

Renewal Assessment Report

***Lecanicillium muscarium* Ve6**

- Mycotal -

Volume 3MP – B.2 Physical and chemical properties

January 2018

Rapporteur Member State: The Netherlands

Co-Rapporteur Member State: France

Version history

When	What
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Table of contents

B Summary, evaluation and assessment of the data and information

B.2	Physical and chemical properties of the plant protection product Mycotal.....	4
B.2.1	Appearance.....	4
B.2.2	Explosive and oxidising properties.....	5
B.2.3	Flammability and auto-flammability	5
B.2.4	Acidity/alkalinity and pH value.....	5
B.2.5	Viscosity and surface tension	6
B.2.6	Relative density and bulk density	6
B.2.7	Storage stability and shelf-life: effects of temperature on technical characteristics of the plant protection product.....	7
B.2.8	Technical characteristics of the plant protection product	9
B.2.8.1	Wettability	9
B.2.8.2	Persistence foaming	9
B.2.8.3	Suspensibility.....	10
B.2.8.4	Degree of dissolution and dilution stability	10
B.2.8.5	Particle size distribution, dust content, attrition and mechanical stability	11
B.2.8.5.1	Particle size distribution	11
B.2.8.5.2	Dust content.....	11
B.2.8.5.3	Attrition	12
B.2.8.5.4	Hardness and integrity.....	12
B.2.8.6	Emulsifiability, re-emulsifiability, emulsion stability	12
B.2.8.7	Flowability, pourability and dustability	12
B.2.9	Physical and chemical compatibility with other products including plant protection products with which its use is to be authorised.....	13
B.2.10	Adherence and distribution to seeds	14
B.2.11	Other studies.....	14
B.2.12	References relied on.....	16

B.2 Physical and chemical properties of the plant protection product Mycotal

Note to reader:

Information from the original DAR and/or addenda to the DAR is highlighted grey.

The in use concentration of Mycotal is 0.1% (Remark: maximum concentration in tank mix should not exceed 0.1%).

A storage stability study (6 months of storage at $4 \pm 2^\circ\text{C}$) for MYCOTAL WG in aluminium bags is ongoing and will be submitted when available. Initial results for the relevant physical and chemical properties are available in the interim report by Biagioni Angeli (2016).

Test or Study & Data point	Guideline and method	Test material purity and specification	Used methods / Results	Comments (Acceptable / Non acceptable)	GLP	Reference
B.2.1 Appearance						
Physical state and colour B.2.1/01	Visual determination	-	colour: beige, odour: not characteristic, physical state: powder	Acceptable	-	Krips, H.J., 2000
	Visual and olfactory assessment CIPAC MT 170 CIPAC MT 171	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	Appearance: Fine powder (particle size: 0.075 – 0.5 mm; essentially non-dusty) Colour: Light ivory Odour: Characteristic	Acceptable	Y	Biagioni Angeli, 2016

B.2.2 Explosive and oxidising properties						
Explosive properties B.2.2/01	-	Theoretical assessment, based on the components in the composition.	No explosive or oxidizing formulants.	Acceptable The formulation does not contain any explosive or oxidising components, therefore the formulation is considered not explosive or oxidising. See for comment MP 2.2.1	-	-
Oxidising properties B.2.2/02			See above			
B.2.3 Flammability and auto-flammability						
Flash point of the liquids formulations B.2.3/01			Not applicable			
Flammability of solid formulations B.2.3/02	-	Theoretical assessment, based on the components in the composition.	No flammable formulants.	Acceptable The formulation does not contain any flammable components, therefore the formulation is considered not (highly) flammable. See for comment MP 2.2.1	-	-
Self-heating of formulations B.2.3/03			No self-heating formulants	Acceptable The formulation does not contain any self-heating components. Therefore, the formulation is considered not self-heating.		
B.2.4 Acidity/alkalinity and pH value						
pH of the neat aqueous	pH meter and	MYCOTAL	Acidity/alkalinity not applicable.	Acceptable	Y	Krips, H.J.,

formulation B.2.4/01	glass electrode.	14.5%	MYCOTAL is a neutral preparation, and has a pH of 6.76.			2000
pH of a 1 % dilution of the solid or non aqueous formulation B.2.4/02	CIPAC MT 75.3	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	pH (1% w/v): 7.20 Acidity/alkalinity: not relevant	Acceptable	Y	Biagioni Angeli, 2016
Acidity / Alkalinity B.2.4/03			Not relevant (pH 4 – 10)			
B.2.5 Viscosity and surface tension						
Viscosity of the liquid formulation B.2.5/01	-	-	Not applicable. MYCOTAL is not a non-Newtonian fluid and not a liquid preparation. Old solid WP-formulation (Wettable powder). New solid formulation: WG-formulation (Water dispersible granules).	Not applicable Viscosity and surface tension are not applicable for solid formulations.	-	-
Surface tension of the formulation B.2.5/02			See above			
B.2.6 Relative density and bulk density						
Relative density of the liquid formulation B.2.6/01			Not applicable			
Bulk density (pour and tap) of powder or granules B.2.6/02	CIPAC MT 186	MYCOTAL WG Batch 16M07 1.2×10^{10}	Pour density: 0.499 g/mL Tap density: 0.515 g/mL	Acceptable	Y	Biagioni Angeli, 2016

		spores/g 6.8 × 109 CFU/g																						
B.2.7 Storage stability and shelf-life: effects of temperature on technical characteristics of the plant protection product																								
Stability after accelerated storage (54°C during 14 days, 8 weeks at 40°C, 12 weeks at 35°C or 18 weeks at 30°C) B.2.7/01			Not applicable																					
Effect of low temperature on stability of liquid formulation B.2.7/02			Not applicable																					
Shelf life following storage at ambient temperature B.2.7/03	Stored at 2 - 6°C for 6 months. Properties determined before and after storage	MYCOTAL 14.5%	<table><tr><th>Test</th><th>Initial</th><th>6 months at 2-6°C</th></tr><tr><td>Physical state and colour (subjective assessment)</td><td>Beige powder</td><td>Beige powder</td></tr><tr><td>Odour (subjective assessment)</td><td>No characteristic odour</td><td>No characteristic odour</td></tr><tr><td>pH of a 1% dispersion. in water (Method)</td><td>6.76 pH meter and glass electrode</td><td>6.74 pH meter and glass electrode</td></tr><tr><td>Wettability (Method)</td><td>64 s (with swirling)</td><td>34 s (with swirling)</td></tr><tr><td>Persistent foaming (Method)</td><td>10s: 5.8 mL 1 min:5.8 mL 3 min:5.8 mL 12 min 3.6 mL</td><td>n.d.</td></tr></table>	Test	Initial	6 months at 2-6°C	Physical state and colour (subjective assessment)	Beige powder	Beige powder	Odour (subjective assessment)	No characteristic odour	No characteristic odour	pH of a 1% dispersion. in water (Method)	6.76 pH meter and glass electrode	6.74 pH meter and glass electrode	Wettability (Method)	64 s (with swirling)	34 s (with swirling)	Persistent foaming (Method)	10s: 5.8 mL 1 min:5.8 mL 3 min:5.8 mL 12 min 3.6 mL	n.d.	Acceptable Although the active substance content (in % w/w, or in CFU/g) is not provided before and after storage according to Reg. (EU) 284/2013, the efficacy on whiteflies after storage of the product for 4, 12, 20, 24 and 26 weeks (reported below in study by Van Buysen, A.C., 1992) show no decrease and therefore this is found acceptable. Storage is performed in standard commercial packaging. (aluminium foil bag), provided in Vol. 3MP B-4 under point MP. 4.1. The study is executed with a	Y	Krips, H.J., 2000
Test	Initial	6 months at 2-6°C																						
Physical state and colour (subjective assessment)	Beige powder	Beige powder																						
Odour (subjective assessment)	No characteristic odour	No characteristic odour																						
pH of a 1% dispersion. in water (Method)	6.76 pH meter and glass electrode	6.74 pH meter and glass electrode																						
Wettability (Method)	64 s (with swirling)	34 s (with swirling)																						
Persistent foaming (Method)	10s: 5.8 mL 1 min:5.8 mL 3 min:5.8 mL 12 min 3.6 mL	n.d.																						

			<table><tr><td></td><td>mL foam at 200 mL CIPAC standard water D</td><td></td></tr><tr><td>Suspensibility/Suspension stability (Method)</td><td>94%</td><td>77%</td></tr><tr><td></td><td>After 30 minutes at 30.0 ± 1°C</td><td>After 30 minutes at 30.0 ± 1°C</td></tr><tr><td>Wet sieve test (Method)</td><td>1.20% Retention percentage on a 75 µm sieve</td><td>1.45% Retention percentage on a 75 µm sieve</td></tr></table> <p>The test material has been determined to undergo no significant physical or chemical change during the storage</p> <p>The test indicates that the shelf life at 2 - 6°C is at least 6 months. The method and the results are acceptable.</p>		mL foam at 200 mL CIPAC standard water D		Suspensibility/Suspension stability (Method)	94%	77%		After 30 minutes at 30.0 ± 1°C	After 30 minutes at 30.0 ± 1°C	Wet sieve test (Method)	1.20% Retention percentage on a 75 µm sieve	1.45% Retention percentage on a 75 µm sieve	<p>different ('old' WP-formulation). To be able to conclude whether this significant composition change has no major impact on the phys-chem characteristics of the new formulation, the final shelf-life study by Biagioni, A. (2016) with the after storage results should be provided, as this is a considered a data gap.</p> <p>The contaminants should also be addressed.</p>		
	mL foam at 200 mL CIPAC standard water D																	
Suspensibility/Suspension stability (Method)	94%	77%																
	After 30 minutes at 30.0 ± 1°C	After 30 minutes at 30.0 ± 1°C																
Wet sieve test (Method)	1.20% Retention percentage on a 75 µm sieve	1.45% Retention percentage on a 75 µm sieve																
	Bio-assays on whiteflies with product stored at 6°C for 4, 12, 20, 24 and 26 weeks		<p>Storage for 26 weeks at 2-6°C does not reduce efficacy of MYCOTAL on whiteflies.</p>	<p>Acceptable</p> <p>This study shows that the active substance is stable after storage up to 26 weeks.</p> <p>See for comment MP 2.2.1</p>	N	Van Buysen, A.C., 1992												
	Stored at 2 - 4°C for 6 months. Properties determined before and after storage	1.2x10 ¹⁰ spores/g, 6.8x10 ⁹ CFU/g	<p>Study is ongoing, only initial results reported, which can be found under the separate annex points.</p>	<p>The study was indicated to be available, but it was not yet submitted.</p> <p>Data gap:</p> <p>Storage stability study (shelf-life) for 6 months at 2-4°C in commercial packaging should be provided.</p>	Y	Biagioni, A. 2016												

B.2.8 Technical characteristics of the plant protection product						
B.2.8.1 Wettability						
Wettability of solid formulation B.2.8.1/01	Method not divined	MYCOTAL 14.5%	The wetting time of MYCOTAL was 64 seconds, and could not be wetted completely within 15 minutes without swirling.	The wetting time of the new formulation is <<60 sec (See study by A. Biagioni 2016 below), and therefore this study is obsolete.	Y	Krips, H.J., 2000
	CIPAC MT 53.3.1 (static) CIPAC MT 53.3.2 (dynamic)	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	Wettability static: 1.5 s dynamic: 1.0 s	Acceptable	Y	Biagioni Angeli, 2016
B.2.8.2 Persistence foaming						
Persistence of foaming of the diluted formulation B.2.8.2/01	Method not divined	MYCOTAL 14.5%	Preparation of 200 mL of the product causes foaming 5.8 mL at 10 seconds, 1 and 3 minutes, and 3.6 mL at 12 minutes.	Acceptable	Y	Krips, H.J., 2000
	CIPAC MT 47.2	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	Persistent foaming (at 0.1%): 10 sec 10 mL 1 min 8 mL 3 min 8 mL 12 min 8 mL	Acceptable The persistence of foaming at the highest in-use concentration (0.1%) after 1 min. is within limits (<60 mL), and therefore found acceptable.	Y	Biagioni Angeli, 2016

B.2.8.3 Suspensibility						
Suspensibility of water dispersible formulation B.2.8.3/01	Method not divined	MYCOTAL 14.5%	Suspensibility of a 0.1% (w/v) suspension of MY- COTAL in water is 94%.	no CIPAC water D is used, however study is obsolete as the suspensibility is tested in CI- PAC water D in study by A. Biagioni 2016 (see below).	Y	Krips, H.J., 2000
	CIPAC MT 184	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	Suspensibility (0.1% in CIPAC Water D): 93.2%	Acceptable The suspensibility at the high- est/lowest in-use concentration (0.1%) in CIPAC water D is above the limit (60%), and therefore found acceptable.	Y	Biagioni Angeli, 2016
Spontaneity of dispersion of water dispersible formulation B.2.8.3/02						
Dispersion stability of SE, OD or EG formulation B.2.8.3/03			Not applicable			
B.2.8.4 Degree of dissolution and dilution stability						
Degree of dissolution of water soluble formulation B.2.8.4/01			Not applicable			
Dilution stability of water soluble formulation B.2.8.4/02			Not applicable			

B.2.8.5 Particle size distribution, dust content, attrition and mechanical stability						
B.2.8.5.1 Particle size distribution						
Wet sieve test of water dispersible formulation B.2.8.5.1/01	Method not defined	MYCOTAL 14.5%	The wet sieve test indicated a percentage retention on a 75 µm sieve of 1.20%.	Acceptable	Y	Krips, H.J., 2000
	CIPAC MT 170 CIPAC MT 185	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	Dry sieve analysis (particle size distribution): 0.075 – 0.5 mm Wet Sieve Test (Residue at mesh – 75 µm ASTM): 0.2325%	Acceptable The wet sieve test acceptable, as < 2% is retained on a 75µg sieve.	Y	Biagioni Angeli, 2016
Size distribution of particles of powder or suspension concentrate formulation B.2.8.5.1/02	CIPAC MT 170 CIPAC MT 171 CIPAC MT 178.2	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	Dry sieve analysis (particle size distribution): 0.075 – 0.5 mm Dust content: Essentially non-dusty (15.85 mg) Friability and attrition resistance: 98.7%	Acceptable	Y	Biagioni Angeli, 2016
Nominal size range of granule B.2.8.5.1/03	Method not defined	MYCOTAL 14.5%	The median particle size L_{50} of the test item deduced from the particle size distribution was 64.2 µm.	WP-formulation, therefore no attrition and friability resistance and dust content provided.	Y	Krips, H.J., 2000
B.2.8.5.2 Dust content						
Dust content of granular formulation B.2.8.5.2/01			Not applicable			

B.2.8.5.3 Attrition						
Attrition characteristics of granules and tablets B.2.8.5.3/01			Not applicable			
B.2.8.5.4 Hardness and integrity						
Hardness of tablets B.2.8.5.4/01			Not applicable			
Integrity of tablets B.2.8.5.4/02			Not applicable			
B.2.8.6 Emulsifiability, re-emulsifiability, emulsion stability						
Emulsifiability, emulsion stability and re-emulsifiability of formulation B.2.8.6/01	-	-	Not applicable. MYCOTAL does not form an emulsion	Not applicable WP-formulation	-	-
B.2.8.7 Flowability, pourability and dustability						
Flowability of granular formulation B.2.8.7/01	CIPAC MT 172	MYCOTAL WG Batch 16M07 1.2×10^{10} spores/g 6.8×10^9 CFU/g	The sample didn't flow naturally through the 5 mm sieve. After 5 liftings: 55.18% of residue on the sieve After 20 more liftings: 15.82% of residue on the sieve	Acceptable The fact that the sample didn't flow freely and that after 20 liftings the remaining residue on the sieve was ~16%, some lumping/clumping can be expected after storage. Although the suspensibility of the fresh product is acceptable, it is possible that storage will negatively affect the suspensibility of the product, also considering the final shelf-life study of the product is not yet available. A previous version of the product	Y	Biagioni Angeli, 2016

				<p>showed a decrease in suspensibility after storage, although it remained acceptable.</p> <p>Considering the product is stored in bags, it is not expected that packaging cannot be adequately emptied. This should be reconsidered if the product is marketed in non-flexible packaging types, like tubs or buckets.</p> <p>The label should include the phrase “agitate spray solution when applying the formulation” to reduce solids in the spray solution.</p>		
Pourability of suspensions B.2.8.7/02			Not applicable			
Dustability of dustable powders after accelerated storage B.2.8.7/03			Not applicable			
B.2.9 Physical and chemical compatibility with other products including plant protection products with which its use is to be authorised						
Physical and biological compatibility of tank mixtures B.2.9/01			Not applicable			

B.2.10 Adherence and distribution to seeds						
Distribution and adhesion to seeds B.2.9.10/01			Not appliable			
B.2.11 Other studies						
			None submitted or required			

Summary and evaluation of data presented under points 2.1 to 2.9

The product MYCOTAL is a light ivory solid formulation with particle size between 0.075 – 0.5 mm and essentially non-dusty. The formulation is not explosive or oxidizing nor is it flammable or auto-flammable. The formulation does not have any corrosion characteristics as the pH of a 1% dilution is 7.2. The pour density is 0.499 g/mL and the tap density is 0.515 g/mL. All physical, chemical properties indicate that no particular problems are to be expected when used and stored as recommended on the label. The technical properties were acceptable, however the flowability was not within specs as after 20 liftings ~16% remained on the sieve. Therefore the label should include the phrase “agitate spray solution when applying the formulation” to reduce solids in the spray solution. The shelf-life study for 6 months at 2-6°C is executed with a different (‘old’ WP-formulation).

To be able to conclude whether this significant composition change has no major impact on the phys-chem characteristics of the new formulation, the final shelf-life study by Biagioni, A. (2016) with the after storage results should be provided, as this is a considered a data gap.

Results from the DAR, August 2008: Volume 3, Annex B, Point B.2.2 from the Annex I inclusion of *Lecanicillium muscarium* Ve6 (Mycotal) are highlighted in grey and are considered relevant in support for the renewal.

Data gap: A storage stability study (6 months of storage at $4 \pm 2^\circ\text{C}$) for MYCOTAL in aluminium bags is required. The applicant has indicated the study has become available, but it has not yet been submitted.

Additional note: only data to support an in-use concentration of 0.1% was provided. Based on the GAP, it appears 0.3% concentrations are also possible. Considering the product will need to be agitated during use, only the foam persistence at 0.3% may still need to be addressed. Still, the applicant should clarify at what concentration the product should be applied and provide the corresponding data on the technical properties of the formulation.

B.2.12 References relied on

Data point	Author(s)	Year	Title Owner Report No. Source (where different from owner) GLP or GEP status Published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KMP 2.1/01 KMP 2.5/01 KMP 2.7.1/01 KMP 2.7.2/01 KMP 2.7.3/01 KMP 2.7.4/01 KMP 2.7.5/01 KMP 2.7.7/01	Biagioni Angeli, E.	2016	PHYSICAL-CHEMICAL PROPERTIES OF PRODUCT MYCOTAL BEFORE AND AFTER 6 MONTHS SHELF LIFE Koppert, BT154/16 Biotechnologie BT Srl, Fraz. Pantalla, Italy GLP/GEP: yes Published: no	no	yes	New data for new formulation, not previously submitted nor evaluated	KBS
KMP 2.2.1/01	Registration department Koppert B.V.	2017	DECLARATION ON PACKAGING Koppert, not stated Koppert B.V., Berkel en Rodenrijs, NL GLP/GEP: no Published: no	no	yes	New data for existing formulation, not previously submitted nor evaluated	KBS
KMP 2.2.1/01	Krips, H.J.	2000	Determination of the appearance, pH, suspensibility, wet sieving and wettability before and after storage at 2-6°C for 6 months of MYCOTAL. NOTOX B.V., Hambakenwetering 7, 5231 DD 's -Hertogenbosch, The Netherlands Koppert Beheer BV 290993 yes unpublished report	no	no	Annex I data, already peer reviewed at EU level.	KBS