

European Commission



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

Mecoprop-P

**Volume 3 – B.5 (PPP) – Mecoprop-P K 600 g/L
(CA3015)**

Rapporteur Member State : United Kingdom
Co-Rapporteur Member State : Ireland

Version History

When	What
31/03/2016	Initial Renewal Assessment Report (RAR)

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B.5. METHODS OF ANALYSIS

B.5.1. METHODS USED FOR THE GENERATION OF PRE-AUTHORISATION DATA

B.5.1.1. Analysis of the plant protection product

a) Active substance in the plant protection product

Report:	CP 5.1.1/01, Wilson, I. (2009)
Title	Duplosan KV (Mecoprop-P K 600 g/L) – Accelerated Storage and Dilution Stability. Report No: 09/0523
Guidelines:	EC Directive 94/37/EC amending Directive 91/414/EEC, SANCO 3030/99 rev 4
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

The determination of the mecoprop-P content in the plant protection product was conducted using an HPLC method with UV detection and internal standard methodology. The following method conditions were noted:

HPLC Column:	Nucleodex alpha PM or equivalent, 5 µm, 200 × 4.6 mm i.d.
Mobile phase:	65% Methanol / 35% buffer solution v/v (50 mM NaH ₂ PO ₄ adjusted to pH 3 with phosphoric acid)
Flow rate:	0.8 ml/min
Injector volume:	10 µl
Detector wavelength:	280 nm
Column Temperature:	30°C
Run Time:	20 minutes

The method used to determine the mecoprop-P content in the plant protection product is CIPAC MT 475. This is applicable to all SL formulations containing mecoprop-P. As this is a standard method full validation data are not required, the specificity of the method is reported below.

Specificity: No significant interference at the retention time of mecoprop-P (*ca.* 5.6 min) or internal standard (*ca.* 3.4 min) was observed in the formulation blank, test item or calibration solutions. Injection of a solution of racemic reference standard confirmed separation of the optical isomers (mecoprop s-isomer = *ca.* 7 min).

Conclusion

The method was considered to have the required specificity and was validated in accordance with SANCO/3030/99/ rev.4.

b) Relevant impurities in the plant protection product identified in the technical material or which may be formed during manufacture of the plant protection product or from degradation of the plant protection product during storage

The active ingredient mecoprop-P contains the relevant impurity 4-chloro-2-methylphenol (PCOC, IMP-5). The analytical method for the detection of PCOC (determined as ‘free phenols’) in the plant protection product is CIPAC MT 69 (UV-spectrophotometry)

Report:	CP 5.1.1/02, Wilson, I. (2009)
Title	Duplosan KV (Mecoprop-P K 600 g/L) – Accelerated Storage and Dilution Stability. Report No: 09/0523
Guidelines:	EC Directive 94/37/EC amending Directive 91/414/EEC, SANCO 3030/99 rev 4
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

CIPAC MT69 is a standard method therefore validation data are not required. It is noted that the lowest calibration point presented in CIPAC MT69 is equivalent to 0.4% PCOC based on nominal mecoprop-P content. As the test material PCOC content may be below this, the amount of sample taken for analysis is doubled. The procedure was validated with this higher sample aliquot and the results are summarised below.

Table Error! No text of specified style in document.-1 Method validation for CIPAC MT69 with higher sample aliquot

Matrix	LOQ	Linearity	Precision, %RSD (n)	Fortification levels (mg) and recovery range (mean), %	Interference
PCOC in Mecoprop-P K 600 (CA3015)	0.5 mg	0.02 – 0.12 mg n = 7 r ² = 0.998	5.1 (5) @ 0.107 % w/w Horwitz %RSD = 3.75	0.5 102.0 - 106.4 (104.1, n = 4) within SANCO acceptable range %	No interference ¹

¹The accuracy determination was carried out by fortification of a solution of mecoprop-P and the appropriate co-formulants in the form of a formulation blank. An acceptable mean recovery was achieved, if there was any significant interference from the co-formulants this would have affected the recovery value.

Conclusion

The method was validated in accordance with SANCO/3030/99/ rev.4.

The manufacturing process for the plant protection product Mecoprop-P K 600 is a mixing process, which involves no chemical reactions other than the conversion of the phenoxy acid mecoprop-P to its potassium salt. Therefore, no additional impurities of toxicological, ecotoxicological or environmental concern are formed during the manufacturing process of the plant protection product.

Furthermore, no additional impurities of toxicological, ecotoxicological or environmental concern arise from degradation of the plant protection product during storage. Note that PCOC may form on storage, but this has been addressed in the storage studies conducted on the representative product (see Volume 3 of the product dossier, section B.2.7). Data show that the plant protection product is stable for 14 days when stored at 54°C and for at least 2 years in ambient conditions. No further consideration is required.

Please refer to Confidential volume 4, section C.1.5. for details of the analytical methods for the determination of significant impurities in the active substance as manufactured.

c) Relevant co-formulants or components of co-formulants, where required by the national competent authorities

No methods are required for co-formulants or components of co-formulants.

B.5.1.2. Methods for the determination of residues

B.5.1.2.1. Methods In soil, water, sediment, air and any additional matrices used in support of environmental fate studies

Analytical methods used in support of environmental fate studies all include radio-isotopes. Therefore according to Regulation (EU) 283/2013 and 284/2013 methods are not necessary. No additional methods are required.

B.5.1.2.2. Methods in soil, water and any additional matrices used in support of efficacy studies

Table Error! No text of specified style in document.-2 Summary of efficacy methods submitted for purposes of renewal

	Study Reference	Analyte	Validation Data
Jar Test	Wilson, I (2012) Study number: 12/0710	Mecoprop-P	Method validation performed within the study (Appendix 7.1).
Cleaning Effectiveness	Stadler, R. (2002) BASF DocID 2002/1005097	Duplosan KV (SL formulation containing 600 g/L mecoprop-P)	None provided

Report:	CP 5.1.2/07, Wilson, I. (2012)
Title	Mecoprop-P K 600 g/l – Small Scale Jar Test Report No. 12/0710
Guidelines:	EC Directive 94/37/EC amending Directive 91/414/EEC, CRD Efficacy Guideline 305
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

Mecoprop-P in acetonitrile rinse solution and tank mix solutions was determined using HPLC with UV detection and external standard methodology. The following chromatography conditions were noted:

HPLC Column:	Nucleodex alpha PM or equivalent, 5 µm, 200 × 4.6 mm i.d.
Mobile phase:	65% Methanol / 35% buffer solution v/v (50 mM NaH ₂ PO ₄ adjusted to pH 3 with phosphoric acid)
Flow rate:	0.8 ml/min
Injector volume:	10 µl
Detector wavelength:	280 nm
Column Temperature:	30°C

Run Time: 15 minutes

Tank mix samples were prepared by dilution of a 1ml aliquot of tank mix in duplicate to a 100 ml volumetric flask. The volume was made up to the mark with acetonitrile. Acetonitrile rinse solutions were analysed directly with no further dilution, however samples were diluted further if required. A summary of the validation data are displayed in Table 5.1.2.2-2.

Table Error! No text of specified style in document.-3 Validation of small scale jar test method

Matrix	Support ed LOQ (% w/w)	Linearity	Precision*, %RSD (n)	Fortification levels (% w/w) and recovery range (mean), %		Interference
Mecoprop-P	0.04 %	0.0004 – 0.5 mg/mL n = 5 r = 0.9975	15.35 (5) @ 0.00005 % w/w Horwitz %RSD _r = 11.9 0.139 (5) @ 0.04 % w/w Horwitz %RSD _r = 4.35	0.00005 0.04	62.1 – 90.3 (79.8, n=5) 102.2 – 102.6 (102.4, n=5) SANCO acceptable range = 80 – 120 %.	Chromatograms of standard, blanks (water and acetonitrile) and samples show no interference at retention times of interest in tap water or acetonitrile. Retention time (mecoprop-P) = ca. 6.2 min

*Precision of accuracy samples is reported. This is acceptable.

Conclusion

The method is not strictly validated in accordance with the EU guidance document SANCO/3030/99/rev. 4, due to the mean recovery and precision at the lower fortification level being outside the acceptable SANCO range 80 – 120 %. Additionally, the individual recoveries at the lower fortification level fall well below this range.

Report:	CP 4.1.2/07, Stadler, R. (2002)
Title	BAS 037 32H – Duplosan Effectiveness of procedures for cleaning application equipment and protective clothing Report No. 2002/1005097
Guidelines:	-
GLP:	-
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

The determination of mecoprop-P in water samples taken throughout the cleaning procedure were analysed by HPLC with UV detection (method CF-A 514). The following conditions were noted:

Stationary phase:	Stainless steel column, Nucleosil C18, 5 µm, 200 x 4 mm
Mobile phase:	20,0 g Natriumdihydrogenphosphat, 770 ml water (milli-Q water) and 230 ml Acetonitril
Flow of the mobile phase:	1.0 ml/min
Injected volume:	5 µl (the washing sample with acetonitrile were injected with 100µl)
Detection:	280 nm
Temperature:	30°C
Analysis run:	12 min

Conclusion

The method is not validated in accordance with the EU guidance document SANCO/3030/99/rev. 4. No method validation data was provided.

B.5.1.2.3. Methods in feed, body fluids and tissues, air and any additional matrices used in support of toxicological studies

Analytical methods used in support of toxicological studies on the plant protection have previously been reviewed under Directive 91/414/EEC. A summary is provided below, but no further consideration is required.

Table Error! No text of specified style in document.-4 Summary of toxicology methods previously reviewed in support of studies

Annex Point	Reference	Matrix	Analytical technique	Validation Data
Acute Oral Toxicity (CP 7.1.1)	██████████ (1994a) ██████████	Water	HPLC-UV	Recovery 101.7% @ 20g/100ml
Acute Inhalation (CP 7.1.3)	██████████ 1994) ██████████	Acetonitrile-Bidistilled water (1:1 ratio)	HPLC-UV	Linear Range : 1-41 mg/50ml Std Dev 0.46% @ 5.4mg/l (n=4)

B.5.1.2.4. Methods in body fluids, air, and any additional matrices used in support of operator, worker, resident and bystander exposure studies

No analytical methods have been supplied in support of operator, worker, resident and bystander exposure studies.

B.5.1.2.5. Methods in or on plants, plant products, processed food commodities, food of plant and animal origin, feed and any additional matrices used in support of residues studies

Analytical methods used in support of residues studies have been submitted in the Chemical Active dossier for this submission. Please see volume 3 section B-5.

B.5.1.2.6. Methods in soil, water, sediment, feed and any additional matrices used in support of ecotoxicology studies

The following analytical methods have been submitted for the purposes of renewal in support of ecotoxicological studies.

Table Error! No text of specified style in document.-5 Summary of ecotoxicology methods submitted for purposes of renewal

Matrix	Analyte	Method	Reference
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Fish	Mecoprop-P K 600 (CA3015)	HPLC-UV	██████████ 2014a. Study ██████████
Daphnia	Mecoprop-P K 600 (CA3015)	HPLC-UV	Liedtke, 2014b. Study D76033
Algal	Mecoprop-P K 600 (CA3015)	HPLC-UV	Liedtke, 2013a. Study D76044
Lemna	Mecoprop-P K 600 (CA3015)	HPLC-UV	Liedtke, 2013b. Study D76055
Test medium and sediment	Mecoprop-P K 600 (CA3015)	HPLC-MS/MS	Gonsoir, 2015. Study S13-04889
Test medium	Mecoprop-P K 600 (CA3015)	HPLC-MS/MS	Seyland-Fremer & Mosch, 2015. Study 91411215

Report:	CP 5.1.2/01 ██████████ (2014a)
Title	Mecoprop-P K, 600 (CA3015): Acute Toxicity to Rainbow Trout (<i>Oncorhynchus mykiss</i>) in a 96-Hour Test. Report ██████████
Guidelines:	EC Directive 94/37/EC amending Directive 91/414/EEC, SANCO 3030/99 rev 4
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

Report:	CP 5.1.2/02, Liedtke, A (2014b)
Title	Mecoprop-P K, 600 (CA3015): Acute Toxicity to <i>Daphnia magna</i> in a 48-Hour Immobilization Test. Report No: D76033
Guidelines:	EC Directive 94/37/EC amending Directive 91/414/EEC, SANCO 3030/99 rev 4
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

Report:	CP 5.1.2/03, Liedtke, A (2013a)
Title	Mecoprop-P K, 600 (CA3015): Toxicity to <i>Pseudokirchneriella subcapitata</i> in a 72-hour algal growth inhibition test. Report No: D76044
Guidelines:	EC Directive 94/37/EC amending Directive 91/414/EEC, SANCO 3030/99 rev 4
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

Report:	CP 5.1.2/04, Liedtke, A (2013b)
Title	Mecoprop-P K, 600 (CA3015): Toxicity to the Aquatic Higher Plant <i>Lemna gibba</i> in a 7-day Growth Inhibition Test. Report No: D76055
Guidelines:	EC Directive 94/37/EC amending Directive 91/414/EEC, SANCO 3030/99 rev 4
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

The mecoprop-P content in aqueous solution in the trout, daphnia, algal and lemna studies above was determined by HPLC with UV detection and external standard methodology. Samples were stored frozen at -20°C immediately after sampling until analysis. It is stated in the study that the active

ingredient proved to be stable under these storage conditions in pre-experiments, though these results were not reported. Test samples were thawed at room temperature for 2 hours and shaken to obtain homogeneous solutions. In the case of the algal study (D76044), some samples were centrifuged due to the presence of algae. Samples were diluted into the calibration range with test water before analysis by HPLC. The following chromatography conditions were noted:

HPLC Column:	Luna C8 (2); 3 μ m, 150 \times 4.6 mm i.d.																		
Mobile phase:	Eluent A: Methanol/water (v/v, 1:9) + 10mM ammonium acetate Eluent B: Methanol/water (v/v, 9:1) + 10mM ammonium acetate																		
Gradient :	<table> <thead> <tr> <th>Minutes</th> <th>% Eluent A</th> <th>% Eluent B</th> </tr> </thead> <tbody> <tr> <td>0</td> <td>60</td> <td>40</td> </tr> <tr> <td>10</td> <td>20</td> <td>80</td> </tr> <tr> <td>13</td> <td>20</td> <td>80</td> </tr> <tr> <td>13.1</td> <td>60</td> <td>40</td> </tr> <tr> <td>19</td> <td>60</td> <td>40</td> </tr> </tbody> </table>	Minutes	% Eluent A	% Eluent B	0	60	40	10	20	80	13	20	80	13.1	60	40	19	60	40
Minutes	% Eluent A	% Eluent B																	
0	60	40																	
10	20	80																	
13	20	80																	
13.1	60	40																	
19	60	40																	
Flow rate:	0.8 ml/min																		
Injector volume:	20 μ l																		
Detector wavelength:	230 nm																		
Retention Time (mecoprop-P):	ca. 9.4 minutes																		

The validation data for each study are reported in the following table.

Table Error! No text of specified style in document.-6 Validation of method for determination of Mecoprop-P K 600 (CA3015) in aqueous medium

Matrix	LOQ	Linearity*	Precision, %RSD (n)	Fortification levels‡ (mg/L) and recovery range (mean), %		Interference
Water (fish, D76022)	47.2 mg/L [0.00472 % w/w]	1.03 – 14.8 mg/L n = 7 r ² = 1.0000	4 (5) %RSD < 20	47.2 (0.00472 % w/w)	92 - 102 (99, n=5) within SANCO acceptable range 80 – 100 %	Chromatograms of control, test and analytical standard samples showed no interference at retention time of interest.
Water (daphnia, D76033)	48.8 mg/L [0.00488 % w/w]	0.126 – 10.1 mg/L n = 9 r ² = 0.9999	3 (4) %RSD < 20	48.8 (0.00488 % w/w)	102 - 126† (105, n=4) within SANCO acceptable range 80 – 100 %	Chromatograms of control, test and analytical standard samples showed no interference at retention time of interest.
Water (Algal, D76044)	5.10 mg/L [0.00051 % w/w]	0.524 – 14.0 mg/L n = 6 r ² = 0.9994	1 (5)	5.10 (0.00051 % w/w)	100 -102 (101, n=5)	Chromatograms of control, test and analytical standard samples showed no interference at retention time of interest.
			0.3 (5)	56.1 (0.00561 % w/w)	100 (100, n = 5)	
			%RSD < 20 at both fortification values	Centrifuged samples:		
				10.8 (0.00108 % w/w)	102 (102, n=2)	
	119 (0.0119 % w/w)	101- 102 (102, n=2) within SANCO acceptable range 80				

Matrix	LOQ	Linearity*	Precision, %RSD (n)	Fortification levels‡ (mg/L) and recovery range (mean), %		Interference
					- 100 %	
Water (Lemna, D76055)	0.141 mg/L [0.0000141 % w/w]	0.0998 – 4.99 mg/L n = 7 r ² = 0.9999	0.4 (5) 1 (5) %RSD < 20 at both fortification values	0.141 (0.0000141 % w/w) 47.5 (0.00475 % w/w)	105 - 106 (104, n=5) 100 - 102 (100, n=5) within SANCO acceptable range 80 - 100 %	Chromatograms of control, test and analytical standard samples showed no interference at retention time of interest.

*Samples are diluted to fall within linear range.

†Recovery of 126 classed as an outlier and not used in statistical calculations. This is acceptable.

‡Expressed as levels of mecoprop-P added.

Conclusion

The supported LOQ does accommodate the lower levels of residues expected in the test samples. Therefore, even though only one fortification level has been tested in some cases, it is at a more critical level than that used in the study, therefore the method is validated according to SANCO/3029/99/rev. 4. It should be noted that the fortifications seem to have been carried out in test water, rather than substrate and this should be considered in the ecotoxicology assessment.

Report:	CP 10.2.1/05, Gonsoir, G (2015)
Title	Mecoprop-p K 600 g/L: Growth Inhibition of <i>Myriophyllum spicatum</i> in a Water/Sediment System, Report No.: S13-04889
Guidelines:	OECD Draft Guideline: Water-Sediment Myriophyllum sp Toxicity Test based on Draft AMRAP Method: Growth Inhibition Test for the Rooted Aquatic Macrophyte, <i>Myriophyllum sp.</i> Submitted to OECD for Evaluation, 22 July 2013.
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

The mecoprop-P content in test medium (water plus nutrients) and wet sediment was determined by HPLC with MS/MS detection (ESI, negative mode, quantifier ion m/z 213→141; qualifier ion m/z 215→143). The following chromatography conditions were noted:

Column:	Thermo Accucore qQ, 50 mm x 2.1 mm i.d. 2.6 µm mean particle size (Thermo no. 17326-052130) with 4 mm guard column
Column temperature:	40 °C
Injection volume:	20 µL
Mobile phase:	A: Water B: Methanol

Samples were stored frozen at ≤ -18 °C until analysis. The maximum storage period from sampling to analysis was 21 days for test medium and 7 days for sediment. No storage stability data is required as the samples were stored for < 30 days. Once thawed, test medium samples were prepared by the addition of ammonia and solid phase extraction (SPE) and sediment samples were extracted with methanol/water (8:2) + 10 mM NaOH before cleanup by SPE.

The validation data for each matrix is displayed in the following table. Sediment recovery samples were prepared by spiking sediment with analytical standard and test medium recovery samples were prepared by spiking with test item (60 % TGAI, at 86 % purity). Samples were diluted so as to fall within the linear range.

Matrix	LOQ	Linearity	Precision ¹ , %RSD (n)	Fortification levels and recovery range (mean), %		Interference
Test medium	0.2 µg/L (0.096 µg/L mecoprop-P)	0.1 - 20 ng/mL [equiv. to 0.1 - 20 µg/L] n = 9 r ² = 0.999	2 (5)	0.2 µg/L (equivalent to 0.096 µg/L mecoprop-P)	88 – 93 (91, n=5)	Chromatograms of test sample, control sample and analytical standard samples showed no interference at retention time of interest (ca. 5 min mecoprop-P).
			2 (5) %RSD < 20	250 µg/L (equivalent to 120 µg/L mecoprop-P)	94 – 98 (96, n=5) Mean within SANCO acceptable range 80 – 100 %	
Sediment	0.005 mg/kg		5 (5) 9 (5) %RSD < 20	0.005 mg/kg mecoprop-P 0.1 mg/kg mecoprop-P	86 – 96 (93, n=5) 79 – 101 (93, n=5) Mean within SANCO acceptable range 80 – 100 %	Chromatograms of test sample, control sample and analytical standard samples showed no interference at retention time of interest (ca. 5 min mecoprop-P).

¹The precision of recovery was reported for each fortification level.

Conclusion

The method for detecting mecoprop-P in test medium and sediment is validated in accordance with SANCO/3029/99/rev.4. The LOQ in test medium is 0.096 µg/L mecoprop-P and in sediment is 0.005 mg/kg mecoprop-P.

Report:	CP 10.2.1/06, Seeland-Fremer, A & Mosch, W (2015)
Title	Toxicity of Mecoprop-p K 600 g/L to the Aquatic Plant <i>Myriophyllum spicatum</i> in a Static Growth Inhibition Test with Prior Rooting Phase, Report No.: 91411215
Guidelines:	GLP compliant study based on OECD Draft New Guideline 239: Water-Sediment <i>Myriophyllum spicatum</i> Toxicity Test, May 20, 2014
GLP:	Yes
Deviations	None
Previous evaluation:	None; Submitted for the purpose of renewal under Regulation 844/2012.

The mecoprop-P content in test medium (water plus nutrients) and wet sediment was determined by HPLC with MS/MS detection. The following chromatography conditions were noted:

LC:	Agilent Series 1290 pump and autosampler
Column:	Synergi Hydro RP 80A (150 x 3 mm, 4µm)
Temperature:	40 °C
Mobile Phase:	50% methanol /50% HPLC water
Flow Rate:	0.3 mL/min
Injection Volume:	3 µL
Mass Spectrometer:	Mass spectrometer API 4000
Ion Source:	Electrospray negative
Mass Transition:	quantifier ion: 213 to 141 amu qualifier ion: 213 to 71 amu

Samples were stored frozen at ≤ -10 °C until analysis. The maximum storage period from sampling to analysis was not reported in the study, but the analysis of fresh and aged samples (14 days) gave acceptable and comparable recoveries, demonstrating the stability of the samples over 14 days. It is assumed that this is the maximum time period from sampling to analysis.

The validation data for each matrix is displayed in the following table. Samples were diluted with acetonitrile / test water (1:1) by a factor of two and further, if required to fall within the linear range. Recovery samples were prepared by dilution of a 1 g/L test item solution. This is not considered an acceptable approach in accordance with SANCO/3029/99/rev.4, which states a known amount of analyte should be added to an untreated commodity.

Matrix	LOQ	Linearity	Precision ¹ , %RSD (n)	Fortification levels and recovery range (mean), %		Interference
Test medium	5 µg test item/L	0.5 – 74 µg/L	1 (5)	5 µg/L (equivalent to ca. 2.5 µg/L mecoprop-P)	96 – 99 (97, n=5)	Chromatograms of test sample, blank control sample and analytical standard samples showed no interference at retention time of interest (ca. 2.3 min mecoprop-P).
	(ca. 2.5 µg/L mecoprop-P)	n = 9 r ² = 0.9999	1 (5)	50 µg/L (equivalent to ca. 25 µg/L mecoprop-P)	101 - 104 (102, n=5)	
			2 (5) %RSD < 20	1250 µg/L (equivalent to ca. 620 µg/L mecoprop-P)	100 – 106 (103, n=5) Mean within SANCO acceptable range 70 – 110 %	

¹The precision of recovery was reported for each fortification level.

Conclusion

The method for detecting mecoprop-P in test medium is not validated in accordance with SANCO/3029/99/rev.4 due to the recovery experiments not being conducted by spiking a blank matrix

with a known amount of standard. The LOQ in test medium is 2.5 µg/L mecoprop-P or 5 µg/L test item. The acceptability of the method will be considered in the ecotoxicology assessment.

B.5.1.2.7. Methods in water, buffer solutions, organic solvents and any additional matrices resulting from the physical and chemical properties tests

Table Error! No text of specified style in document.-7 Summary of new physical chemistry studies with supporting analytical data

Physical and chemical property	Study Reference	Analyte	Validation Data
Accelerated storage stability	Mahmood, T (2012) Study number: 12/0697	Mecoprop-P PCOC	Validation of MCPP-P content by HPLC-UV and free phenols as PCOC by UV is contained in study 09/0523 (Wilson, 2009). This has been validated in section B.5.1.1.
Two Year Storage Stability	Mahmood, T (2014) Study Number: 12/0717	Mecoprop-P PCOC	Validation of MCPP-P content by HPLC-UV and free phenols as PCOC by UV is contained in study 09/0523 (Wilson, 2009). This has been validated in section B.5.1.1.

B.5.2. METHODS FOR POST-APPROVAL CONTROL AND MONITORING PURPOSES

Methods for post-approval control and monitoring purposes are summarised in the Chemical Active dossier. Please refer to Volume 3, Section B-5.2 of the Active RAR for details.

B.5.3. REFERENCES RELIED ON

Regarding the literature search undertaken by the applicant (report dated 15/07/2015). It is considered that the search is acceptable in terms of databases searched and the search criteria applied. The search did not reveal any references of relevance to this section.

The references relied on list has been updated to include the newly submitted data relied on as well as those original submitted tests and studies (in *italics*) that are still considered relevant to support the application for renewal.

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
CP 5.1.1/01	Wilson, I	2009	Duplosan KV (Mecoprop-P K 600 g/L) – Accelerated	N	Y	New data to new guidelines	Nufarm	Submitted for purposes of renewal

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
			Storage and Dilution Stability. Report No: 09/0523 Nufarm UK Limited GLP Not published					
CP 5.1.2 & CP 7.1.1	████████	1994a	Study on the acute oral toxicity of BAS 037 32 H in Rats ████████ ████████ ████████ GLP Not published	Y	N	-	Nufarm	In DAR (1998)
CP 5.1.2 & CP 7.1.3	████████ █	1994	Study on the acute inhalation toxicity LC50 of BAS 037 32 H as a liquid aerosol in rats 4-hour exposure ████████ ████████ ████████ GLP Not published	Y	N	-	Nufarm	In DAR (1998)
CP 5.1.2/01	████████	2014a	Mecoprop-P K, 600 (CA3015): Acute Toxicity to Rainbow Trout (<i>Oncorhynchus mykiss</i>) in a 96-Hour Test ████████ ████████ ████████ GLP Not published	Y	Y	New data submitted	Nufarm	Submitted for purposes of renewal
CP 5.1.2/02	Liedtke, A	2014b	Mecoprop-P K, 600 (CA3015): Acute Toxicity to <i>Daphnia magna</i> in a 48-Hour Immobilization Test. Harlan	N	Y	New data submitted	Nufarm	Submitted for purposes of renewal

Data Point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Verteb rate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner	Previous evaluation
			Laboratories Ltd, Switzerland Report No: D76033 GLP Not published					
CP 5.1.2/03	Liedtke, A	2013a	Mecoprop-P K, 600 (CA3015): Toxicity to <i>Pseudokirchneriella subcapitata</i> in a 72-hour algal growth inhibition test. Harlan Laboratories Ltd, Switzerland Report No: D76044 GLP Not published	N	Y	New data submitted	Nufarm	Submitted for purposes of renewal
CP 5.1.2/04	Liedtke, A	2013b	Mecoprop-P K, 600 (CA3015): Toxicity to the Aquatic Higher Plant <i>Lemma gibba</i> in a 7-day Growth Inhibition Test. Harlan Laboratories Ltd, Switzerland Report No: D76055 GLP Not published	N	Y	New data submitted	Nufarm	Submitted for purposes of renewal
CP 10.2.1/05	Gonsoir, G	2015	Mecoprop-p K 600 g/L: Growth Inhibition of <i>Myriophyllum spicatum</i> in a Water/Sediment System Report No.: S13-04889 GLP Not published	N	Y	New data submitted	Nufarm	Submitted for purposes of renewal