Survey on laboratories for quality control

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- Every MS is responsible for ensuring control of quality of ppp's
- Control is not harmonized.
- Activities of the Working Group on SUD (subgroup on formulation labs)
- Guidelines on its final steps – but just guidelines
- A lot of discussion on control of quality, official control, fighting with illegal and counterfeit ppp's, system of control but the topic on laboratories was missing
- Survey sent to Memer States on 27.09.2019
- 8 filled in forms from: Bulgaria, Czech Republic, Germany, Lithuania, Poland, Slovakia, Slovenia and Hungary.

SURVEY ON OFFICIAL CONTROLS OF PPPs

Member state	
Organisation	
Contact person name	
Date	

AUTHORISATION OF PPPs

Is submission of the reference sample of PPP requested during authorization process?	☐ YES	☐ NO	☐ I don´t know
Is submission of the analytical standard(s) requested during authorization process?	☐ active substance	☐ impurity	☐coformulant(s)
Are the provisions on prolongation of authorization of PPPs in the national legislation?	☐ YES	☐ NO	☐ I don´t know
Are the provisions on prolongation of expiration of PPPs in the national legislation?	☐ YES	☐ NO	☐ I don´t know

AUTHORISATION OF PPPs AND LABORATORY

Does the laboratory have access to documents and materials for authorizations?			
Composition of PPP incl. coformulants	☐ YES	☐ NO	☐ I don´t know
Analytical methods	☐ active substance	☐ impurity	☐coformulant(s)
Limit values of physico-chemical properties	☐ YES	☐ NO	☐ I don´t know

Does the laboratory have a quality system in place?	☐ ISO 17025	☐ GLP	☐ NO
Does the laboratory meet the requirements of Regulation 2017/625 for all analytical methods?	☐ YES	☐ NO	☐ I don´t know
Does the laboratory interpret the results?	☐ YES	☐ NO	☐ I don´t know
Does the laboratory analyze samples after expiration?	☐ YES	☐ NO	☐ I don´t know
Does the laboratory perform profile tests?	☐ GCMS	☐ FTIR	☐ Raman
Used methods	☐ CIPAC	☐ In house	☐ Authorised
Do you use multimethods to determine the active substance content?	☐ YES	☐ NO	☐ I don´t know

SCOPE OF PHYS. – CHEM. DETERMINATIONS (PARAMETERS)
SCOPE OF DETERMINATION OF ACTIVE SUBSTANCES
SCOPE OF DETERMINATION OF COFORMULANTS
STAFF and EQUIPMENT (chromatographs and detectors)

RESULTS of the SURVEY

Is submission of the reference sample of PPP requested during authorization process?			
Bulgaria		NO	
Czech Republic	YES (always when RMS)		
Lituania		NO	
Germany	YES		
Poland	NO		
Slovenia		NO	
Slovakia	YES		
Hungary		NO	

Is submission of the analytical standard(s) requested during authorization process?			
	active substance	impurity	coformulant(s)
Bulgaria	NO	NO	NO
Czech Republic	NO	NO	NO
Lituania	NO	NO	NO
Germany	YES	NO	NO
Poland	NO	NO	NO
Slovenia	YES	NO	NO
Slovakia	NO	NO	NO
Hungary	NO	NO	NO

Are the provisions on prolongation of authorization of PPPs in the national legislation?			
Bulgaria	YES		
Czech Republic	YES		
Lituania		NO	
Germany	no answer		
Poland	YES		
Slovenia		NO	
Slovakia	YES		
Hungary	YES		

Are the provisions on prolongation of expiration of PPPs in the national legislation?			
Bulgaria	YES		
Czech Republic	YES		
Lituania	YES		
Germany	no answer		
Poland	YES		
Slovenia		NO	
Slovakia		NO	
Hungary	YES		

Does the laboratory have access to documents and materials for authorizations?

	Composition of PPP incl. coformulants	Analytical methods			Limit values of physico-chemical properties
		active substance	impurity	coformulant(s)	
Bulgaria	YES	YES	NO	NO	YES
Czech Republic					
Lituania	NO	NO	NO	NO	NO
Germany	YES	YES	NO	In general methods available	YES
Poland	YES	YES	YES	NO	YES
Slovenia	YES	YES	YES	NO	YES
Slovakia	YES	YES	YES	YES	NO
Hungary	YES	YES	NO	NO	YES

	Does the laboratory have a quality system in place?	Does the laboratory meet the requirements of Regulation 2017/625 for all analytical methods?	Does the laboratory interpret the results?
Bulgaria	ISO 17025	NO	NO
Czech Republic			
Lituania	ISO 17025	don’t know	NO
Germany	ISO 17025	YES	YES
Poland	GLP	YES	YES
Slovenia	ISO 17025	NO	NO
Slovakia	GLP	NO	NO
Hungary	ISO 17025	NO	YES

	Does the laboratory analyze samples after expiration?	Does the laboratory perform profile tests?	Do you use multimethods to determine the active substance content?
Bulgaria	NO	NO	YES
Czech Republic			
Lituania	YES	NO	NO
Germany	YES	GCMS	NO
Poland	YES	GCMS	YES
Slovenia	YES	NO	YES
Slovakia	YES	GCMS	NO
Hungary	YES	FTIR	YES

Used methods

	CIPAC	In house	authorised
Bulgaria	x	x	x
Czech Republic			
Lituania	x	x	
Germany	x	x	x
Poland	x	x	x
Slovenia	x		
Slovakia	x	x	x
Hungary	x		

Scope of phys.-chem. determination (parameters)	
Bulgaria	emulsion characteristics of emulsifiable concentrates, suspension stability, titrimetric methods (Mancozeb, copper oxy chloride , copper sulphate - CS2, Cu)
Czech Republic	
Lituania	density, pH
Germany	color density, emulsion characteristics, foaming, suspensibility, pH, suface tension, dispersion stability, wet sieve, dustiness
Poland	CIPAC methods: MT 3 - Relative density, MT 36.3 - Emulsion stability, re-emulsion, MT 39.3 - Low temperature stability, MT 41 - Dilution stability of herbicide aqueous solutions, MT 46 - Accelerated storage procedure, MT 47.3 - Persistent foaming, MT 53.3 – Wettability, MT 160 - Spontaneity of despersion, MT 179 - Degree of dissolution and solution stability, MT 180 - Dispersion stability of suspo-emulsions, MT 181 - Solubility in organic solvents, MT 184 – Suspensibility, MT 185 - Wet sieve, MT 71 – Solubility in sodium hydroxide, MT 75 – Determination of pH values, MT 157 – Water solubility, MT 171 – Dustiness of granular products, MT 179 – Dissolution degree and solution stability, MT 186 – Bulk density, MT 191 – Acidity of alkalinity of formulations, EEC methods: Melting point A.1, Surface tension A.5, Density A.3
Slovenia	color (visual), water loss (CIPAC MT 17), density (CIPAC MT 3.3), pH (CIPAC MT 75), persistent foam (CIPAC MT 47), emulsifiability, emulsion characteristics (CIPAC MT 36), flammability (Abely-Pensky up to 50 deg. C), wet sieve test (CIPAC MT 185), suspensibility (CIPAC MT 184), wettability (CIPAC MT 184), bulk density, tap density,
Slovakia	Wettability MT 53.3, Persistent foam MT 47.3, Dispersibilty MT 174, Suspebsibility MT 184.1, Density MT 3, OECD 109, Persistent foam MT 180, Spontaneity of dispersion MT 160, Suspesibility MT 184, Dispersion stability, MT 180, Emulsion stability MT36.3, pH MT 75,3
Hungary	Quality control of the plant protection products on the Hungarian market.

Scope of determination of active substances	
Bulgaria	34 - within the scope of accreditation Azoxystrobin, alpha-Cypermethrin, Acetamiprid, Glyphosate, Deltamethrin, Dimethoate, Dithianon, Etofenprox, Imidacloprid, Lambda-cyhalothrin, Metalaxyls M, Methiocarb, Metribuzin, Metsulfuron – methyl, Nicosulfuron, Pendimethalin, Pyraclostrobin, Propamocarb hydrochloride, Cymoxanil, Thiram Tebuconazole, Thiacloprid, Thiophanate-methyl, Triadimenol, Tribenuron methyl, Fluazifop-P-butyl, Chlorpyrifos ethyl, Cycloscim, Cypermethrin
Czech Republic	
Lituania	Glyphosate*, epoxiconazole*, propiconazole*, * - accredited method Laboratory carry out tests on 42 active substances according to CIPAC methods, 44 on the basis of methods provided by the manufacturer of plant protection products, 39 – on the basis of in-house methods.
Germany	About 150 different AS with HPLC-UV, 5 AS and 2 impurities with LC/MS, about 20 AS with GC/FID and 10 impurities, 2 AS and 5 impurities with GC/MS.
Poland	Almost all of registered
Slovenia	List of 78 as
Slovakia	List of 49 as (2018)
Hungary	The active substance content is the main parameter checked during the testing.

Scope of determination of coformulants	
Bulgaria	NO
Czech Republic	
Lituania	not done
Germany	about 30 coformulants or foreign substances with GC/FID, one multimethod containing 24 substances.
Poland	about 20
Slovenia	none
Slovakia	no
Hungary	Coformulants are not determined in the plant protection products

Laboratory Staff and number of samples per year

	chemists/analyst	technical staff	Number of samples
Bulgaria	2	2	100 (official control)
Czech Republic			
Lituania	3		~ 300 (not official control)
Germany	2	5	260
Poland	5	3	310 (official control) + 30 (order from other MS)
Slovenia	5	10	36(2016), 51(2017), 44(2018)
Slovakia	3	2	143
Hungary	2		1700 samples from 913 batches (2018)

Equipment				
	Gas chromatographs	detectors	HPLC	detectors
Bulgaria	1	FID (1)	3	UV-VIS (2), DAD (1)
Czech Republic				
Lituania	5	FID (4), NPD (1), ECD (1), MSD (1)	1	UV-VIS (1), DAD (1), FLD (1)
Germany	5	FID, MSD,	6	UV-VIS, DAD, MSD
Poland	4	FID (3), MSD (1)	4	DAD (3), MS/MS (1)
Slovenia	4	FID (1), ECD (1), MSD (1)	4	DAD (3), RID (1), FLD (1), MSD (2)
Slovakia	2	FID (1), MSD (1)	2	UV-VIS (2), DAD (1), FLD (1)
Hungary	1	FID (2)	1	UV-VIS (1)

THANK YOU VERY MUCH

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