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Pesticides — Methods of sampling



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Foreword

Rwanda Standards are prepared by Technical Committees and approved by Rwanda Standards Board (RSB) Board of Directors in accordance with the procedures of RSB, in compliance with Annex 3 of the WTO/TBT agreement on the preparation, adoption and application of standards.

The main task of technical committees is to prepare national standards. Final Draft Rwanda Standards adopted by Technical committees are ratified by members of RSB Board of Directors for publication and gazettment as Rwanda Standards.

DRS 405 was prepared by Technical Committee RSB/TC 024, *Chemicals and Consumer Products*.

In the preparation of this standard, reference was made to the following standard:

ES 694: Pesticides – Methods of sampling

The assistance derived from the above source is hereby acknowledged with thanks.

Committee membership

The following organizations were represented on the Technical Committee on Chemicals and Consumer Products (RSB/TC 024) in the preparation of this standard.

Star Construction and Consultancy (SCC) Ltd

University of Rwanda/College of Science and Technology (UR/CST)

HORIZON/SOPYRWA Ltd

University of Rwanda/College of Education

AGROPY Ltd

Rwanda Standards Board (RSB) – Secretariat

Pesticides — Methods of sampling

1 Scope

This Draft Rwanda Standard prescribes the methods of sampling of representative test samples of liquid and solid agricultural pesticides

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

DRS xxx, *Pesticides — Terminology*

RS ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

3 Terms and definitions

For the purposes of this standard, the terms and definitions given in **DRS** 406 and the following apply.

3.1

analytical portion

a portion of a suitably prepared and appropriately homogenized laboratory sample (see 3.11) which is analyzed or tested; also known as a test portion

3.2

bulk sample

the combined total of the primary samples taken from the batch. The bulk sample should be mixed thoroughly before dividing it into (at least three) equal laboratory samples. The bulk sample should not be less than 300 g or 300 ml for technical materials, 600 ml for liquid formulations and 1800 g for solid formulations. These requirements may be increased, depending upon the tests required. Where the bulk sample is comprised of small packages (e.g. sachets), each containing less than these quantities: **either** the packaged should be opened and the contents thoroughly, but carefully, mixed before subdivision into the laboratory samples; **or** the intact packages should be divided equally and randomly between the laboratory samples but, in the case, each laboratory sample must consist of at least three individual containers. Where a batch of a formulation is stored or transported in single bulk container, the bulk sample should consist of samples taken from a number of points in the batch.

3.3

consignment

a quantity of one or more materials delivered at one time. A consignment of pesticides may consist of one or more batches or parts of batches.

3.4

distribution

the process by which pesticides are supplied through trade channels to local or international markets

3.5

end - user

the persons or organizations using the pesticide for its intended purpose

3.6

IATA

International Air Transport Association

3.7

ICAO

International Civil Aviation Organization

3.8

IMO

International Maritime Organization

3.9

Inspector (Sampling Officer)

a person who has been properly trained in reliable and safe sampling, and who is authorized by the responsible authority to examine pesticides and take samples for controlling the quality of pesticides and their packaging

NOTE The inspector should carry an appropriate identification or authorization document.

3.10**label**

the written or graphic information on, or attached to, the immediate container of the pesticide and its external packaging, if any

3.11**laboratory sample**

the portion of material which is obtained by the specified sampling procedure, and which is sent to the laboratory for testing

NOTE The laboratory sample should not be less than 100 g or 100 ml for technical materials, 200 ml for liquid formulations and 600 for solid formulations. These requirements may be increased, depending upon the tests required. Where the laboratory sample is comprised of more than one intact container of a formulation, each container shall be analyzed individually, if practicable. In this case, each container shall meet the specification and, in case of dispute, the containers comprising the replicate laboratory samples shall also be analyzed separately.

3.12**packaging**

the container together with the protective wrapping used to transport pesticides, via wholesale or retail distribution, to end users

3.13**packaging unit**

an individual container containing pesticide, and/or a retail package containing a number of smaller packages or containers, each usually less than 2L or 2 Kg, of pesticide

3.14**primary sample**

a quantity of material, loose or packaged, taken, with or without a sampling tool, from a single sampling position in a container or batch

NOTE Where a formulation is packaged for the end user in quantities larger than that required for the bulk sample, the primary sample and the bulk sample may be indistinguishable as the corresponding laboratory samples are withdrawn from a single container. For small packages containing less material than the minimum amount required for the sample, the next larger packaging unit (e.g.: a box containing a sufficient number of small packages) may be opened to take randomly the necessary number of small packages (i.e. primary samples) to prepare the bulk sample.

3.15**RID**

International Regulation concerning the Carriage of Dangerous Goods by Rail

3.16

random sampling

a sampling procedure in which each package or portion of material has an equal chance of being chosen

3.17

referee analysis

an analysis performed in an independent laboratory staffed by suitably experienced personnel, agreed by the parties to a dispute, in order to certify the quality of a disputed sample

3.18

registration certificate

a document issued by the responsible government organization which sets out specification for a formulated pesticides

3.19

responsible authority

the government agency or agencies responsible for regulating the manufacture, distribution and/or use of pesticides, and more generally for implementing registration

3.20

sampling assistant

a person allocated to help the inspector in handling containers, sampling equipment etc.

NOTE A sampling assistant can only take samples under supervision from the inspector

3.21

sampling report

the standard report from completed one to be attached to each laboratory sample and one to be retained by the inspector for his/her own record.

NOTE At least four copies are required, one to be attached to each laboratory sample and one to be retained by the inspector for his/her own record

3.22

testing laboratory

a laboratory which is authorized by the responsible authority to test pesticides for their compliance with quality specifications.

4 Objective

The objective of this sampling procedure is to provide sufficient representative material for testing the packaging and physical and chemical properties of pesticides, to ensure that pesticides moving in trade meet their minimum quality specifications, and that their physical state and chemical composition are suitable for safe and efficient use.

The procedure has been established harmonization to ensure that samples are collected safely and at an appropriate stage, are representative, and will arrive at their destination intact. The procedure may be used for commercial or official regulatory purposes.

5 Safety precautions

Pesticides are toxic chemicals, which can poison if handled improperly. These sampling guidelines are not intended to provide detailed safety instructions and therefore sampling inspectors should be informed of, and comply with, the safety precautions required for specific pesticides and should wear appropriate protective clothing as indicated by labels or pictograms. The general precautions to be taken include the following.

- Take great care to avoid contamination of the eye, skin or clothing, ingestion of pesticides, or inhalation of dusts and vapours. Take similar care to avoid contamination of personal belongings and the local environment. Do not keep food in the immediate vicinity. If practicable, sample pesticides only in a well-ventilated environment.
- Take care to avoid spillage or splashing of liquids or spreading of dusts. Take particular care in handling leaking containers or those with a build up of material around the opening.
- Before sampling make sure washing facilities are available, in case of accidental spillage and for adequate washing after sampling is completed.
- Do not eat, smoke or drink during sampling or before removing protective clothing and washing thoroughly.
- Attach labels to sample containers before starting the sampling. As far as practicable, ensure that the outsides of sample containers are not contaminated with the sample material.
- Ensure the safe and proper cleaning of equipment and the safe disposal of contaminated materials such as personal protective clothing and equipment, tissue paper, etc.

6 General principles of sampling

Sampling and subsequent analysis of the sample taken constitute the most effective way to monitor compliance with quality specifications. Although sampling should preferably be done by removing material from positions in a batch which are chosen in a statistically random manner, in practice the positions may be limited by accessibility and safety. If random sampling is not practicable, the method of selecting primary samples should be noted in the Sampling Report under Remarks.

6.1 Sampling protocol for on-site examination of packaging

The general sampling protocol for on-site examination of packaging is as follows:

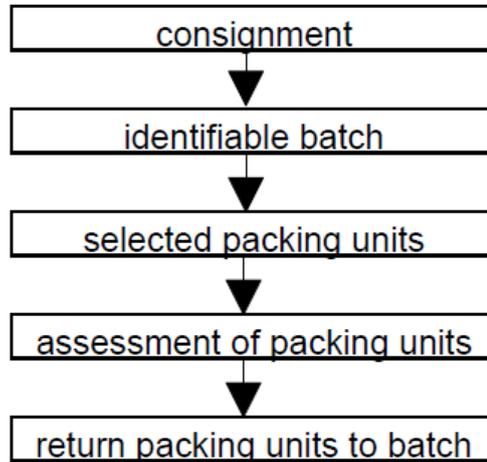


Figure 1 – General sampling protocol for on-site examination

6.2 Sampling protocol for laboratory testing

6.2.1 The general sampling protocol for laboratory testing is as follows:

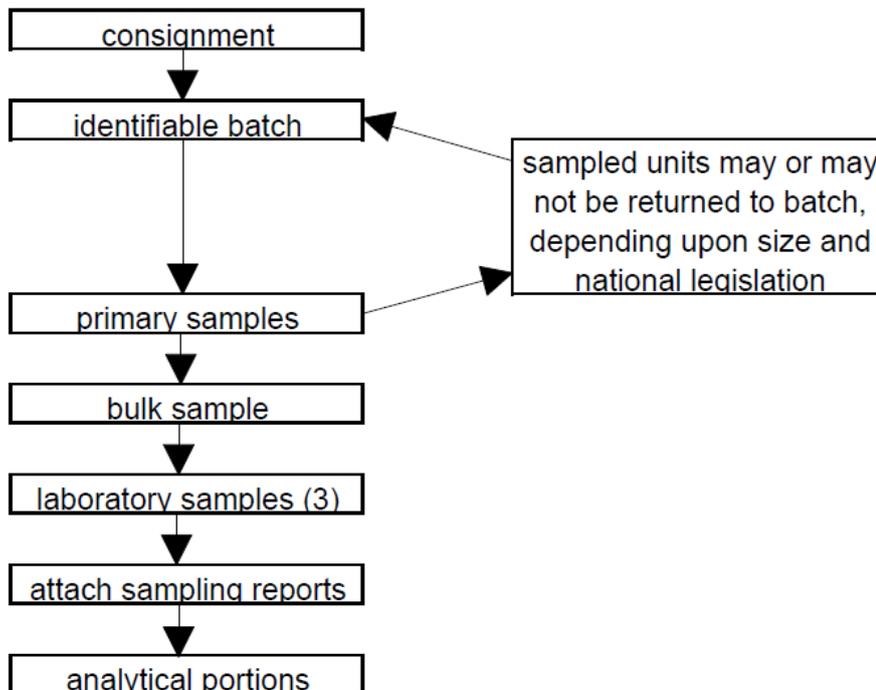


Figure 2 – General sampling protocol for laboratory testing

6.2.2 Where applicable, the samples for laboratory testing may be taken from packaging units, which have been selected for on-site examination.

6.2.3 Sampling may be carried out at any point in the distribution of pesticides, from the factory to the retail outlet.

6.2.4 The role of the inspector in collecting samples and forwarding them to the testing laboratory is crucial to the success of the testing carried out. The inspector must be well trained in sampling procedures. In all cases the sampling technique used must ensure that the samples taken enable the analyst to provide results which are representative of the material sampled. The inspector must therefore follow established procedures for sampling, handling and packaging.

6.2.5 The basic principles of sampling of technical active ingredients and formulations differ because of their subsequent uses. Technical active ingredients are processed into formulations. During this procedure the raw material is likely to be mixed thoroughly and consequently its average properties should be determined from composite samples representing the batch. On the other hand, each container of a formulation should meet the quality specification. Consequently the material removed from each package should be analyzed separately.

6.2.6 The quality of technical active ingredients should be tested at manufacturing or formulating plants, etc., before formulation.

6.2.7 Formulated pesticides may be checked before distribution at manufacturing, formulation and packaging plants or, in the case of imported formulations, at central depots or stores. If practicable, samples should be taken from retail outlets before the beginning of the normal season of use for the pesticide, so that effective corrective action can be taken, if required.

6.2.8 The bulk sample should be thoroughly mixed and divided into three equal laboratory samples. The three laboratory samples are provided for the testing laboratory; the organization agreed by both parties to retain referee samples; and the person designated responsible for the batch at time of sampling. A Sampling Report should be completed in a minimum of four copies. One copy of the sampling report should accompany each laboratory sample and the last copy should be retained by the Inspector.

6.2.9 The recommended form of sampling report is shown in **annex D**. In the remarks section of the report, the Inspector should record his/her observations of the suitability of the storage conditions; whether the pesticide was exposed to frost, water, prolonged sunshine or high temperature; whether opened containers were present; the apparent homogeneity of the batch; whether segregated fractions of the formulation were sampled separately and how many such samples were taken from the batch.

7 Preparations for sampling

7.1 Sampling devices and sample bottles

7.1.1 Appropriate, clean equipment and containers must be used to take and retain samples, to avoid contamination from outside sources, to ensure that contamination of the sampling officer is minimized and to enable the analyst to analyze the submitted material satisfactorily.

7.1.2 Bottles, their seals and sampling equipment must be approved by the testing laboratory. Bottles should be made of glass but solvent-resistant plastic may be satisfactory in certain cases.

7.2 Sampling preparation

a) Collect information on:

- The toxicity of, and handling instructions for, the pesticides to be sampled;
- The amount of laboratory sample required for the tests;
- The nature and identify of pesticide materials to be sampled and the number and sizes of packaging units involved.

b) Select appropriate:

- Sampling apparatus, e.g. 50 – 100 mL pipettes; 3-way pipette fillers; siphon-and-lift hand pumps (possibly with interchangeable non-drip discharge tubes suitable for hydrocarbons); dip tubes; sample triers, scoops, etc.; sample bottles (preferably glass containers with caps that can be tightly closed); plastic bags (without ventilation holes); plastic sheets; tools for opening pesticide containers; containers for pesticides where the original containers are to be emptied;
- Portable balance(s) with a suitable weighing range;
- Labels which can be firmly glued or otherwise attached to the sample containers;
- Sealing tape and wax seal, or official printed tape to certify the authorized opening of containers and to indicate the amount taken as sample (only where national legislation permits selling resealed containers);
- Personal safety devices, e.g. appropriate gloves (suitable for handling drums, cans packages, sampling devices and sample containers), aprons, dust masks, an effective respirator where necessary, safety goggles, tissue paper, first aid kit, soap, towel and a supply of water for washing;
- Case for sampling equipment and sample containers which enables them to be carried and transported safely;
- Absorbent material (e.g. sawdust, vermiculite or similar material) for filling the space around sample containers.

Note Newspaper, polystyrene granules or wood are not satisfactory absorbents.

c) Check the availability of:

- A sufficient number of sampling report sheets;

- Writing and marking pens;
- A valid identification document or authorization of the Inspector (Sampling Officer);
- Sampling Assistants;
- Vehicle for carrying sampling personnel, equipment and samples; and
- Transportation for the samples of the laboratory.

d) Notify

- The individual responsible for the site in advance of the sampling date to ensure the availability of responsible personnel to handle pesticide containers during the sampling operations; and
- The laboratories of the types and numbers of samples likely to be sent to analysis.

Note Merchants or retailers who are re-packing pesticide formulations for sale in small quantities should not be notified in advance.

8 Monitoring the properties of pesticide packages

The quality of packaging is assessed by external examination of original intact containers or packages. The number of packaging unit to be selected from randomly chosen positions depends on the size of the batch, as follows: up to 5 units, each unit; for 6 – 100 units, 5 units; for more than 100 units, 1 for every 20 units. Each selected packaging unit shall meet the quality specification.

NOTE 1 The packaging is acceptable if all containers selected randomly as samples are intact, their contents do not leak during shaking or turning, there is no observable sign of pesticide contamination on the surface of the containers, and the material and size of the packaging comply with the Registration Certificate. Deformation of containers is unacceptable if the contents are thereby pressurized or if the containers are rendered more difficult or hazardous to store, to transport or to use.

NOTE 2 The permanent label and any attached documents are acceptable if they are clearly legible, intelligible and give the information which is specified in the Registration Certificate. Secondary packaging, if any, of the pesticide containers is acceptable if it indicates clearly the nature of the contents and the hazard involved.

NOTE 3 Where the nominal gross mass (or the nominal mass of the container) is specified, the mass of the contents of pesticide containers should be checked by weighing the intact containers with their contents.

Where only the net mass or volume is specified, the mass of the contents of pesticide containers should be checked by weighing the intact containers complete with contents. The mass of the container (and of the contents where the volume is specified) must then be determined. Where applicable, the following procedure should be performed in the laboratory but if not suitable alternative containers are required on site and the Inspector must take due care in transferring the pesticide. The measurements should be made on a minimum of 3 containers. Carefully and completely remove the entire contents into a suitable alternative container. Where the net volume is specified, calculate the nominal net mass either from the measured specific gravity or from the specific gravity, quoted in the pesticide specification. Sum the mean mass of the empty containers with the nominal mass content to obtain the estimated nominal gross mass. Compare the measured gross masses of the selected containers with the estimated nominal gross mass and, where the deviation exceeds the specified value (or that permitted by national legislation), determine the net masses or volumes by removal of the

contents. Where the same pesticide, in identical containers, is to be checked on a number of occasions, the nominal gross mass estimated for the first batch might be used to check subsequent batches.

The accuracy of the mass or volume measurements should be equal to or better than 1/4th of the acceptable deviation from the nominal mass or volume, in the quality specification. For example, if the nominal gross mass is 550 g and the permitted deviation is $\pm 2\%$ then its mass equivalent is 11 g. consequently the accuracy of the weighing should be ± 2.5 g or better (and the resolution of the balance should be ± 1 g).

NOTE 4 The inspector should pay attention to the general condition of the packages during sampling. If any deficiency (deformation, leakage, loose labels, etc.) is observed, the packages in the remainder of the batch should be examined individually and the defective ones removed. Their further use should be determined on a case-by-case basis depending on national legislation and the corrective actions, which are practicable.

9 Sampling for testing physical and chemical properties

9.1 Technical active ingredients

9.1.1 In the case of packaged pesticides the number of primary samples to be taken and aggregated into a bulk sample is as follows: up to 5 packaging units, 1 primary sample from each unit; 6 – 100 units, 1 primary sample from 5 units; over 100 units, 1 primary sample per 20 units.

9.1.2 Where the technical active ingredient is transported or stored in a single, large bulk, 15 primary samples should be taken from randomly selected parts of the batch.

9.1.3 The minimum mass of the bulk sample should be 300 g and 3 equal laboratory samples should be withdrawn from it. After mixing one is to be sent to the testing laboratory, the second to be retained by the supplier of the pesticide and the third to be kept for referee analysis in case of dispute. The referee sample should be retained by an organization agreed by both parties.

NOTE Bulk samples and laboratory samples of technical active ingredients must be made homogeneous, as far as is practicable, before subdivision. In order to facilitate homogenization of laboratory samples, liquid pesticides may be carefully warmed to a maximum of 40 °C, in the laboratory. Warning of bulk samples in the field should not be attempted.

9.2 Formulations

In an acceptable batch of formulated pesticide, the material in each individual container should meet the specification. Therefore each bulk sample taken for testing should normally be obtained from a single packaging unit and may consist of one or more primary samples taken from that packaging unit. The packaging unit and the corresponding method for obtaining primary/bulk samples should be identified as follows.

- i. For end-user containers which each holds sufficient formulation to be subdivided into three laboratory samples (i.e. holding 600 ml or more for liquid formulations and 1800 g or more for solid formulations), the bulk sample is obtained from a single container and no primary sampling is required. The contents should be well mixed before opening the container and removal of the three laboratory samples. In the case of large containers, each laboratory sample should consist of material removed from the top, middle and bottom of the container, as far as is practicable.
- ii. For end-user containers which each hold less than is required for three laboratory samples (i.e. holding less than 600 ml for liquid formulations or less than 1800 g for solid formulations).

The bulk sample should be taken from a single packaging unit consisting of multiple small containers (the primary samples) which, when aggregated, will provide sufficient formulation to be subdivided into three laboratory samples. If individual containers are not packaged into larger units at the time of sampling, the bulk sample should be aggregated from sufficient containers taken at one point in the batch.

The individual containers of the bulk sample should normally be opened, their contents should be mixed thoroughly (taking special care with granules and water-dispersible formulations) and subdivided into three laboratory samples. Alternatively, if required (where the containers are also to be tested by the laboratory, for example), the individual containers may remain intact but each laboratory sample should then consist of a minimum of three containers and each should be analyzed separately.

- iii. Bulk tankers or wagons should be sampled by taking three primary samples, each a minimum of 200 ml (of liquid formulations) or 600 g (of solid formulations), from different depths of the tank or at the beginning, middle and end of discharging. The primary samples should be aggregated into bulk sample, well mixed and subdivided into three laboratory samples.

One of the laboratory samples should be sent to the testing laboratory, the second should be retained by the supplier of the pesticide, and the third should be kept for referee analysis in case of dispute. The referee sample should be retained by an organization agreed by both parties.

Where an identifiable batch of end-user packaged formulation cannot be presumed uniform (e.g. where there are no data from previously satisfactory similar batches, etc.), separate bulk samples should be taken from the number of sampling positions indicated in [table 1](#). These bulk samples must not be aggregated but should be identified and analyzed separately. If a batch may be presumed uniform, bulk samples may be taken from fewer positions (a minimum of one) in the batch. Batches of solid formulations, particularly granular formulations, are less easily judged to be uniform than are liquid formulations and should normally be sampled in accordance with the requirements of [table 1](#).

Table 1 – Number of bulk samples to be selected randomly for testing physical and chemical properties of formulated pesticides

Number of packaging units in the batch	Number of packaging units from which primary/bulk samples are to be taken
Up to 10	1
11 – 20	2
21 – 40	3
> 40	3 plus 1 for every additional 20 units up to a maximum of 15 sampled units

The sampling report should indicate whether the storage conditions could have been unsatisfactory for formulations, which must be protected from frost, extreme heat or moisture.

In national legislation permits, the net content of open containers may be restored (where samples have been taken from several containers) by combining their contents if the samples removed represent more than 10% of the net contents. In this case, reseal the containers with official tape to indicate that they have been sampled.

9.2.1 Liquid formulations (solutions, emulsifiable concentrates, suspension concentrates, emulsions)

Using appropriate care, containers which are to be opened for sampling should be shaken, rolled, tumbled or otherwise agitated (using methods which could be adopted by end users) before sampling, to try to achieve physical homogeneity. Before taking samples, the contents of containers of liquid pesticides should be visually checked, where practicable, for any physical heterogeneity, such as crystallization, precipitation,

sedimentation or separation. A rod may be used to check for the presence of dense sediment. After complete or partial emptying, inspect containers again for sediment, which was not re-suspended.

Where the separated components of formulations cannot be re-dissolved by methods which can also be used in the field before application of the formulation, this must be recorded in the sampling report.

NOTE Such a formulation is not suitable for use and separate samples should be taken from the segregated portions as evidence of the deteriorated condition. Subsequent analysis may be omitted. An estimate should be made of the amount or depth of any sediment or separated layers remaining.

The samples from containers, which are to be opened, should be collected, using appropriate apparatus such as a pump or pipette, into glass bottles or other containers that can be tightly closed.

9.2.2 Particulate solids (dusts, dispersible powders, water dispersible granules, granular formulations)

Particulate solid formulations are not usually sensitive to frost, although they are frequently affected by high temperatures and moisture, a batch that has been stored in an exposed state (e.g. in inadequately sealed bags) must be regarded as non-uniform and the number of bulk samples taken should be in accordance with [table 1](#).

Granular formulations (especially those such as GR, WG, SG, etc.) may undergo mechanical segregation of the different particle sizes during transport and when being transferred to other containers. At the initial sampling and when samples are subdivided for preparing the laboratory sample and analytical portions, the utmost care must be exercised to obtain a representative portion of the material.

Where applicable, [annex A and annex B](#) should be used for sampling solids. Otherwise, bags may be sampled through a top corner. The primary samples from a single bag should be collected with an appropriate dip tube, trier, sampling probe or scoop and aggregated, to form bulk sample, in a glass bottle, plastic bag or other container that can be tightly closed. The dip tube, etc., should be inserted through the opening diagonally across the bag and should be long enough to reach the bottom. Using a long-handled scoop, the bag may be tilted so that primary samples can be taken from the top, middle and bottom parts of the bag. Other types of container should be opened appropriately and sampled in a similar way to bags.

The bulk sample from each bag, or other container of the pesticide, should be divided into 3 equal laboratory samples, preferably using a mechanical sample divider. If a mechanical divider is not available, laboratory samples should be prepared by manual division, paying the utmost attention to avoiding contamination of the working environment, as follows.

- Transfer the bulk sample into a polyethylene bag large enough to be about one third full.
- Mix the contents by inverting the securely closed bag at least 10 times, put the bag on a flat surface and spread the material within it over as large an area as possible. (The sample layer should be approximately 1 cm deep).
- Divide the spread material into 6 approximately equal portions and combine pairs of portions to form each laboratory sample (e.g. 1st and 4th; 2nd and 5th; 3rd and 6th).

Where a formulation in water-soluble bags is to be sampled, intact bags must be taken complete with contents, preferably from freshly opened commercial packs. The individual bags must not be opened but dispatched for testing as quickly as possible.

10 Shipping and transporting samples

Care must be taken to avoid spillage, leakage or deterioration of samples during packaging and transport. Pesticide samples, which are packaged improperly and broken during transport, can endanger the health of both transport handlers and laboratory staff.

The following example procedure may be adopted when packaging and shipping pesticide samples.

- a) Place each sealed sample container, clearly labelled with a sample number which corresponds to that on the accompanying sampling report, in a plastic bag and seal with tape.
- b) Line a robust container of about 4 L capacity (e.g. a plastic or metal can with a securely fitting lid) with a suitably large plastic bag.
- c) Half-fill the plastic-lined container with absorbent material to immobilize the sample bottles and to absorb any leakage from broken bottles.
- d) Place the sampling reports in a separate plastic bag, seal it and place it in the container. Fill the remainder of the container with absorbent material.
- e) Close the container and seal its lid. Attach securely labels showing:
 - i) The address of the testing laboratory, and the name of the contact unit or person;
 - ii) The appropriate hazard classification of the pesticide;
 - iii) Arrow(s) indicating the "up" position of the samples.

When pesticide samples are transported, the regulations of ICAO, IMO, RID or IATA must be met as applicable.

Annex A (normative)

Sampling of water dispersible granules

A.1 Scope

The method is suitable for preparing samples of water dispersible granules for the determination of the amount of active ingredient content or the physical properties of the formulation.

A.2 Outline of method

Water dispersible granules (WG) may have a relatively broad particle size spectrum so that segregation into different particle sizes may occur during transportation or handling of the products.

It is, therefore, essential to divide samples with great care. This may be achieved by using a dividing apparatus or by standardized manual dividing.

A.3 Apparatus

A.3.1 Mechanical sample divider

A.3.2 Polyethylene bag size about 30 cm square

A.3.3 Laboratory spoon-spatula

A.4 Procedure

A.4.1 With the dividing apparatus

Mechanical sample division is most suitable for taking representative individual samples. For the handling of the apparatus, refer to the instructions of the manufacturer.

A.4.2 Manual

Put a representative sample -of about 200 g of the WG into a sufficiently large polyethylene bag, filling this by at least one third.

NOTE If the original sample is bigger than 200 g, start the manual sampling procedure carefully. Spread out the contents of the entire container and take sub samples by spoon from 20 – 25 different positions per kg to get a representative sample of 200 g.

Mix the contents by turning the closed bag end over at least 10 times. Put the polyethylene bag on a flat surface and spread the granules within the bag over as large an area as possible. The sample layer should be approximately 1 cm thick.

Take the required sub sample from a minimum of five positions from the sample layer.

For samples taken from large container, a mechanical sample divider is preferred.

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Annex B (normative)

Dust content and apparent density of granular pesticide formulations

B.1 Sampling

B.1.1 Apparatus

Metal or plastic boxes 10 x 10 x 3.5 cm, with well-fitting lids

B.1.2 Procedure

Gently rotate the bulk containers for at least 10 full cycles and then allow standing for 2 min to allow any dust to settle before the package is opened.

Take at least three samples (350 ml x 3) of which one must be from the top, one from the middle and one from the bottom of container. For ease of sampling, tilt the container and fill the sampling boxes to the brim by using them as scoops. Leave a small air space (5 ml) in each box, secure the lids and label "for sieve analysis". Take a fourth sample (100 ml), secure the lid and label "for density determination".

Take care not to disperse dust into the air when sampling from bulk containers. Do not pour the material into the sampling boxes.

B.2 Preparation of sample

Remove the lids from the sample boxes, and dry at 18 °C to constant weight in an evacuated desiccators, using phosphorus (V) oxide as the desiccant.

B.2.1 Apparatus

B.2.1.1 Test sieves

RS ISO 565 or equivalent, 200 µm diameter, nominal aperture sizes µm: 150, 250, 355, 420, 500, 710, 850.

B.2.1.2 Lid and a receiver pan for the sieves

B.2.1.3 Soft brush (2.5 cm flat) for cleaning the finer mesh sieves

B.2.1.4 Stiff brush for cleaning the coarser mesh sieves

B.2.1.5 Balance sensitive to 0.1 g, with a precision of ± 0.05 g

B.2.1.6 Beakers 10 to 300 ml

B.2.1.7 Sheets of glazed paper not less than 38 cm square

B.2.1.8 Incline test sieve shaker

Model No 1, 8-inch (about 20 cm) platform. The table of the incline sieve shaker, on which the platform rests, is inclined to the axis of the unit at an angle of 4.5 degrees and its movement is like an inclined disc rolling around its perimeter, the speed of rotation being normally 2.5 rpm. A few models have been marketed with rotational speeds of approximately 2.1 rpm but the manufacturer can rapidly modify them. This gyrator simultaneously vibrates up and down through 4 mm, at a frequency of about 300 vibrations per minute. The rotary and vibratory movements are governed by the same mechanism; hence it is sufficient to measure the speed of rotation of the fraction inclined table to check the characteristics of a particular machine. The period allowed for sieving is 45 min at 2.5 ± 0.04 rpm. If it is outside this range, the granular material collected or machine is unsuitable.

B.2.2 Procedure

Assemble the nest of sieves in the correct order above the receiver pan.

Weigh a sample box and its contents. Empty the contents carefully on the coarsest sieve (850 μm) and brush out any residue into the sieve. Weigh the empty sample box and record the mass of sample transferred to sieve. Fit the lid to the nest of sieves and amount in the shaking machine. Shake for a period of 45 min.

The volume of sample should be between 300 and 350 ml and the approximate mass of samples of different apparent densities should be as follows:

Apparent density (g/ml)	Mass of sample (g)
1.0	300
Between 0.7 and 1.0	210 to 300
Between 0.4 and 0.7	120 to 210

Remove the nest of sieves from the machine after allowing time for suspended dust to settle (about 2 min). Carefully remove the lid and invert each sieve over separate sheets of paper, tap the side of the sieve frame and carefully brush the uppermost surface. Invert the sieve and brush out any loose particles remaining, inside the sieve. Add the brushings to the bulk of the material taken from each sieve. Follow this procedure for the 850 μm to 250 μm sieves inclusive and record the mass to the nearest 0.1 g. also record the mass (to the nearest 0.1 g) of the material collected on the 150 μm sieve and in the receiver pan.

B.2.2.1 Loss of dust during sieving

The mass collected on the sieves, together with the mass of material collected in the receiver pan, are added together and subtracted from the mass of the sample taken. This gives the loss during sieving which should be less than 0.25% of the sample mass. If not, repeat the test.

Add the mass of sample lost on sieving (less than 0.25% of the total mass) to that of the fraction passing the 150 μm sieve. Express the mass of each sieved fraction as a percentage of the sample mass, to the nearest 0.1%.

B.2.2.2 A typical result

Fraction	Mass percent distribution
Granular material collected on 850 µm test sieves (inclusive)	96.2
Dust fraction through 250 µm and retained on 150 µm test sieve	3.1
Dust fraction through 150 µm test sieve in receiver pan (including loss during sieving)	0.7
	Total 100.0

Calculate the cumulative percentage by mass of the sample passing through the 250 µm sieve, by adding the calculated percentage retained on the 150 µm sieve to that passing through it into the receiver pan. In the above example, this would be 3.8% of the sample mass.

B.2.2.3 Interpretation of results

In the United Kingdom, legislation that the more toxic pesticides in granular form shall have no more than four percent by mass (cumulative percentage) passing the 250 µm sieve, and not more than one percent passing the 150 µm sieve. Otherwise appropriate protective clothing must be worn. The accuracy of the sieving procedure in determining this percentage distribution of the dust fractions (particles less than 250 µm diameter) is normally no more than 10% of the specified limits. Hence, a product found to have 4.4% by mass passing the 250 µm mesh sieve and 1.1% passing the 150 µm mesh sieve just complies with the specification.

B.3 Apparent density after compaction without pressure

B.3.1 Apparatus

B.3.1.1 Dropping box

Firmly screwed to a rigid table or bench (figure 1).

B.3.1.2 Measuring cylinder

Glass cylinder without spout and fitted with a rubber bung; the base of the cylinder shall be ground flat, and the weight of the cylinder and rubber bung shall be 250 ± 2 mm, i.e. the total lift of the cylinder shall be adjusted to 25 ± 2 mm by the application of suitable packaging to the under side of the shelf.

B.3.1.3 Rubber base pad

The rubber base pad shall have a BS hardness of 35 to 50.

B.3.1.4 Timing device to indicate seconds

B.3.1.5 Balance or scales

A type providing easy access to the pans, the pans being at least 10 cm in diameter. It is necessary that the pointer should show a significant deflection for a change of load of 0.25 g.

B.3.1.6 Sampling paper black glazed

B.3.1.7 Finger stalls or gloves smooth rubber

B.3.1.8 Test sieve 500 μm

B.3.2 Procedure

Weigh 40 g of sample into a beaker, pour gently into the cylinder and fit the rubber bung into the cylinder without jolting. Put it carefully in the dropping box and start the timing device.

With the thumb and forefinger of one hand gently hold the upper part of the cylinder and, during 1 sec, lift it to the full extent of its travel. Avoid any undue impact against the upper stop so that no jar is given to the sample. At the beginning of the next second, smartly release the cylinder by quickly and completely withdrawing the thumb and forefinger. Continue the process of lifting and dropping until 50 counted drops are completed, the cylinder falling once every 2 sec. rotation of the cylinder through an arc of about 10 degrees should be given during the lifting which precedes each drop, since this will help to impart a level surface to the sample for the final volume reading.

Immediately the 50 drops are completed, remove the cylinder from the dropping box and note the volume to the nearest 1 ml (V). Any further drop in the level after standing should be ignored.

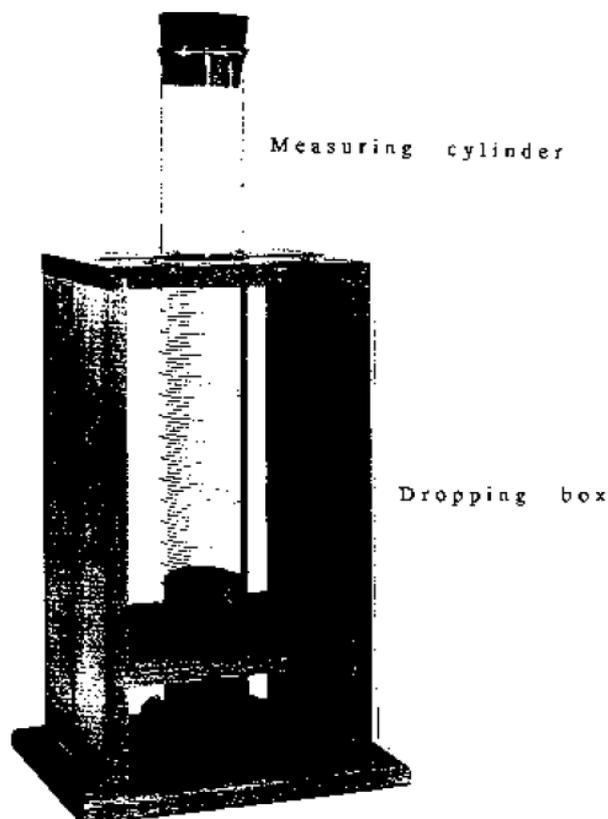


Figure B1 – Tap density apparatus

B.3.3 Calculation of results

Calculate the apparent density, D after compaction of the sample to two significant figures.

$$D = \frac{40}{V} \text{ g/ml}$$

B.3.4 Interpretation of results

The statutory specification for pesticides in granular form requires the apparent density by compaction without pressure to be not less than 0.4 g/ml.

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Annex C (Informative)

Supply and certification of reference substances of pesticide active ingredients

C.1 Introduction

Reference substances of known content are often essential for the quantitative determination of pesticide active ingredients in different types of samples and at various concentration levels. They are available from various sources. It is important that the active ingredient content is declared and certified.

C.2 Supply of reference substances

There are two sources for the supply of certified reference substances:

- a) The manufacturer of the active ingredient in question;
- b) National Standards Bodies or companies selling substances

C.3 Supply by the manufacturer

The manufacturer of a pesticide technical grade active ingredient, or its formulations, is obliged to supply a certified reference substance in the following cases:

C.3.1 Trade relations between customer and manufacturer

The customer, or an independent laboratory, might want to check the active ingredient content of material delivered by the manufacturer. This is to be done on the basis of the specification and using the analytical method stated therein. The manufacturer's certified reference substances should be made available to the customer so as to avoid any discrepancy in the analytical results, which could otherwise be derived from differences in the purity of the reference substances used by the two parties.

C.3.2 Relations between manufacturer and registration authorities

Each company (not only the inventor company) applying for the registration of a pesticide (new a.i. or formulation) must supply its own certified reference substance, if required by the registration authorities.

C.3.3 Supply by reference substances producers or suppliers

There are various standards institutions and commercial companies, which offer reference substances. They should normally be approached if reference substances are required in situations different from those mentioned above. On request manufacturers may, at their discretion, also supply reference substances in such cases.

C.4 Certification

A certificate of identity and determined content, together with the corresponding measurement uncertainty and a reference to the method(s) of analysis used, must always accompany each sample of a certified reference substance. Descriptions such as “greater than ...%” are unacceptable. The recommended storage conditions and an expiry date shall be stated.

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Annex D
(Informative)

Model sampling report

Name and address of store or plant:

.....
.....
.....

Name of pesticide:

Manufacturer/supplier:

Batch No.:

Batch total quantity (kg, l or pieces):

Date of manufacture:

External tests

Number of packaging units selected:

Minimum and maximum measured gross masses of containers:

Mean measured mass of empty containers (if applicable):

Estimated nominal gross mass of containers (if applicable):

Quality of packaging:

Quality of label:

Sampling for laboratory tests

Number of packaging units sampled:

Amount and number of primary samples taken for bulk sample:

Location of the reference sample and name of responsible person:

Remarks:

.....
.....
.....

Date:

.....

Name and signature of Inspector

.....

Name and signature of owner or

Representative of store/plant

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