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BUREAU OF INDIAN STANDARDS
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भारतीय मानक मसौदा

कृषि और खाद्य वस्तुओं में कीटनाशक अवशेषों का विश्लेषण — नमूना लेने की विधि

(IS 11380 का पहला पुनरीक्षण)

Draft Indian Standard

Pesticide Residues Analysis in Agricultural and Food Commodities — Method of Sampling

(First Revision of IS 11380)

ICS 65.100.01

Pesticide Residues analysis Sectional Committee, FAD 27	Last date of comments: **11 February 2025**

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.

Originally, this standard was published in 1985 and was in line with the Codex document 'Recommended Method of Sampling for the Determination of Pesticide Residues', CAC/Vol XIII-First Edition, 1983.

In preparing this revised standard, assistance has been derived from Codex document CAC/GL 33 'Recommended methods of sampling for the determination of pesticide residues for compliance with MRLs CAC/GL 33-1999' and major modifications are as under:

a) A number of new terms relating to sampling have been defined;
b) The minimum number of primary samples to be taken from a lot has been revised;
c) The procedure for preparation of the aggregate sample has been modified;
d) The minimum size of laboratory samples has been included in detail under different heads such as meat and poultry products, plant products, egg, and dairy products;

e) Sample sizes for processed foods have been detailed under different heads; and
f) Specific sample sizes for high value products (for example, spices like saffron) have been introduced

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 the samples for the examination of a lot of agricultural and food commodities with respect to pesticide residue analysis.

2 TERMINOLOGY

For the purpose of this standard, following definitions shall apply:

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.

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 sent 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 quantity of a commodity delivered at one time. It may consist of either a portion of a lot, or 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 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

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

4.2 Collection of Primary/Incremental Samples — The minimum number of primary samples drawn from a lot is pre-determined, considering that the primary samples should be sufficient to provide the laboratory sample(s) for analysis. Minimum number of primary samples to be taken from a lot is given in Table 1. 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* 4.2)

Sl No.	Product	Minimum number of primary samples to be taken from the lot
(1)	(2)	(3)
a)	**Meat and poultry**	1
b)	**Other products**	
	Products, packaged or in bulk, which can be assumed to be well mixed or homogeneous	1
1)	Weight of lot (in kg)	
i)	< 50	3
ii)	50-500	5
iii)	> 500	10
2)	Number of cans, cartons or other containers in the lot	
i)	1-25	3
ii)	26-100	5
iii)	> 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.		

4.3 Preparation of Bulk/Aggregate Sample — For meat and poultry, each primary sample is considered to be a separate bulk sample. Description of primary samples and minimum size of laboratory samples for meat and poultry is specified in 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. Description of primary samples and minimum size of laboratory samples for plant products and egg or dairy is given in Tables 3 and Table 4, respectively. 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.

4.4 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 unless 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.

4.5 Sampling Record

The laboratory shall retain following records related to sampling:

a) The nature and origin of the lot;
b) The farmer/supplier/manufacturer's name;
c) Reference to the sampling method and plan used;
d) Date and time of sampling;
e) Purpose of sampling;
f) Details to identify and describe the sample (e.g. lot number, amount, name etc);
g) Identification of the personnel performing sampling;
h) Identification of the equipment used;
j) Environmental or transport conditions;
k) Diagrams or other equivalent means to identify the sampling location, when appropriate; and
m) 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.

4.6 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.

4.7 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).

4.8 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 where any other practical alternatives are not possible; the analytical portion may consist of a whole laboratory sample. In case, the situation arises for the storage of analytical sample, 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.

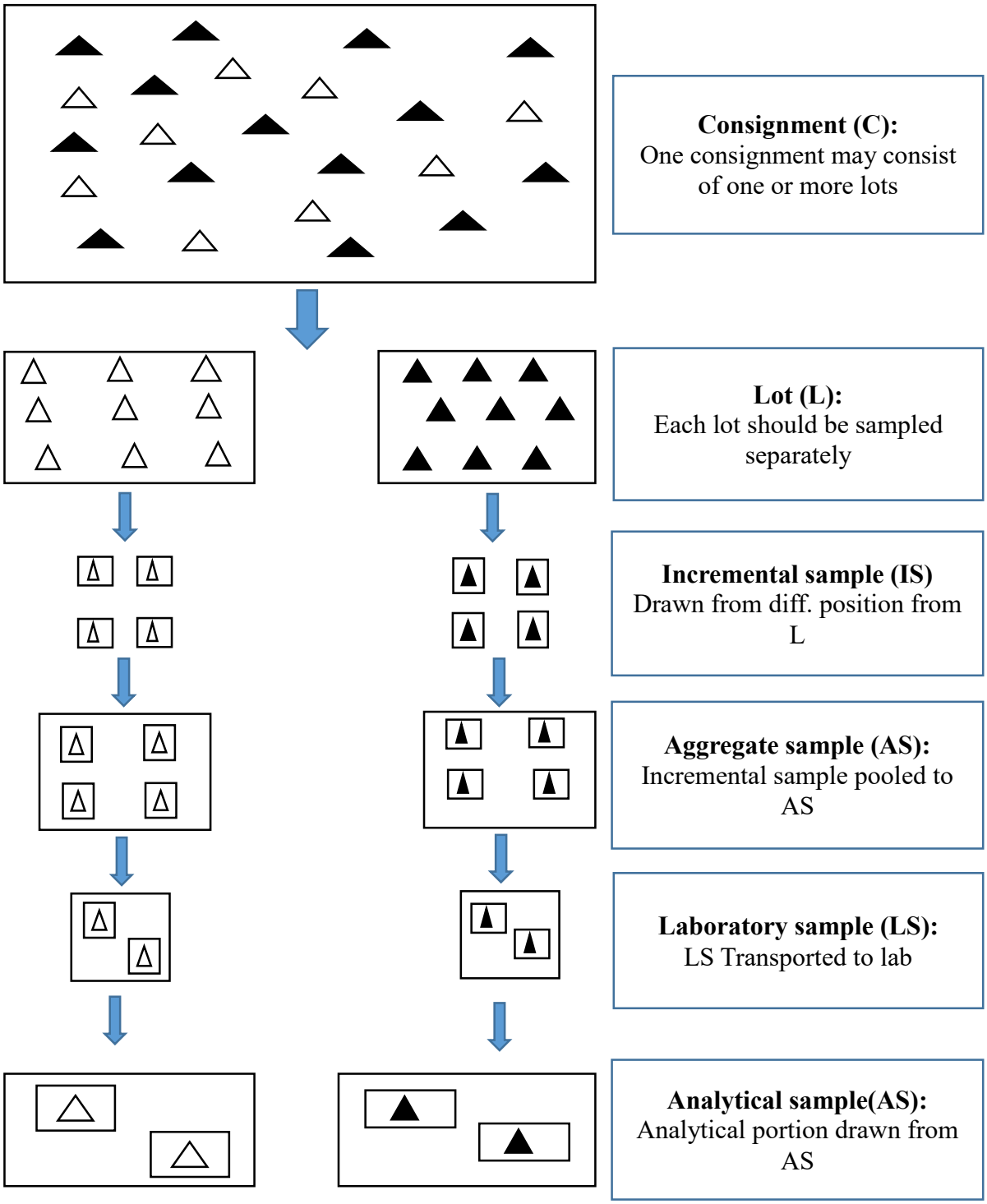

FIG 1 SCHEMATIC DIAGRAM ON DRAWING SAMPLE FROM A CONSIGNMENT/LOT

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

(*Clause* 4.3 *and* 4.4)

Sl No.	Commodity Classification	Examples	Nature of Primary Sample to be taken	Minimum size of each Laboratory Sample
(1)	(2)	(3)	(4)	(5)
A. Primary Food Commodities				
a) Mammalian meats				
i)	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
ii)	Small mammals, whole carcass	Rabbits	Whole carcass or hind quarters	0.5 kg, after removal of skin and bone
iii)	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
iv)	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
b) Mammalian fats, including carcass fat				
i)	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
ii)	Small mammals, at slaughter, whole or half carcass <10 kg	-	Abdominal or subcutaneous fat from one or more animals	0.5 kg
iii)	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

Sl No.	Commodity Classification	Examples	Nature of Primary Sample to be taken	Minimum size of each Laboratory Sample
iv)	Mammal bulk fat tissue	-	Units taken with a sampling device from at least 3 positions	0.5 kg
c) Mammalian offals				
i)	Mammal liver, fresh/chilled/frozen	-	Whole liver(s), or part of liver	0.4 kg
ii)	Mammal kidney, fresh/chilled/frozen	-	1 or both kidneys from 1 or more animal	0.2 kg
iii)	Mammal heart, fresh/chilled/frozen	-	Whole heart(s), or ventricle portion only, if large	0.4 kg
iv)	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
d) Poultry meats				
i)	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
ii)	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
iii)	Birds, small-sized carcass <500 g carcass	Quail pigeon	Carcasses from at least 6 birds	0.2 kg of muscle tissue
iv)	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
e) Poultry fats, including carcass fat				
i)	Birds, at slaughter, whole or part-carcass	Chickens turkeys	Units of abdominal fat from at least 3 birds	0.5 kg
ii)	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

Sl No.	Commodity Classification	Examples	Nature of Primary Sample to be taken	Minimum size of each Laboratory Sample
iii)	Bird fat tissue in bulk	-	Units taken with a sampling device from at least 3 positions	0.5 kg
f) Poultry offals				
i)	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
ii)	Goose and duck fat liver and similar high value products	-	Unit from 1 birds or container	0.05g
B. **Processed Foods**				
a)	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%

Table 3 Plant Products — Description of Primary Samples and Minimum Size of Laboratory Samples

(*Clause* 4.3 *and* 4.4)

Sl. No.	Commodity Classification	Examples	Nature of Primary Sample to be taken	Minimum size of each Laboratory Sample
(1)	(2)	(3)	(4)	(5)
A. Primary Food Commodities*				
a) Fresh Fruits and Fresh Vegetables				
i)	Small sized fresh products units generally < 25 g	Berries peas olives	Whole units, or packages, or units taken with a sampling device	1 kg
ii)	Medium sized fresh products units generally 25-250 g	Apples, oranges	Whole units	1 kg (at least 10 units)
iii)	Large sized fresh products units generally > 250 g	Cabbages, cucumbers,	Whole units	2 kg (at least 5 units)

Sl. No.	**Commodity Classification**	**Examples**	**Nature of Primary Sample to be taken**	**Minimum size of each Laboratory Sample**
(1)	(2)	(3)	(4)	(5)
		grapes (bunches)		
b)	Pulses	Soyabeans	Units taken with a sampling device	1 kg
c)	Cereal grains	Rice, wheat	Units taken with a sampling device	1 kg
d)	Tree nuts	Almond, cashew, except coconuts	Units taken with a sampling device	1 kg
e)	Oilseeds	Peanuts	Units taken with a sampling device	5 units 500 g
f)	Seeds for beverages and sweets	Coffee beans	Units taken with a sampling device	500 g
g) Herbs and Spices				
i)	Herbs	Fresh parsley Others	Whole units	0.5 kg 0.2 kg
ii)	Spices (Small size)	Dried pepper, cardamom, cumin	Whole units or taken with a sampling device	0.2 kg
iii)	Spices (Larger size)	Large cardamom, dry ginger, dry turmeric	Whole units or taken with a sampling device	0.5 kg
iv)	Spices (High value)	Saffron	Whole units or taken with a sampling device	10 g
B. Primary Animal Feed Commodities				
a)	Legume animal feeds, and other forages and fodders		Whole units, or units taken with a sampling device	1 kg (at least 10 units)
b)	Straw, hay and other dried products		Units taken with a sampling device	0.5 kg (at least 10 units)
C. Processed Foods**				
a)	Solid products of low bulk density	Hops Tea	Packaged units, or units taken with a sampling device	0.2 kg

Sl. No.	Commodity Classification	Examples	Nature of Primary Sample to be taken	Minimum size of each Laboratory Sample
(1)	(2)	(3)	(4)	(5)
b)	Other solid products	Bread Flour Apple pomace Dried fruit	Packages or other whole units, or units taken with a sampling device	0.5 kg
c)	Liquid products	Vegetable oils Juices	Packaged units, or units taken with a sampling device	0.5 litre 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. **Including products with ingredients of animal origin where the ingredient(s) of plant origin predominate(s).				

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

(*Clause* 4.3 *and* 4.4)

Sl. No.	Commodity Classification	Examples	Nature of Primary Sample to be taken	Minimum size of each Laboratory Sample
(1)	(2)	(3)	(4)	(5)
A. Primary Food Commodities				
a) Poultry eggs				
i)	Eggs, except quail and similar	-	Whole eggs	12 whole chicken eggs, 6 whole goose or duck eggs
ii)	Eggs, quail and similar	-	Whole eggs	24 whole eggs
b)	Milk		Whole unit(s), or unit(s) taken with a sampling device	0.5 litre
B. Processed Foods*				
a)	Liquid milks, milk powders, evaporated milks and creams, creams, dairy ice creams, yoghurts[1)]		Packaged unit(s), or unit(s) taken with a sampling device	0.5 litre or 0.5 kg
b)	Butter and butter oils	Butter, whey butter, low fat spreads containing	Whole or parts of packaged unit(s), or unit(s) taken with a sampling device	0.2 kg or 0.2 litre

		butter fat, anhydrous butteroil, anhydrous milkfat		
c) Cheeses, including processed cheeses[2)]				
i)	units ≥0.3 kg		Whole unit(s) or unit(s) cut with a sampling device	0.5 kg
ii)	units < 0.3 kg		Whole unit(s), or unit(s) cut with a sampling device	0.3 kg
d)	Liquid, frozen or dried egg products		Unit(s) taken aseptically with a sampling device	0.5 kg

***** Including products with ingredients of plant origin where the ingredient(s) of animal origin predominates.

1)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. Milk powders in bulk should be sampled aseptically, passing a dry borer tube through the powder at an even rate. Creams in bulk should be mixed thoroughly with a plunger before sampling but foaming, whipping and churning must be avoided.

2)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.