1 Summary

The present method validation was performed for the determination of deltamethrin in soil and sediment by means of HPLC-MS/MS detection. This method is able to quantify cis-deltamethrin as well as its isomers trans-deltamethrin and alpha-R-deltamethrin. The method was optimised and validated using cis-deltamethrin.

Soil and sediment samples of 20 g are extracted in a microwave extractor with 40 mL of a mixture of acetonitrile/ammonium acetate 10 mMol/L in water (900/100, v/v). After extraction, portions of the samples are centrifuged to remove fine particles of the soil. Identification and quantitation of the test item is done by high performance liquid chromatography using MS/MS detection in the Multiple Reaction Monitoring mode. Possible matrix effects of deltamethrin are eliminated by using an internal standard solution of the isotopically labelled analytical standard. This solution is added to the sample solutions after extraction.
The method was validated with two different soils (silt and sandy loam soil) as well as a sediment (silt loam).

The limit of quantitation of the method is 0.1 µg/kg for cis-deltamethrin.

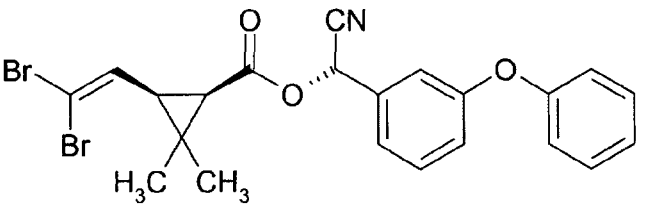
The limit of detection of the method is 0.03 µg/kg for cis-deltamethrin.

2 Introduction

The objective of the study was to validate a new residue analytical method for the determination of the total residue of deltamethrin in soil and sediment by means of HPLC-MS/MS. The total residue is defined as the sum of cis-deltamethrin (AE F032640), α-R-deltamethrin (AE F108569) and trans-deltamethrin (AE F0035073).
It is known that cis-deltamethrin can be transformed by chemical or biological processes to its diasteromeres α-R-deltamethrin and trans-deltamethrin. For ecotoxicological risk assessment purposes α-R- and trans-deltamethrin are assumed to have the same biological/pesticidal activity as cis-deltamethrin. This method is able to quantify cis-deltamethrin as well as its isomers trans-deltamethrin and alpha-R-deltamethrin. The method was optimised and validated using cis-deltamethrin.
The method was validated in accordance to the Guidance Document on Residue Analytical Methods (Ref. 1), the Commission Directive 96/46/EC (Ref. 3) and BBA Guideline for Residue Analytical Methods (Ref. 2).

3 Test and Reference Items

cis-deltamethrin:

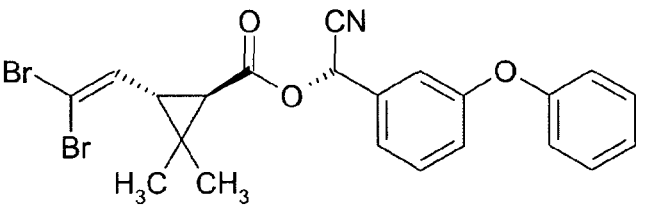


Common name:	Deltamethrin	
Code name:	cis-deltamethrin	
Chemical code:	AE F032640	
Chemical name (IUPAC):	(S)–α-cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate	
Chemical name (CA):	[1R-[1α(S*),3α]]-cyano(3-phenoxyphenyl)methyl3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate	
CAS No.:	52918-63-5	
Molecular formula:	$C_{22}H_{19}Br_2NO_3$	
Molecular weight:	505.2 g/mol	
Solubility in water:	<5 µg/L (20 °C)	
Vapour Pressure:	1.24 x 10^{-8} Pa (25 °C)	
Octanol-water partition coefficient log Pow:	4.6 (25 °C)	
Hydrolytic stability:	pH 5 & 7:	$t_{1/2}$ >30 days (25 °C)
	pH 9:	$t_{1/2}$ 2.5 days (25 °C)

Reference standard:

Certificate of analysis:	AZ 10090
Batch ID:	97B0276B3
Purity:	99.6%
Expiry date:	July 2007
Origin:	Bayer CropScience GmbH, PT – Analytics Frankfurt, D-65926 Frankfurt am Main, Germany

trans-deltamethrin:

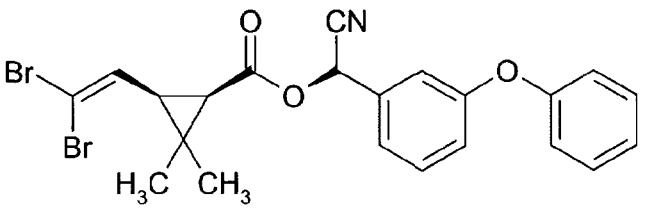


Code name:	trans-deltamethrin
Chemical code:	AE 0035073
Chemical name (IUPAC):	(S)–α-cyano-3-phenoxybenzyl (1R,3S)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate
Chemical name (CA):	[1R-[1α(S*),3β]]-cyano(3-phenoxyphenyl)methyl3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate
CAS No.:	64363-96-8
Molecular formula:	$C_{22}H_{19}Br_2NO_3$
Molecular weight:	505.2 g/mol

Reference standard:

Certificate of analysis:	AZ 09021
Batch ID:	5E0551
Purity:	94.0%
Expiry date:	March 2005
Origin:	Bayer CropScience GmbH, PT – Analytics Frankfurt, D-65926 Frankfurt am Main, Germany

alpha-R-deltamethrin:

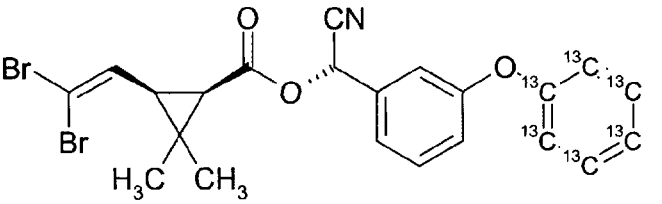


Code name:	alpha-R-deltamethrin
Chemical code:	AE F108569
Chemical name (IUPAC):	(R)–α-cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate

Chemical name (CA): [1R-[1α(R*),3α]]-cyano(3-phenoxyphenyl)methyl3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate
CAS No.: 55700-99-7
Molecular formula: $C_{22}H_{19}Br_2NO_3$
Molecular weight: 505.2 g/mol

Reference standard:
- Certificate of analysis: AZ 11194
- Batch ID: SEL/1322
- Purity: 93.1%
- Expiry date: Nov. 2004
- Origin: Bayer CropScience GmbH, PT – Analytics Frankfurt, D-65926 Frankfurt am Main, Germany

cis-deltamethrin-^{13}C6:
cis-deltamethrin-^{13}C6 is used as internal standard.



Code name: [phenoxy-^{13}C6]deltamethrin
Chemical name (IUPAC): (S)–cyano(3-phenoxyphenyl)methyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate
Empirical formula: $C_{22}H_{19}Br_2NO_3$
Molecular weight: 511.14 g/mol

Reference standard:
- Batch ID: BECH 1068-1-1
- Chemical purity: >99%
- Isotope enrichment: total ^{13}C: 99.6%
- Expiry date: not given
- Origin: Bayer CropScience AG, PT – Isotope Chemistry, D-42096 Wuppertal, Germany

4 Test System

The method was validated using the two German soils *Höfchen* and *Laacher Hof* and the German sediment *Nesten*. Two different soils and the sediment were used in order to assess a possible influence of different soil properties. The soil and sediment samples were classified according to DIN and/or USDA specifications. Soil characteristics are summarised in Table 1.
Complete soil and sediment parameterisation is reported in the Appendix (Table 7 to Table 9).

Table 1: Soil Types

Designation	Soil type (Texture)	Organic matter [%]
Höfchen	silt loam (USDA)	1.6
Laacher Hof	sandy loam (USDA)	2.1
Sediment	silt loam (USDA)	7.2

5 Instruments

Microwave Extractor: MLS-Ethos
MWS Vertriebs GmbH
88299 Leutkirch, Germany

Balance: PC 4400, PM 4800 and AT 261
Mettler Instruments GmbH
35387 Giessen, Germany

Ultrasonic bath: Transsonic 890/H
Heinrich Faust
51145 Cologne, Germany

Liquid chromatograph: HP 1100 Column Compartment G1316A
HP 1100 Binary Pump G1312A
HP 1100 Isocratic Pump G1310A
HP 1100 Degasser G1322A
Agilent
40880 Ratingen, Germany

Autosampler: HTC PAL System
CTC Analytics AG
4222 Zwingen, Switzerland

Column: Purospher STAR RP-18e
Li ChroCard
Size: 55 x 4 mm
Cat. No.: 1.50231
Merck Eurolab GmbH
64293 Darmstadt, Germany

Mass spectrometer:	API 4000 with turbo-ionspray interface mass selective detector (MS/MS) Perkin Elmer Sciex Instruments 64331 Weiterstadt, Germany

6 Reagents and Equipment

Magnetic stirring bar:	plain (large, e.g. 35 x 8 mm [length x i.d.]) or "dumb-bell" type (e.g. 35 x 8 mm [length x i.d.], diameter of end disk is 20 mm, from COWIE Technology, parts no. 1.1335)
Acetonitrile:	for HPLC, super gradient grade Riedel de Haen, No. 34998 30926 Seelze, Germany
Methanol:	for HPLC Promochem GmbH 46469 Wesel, Germany
Toluene	for analysis Merck, No.1.08325.2500 64271 Darmstadt, Germany
Ammonium acetate	extra pure Merck, No.1.01115.1000 64271 Darmstadt, Germany
Water:	purified in a Milli-Q unit Milli-Pore GmbH 65731 Eschborn, Germany
Solvent I:	acetonitrile/ammonium acetate 10 mMol/L in water(900/100; v/v)

Volumetric flasks, pipettes and other instruments commonly used in the laboratory.

7 Standard Solutions

The following Sections 7.1 to Section 7.3 describe general working procedures for the preparation of standard solutions. Therefore, given names, weights and volumes do not correspond exactly to the used names, weights, volumes and concentrations documented in the raw data.

7.1 Standard Stock Solutions

(STMDEL) 500 mg/L stock solution of cis-deltamethrin:
Weigh approximately 10.0 mg* cis-deltamethrin into a 20-mL volumetric flask. Dilute to volume with toluene.

(STM13C) 1000 mg/L stock solution of [phenoxy-^{13}C6]deltamethrin:
Weigh approximately 10.0 mg* [phenoxy-^{13}C6]deltamethrin into a 10-mL volumetric flask. Dilute to volume with toluene.

*: 0.01 mg characterises the precision of the balance, not the precision of the weighed reference substance

Before further use, the standard stock solution has to be ultrasonicated for about one minute to achieve complete dissolution of the test substance.

7.2 Working Standard Solutions

The working standard solutions are used for the preparation of the calibration standard solutions and for sample fortification.

(1 LSGDEL) 1000 µg/L solution of cis-deltamethrin:
Pipette 0.2 mL of (STMDEL) into a 100-mL volumetric flask and dilute to volume with Solvent I.

(2 LSG13C) 1000 µg/L solution of [phenoxy-^{13}C6]deltamethrin:
Pipette 0.1 mL of (STM13C) into a 100-mL volumetric flask and dilute to volume with Solvent I.

(3 LSGDEL) 100 µg/L solution of cis-deltamethrin:
Pipette 10 mL of (1LSGDEL) into a 100-mL volumetric flask and dilute to volume with Solvent I.

(4 LSG13C) 2 µg/L solution of [phenoxy-^{13}C6]deltamethrin:
Pipette 0.5 mL of (2LSG13C) into a 250-mL volumetric flask and dilute to volume with Solvent I.

(5 LSGDEL) 10 µg/L solution of cis-deltamethrin:
Pipette 1 mL of (1LSGDEL) into a 100-mL volumetric flask and dilute to volume with Solvent I.

7.3 Solvent Standard Solutions

(1 MIXDEL) mixed solution of 5 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]-deltamethrin:
Pipette 0.25 mL of (1 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

(2 MIXDEL) mixed solution of 2.5 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]-deltamethrin:
Pipette 0.125 mL of (1 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

(3 MIXDEL) mixed solution of 0.5 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]-deltamethrin:
Pipette 0.25 mL of (3 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

(4 MIXDEL) mixed solution of 0.2 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]-deltamethrin:
Pipette 0.1 mL of (3 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

(5 MIXDEL) mixed solution of 0.1 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]-deltamethrin:
Pipette 0.05 mL of (3 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

(6 MIXDEL) mixed solution of 0.08 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]deltamethrin:
Pipette 0.4 mL of (5 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

(7 MIXDEL) mixed solution of 0.05 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]deltamethrin:
Pipette 0.25 mL of (5 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

(8 MIXDEL) mixed solution of 0.015 µg/L cis-deltamethrin and 2 µg/L of [phenoxy-^{13}C6]deltamethrin:
Pipette 0.075 mL of (5 LSGDEL) and 0.1 ml of (2LSG13C) into a 50-mL volumetric flask and dilute to volume with Solvent I.

All standard solutions need to be stored in a refrigerator.

8 Safety Precautions

The German guidelines for laboratories issued by the Trade Co-operative Association (e.g. Bulletin M006) or comparable guidelines in other countries must be considered when working according to this method.
The following solvents and chemicals are used which are classified according to the Hazardous Substances Regulations:

cis-deltamethrin	toxic T and irritant Xi (R-phrases 21, 23/25, 36/38)
alpha-R-deltamethrin	toxic T *)
trans-deltamethrin	toxic T *)
[phenoxy-^{13}C6]-deltamethrin	toxic T *)
Ammonium acetate	
Acetonitrile	harmful Xn, highly flammable F
Toluene	harmful Xn, highly flammable F
Methanol	toxic T, highly flammable F

*) A classification is not yet available. Due to this fact the compound has to be treated as very toxic substance.

This classification is based on German guidelines and has to be adapted according to the respective national guidelines in case the method is used outside of Germany. While working with these substances, the relevant safety regulations are to be considered (see R- and S-rules).

9 Performance of Analysis

Within this method validation it was demonstrated that cis-deltamethrin and its isomers trans-deltamethrin and alpha-R-deltamethirn show the same selectivity for MS/MS detection (see Figure 2 to Figure 6). This will allow to minimize time for chromatographic separation and to elute the isomer mixture from the separation column as a single peak. Since the current method was validated using MS/MS parameters which are optimised for cis-deltamethrin these settings may not be optimum for quantitation of the isomers alpha-R-deltamethrin and trans-deltamethrin. This has to be considered when quantifying deltamethrin residues in unknown samples. The current method validation was performed using control material fortified with cis-deltamethrin only.

9.1 Fortification

The method was validated by analysing control samples and control samples fortified with cis-deltamethrin at and above the limit of quantitation.
Sample fortification was done by adding a certain amount of a fortification standard solution to 20 g of soil or sediment. After a waiting time of one hour between fortification and begining of the extraction, to allow for the standard to be soaked into the soil, the samples were extracted according to the procedure described in section 9.2.

The following fortification levels were analysed:

0.1 µg/kg:	addition of 200 µL of (5 LSGDEL) to 20 g of soil or sediment
1 µg/kg:	addition of 200 µL of (3 LSGDEL) to 20 g of soil or sediment

The preparation of the fortification standards is described in section 7.2.

9.2 Extraction

1. Weigh 20 g of the soil or sediment sample into a 100-mL beaker containing a magnetic stirrring bar. (Large stirring bar necessary to ensure soil/solvent mixture is completely mixed during extraction.)
2. Add 40 mL of a mixture of acetonitrile/ammonium acetate 10 mMol/L in water (9/1 v/v).
3. Place ten beakers with soil/sediment and solvent mixture into the microwave extractor.
4. Switch on the magnetic stirrer.

5. Extract for three minutes at 250 W.
6. After extraction, add 80 µL of internal standard solution (2 LSG13C) into the sample and stir for one minute.
7. Transfer about 1.5 mL of the extract into a centrifuge tube.
8. Centrifuge for 3 minutes at >12000 g to remove fine particles of the soil.
9. Transfer a portion of the sample solution into an HPLC vial.
10. Inject an aliquot of 50 µL into the HPLC-MS/MS system.

A flow diagram of the analytical procedure is given in Figure 1.

9.3 Liquid Chromatographic Conditions

Column: LiChroCART 55-4
Purospher STAR RP-18e, 3 µm, length 5.5 cm, i.d. 4.0 mm

Injection volume: 50 µL
Oven temperature: 40 °C
Mobile phases: A: water with ammonium acetate 10 mMol/L / methanol (9/1 v/v)
B: methanol with ammonium acetate 10 mMol/L

Run time: 4 min
Flow rate (column): 1.0 mL/min
Flow rate (interface): 1.0 mL/min

Isocratic Pump (for flushing the interface):
Mobile phase: water with ammonium acetate 10 mMol/L / methanol (1/1 v/v)

Retention time:

cis-deltamethrin (AE F032640):	approx.1.2 min
alpha-R-deltamethrin (AE F108569) :	approx.1.2 min
trans-deltamethrin (AE F0035073) :	approx.1.2 min
[phenoxy-^{13}C6]deltamethrin:	approx.1.2 min

Table 2: HPLC Time Table

Time [min]	Setting
0.10	switching eluent stream to waste
0.50	switching eluent stream to interface
3.00	switching eluent stream to waste

Remark: For the time, the eluent stream of the binary pump is switched to the interface, the eluent stream of the isocratic pump is switched to waste and vice versa.

9.4 Mass Spectrometry - Principle of Measurement

Substances introduced into the mass spectrometer are ionised using a turbo-ionspray interface. Sample ions are accelerated by an adequate voltage regulation and separated by mass in the first quadrupole (Q1). The most abundant ions (the protonated and deprotonated ions) of the analyte (parent ions) are impulsed with nitrogen in the collision cell (Q2). Fragments of these ions (daughter ions) are separated by mass in the third quadrupole (Q3) and detected. The mass spectrometric parameters for the analytes and the selected ions are listed in Table 3.

9.5 Mass Spectrometric Parameters

The reported parameters are examples for an optimum adjustment of the mass spectrometer for cis-deltamethrin. With these parameters, the results in sections 11 to 15 were obtained. From time to time these parameters have to be checked and adjusted if necessary.

Table 3: MS/MS Operating Parameters

	cis-deltamethrin	[phenoxy-^{13}C6]deltamethrin
Q1 Mass [amu]	523 *	529 *
Q3 Mass [amu]	281	281
Dwell [msec]	500	500
Ionisation Mode	ESP+	ESP+
IS [V]	5500	5500
EP [V]	10	10
DP [V]	51	51
CE [V]	21	21
CXP [V]	12	12
Turbo Gas	50	50
Curtain Gas	50	50
Nebuliser Gas	50	50
Collision Gas [L/min]	0.92	0.92
Turbo gas Temp. [°C]	500	500

* masses m/z 523 and m/z 529 correspond to $[M+2+NH_4]^+$

IS: Ion Spray Voltage EP: Entrance Potential DP: Declustering Potential
CE: Collision Energy CXP: Collision Cell Exit Potential
ESP+: positive ion mode, i.e. production of positive ions

The mass spectra of the parent and the product ions of cis-deltamethrin, trans-deltamethrin, alpha-R-deltamethrin and [phenoxy-^{13}C6]deltamethrin are presented in the Appendix (Figure 2 to Figure 6).

10 Calculations

For calculation of the concentrations, multi-point calibration curves were used. These curves were calculated automatically after each sequence run with the Perkin-Elmer quantitation software *Analyst (vers. 1.3)* using linear regression. Further calculations were performed using the software *EXCEL 97 (Office 97®)*.
Matrix effects for cis-deltamethrin are eliminated by using an internal standard solution of the isotopically labelled test item ([phenoxy-^{13}C6]deltamethrin). This solution will be added to the sample solutions after extraction.

The linear equation for calibration is expressed as:

$$y = \text{Intercept} + \text{Slope} \cdot x$$

y = Area, x = Concentration

When an internal standard is used:

$$y = \frac{\text{Area}_{\text{Standard}}}{\text{Area}_{\text{Internal Standard}}} = \text{Int.Ratio} \quad \text{and} \quad x = \frac{\text{Conc}_{\text{Standard}}}{\text{Conc}_{\text{IS}}} = \text{Conc}_{\text{ratio}}$$

Int.Ratio = Intensity ratio
$Conc_{Standard}$ = Concentration of standard solution [µg/L]
$Conc_{IS}$ = Concentration of internal standard solution [µg/L]
$Conc_{ratio}$ = Concentration ratio

If the concentration of the isotopically labeled internal standard is the same in all solutions that are injected into the HPLC instrument, it has not to be taken into consideration. However, the concentration of the internal standard solution should be in the range of the concentration of the sample solutions.

By means of the linear equation, the content of deltamethrin in dry soil can be calculated as follows:

$$\text{Dilution}_{\text{Factor}} = \frac{\text{Volume}_{\text{final}}}{\text{Weight}}$$

$$\text{Conc}_{\text{Analyte}} = \frac{\text{Int.Ratio} - \text{Intercept}}{\text{Slope}}, \quad \text{Int.Ratio} = \frac{\text{Area}_{\text{Analyte}}}{\text{Area}_{\text{Internal Standard}}}$$

$$\text{Conc}_{\text{Soil Wet}} = \text{Conc}_{\text{Analyte}} \cdot \text{Dilution}_{\text{Factor}}$$

$$\text{Conc}_{\text{Soil Dry}} = \text{Conc}_{\text{Soil Wet}} \cdot \frac{100\%}{100\% - \text{Moisture}}$$

$Volume_{final}$ = Volume of the extraction solution added [L]
Weight = Weight of the soil sample [kg]
$Conc_{Analyte}$ = Concentration of the analyte in the sample solution [µg/L]
$Conc_{Soil\ Wet}$ = Concentration of the analyte in wet soil [µg/kg]
$Conc_{Soil\ Dry}$ = Concentration of the analyte in dry soil [µg/kg]
Moisture = Moisture (water content) of the sample [%]
$Area_{Analyte}$: mean area of the analyte in the sample solution
$Area_{Internal\ Standard}$: mean area of the internal standard in the sample solution

The recovery is calculated according to the following equation:

$$\text{Recovery} = \frac{\text{Conc}_{\text{Soil Wet}} \cdot 100\%}{\text{Conc}_{\text{Soil Spiked}}}$$

$Conc_{Soil\ Spiked}$ = *Concentration of the reference item spiked [µg/kg]*

Example calculation for recovery of cis-deltamethrin in soil Höfchen, 1.0 µg/kg (recovery 7, file: 040601a9_us.wiff, sample ID: 49)

$$\text{Dilution}_{\text{Factor}} = \frac{0.04\,L}{0.02\,kg} = 2.0\,L/kg \qquad \text{Dilution}_{\text{Factor}} = \frac{\text{Volume}_{\text{final}}}{\text{Weight}}$$

$$\text{Conc}_{\text{Analyte}} = \frac{0{,}27338 - 0.00367069}{0.513769} = 0{,}52496\,\mu g/L \qquad \text{Conc}_{\text{Analyte}} = \frac{\text{Int.Ratio} - \text{Intercept}}{\text{Slope}}$$

$$\text{Conc}_{\text{Soil Wet}} = 0{,}52496\,\mu g/L \cdot 2.0\,L/kg = 1{,}0499\,\mu g/kg \qquad \text{Conc}_{\text{Soil Wet}} = \text{Conc}_{\text{Analyte}} \cdot \text{Dilution}_{\text{Factor}}$$

$$\text{Recovery} = \frac{1{,}0499\,\frac{\mu g}{kg} \cdot 100\%}{1.0\,\frac{\mu g}{kg}} = 105\% \qquad \text{Recovery} = \frac{\text{Conc}_{\text{Soil Wet}} \cdot 100\%}{\text{Conc}_{\text{Soil Spiked}}}$$

Remark: Calculations were performed using the computer software *Analyst (vers. 1.3)* and *EXCEL 97*. The results given are rounded values. Thus, rounding deviations may occur if recalculations are made using the rounded figures.

11 Detector Linearity

Standard solutions containing cis-deltamethrin, were measured in a concentration range of about 0.015 to 5 µg/L corresponding to a concentration in soil of 0.03 - 10 µg/kg. In this concentration range, the mass spectrometric detector showed linear correlation between concentration and peak area ratio (area ratio = analyte area / internal standard area) (Table 4, 1/x weighted linear regression).
The graphical presentation of the linearity curve is included in the Appendix (Figure 7).

Table 4: Correlation Between Concentration and Area Ratio

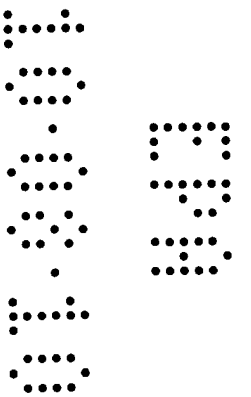
Compound	Concentration range [µg/L]	Correlation coefficient r
cis-deltamethrin	0.015 – 5	0.9999

17 References

Ref. 1 EC Guidance Document on Residue Analytical Methods, SANCO/825/00 rev.7 of March 17, 2004

Ref. 2 BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998

Ref. 3 Commission Directive 96/46/EC amending Council Directive 91/414/EEC of July 16, 1996



18 Appendices

Figure 1: Flow Diagram of the Extraction Procedure