Section A4.1
Annex Point IIA4.1 IIIA-IV.1

Analytical Methods for Detection and Identification

Official use only

1 REFERENCE

Reference

A4.1/01:

[redacted] Verfahren zur Prüfung der Gehaltsbestimmung von Sorbinsäure, Kaliumsorbat, Kaliumsorbat Lösung 50 % und Calciumsorbat, [redacted]

A4.1/02:

[redacted] Verfahren zur Bestimmung des Wassergehaltes in Kaliumsorbat Pulver und Granulat sowie Calciumsorbat und Sorbinsäure, [redacted]

The details of the analytical method are considered to represent commercially sensitive and therefore confidential data by [redacted]

The report is summarised in detail in the file of confidential information of [redacted].

Evaluation by Competent Authorities

Section A4.1 **Annex Point IIA4.1 IIIA-IV.1**	**Analytical Methods for Detection and Identification**
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	[redacted]
Materials and methods	[redacted]
Conclusion	[redacted]
Reliability	[redacted]
Acceptability	[redacted]
Remarks	[redacted]
	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	

Section A4.2 Annex Point IIA4.2	Analytical methods for detection and identification of the active substance in (a) soil	Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/01:** [redacted] Validation of an analytical method for the determination of Sorbic acid in soil. [redacted]
1.2	**Data protection**	Yes
1.2.1	Data owner	[redacted]
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/IA.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	SANCO/825/00 rev. 6.
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	No

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	Residues of Sorbic acid were extracted with acetonitrile/water (1/1), filtrated and evaporated to dryness.
3.1.2	Clean-up	Further clean-up was performed by water steam distillation using an automated water steam distillation device and solid-phase extraction of the acidified distillate on PPL-SPE-cartridges. Residues were eluted with methanol. After evaporation, residues were derivatised using BF_3/methanol solution.
3.2	**Detection**	
3.2.1	Separation method	Capillary gas chromatography with an mass selective detector (GC-MSD).
3.2.2	Detector	Mass selective detector (m/z = 111 for quantification and m/z = 126, 95 for verification).
3.2.3	Standard(s)	Sorbic acid, batch no. 5661, purity 99.8% Soil standard: Soil 3A (LUFA Speyer, Germany), batch no. F3A0303, pH 7.1, dry matter 90%, organic carbon content 2.6%, soil type loam.

Section A4.2
Annex Point IIA4.2

Analytical methods for detection and identification of the active substance in (a) soil

3.3	**Linearity**	
3.3.1	Calibration range	The detector response for the analytical standard was found to be linear between 0 and 1000 ng/ml in sample extracts.
3.3.2	Number of measurements	The calibration curve was plotted based on seven different concentrations.
3.3.3	Linearity	The equation of a typical standard calibration function for Sorbic acid was determined as (graph forced through the origin) $y = 0.128\,x^2 + 354\,x$ with a regression coefficient of $r^2 = 1.000$ where y is the response in the chromatogram, and x the concentration of the substance [ng/ml].
3.4	**Specificity: interfering substances**	The method is suitable for the specific determination of residues of Sorbic acid in soil. As three fragment ions can be used for the determination, the method can be classified as highly specific. Under the chromatographic conditions used in this study, the retention time was approx. 5.6 min for Sorbic acid. Analysed blank control samples gave no interfering signals (< 30% of LOQ).
3.5	**Recovery rates and standard deviations at different levels**	The results are presented in Table A4.2- 1.
3.6	**Limit of determination**	The limit of quantification (LOQ) is 0.05 mg/kg.
3.7	**Precision**	
3.7.1	Repeatability	The average recovery was in a range of 70 and 110%, the relative standard deviation was below 20%.
3.7.2	Independent laboratory validation	Not required.

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	Residues of Sorbic acid were extracted with acetonitrile/water (1/1). Further clean-up was performed by water steam distillation and solid-phase extraction on PPL-SPE-cartridges. After derivatisation with BF_3 in methanol, determination was performed by GC-MSD.
4.2	**Conclusion**	Average recoveries were in the range of 70 - 110% with relative standard deviations below 20%. No interfering blanks were observed. Therefore, this method fulfils the requirements of SANCO/825/00 rev.6 and can be used as an enforcement method for the determination of residues of Sorbic acid in soil. Since the analytically relevant moiety of Sorbic acid as well as of Potassium sorbate is the Sorbate anion, the submitted method is likewise recommended as a suitable technique for detection and identification of Potassium sorbate residues.

Section A4.2 **Annex Point IIA4.2**	**Analytical methods for detection and identification of the active substance in (a) 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
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	[redacted]
Materials and Methods	[redacted]
Results and discussion	[redacted]
Conclusion	[redacted]
Reliability	[redacted]
Acceptability	[redacted]
Remarks	[redacted]
	COMMENTS FROM ...
Date	
Materials and Methods	
Results and discussion	
Conclusion	
Reliability	
Acceptability	
Remarks	

Table A4.2- 1: Recovery rates of Sorbic acid in soil.

Fortification level [mg/kg]	n	Recovery [%]		
		Range	**Mean ± RSD**	**Overall ± RSD**
0.05	5	84 – 123	99 ± 20%	96 ± 16%
0.5	5	77 – 108	97 ± 13%	

RSD = relative standard deviation
n = number of determinations

Section A4.2 **Annex Point IIA4.2**	**Analytical methods for detection and identification of the active substance in (b) air**	
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	**Official use only**
Other existing data [ ] **Limited exposure [X]**	**Technically not feasible [ ]** **Scientifically unjustified [X]** **Other justification [ ]**	
Detailed justification:	A method for the detection of residues in air is not suggested, since Potassium sorbate is neither volatile nor intended to be sprayed or applied in any other way resulting in occurrence of Potassium sorbate in air.	
Undertaking of intended data submission [ ]		

	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)	
Date	[redacted]	
Materials and Methods		
Results and discussion		
Conclusion		
Reliability		
Acceptability	[redacted]	
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 of the active substance in (c) water**

Official use only

1 REFERENCE

1.1	**Reference**	**A4.2/02:** [redacted] Validation of an analytical method for the determination of Sorbic acid in surface water. [redacted]
1.3	**Data protection**	Yes
1.2.1	Data owner	[redacted]
1.2.3	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/IA.

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	SANCO/825/00 rev. 6.
2.2	**GLP**	Yes (certified laboratory)
2.3	**Deviations**	No

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	Residues of Sorbic acid were extracted by solid-phase extraction of the acidified sample on PPL-SPE-cartridges. Residues were eluted with methanol and evaporated.
3.1.2	Clean-up	No further clean up was performed. Residues of Sorbic acid were derivatised using BF_3/methanol solution.
3.2	**Detection**	
3.2.1	Separation method	Capillary gas chromatography with an mass selective detector (GC-MSD)
3.2.2	Detector	Mass selective detector (m/z = 111 for quantification and m/z = 126, 95 for verification).

Section A4.2 — Analytical methods for detection and identification of the active substance in (c) water

Annex Point IIA4.2

3.2.3	Standard(s)	Sorbic acid, batch no. 5661, purity 99.8%.
		Specification of surface water (sample was taken from the stream 'Federbach' in Malsch/Germany):
		Appearance: slightly brown and weakly turbid, odourless
		pH (20°C): 7.5
		Specific electric conductivity (20°C): 120 µS/cm
		Total hardness: 0.42 mmol/l
		Spectral absorption coefficient (254 nm): 12.6 m^{-1}
		Dissolved organic carbon: 4.4 mg C/l.
3.3	**Linearity**	
3.3.1	Calibration range	The detector response for the analytical standards was found to be linear between 0 and 250 ng/ml.
3.3.4	Number of measurements	The calibration curve was based on seven different concentrations.
3.3.5	Linearity	The equation of a typical standard calibration function for Sorbic acid was determined as (graph forced through the origin) $y = 2090\,x$ with a regression coefficient of r^2=0.999 where y is the response in the chromatogram, and x the concentration of the substance [ng/ml].
3.4	**Specificity: interfering substances**	The method is suitable for the specific determination of residues of sorbic acid in water. As three fragment ions could be used for determination, the method can be classified as highly specific. Under the chromatographic conditions used in this study, the retention time was approx. 5.6 min for sorbic acid. Blank control samples analysed gave no interfering signals (< 30% of LOQ).
3.5	**Recovery rates and standard deviations at different levels**	Please refer to Table A4.2- 2.
3.6	**Limit of determination**	The limit of quantification (LOQ) is 0.1 µg/l.
3.7	**Precision**	
3.7.1	Repeatability	The average recovery is in a range between 70 – 110% and the relative standard deviation is less than 20%.
3.7.2	Independent laboratory validation	Not required.

4 APPLICANT'S SUMMARY AND CONCLUSION

Section A4.2 — Analytical methods for detection and identification of the active substance in (c) water

Annex Point IIA4.2

4.1	**Materials and methods**	Residues of sorbic acid were extracted by solid-phase extraction on PPL-SPE-cartridges. Residues were eluted with methanol. After derivatisation with BF_3 in methanol determination was performed by GC-MSD.
4.2	**Conclusion**	Average recoveries were in the range of 70 – 110% with relative standard deviations < 20%. No interfering blanks were observed. Therefore, this method fulfils the requirements of SANCO/825/00 rev.6 and can be used as an enforcement method for the determination of residues of sorbic acid in surface, ground and drinking water. Since the analytically relevant moiety of Sorbic acid as well as of Potassium sorbate is the Sorbate anion, the submitted method is likewise recommended as a suitable technique for detection and identification of Potassium sorbate residues.
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	[redacted]	
Materials and Methods	[redacted]	
Results and discussion	[redacted]	
Conclusion		
Reliability		
Acceptability	[redacted]	
Remarks	[redacted]	
	COMMENTS FROM ...	
Date		
Materials and Methods		
Results and discussion		
Conclusion		
Reliability		
Acceptability		
Remarks		

Table A4.2- 2: Recovery rates of Sorbic acid in surface water.

Fortification level [µg/l]	n	Recovery [%]		
		Range	Mean ± RSD	Overall ± RSD
0.1*	5	86 – 122	100 ± 14%	90 ± 17%
1.0	5	67 – 91	81 ± 12%	

RSD: relative standard deviation
n: number of determinations

Section A4.2 **Annex Point IIA4.2**	**Analytical methods for detection and identification of the active substance in** **(d) animal and human body fluids and tissues**	
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	**Official use only**
Other existing data [ ]	**Technically not feasible [ ]** **Scientifically unjustified [X]**	
Limited exposure [X]	**Other justification [ ]**	
Detailed justification:	A method for the detection of residues of the active substance in animal and human body fluids and tissues is not submitted, since Potassium sorbate is not classified as toxic (T) or as highly toxic (T+).	
Undertaking of intended data submission [ ]		

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

Section A4.3 **Annex Point IIIA 6.1**	**Analytical methods including recovery rates and the limits of determination of 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 [ ] **Limited exposure [X]**	**Technically not feasible [ ]** **Scientifically unjustified [ ]** **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 recommendations for use of Potassium sorbate as a wood protection agent clearly precludes any such contamination, which is why the submission of such methods 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
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	[redacted]
Evaluation of applicant's justification	[redacted]
Conclusion	[redacted]
Remarks	[redacted]
	COMMENTS FROM ...
Date	
Evaluation of applicant's justification	
Conclusion	
Remarks	