

# **FINAL REGISTRATION REPORT**

## **Part B**

### **Section 5**

#### **Analytical Methods**

Detailed summary of the risk assessment

Product code: SHA 0100 Y

Product name: DECIDE

Chemical active substance:

Deltamethrin, 50 g/L

Central Zone

Zonal Rapporteur Member State: Poland

**CORE ASSESSMENT/**

Applicant: SHARDA Cropchem España S.L.

Submission date: August 2019

Finalisation date: 09/2021; 04/2022

## Version history

When	What
September 2021	Assessment finalised by RMS
April 2022	The final version of RR

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## 5 Analytical methods

### 5.1 Conclusion and summary of assessment

Sufficiently sensitive and selective analytical methods are available for the active substance(s) and relevant impurities in the plant protection product.

Noticed data gaps are:

none

Sufficiently sensitive and selective analytical methods are available for all analytes included in the residue definitions.

Noticed data gaps are:

none

Commodity/crop	Supported/ Not supported
Brassicas	Supported
Strawberries	Supported
Tomatoes	Supported
Ornamentals	Not required

### 5.2 Methods used for the generation of pre-authorization data (KCP 5.1)

#### 5.2.1 Analysis of the plant protection product (KCP 5.1.1)

##### 5.2.1.1 Determination of active substance and/or variant in the plant protection product (KCP 5.1.1)

An overview on the acceptable methods and possible data gaps for analysis of Deltamethrin in plant protection product is provided as follows:

Comments of zRMS:	The proposed analytical method is suitable for the determination of deltamethrin in plant protection product Decide (Deltamethrin 5% CS). The proposed analytical method has been fully validated in terms of the interference, specificity, linearity, accuracy and precision. Proposed method fulfils the requirements of SANCO/3030/99 rev.4 guidance. Proposed method fulfils also the requirements of SANCO/3030/99 rev.5 guidance. The validation of the analytical method has been accepted.
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Reference: KCP 5.1.1/01

Report Accelerated storage stability test by heating at elevated temperature of Deltamethrin 5% CS. XXX V.S., 2018, report No. G13965.

Guideline(s): Yes, SANCO/3030/99 rev. 4

Deviations: No  
 GLP: Yes  
 Acceptability: Yes

## Materials and methods

Test item solution was prepared in water/CAN and active ingredient determination was performed by HPLC-DAD chromatographic system using deltamethrin standard as external reference item prepared in ACN. HPLC column Zorbax SB-C8, 3.5 µm, 150x4.6 mm was used and quantification was carried out at 202 nm.

## Validation - Results and discussions

**Table 5.2-1: Methods suitable for the determination of Deltamethrin in plant protection product Deltamethrin 5% CS**

	Deltamethrin
Author(s), year	XXX, V.S., 2018
Principle of method	HPLC-DAD
Linearity (linear between mg/L / % range of the declared content) (correlation coefficient, expressed as r)	$Y = 4565623.21x + 1389528.19$ (linear between 4.99 to 149.55 µg/mL) $r = 1.000$
Precision – Repeatability Mean n = 5 (%RSD)	0.40 % RSD
Accuracy n = 6 (% Recovery)	98.92%
Interference/ Specificity	Specific, no interferences
Comment	

## Conclusion

Analytical method (HPLC-DAD) is suitable for the determination of Deltamethrin in the formulation. The method is validated according to SANCO/3030/99 rev4 and meets the requirements set out in this guideline.

### 5.2.1.2 Description of analytical methods for the determination of relevant impurities (KCP 5.1.1)

No relevant impurities are present and/or expected in the formulation, therefore no methods are provided and required.

Comments of zRMS:	Comment on study; acceptable or not; deficiencies, corrections, according to recent guidelines or not, used in evaluation or only as additional information
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### 5.2.1.3 Description of analytical methods for the determination of formulants (KCP 5.1.1)

Not relevant.

### 5.2.1.4 Applicability of existing CIPAC methods (KCP 5.1.1)

A CIPAC method No. 333 is available for Deltamethrin.

### 5.2.2 Methods for the determination of residues (KCP 5.1.2)

Please refer to the post-registration method.

## 5.3 Methods for post-authorization control and monitoring purposes (KCP 5.2)

### 5.3.1 Analysis of the plant protection product (KCP 5.2)

Analytical methods for the determination of the active substance and relevant impurities in the plant protection product are already submitted in accordance with the requirements set out in point 5.2.1.

### 5.3.2 Description of analytical methods for the determination of residues of Deltamethrin (KCP 5.2)

#### 5.3.2.1 Overview of residue definitions and levels for which compliance is required

The residue definition stated in Regulation (EU) No. 2018/832 is the following:

- For food of plant origin: Deltamethrin (*cis*-Deltamethrin)
- For food of animal origin: Deltamethrin (*cis*-Deltamethrin)

The last established residue definition is therefore Deltamethrin (*cis*-Deltamethrin) for both plant and animal products. Analytical methods for residues in plant and animal matrices provided in this dossier are therefore complying with the latest residue definition stated in Reg. (EU) 2018/832.

**Table 5.3-1: Relevant residue definitions for monitoring/enforcement and levels for which compliance is required**

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high water content	Deltamethrin ( <i>cis</i> -deltamethrin)	0.01 mg/kg	Reg. (EU) 2018/832
Plant, high acid content		0.01 mg/kg	Reg. (EU) 2018/832
Plant, high protein/high starch content (dry commodities)		0.02 mg/kg	Reg. (EU) 2018/832
Plant, high oil content		0.01 mg/kg	Reg. (EU) 2018/832
Muscle	Deltamethrin ( <i>cis</i> -	0.02 mg/kg	Reg. (EU) 2018/832

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Milk	deltamethrin)	0.05 mg/kg	Reg. (EU) 2018/832
Eggs		0.02 mg/kg	Reg. (EU) 2018/832
Fat		0.1 mg/kg	Reg. (EU) 2018/832
Liver, kidney		0.02 mg/kg	Reg. (EU) 2018/832
Soil (Ecotoxicology)	Deltamethrin (cis-deltamethrin)	0.05 mg/kg	common limit
Drinking water (Human toxicology)	Deltamethrin (cis-deltamethrin)	0.1 µg/L	general limit for drinking water
Surface water (Ecotoxicology)	Deltamethrin (cis-deltamethrin)	0.0041 µg/L	Chronic NOEC Daphnia
Air	Deltamethrin (cis-deltamethrin)	2250 µg/m <sup>3</sup>	AOEL sys: 0.0075 mg/g bw/d
Tissue (meat or liver)	Deltamethrin (cis-deltamethrin)	0.1 mg/kg	common limit
Body fluids		0.05 mg/L	common limit

### 5.3.2.2 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Deltamethrin in plant matrices is given in the following tables. For the detailed evaluation of new/additional studies it is referred to Appendix 2.

**Table 5.3-2: Validated methods for food and feed of plant origin (required for all matrix types, “difficult” matrix only when indicated by intended GAP)**

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)				
Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
High water content	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Marten, 1998a,c) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
		0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)
	Confirmatory (if required)	0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)
High acid content	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Marten, 1998c) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
		0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)				
Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing / EU agreed
	Confirmatory (if required)	0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)
High oil content	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Marten, 1998a) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
		0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)
	Confirmatory (if required)	0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)
High protein/high starch content (dry)	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Marten, 1998a) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
		0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)
	Confirmatory (if required)	0.005 mg/kg	MRM (GC-MS)	EURL-FV 2014 M12 (EURL, 2014)

For any special comments or remarkable points concerning the analytical methods for the determination of residues in plant matrices, please refer to Appendix 2.

**Table 5.3-3: Statement on extraction efficiency**

	Method for products of plant origin
Required, available from:	-
Not required, because:	Not presented in the Addendum to the Monograph Annex B, 2002

### 5.3.2.3 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Deltamethrin in animal matrices is given in the following tables. No additional studies are submitted.

**Table 5.3-4: Validated methods for food and feed of animal origin (if appropriate)**

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)				
Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing
Milk	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Martens, 2000) Addendum to the Monograph Annex B, 2002

<b>Component of residue definition: Deltamethrin (<i>cis</i>-deltamethrin)</b>				
<b>Matrix type</b>	<b>Method type</b>	<b>Method LOQ</b>	<b>Principle of method (i.e. GC-MS or HPLC-UV)</b>	<b>Author(s), year / missing</b>
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
	Confirmatory (if required)	0.01 mg/kg	GC-MSD	DFG S 19 Standard method (EFSA, 2017)
Eggs	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Martens, 2000) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
	Confirmatory (if required)	0.01 mg/kg	GC-MSD	DFG S 19 Standard method (EFSA, 2017)
Muscle	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Martens, 2000) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
	Confirmatory (if required)	0.01 mg/kg	GC-MSD	DFG S 19 Standard method (EFSA, 2017)
Fat	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Martens, 2000) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
	Confirmatory (if required)	0.01 mg/kg	GC-MSD	DFG S 19 Standard method (EFSA, 2017)
Kidney, liver	Primary	0.02 mg/kg	MRM-1 (GC-ECD)	(Martens, 2000) Addendum to the Monograph Annex B, 2002
	ILV	0.02 mg/kg	MRM-1 (GC-ECD)	(Haines, 2001) Addendum to the Monograph Annex B, 2002
	Confirmatory (if required)	0.01 mg/kg	GC-MSD	DFG S 19 Standard method (EFSA, 2017)

For any special comments or remarkable points concerning the analytical methods for the determination of residues in animal matrices, please refer to Appendix 2.

**Table 5.3-5: Statement on extraction efficiency**

	<b>Method for products of animal origin</b>
Required, available from:	-
Not required, because:	Not presented in the Addendum to the monograph Annex B, 2002. Extraction efficiency has not been addressed. This is considered acceptable as residues >LOQ are not expected in products of animal origin.

### 5.3.2.4 Description of methods for the analysis of soil (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Deltamethrin in soil is given in the following tables. For the detailed evaluation of new studies it is referred to Appendix 2.

**Table 5.3-6: Validated methods for soil (if appropriate)**

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)			
Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing
Primary	0.001 mg/kg	GC-ECD	(Benwell, 1992), DAR, 1998
	0.002 mg/kg	GC-ECD	(Grigor, 1994), DAR, 1998
	0.0001 mg/kg	LC-MS/MS	XXX R., 2013a (new)
Confirmatory	0.0001 mg/kg	LC-MS/MS	XXX R., 2013a (new)

For any special comments or remarkable points concerning the analytical methods for soil please refer to Appendix 2.

### 5.3.2.5 Description of methods for the analysis of water (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Deltamethrin in surface and drinking water is given in the following tables. No new/additional studies are submitted

**Table 5.3-7: Validated methods for water (if appropriate)**

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)				
Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing
Drinking water	Primary	0.05 µg/L	GC-ECD	(Marten, 1999) Addendum to the Monograph Annex B, 2002
	ILV	0.003 µg/L	GC-ECD (GC-MS/MS for confirmation)	(Class, 2001a) Addendum to the Monograph Annex B, 2002
	Confirmatory	0.05 µg/L	GC-ECD (confirmation by using different column)	(Marten, 1999) Addendum to the Monograph Annex B, 2002
		0.003 µg/L	GC-ECD (GC-MS/MS for confirmation)	(Class, 2001a) Addendum to the Monograph Annex B, 2002
Surface water	Primary	0.05 µg/L	GC-ECD	(Marten, 1999) Addendum to the Monograph Annex B, 2002
		0.003 µg/L	GC-ECD (GC-MS/MS for confirmation)	(Class, 2001a) Addendum to the Monograph Annex B, 2002
	Confirmatory	0.05 µg/L	GC-ECD (confirmation by using different column)	(Marten, 1999) Addendum to the Monograph Annex B, 2002
		0.003 µg/L	GC-ECD (GC-MS/MS)	(Class, 2001a) Addendum to

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)				
Matrix type	Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing
			for confirmation)	the Monograph Annex B, 2002

For any special comments or remarkable points concerning the analytical methods for water please refer to Appendix 2.

### 5.3.2.6 Description of methods for the analysis of air (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Deltamethrin in air is given in the following tables. For the detailed evaluation of new studies please refer to Appendix 2.

**Table 5.3-8: Validated methods for air (if appropriate)**

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)			
Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing
Primary	0.27 µg/m <sup>3</sup>	GC-ECD/MS	(Class, 2001b) Addendum to the Monograph Annex B, 2002
Confirmatory	0.27 µg/m <sup>3</sup>	GC-MS	(Class, 2001b) Addendum to the Monograph Annex B, 2002

For any special comments or remarkable points concerning the analytical methods for air it is referred to Appendix 2.

### 5.3.2.7 Description of methods for the analysis of body fluids and tissues (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Deltamethrin in body fluids and tissues is given in the following table. For the detailed evaluation of new studies it is referred to Appendix 2.

**Table 5.3-9: Methods for body fluids and tissues (if appropriate)**

Component of residue definition: Deltamethrin ( <i>cis</i> -deltamethrin)			
Method type	Method LOQ	Principle of method (i.e. GC-MS or HPLC-UV)	Author(s), year / missing
Primary	0.01 mg/kg (urine, faeces)	GC-ECD	(Akhtar, 1982) DAR, 1998
	0.02 mg/L (human plasma)	GC-ECD	(Tillier, 1998) DAR, 1998
	0.05 mg/L (blood)	LC-MS/MS	XXX, R. 2013b (new)
	0.1 mg/kg (liver)	LC-MS/MS	XXX, R. 2013b (new)
Confirmatory	0.05 mg/L (blood)	LC-MS/MS	XXX, R. 2013b (new)
	0.1 mg/kg (liver)		

For any special comments or remarkable points concerning the analytical methods for body fluids and tissues please refer to Appendix 2.

#### **5.3.2.8 Other studies/ information**

No new or additional studies have been submitted.

## Appendix 1 Lists of data considered in support of the evaluation

Tables considered not relevant can be deleted as appropriate.  
 MS to blacken authors of vertebrate studies in the version made available to third parties/public.

### List of data submitted by the applicant and relied on

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCP 5.1.1/01	XXX, V.S.	2018	Accelerated storage stability test by heating at elevated temperature of Deltamethrin 5% CS. Advinus report no. G13965. GLP Unpublished	N	Sharda
KCP 5.2/01	XXX, R.	2013a	Method validation-Determination of residues of deltamethrin in soil and sediment. Battelle report no. YV/12/013 GLP Unpublished	N	Sharda
KCP 5.2/03	XXX, R.	2013b	Method validation-Determination of residues of deltamethrin in blood and liver. Battelle report no. YV/12/007 GLP Unpublished	N	Sharda
KCP 5.2/04	Anonymous	2014	Dutch mini-luke (“NL-“) extraction method followed by LC and GC-MS/MS for multiresidue analysis of pesticides in fruits and vegetables. EURL-FV 2014 M12. Non GLP Published	N	N

**List of data submitted or referred to by the applicant and relied on, but already evaluated at EU peer review**

<b>Data point</b>	<b>Author(s)</b>	<b>Year</b>	<b>Title Company Report No. Source (where different from company) GLP or GEP status Published or not</b>	<b>Vertebrate study Y/N</b>	<b>Owner</b>
-	-	-	-	-	-

The following tables are to be completed by MS

**List of data submitted by the applicant and not relied on**

<b>Data point</b>	<b>Author(s)</b>	<b>Year</b>	<b>Title Company Report No. Source (where different from company) GLP or GEP status Published or not</b>	<b>Vertebrate study Y/N</b>	<b>Owner</b>
-	-	-	-	-	-

**List of data relied on not submitted by the applicant but necessary for evaluation**

<b>Data point</b>	<b>Author(s)</b>	<b>Year</b>	<b>Title Company Report No. Source (where different from company) GLP or GEP status Published or not</b>	<b>Vertebrate study Y/N</b>	<b>Owner</b>
-	-	-	-	-	-

## Appendix 2 Detailed evaluation of submitted analytical methods

### A 2.1 Analytical methods for Deltamethrin

#### A 2.1.1 Methods used for the generation of pre-authorization data (KCP 5.1)

No new or additional studies have been submitted

#### A 2.1.2 Methods for post-authorization control and monitoring purposes (KCP 5.2)

##### A 2.1.2.1 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

No new or additional studies have been submitted

##### A 2.1.2.2 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

No new or additional studies have been submitted

##### A 2.1.2.3 Description of Methods for the Analysis of Soil (KCP 5.2)

###### A 2.1.2.3.1 Analytical method 1

###### A 2.1.2.3.1.1 Method validation

Comments of zRMS:	Method is accepted
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<b>Reference</b>	KCP 5.2/01
<b>Report:</b>	Method Validation - Determination of Residues of Deltamethrin in Soil and Sediment, XXX, R. (2013a), report No YV/12/013
<b>Guidelines:</b>	SANCO/825/00 rev 8.1
<b>Deviations:</b>	No
<b>GLP:</b>	Yes

### Materials and methods.

Residues of deltamethrin were extracted by shaking with acetone/hexane (1:1). An aliquot was evaporated and reconstituted in water/methanol (3:7) containing 0.01M ammonium acetate and 5 ng/mL internal standard and analysed by LC-MS/MS.

### LC-MS/MS Analysis parameters:

Liquid Chromatography	
Instrument:	Agilent 1100 HPLC System
Column:	Purospher STAR RP-18 endcapped 55 mm × 4 mm, 3µm (Part number 1.50242.0001)
Column Temperature:	40 °C
Mobile Phase A:	Water/methanol (9:1) containing 0.01M ammonium acetate
Mobile Phase B:	Methanol containing 0.01M ammonium acetate
Mass Spectrometry	
Instrument:	API 5000 LC-MS/MS, AB Sciex
Ion Source:	Electrospray, (ESI, Turbolon Spray)
Polarity:	Positive

### Validation data:

#### Linearity

Six calibration standards between 0.015 to 1 ng/mL were injected, covering the required range from 30% of the LOQ to 20% above the highest fortification level. Calibration curves with correlation co-efficients  $r \geq 0.999$  were achieved for both transitions, demonstrating acceptable linearity. Calibration standards were prepared in solvent but using an internal standard to compensate matrix effect.

#### Accuracy & Precision

Average recoveries at each fortification level were all within the acceptance range of 70-120 % (for both quantitation and confirmatory transitions). The relative standard deviation (RSD) did not exceed 20 % at any fortification level and any interference was below 30 % of the LOQ (for both quantitation and confirmatory transitions).

#### Deltamethrin in Soil

Transition m/z	Fortification Level (mg/kg)	Individual Results (%)	n	Mean Recovery (%)	RSD (%)	Overall Mean Recovery (%)	Overall RSD (%)
523 – 281	0.0001	91, 86, 100, 92, 91	5	92	5.5	90	4.8
	0.001	93, 90, 86, 86, 88	5	89	3.3		
525 - 283	0.0001	85, 95, 104, 85, 97	5	93	8.8	92	6.8
	0.001	96, 96, 85, 90, 91	5	92	5.0		

#### Deltamethrin in Sediment

Transition m/z	Fortification Level (mg/kg)	Individual Results (%)	n	Mean Recovery (%)	RSD (%)	Overall Mean Recovery (%)	Overall RSD (%)
523 – 281	0.0001	107, 84, 107, 95, 84	5	95	12.1	97	9.2
	0.001	103, 107, 94, 95, 92	5	98	6.6		
525 - 283	0.0001	99, 88, 97, 102, 101	5	97	5.7	97	6.9
	0.001	106, 106, 88, 95, 91	5	97	8.7		

#### Specificity

No interference/contamination peak above 30% of the LOQ was detected at the retention time of deltamethrin in any control sample. The specificity and confirmation of the method has been determined by the quantification of two LC-MS/MS transitions (m/z 523 -> 281: m/z 525 -> 283).

#### Conclusion:

The method was successfully validated and is suitable for the determination of residues of deltamethrin in soil and sediment. The LOQ was 0.0001 mg/kg. Satisfactory validation data was also achieved for the

second transition demonstrating that any mass transition can be used for quantification and/or confirmation.

#### A 2.1.2.4 Description of Methods for the Analysis of Water (KCP 5.2)

No new or additional studies have been submitted

#### A 2.1.2.5 Description of Methods for the Analysis of Air (KCP 5.2)

No new or additional studies have been submitted

#### A 2.1.2.6 Description of Methods for the Analysis of Body Fluids and Tissues (KCP 5.2)

Comments of zRMS:	Method is accepted
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<b>Reference</b>	KCP 5.2/03
<b>Report:</b>	Method Validation - Determination of Residues of Deltamethrin in whole blood and liver, XXX, R. (2013b), report No YV/13/007
<b>Guidelines:</b>	SANCO/825/00 rev 8.1
<b>Deviations:</b>	No
<b>GLP:</b>	Yes

#### Materials and methods.

Residues of deltamethrin were extracted by vortexing with acetone, sonicating with ethyl acetate/dichloromethane (2:1) and sonicating with hexane. The extracts were transferred to a kiesulguhr filtration column and the eluent was collected. An aliquot was evaporated, reconstituted in water/methanol (3:7) containing 0.01M ammonium acetate and 5 ng/mL internal standard and analysed by LC-MS/MS.

#### LC-MS/MS Analysis parameters:

<b>Liquid Chromatography</b>	
Instrument:	Agilent 1100 HPLC System
Column:	Phenomenex Luna 5µ C18(2) 100A 50 mm × 2 mm (00B-4252-B0)
Column Temperature:	40 °C
Mobile Phase A:	Water/methanol (9:1) containing 0.01M ammonium acetate
Mobile Phase B:	Methanol containing 0.01M ammonium acetate
<b>Mass Spectrometry</b>	
Instrument:	API 5000 LC-MS/MS, AB Sciex
Ion Source:	Electrospray, (ESI, Turbolon Spray)
Polarity:	Positive

#### Validation data:

##### Linearity

Seven calibration standards between 0.25 to 10 ng/mL were injected, covering the required range from 30% of the LOQ to 20% above the highest fortification level. Calibration curves with correlation co-

efficients  $r \geq 0.999$  were achieved for both transitions, demonstrating acceptable linearity. Calibration standards were prepared in solvent but using an internal standard to compensate matrix effect.

#### Accuracy & Precision

Average recoveries at each fortification level were all within the acceptance range of 70-120 % (for both quantitation and confirmatory transitions). The relative standard deviation (RSD) did not exceed 20 % at any fortification level and any interference was below 30 % of the LOQ (for both quantitation and confirmatory transitions).

#### Deltamethrin in Whole Blood

Transition <i>m/z</i>	Fortification Level (mg/L)	Individual Recovery Results (%)	n	Mean Recovery (%)	RSD (%)
523 – 281	0.05	80, 80, 81, 78, 84	5	81	2.7
525 - 283	0.05	81, 83, 80, 78, 83	5	81	2.6

#### Deltamethrin in Liver

Transition <i>m/z</i>	Fortification Level (mg/kg)	Individual Recovery Results (%)	n	Mean Recovery (%)	RSD (%)
523 – 281	0.1	84, 84, 88, 87, 80	5	85	3.7
525 - 283	0.1	83, 85, 80, 82, 85	5	83	2.6

#### Specificity

No interference/contamination peak above 30% of the LOQ was detected at the retention time of deltamethrin in any control sample. The specificity and confirmation of the method has been determined by the quantification of two LC-MS/MS transitions (*m/z* 523 -> 281: *m/z* 525 -> 283).

#### **Conclusion:**

The method was successfully validated and is suitable for the determination of residues of deltamethrin in whole blood and liver. The LOQ of the method was validated at 0.05 mg/L for deltamethrin in whole blood and 0.1 mg/kg for deltamethrin in liver during the validation study. Satisfactory validation data was also achieved for the second transition demonstrating that any mass transition can be used for quantification and/or confirmation.

#### **A 2.1.2.7      A.2.A.9      Other Studies/ Information**

No new or additional studies have been submitted