

FINAL REGISTRATION REPORT

Part B

Section 5

Analytical Methods

Detailed summary of the risk assessment

Product code: SAP09I

Product name: VOLKETE MAX

Chemical active substance:

Spinosad, 480 g/L

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT

(New product authorization according to article 34 of Regulation (EC) 1107/2009)

Applicant(s): Albaugh TKI d.o.o.

Submission date: January 2025

MS Finalisation date: 12/2025

Version history

When	What
January 2025	V0 – Original dRR version from applicant Albaugh TKI d.o.o. for submission to zRMS Poland in the frame of a new authorization according to Article 33/34 of Regulation (EC) No. 1107/2009.
April 2025	Dossier sent for evaluation
October 2025	zRMS evaluation of dRR
December 2025	Final RR

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zRMS comments:

The text highlighted in grey was provided by the zRMS.

5 Analytical methods

5.1 Conclusion and summary of assessment

Methods for residues were provided in the dossier for the authorization of the product SPINTOR 480 SC and they are valid for the product SAP09I.

According to the EFSA Journal 2012;10(3):2630: “During the peer review under Directive 91/414/EC, an analytical method using HPLC-UV (DAD) and its ILV were evaluated and validated for the determination of spinosad (sum of spinosyn A and spinosyn D) in plant matrices with for each component a LOQ of 0.01 mg/kg in high water content (tomatoes, apples, peppers) and acidic (strawberries, grapes) commodities (The Netherlands, 2004);the combined LOQ is 0.02 mg/kg. Additionally, analytical methods using HPLC-UV or MS were evaluated and validated in JMPR report for the determination of the spinosad (sum of spinosyn A and spinosyn D) in plant matrices with for each component a LOQ of 0.01 mg/kg in high water content, acidic, dry and high oil content commodities (FAO, 2001); the combined LOQ is 0.02 mg/kg.

The multi-residue QuEChERS method using HPLC-MS/MS described in the European Standard EN15662:2008 validated with a LOQ of 0.01 mg/kg for the determination of each component in highwater content, dry and acidic commodities is also applicable (combined LOQ of 0.02 mg/kg).

Hence, according to the overall availability of methods, it is concluded that spinosad (sum of spinosyn A and spinosyn D) can be enforced in food of plant origin with a LOQ of 0.01 mg/kg for each substance in high water content, high fat content, acidic and dry commodities (combined LOQ of 0.02 mg/kg).

According to the PROFile and the JMPR report (FAO, 2001), an analytical method using HPLC-UV or MS was validated and can be enforced for the determination of spinosad (sum of spinosyn A and spinosyn D) in food of animal origin (milk, liver, kidney, fat, muscle and eggs) with a LOQ of 0.01mg/kg for each component (combined LOQ of 0.02 mg/kg).”

Sufficiently sensitive and selective analytical methods are **not** 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 **not** available for all analytes included in the residue definitions.

Please refer to the reference product Dossier of SPINTOR 480 SC.

Commodity/crop	Supported/ Not supported
Head cabbage	Supported
Broccoli, cauliflower	Supported
Onion, garlic, shallot	Supported
Leek	Supported
Apple, pear, crab apple, quince	Supported

Please refer to the reference product Dossier of SPINTOR 480 SC in Spain (Registration no. 22839).

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 Spinosad in plant protection product is provided as follows:

Comments of zRMS:	Accepted
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Reference: KCP 5.1.1/01

Report Spinosad 480 g/L SC (SAP09I): Physical, chemical and technical of the plant protection product, Morais, F., 2021, Report No. EF/359/21 – Interim Report Annex 1

Guideline(s): Yes, SANCO 3030/99, rev. 5 (22/03/2019)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods

The sample is dissolved in methanol, the solution is placed in an ultrasonic bath and filtered. Spinosad content is determined by HPLC using DAD detection. Spinosad was identified using a LC-MS method.

Chromatographic conditions – HPLC-DAD for active substance quantification

Mobile phase:	40 % Acetonitrile + 40 % Methanol + 20 % Buffer solution
Run time:	20 minutes
Flow:	1.500 mL/min
Column:	YMC-Pack ODS-AQ; 150 mm x 4.6 mm; 5µm
Column temperature:	35.0° C
Detection wavelength:	250 nm
Injection volume:	20.0 µL
Retention time:	Around 11.2 minutes (Spinosad A) Around 14.2 minutes (Spinosad D)

MS Conditions for active substance identification

Mobile phase:	80 % Acetonitrile + 20 % Water with 0.1 % ammonium acetate
Run time:	5 minutes
Flow:	0.300 mL/min
Column:	Acquity UPLC BEH; 50 mm x 2.1 mm; 1.7µm
Detection wavelength:	190 - 400 nm
MS type:	MS2 Scan
MS ion mode:	ES ⁺
Data Format:	Centroid
Mass range:	50 m/z – 900 m/z
Cone voltage:	60 V

Retention time:	Around 1.9 minutes (Spinosad A) Around 2.4 minutes (Spinosad D)
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Buffer solution preparation

Weigh approximately 20 g of ammonium acetate in 100mL of ultrapure water. Adjust pH of this solution at 5.3 with acetic acid.

Standard solution preparation

Weigh approximately 20 mg $\pm$ 2.0 mg of spinosad reference material into a 10 mL volumetric flask. Dissolve and complete the volume with methanol (2.0 mg/mL). From this solution transfer 0.875 mL into a 5 mL volumetric flask and complete the volume with methanol (0.35 mg/mL). Prepare in duplicate.

Sample solution preparation

Weigh approximately 81 mg $\pm$ 8.1 mg of sample into a 100 mL volumetric flask. Dissolve and complete the volume with methanol. Place the solution in an ultra-sonic bath, filter using a 0.20 μ m disk filter (0.81 mg/mL). Prepare in duplicate.

Validation - Results and discussions

Table 5.2-1: Methods suitable for the determination of active substances Spinosad in plant protection product SAP09I

	Spinosad
Author(s), year	Morais, F. (2021)
Principle of method	HPLC-DAD/MS
Linearity (linear between mg/L / % range of the declared content) (correlation coefficient, expressed as r) n=5	Range: [13.03 % - 130.86 %], [0.1055 mg/mL – 1.0600 mg/mL] r = 1.0000 (y = 10204.5745x – 111.5394)
Precision – Repeatability Mean n = 5 (%RSD)	Instrumental Repeatability: RSD = 0.20 % Hr = 0.14 Method Repeatability: RSD = 0.32 % Hr = 0.20 (expected content: 35.23 % of spinosyn A) Method Repeatability: RSD = 0.57 % Hr = 0.29 (expected content: 8.84 % of spinosyn D) Method Repeatability: RSD = 0.34 % Hr = 0.23 (expected content: 48 % of spinosad)
Accuracy n = 5 (% Recovery)	1 st level (24.69 %): 102.63 % (RSD = 0.26 %) (RSD criterion < 1.65) 2 nd level (43.21 %): 102.48 % (RSD = 0.32 %) (RSD criterion < 1.52)
Interference/ Specificity	Mass spectra and retention time were obtained from standard and sample and confirm compound identity. Specific method. Positive identity with more than three ions. There are no interfering peaks (Injection of blank, standard (A+D), sample and blank formulation solution). Spinosyn A and spinosyn D was also injected.
Comment	-

Conclusion

The analytical methods for the determination of active substance Spinosad in the plant protection product SAP09I has been described and validated according with SANCO/3030/99 rev. 5. The method accomplishes with all parameters.

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

According to commission implementing regulation (EU) o 540/2011 of 25 May 2011, there are no relevant impurities.

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

5.2.1.3 Description of analytical methods for the determination of formulants (KCP 5.1.1)

There are no formulants or constituents of formulants within the preparation or formed during storage, that are of toxicological, ecotoxicological or environmental relevance. Therefore, this point is not relevant.

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

5.2.1.4 Applicability of existing CIPAC methods (KCP 5.1.1)

CIPAC method 636 is available for the determination of Spinosad in suspension concentrate formulation described in CIPAC HANDBOOK L page 121.

Outline of the method:

Spinosad is dissolved in methanol and determined by high-pressure liquid chromatography using an octadecylsilyl column, ultraviolet detection at 250 nm and external standardisation.

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

5.2.2 Methods for the determination of residues (KCP 5.1.2)

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

5.3.1 Analysis of the plant protection product (KCP 5.2)

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

5.3.2.8 Other studies/ information

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

Appendix 1 Lists of data considered in support of the evaluation

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	Morais, F.	2021	Spinosad 480 g/L SC (SAP09I): Physical, chemical and technical properties of the plant protection product Study EF/359/21 - Interim report ASCENZA Agro, S.A. Physical-chemical laboratory GLP Unpublished	N	Albaugh Europe Sàrl Ascenza Agro SA Lainco SA

Appendix 2 Detailed evaluation of submitted analytical methods

A 2.1 Analytical methods

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

See 5.2.1 above – full summary of the method validation is provided, see submitted report KCP 5.1.1.

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)

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).

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

Please refer to the reference product Dossier of SPINTOR 480 SC in Poland (Registration No. R-47/2018wu of 19 October 2018).