outdoor environment, thus analytical methods for soil and water are not submitted.	
Undertaking of intended data submission [ ]	Not applicable	

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	*06/06/07, 10/02/17*
Evaluation of applicant's justification	

Conclusion	*Add heading 'Food and feeding stuffs'. Under 'Detailed justification' for this heading, add 'Analytical methods for food and feeding stuffs are not considered to be necessary - the product is intended to be used for spot, crack and crevice treatment in domestic or restaurant kitchens and not as a general broadcast surface spray treatment over kitchen surfaces. Its use will be restricted to more inaccessible areas in the kitchen.'* *Applicant's justification is acceptable for soil, sediment and ground water.* *For drinking water, in accordance with the requirements of 'Guidance on the Biocidal Products Regulation, Volume 1, Part A Information Requirements, Version 1 dated November 2014', as imiprothrin falls within the definition of a pesticide given in Annex I to Council Directive 98/83/EC, an analytical method for the determination of imiprothrin in drinking water acceptably validated in accordance with the parameters as specified within Directive 98/83/EC is required.* *For surface water, the environmental risk assessment concludes that potential exposure of surface water to imiprothrin via sewage treatment is assumed. Consequently, a method of analysis to monitor for imiprothrin in surface water is required.*
Remarks	*A data gap was initially set for method of analysis for determination of imiprothrin in surface water and drinking water acceptably validated in accordance with the parameters as specified within Directive 98/83/EC. New methods to address these requirements have subsequently been submitted and have been evaluated within a monitoring methods addendum. The method of analysis for drinking water is considered to be acceptable whilst further validation data are required to support the proposed method for surface water to validate a LOQ in accordance with the proposed PNEC of 0.038 µg/L – this shall be set as a data gap.*
	COMMENTS FROM OTHER MEMBER STATE *(specify)*
Date	*Give date of comments submitted*
Evaluation of applicant's justification	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Remarks	