European Commission

Draft Renewal Assessment Report prepared according to the Commission Regulation (EU) N° 1107/2009

BLOOD MEAL

Volume 3 – B.5 (AS)

Rapporteur Member State: Austria
Co-Rapporteur Member State: Lithuania

Version History

When	What
2018/02	Original dossier submission by applicant
2018/04	Revised dossier submission by applicant
2018/12	Draft RAR by RMS AT
2019/02	Draft RAR by RMS AT after commenting by Co-RMS LT

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B.5. METHODS OF ANALYSIS

Blood meal was included in the Annex I of Directive 91/414 under Inclusion Directive 2008/127/EC RMS for assessment of Blood meal was Belgium. The Regulation (EU) No 1107/2009 repealed and replaced the Directive 91/414/EEC and the active substance Blood meal is deemed to be approved under that Regulation and included in the Annex to Regulation (EC) No 540/2011 amended by Commission Implementing Regulation (EU) No 369/2012 and Commission Implementing Regulation (EU) 2017/195.

Blood meal was included in Annex I under provision as use in game repellent. The SANCO report for Blood meal (SANCO/2604/08 - rev 1-4 dated 11th July 2014) and Peer review document EFSA 2011 (EFSA Journal 2011;9(10):2394) are considered to provide the relevant information for the re-registration of Blood meal. The reference product Certosan contains 99.8% blood meal and can therefore considered being identical with the active ingredient. Data obtained with the product can be used also for the active substance Blood meal.

B.5.1. METHODS USED FOR THE GENERATION OF PRE-AUTHORISATION DATA (CA 4.1)

B.5.1.1. Methods for the analysis of the active substance as manufactured (CA 4.1.1)

Reference:	**Determination of the solubility in water of Blood Meal (Certosan) according to OECD 105 resp. EU A.6**
Author(s), year:	Affolter, O. (2013)
Report/Doc. number:	13022801G910
Guideline(s):	OECD Guidelines for the testing of Chemicals, method No.105; Method A6: "Water solubility"
GLP:	Yes

Principle of the method:

The concentration of Blood meal (Certosan) was determined through a mehod using ICP-OES via measurement of Iron the filtratered test solutions.

Material and methods:

Test material:

Technical batch of Blood meal (Certosan)

Analyte:

Blood Meal (Certosan), batch no. 94015FO/1, WP 99.8% blood meal, [redacted]

Analytical Instrument:

ICP-OES 720, Agilent; Software Agilent ICP Expert II Version 2.0.4.280

Method Parameters:

Wavelength Fe: 238.204 nm

TOC Analyser, multi N/C 2100S, Analytic Jena AG

Findings:

- Specificity: The specificity of the method was demonstrated by comparison of the retention time with the relevant reference item and by analysis of a reagent blank. No interferences were detected.
- Calibration (Linearity): The linearity was determined from 2 injections of six levels of standard covering the validation range relevant to the concentration of the active substance. The calibration curve was linear within the measured range of 1-50 µg/L Fe, equivalent to a Blood meal (Certosan) concentration of 0.05 to 100% (w/w).
- Accuracy (Recovery): This method was considered to have satisfactory accuracy from the results of specificity, linearity and precision.
- Precision (Repeatability): The relative standard deviation was calculated from 6 seperate determinations of the content of Blood meal (Certosan). The method is suitably precise with mean relative standard deviations inside the recommended values (modified Horwitz value RSDr). No outlier was discarded.
- Confirmation of identification: Analyte confirmation was shown via ICP-OES chromatograms of the test substance compared to analytical reference standards.

Conclusion:

The method is acceptable and allows the determination of the active substance Blood meal (Certosan) in the technical material. The limit of quantification of the method (LOQ) 1 µg/L for Iron was detrmined.

Table 5.1.1-1: Validation data summary for the determination of Blood meal (Certosan) in the technical material

References	Method	Analyte	Specificity / interferences	Calibration	Precision
Affolter, O.. (2013)	ICP-OES	Fe	No interferences	n = 6 0.05 – 100 %w/w R > 0.9996	n = 6 RSD 3.51 %
				n = 6 0.05 – 100 %w/w R > 0.9995	n = 6 RSD 3.56 %

Applicability of existing CIPAC methods

No CIPAC method is available for the determination of Blood meal (Certosan) in technical material.

B.5.1.2. Methods for risk assessment (CA 4.1.2)

No methods are required for soil, water and air since blood meal is a natural non-toxic compound.
There are clear indications that it may be expected that blood meal does not have any harmful effects on human or animal health or on groundwater or any unacceptable influence on the environment (SANCO/2604/08 – rev 4, 11 July 2014)

B.5.1.2.1. *Residue Data*

No new studies were submitted/evaluated for the purpose of renewal.

No methods required, since no MRL is proposed.

B.5.1.2.2. *Toxicology and Metabolism Data*

Since no systemic effects were observed in the toxicity studies and the test item was considered to be stable, no analytical work was performed. The evaluations of the results were based on nominal concentrations of the test item Blood meal (Certosan).

B.5.1.2.3. *Environmental Fate and Behaviour*

No new studies were submitted/evaluated for the purpose of renewal
Since no systemic effects were observed in the toxicity studies and the test item was considered to be stable, no analytical work was performed. The evaluations of the results were based on nominal concentrations of the test item Blood meal (Certosan).

B.5.1.2.4. *Ecotoxicological Data*

No new studies were submitted/evaluated for the purpose of renewal
Since no systemic effects were observed in the toxicity studies and the test item was considered to be stable, no analytical work was performed. The evaluations of the results were based on nominal concentrations of the test item Blood meal (Certosan).

B.5.2. METHODS FOR POST-APPROVAL CONTROL AND MONITORING PURPOSES (CA 4.2)

B.5.2.1. Plant matrices

No data necessary. Blood meal is applied as a repellent in ornamentals and forestry. No data on the metabolism in plants is required. In addition blood meal is included in Annex IV of Reg. (EC) No 396/2005, making a definition of the residues and setting of MRLs unnecessary.

Thus no analytical methods are required.

B.5.2.2. Animal matrices

No data necessary. Blood meal is applied as a repellent in ornamentals and forestry. No data on the metabolism in plants is required. In addition blood meal is included in Annex IV of Reg. (EC) No 396/2005, making a definition of the residues and setting of MRLs unnecessary.

Thus no analytical methods are required.

B.5.2.3. Soil

According to the EFSA Scientific Report, EFSA Journal (2011); 9(10):2394, the residue definition for monitoring in soil Blood meal (Certosan) is a natural non-toxic compound.

Therefore no analytical method is required.

B.5.2.4. Water

According to the EFSA Scientific Report, EFSA Journal (2011); 9(10):2394, the residue definition for monitoring in water is Blood meal (Certosan). Data gaps need to be filled before a residue definition and need for a method can be concluded on.

Reference:	**Validation of an Analytical Method using ICP-OES for the detrmination of Blood Meal (Certosan) in the matrices demineralised water, algal test medium, Daphnia and Danio test medium**
Author(s), year:	Affolter, O. (2013c)
Report/Doc. number:	13022801G926
Guideline(s):	-
GLP:	Yes

Principle of the method:

An ICP-OES-Method for the determination of Iron in the test item solution was validated. Iron was measured at 238.204 nm.

Material and methods:

Materials

Blood Meal (Certosan), batch No. 94015FO/1, purity 99.8%, [redacted]

Iron standard ; 1000 mg/L, Merck, Titrisol, batch No. HC932314

Analytical Instrument ICP-OES

ICP-OES 720, Agilent; Software Agilent ICP Expert II Version 2.0.4.280

Method Parameters:

Wavelength Fe: 238.204 nm

TOC Analyser, multi N/C 2100S, Analytic Jena AG

Findings:

- Specificity: The specificity of the method was checked by twelvefold determination of the blank value in algal test medium, Danio and Daphnia test medium in the comparison with twelvefold determination of the solvent for calibration (demineralised water). Specificity is not given, as Iron was measured in all test media.
- Calibration (Linearity): The calibration was determined from six levels of standard covering the validation range relevant to the concentration of the active substance. The calibration curve was linear within the measured range of 1-50 µg/L Fe thrice measurements. Linearity was not determined, as a quadratic function was used.
- Accuracy (Recovery): For the recovery rates were used 1 µg/L Fe in algal and 20 µg/L Fe in Danio test medium, in comparison to 1 µg/L and 20 µg/L Fe in demineralised water.
- Precision (Repeatability): The relative standard deviation was calculated from 6 seperate determinations of the content of Blood meal (Certosan). The quadratic calibration function was used.
- Confirmation of identification: Analyte confirmation was shown via ICP-OES chromatograms of the test substance compared to analytical reference standards.

The calibration was performed in demineralised water in the range 1 – 50 µg/L Iron. The ICP OES-method was validated for the parameter linearity, accuracy, precision and limit of detection and quantification.

The calibration was performed in demineralised water using certified Iron stock solution (1000 mg/L). All validation conditions for the calibration range 1 – 50 µg/I Fe are fulfilled, if a quadratic function is used for the calculation of the Iron content in the test solution.

Recovery rates from Danio test medium for the concentration 20 µg/L Fe were determined as 99 %, indicating that no matrix effects were present. Accuracy is not given in algal and Daphnia test medium for the concentration 1 µg/L Fe, as the concentration of 1 µg/L is the LOQ of Iron and the measurements in this range are inexact. Therefore, in the ecotoxicological studies, the blank value should be subtracted from the values of the test solutions.

Determination of LOQ and LOD

The lowest concentration of the calibration (1 µg/L Fe) was stated as limit of determination and quantification.

No observations arousing doubts concerning the accuracy of the results and the validity were made.

Table 5.2.4-1: Validation data summary for the determination of Blood meal (Certosan) in the matrices demineralised water, algal test medium, Daphnia and Danio test medium

References	Method	Analyte	Specificity / interferences	Calibration	Precision
Affolter, O.. (2013c)	ICP-OES	Fe	No interferences	n = 6 0.05 – 100 %w/w R > 0.9996	n = 6 RSD 3.20 %

Conclusion:

The method is acceptable. It filfull the requirements of SANCO 825/00 rev.8.1.

B.5.2.5. Air

According to the EFSA Scientific Report, EFSA Journal (2011); 9(10):2394, the residue definition for monitoring in soil Blood meal (Certosan) is a natural non-toxic compound.

Therefore, no analytical method is required.

B.5.2.6. Body fluids and tissues

According to the EFSA Scientific Report, EFSA Journal (2011);9(10):2394, the residue definition for monitoring in soil Blood meal (Certosan) is a natural non-toxic compound.

Therefore no analytical method is required.

B.5.3. REFERENCES 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	Data protection claimed Y/N	Justification if data protection is claimed	Owner	Previous evaluation
KCA 4.1.1	Affolter, O.	2013	Determination of the solubility in water of Blood Meal (Certosan) according to OECD 105 resp. EU A.6 Study No. 13022801GH910 LAUS GmbH GLP: yes unpublished	N	Y	-	FLU	N
KCA 4.1.2	Affolter, O.	2013 c	Validation of an Analytical Method using ICP-OES for the determination of Blood Meal (Certosan) in the matrices demineralised water, algal test medium, Daphnia and Danio test medium Report No. 13022801G926 LAUS GmbH GLP: yes unpublished	N	Y	-	FLU	N

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