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European and Mediterranean Plant Protection Organization
Organisation Européenne et Méditerranéenne pour la Protection des Plantes PP 1/292 (1)

Efficacy evaluation of plant protection products
Evaluation biologique des produits phytosanitaires

PP 1/292 (1) Cleaning pesticide application equipment (PAE) – efficacy aspects

Specific scope

This Standard describes methods used to examine whether cleaning procedures are sufficient to ensure that residues of plant protection products do not remain in the pesticide application equipment (PAE) after cleaning, and that there is no unacceptable risk to subsequently treated crops.

Specific approval and amendment

First approved in 2016-09.

1. Introduction

This Standard describes a decision support scheme and suitable methods for assessing the efficiency of cleaning procedures following application of a plant protection product. The methods determine whether these procedures are feasible and sufficient to ensure that any residues of plant protection products remaining in the application equipment after cleaning do not pose a risk to subsequently treated crops.[1]

When the same machinery is used to apply another product, usually to another crop, any residues remaining in the PAE could be diluted or re-suspended, and applied onto this subsequently treated crop or land. If this crop, or a crop that is grown on the contaminated land, is sensitive to the levels of the active substance present then crop damage can occur.

The Standard not only gives information on the design of a particular trial, but provides a stepwise, tiered guide to identifying the risk of crop damage from residues, and what assessments should be carried out. This includes incorporating available information from trials conducted for other purposes, such as non-target plant testing. However, results generated with one formulation may not be wholly applicable to other formulations of the same active substance. Any mandatory mixture should be considered.

The properties of a plant protection product can be investigated in preliminary laboratory or glasshouse trials. Its behaviour and biological activity in these trials, together with the predicted effectiveness of any tank cleaning procedure, will determine whether field trials are required and, if so, their extent and type. Data generated for environmental risk assessments and efficacy studies can also be used to avoid additional testing. For many plant protection products, further testing will not be required.

Where effects are predicted from preliminary laboratory or glasshouse trials, observational trials on small plots can be carried out to examine whether any residues of the plant protection product remaining in application equipment following cleaning can cause phytotoxic effects under field conditions.

If effects are observed on sensitive crops in field trials, a more robust cleaning method will be required to minimize risks. This may include additional rinses with water or the use of a specific cleaning agent.

The conclusions of the risk assessment and any further testing (as required) should form the basis for proposed cleaning instructions on the label of the plant protection product. These may range from simple statements relating to rinsing, through to prescriptive detailed washing procedures.

2. Decision-support scheme for the risk assessment for the effectiveness of cleaning procedures

The scheme follows a sequential or tiered approach. Appendix 1 provides an overview of the decision-making scheme and determination of need for further testing.

Tier 0: If no cleaning of application equipment is required no further testing is necessary (e.g. seed treatment,

[1]This risk relates only to potential damage to the crops, and not any human health issues.

use of granules, and products in disposable ready-to-use containers).

Tier 1: If application equipment is used for subsequent treatments with other plant protection products (e.g. field sprayers) the phytotoxic properties of the plant protection product should be assessed using single-dose phytotoxicity screening data for crop plants. Testing should be at the maximum application rate on a range of representative species. Selection of species should be based on preliminary glasshouse tests, knowledge of the mode of action of the product and the potential crops that may be subsequently treated with that equipment. This data can usually be taken from non-target plant testing, as this testing nearly always includes crop plants, as well as from other greenhouse or laboratory tests, or from efficacy studies for fungicides and insecticides if a range of sensitive crops have been tested. Appendix 2 gives guidance on how to conduct such plant tests. If the plant protection product causes no symptoms of phytotoxicity on the plant species tested, no further testing is necessary.

Tier 2: If significant (>50%) phytotoxicity is observed, conduct dose–response relationships for species representing plant families for which significant negative activity has been found. If there is a clear indication that the activity via one route of exposure (soil or leaves) is far stronger than by the other, tests should be limited to that exposure route. These data can also be taken from the non-target plant (ecotoxicology) section as well as from other greenhouse or laboratory tests. See Appendix 2 for details of plant testing.

Toxicity values derived in these tests are then compared with predicted concentrations after spraying to develop the toxicity:exposure ratio (TER; calculated as the ED_{50} value divided by the amount of residue remaining in the spray tank). This information may be obtained, for example, from tests to determine the effectiveness of the cleaning procedure under Annex IIIA 4.2 under EU Regulation 284/20135. The TER is then compared with a trigger value that is based on expert judgement or derived empirically. If the TER value of the most sensitive crop is >1 (or the specific national level, if higher), no further testing is necessary.

Progression to the next tier is warranted if the safety margin is not met, while testing is stopped if the safety margin is met or exceeded.

There are several methods by which the residue in the PAE may be determined: by calculation for highly water-soluble formulations, by the use of small-scale tests (see Appendix 1 for an example) or by the testing using commercial-scale equipment.

Tier 2a: Calculation of residues left in the PAE.

When a sprayer is emptied, dilute spray solution will be retained in the sprayer. According to ISO standards,[2] for example, up to 52 L of spray solution are allowed to remain in the parts of a 2000 L sprayer with a 21 m boom. An example of how this can be used to calculate residues left in the PAE is included in Appendix 4. For *soluble active substances* it may be possible to calculate the worst-case concentration for contamination of application machinery. This is determined by taking the average amount of the original spray dilution that will be left after application. Then calculate the extent of dilution resulting from the recommended sprayer cleaning regime and subsequent refilling of the sprayer before use of the next product. The resulting value can then be used in the TER calculation to demonstrate if a sufficient level of cleaning has been achieved. It is unlikely, however, that sufficient information would be available to calculate the effect of using a cleaning agent if this is recommended during the cleaning procedure.

However, for most active substances analytical testing will be required to determine the amount of active substance remaining in the sprayer after application of the plant protection product and subsequent re-filling before the next application. The amount of active substance remaining in the PAE will be affected by the cleaning procedure adopted.

Tier 2b: Small-scale/large-scale tests

For the initial examination of the effectiveness of the cleaning procedure small-scale tests in bottles or jars can provide more consistent results than results from a full-scale test. An example protocol for small-scale jar tests is given in Appendix 3. In small-scale tests there is a greater surface area to volume ratio, increasing the likelihood that spray residues would adhere to the bottle. The small-scale containers should be made of a similar material to that used for farm-scale application machinery, such as high-density polyethylene (HDPE). The use of small-plot spray equipment is not recommended because this is commonly made of metal and the spray is often delivered using CO_2 pressure, neither of which represent normal conditions of application. Small sprayers used in domestic gardens can be suitable as these may be made of HDPE. Full-scale tests with commercial sprayers may also be used according to ISO Standard ISO 16119-2:2013 (Agricultural and forestry machinery – Environmental requirements for sprayers. Part 2: Horizontal boom sprayers).

Single products or mixtures can be tested, being added to the bottles at their highest proposed recommended concentration on the label. The bottles can then be washed using the method recommended on the label and then the final rinsate analysed for residues of the active substance(s) concerned. The solvent to be used in the final rinsate should be considered in light of the chemical properties of the active substances under test and need not necessarily be water. Some active substances are not very soluble in water or water-insoluble deposits can be formed. In these cases the final rinse which is analysed should be an organic solvent, such as isophorone, instead of water. (This is not

[2]EN/ISO 16119-2:2013.

unrepresentative of field conditions because organic solvents can often be found in pesticide formulations). If the product is contained in water-soluble packaging then an appropriately sized piece of the packaging should be added to the container at the beginning of the test.

As mentioned previously, the methodology and results of such tests are often presented under Annex IIIA 4.2 under EU Regulation 284/20135.

The amounts of active substance remaining are then used to derive a TER value. This is calculated by comparing the biological activity (the ED_{50} value for each plant species) to the residue in the sprayer in order to predict the likelihood of effects on subsequently sprayed crops.

If the TER value of the most sensitive crop is >1 (or the specific national level, if higher), no further testing is necessary. If the TER value is <1 it is likely that damage will occur when a sensitive crop is subsequently treated. Field testing will be necessary to examine the extent of effects.

If, following Tier 2, it is concluded that that even with the cleaning procedure there is a risk of damage, the 'small-plot' field tests described below should be conducted with an effective cleaning procedure. These could include treatments without the proposed cleaning procedure and with the proposed cleaning procedure. If the phytotoxic effects do not result in significant reductions in biomass no further testing is necessary. However, if phytotoxic symptoms lead to biomass reductions, additional refinements to the cleaning procedure will be required until an effective cleaning procedure can be derived. The resulting procedure that gives rise to an effective cleanout, should be described on the label of the plant protection product.

Tier 3: If phytotoxic effects are still possible then a series of semi-field or field tests is necessary as described below. The first step would be to undertake field screening (which may be unreplicated) pre-emergence and/or post emergence over a sufficient test period, using the crop species known to be the most sensitive following testing at *Tiers 1* and *2*. The doses applied should be representative of the likely residues remaining in the PAE following the cleaning procedure and the crop growth stages likely to be present at the proposed time of application of the plant protection product, and phytotoxic effects (observed as visible plant damage or shoot weight reduction) should be assessed. Any crop species found to be sensitive (showing phytotoxic effects) following this testing would need to undergo further field testing.

In the following step 'small-plot' field tests should be conducted using the *most sensitive representative crops* that may be treated. This should use doses representative of the residues remaining in the PAE following a cleaning procedure and crop growth stages likely to be present at the proposed time of application of the plant protection product, assessing both phytotoxic effects (observed as visible plant damage or a reduction of shoot weight) and effects on biomass. If the phytotoxic effects do not result in significant reductions in biomass, no further testing is necessary. However, if phytotoxic symptoms lead to biomass reductions, additional refinements to the cleaning procedure will be required until an effective cleaning procedure can be derived.

3. Field trials

To further refine the results of small-scale testing, field trials can be carried out using full-size or scaled-down farm application machinery and representative crops. It is recommended that these tests are only used to test methods that have been designed following smaller-scale tests. Products are mixed as recommended in the test apparatus and disposed of by spraying though representative nozzles. The test apparatus is then cleaned according to label recommendations and the tank refilled. The tank's contents at this stage are analysed to validate the doses that should be used in field testing.

This method is generally acceptable as long as the large-scale test apparatus is representative of sprayers that will be used in practice, the recommended cleaning technique is tested and the crops used in the test can be shown to be representative and sensitive to the active substance(s) in question. It allows plots of sensitive crops to be treated so that the extent and duration of any damage can be recorded and, if necessary, the yield measured.

As a range of different crops may be subsequently treated, all the trial parameters should be consistent with the specific Standard for the named crop.

3.1 Experimental conditions

3.1.1 Selection of crop and cultivar

The trial should be performed on crops that are normally sprayed at a similar time to the crop(s) specified for the intended use. According to the proposed use and time of application of the plant protection product, the crops may already have been planted (post-emergence) or be in the process of germination (pre-emergence). For each crop, the selected varieties should include the most common ones.

3.1.2 Trial conditions

The trial should be set up in the field. Cultural conditions (e.g. soil type, fertilization, tillage) should be uniform for all plots of the trial and should conform to local agricultural/horticultural practice. The preceding crop should be recorded as well as any plant protection products used on or after it. Sites treated with plant protection products known to have phytotoxic effects on the test crop should be avoided.

The trial should form part of a series carried out in different regions with distinct environmental conditions and preferably in different growing seasons (see EPPO Standard

PP 1/181 *Conduct and reporting of efficacy evaluation trials, including good experimental practice*).

3.1.3 Design and lay-out of the trial

Treatments: test product(s) and untreated control, arranged in a suitable statistical design. Plots and replicates should be as specified in the specific EPPO Standard PP 1 *Efficacy evaluation of plant protection products*.

For further information on trial design see EPPO Standard PP 1/152 *Design and analysis of efficacy evaluation trials*.

3.2 Application of treatments

3.2.1 Test product(s)

The product(s) under investigation should be the named formulated product(s) and should be applied as specified for the intended use (e.g. with an adjuvant): see EPPO Standard PP 1/181 *Conduct and reporting of efficacy evaluation trials, including good experimental practice*.

3.2.2 Mode of application

Applications should comply with good standard practice.

3.2.2.1 Type of application. The type of application should be as specified for the intended use.

3.2.2.2 Type of equipment. Application(s) should be made with suitable equipment providing an even distribution of product on the whole plot or accurate directional application where appropriate. Factors such as volume rate, operating pressure and nozzle type, should be chosen in relation to the intended use.

3.2.2.3 Time and frequency of application. The product should be applied once. The date of application should be as specified for the intended use. The state (emergence, growth stage) of the crop and date of each application should be recorded. If crop types or cultivars can be treated at a range of timings in the year, then application to the crop(s) should be done over a range of representative timings.

3.2.2.4 Doses and volumes. The product should be applied at the doses likely to be present following use of the specified cleaning procedure and after dilution when refilling the sprayer.

The dosage applied should normally be expressed in kg (or L) of formulated product per hectare and the volume of water (L ha^{-1}) should also be recorded for sprays. It may also be useful to record the dose in g of active substance per ha or the concentration (%).

3.2.2.5 Data on other plant protection products. If other plant protection products (or any biocontrol agents) have to be used they should be applied uniformly to all plots, separately from the test product. Possible interference with these should be avoided.

3.3 Mode of assessment, recording and measurements

3.3.1 Meteorological and edaphic data

3.3.1.1 Meteorological data. Around the date of application (e.g. 7 days before and 7 days after the application), meteorological data which is likely to affect the development of the crop and/or the performance of the active substance should be recorded. This normally includes at least precipitation and temperature. All data should preferably be recorded on the trial site, but may be obtained from a nearby meteorological station. The location and distance of the meteorological station from the trial site should be noted.

On the date of application, meteorological data should be recorded which is likely to affect the quality and persistence of the treatment, and should preferably be recorded on the trial site. This normally includes at least precipitation (amount in mm and the time between treatment and start of precipitation), temperature (average, maximum and minimum in °C), wind speed and direction (at the trial site during application) and relative humidity. Record whether leaves are wet at the time of treatment. Any significant change in weather should be noted.

Throughout the trial period, extreme weather conditions such as severe or prolonged drought, heavy rain, late frosts, hail, etc., which are likely to influence the results, should also be reported. All data concerning irrigation should be recorded, as appropriate.

3.3.1.2 Edaphic data. The following characteristics of the soil should be recorded: pH, organic matter content, soil type (according to a specified national or international standard), moisture (e.g. dry, wet, waterlogged), seed-bed quality (tilth, if appropriate) and fertilizer regime.

3.3.2 Type, time and frequency of assessment

The state of the crop at application and assessment should be recorded. This usually includes the BBCH growth stage and general condition of a crop.

3.3.2.1 Type. The test crops should be examined for the presence of phytotoxic effects. In addition, any positive effects should be noted. The type and extent of such effects should be recorded and, if there are no effects, this fact should also be recorded.

Phytotoxicity should be scored as follows:

(1) if the effect can be counted or measured, it should be expressed in absolute figures
(2) in other cases, the frequency and degree of damage should be estimated. This may be done in either of two ways: each plot is scored for phytotoxicity by reference to a scale, or each treated plot is compared with an untreated plot and % phytotoxicity estimated.

In all cases, unintended effects to the crop should be accurately described (stunting, chlorosis, deformation, delay in emergence, etc.). For further details see EPPO Standard PP 1/135 *Phytotoxicity assessment*, which contains sections on individual crops and specific EPPO Standards in series PP 1.

The assessment relates to damage due to both the test product and to other influences. The latter are determined in the untreated plot. It is important to consider the possible interaction between phytotoxicity and stress factors (damage due to cultural operations, lodging, attacks of pests, prolonged heat or cold, etc.).

3.3.2.2 Time and frequency. As a guide, the following observation times may be chosen. In the case of successive applications it is necessary to make an assessment before each application. An assessment before the first application is only needed if the biomass of the crops shows clear visual differences between individual plots.

(1) For pre-emergence application

1st assessment: during emergence (in order to be able to assess any delay in emergence or thinning, preferably determined by counting the plants).

2nd assessment: at the end of emergence.

3rd assessment: at the 2–3 leaf stage.

(2) For post-emergence application

1st assessment: at application of the test product to make sure that the crop shows no abnormal symptoms before beginning the trial.

2nd assessment: 1–2 weeks after application. Numbers of crop plants present should be estimated.

3rd assessment: 3–4 weeks after application.

Further phytotoxicity assessments should be made during the life of the crop.

3.4 Quantitative and qualitative recording of yield

Where trials are harvested, the method of recording yield or components of yield should be appropriate to the test crop. This is described for some crops in EPPO Standard PP 1/135 *Phytotoxicity assessment*. See specific EPPO Standards in series PP 1 if the test product is a herbicide or growth regulator.

4 Results

The results should be reported in a systematic form and the report should include an analysis and evaluation. Original (raw) data should be available. Statistical analysis should normally be done using appropriate methods which should be indicated. If statistical analysis is not done this should be justified. See also EPPO Standard PP 1/152 *Design and analysis of efficacy evaluation trials*.

References

OECD (2006a) *Guidelines for Testing of Chemicals*. Section 2: Effects on Biotic Systems. Test No. 208: Terrestrial Plant Test: Seedling Emergence and Seedling Growth Test. OECD, Paris (FR).

OECD (2006b) *Guidelines for Testing of Chemicals*. Section 2: Effects on Biotic Systems. Test No. 227: Terrestrial Plant Test: Vegetative Vigour Test. OECD, Paris (FR).

ISO 16119-2:2013Agricultural and forestry machinery – environmental requirements for sprayers – Part 2: Horizontal boom sprayers. Available at: http://www.iso.org/iso/catalogue_detail.htm?csnumber=55706 (last accessed 28 June 2016).

ISO 22368-:2004 Crop protection equipment – test methods for the evaluation of cleaning systems – Part 1: Internal cleaning of complete sprayers. Available at: http://www.iso.org/iso/home/store/catalogue_tc/catalogue_detail.htm?csnumber=34976. Part 2: External cleaning of sprayers available at: http://www.iso.org/iso/iso_catalogue/catalogue_tc/catalogue_detail.htm?csnumber=34978.
Part 3: Internal cleaning of tank available at http://www.iso.org/iso/home/store/catalogue_tc/catalogue_detail.htm?csnumber=36321 (last accessed 28/06/2016).

Appendix 1 – Decision-support scheme for the risk assessment for the effectiveness of cleaning procedures

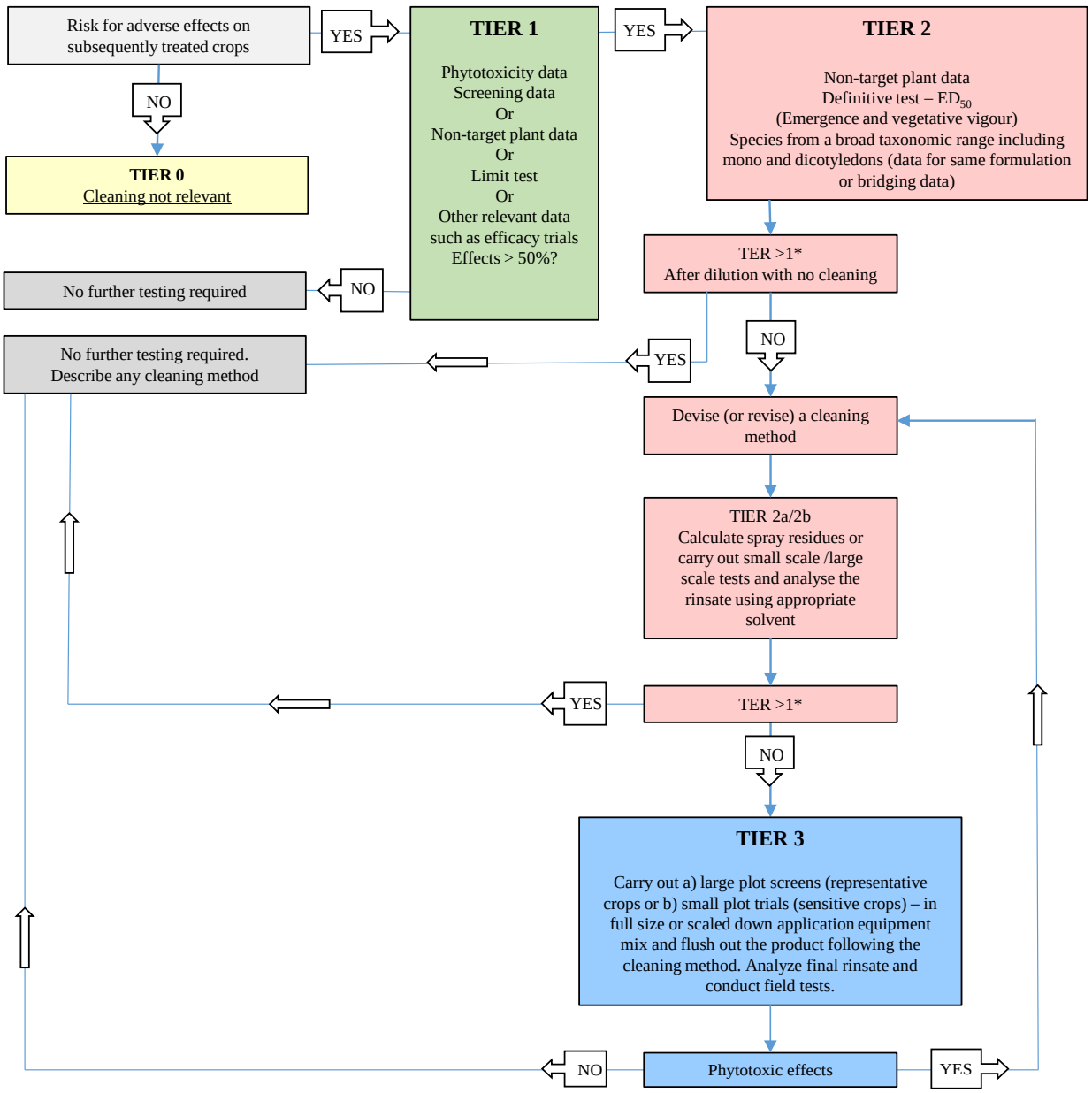

Fig. 1 Decision-support scheme for the risk assessment for the effectiveness of cleaning procedures. *TER = ED_{50} Residue in the PAE (calculated/actual).

Appendix 2 – Method for screening the sensitivity of crop species to active substances

Test plants are sown in pots. Test species are chosen to be representative of the range of crops present at the time of application of the plant protection product (proposed use) and may also be adjacent crops. The bioassay should also include species already demonstrated to be very sensitive to the active substance. Test plants should be sown so that sufficient numbers of plants emerge for the purpose of the test. To test post-emergence activity the plants can be

transplanted. The test should be replicated and randomized, and plants should be grown in controlled conditions so that growing conditions are the same for all plants. An assessment should be made of emergence (for pre-emergence testing only) and all aspects of growth of the test plants in the treated pots compared with untreated plants.

Plant weight should be measured after a sufficiently long interval for effects of the active substance to be seen; this depends on the mode of action of the active substance.

For further information see OECD (2006a,b).

Appendix 3 – Protocols for small-scale tests to evaluate cleaning of pesticide application equipment (PAE)

This appendix outlines an example protocol for small-scale tests to evaluate the cleanout efficiency of a tank cleaner and/or a specific tank mix using a single full-tank cleanout procedure. Tests should be run in triplicate and the results averaged. Alternative procedures may be substituted where appropriate. The resulting procedure that gives rise to an effective tank cleanout, should be described on the label of the plant protection product in line with national requirements.

1. Tank mix preparation

300 mL of CIPAC water D is placed into a 400 mL beaker and stirred and the appropriate amount of the pesticide is added. After 2 min of stirring, the appropriate amount of a tank partner is added, if desired, and stirring is continued for an additional 2 min. 100 mL aliquots are poured into three polyethylene bottles (about 45 mm in diameter and 90 mm high); these are capped and allowed to stand at room temperature overnight.

2. Tank cleaning procedures

Generic cleanout, with tank cleaner

(i) Each polyethylene bottle is subjected to a standard cleanout procedure.
(ii) The bottle is inverted twice (and shaken if needed) to re-suspend any settled material. The tank mix is then discarded.
(iii) 10 mL of tap water is added. The bottle is inverted twice, and the rinsate is discarded.
(iv) 100 mL of tap water is added with the appropriate amount of a tank cleaner. The bottle is inverted twice and allowed to stand for 15 min. The bottle is then inverted twice and the liquid is discarded.
(v) Repeat Step (iii).
(vi) 10 mL of acetonitrile is added and the bottle is shaken to coat all surfaces. The acetonitrile is used to extract residual pesticide from the bottle surfaces. Alternate solvents can be used if appropriate.
(vii) The acetonitrile sample is analysed for pesticide content.

Alternative cleanout procedure, with tank cleaner

(i) Each polyethylene bottle is subjected to a standard cleanout procedure.
(ii) The bottle is inverted twice (and shaken if needed) to re-suspend any settled material. The tank mix is then discarded.
(iii) 100 mL of tap water is added with the appropriate amount of a tank cleaner. The bottle is inverted twice and allowed to stand for 15 min. The bottle is then inverted twice and the liquid is discarded.
(iv) 10 mL of acetonitrile is added and the bottle is shaken to coat all surfaces. The acetonitrile is used to extract residual pesticide from the bottle surfaces. Alternative solvents can be used if appropriate.
(v) The acetonitrile sample is analysed for pesticide content.

Possible cleanout procedures, without tank cleaner

(a) Single-rinse procedure:
(i) The bottle is inverted twice (and shaken if needed) to re-suspend any settled material. The tank mix is then discarded.
(ii) 10 mL of tap water is added. The bottle is inverted twice, and the rinsate is discarded.
(iii) 10 mL of acetonitrile is added and the bottle is shaken to coat all surfaces. The acetonitrile is used to extract residual pesticide from the bottle surfaces. Alternative solvents can be used if appropriate.
(iv) The acetonitrile sample is analysed for pesticide content.

(b) Double-rinse procedure:
(i) The bottle is inverted twice (and shaken if needed) to re-suspend any settled material. The tank mix is then discarded.
(ii) 10 mL of tap water is added. The bottle is inverted twice, and the rinsate is discarded.
(iii) Repeat Step (ii).
(iv) 10 mL of acetonitrile is added and the bottle is shaken to coat all surfaces. The acetonitrile is used to extract residual pesticide from the bottle surfaces. Alternative solvents can be used if appropriate.
(v) The acetonitrile sample is analysed for pesticide content.

(c) Triple-rinse procedure:
(i) The bottle is inverted twice (and shaken if needed) to re-suspend any settled material. The tank mix is then discarded.
(ii) 10 mL of tap water is added. The bottle is inverted twice, and the rinsate is discarded.
(iii) Repeat Step (ii).
(iv) Repeat Step (ii) again.

(v) 10 mL of acetonitrile is added and the bottle is shaken to coat all surfaces. The acetonitrile is used to extract residual pesticide from the bottle surfaces. Alternative solvents can be used if appropriate.
(vi) The acetonitrile sample is analysed for pesticide content.

3. Analysis

The acetonitrile sample is analysed by high-performance liquid chromatography (HPLC) for pesticide active substance (a.s.) using an external standard. The results of the 3 bottles are averaged. Values should generally be reported down to the limit of detection/determination (at least 0.5–1 ppm with a linear range up to 300–500 ppm) and the limit of determination/detection should be quoted in the results. Values greater than the highest standard or less than the lowest standard are reported as such. Where a tank mix is being considered, if a case can be made that one of the tank-mix partners poses no risk to crops then this may be sufficient and this partner may not need to be analysed.

4. Evaluation

To calculate the '% Removed' from the bottle:

$$\%\text{Removed} = \left(1 - \frac{\text{mg pesticide a.s. recovered}}{\text{mg pesticide a.s. charged}}\right) \times 100.$$

5. Notes

Any deviations to the procedure(s) should be noted and fully justified in the report.

The selection of CIPAC water D, the tank cleaner to be proposed, the tank mixtures chosen for the test and the use of acetonitrile as the 'final extraction agent' should be critically examined (to determine their appropriateness) before undertaking the test.

This methodology is designed to be conservative. Residues generally adhere to the surfaces of a container or spray tank. The surface area to volume ratio in this small-scale test is at least 20 times greater than the surface area to volume ratio in a commercial spray tank.

In the case of tank mixes consider the biological activity of the tank-mix partners. Analysis is required only for active substances that pose a risk of damage to crops.

As analysis is via HPLC, the relevant methods of analysis for the substance(s) under test are required for accurate determination of residues. If the relevant methods of analysis are unavailable for the substance(s) under test (e.g. the tank-mix partner) it may be necessary to request information from the approval holder on the appropriate method of analysis for that test substance. Where this is not possible, the alternative action taken should be stated in the report (e.g. non-standard method of analysis used, or no analysis for that test substance undertaken).

Acknowledgements

This protocol for small-scale tests to evaluate cleaning of PAE is based on that kindly provided by DuPont (UK) Ltd. for analysis of highly active herbicides (including ALS inhibitors, e.g. sulphonyl ureas).

Appendix 4 – 4. Example calculation for estimating residues within PAE based on ISO 16119

Up to 2.6% of the spray solution will remain in the PAE following application (according to ISO 16119)

Assuming a dose of 3.0 L ha^{-1} and a product containing 500 g L^{-1} of active substance (a.s.) the following would therefore apply:

Calculations:		
Amount of a.s. in 1000 L sprayer (assuming 100 L ha^{-1} water)	1000/100 = 10 × 3.0 L ha^{-1} = 30 L ha^{-1} applied at 500 g a.s. L^{-1} = 15 000 g a.s. ha^{-1}	15 000 g a.s.
Amount left after spraying (2.6%)	15 000 × 2.6%	390 g a.s.
Amount left after 1st stage of washout procedure (2.6%)	390 × 2.6%	10.14 g a.s.
Amount left after 2nd stage of washout procedure (2.6%)	10.14 × 2.6%	0.26364 g a.s.
Amount after re-filling sprayer (1000 L)		0.26364 g a.s.
Dose applied (at 400 L ha^{-1}) to 2.5 ha	0.26364/2.5	0.105456 g a.s. ha^{-1}

In the table above, the maximum dose liable to be applied following a two-stage cleaning procedure is 0.105456 g a.s. ha^{-1}. This dose is then used to calculate the toxicity:exposure ratio.