

Effectiveness of procedures for cleaning application equipment

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1. Objectives

This is important that residues of plant protection products do not remain in the spray tank after cleaning such that there is no risk to the following crops or to the operators. There is a general requirement for the procedures for cleaning of application equipment to be described in detail and for the effectiveness of these procedures to be investigated, under the 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.

A suitable protocol (small scale test) to evaluate the effectiveness of procedures for cleaning of application equipment was developed on plant protection products containing herbicides, insecticides or fungicides. This protocol is based on the CIPAC method MT 148 (pourability / rinsability) but with the determination of residues of active substance(s) after rinsing. Each analytical method was validated in term of specificity, linearity of chromatographic response, limit of quantification (LOQ), accuracy and repeatability of the procedure of rinsing.

2. Method

Sample preparation

Dispersion of formulated product at the maximum use rate in water in a 500 mL polyethylene bottle
Standing of the undisturbed bottle for 24 hours

Analysis

Pouring of the bottle
Gravimetric determination of residue after pouring
Rinsing of the bottle following the plant protection product label
Gravimetric determination of residue after rinsing
Rinsing of the bottle with an appropriate solvent for the chromatographic analysis of active substance content
Injection in HPLC-DAD or in GC-FID

Validation of the chromatographic method

Repeatability of the entire procedure
Specificity and non-analyte interference
Linearity of the chromatographic response
Limit of quantification (LOQ)
Accuracy at the level of LOQ and at the level of the residue found after cleaning

3. Examples of results on plant protection products

Class of active substance	Herbicides		Insecticides				Fungicides		
	Sulfonylurea	Isoxazolidinone	Neonicotinoid		Organo-phosphate	Pyrethroid	Dinitroaniline	Phenylamide : acylalanine	Inorganic
Type of formulation	Suspension concentrate (SC)	Wettable powder in water soluble bag (WP-SB)	Suspension concentrate for seed treatment (FS)	Soluble concentrate (SL)	Mixed formulation of CS and EW (ZW)		Suspension concentrate (SC)	Wettable powder in water soluble bag (WP-SB)	
Residue of formulated product after pouring (gravimetrically)	0.20%	0.29%	2.20%	0.05%	0.37%	0.38%	0.82%	0.98%	1.88%
Residue after rinsing									
- with water	0.07%	0.10 %	0.10 %	0.01%	0.06 %	0.07%	0.07 %	0.22%	0.10%
- with tank-cleaner	0.09%	-	-	-	-	-	-	-	-
Analytical method	HPLC-DAD	HPLC-DAD	HPLC-DAD	HPLC-DAD	HPLC-DAD	HPLC-DAD	HPLC-DAD	GC-FID	electrolytic
LOQ (% of a.s content in the spray tank)	0.0015%	0.012%	0.00007%	0.00009%	0.0013%	0.28%	0.00070%	0.0087%	0.040%
Accuracy at the LOQ	108.1 %	100.0%	96.9%	89.9%	101.1%	94.4%	101.7%	101.3%	99.3%
Accuracy at the found level	101.4%	98.7%	98.9%	92.4%	101.2%	105.1%	98.7%	113.2%	-
Residue after rinsing (% of a.s content in the tank spray)									
- with water	0.30 % (RSD 26.9%)	0.065 % (RSD 24.2%)	0.00145 % (RSD 23.8%)	0.00092% (RSD 23.6%)	0.0723 % (RSD 25.0%)	1.435 % (RSD 15.1%)	0.20 % (RSD 50.7%)	0.085 % (RSD 37.2%)	< LOQ
- with tank-cleaner	0.00038% (RSD 45.2%)	-	-	-	-	-	-	-	-

4. Conclusion

The method to evaluate the effectiveness of procedures for cleaning of application equipment has been tested and successfully validated on different types of formulations and different active substances. In general, except in the case of sulfonylurea and pyrethroids, cleaning procedures with water permit to eliminate correctly the pesticides remaining in the spray tank.

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