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DRAFT EAST AFRICAN STANDARD

Carbaryl dusting powders — Specification

EAST AFRICAN COMMUNITY

Second Edition 2021

Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS) and other deliverables. The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the principles and procedures for development of East African Standards.

East African Standards and other deliverables are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 069, *Organic and Inorganic chemicals.*

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

This second edition cancels and replaces the first edition (EAS 361:2004), which has been technically revised.

Carbaryl dusting powders — Specification

1 Scope

This Draft East African Standard prescribes the requirements, sampling and test methods for carbaryl dusting powders.

2 Normative references

The following referenced documents are indispensable for the application 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.

3 Terms and definitions

No terms and definitions are listed in this document. ISO and IEC maintain terminological databases for use in standardization at the following addresses: — ISO Online browsing platform: available at http://www.iso.org/obp

4 Requirements

4.1 General requirements

4.1.1 The Carbaryl dusting powders shall be in the form of a powder. It shall be free-flowing and devoid of lumps.

Carbaryl technical, used in the manufacture of the Carbaryl dusting powders shall have been uniformly incorporated in a suitable filler.

4.1.2 The colour of the Carbaryl dusting powders shall be either cream, or grey.

4.1.3 The Carbaryl dusting powders, when dusted from a hand rotary, shall issue freely without clogging or bridging.

4.2 Specific requirements

The Carbaryl dusting powders shall comply with specific requirements given in Table 1 when tested according to the methods prescribed.

Table - 1: **Specific requirements for carbaryl dusting powders**

S/No.	Characteristic	Requirement	Test Method
i	Carbaryl content %m/m, min.	5% of declared value	Annex A
ii	Material passing through 75-micron sieve, % m/m, min	90	Annex B
iii	Bulk density, % m/m, max	60	Annex C
iv	Acidity (as H_2SO_4) %m/m, max or Alkalinity (as NaOH), %m/m, max	0.05	Annex D

NOTE: The actual value of the technical Carbaryl dusting powders in the formulation shall be calculated to the second decimal place for rounding off the first decimal place before applying the tolerance.

5 Packaging

The Carbaryl dusting powders shall be packed in securely closed suitable containers that protects its integrity.

6 Labelling

6.1 Each package shall be legibly and indelibly labelled in English and/or any other official language (French, Kiswahili, etc) used in the importing East African Partner State with the following information:

a) the name of the product as “Carbaryl dusting powder”

b) manufacturer’s name and physical address;

c) carbaryl content

d) net content in grams

e) country of origin;

f) batch number or lot number;

g) date of manufacture and expiry date.

h) cautions for safety such as signal words, hazard pictogram, precaution statement, and hazard statement

6.2 Carbaryl dusting powders shall be supplied with the safety data sheet which is written in language as depicted in 6.1

7 Sampling

Random samples of Carbarly dusting powder shall be drawn for test in accordance with Annex E.

Annex A

(Normative)

Determination of carbaryl content

A.1 Polarimetric method

Principle of the method

The Carbaryl dusting powders is heated with methanolic sodium hydroxide. To the resultant 1-naphnol, iodine solution (see A.2.2.3) is added and the violet colour thus produced is measured in a colorimeter and the carbaryl content is calculated by running a sample of analytical grade carbaryl along with the Carbaryl dusting powders under analysis.

A.2 Reagents

A.2.1 Methyl alcohol

Distilled, boiling range 64 to 65 °C.

A.2.2 Standard methanolic sodium hydroxide solution

0.5 N, prepared by weighing 10 g of sodium hydroxide pellets, dissolving in 10 ml of water and making up 500 ml with distilled methanol in a volumetric flask.

A.2.3 Colour reagent solution

Prepared by dissolving 1.27 g of iodine, 4 g of potassium iodine in 50 ml water in a beaker. This is then transferred to a 100 ml volumetric flask, shaken well and made to mark with water. It is to be ensured that all iodine goes into solution. This solution is diluted ten times its volume in water. This diluted solution is used for developing colour.

A.3 Procedure

A.3.1 Preparation of sample solution

Weigh enough Carbaryl dusting powders containing carbaryl close to 0.06 g in a 100 ml Erlenmeyer flask. Pipette exactly 1 ml of standard methanolic sodium hydroxide solution followed by 10 ml methyl alcohol into the flask. Heat to boil. Transfer the solution quantitatively into 100 ml volumetric flask. Wash the Erlenmeyer flask with methyl alcohol two or three times and add washings to the volumetric flask and finally make up to the mark with methyl alcohol. Pipette out 5 ml of the above stock solution into 500 ml volumetric flask. Add 300 ml of water and 20 ml of colour reagent solution, and finally make up to mark with water. Shake well.

A.3.2 Preparation of standard carbaryl solution

Weigh carbaryl, analytical grade (see note), close to 0.06 g in 100 ml Erlenmeyer flask and proceed as described in A.3.1.

NOTE Carbaryl, analytical grade, may be prepared by reported recrystallization of carbaryl, technical from toluene.

A.3.3 Preparation of blank solution

Weigh the same amount of carbaryl formulation that is equivalent to the sample weighed for analysis of carbaryl content. Transfer to 100 ml volumetric flask and make up to the mark with methyl. Shake well. Pipette out 5 ml of this solution to 500 ml volumetric flask containing 20 ml of colour reagent solution and 300 ml of water. Make up to the 500 ml mark with water, shake well and use this solution as blank.

A.3.4 Measurement of absorbance

Take the above three solutions to the colorimeter and fill one of the cells with the blank solution and adjust the colorimeter to zero absorbance at wavelength of 540 nm. Fill another cell with standard carbaryl solution and measure the optical density at 540 nm. Similarly, measure the optical density for the sample solution.

NOTE Measurement of optical density shall be carried within 15 minutes after addition of colour reagent.

A.4 Calculation

Carbaryl content, percent by mass = $\frac{A \times B}{C \times D} \times 100$

where,

- *A* Mass in g of the material in standard solution;
- *B* Optical density of sample;
- *C* Mass in g of the material in sample solution; and
- *D* Optical density of the standard solution.

Annex B

(Normative)

Test for sieving requirement for particle size

B.1 Apparatus
Test sieve prepared for the test by removing any film, grease or other water repellent material and then by drying.

B.2 Procedure
B.2.1 Dusting powders - Weigh accurately 10·0 g of the material and transfer it to the test sieve. Cover the sieve and screen the material in a Ro-Tap or a similar machine for 20 minutes, two small square rubber cubes are introduced along with the material on the sieve to facilitate the breaking-up of any soft -lumps of the caked material. After 20 minutes stop the machine and brush the residue on the sieve into a tared weighing dish. Weigh the dish and determine the mass of the residue.

B.2.2 Granules - Weigh 100 g of the material and transfer to a deck of sieves consisting of sieves of upper declared limit, lower declared limit and a receiver. Screen for 15 minutes, collect separately the fractions retained on sieves. Weigh separately fractions retained on the sieve of upper declared-limit and collected in the receiver. Determine the oversize retained on the sieve of upper declared limit and that passing through the sieve of lower declared limit.

B.3 Calculation

Materials passing through specified sieve in percentage by mass is calculated as follows:

$$100\,(1-\frac{m}{M})$$

Where,

m = mass of the materials retained on the- test sieve, in g and
M = mass of the material taken for the test in g.

Annex C

(Normative)

Test for balk density after compacting

C.1 Apparatus
C.1.1 Graduated cylinder - of 100 ml capacity with internal diameter of 27 to 29 mm
C.1.2 Funnel - wide and short-stemmed

C.2 Procedure
C.2.1 Bulk density before compacting - Rest the funnel over the top edge of the tared graduated cylinder. Fill the cylinder to the 100-ml mark by pouring the material through the funnel, without tapping, and level the powder with the minimum of disturbance. Leave the cylinder untouched for 5 minutes and add more powder if necessary, to bring the contents to the 100-ml mark, level again with the minimum of disturbance. Weigh the filled cylinder and calculate the bulk density before compacting (mass of the material/volume occupied by the material).

C.2.2 Bulk density after compacting - Stopper the filled cylinder (see. C.2.1), and drop it 20 times through a height of 15 cm on to a felt pad resting on a hard surface. Note the volume of the material after compacting, Calculate the bulk density of the material after compacting (mass of the material/volume occupied by the material after compacting).

C.3 Test Evaluation - The value obtained in C.2.2 shall not exceed the value obtained in C.2.1 by more than 60 percent.

Annex D

(Normative)

Test for Acidity or Alkalinity

D.1 Qualitative test

Procedure - Take about 0.5 g of the material in a test-tube and mix with about 1 ml of distilled water. Test the mixture for acidity or alkalinity with a litmus paper. Determine the acidity (see D.2) or the alkalinity (see D.3), as the case may be.

D.2 Determination of acidity

D.2.1 Reagents

D.2.1.1 Methyl red indicator solution-aqueous – 1% (m/v).
D.2.1.2 Bromocresol purple indicator solution – 1% (m/v) in ethyl alcohol.
D.2.1.3 Standard sodium hydroxide solution – 0.05 N.
D.2.1.4 Standard hydrochloric acid – 0.05 N.

D.2.2 Procedure

Weigh accurately 10.0 g of the material into a dry conical flask, add 5 ml of acetone and mix. Warm the flask gently to affect the solution of the active ingredient present. Add 75 ml of water and let it stand for an hour. Filter the supernatant aqueous extract and take 50 ml filtrate. Titrate immediately with the standard sodium hydroxide solution using methyl red or bromocresol purple as the indicator. Carry out a blank determination on an aliquot of 50 ml made from 25 ml acetone and 75 ml water.

D.2.3 Calculation

Acidity (as H_2SO_4), percent by mass is calculated as follows:

$$\frac{4.9 \times 2\,(V - v)\,N}{M}$$

where

V = volume in millilitres of the standard sodium hydroxide solution required for the test with the material,
v = volume in millilitres of the standard sodium hydroxide solution required for the blank determination,
N = normality of the standard sodium hydroxide solution, and
M = mass in g of the material taken for the test.

In case the blank shows alkaline reaction, neutralize with the standard hydrochloric acid and calculate the acidity as follows:

$$\frac{4.9 \times 2\,(VN_1 - vN_2)\,N}{M}$$

where,

V = volume in millilitres of the standard sodium hydroxide solution required for the test with the material,
N_1 = normality of the standard sodium hydroxide solution,
v= volume in millilitres of the standard hydrochloric acid required for the blank determination,
N_2 = normality of the standard hydrochloric acid, and
M = mass in g of the material taken for the test.

D.3 Determination of Alkalinity

D.3.1 Reagents

D.3.1.1 Methyl red indicator solution – aqueous, 1% (m/v).
D.3.1.2 Bromocresol purple indicator solution – 1% (m/v) in ethyl alcohol.
D.3.1.3 Standard hydrochloric acid – 0.05 N.
D.3.1.4 Standard sodium hydroxide solution – 0.05 N.

D.3.2 Procedure - Weigh accurately 10.0 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to affect the solution of the active ingredient present, add 75 ml of water and let it stand for an hour. Filter the supernatant aqueous extract and take 50 ml of filtrate, Titrate immediately with the standard hydrochloric acid using methyl red or bromocresol indicator as the indicator. Carry out a blank determination on 50 ml aliquot made from 25 ml acetone and 75 ml water.

D.3.3 Calculation

Alkalinity (as NaOH), percent by mass is calculated as follows:

$$\frac{4.0 \ \times 2\ (V - v)\ N}{M}$$

where

V = volume in millilitres of the standard hydrochloric acid required for the test with the material,
v = volume in millilitres of the standard hydrochloric acid required for the blank determination,
N = normality of the standard hydrochloric acid, and
M = mass in g of the material taken for the test.

In case the blank shows acid reaction, neutralize with the standard sodium hydroxide solution and calculate the alkalinity as follows:

$$\frac{4.0 \ \times 2\ (VN_1 - vN_2)\ N}{M}$$

where,

V = volume in millilitres of the standard hydrochloric acid required for the test with the material,
N_1 = normality of the standard hydrochloric acid,
v= volume in millilitres of the standard sodium hydroxide solution required for the blank determination,
N_2 = normality of the standard sodium hydroxide solution, and
M = mass in g of the material taken for the test.

Annex E

(Normative)

Sampling of carbaryl dusting powder

E.1 General requirements of sampling

In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

E.1.2 Samples shall be neither taken nor stored at a place exposed to such weather as would effect basic change in its characteristics.

E.1.3 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

E.1.4 The sampling instrument shall be made of glass or a metal on which the material has no action and shall be clean and dry when used.

E.1.5 The sample containers shall be of such a size that they are almost but not completely filled by the sample. They shall be clean, dry, leakproof and be made of the material which shall not react with the contents of the sample.

E.1.6 Once the container is opened, care shall be taken to avoid the risk of deterioration of contents due to the presence of various factors, such as air, light and moisture.

E.1.7 Each sample container shall be sealed airtight after filling and marked with full details of sample such as date of manufacture, date of sampling, name of the manufacturer and other particulars of the consignment.

E.1.8 The sample or its container shall be marked to indicate the nature of the material and the risks associated with it, wherever necessary.

E.1.9 The sampler should always be alert for possible biases arising from the use of a particular sampling device or from the segregation of material.

E.1.10 Before drawing the test samples, contents of each container selected for sampling shall be, as far as possible, thoroughly mixed by suitable means.

E.1.11 In the case of liquid formulations the proper selection of a representative sample involves a consideration of the physical laws of liquids, their chemical activity, the miscibility of all constituents and the interference of any insoluble materials carried by the liquid.

E.1.12 Avoid ignition of the material from open flames or sparks produced by static electricity or by metal equipment or tools.

E.1.13 The operator shall have safe access to and from the place where the sample is to be taken. He shall also have a safe working place with necessary light and ventilation.

E.1.14 The person taking the sample shall be made fully aware of the nature of the hazards involved and precautions to be taken. In the case of toxic nature of material, the person should be instructed that in the event of his feeling unwell he should immediately report to the concerned authorities for medical attention.

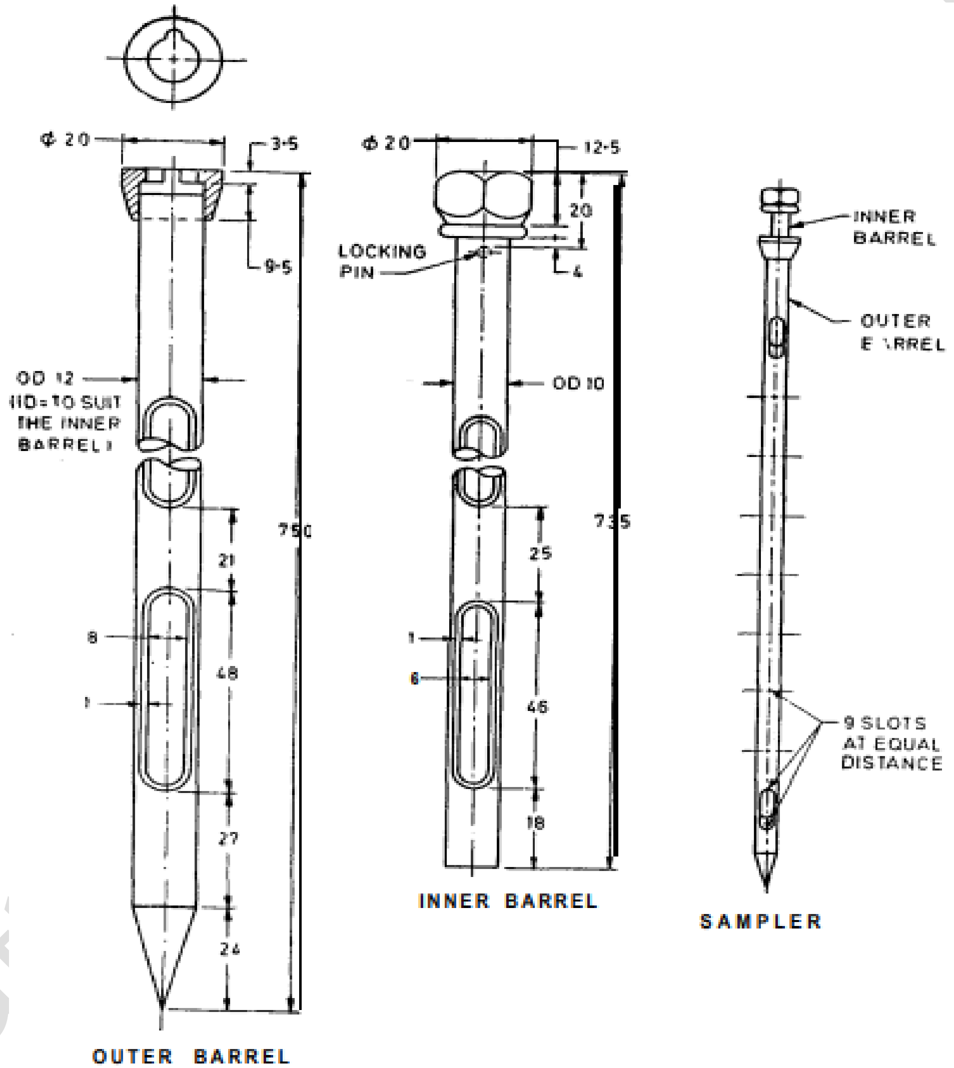
E.1.15 The operator engaged in sampling should have clean hands. It may be essential for the operator to wear clean gloves to safeguard against health or other hazards.

E.1.16 In the case of those formulations which change shape according to temperature, the material may have to be heated to a suitable temperature to bring it into the liquid state from the solid or cooled to a suitable temperature to bring it to solid crystalline state.

E.2 Sampling implements

Carbaryl dusting powder - Generally a sampler (or bag trier) consisting of two barrels (outer and inner) having slots at equal intervals, as shown in Fig. 1, is recommended. The working end of the outer barrel is pointed for easy insertion into the bag. The sampler shall be made of steel, brass or any other suitable material for taking the material from different layers of the bag. The slots of the sampler can be opened or closed by rotating the inner-barrel-head. The sampler is inserted into the bag in a closed position. After inserting the sampler to the desired depth, the sample material is collected by opening the slots of the sampler and then closing them. The sampler after this shall be taken out from the bag and then emptied on a smooth flat surface table/paper/any other suitable container. Thus, the sample of requisite quantity is collected and difference in the material taken out from different layers of the bag can also be studied.

NOTE - The tops of the inner and the outer barrels should be convenient for handling.



All dimensions in millimetres.

Fig. 1 Typical sampler for formulations in solid form

E.3 Process control

E.3.1 In order to ensure the desired quality of finished product, appropriate checks shall be made at raw materials and intermediate stages of the process of manufacture.

E.3.2 Each consignment of raw materials received in the factory shall be tested on the basis of a representative sample for technical content, moisture and acidity/alkalinity requirements. Alternately, a test certificate shall be obtained from the supplier that the material conforms to the specified requirements.

E.3.3 Appropriate checks shall be carried out on the bias of weighing machine, time of mixing various ingredients and performance of the -stirrer used for mixing in the case pesticidal formulations in liquid form.

E.3.4 Formulations in solid - The process of manufacture may be a batch or a continuous one. In a batch process various raw material are mixed at a time and fed to a grinder or a pulveriser from where the same are conveyed for homogenizing to a blender of known capacity. No further feeding of grinder or pulveriser is possible till such time the entire ground material is homogenized in the blender and the same is emptied out. For a continuous process of manufacture various raw materials are continuously fed to the pulveriser from where the same are continuously homogenized in a blender which is continuous to the grinding or the pulverizing unit. In this case there is no pause in the continuity of feeding the pulveriser and the finished material coming out of the blender for final packing.

E.3.5 Batch process - The quantity of material obtained by mixing various ingredients in a single operation in a mixer, not exceeding one tonne shall constitute a control unit (usually known as a batch in industry). Two representative samples shall be tested from each control unit, before packing, for active ingredient, and requirements. For remaining requirements, a composite sample prepared by mixing portions of material drawn at regular intervals shall be tested.

E.3.6 Continuous process - Five tonnes or part thereof, not exceeding a day's production, shall constitute a control unit. Five representative samples shall be taken at regular intervals from each control unit before packing, and tested for active ingredient. The remaining requirements shall be tested on the composite sample.

E.4 Lot inspection

E.4.1 General

If the manufacturer has maintained an adequate and satisfactory system of quality control in the manufacture of pesticidal formulations, the resulting data and information may be made available to the purchaser along with the material supplied to enable him to judge the acceptability of the consignment.

the following clauses shall be followed for determining the conformity of the material to the requirements of the specification.

E.4.2 Scale of Sampling

E.4.2.1 Lot - In a single consignment, all the containers of the same type, same grade and belonging to the same batch of manufacture shall be grouped together to constitute a lot.

NOTE - The definition of batch shall depend on the type of process of manufacture, that is, whether a batch process or a continuous one. The distinction between the two processes is explained in E.3.4.

E.4.2.2 For ascertaining the conformity of the material to the requirements of the individual specification, samples shall be tested from each lot separately.

The number of containers to be chosen from a lot shall depend on the size of the lot and shall be according to Table 2

Table 2 – Number of containers to be selected for sampling

Quantity of material in the lot (kg)	For containers of up to 25 kg	For containers of more than 25 kg
Up to 1 000	3	2
1 001 to 3 000	5	3
3 001 to 10 000	7	5
10 001 to above	10	7

E.4.3 Test samples and referee samples

E.4.3.1 Test samples

E.4.3.1.1 Before drawing the test samples, mixing the contents of each container by shaking is usually not practical. Draw small portions of the material from as many places as practicable throughout the volume of each selected container (see Table 2). The total quantity of material drawn from each container shall be sufficient so as to make triplicate determinations for all the characteristics given in the individual specification, mix thoroughly all the portions of material from the same container.

E.4.3.1.2 A small but approximately equal quantity of material shall be taken from each of the selected containers and shall be well mixed so as to form the composite sample. The quantity of material in the composite sample shall be sufficient so as to make triplicate determinations for the specification requirements to be tested on the composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the vendor and the third for the referee.

E.4.3.1.3 The remaining portions of the material (after the quantity needed for the formation of the composite sample has been taken) shall be divided into three equal parts. These parts shall be immediately transferred to thoroughly dried sample containers which are then sealed air-tight and labelled with all the particulars of sampling. The material in each sealed sample container shall constitute an individual test sample. These individual test samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each selected container. One of these three sets shall be marked for the purchaser, another for the vendor and the third for the referee.

E.4.3.2 Referee Samples –

Referee samples shall consist of a composite test sample and a set of individual test samples marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept in a cool and dry place as agreed to between the purchaser and the vendor so as to be used in case of dispute between the two.

E.4.4 Number of Tests

Active ingredient and sieving (particle size) shall be tested on individual samples and the remaining requirements on the composite sample.

E.4.5 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification if E.4.5.1, E.4.5..2 and E.4.5.3 are satisfied.

E.4.5.1 5.5.1.1 All the test results for each of the characteristics tested on individual samples (see 5.4.1 and 5.4.2) satisfy the relevant specification requirements.

E.4.5.2 The average of all the test results for active ingredient is greater than or equal to the nominal value.

E.4.5.3 For the remaining requirements all the test results on the composite sample satisfy the relevant specification requirements.

Bibliography

EAS 361:2004 Carbarly powders – specification

IS 6940:1982 Methods of test for pesticides and their formulations

IS 10627:1983 Methods for sampling of pesticidal formulations