**BUREAU OF INDIAN STANDARDS**

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| *भारतीय मानक मसौदा*  **कृषि एवं खाद्य वस्तुओं में कीटनाशक अवशेषों के निर्धारण के लिए नमूना लेने की विधि** |
|  |
| *Draft Indian Standard* |
| **Pesticide Residues Analysis in Agricultural and Food Commodities –**  **Method of Sampling** |
|  |
| **ICS No.** |

|  |  |
| --- | --- |
| Pesticide Residues analysis Sectional Committee, FAD 27 | Last Date of Comments: |

**FOREWORD**

(*Formal clause would be added later*)

For the examination of a lot in order to decide whether it complies with maximum ‘limits for pesticide residues, it is necessary to provide a representative sample for analysis. The objective of this sampling procedure is to obtain a final sample representative of the lot in order to determine

its average pesticide residue content in agricultural and food commodities.

Standardized sampling approach for pesticide residue analysis in food is essential to ensure accuracy, regulatory compliance, consumer safety, cost efficiency, and international harmonization. This leads to better protection of public health and facilitates smooth trade and research activities.

This standard was first published in 1985. This revision is being undertaken to update the sampling techniques and guidelines with the latest practices.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding-off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

**1 SCOPE**

This standard prescribes the procedures for drawing, transport and storing the samples for the examination of a lot of agricultural and food commodities with respect to pesticide residue analysis.

**2 TERMINOLOGY**

**2.1 Sampling Plan –** A predetermined sequential activity for the selection, withdrawal, preservation, and preparation of a sample from a lot in order to get the information needed, such as a decision on compliance status of the lot.

**2.2 Sampling –** The procedure used to draw and constitute a sample from a lot.

**2.3 Sampling Device** – Tools such as a scoop, dipper, borer, knife, or spear used to remove a primary or incremental sample (primary unit) from a lot.

**2.4 Sampling Officer –** A person trained in sampling procedures and, where required, authorized by the appropriate authorities to take samples. The sampling officer is responsible for sampling, sample packing and shipping of the laboratory sample(s).

**2.5 Unit –** The smallest discrete portion in a lot which should be withdrawn to form the whole or part of a primary sample/incremental sample. For example, each whole fruit, vegetable, or natural bunch/bunchlets.

**2.6 Sample (Representative Sample) –** One or more units or a portion of the material selected from a larger quantity of material or a population. A representative sample is intended to elaborate the lot regarding its targeted attribute (pesticide residue content).

**2.7 Primary Sample/Incremental Sample –** One or more units are taken from one position in a lot. The primary sampling positions should be chosen randomly, covering the entire lot. Units taken for sampling should not be cut or broken to produce the primary sample(s), except where a subdivision of units is explicitly specified. The number of units and quantity required for the primary sample should be decided based on the minimum quantity and number of laboratory samples required.

**2.8 Lot –** The definite quantity of food material identified by the sampling officer having uniform characteristics such as origin, farmer/owner, variety, packer, type of packing, markings, consignor, etc. A consignment comprises lots that can be identified as originating from different growers, etc. A consignment may consist of one or more lots and each lot should be considered separately.

**2.9 Bulk Sample/Aggregate Sample –** The primary samples combined and well-mixed taken from a lot. The primary samples should be sufficient to form the aggregate sample so that the required laboratory sample can be withdrawn from the bulk sample.

**2.10 Laboratory Sample –** The sample send to or received by the laboratory. A representative quantity of material is removed from the bulk sample. The laboratory sample should be the whole or a part of the bulk sample/aggregate sample.

**2.11 Consignment –** A consignment is a quantity of some commodity delivered at one time. It may consist in either a portion of a lot, either a set of several lots. If the consignment is a portion of a lot, each portion is considered as a lot for the inspection.

**2.12 Analytical Sample –** The material prepared for analysis from the laboratory samples, with separation of the portion to be analysed followed by mixing, grinding, fine chopping etc. for removal of analytical portion with minimal sampling error.

**2.13 Analytical Portion –** A representative quantity of material taken from the analytical sample, of proper size/ quantity for measurement of residue concentration.

**3 GENERAL REQUIREMENTS OF SAMPLING**

**3.1 Precautions to be taken** – Contamination and deterioration of samples must be prevented at all stages, because they may affect the analytical results.

**3.2** Transport condition should be maintained to prevent any sought of contamination and deterioration of samples which affect the analytical results.

**3.3** Sampling device should be selected such a way that it will not physically damage the sampling units affecting the lot integrity

**3.4** Traceability of the sample should be maintained at lot level.

**4 SAMPLING PROCEDURE**

Each lot that is to be examined must be sampled separately. The laboratory samples to be collected varies with the commodities.

**4.1 Collection of Primary/Incremental Samples –** The minimum number of primary samples drawn from a lot is pre-determined (Table 1), considering that the primary samples should be sufficient to provide the laboratory sample(s) for analysis. The primary sample may be taken from randomly chosen positions in the lot as prescribed.

**Table 1 Minimum Number of Primary Samples to be Taken from a Lot**

(*Clause* 4.1)

|  |  |  |
| --- | --- | --- |
| **Sl No.** | **Product** | **Minimum number of primary samples to be taken from the lot** |
|  |  |  |
|  | **Meat and poultry** |  |
|  | a non-suspect lot | 1 |
| b) | **Other products** |  |
|  | Products, packaged or in bulk, which can be assumed to be well mixed or homogeneous | 1 |
|  | Weight of lot (Kg) |  |
|  | < 50 | 3 |
|  | 50-500 | 5 |
|  | > 500 | 10 |
|  | Number of cans, cartons or other containers in the lot |  |
|  | 1-25 | 3 |
|  | 26-100 | 5 |
|  | > 100 | 10 |
| NOTE – For products comprised of large units, the minimum number of primary samples should comply with the minimum number of units required for the laboratory sample (*see* Table 3). | | |

**4.2 Preparation of Bulk/Aggregate Sample –** For meat and poultry, each primary sample is considered to be a separate bulk sample (Table 2). For plant products, eggs or dairy products, the primary samples should be combined and mixed well, if practicable, to form the bulk sample (Tables 3 and 4). An alternative procedure may be adopted where mixing to form the bulk sample is inappropriate or impractical. Where units may be damaged (and thus residues may be affected) by the processes of mixing or sub-division of the bulk sample, or where large units cannot be mixed to produce a more uniform residue distribution, the units should be allocated randomly to replicate laboratory samples at the time of taking the primary samples. In this case, the result to be used should be the mean of valid results obtained from the laboratory samples analyzed.

**4.3 Preparation of the Laboratory Sample –** The laboratory sample may be prepared from the bulk sample, if the bulk sample is larger than the required laboratory sample. An appropriate size reduction process, such as quartering, may be used. However, fresh plant products should not be cut or broken otherwise specified. If required, replicate laboratory samples may be withdrawn at this stage. For the minimum sizes required for laboratory, refer to tables 2, 3 and 4.

**4.4 Sampling Record –**

The laboratory shall retain following records related to sampling;

1. The nature and origin of the lot;
2. The farmer/owner's name;
3. Reference to the sampling method and plan used;
4. Date and time of sampling;
5. Purpose of sampling;
6. Details to identify and describe the sample (e.g. lot number, amount, name etc);
7. Identification of the personnel performing sampling;
8. Identification of the equipment used;
9. Environmental or transport conditions;
10. Diagrams or other equivalent means to identify the sampling location, when appropriate; and
11. Deviations, additions to or exclusions, if any; from the sampling method and plan.

The sampling record should be signed by the sampling officer and must accompany each replicate laboratory sample, and the sampling officer should retain a copy of the record. If sampling records are produced in computerized form, the soft copy should be distributed to the recipients in order to maintain an audit trail.

**4.5 Packaging and Transmission of the Laboratory Sample –** The laboratory sample must be packed in a clean, inert container that prevents contamination, damage, and leakage. The container should be sealed, labelled, and the sampling record must be attached. The sample must be transported to the laboratory as soon as possible. Any kind of spoilage which affects the integrity of the sample should be avoided.

**4.6 Preparation of the Analytical Sample –** On receipt in the laboratory, the laboratory sample should be given a unique identification number, date, and sample size. From the laboratory sample, the analytical sample should be separated as soon as possible. Where the residue level is calculated, including parts that are not analyzed, the weights of the separated parts must be recorded (e.g., In the case of Mango, Mango stone is not used for analysis; however, the weight of the stone must be accounted at the time of reporting the residue content).

**4.7 Preparation and Storage of the Analytical Portion –** The analytical sample should be homogenized using the appropriate device to enable representative analytical portions. The size of an analytical portion should be determined considering the analytical method and the extent of homogenization. The method used for homogenization should not affect the targeted analyte concentration in the analytical samples. If required, the analytical sample may be processed under sub-zero temperature to minimize any degradation during homogenization. Homogenization could affect residues content, and any other practical alternatives are not possible; the analytical portion may consist of a whole laboratory sample. If the situation arises for the analytical sample's storage, the condition and storage duration should not affect the residues. If required, additional portions may be withdrawn for replicate and confirmatory analyses. A schematic diagram on drawing sample from a consignment is mentioned at Fig. 1.

**Consignment (C):**

One consignment may consist of one or more lots

**Lot (L):**

Each lot should be sampled separately

**Incremental sample (IS)**

Drawn from diff. position from L

**Aggregate sample (AS):**

Incremental sample pooled to AS

**Laboratory sample (LS):**

LS Transported to lab

**Analytical sample(AS):**

Analytical portion may be dram from AS

FIG 1SCHEMATIC DIAGRAM ON DRAWING SAMPLE FROM A CONSIGNMENT/LOT

**Table 2 – Meat and Poultry – Description of Primary Samples and Minimum Size of Laboratory Samples**

(*Clause* 4.2 *and* 4.3)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl No.** | **Commodity Classification** | **Examples** | **Nature of Primary Sample to be taken** | **Minimum size of each Laboratory Sample** |
| **(1)** |  |  |  |  |
| **Primary Food Commodities of Animal Origin** | | | | |
| 1. Mammalian meats | | | | |
|  | Large mammals, whole or half carcass, usually 10 kg or more | cattle sheep pigs | whole or part of diaphragm, supplemented by cervical muscle, if necessary | 0.5 kg |
|  | Small mammals, whole carcass | rabbits | whole carcass or hind quarters | 0.5 kg, after removal of skin and bone |
|  | Mammal meat parts, loose fresh/chilled/frozen packaged or otherwise | Quarters  Chops  steaks  shoulders | whole unit(s), or a portion of a large unit | 0.5 kg, after removal of bone |
|  | Mammal meat parts, bulk frozen | quarters  chops | Either a frozen cross-section of a container or the whole (or portions) of individual meat parts | 0.5 kg, after removal of bone |
| 1. **Mammalian fats, including carcass fat** | | | | |
|  | Large mammals, at slaughter, whole or half carcass Usually 10 kg or more | cattle  sheep  pigs | kidney, abdominal or subcutaneous fat cut from one animal | 0.5 kg |
|  | Small mammals, at slaughter, whole or half carcass <10 kg | ----- | abdominal or subcutaneous fat from one or more animals | 0.5 kg |
|  | Mammal meat parts | Legs  chops  steaks | either visible fat, trimmed from unit(s)  or whole unit(s) or portions of whole unit(s), where fat is not trimmable | 0.5 kg  2 kg |
|  | Mammal bulk fat tissue | ----- | units taken with a sampling device from at least 3 positions | 0.5 kg |
| 1. **Mammalian offals** | | | | |
|  | Mammal liver, fresh/chilled/frozen |  | whole liver(s), or part of liver | 0.4 kg |
|  | Mammal kidney, fresh/chilled/frozen |  | 1 or both kidneys from 1 or more animal | 0.2 kg |
|  | Mammal heart, fresh/chilled/frozen |  | Whole heart(s), or ventricle portion only, if large | 0.4 kg |
|  | Other mammal offal, fresh/chilled/frozen | intestines  brains | Part or whole unit from 1 or more animals, or a cross-section taken from bulk frozen product | 0.5 kg |
| 1. Poultry meats | | | | |
|  | Bird, large-sized carcass >2 kg | Turkey  Goose  mature chicken | thighs, legs and other dark meat | 0.5 kg after removal of skin and bone |
|  | Birds, medium-sized carcass 500 g-2 kg | duckling guinea fowl young chicken | thighs, legs or other dark meat from at least 3 birds | 0.5 kg after removal of skin and bone |
|  | Birds, small-sized carcass <500 g carcass | quail pigeon | carcasses from at least 6 birds | 0.2 kg of muscle tissue |
|  | Bird parts fresh/chilled/frozen, retail or wholesale packaged | legs quarters | packaged units, or individual parts | 0.5 kg (after removal of skin and bone) |
| 1. **Poultry fats, including carcass fat** | | | | |
|  | Birds, at slaughter, whole or part-carcass | chickens  turkeys | units of abdominal fat from at least 3 birds | 0.5 kg |
|  | Bird meat parts | legs breast muscle | either visible fat, trimmed from unit(s)  or whole unit(s) or portions of whole unit(s), where fat is not trimmable | 0.5 kg  2 kg |
|  | Bird fat tissue in bulk |  | units taken with a sampling device from at least 3 positions | 0.5 kg |
| 1. **Poultry offals** | | | | |
|  | Edible bird offal, except goose and duck fat liver and similar high value products |  | units from at least 6 birds, or a cross-section from a container | 0.2 kg |
|  | Goose and duck fat liver and similar high value products |  | unit from 1 birds or container | 0.05 kg |
| 1. **Processed Foods\*** | | | | |
|  | Mammal or bird, comminuted, cooked canned, dried, rendered, or otherwise processed products, including multi-ingredient products | Ham  Sausage  minced beef  chicken paste | packaged units, or a representative cross-section from a container, or units (including juices, if any) taken with a sampling device | 0.5 kg or  2 kg if fat content is less than 5% |
| NOTE – \*Processed foods will include secondary food commodities of animal origin, dried meats; derived edible products of animal origin, processed animal fats; manufactured food (single ingredient) of animal origin; manufactured food (multi-ingredient) of animal origin | | | | |

**Table 3 – Plant products – Description of Primary Samples and Minimum Size of Laboratory Samples**

(*Clause* Table 1, 4.2 *and* 4.3)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl No.** | **Commodity Classification** | **Examples** | **Nature of Primary Sample to be taken** | **Minimum size of each Laboratory Sample** |
| **Primary Food Commodities of Plant Origin\*** | | | | |
| 1 | **All fresh fruits**  **All fresh vegetables**, **except** (dry pulses) | | | |
| 1.1 | **Small sized fresh products** units generally < 25 g | berries peas olives | whole units, or packages, or units taken with a sampling device | **1 kg** |
| 1.2 | **Medium sized fresh products** units generally 25-250 g | apples oranges | whole units | **1 kg (at least 10 units)** |
| 1.3 | **Large sized fresh products** units generally > 250 g | cabbages cucumbers grapes(bunches) | whole units | **2 kg (at least 5 units)** |
| 2 | **Pulses**  **Cereal grains**  **Tree nuts** | soya beans  rice, wheat  except coconuts | units taken with a sampling device | **1 kg**  **1 kg**  **1 kg** |
| **Oilseeds** | peanuts | units taken with a sampling device | **5 units**  **500 g** |
| **Seeds for beverages and sweets** | coffee beans | units taken with a sampling device | **500 g** |
| 3 | **Herbs** | fresh parsley  others, fresh | whole units | **0.5 kg**  **0.2 kg** |
| 3.1 | **Spices- small size** | Dried-**(pepper, cardamom, cumin etc)** | whole units or taken with a sampling device | **0.2 kg** |
| 3.2 | **Spices- larger size** | Dried- (**(large cardamom, dry ginger, dry turmeric etc.)** | whole units or taken with a sampling device | **0.5 kg** |
| 3.3 | **Spices- High value** | Saffron | whole units or taken with a sampling device | **10 g** |
| **Primary Animal Feed Commodities** | | | | |
| 4 | **Primary feed commodities of plant origin** | | | |
| 4.1 | **Legume animal feeds, and other forages and fodders** |  | whole units, or units taken with a sampling device | **1 kg**  **(at least 10 units)** |
| 4.2 | **Straw, hay and other dried products** |  | units taken with a sampling device | **0.5 kg**  **(at least 10 units)** |
| **Processed Foods of Plant Origin** | | | | |
| 5 | **Secondary food commodities of plant origin**- dried fruits, vegetables, herbs, milled cereal products  **Derived products of plant origin**- teas, vegetable oils, juices, by-products for animal feed and miscellaneous products  **Manufactured foods (single ingredient) of plant origin**  **Manufactured foods (multi-ingredient) of plant origin**- including products with ingredients of animal origin where the ingredient(s) of plant origin predominate(s), and breads | | | |
| 5.1 | **Products of high unit value** | Saffron | packages or units taken with a sampling device | **0.1 kg\*** |
| 5.2 | **Solid products of low bulk density** | hops  tea | packaged units, or units taken with a sampling device | **0.2 kg** |
| 5.3 | **Other solid products** | bread  flour  apple pomace  dried fruit | packages or other whole units, or units taken with a sampling device | **0.5 kg** |
| 5.4 | **Liquid products** | vegetable oils  juices | packaged units, or units taken with a sampling device | **0.5 L or 0.5 kg** |
| \* A smaller laboratory sample may be taken from a product of exceptionally high value but the reason for doing so should be noted in the sampling record. | | | | |

**Table 4 Egg and Dairy Products – Description of Primary Samples and Minimum Size of Laboratory Samples**

(*Clause* 4.2 *and* 4.3)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl No.** | **Commodity Classification** | **Examples** | **Nature of Primary Sample to be taken** | **Minimum size of each Laboratory Sample** |
| **Primary Food Commodities of Animal Origin** | | | | |
| 1 | **Poultry eggs** | | | |
| 1.1 | **Eggs, except quail and similar** |  | whole eggs | **12 whole chicken eggs, 6 whole goose or duck eggs** |
| 1.2 | **Eggs, quail and similar** |  | whole eggs | **24 whole eggs** |
| 2 | **Milks** |  | whole unit(s), or unit(s) taken with a sampling device | **0.5 L** |
| **Processed Foods of Animal Origin** | | | | |
| 3 | **Secondary food commodities of animal origin**- skimmed milks, evaporated milks and milk powders  **Derived edible products of animal origin**- milkfats, butters, butteroils, creams, cream powders, caseins, etc.  **Manufactured food (single ingredient) of animal origin**  **Manufactured food (multi-ingredient) of animal origin** (including products with ingredients of plant origin where the ingredient(s) of animal origin predominates(s)) | | | |
| 3.1 | **Liquid milks, milk powders, evaporated milks and creams, creams, dairy ice creams, yoghurts** |  | packaged unit(s), or unit(s) taken with a sampling device | **0.5 L** (liquid) or  **0.5 kg** (solid) |
|  | NOTES –  (i) Evaporated milks and evaporated creams in bulk must be mixed thoroughly before sampling, scraping adhering material from the sides and bottom of containers and stirring well. About 2-3 l should be removed and again stirred well before removing the laboratory sample.  (ii) Milk powders in bulk should be sampled aseptically, passing a dry borer tube through the powder at an even rate.  (iii) Creams in bulk should be mixed thoroughly with a plunger before sampling but foaming, whipping and churning must be avoided. | | | |
| 3.2 | **Butter and butteroils** | butter, whey butter, low fat spreads containing butter fat, anhydrous butteroil, anhydrous milkfat | whole or parts of packaged unit(s), **or** unit(s) taken with a sampling device | **0.2 kg or 0.2 L** |
| 3.3 | **Cheeses, including processed cheeses** units 0.3 kg or greater |  | Whole unit(s) or unit(s) cut with a sampling device | **0.5 kg** |
| units < 0.3 kg |  | whole unit(s), or unit(s) cut with a sampling device | **0.3 kg** |
|  | NOTE – Cheeses with a circular base should be sampled by making two cuts radiating from the centre. Cheeses with a rectangular base should be sampled by making two cuts parallel to the sides. | | | |
| 3.4 | **Liquid, frozen or dried egg products** |  | unit(s) taken aseptically with a sampling device | **0.5 kg** |

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