AUG. 2020

TECHNICAL BROCHURE

STANDARD OPERATING PROCEDURE (SOP) FOR THE IMPLEMENTATION OF PLANT PROTECTION PRODUCT (PPP) FIELD TRIALS

COLEACP

This publication has been prepared by the COLEACP as part of co-operation programmes funded by the European Union (European Development Fund – EDF), the Agence Française de Développement (AFD) and the Standards and Trade Development Facility (STDF).

This publication is an integral part of a COLEACP collection, which is made up of educational and technical tools and materials. All of them are suited to different types of learners and beneficiaries and levels of education found in agricultural supply chains, production and sales.

This collection is available online for COLEACP members and beneficiaries.

AFD AGENCE FRANÇAISE DE DÉVELOPPEMENT

STDF

COLEACP

CONTENT

1. Introduction 5

2. Description of roles and responsibilities 5

3. Distribution, receipt and handling of plant protection products 7

4. Spray application equipment - recommended equipment, handling maintenance and calibration 7

5. Number of trials 11

6. Number of replicates 11

7. Trial design 12

8. Untreated control 13

9. Reference product 13

10. Test treatments 13

11. Location/site selection for trial 14

12. Plot size and shape 14

13. Marking the plots 15

14. Application of PPP(s) 15

15. Data collection 17

16. Content of trial report 19

17. SOP Summary check sheets 21


BB JUNIOR
OPEN 2017

1. Introduction

This document describes in detail the requirement (Standard Operating Procedure - SOP) for implementing both efficacy and residues field trials for plant protection products (PPPs). Much of the procedures are the same for both types of trials, but – especially with sampling – there are difference. This is indicated throughout the text when necessary. Also included are SOP summary checklists for both types of trial that are based on the SOPs used by the COLEACP.

This document is a generic one and can be used to develop specific SOPs for trials. While the SOP summary checklists provide an easy to use document, the detail in the text describes how PPP trials should be planned, and how PPP(s) should be handled, applied and samples/data managed in order to obtain the required results and therefore must be closely followed.

2. Description of roles and responsibilities

For all types of field trials, COLEACP normally contracts a (local) organization for implementation, which would have trained staff, equipment and experience in pesticide field trials. This is often referred to as the testing unit. The testing unit should be managed in a structured and transparent manner and be able to ensure that trials are run according to Good Experimental Practice (GEP). The organisation should have:

- a manager;
- a study director;
- a chief trial technician;
- other staff, including other trial technicians and labourers, as needed.

In some cases, an individual may have more than one role e.g. Manager and Study Director, but still needs to cover the relevant responsibilities outlined below.

The responsibilities of each are listed below.

2.1 Manager (or management unit)

The management has overall responsibility over the testing unit and must ensure that the testing unit has:

- scientific and technical staff with the education, training, technical knowledge and experience necessary to conduct properly the assigned tasks of the field trial;
- the facilities and equipment (correctly maintained and calibrated) necessary to conduct the trials properly;
- suitable trial areas and storage facilities at its disposal;
- agreed SOPs, trial plans and safety regulations that are available, are in compliance with local regulation and norms and are followed by relevant staff, all staff of which know their responsibility;
- oversight to ensure that the quality of the work is consistent with the type, extent and intention of the trials;

- records of calibration schedules, raw data and the final reports, etc. that is available to COLEACP, or other agreed authority, on request;
- the manager liaises with, and provides reports to, COLEACP, as required.

2.2 Study director

The study director is responsible for:

- preparation and maintenance of SOPs required for the specific trial and based on this document;
- planning, testing and reporting on the trial;
- ensuring that the trials are conducted in accordance with the procedures specified in the trial plan and the SOPs and in accordance with local regulations and norms;
- ensuring that all required data and any changes to the trial plan are well documented
- filing data.

2.3 Chief trial technician

The chief trial technician is responsible for:

- receipt and handling of the PPP test samples;
- the individual trial being taken care of in accordance with the trial plan and the SOPs;
- all the deviations being documented and reported to the study director;
- all products and samples being treated with the necessary care.

2.4 Other staff

Other staff are responsible for:

- undertaken the trial according to their assigned roles and in accordance with the SOP and trial plan;
- recording all information/data required in the agreed format and submitting to the chief trial technician;
- recording and informing the chief trial technician of any deviations from the SOP and trial plan.

Trial plan and protocols should be sufficiently detailed so that third parties can understand the contents of the study and verify its quality. Trial protocols should be acceptable to the relevant registration authorities (as required) and they should follow national or regional standards, where available. SOPs must be defined at least for the following activities: distribution, receipt and handling of PPP(s); pesticide application; calibration and use of application equipment, use of weighing apparatus/measuring equipment, sowing, planting and harvesting equipment; maintenance of equipment; sampling of PPP(s) (for quality control); recording of results. SOPs and protocols should be made available to all relevant personnel involved in the trials.

3. Distribution, receipt and handling of plant protection products

Handling, storing and any destruction of the trial material must comply with the SOPs of national and local regulations and adhering to best practices.

3.1 Distribution and receipt

COLEACP will arrange the acquisition of the PPP(s) from the registrant, which will be delivered to the testing unit. The trial technician is responsible for filing out the information that identifies the test PPP (name, % active ingredient, formulation, lot/batch number), as well as the condition, quantity received and the date of receipt. The formulation to be used should be the same of that which will be used in normal field use. It is recommended to photograph the label and container of the received material. The marking of sample substances must correspond to the nomenclature used in the trial plan.

3.2 Storage

Store PPP(s) in a secure, clean, dry area, not in direct sunlight and below 20 °C or according to the manufacturer's instructions (whichever is lower). Storage temperatures should be monitored and documented. Liquid products of less than 10 ml must not be stored for more than 7-10 days, due to photosensitivity and possible evaporation, unless the container is made of High Density Polyethylene (HDPE). The test substance(s) should be stored under appropriate conditions for the study duration and applied soon after preparation or mixing. Some authorities require that the test substance(s) is retained until the final study report is completed.

4. Spray application equipment - recommended equipment, handling maintenance and calibration

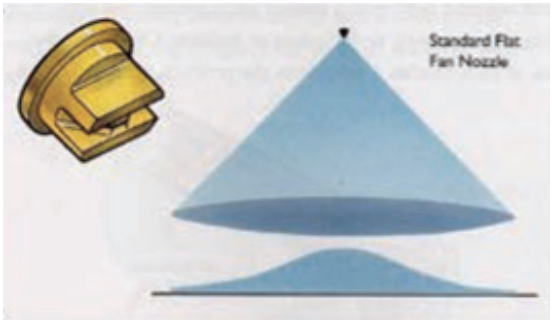
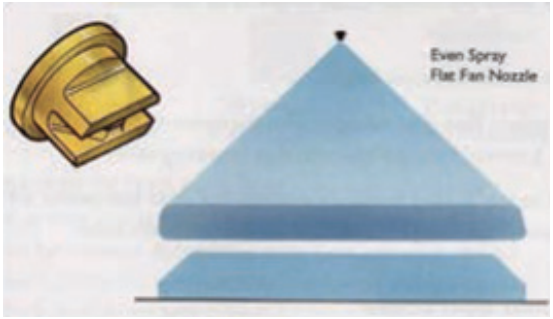
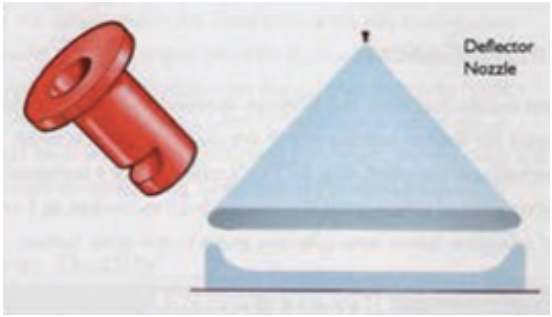
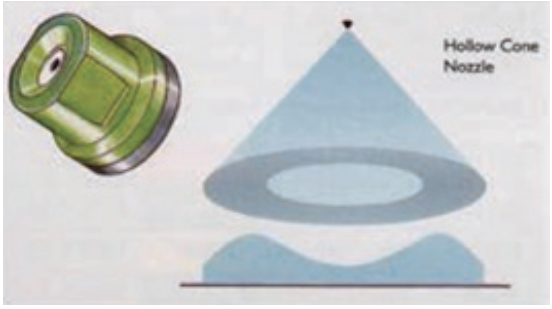
4.1 Equipment type

The equipment and method of application of the PPP(s) should normally be the same or similar to the one to be recommended on the label and that is generally used for pest control in country/region and the target crop/pest. For field crops in ACP countries this is likely to be a lever-operated backpack sprayer for field crops and for high (tree) crops a motorized backpack sprayer or mistblower. However, especially in small-scale trials hand-held equipment may be used, even if the product is later to be applied using vehicle-mounted equipment. Application rates (both the product dose and, for sprays, the volume application rate) and dilution ratios (as appropriate) should be the same as recommended on the product label, except if the influence of the dose on efficacy is being evaluated. Treatments should be timed as recommended on the label. Where repeated application is recommended, the same application frequency should be tested (different application frequencies may be used in trials to determine the optimum interval between applications).

With lever-operated sprayers, a control valve (preferably) or pressure gauge should be fitted in the lance as closely to the nozzle as possible. The lever should be operated evenly with a full stroke to maintain a uniform pressure – this needs to be practiced by the operator. Pressure gauges or control valves are, or should be, included with other hydraulic sprayers.

Nozzles should be selected according to standard recommendations (Figure 1).

Figure 1 Main types of nozzle for application of pesticides

Type of nozzle	
	**Flat fan nozzle:** used on boom sprayers where spray from one nozzle has to overlap spray from adjacent nozzles to give uniform coverage across the boom.
	**Even spray flat fan nozzle:** used as a single nozzle on a knapsack sprayer. Gives uniform deposit of spray. Air-induction versions reduces spray drift.
	**Deflector (flood jet) nozzle:** used on knapsack sprayers. Gives wide coverage and produced large droplets
	**Hollow cone nozzle:** can be used on a knapsack or boom sprayer. Produces fine spray.

Recommended nozzles for different pesticide categories are given below. It can be seen that if only one nozzle type can be used, the fan nozzle provides a compromise suitable for all. Furthermore, air-induction nozzles reduce drift and are thus good for small plots.

Table 1: Use of nozzles

Type of nozzle	Herbicide (1 bar)	Insecticide (3 bar)	Fungicide (3 bar)
Cone (small to medium sized drops)	*	***	***
Fan (medium-sized drops)	**	**	**
Deflector (large drops)	***	*	*

*unsuitable
**acceptable
***most acceptable

Generally, fan nozzles with flow rates of 0.2 – 0.5 l/min should be used, ideally air-induction versions, unless otherwise recommended by the manufacturer of the pesticide being used.

4.2 Sprayer maintenance

All application equipment should be well-maintained and in good working order, having been checked for any leaks. It should have been thoroughly cleaned after the last time it was used, and also prior to the current use. Cleaning should involve triple-rinsing with water, including passage of water through the hoses and lance. Nozzles should be dismantled and the individual components (filter, tip and cap) cleaned and replaced. Similarly, all other filters should be removed, cleaned and replaced, as well as removable seals e.g. inside the lid. Note that with plastic sprayers some of the chemical, albeit very small, will be absorbed by the plastic and will leach out. This may be an issue (risk of cross contamination) for example with some highly active herbicides, and can be reduced by decontamination with charcoal and replacement of hoses. Alternatively, new equipment can be used for each trial.

Visually inspect pumps, hoses, pipes, fittings, regulators, gauges, O-rings, screens, filters, and tanks for obvious wear or potential leaks and repair or replace as necessary.

Basic maintenance can be undertaken by the user for lever-operated and other hydraulic sprayers. Maintenance of mistblowers should be undertaken by professional mechanics and in accordance with the manufacturer's instructions.

Application equipment should be properly calibrated to give the intended application rate and droplet spectrum. Details of the sprayer, its operating pressure, nozzle type, forward speed and volume application rate need to be specified, as well as dose, spray concentration, and formulation used. To confirm the quality of the treatments, the actual applied volume application rate is also determined and reported, rather than only the rate based on the calibration.

4.3 Calibration

Normally, the concentrated product is diluted in water and sprayed onto the crop. The amount of diluted (mixed) spray liquid applied per hectare is known as the volume application rate. For backpack sprayers, optimum volume application rate are presented in Table 2.

Table 2 General volume application rate for field crops

Target type	Volume application rate (litres/hectare, l/ha)
Bare soil and small plants e.g. herbicide application to soil or small weeds; insecticide or fungicide application to young crops	150 – 200 l/ha
Medium sized weeds/crop	200 – 250 l/ha
Dense crop	300 l/ha

Note: The pesticide label may give a recommendation for the volume application rate to use.

The volume application rate is determined by the type of sprayer, type of spray nozzle, pressure at which the spray is applied, width of the spray (in cotton this will often be the distance between plant rows) and speed that the sprayer/spray operator passes through the crop. The sprayer is calibrated under the conditions that it is going to be used, which will account for all of the above. The simplest way to do this is:

Spray a known volume of water into the target (either crop or weed) and measure the area it covers

Or

Have a known area of target and measure how much water is necessary to cover it.

The application volume (litres per hectare) is calculated as:

$$\text{Application volume (litres per hectare)} = \frac{\textit{10,000 (one hectare in square metres) X Number of litres sprayed}}{\textit{Area sprayed (in hectare)}}$$

If the volume is very different from the amounts shown in Table 2 either change the nozzle or adjust your walking speed.

The applicator can then determine the volume required to spray the area to be treated:

$$\text{Volume to treat required area (l)} = \text{Application volume (l/ha) x area to be treated (ha)}$$

And the number of sprayer loads required:

$$\text{Number of spray loads} = \frac{\textit{Volume to treat required area (in litres)}}{\textit{Sprayers capacity (in litres)}}$$

The amount of formulated product to be added to each sprayer load will be according to the dose on the product label and may be in millilitres per hectare, from which the amount per sprayer load can be calculated.

$$\text{Amount of formulated product per sprayer load (ml)} = \frac{\textit{Dose of formulated product (ml/ha) x area to be treated (ha)}}{\textit{Number of sprayer loads}}$$

Alternatively, the amount or % of formulated product to be added to a litre of water may be given.

5. Number of trials

The number of trials is dependent on the range of climatic/environmental conditions under which the PPP is likely to be used (at least two in the norm), usually undertaken over at least two years. For efficacy trials they should be done in situations subject to challenging pest attacks or where significant attacks are anticipated.

Reduced number of trials can be made if:

- tThere is a large amount of supporting evidence from the same product, similar product or active ingredient on closely related pests or the same pest on similar crops;
- for Minor pest or crops;
- special conditions, where very large plots are required e.g. testing pheromones.

Table 3 summarises the number of trials required.

Table 3 Number of trials required for different scenarios

Scenario	Number of trials
Major pest on major crop	10 (range 6 – 15)
Minor uses	3 (range 2 – 6)
Major use (protected conditions)	6 (range 4 – 8)

6. Number of replicates

Within a trial the treatments are replicated to take into account variation in effects that occur as a result of factors other than the treatment (residual effects). The number of replicates depend on the likely variation and the requirements of statistical analysis. Ultimately, the difference resulting from different treatments needs to be significantly higher than differences resulting from other factors. To analyse this statistically, the 'degrees of freedom' for the residual variation needs to be sufficient – this should be greater than 12. As an example, a trial with 6 treatments and four replicates will have (6 x 4) – (6 – 1) – (4 – 1) = 15 residual degrees of freedom. As a general rule, the number of replicates should be four, which could be reduced to three if the trial is undertaken on several different sites.

7. Trial design

All trials should normally be a randomized design. Two types are the most common – completely randomized or randomized complete block. Completely randomized should only be used if the trial area has a homogenous environment (no environmental difference such as difference in drainage, soil etc.). This is unlikely to be the case, so normally a randomized complete block design is used. An example (monofactorial) is given below using four test treatments, a reference treatment and untreated control (i.e. six treatments), replicated 4 times. This is illustrated below for different scenarios.

Example (source : OEPP (2012))

In the case of a known environmental gradient on a trial site the following layout can be used (there are several variations on this):

Block 1	2	3	1	5	4	6
Block 2	5	2	6	3	1	4
Block 3	3	6	2	4	5	1
Block 4	2	5	1	6	4	3

GRADIENT

Block 1						Block 2						Block 3						Block 4					
2	3	1	5	4	6	5	2	6	3	1	4	3	6	2	4	5	1	2	5	1	6	4	3

Figure 2 Possible arrangement of blocks and plots in randomized blocks in field trials. An environmental gradient down the field is accounted for, by arranging blocks down the gradient. Plots within blocks placed across the gradient are affected equally by the environmental variable.

The most likely layout is where a gradient is unclear or unknown:

Block 1			Block 2		
2	3	1	5	2	6
5	4	6	3	1	4
3	6	2	2	5	1
4	5	1	6	4	3
Block 3			Block 4		

Figure 3 Possible arrangement of blocks and plots in randomized field trial where an environmental gradient in unknown or unclear.

Other randomized trial designs are shown in EPPO guidelines (https://pp1.eppo.int/standards/PP1-152-4).

8. Untreated control

The main feature of 'untreated controls' is that they usually have not been subjected to any of the plant protection treatments under study. Untreated controls should, however, receive all the measures which are uniformly applied throughout the trial, in particular cultural measures and applications against pests not being studied in efficiency trials. The untreated control normally receives no treatment at all against the pest being studied, unless there are preventative control measures that are included in the treatments and do not involve the test PPP(s). Also, in some circumstances the untreated control may include certain operations received by the other treatments. For example, where the treatments are applied in high volume of water, the untreated control may be modified to include treatment with water alone.

Although there are different types of arrangement for untreated control (within or outside the treated area), for trials where the untreated control is included in the statistical analysis 'adopted controls' should be used where the controls are considered like any other treatment and are the same shape and size as the other plots, and are randomized in the trial layout.

9. Reference product

A reference product should be included in an efficacy trial whenever possible. The reference product serves to compare the test product with a PPP with known efficacy in practice, and so check the quality of the trial. Furthermore, the inclusion of a reference product facilitates the comparison between different trials in a series. Generally, the test product is compared against the reference product (applied at the label recommended rate) when assessing if efficacy is acceptable. As far as possible, the reference product should be registered in the country for the same use (crop/pest) as the test product is being proposed.

10. Test treatments

The number of test treatments will have been agreed with COLEACP.

For efficacy trials the rate of product applied (mg or ml/ha) should be as stated on the label/SDS (for a similar crop) or as advised by the manufacturer, if only one rate is tested. In a number of cases more than one rate is tested in order to determine the minimum effective dose. In this case, a rule of thumb is to apply the (label) recommended rate, three times that rate and a three times lower rate.

For residue trials normally only the recommended (label) rate is applied.

11. Location/site selection for trial

Trials should be conducted in locations which represent the range of agronomic, plant health, environmental and climatic conditions likely to be encountered in practice in the area of proposed use. The crops in the experimental locations may be specifically grown for the trials or be part of crops grown for commercial purposes, but in all cases should be grown according to normal agronomic or commercial practice. Any cultural operations in the fields, apart from the one being tested, should be according to normal practice.

If there is a possibility that different growing conditions like soil types may affect the efficacy of a PPP, the various trial sites should be chosen to be representative of the range of growing conditions that can be encountered in the proposed use patterns of the product. Within each trial site, environmental and agronomic conditions should be as uniform as possible.

12. Plot size and shape

The experimental units should be chosen to be representative of the population the trial is testing and to be as uniform as possible. In general, plots should be rectangular and of the same size in one trial and of similar size for a single trial series. Square plots reduce the risk of interference between plots and allow better for variation in wind direction. For observations of aggregated pests, such as some weeds and soil borne diseases, a greater number of smaller plots are better than fewer larger plots.

Plot sizes are given in specific EPPO standards for particular crop/pest combinations (EPPO (2012)). In cases where interference between plots is liable to occur, the plots will be larger (gross plot) and the observations will be limited to the central area (net plot). The difference between the net plot and the gross plot is called a discard area. One common source of interference is movement of the product (for example spray or vapour drift, or lateral movement on/in soil) outside the plot to contaminate adjacent plots. This can be particularly important for sprays applied to tall crops. However, with greater discard areas, the experimental error can often be minimized. Another common source of interference is spread of the pest (for example air borne fungi or highly mobile insects) from untreated plots or from plots where control of the pest is poorer. Such spread can both increase the pest population in plots with more efficacious treatments and decrease it in plots with less good ones. Similarly, if a product is being tested in a crop where integrated control is practised, adverse effects on predators and parasites may be masked by their migration between plots.

Thus the most suitable plot size depends upon many factors such as the mobility of the target organism (larger plots are generally needed for more mobile organisms), the technique of application (application techniques that deposit the product in a more precise manner require smaller plots than less precise techniques such as mist blowing), the size and type of the crop/plant. For instance, trials with backpack sprayers may already be done on fairly small plots, but there needs to be an appropriate buffer strips between plots or other means of reducing cross-contamination. Generally, 20 m x 20 m plots are suitable. Guard or buffer rows/strips are needed to minimize interference between plots (e.g. if drift or spread of the plant protection product is likely to occur or pest movement can be expected). Ideally, a buffer strip should be around 5 metres minimum; with a mistblower directed at a tall crop up to 30 m is needed. Such large buffer strips are not always possible; thus, the emphasis should be on careful and accurate application using equipment that minimises drift and

limiting observations and sampling and/or harvesting to the centre part of the plot (having discard areas). Accuracy increases with plot size, but only up to a certain degree, because variability in soil and infestation levels may also increase.

13. Marking the plots

Each plot within the trial needs to be clearly marked and labelled. It is recommended that a labelled pole is place at the lower, left hand corner of each plot. It should be labelled with the appropriate trial and plot number or designation (that refers to the treatment details). A map showing the trial layout and plot designations, as well as the surrounding crop area, should be drafted and included in reports. As a minimum, the trial technician should have a clear picture of each plot and its treatment.

14. Application of PPP(s)

All personnel involved in the mixing, application, storage, cleaning of equipment should be properly trained and wear the recommended Personal Protective Equipment (PPE). The handling and safety instructions on the product label and Safety Data Sheet (SDS) should always be followed, along with other best practices recommended by the manufacturer.

14.1 Weighing or measuring

Although some protocols state that chemicals must be weighed out, measuring is accepted (for liquids). It is accepted that the measuring takes place in the field if it is carried out carefully and in accordance with well-described SOPs. Accurate and calibrated weighing scales, measuring cylinders or syringes should be used for all measurements. Appropriately sized measuring cylinders should be used, the measure being read at the lower part of meniscus of the liquid. For volumes less than 10 ml a syringe is recommended. With low-dose pesticides a stock solution can be made in the laboratory and then taken along to the field. The solution must be used within 6 hours. If not, it must be documented that the pesticides do not hydrolyse in aqueous solutions. Records should be taken on all measuring operations.

Clear, written instructions on the amount of product and volume required for each plot, which has been previously calculated should be available, and followed.

14.2 Mixing

The application rate used (amount of active ingredient and volume application rate) should be agreed according to the recommendations on the PPP label and should be agreed with COLEACP during trial planning. More than one dose may be agreed for efficacy trials.

1. Add water to the mixing container to be about 25% of the final spray volume.
2. Measure out the required amount of PPP and add to the mixing container.
3. Triple rinse the graduated cylinder or syringe, adding the rinsate to the mixing container.

4. Add the remaining volume of water to reach the calculated spray volume (liquids) into the container. Add water by pouring against the side of the container opposite to the volume marks to reach the correct volume.

Mix the spray liquid by agitation or stirring with a stick until the PPP is well mixed. Pour the spray solution into the spray tank and re-check that the spray volume is correct.

14.3 Weather conditions

Application should not be made in high wind speeds (not greater than 15kph; range 3 – 15 kph). Application should also not be made at very low wind speeds (less than 1 kph) as the spray droplets are likely to stay in the air and drift. In hot climates, due to potential for droplet evaporation and resulting drift, as well as extreme working conditions, applications at the hottest time of the day should be avoided and restricted to the morning (preferably) or early evening. Application should not be made at temperatures greater than 30 °C. Dew should also have mostly evaporated. Do not apply is there is, or is likely to be, rain during the day.

14.4 Application

The follow rules should be followed:

- Make sure all settings of pressure, speed, boom height, granular flow, etc. are set according to specification from the calibration as previously performed.
- Agitate the spray mix before and during application to insure an even mix of the pesticide and water.
- If more than one concentration of a PPP is being applied start with the lowest concentration and work up to the highest concentration.
- Always spray to the side, with the nozzle aimed downwind.
- Cross-contamination of plots is reduced if buffer rows are included in the trial design. It can be further reduced by holding or fixing (preferably) cloth sheeting at the downwind edge(s) of the plot being treated. However, it should be noted that on hitting the barrier wind will generally rise up and over it, so it will not completely prevent contamination. Buffer rows should therefore always be used in combination.
- During spraying walk at the same speed as during the sprayer calibration.
- Placement of water sensitive paper on the edge of the adjacent plots will show if there has been any drift into those plots. These should be labelled according to the plot in which they were placed and the plot where the treatment was being applied and photographed.
- Placement of water sensitive paper in the treatment plot will also confirm adequate coverage – particularly on the top part of tree crops. This should also be labelled accordingly and photographed.
- On completion of spraying to a plot any residual spray liquid remaining in the sprayer should be measured so that the actual amount applied is recorded. Note if part of the plot is left unsprayed due to the spray liquid being exhausted. From this, the actual dose applied to the treated area can be calculated.

- It is recommended that a different sprayer (calibrated to the same volume application rate) is used for treatment with different PPP(s). If this is not possible, the sprayer should be properly rinsed (cleaned) between treatments with PPP(s).
- Throughout treatment environmental conditions (cloud cover, wind speed and direction, relative humidity and temperature) should be monitored and recorded.
- After spraying, all equipment should be washed properly. Do not spray the rinsate onto the plot, but onto border rows around the plot and where it cannot drift into the plots.

The need to apply more than one treatment application over the season needs to be assessed based on label recommendations, recommendations from the manufacturer and level of pest attack (threshold) and trial requirements. This, including the criteria for initial and repeat applications, should be agreed with COLEACP prior to the trial, but regularly reviewed on the basis of pest monitoring and damage levels throughout the season.

15. Data collection

Data collection is dependent on what the trial is being set up to measure – efficacy or residue levels. Some environmental conditions should be measured throughout for both type of trial:

- daily maximum and minimum temperature;
- relative humidity;
- rainfall.

Additionally, throughout the trial records should be kept of all agronomic and other pest management activities, which are carried out over the entire trial area (all plots treated, control and reference) being managed in exactly the same way, both prior to and during the trial. This could take the form of a cropping calendar and supporting field record book, which can show planned and actual agronomic activities.

For both efficacy and residue trials, data and sample selection should be taken from a central subsection (net plot) of the plot (gross plot) to avoid edge effects and reduce the possibility of effects from cross-contamination during treatment.

- With field crops data/samples should not be collected from the areas of the plot that are within three metres of its edges (for a 20 x 20 m plots and above, for smaller plots this will have to be reduced to 1 – 1.5 m).
- For tree crops, the sampling should not be made within three trees from the edges of the plots. Do not sample from areas that had been left unsprayed (if the spray liquid was exhausted before the plot had been fully covered).

The following sections describe the data collection activities specific to each type of trial.

15.1 Efficacy trials data collection

Efficacy of a PPP is ultimately assessed on the basis of yield and marketability/quality of the produce. However, pest levels also need to be assessed to determine whether the target pest attack was sufficient to affect yield (including impact on beneficial organisms and if they have impacted the pest population) and whether other (non-target) pests may have affected yield (this is needed for interpretation of results: comparison to the untreated control and reference will not give reasons for differences that may occur).

Thus, plots should be assessed for pest presence (and ideally beneficial organisms) and damage (one day) prior to treatment and weekly until harvest. It is recommended that 10 random assessments are made in each plot. The methodology for assessment (use of quadrats, whole plant assessments, etc.) will depend on the pest and the crop and should be agreed with COLEACP during planning of the trial. Visual damage assessments should also be made along with plant growth observations (size of plant, stage of growth, flowering etc.).

At harvest, a sample should be taken from each plot to assess quality/marketability of the crop. Ten samples should be taken at random from each plot and (visually) scored/graded. The type and size of sample taken will be dependent on the crop. Normally 10 – 50 samples should be sufficient.

Yield assessment ideally could be on a whole plot basis; however, this is not often practical; In this case a sub-sample needs to be taken and, normally, weighed. This sub-sample could be a specific area within the crop (two one meter square areas selected randomly) or several fruits selected randomly from different bushes or trees. Again, the actual method chosen will depend on the crop.

15.2 Residue trials data collection

For residue trial samples are normally collected at fixed intervals following treatment.It could also be beneficial to take a set of samples before treatment to ensure that there is no contamination from activities prior to trial initiation. This will be evaluated on a case by case basis and agreed with COLEACP.

Generally, samples are taken at random, or in a zig-zag pattern (Figure 4) across the sampling area in each plot. Collect two samples per plot, each sample being collected in a separate run through the plot. The total sample collected from each plot should be enough to allow testing for residues, but generally should be 1 to 5 kg. Collection should start with the untreated control and lower doses of the treatment (if there is more than one dose, which is not likely with residue trials). Individuals doing the sampling should wear PPE, and gloves should be changed or properly washed between collecting from different plots. Harvesting tools should also be cleaned to avoid cross-contamination. Samples should be placed in a clean sample bag, that has been clearly labelled with the plot number, treatment and collection date and time. The sample bag should be made of a material that does not interfere with the subsequent chemical analysis. If plastic or plastic-lined is used, this material should be polyethylene, not PVC. Wrapping individual samples in aluminium foil before placing in the bag reduces the risk of chemical interference and contamination.

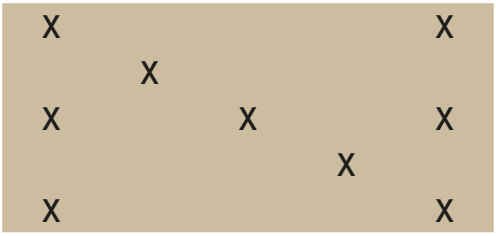


Figure 4 Example of sampling plan for the collection of samples on a cultivated plot

Generally, samples should be taken over a time series following application, normally 0 (immediately following application, once the spray droplets have dried) and 1, 3, 7, 14 and 21 days after application. This may vary depending on the trial and needs to be confirmed/ agreed with COLEACP during trial planning.

If possible, samples should be transferred immediately to the analytical laboratory. If this is not possible, there are three possible scenarios:

- storage in a refrigerator (4 °C), if the samples can be transferred to the laboratory within 48 hours;
- storage in a freezer (-18 °C) for a longer time and transferred to the laboratory as soon as possible;
- finally, if there is a risk of deterioration of the samples within 48 hours, they should be immediately placed in a freezer (-18 °C).

Freezer logs should be maintained if samples are stored (additions to and removal from storage). Samples should be maintained at freezing temperatures during shipment, for example by addition of ice to the shipment containers. All samples from an individual trial should be stored in exactly the same way. The analytical laboratory should be informed by email of shipments being made and details of the shipment. In return, they should inform the chief trial technician of the arrival of the complete shipment..

The residue analysis should be undertaken by a Good Laboratory Practice (GLP) certified laboratory using standard methodologies. The report giving the results should also include the date of extraction and analysis, as well as the methodology used. This should be sent to the trial manager and collated with a full description of the trial (including the field data book and crop calendar) in a report to COLEACP.

16. Content of trial report

For both efficacy and residue trials the field data book needs to be part of the report submitted to COLEACP. The content of the field data book should be detailed enough to reconstruct the field trial, and as a minimum contain the following:

- names of all personnel conducting specific research functions;
- amendments and deviations from the protocol and standard operating procedures agreed with COLEACP prior to the trial;
- test site information;
- plot maps;
- test substance receipt and details;

- test substance storage conditions (including timing and temperatures);
- data for calibration and use of application equipment;
- details of sample preparation (mixing, volumes prepared for each plot etc.);
- treatment application information data (including measures for drift reduction, wind speed, humidity and temperature, actual volume applied, any area where application did not occur, observations of droplet disposition);
- crop maintenance, PPP and cultural practices (fertilisation, irrigation, hand weeding etc.), test plot history, and soil information.

16.1 Efficacy trials report content

For efficacy trials the following should be included:

- daily records for temperature, relative humidity, windspeed, rainfall;
- table/Graph of weekly pest and beneficial organism numbers;
- table/Graph of damage assessments;
- table/Graph of crop yield kg/ha;
- table/Graph of marketable yield or yield in each quality category kg/ha.

Pest/beneficial and damage assessments can be analysed by analysis of variance at the end of the season to provide a measure of % control. Yield and quality data should be analysed statistically using analysis of variance. It is expected that effective test materials should provide control/yield of at least 90% of the reference material.

16.2 Residue trials report content

For residue trials the following should be included:

- residue sample identification, collection, storage conditions and handling;
- residue sample shipping information;
- daily records for temperature, relative humidity, windspeed, rainfall;
- validated results from the analytical laboratory (copy of the certified report from the laboratory should be included);
- analysis of results: this would normally consist of a breakdown graph, with standard error for each point, based on the mean residue figure for each sample time.

In addition to a written report, a SOP summary check sheet can be included for each trial. Examples are given below.

In order to ensure compliance with this SOP, check sheets containing all the key elements of this document will be used. These check sheets will be adapted for each trial and will be based on the examples below.

The check sheets will be attached to the trial documents (protocol, report, etc.).

SOP SUMMARY CHECK SHEETS

1. Efficacy trials check sheet

Title of trial – Brief description

i. *General information*

TRIAL NUMBER *Assigned by COLEACP*	

TRIAL OBJECTIVES

ADMINISTRATIVE INFORMATION	
Testing Unit Manager	
Chief Trial Technician	
Testing Unit: *Name of contracted organisation*	
Sponsor	COLEACP Rue du Trône, 130 B-1050 Bruxelles (Belgium) Telephone : 32 2 508 10 98 E-mail :
Trial supervisor	Name : COLEACP Rue du Trône, 130 B-1050 Bruxelles (Belgium) Telephone : 32 2 508 10 98 E-mail :

STUDY SCHEDULE	
Estimated starting date	
Estimated date for the end of trials (last harvesting)	

NUMBER OF SITES

EXPERIMENTAL DESIGN PER SITE	
Crop	
Variety	
Experimental treatments: *Description of treatments (including reference and untreated control)*	
Surface of an individual plot	
Total surface of the trial	

Trial Map:

ii. *Description of experimental treatments*

TEST TREATMENTS				
Active substance		Product		
Name	*g/ha*	*Trade name **	Company	Dose/ha

* original label and SDS are provided and added in the final report

REFERENCE TREATMENTS				
Active substance		Product		
Name	*g/ha*	*Trade name **	Company	Dose/ha

* original label and SDS are provided and added in the final report

CONTROL PLOT: *Description of any treatments e.g. water alone.*

TYPE OF APPLICATION: *e.g. spray application.*
APPLICATION DEVICE: *e.g. backpack-sprayer: type of sprayer, nozzle size, pressure.*
FURTHER APPLICATION DETAILS: *The details during each application must be recorded such as:* *sky coverage of clouds, wind direction, measured wind speed, buffer between each plot at the sites should be at least 1.5 m from each other to avoid spray drift, coverage of plants (from water sensitive paper) and run off observations, if any.* *Each application should be done under dry conditions – no rainfall should occur before, during and shortly after application. Dew should have evaporated.*
CALIBRATION OF SPRAYER: *A calibration must be done before each treatment. Details of calibration – volume application rate needs to be recorded here.*

PREPARATION OF THE MIXTURE

The quantity of commercial product in the mixture (quantity per litre of water) will be calculated based on:

- *volume application rate and plot size,*
- *concentration of active ingredient in product,*
- *recommended rate.*

These need to be listed here for each treatment.
Volume of water per hectare used for each plot application must be recorded.

ADDITIONAL TREATMENT:

As needed and agreed by COLEACP

CLIMATOLOGIC OBSERVATIONS:

Record of daily temperatures (°C.): min. and max.
Record of daily rainfalls (mm)
Relative humidity records (%)

ADDITIONAL DATA RELATED TO THE FIELD PHASE:

The field history for pesticide applications during the last 3 years will be documented, as well as the soil type.
Sowing or planting of the trial crop system, beginning and end of flowering and commercial harvest date. The plot maintenance, e.g. fertilization, irrigation and additional pesticide application will be performed according to common agricultural practice and documented (including presentation as a crop calendar). No maintenance products containing the test product(s) should be used unless previously agreed with COLEACP. All treatments must be authorised by COLEACP.

iii. *Treatments and sampling schedule*

Remark: number of applications and interval between applications are agreed with COLEACP after consultation with the PPP manufacturers

APPLICATIONS (EXAMPLE)		
T1 Date:	T2 Date:	T3 Date:
Product 1:		Product 1:
	Product 2:	Product 2:

T1, 2 and 3 = Treatments 1, 2 and 3;

CONTROL PLOT: *If pest control applications are required for non-target pests, active substances must be different to the PPP(s) tested. These treatments should also be made to the treated plots.*

iv. *Schedule of pests and diseases monitoring*

PRINCIPLE: *Monitoring of plots to determine the necessity of extra treatments to protect the crop.*

PERIODS OF MONITORING: *1 monitoring per week, starting from emergence.*

AGRICULTURAL OBSERVATIONS (CROP DEVELOPMENT): *At each pest and disease monitoring note the growth stages (BBCH scale) and development of plant density.*

v. *Sampling*

SAMPLE NATURE: *Pests and beneficial counts, damage assessments, 10 observations per plot.*
WEIGHT/QUALITY OF A SAMPLE AT HARVESTING: *20 fruits/plants per plot – assess weight and score quality. Take pictures of whole sample.*

Sampling schedule (in days) related to the harvest and analysis to be done (X) on samples

e.g.

TREATMENT, REFERENCE AND CONTROL	S1	S2, etc.	SX (HARVEST)
a.	X One week After emergence	X Weekly	X
b.	X	X	X

S = sampling points;

vi. *Final report*

The final report must include the following

OBJECTIVES OF THE TRIALS: *Short sentence about the purpose of these trials*

Identification number of trials	
Person in charge of the administration (Testing Unit Manager)	
Person in charge of the technical follow-up of trial (Chief Trial Technician)	
Location of the trials	
Host companies of the trials	
Sponsor	
Trial supervisor for COLEACP	

ACTIVE SUBSTANCES, COMMERCIAL PRODUCTS AND GAP TESTED: - *Table 1 showing providers of test items,* - *Table 2 on description of the experimental treatments and number of replicates,* - *Table 3 of the treatments schedule (see protocol).*
EXPERIMENTAL SETTING: *Description of plots (including crop, variety, surface, number of plots per treatment and combinations and a diagram with exact plots size and directions as well as neighbouring crops and /or fallow).*
CHARACTERISTICS OF THE TRIAL SITE: *Site characteristics, plot characteristics, previous crops, additional treatments, previous treatments, agricultural observations.*
TREATMENTS APPLIED: *Type of application and application details (water volumes, calibration of sprayer, etc.), including control. Remaining spray liquid after treating a plot, etc.*
ADDITIONAL TREATMENTS DONE: *Herbicides, fungicides, insecticides, nematicides, seed treatments.*
CULTURAL/AGRONOMIC ACTIONS:

Crop calendar.
SAMPLING: *Pest and beneficial observations (Table/Graph), yield amount and quality (Table/Graph).*
AGRICULTURAL OBSERVATIONS: *Crop Development and plant density (can be included in crop calendar).*
PHYTOSANITARY OBSERVATIONS: *Give results of monitoring.*
METEOROLOGICAL OBSERVATIONS: *Temperatures, humidity and rainfall during the period of field trials.*
ANALYSIS RESULTS: *Analysis of variance.*

2. Residue trials check sheet

Title of trial – Brief description

i. *General information*

TRIAL NUMBER *Assigned by COLEACP*	

TRIAL OBJECTIVES

ADMINISTRATIVE INFORMATION	
Testing Unit Manager	
Chief Trial Technician	
Testing Unit *Name of contracted organization*	
Sponsor	COLEACP Rue du Trône, 130 B-1050 Bruxelles (Belgium) Telephone : 32 2 508 10 98 E-mail :
Trial supervisor	Name : COLEACP Rue du Trône, 130 B-1050 Bruxelles (Belgium) Telephone : 32 2 508 10 98 E-mail :

STUDY SCHEDULE	
Estimated starting date	
Estimated date for the end of trials (last harvesting)	

NUMBER OF SITES

EXPERIMENTAL DESIGN PER SITE	
Crop	
Variety	
Experimental treatments: *Description of treatments (untreated control)*	
Surface of an individual plot	
Total surface of the trial	

Trial Diagram:

ii. *Description of experimental treatments*

TEST TREATMENTS				
Active substance		Product		
Name	*g/ha*	*Trade name **	Company	Dose/ha

* original label and SDS are provided and added in the final report

CONTROL PLOT *Description of any treatments e.g. water alone.*

TYPE OF APPLICATION: *e.g. spray application.*
APPLICATION DEVICE: *e.g. backpack-sprayer: type of sprayer, nozzle size, pressure.*
FURTHER APPLICATION DETAILS: *The details during each application must be recorded such as:* *sky coverage of clouds, wind direction, measured wind speed, buffer between each plot at the sites should be at least 1.5 m from each other to avoid spray drift, coverage of plants (from water sensitive paper) and run off observations, if any:* *Each application should be done under dry conditions – no rainfall should occur before, during and shortly after application. Dew should have evaporated.*
CALIBRATION OF SPRAYER: *A calibration must be done before each treatment. Details of calibration – volume application rate needs to be recorded here.*

PREPARATION OF THE MIXTURE:

The quantity of commercial product in the mixture (quantity per litre of water) will be calculated based on:

- *volume application rate and plot size,*
- *concentration of active ingredient in product,*
- *recommended rate.*

These need to be listed here for each treatment.
Volume of water per hectare used at for each plot.

ADDITIONAL TREATMENT:

As needed and agreed by COLEACP.

CLIMATOLOGIC OBSERVATIONS:

Record of daily temperatures (°C.): min. and max.
Record of daily rainfalls (mm)
Relative humidity records (%)

ADDITIONAL DATA RELATED TO THE FIELD PHASE:

The field history for pesticide applications during the last 3 years will be documented, as well as the soil type.
Sowing or planting of the trial crop system, beginning and end of flowering and commercial harvest date. The plot maintenance, e.g. fertilization, irrigation and additional pesticide application will be performed according to common agricultural practice and documented (including presentation as a crop calendar). No maintenance products containing the test product(s) will be used unless previously agreed with COLEACP All treatments must be authorised by COLEACP.

iii. *Treatments and sampling schedule*

Remark: number of applications and interval between applications are agreed with COLEACP after consultation with the PPP manufacturers

TREATMENT							
Applications (example)[a]			Samplings (example)[b]				
T1 15 days before harvesting	T2 11 days before harvesting	T3 1 day before harvesting	S1 1 DALA (at 1st harvest)	S2 3 DALA	S3 7 DALA	S4 14 DALA	S5 21 DALA
Product 1		Product 1	X	X	X	X	X
	Product 1	Product 1	X	X	X	X	X

[a] T1, 2 and 3 = Treatments 1, 2 and 3;
[b] S1, 2, 3, 4 = Sampling points at X DALA (Days After Last Application)

CONTROL PLOT: *Apply if required. Active substances that must be different in terms of residues from active substances to be analysed in test products. These treatments should also be made to the treated plots.*

iv. *Schedule of pests and diseases monitoring*

PRINCIPLE: *Monitoring of plots to determine the necessity of extra treatments to protect the crop.*

PERIODS OF MONITORING: *1 monitoring per week starting from emergence.*

AGRICULTURAL OBSERVATIONS (CROP DEVELOPMENT): *At each pest and disease monitoring note the growth stages (BBCH scale) and development of plant density.*

v. *Sampling*

SAMPLE NATURE: *Produce, export quality.*
WEIGHT OF A SAMPLE AT HARVESTING: *1 – 5 kg on a minimum of 20 plants (previously agreed with COLEACP). Take samples at various heights of the plants in order to reflect different exposure to the spray.*
WEIGHT OF SAMPLE DEEP-FROZEN: *e.g. 0.5 kg per sample.*
NUMBER OF SAMPLE/PLOT: *2 (1 will be sent frozen to laboratory, the other one will be kept frozen as retained sample).*

NUMBERING OF SAMPLES: *Codification.*
PACKAGING: *Sealed plastic bags (wrapped in aluminium foil and placed in polyethylene bags).*
CONSERVATION (STORAGE CONDITIONS OF SAMPLES SENT FOR ANALYSIS AND SAMPLES RETAINED): *Transfer samples immediately to the analytical laboratory. If this is not possible:* *- store in a refrigerator (4°C) and transfer to the laboratory within 48 hours;* *- immediately store in a freezer (-18 ° C) if there is a risk of deterioration of the samples within 48 hours and transfer to the laboratory as soon as possible;* *- store in a freezer (-18 ° C) for a longer time and transfer to the laboratory as soon as possible.* *Before destruction of stock, person in charge of the trial will ask authorization to COLEACP.*
LABELLING BEFORE SHIPMENT: *Number of sample* *Date and time of sampling* *Shipper* *Analysis to be undertaken*

SHIPMENT: *Box with carbo ice (weight of carbo ice = more or less 50 % of the weight of samples in a box).* *Shipment of 1 sample out of 2 identical samples to the analytical laboratory.* *Sampling report with full address (sender and address), number of the trial, list of sent samples with their number and analyses to be undertaken (according to label). A copy of this document will be sent to COLEACP and to the Trial Manager.* *All samples will be sent in one batch.*
SHIPMENT ADDRESS: *Following indications given by COLEACP.*

Sampling schedule (in days) related to the harvest and analysis to be done (X) on samples

e.g.

TREATMENT	S1 (1 DAY AFTER LAST SPRAYING)	S3	S7	S14	S21
a.	X (1 d. PHI)	X (3 d. PHI)	X (7 d. PHI)	X (14 d. PHI)	X (21 d. PHI)
b.	X (1 d. PHI)	X (3 d. PHI)	X (7 d. PHI)	X (14 d. PHI)	

CONTROL C0	S1 (1 DAY AFTER LAST SPRAYING)	S7
All active substances tested to be analysed	X (1 d. PHI)	X (7 d. PHI)

S = sampling points; PHI = Pre harvest intervals

vi. *Final report*

The final report must include the following

OBJECTIVES OF THE TRIALS: *Short sentence about the purpose of these trials.*	
Identification number of trials	
Person in charge of the administration (Testing Unit Manager)	
Person in charge of the technical follow-up of trial (Chief trial Technician)	
Location of the trials	
Host companies of the trials	
Sponsor	
Trial supervisor for COLEACP	
ACTIVE SUBSTANCES, COMMERCIAL PRODUCTS AND GAP TESTED: *Table 1 showing providers of test items* *Table 2 on description of the experimental treatments and number of replicates* *Table 3 of the treatments schedule (see this protocol)*	

EXPERIMENTAL SETTING: *Description of plots (including crop, variety, surface, number of plots per treatment and combinations, diagram with exact plots size and directions, as well as neighbouring crops and/or fallow).*
CHARACTERISTICS OF THE TRIAL SITE: *Site characteristics, plot characteristics, previous crops, additional treatments, previous treatments, agricultural observations.*
TREATMENTS APPLIED: *Type of application and application details (water volumes, calibration of sprayer, etc.), including control. Remaining spray liquid after treating a plot, etc.*
ADDITIONAL TREATMENTS DONE: *Herbicides, fungicides, insecticides, nematicides, seed treatments.*
CULTURAL/AGRONOMIC ACTIONS: *Crop calendar.*

AUGUST 2020 - Ref: 00430-03

GROWING PEOPLE