

Study N° 22973

# 10.2.e

# Physical and chemical properties and low temperature storage stability of PMV-01

#### **REPORT**

Project: DCM / FO 22973 / Ch.5324 / 2012 / A

Study starting: June 25, 2012 Study completion: July 17, 2012

#### **TEST FACILITY**

Centre wallon de Recherches agronomiques





#### Authenticity and confidentiality statement

 Study plan number : 22973.
 10.1.c

 Wob juncto
 63.2.d Vo

 1107/2009
 1107/2009

 Study director
 10.2.e

Study title: Physical and chemical properties and low temperature storage stability of PMV-01.

We, the undersigned, hereby declare that the work described in this report was performed under our supervision in accordance with the agreed study plan, and that the report provides a true and accurate record of the results obtained.

The information contained in this report is confidential and must not be discussed without the written consent of **De Ceuster N.V.**.

The Plant Protection Products and Biocides Physico-chemistry and Residues Unit of CRA-W undertakes not to disclose any of the results, work practices or functions of **De Ceuster N.V.** conveyed to the Plant Protection Products and Biocides Physico-chemistry and Residues Unit of CRA-W during the course of this study.

5030 - GEMBLOUX, July 17, 2012.



# Statement of GLP compliance

Study plan number: 22973.

Project: DCM / FO 22973 / Ch.5324 / 2012 / A.

10.1.c wob juncto 63.2.d Vo 1107/2009

Test item: PMV-01

isolate 1900) SC.

Pepino mosaic virus, CH2 strain,

180

10.2.e

**Study director**:

Study title: Physical and chemical properties and low temperature storage stability of PMV-01.

This study was performed in compliance with the OECD Principles of Good Laboratory Practice.

The characterisation of the test item was not performed in this study. A certificate of analysis was provided by the sponsor (non GLP certificate).

The Plant Protection Products and Biocides Physico-chemistry and Residues Unit of CRA-W has been certified for GLP compliance since October 1994.

July 17, 2012.

10.2.e



## **Statement of Quality Assurance**

Study plan number: 22973.

10.1.c Wob juncto 63.2.d Vo 1107/2009

Project: DCM / FO 22973 / Ch.5324 / 2012 / A.

Test item: PMV-01

Pepino mosaic virus, CH2 strain,

isolate 1906) SC.

**Quality assurance**: Lic. Vanessa HERION

Valérie DE COCK.

Study title: Physical and chemical properties and low temperature storage stability of PMV-01.

The following inspections have been carried out in relation to this study:

Quality control	Date of inspection	Date reported to study director	Date reported to test facility manager		
Study plan	June 25, 2012	June 25, 2012	June 26, 2012		
Critical phase PA-U10-METDESCR	June 27, 2012	June 27, 2012	June 28, 2012		
Raw data and draft report	July 10, 2012	July 10, 2012	July 12, 2012		
Report	July 17, 2012	July 17, 2012	July 17, 2012		

In addition the repetitive procedures and analytical methods are covered by process based inspections and were inspected within the framework of other similar GLP studies.

Similarly an inspection of the facility where this study was conducted was carried out on an annual basis.

I, Quality Assurance Inspector, hereby declare that the work described in this report is in accordance with the agreed study plan, and that the results in the report totally and accurately reflect the raw data.

July 17, 2012.



10.2.e

Quality Assurance Inspector.

# **Table of contents**

Authenticity and confidentiality statement	2
Statement of GLP compliance	3
Statement of Quality Assurance	4
Table of contents	5
Summary of results	6
General information	8
REPORT	11
1 Study objective	11
2 Materials	11
2.1 Analytical standard	11
2.2 Test item	11
3 Analytical method	13
3.1 Appearance of the test item	13
3.2 Appearance of the commercial type pack	13
3.3 pH of the test item	13
3.4 pH at 1 % in water	13
3.5 Viscosity of liquids by rotational viscometry (dynamic viscosity)	13
3.6 Density at $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ and relative density	
3.7 Persistent foaming	14
3.8 Wet sieve test	14
3.9 Storage for 7 days at $0^{\circ}$ C $\pm 2^{\circ}$ C	14
4 Results	15
4.1 Determination on the test item as received	15
4.1.1 Appearance of the test item	15
4.1.2 Appearance of the finished package	
4.1.3 pH of the test item	
4.1.4 pH at 1 % in water	
4.1.5 Density at $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ and relative density	
4.1.6 Viscosity of liquids at 20°C	
4.1.7 Viscosity of liquids at 40°C	
4.1.8 Persistent foaming	
4.1.9 Wet sieve test	21
4.2 Determination after storage for 7 days at $0^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in a closed glass bottle	22
4.2.1 Appearance of the test item	
4.2.2 Wet sieve test	
5 Amendment to the study plan	
6 Deviation to the study plan	
List of figures	
Viscosity of liquids	
Viscosity at 20°C ± 0.5°C	23
Viscosity at $40^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$	
Appendices	
Certificate of analysis of the test item (supplied by the sponsor)	24
Certificate of GLP endorsement from Belgian authorities	

10.1.c Wob juncto

63.2.d Vo 1107/2009

# **Summary of results**

<u>**Test item**</u> : PMV-01.

<u>Type of formulation</u>: suspension concentrate (SC).

Active substance content (nominal concentration):

Pepino mosaic virus, CH2

strain, isolate 1906.

**Batch number** 

Manufacture date:

Expiry date:

10.1.c Wob juncto 63.2.a Vo 1107/2009 juncto 39.2.a Vo 178/2002

Tests	Methods	Initially	After 7 days at 0°C ± 2°C in a closed glass bottle (CIPAC MT 46.3)
Appearance of the test item	PA-U10-METDESCR visual method	Physical state at ambient temperature: liquid with some particles in the bottom. homogeneous after gentle shaking but the particles fall quite quickly Colour: dark green.	Physical state at ambient temperature: liquid with some particles in the bottom. homogeneous after gentle shaking but the particles fall quite quickly Colour: dark green.
		Odour:	Odour :
Appearance of the finished package	PA-U10-METDESCR visual method	Commercial brown translucent PET bottle of 1 L.	
		Closing: with a screw white plastic cap.	
		Sealing: with a small plastic tongue well closed bottle without deterioration or special anomaly. no observable sign of test item contamination on the outer surface. no leak during shaking or turning. no noticeable odour before opening of the package.	-
pH of the test item	CIPAC MT 75.3	5.89	-
pH at 1 % in water	CIPAC MT 75.3	6.52	-
Density at 20°C ± 0.5°C	CIPAC MT 3.3.2	1.0004 g/mL	-
and relative density	Calculation	$D_{4}^{20} = 1.0004$	-

# Walloon Agricultural Research Centre, CRA-W Gembloux, Belgium PLANT PROTECTION PRODUCTS AND BIOCIDES PHYSICO-CHEMISTRY AND RESIDUES UNIT (U10)

Tests	Methods	Initially	After 7 days at 0°C ± 2°C in a closed glass bottle (CIPAC MT 46.3)
Viscosity of liquids $ \begin{tabular}{ll} Temperatures: $20^{\circ}$C$ $\pm 0.5^{\circ}$C \\ & 40^{\circ}$C$ $\pm 0.5^{\circ}$C \\ \end{tabular} $	CIPAC MT 192	No Newtonian flow behaviour 64.3 mPa.s to 4.4 mPa.s [10.58 – 665 s <sup>-1</sup> ] 64.3 mPa.s to 3.6 mPa.s [10.58 – 665 s <sup>-1</sup> ] Dependent on the shear rate applied to the sample	-
Persistent foaming in CIPAC water D Temperature: 30°C ± 2°C Concentration: 2.5 % v/v after 10 seconds after 1 minute after 3 minutes	CIPAC MT 47.2	15 mL 12 mL 10 mL	- -
after 12 minutes		ring (< 0.5 mL)	-
Wet sieve test (material retained on a 75 µm test sieve)	CIPAC MT 185	0.033 % w/w	0.029% w/w

#### **General information**

#### **Guidelines**:

- EU legislation concerning pesticide technical and formulated products and more particularly:
  - Directive 91/414/EEC
  - Directive 2001/36/EC of May 16, 2001 amending the directive 91/414/EEC concerning the placing of plant protection products on the market.
  - Directive 94/37/EC
  - Commission Regulation (EU) No 545/2011 of 10 June 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the data requirements for plant protection products.
  - Commission Regulation (EU) No 440/2008 of 30 May 2008 (EU methods)
  - Draft working document concerning the date requirements for active substances of plant protection products made from plants or plant extracts SANCO/10472/2003 rev .5 (July 06/2004).
- Manual on the development and use of FAO and WHO specifications for pesticides, second revision of the first edition, 2010, Rome.
- CIPAC methods recommended by EU, FAO and WHO.
- OECD guidelines.
- Standard Operating Procedures and methods from the Plant Protection Products and Biocides Physico-chemistry and Residues Unit of the Walloon Agricultural Research Centre (CRA-W).

Sponsor:

De Ceuster N.V.

Fortsesteenweg 30

2860 Sint-Katelijne-Waver

**BELGIUM** 

**Study monitor**:



10.2.e

10.2.e

#### Study director:

Walloon Agricultural Research Centre (CRA-W)

Agriculture and Natural Environment Department (D3)

Plant Protection Products and Biocides Physico-chemistry and Residues Unit (U10)

Carson Building Rue du Bordia, 11 B - 5030 - GEMBLOUX

**BELGIUM** 

**Quality Assurance**:

Walloon Agricultural Research Centre (CRA-W)

Agriculture and Natural Environment Department (D3)

Plant Protection Products and Biocides Physico-chemistry and Residues Unit (U10)

Carson Building Rue du Bordia, 11 B - 5030 - GEMBLOUX

**BELGIUM** 

**Technical personnel**:

**Secretariat** 

Archivist:

**Time schedule**: Study plan

- signed by the study director on June 25, 2012.
- signed by the study monitor on June 29, 2012.

Analysis on the test item as received and after 7 days at  $0^{\circ}$ C  $\pm 2^{\circ}$ C

- from June 27 until July 04, 2012.

#### **Archives**:

All the documentation relating to this study will be kept under the test item number in the archives of the Plant Protection Products and Biocides Physicochemistry and Residues Unit of the Walloon Agricultural Research Centre (CRA-W), presently located at 11, rue du Bordia, B - 5030 - GEMBLOUX, BELGIUM, for a period of at least 10 years starting on the study completion date, unless instructions to the contrary are received from the sponsor.

Archived documents include as minimum:

- study plan and amendment
- all correspondence
- protocols
- raw data
- report
- Quality Assurance Inspection reports.

An aliquot of the remaining original test item after analysis will be stored by the test facility at room temperature in the original container under shelter from direct sunlight for a period of at least 5 years starting on the study completion date. After this period, it will be destroyed unless instructions to the contrary are received from the sponsor.

#### **REPORT**

10.1.c Wob juncto 63.2.d Vo 1107/2009

#### 1 Study objective

The purpose of this study was to determine some physical and physico-chemical properties and the low temperature storage stability of the formulated product **PMV-01**, a formulation suspension concentrate (SC)

Pepino mosaic virus, CH2 strain, isolate 1906.

The tests and the storage for 7 days at 0°C have been carried out by the Plant Protection Products and Biocides Physico-chemistry and Residues Unit of the Walloon Agricultural Research Centre (CRA-W).

This GLP report deals with all the results of the study.

#### 2 Materials

#### 2.1 Analytical standard

Not relevant for this study.

#### 2.2 Test item

**Name**: PMV-01.

**Type of formulation\***: suspension concentrate (SC).

Active substances contents\* (nominal concentrations):

Pepino mosaic virus, CH2 strain, isolate 1906.

Supplier: De Ceuster N.V., Fortsesteenweg 30, 2860 Sint-Katelijne-Waver, Belgium.

**Containers**: 2 PET bottles of 1 litre (commercial pack)

**Batch number**\*:

Manufacture date\*:

Expiry date\*

10.1.c Wob juncto 63.2.a Vo 1107/2009 juncto 39.2.a Vo 178/2002

10.1.c Wob iuncto 63.2.d

1107/2009

Vo

<u>Certificate of analysis</u> (supplied by the sponsor): of June 25, 2012 (see copy on page 24).

**Receipt date at the test facility**: June 22, 2012.

**Registration number at the test facility**: Ch.5324.

<sup>\*:</sup> information supplied by the sponsor.

**Storage**: before the analysis : at  $4^{\circ}C \pm 3^{\circ}C$  in the original container under shelter from direct sunlight.

during the analysis, a sub-sample has been kept at room temperature.

The GLP Principles require that the sponsor provides to the study director the active substance content, the batch number, the estimated expiry date and the stability and homogeneity of the active substance of test item in the vehicle (when applicable) unless the determination of these items is planned in the study. The shipment of the test item until the receipt at the test facility is under the responsibility of the sponsor.

#### 3 Analytical method

#### 3.1 Appearance of the test item

Test facility method PA-U10-METDESCR (visual method).

Physical state, odour and colour of the test item.

Direct inhalation is assumed to present a hazard and is prohibited. However if an odour is detected and identified, it is noted.

Statement of claying and redispersibility.

# 3.2 Appearance of the commercial type pack

Test facility method PA-U10-METDESCR (visual method).

Description of the commercial package, influence of the test item on the commercial package and vice versa.

Statement on compatibility between the test item and the package.

#### 3.3 pH of the test item

CIPAC method MT 75.3, CIPAC Handbook J - pg. 131.

- Apparatus: METROHM 713 pH meter.
- Calibration of the pH meter with commercial pH buffer solutions (pH 2, 4, 7 and 9) at recorded solution temperature (solutions at room temperature).

#### 3.4 **pH** at 1 % in water

CIPAC method MT 75.3, CIPAC Handbook J - pg. 131.

- Apparatus : METROHM 713 pH meter.
- Calibration of the pH meter with commercial pH buffer solutions (pH 2, 4, 7 and 9) at recorded solution temperature (solutions at room temperature).
- Concentration: 1 g test item / 100 mL.
- Water: freshly boiled and cooled distilled water.

## 3.5 <u>Viscosity of liquids by rotational viscometry (dynamic viscosity)</u>

CIPAC method MT 192, CIPAC Handbook L - pg.145.

Method based on OECD guidelines 114.

- Apparatus : rotative viscosimeter RHEOMAT 15T-FC. Contraves with coaxial cylinders.

Bowl A.

- Temperatures :  $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  and  $40^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ .

#### 3.6 Density at $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ and relative density

CIPAC method MT 3.3.2 (density bottle method), CIPAC Handbook F - pg. 19. Method equivalent to EEC method A.3 published in the Commission Regulation (EC) No. 440/2008.

- Temperature :  $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ .

The relative density D  $_{4}^{20}$  is calculated from the result obtained for the density at 20°C  $\pm$  0.5°C and from the water density at 4°C (1.00000) : D  $_{4}^{20}$  = density / 1.

#### 3.7 Persistent foaming

CIPAC method MT 47.2, CIPAC Handbook F - pg. 152.

- Concentration (the highest): at the highest concentration of use: 2.5% v/v corresponding to 4 L / 160 L water
- Water: CIPAC water D, CIPAC method MT 18.1.4, CIPAC Handbook F - pg. 62.
- Temperature :  $30^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .

Determination of the volume of foam after 10 seconds  $\pm$  1 second, 1 minute, 3 minutes and 12 minutes  $\pm$  10 seconds.

#### 3.8 Wet sieve test

CIPAC method MT 185, CIPAC Handbook K - pg. 149.

- Test sieve : 75 μm.

#### 3.9 Storage for 7 days at $0^{\circ}$ C $\pm 2^{\circ}$ C

CIPAC method MT 46.3.1, CIPAC Handbook J - pg. 128.

- Test : 7 days at  $0^{\circ}$ C  $\pm 2^{\circ}$ C.
- Storage of the test item: in a closed glass bottle.

#### 4 Results

10.1.c Wob juncto 63.2.a Vo 1107/2009 juncto 39.2.a Vo 178/2002

#### 4.1 Determination on the test item as received

[Bottle 1/2]

**Dates of analysis**: from June 27 until July 04, 2012.

#### 4.1.1 Appearance of the test item

Date of analysis: June 27, 2012.

Physical state: liquid with some particles in the bottom.

homogeneous after gentle shaking but the particles fall quite quickly

Colour: dark green.

Odour:

#### 4.1.2 Appearance of the finished package

Date of analysis: June 27, 2012.

Outside aspect

- Description: Commercial brown translucent PET bottle of 1 litre

closing: with a screw white plastic cap.

external diameter :  $\pm$  9 cm. height (with cap) :  $\pm$  22.5 cm. sealing : with a small plastic tongue

- well closed bottle without deterioration or special anomaly.
- no observable sign of test item contamination on the outer surface.
- no leak during shaking or turning.
- no noticeable odour before opening of the package.

#### Inside aspect

- no observable alteration of package material by the test item.

Weighing (at the receipt of the test item on June 22, 2012)

bottle 1/2: 1072.3 g

[for analysis on the test item as received and for low temperature storage

of 7 days at 0°C]

bottle 2/2: 1070.4 g [spare sample]

PLANT PROTECTION PRODUCTS AND BIOCIDES PHYSICO-CHEMISTRY AND RESIDUES UNIT (U10)

#### 4.1.3 pH of the test item

Date of analysis: June 27, 2012.

Temperature: 19°C.

Determination	pН
1	5.90
2	5.88
Mean	5.89

#### 4.1.4 pH at 1 % in water

Date of analysis: June 27, 2012.

Temperature: 21°C.

Determination	pН
1	6.53
2	6.50
Mean	6.52

# 4.1.5 Density at $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ and relative density

Date of analysis: June 27, 2012.

Determination	Density (g/mL)	Relative density D <sup>20</sup> <sub>4</sub>		
1	1.0007	1.0007		
2	1.0000	1.0000		
Mean	1.0004	1.0004		

#### 4.1.6 Viscosity of liquids at 20°C

Date of analysis: June 27, 2012.

Temperature :  $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ .

Bowl A.

#### **DETERMINATION 1**

Spe	eed	Shear rate D (s <sup>-1</sup> )	Shear stress	Factor  η %  (mPa.s by scale unity)		Reading (%)		amic osity l Pa.s)
steps	(rpm)				$\rightarrow$	<b>↑</b>	$\rightarrow$	<b>↑</b>
1	5.59	10.58	0.17912	16.93	3.8	3.8	64.3	64.3
2	7.51	14.20		12.61	3.8	3.8	47.9	47.9
3	9.89	18.69		9.59	3.8	3.8	36.4	36.4
4	13.19	24.9		7.18	3.8	3.8	27.3	27.3
5	17.40	32.9		5.45	3.8	3.8	20.7	20.7
6	25.1	47.4		3.78	3.8	3.8	14.4	14.4
7	33.7	63.7		2.81	3.8	3.8	10.7	10.7
8	44.3	83.8		2.14	4.0	3.8	8.6	8.1
9	59.1	111.7		1.603	4.2	3.8	6.7	6.1
10	78.0	147.4		1.215	4.2	3.8	5.1	4.6
11	113.2	214		0.837	4.2	3.8	3.5	3.2
12	152.0	287		0.623	4.8	4.8	3.0	3.0
13	200	378		0.474	7.0	7.0	3.3	3.3
14	267	504		0.355	10.0	10.0	3.6	3.6
15	352	665		0.269	16.2	16.2	4.4	4.4

#### **DETERMINATION 2**

Spe	eed	Shear rate D (s <sup>-1</sup> )	Shear stress  τ %  (Pa by scale unity)	Factor η % (mPa.s by scale unity)	Reading (%)		Reading (%)		(%) η (mPa.	
steps	(rpm)				$\rightarrow$	<b>↑</b>	$\rightarrow$	<b>↑</b>		
1	5.59	10.58	0.17912	16.93	3.8	3.8	64.3	64.3		
2	7.51	14.20		12.61	3.8	3.8	47.9	47.9		
3	9.89	18.69		9.59	3.8	3.8	36.4	36.4		
4	13.19	24.9		7.18	3.8	3.8	27.3	27.3		
5	17.40	32.9		5.45	3.8	3.8	20.7	20.7		
6	25.1	47.4		3.78	3.8	3.8	14.4	14.4		
7	33.7	63.7		2.81	3.8	3.8	10.7	10.7		
8	44.3	83.8		2.14	3.8	3.8	8.1	8.1		
9	59.1	111.7		1.603	3.8	3.8	6.1	6.1		
10	78.0	147.4		1.215	3.8	3.8	4.6	4.6		
11	113.2	214		0.837	4.2	4.0	3.5	3.3		
12	152.0	287		0.623	5.0	4.8	3.1	3.0		
13	200	378		0.474	7.0	7.0	3.3	3.3		
14	267	504		0.355	9.8	10.0	3.5	3.6		
15	352	665		0.269	16.4	16.6	4.4	4.5		

SEE EXAMPLE OF CURVE ON PAGE 23.

Dynamic viscosity  $\eta$  (mPa.s) = factor  $\eta$  % . reading (%)

#### **MEAN VALUES**

Speed steps	Shear rate D (s <sup>-1</sup> )	Shear stress τ % (Pa by scale unity)	Factor  η %  (mPa.s by scale unity)	visc.	amic osity n Pa.s)
	40.50	0.4=0.40	40.00	<u> </u>	
1	10.58	0.17912	16.93	64.3	64.3
2	14.20		12.61	47.9	47.9
3	18.69		9.59	36.4	36.4
4	24.9		7.18	27.3	27.3
5	32.9		5.45	20.7	20.7
6	47.4		3.78	14.4	14.4
7	63.7		2.81	10.7	10.7
8	83.8		2.14	8.4	8.1
9	111.7		1.603	6.4	6.1
10	147.4		1.215	4.9	4.6
11	214		0.837	3.5	3.3
12	287		0.623	3.1	3.0
13	378		0.474	3.3	3.3
14	504		0.355	3.6	3.6
15	665		0.269	4.4	4.5

**CONCLUSION**: no Newtonian flow behaviour.

Dynamic viscosity at  $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  was 64.3 mPa.s to 4.4 mPa.s, dependent on the shear rate applied to the sample.

#### 4.1.7 Viscosity of liquids at 40°C

Date of analysis: June 27, 2012.

Temperature :  $40^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ .

Bowl A.

#### **DETERMINATION 1**

Spe	eed	Shear rate D (s <sup>-1</sup> )	Shear stress	Factor  η %  (mPa.s by scale unity)	Reading (%)		visce 1	amic osity 1 Pa.s)
steps	(rpm)				$\downarrow$	1	$\rightarrow$	<b>↑</b>
1	5.59	10.58	0.17912	16.93	3.8	3.8	64.3	64.3
2	7.51	14.20		12.61	3.8	3.8	47.9	47.9
3	9.89	18.69		9.59	3.8	3.8	36.4	36.4
4	13.19	24.9		7.18	3.8	3.8	27.3	27.3
5	17.40	32.9		5.45	3.8	3.8	20.7	20.7
6	25.1	47.4		3.78	3.8	3.8	14.4	14.4
7	33.7	63.7		2.81	3.8	3.8	10.7	10.7
8	44.3	83.8		2.14	3.8	3.8	8.1	8.1
9	59.1	111.7		1.603	3.8	3.8	6.1	6.1
10	78.0	147.4		1.215	3.8	3.8	4.6	4.6
11	113.2	214		0.837	4.0	4.0	3.3	3.3
12	152.0	287		0.623	4.4	4.2	2.7	2.6
13	200	378		0.474	6.0	6.0	2.8	2.8
14	267	504		0.355	8.4	8.4	3.0	3.0
15	352	665		0.269	13.2	13.2	3.6	3.6

#### **DETERMINATION 2**

Spe	eed	Shear rate D (s <sup>-1</sup> )	Shear stress $\tau$ % (Pa by scale unity)	Factor  η %  (mPa.s by scale unity)	Reading (%)		Dyna visco n (mF	osity
steps	(rpm)				$\rightarrow$	1	$\rightarrow$	<b>↑</b>
1	5.59	10.58	0.17912	16.93	3.8	3.8	64.3	64.3
2	7.51	14.20		12.61	4.0	4.0	50.4	50.4
3	9.89	18.69		9.59	4.0	4.2	38.4	40.3
4	13.19	24.9		7.18	4.2	4.4	30.2	31.6
5	17.40	32.9		5.45	4.4	5.0	24.0	27.3
6	25.1	47.4		3.78	5.0	5.0	18.9	18.9
7	33.7	63.7		2.81	5.2	5.2	14.6	14.6
8	44.3	83.8		2.14	5.4	5.6	11.6	12.0
9	59.1	111.7		1.603	5.8	6.0	9.3	9.6
10	78.0	147.4		1.215	6.2	6.2	7.5	7.5
11	113.2	214		0.837	3.8	3.8	3.2	3.2
12	152.0	287		0.623	4.2	4.2	2.6	2.6
13	200	378		0.474	6.0	6.0	2.8	2.8
14	267	504		0.355	8.6	8.4	3.1	3.0
15	352	665		0.269	13.0	13.0	3.5	3.5

SEE EXAMPLE OF CURVE ON PAGE 23.

Dynamic viscosity  $\eta$  (mPa.s) = factor  $\eta$  % . reading (%)

#### MEAN VALUES

Speed steps	Shear rate D (s <sup>-1</sup> )	Shear stress τ % (Pa by scale unity)	Factor η % (mPa.s by scale unity)	Dynamic viscosity η (mPa.s)	
	40.50	0.47040	40.00	•	, J
1	10.58	0.17912	16.93	64.3	64.3
2	14.20		12.61	49.2	49.2
3	18.69		9.59	37.4	38.4
4	24.9		7.18	28.8	29.5
5	32.9		5.45	22.4	24.0
6	47.4		3.78	16.7	16.7
7	63.7		2.81	12.7	12.7
8	83.8		2.14	9.9	10.1
9	111.7		1.603	7.7	7.9
10	147.4		1.215	6.1	6.1
11	214		0.837	3.3	3.3
12	287		0.623	2.7	2.6
13	378		0.474	2.8	2.8
14	504		0.355	3.1	3.0
15	665		0.269	3.6	3.6

**CONCLUSION**: no Newtonian flow behaviour.

Dynamic viscosity at  $40^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  was 64.3 mPa.s to 3.6 mPa.s,

dependent on the shear rate applied to the sample.

#### 4.1.8 Persistent foaming

Date of analysis: June 27, 2012.

Concentration : 2.5% v/v. Temperature :  $30^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Water : CIPAC water D.

	mL of foam after:				
Determination	10 seconds	1 minute	3 minutes	12 minutes	
1	12	12	10	ring	
2	18	12	10	ring	
Mean	15	12	10	ring	

Ring: volume < 0.5 mL.

PLANT PROTECTION PRODUCTS AND BIOCIDES PHYSICO-CHEMISTRY AND RESIDUES UNIT (U10)

#### 4.1.9 Wet sieve test

Date of analysis: July 04, 2012.

Temperature of tap water : 17.1°C. Flow rate of tap water : 4.75 L/min.

Determination	Material retained on a 75 μm test sieve (% w/w)
1	0.028
2	0.037
Mean	0.033

PLANT PROTECTION PRODUCTS AND BIOCIDES PHYSICO-CHEMISTRY AND RESIDUES UNIT (U10)

#### 10.1.c Wob juncto 63.2.a Vo 1107/2009 juncto 39.2.a Vo 178/2002

# 4.2 <u>Determination after storage for 7 days at $0^{\circ}C \pm 2^{\circ}C$ in a closed glass bottle</u>

[sample coming from the commercial bottle 1/2]

Starting of storage: June 27, 2012.

End of storage: July 04, 2012.

Date of analysis: July 04, 2012.

#### 4.2.1 Appearance of the test item

Date of analysis: July 04, 2012.

Physical state: liquid with some particles in the bottom.

homogeneous after gentle shaking but the particles fall quite quickly

Colour: dark green.

Odour:

No modification of appearance.

#### 4.2.2 Wet sieve test

Date of analysis: July 04, 2012.

Temperature of tap water : 17.1°C. Flow rate of tap water : 4.75 L/min.

Determination	Material retained on a 75 μm test sieve (% w/w)	
1	0.030	
2	0.028	
Mean	0.029	

# 5 Amendment to the study plan

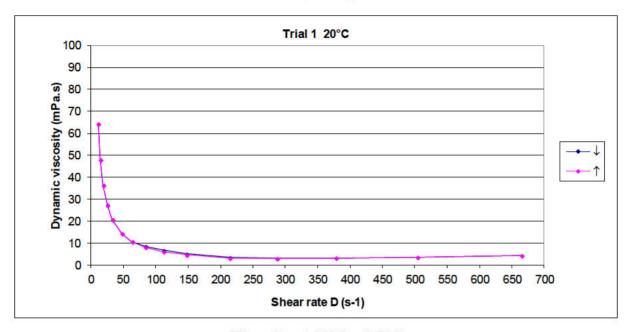
No amendment was issued for this study.

# 6 Deviation to the study plan

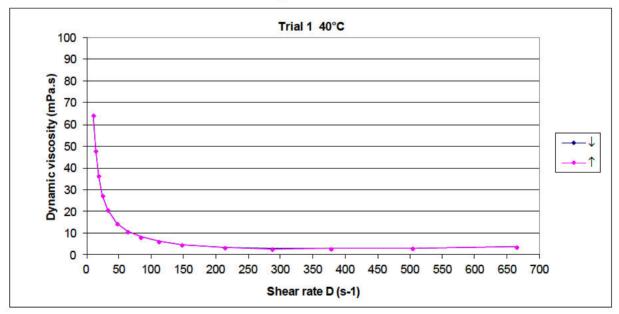
No deviation was issued for this study:

#### List of figures

#### Viscosity of liquids



Viscosity at 20°C ± 0.5°C



Viscosity at  $40^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ 

PLANT PROTECTION PRODUCTS AND BIOCIDES PHYSICO-CHEMISTRY AND RESIDUES UNIT (U10)

10.1.c Wob juncto 63.2.a en d Vo 1107/2009 juncto 39.2.a Vo 178/2002

# Appendices

Vo 178/2002	



# STATEMENT OF GLP COMPLIANCE

Assessment of conformity with GLP according to the directive 2004/9/EC. Date of inspection:  $24 \rightarrow 27$  October 2011

According to the criteria specified in the article 5, § 8 of the Royal Decree of March 6, 2002 the General Director of the Scientific Institute of Public Health, endorses on the advice of the GLP Monitorate, that the Test Facility,

Unité Physico-chimie et Résidus des Produits phytopharmaceutiques et des Biocides

Centre wallon de Recherches agronomiques Rue du Bordia, 11 5030 GEMBLOUX-Belgium Registration No : CO4

carries out physico-chemical, stability, residues, analytical chemistry and field studies with respect to the OECD and the EU principles of Good Laboratory Practices.

The Test Facility is regularly inspected within a cycle of 2 to 3 years.

Brussels, 18,01,2012



Scientific Institute of Public Heelth
Rue Juliette Wytsmanstraat 14, 1050 Brussels, BELGIUM
Tel: +32/(0)2-642.51.11 www.wiv-isp.be/glp Fax: +32/(0)2-642.50.01

Certificate of GLP endorsement from Belgian authorities