

**RECOMMENDED METHODS OF SAMPLING FOR THE DETERMINATION OF PESTICIDE  
RESIDUES FOR COMPLIANCE WITH MRLS**

*CAC/GL 33-1999*

**CONTENTS**

	Pages
OBJECTIVE.....	1
PRINCIPLES .....	1
SAMPLING PROCEDURES.....	2
CRITERIA FOR DETERMINING COMPLIANCE .....	3
TABLE 1. MINIMUM NUMBER OF PRIMARY SAMPLES TO BE TAKEN FROM A LOT.....	4
(a) Meat and poultry .....	4
(b) Other products .....	4
TABLE 2. NUMBER OF RANDOMLY SELECTED PRIMARY SAMPLES REQUIRED FOR A GIVEN PROBABILITY OF FINDING AT LEAST ONE NON-COMPLIANT SAMPLE IN A LOT OF MEAT OR POULTRY, FOR A GIVEN INCIDENCE OF NON- COMPLIANT RESIDUES IN THE LOT.....	5
TABLE 3. MEAT AND POULTRY PRODUCTS: DESCRIPTION OF PRIMARY SAMPLES AND MINIMUM SIZE OF LABORATORY SAMPLES .....	6
TABLE 4. PLANT PRODUCTS: DESCRIPTION OF PRIMARY SAMPLES AND MINIMUM SIZE OF LABORATORY SAMPLES .....	8
TABLE 5. EGG AND DAIRY PRODUCTS: DESCRIPTION OF PRIMARY SAMPLES AND MINIMUM SIZE OF LABORATORY SAMPLES .....	10
ANNEX I. DEFINITION OF TERMS .....	11
ANNEX II.A SCHEMATIC REPRESENTATION OF SAMPLING: MEAT AND POULTRY .....	14
ANNEX II.B SCHEMATIC REPRESENTATION OF SAMPLING: PRODUCTS OTHER THAN MEAT AND POULTRY.....	15
ANNEX III. EXAMPLES.....	20
REFERENCES .....	22

**RECOMMENDED METHODS OF SAMPLING FOR THE DETERMINATION OF  
PESTICIDE RESIDUES FOR COMPLIANCE WITH MRLS**

**1. OBJECTIVE**

The objective of these sampling procedures is to enable a representative sample to be obtained from a lot, for analysis to determine compliance with Codex Maximum Residue Limits (MRLs) for pesticides.

**2. PRINCIPLES**

- 2.1 Codex MRLs are based on Good Agricultural Practice data and foods derived from commodities that comply with the respective Codex MRLs are intended to be toxicologically acceptable.
- 2.2 A Codex MRL for a plant, egg or dairy product takes into account the maximum level expected to occur in a composite sample, which has been derived from multiple units of the treated product and which is intended to represent the average residue level in a lot. A Codex MRL for meat and poultry takes into account the maximum level expected to occur in the tissues of individual treated animals or birds.
- 2.3 In consequence, MRLs for meat and poultry apply to a bulk sample derived from a single primary sample, whereas MRLs for plant products, eggs and dairy products apply to a composite bulk sample derived from 1-10 primary samples.

### 3. SAMPLING PROCEDURES

*Notes. (a) The terms used are defined in Annex I and the procedures are shown schematically in Annexes IIA and IIB.*

*(b) ISO recommendations for sampling of grain<sup>1</sup>, or other commodities shipped in bulk may be adopted, if required.*

#### 3.1 Precautions to be taken

Contamination and deterioration of samples must be prevented at all stages, because they may affect the analytical results. Each lot to be checked for compliance must be sampled separately.

#### 3.2 Collection of primary samples

The minimum number of primary samples to be taken from a lot is determined from Table 1, or Table 2 in the case of a suspect lot of meat or poultry. Each primary sample should be taken from a randomly chosen position in the lot, as far as practicable. The primary samples must consist of sufficient material to provide the laboratory sample(s) required from the lot.

*Note. (a) Sampling devices required for grain<sup>1</sup>, pulses<sup>2</sup> and tea<sup>3</sup> are described in ISO recommendations and those required for dairy products<sup>4</sup> are described by the IDF.*

#### 3.3 Preparation of the bulk sample

##### 3.3.1 Procedure for meat and poultry (Table 3)

Each primary sample is considered to be a separate bulk sample.

##### 3.3.2 Procedure for plant products, eggs or dairy products (Tables 4 and 5)

The primary samples should be combined and mixed well, if practicable, to form the bulk sample.

##### 3.3.3 Alternative procedure 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 subdivision 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.

#### 3.4 Preparation of the laboratory sample

Where the bulk sample is larger than is required for a laboratory sample, it should be divided to provide a representative portion. A sampling device, quartering, or other appropriate size reduction process may be used but units of fresh plant products or whole eggs should not be cut or broken. Where required, replicate laboratory samples should be withdrawn at this stage or they may be prepared as in 3.3.3, above. The minimum sizes required for laboratory samples are given in Tables 3, 4 and 5.

#### 3.5 Sampling record

The sampling officer must record the nature and origin of the lot; the owner, supplier or carrier of it; the date and place of sampling; and any other relevant information. Any departure from the recommended method of sampling must be recorded. A signed copy of the record must accompany each replicate laboratory sample and a copy should be retained by the sampling officer. A copy of

the sampling record should be given to the owner of the lot, or a representative of the owner, whether or not they are to be provided with a laboratory sample. If sampling records are produced in computerised form, these should be distributed to the same recipients and a similar verifiable audit trail maintained.

### 3.6 Packaging and transmission of the laboratory sample

The laboratory sample must be placed in a clean, inert container which provides secure protection from contamination, damage and leakage. The container should be sealed, securely labelled and the sampling record must be attached. Where a bar code is utilised, it is recommended that alphanumeric information is also provided. The sample must be delivered to the laboratory as soon as practicable. Spoilage in transit must be avoided, e.g. fresh samples should be kept cool and frozen samples must remain frozen. Samples of meat and poultry should be frozen prior to despatch, unless transported to the laboratory before spoilage can occur.

### 3.7 Preparation of the analytical sample

The laboratory sample should be given a unique identifier which, together with the date of receipt and the sample size, should be added to the sample record. The part of the commodity to be analysed<sup>5,6</sup>, i.e. the analytical sample, should be separated as soon as practicable. Where the residue level must be calculated to include parts which are not analysed<sup>††</sup>, the weights of the separated parts must be recorded.

### 3.8 Preparation and storage of the analytical portion

The analytical sample should be comminuted, if appropriate, and mixed well, to enable representative analytical portions to be withdrawn. The size of the analytical portion should be determined by the analytical method and the efficiency of mixing. The methods for comminution and mixing should be recorded and should not affect the residues present in the analytical sample. Where appropriate, the analytical sample should be processed under special conditions, e.g. at sub-zero temperature, to minimize adverse effects. Where processing could affect residues and where practical alternative procedures are not available, the analytical portion may consist of whole units, or segments removed from whole units. If the analytical portion thus consists of few units or segments, it is unlikely to be representative of the analytical sample and sufficient replicate portions must be analysed, to indicate the uncertainty of the mean value. If analytical portions are to be stored before analysis, the method and length of time of storage should be such that they do not affect the level of residues present. Additional portions must be withdrawn for replicate and confirmatory analyses, as required.

## 4. CRITERIA FOR DETERMINING COMPLIANCE

- 4.1 Analytical results must be derived from one or more laboratory samples taken from the lot and received in a fit state for analysis. The results must be supported by acceptable quality control data (e.g. for instrument calibration and pesticide recovery - refer to the Guidelines on good laboratory practice in pesticide residue analysis (CAC/GL 40-1993). Results should not be corrected for recovery. Where a residue is found to exceed an MRL, its identity should be confirmed and its concentration must be verified by analysis of one or more additional analytical portions derived from the original laboratory sample(s).
- 4.2 The Codex MRL applies to the bulk sample.
- 4.3 The lot complies with a Codex MRL where the MRL is not exceeded by the analytical result(s).

---

<sup>††</sup> For example, the stones of stone fruit are not analyzed but the residue level is calculated assuming that they are included but contain no residue<sup>5</sup>.

- 4.4 Where results for the bulk sample exceed the MRL, a decision that the lot is non-compliant must take into account: (i) the results obtained from one or more laboratory samples, as applicable; and (ii) the accuracy and precision of analysis, as indicated by the supporting quality control data.

**Table 1. Minimum number of primary samples to be taken from a lot**

	Minimum number of primary samples to be taken from the lot
<b>(a) Meat and poultry</b>	
a non-suspect lot	1
a suspect lot	determined according to Table 2
<b>(b) Other products</b>	
(i) Products, packaged or in bulk, which can be assumed to be well mixed or homogeneous	1 see note (d) under definition of a lot, Annex 1
(ii) Products, packaged or in bulk, which may not be well mixed or homogeneous	see note (i), below
<i>either:</i>	
Weight of lot, kg	
<50	3
50-500	5
>500	10
<i>or</i>	
Number of cans, cartons or other containers in the lot	
1-25	1
26-100	5
>100	10

*Note.* (i) For products comprised of large units, in class A only, the minimum number of primary samples should comply with the minimum number of units required for the laboratory sample (see Table 4).

**Table 2. Number of randomly selected primary samples required for a given probability of finding at least one non-compliant sample in a lot of meat or poultry, for a given incidence of non-compliant residues in the lot**

Incidence of non-compliant residues in the lot %	Minimum number of samples ( $n_0$ ) required to detect a non-compliant residue with a probability of:		
	90%	95%	99%
90	1	-	2
80	-	2	3
70	2	3	4
60	3	4	5
50	4	5	7
40	5	6	9
35	6	7	11
30	7	9	13
25	9	11	17
20	11	14	21
15	15	19	29
10	22	29	44
5	45	59	90
1	231	299	459
0.5	460	598	919
0.1	2302	2995	4603

Notes. (a) The Table assumes random sampling.

(b) Where the number of primary samples indicated in Table 2 is more than about 10% of units in the total lot, the number of primary samples taken may be fewer and should be calculated as follows:

$$n = \frac{n_0}{1 + (n_0 - 1) / N}$$

where  $n$  = minimum number of primary samples to be taken

$n_0$  = number of primary samples given in Table 2

$N$  = number of units, capable of yielding a primary sample, in the lot.

(c) Where a single primary sample is taken, the probability of detecting a non-compliance is similar to the incidence of non-compliant residues.

(d) For exact or alternative probabilities, or for a different incidence of non-compliance, the number of samples to be taken may be calculated from:

$$1-p = (1-i)^n$$

where  $p$  is the probability and  $i$  is the incidence of non-compliant residues in the lot (both expressed as fractions, not percentages), and  $n$  is the number of samples.

**Table 3. Meat and poultry: description of primary samples and minimum size of laboratory samples**

Commodity classification	Examples	Nature of primary sample to be taken	Minimum size of each laboratory sample
<b>Class B, primary food commodities of animal origin</b>			
1. <b>Mammalian meats</b> , type 06, group 030 Note: for enforcement of MRLs for fat soluble pesticides samples must be taken according to section 2 below.			
1.1 <b>Large mammals</b> , whole or half carcass, usually 10 kg or more	cattle sheep pigs	whole or part of diaphragm, supplemented by cervical muscle, if necessary	<b>0.5 kg</b>
1.2 <b>Small mammals</b> whole carcass	rabbits	whole carcass or hind quarters	<b>0.5 kg</b> , after removal of skin and bone
1.3 <b>Mammal meat parts, loose fresh/chilled/frozen</b> packaged or otherwise	quarters chops steaks shoulders	whole unit(s), or a portion of a large unit	<b>0.5 kg</b> , after removal of bone
1.4 <b>Mammal meat parts, bulk frozen</b>	quarters chops	<b>either</b> a frozen cross-section of a container <b>or</b> the whole (or portions) of individual meat parts	<b>0.5 kg</b> , after removal of bone
2. <b>Mammalian fats, including carcass fat</b> , type 06, group 031 Note: samples of fat taken as described in 2.1, 2.2 and 2.3 may be used to determine compliance of the fat, or the whole product, with the corresponding MRLs.			
2.1 <b>Large mammals, at slaughter, whole or half carcass</b> Usually 10 kg or more	cattle sheep pigs	kidney, abdominal or subcutaneous fat cut from one animal	<b>0.5 kg</b>
2.2 <b>Small mammals, at slaughter, whole or half carcass</b> <10 kg		abdominal or subcutaneous fat from one or more animals	<b>0.5 kg</b>
2.3 <b>Mammal meat parts</b>	legs chops steaks	<b>either</b> visible fat, trimmed from unit(s)  <b>or</b> whole unit(s) or portions of whole unit(s), where fat is not trimmable	<b>0.5 kg</b>  <b>2 kg</b>
2.4 <b>Mammal bulk fat tissue</b>	-	units taken with a sampling device from at least 3 positions	<b>0.5 kg</b>
<b>Class B, primary food commodities of animal origin</b>			
3. <b>Mammalian offals</b> , type 06, group 032			
3.1 <b>Mammal liver</b> , fresh/chilled/frozen	-	whole liver(s), or part of liver	<b>0.4 kg</b>
3.2 <b>Mammal kidney</b> , fresh/chilled/frozen	-	1 or both kidneys from 1 or more animal	<b>0.2 kg</b>
3.3 <b>Mammal heart</b> , fresh/chilled/frozen	-	Whole heart(s), or ventricle portion only, if large	<b>0.4 kg</b>
3.4 <b>Other mammal offal</b> , fresh/chilled/frozen	intestines brains	Part or whole unit from 1 or more animals, or a cross- section taken from bulk frozen product	<b>0.5 kg</b>

Commodity classification	Examples	Nature of primary sample to be taken	Minimum size of each laboratory sample
4. <b>Poultry meats</b> , type 07, group 036 Note: for enforcement of MRLs for fat soluble pesticides samples must be taken according to section 5 below.			
4.1 <b>Bird, large-sized carcass</b> >2 kg	turkey goose mature chicken	thighs, legs and other dark meat	<b>0.5 kg</b> after removal of skin and bone
4.2 <b>Birds, medium-sized carcass</b> 500 g-2 kg	duckling guinea fowl young chicken	thighs, legs or other dark meat from at least 3 birds	<b>0.5 kg</b> after removal of skin and bone
4.3 <b>Birds, small-sized carcass</b> <500 g carcass	quail pigeon	carcasses from at least 6 birds	<b>0.2 kg</b> of muscle tissue
4.4 <b>Bird parts</b> fresh/chilled/frozen, retail or wholesale packaged	legs quarters	packaged units, or individual parts	<b>0.5 kg</b> (after removal of skin and bone)
<b>Class B, primary food commodities of animal origin</b>			
5. <b>Poultry fats</b> , including carcass fat, type 07, group 037 Note: samples of fat taken as described in 5.1 and 5.2 may be used to determine compliance of the fat, or the whole product, with the corresponding MRLs			
5.1 <b>Birds, at slaughter, whole or part-carcass</b>	chickens turkeys	units of abdominal fat from at least 3 birds	<b>0.5 kg</b>
5.2 <b>Bird meat parts</b>	legs breast muscle	<b>either</b> visible fat, trimmed from unit(s)  <b>or</b> whole unit(s) or portions of whole unit(s), where fat is not trimmable	<b>0.5 kg</b>  <b>2 kg</b>
5.3 <b>Bird fat tissue in bulk</b>	-	units taken with a sampling device from at least 3 positions	<b>0.5 kg</b>
6. <b>Poultry offals</b> , type 07, group 038			
6.1 <b>Edible bird offal, except goose and duck fat liver and similar high value products</b>		units from at least 6 birds, or a cross-section from a container	<b>0.2 kg</b>
6.2 <b>Goose and duck fat liver and similar high value products</b>		unit from 1 birds or container	<b>0.05 kg</b>
<b>Class E, processed foods of animal origin</b>			
7. <b>Secondary food commodities of animal origin</b> , type 16, group 080 dried meats <b>Derived edible products of animal origin</b> , type 17, group 085 processed animal fats <b>Manufactured food (single ingredient) of animal origin</b> , type 18 <b>Manufactured food (multi-ingredient) of animal origin</b> , type 19			
7.1 <b>Mammal or bird, comminuted, cooked canned, dried, rendered, or otherwise processed products</b> , including multi-ingredient products	ham sausage minced beef chicken paste	packaged units, <b>or</b> a representative cross-section from a container, <b>or</b> units (including juices, if any) taken with a sampling device	<b>0.5 kg</b> or <b>2 kg</b> if fat content <5%

Commodities are classified according to the Codex Alimentarius<sup>6</sup>  
Refer to Table 1 to determine the number of primary samples required.

**Table 4. Plant products: description of primary samples and minimum size of laboratory samples**

Commodity classification	Examples	Nature of primary samples to be taken	Minimum size of each laboratory sample
<b>Class A, primary food commodities of plant origin</b>			
1. <b>All fresh fruits</b> , type 1, groups 001-008 <b>All fresh vegetables</b> , type 2, groups 009-019, <b>except</b> group 015 (dry pulses)			
1.1 <b>small sized fresh products</b> units generally < 25 g	berries peas olives	whole units, or packages, or units taken with a sampling device	<b>1 kg</b>
1.2 <b>medium sized fresh products</b> units generally 25-250 g	apples oranges	whole units,	<b>1 kg (at least 10 units)</b>
1.3 <b>large sized fresh products</b> units generally > 250 g	cabbages cucumbers grapes(bunches)	whole units	<b>2 kg (at least 5 units)</b>
2. <b>Pulses</b> , type 2, group 015 <b>Cereal grains</b> , type 3, group 020 <b>Tree nuts</b> , type 4, group 022  <b>Oilseeds</b> , type 4, group 023  <b>Seeds for beverages and sweets</b> , type 4, group 024	soya beans rice, wheat except coconuts coconuts  peanuts		<b>1 kg 1 kg 1 kg  5 units 500 g  500 g</b>
3. <b>Herbs</b> , type 5, group 027 <i>(for dried herbs see: Class D, type 12, in section 5 of this Table)</i>  <b>Spices</b> , type 5, group 028	fresh parsley others, fresh  dried	whole units  whole units or taken with a sampling device	<b>0.5 kg 0.2 kg  0.1 kg</b>
<b>Class C, primary animal feed commodities</b>			
4. <b>Primary feed commodities of plant origin</b> , type 11			
4.1 <b>Legume animal feeds, and other forages and fodders</b>		whole units, or units taken with a sampling device	<b>1 kg (at least 10 units)</b>
4.2 <b>Straw, hay and other dried products</b>		units taken with a sampling device	<b>0.5 kg (at least 10 units)</b>
<b>Class D, processed foods of plant origin</b>			
5. <b>Secondary food commodities of plant origin</b> , type 12, dried fruits, vegetables, herbs, milled cereal products <b>Derived products of plant origin</b> , type 13, teas, vegetable oils, juices, by-products for animal feed and miscellaneous products <b>Manufactured foods (single ingredient) of plant origin</b> , type 14 <b>Manufactured foods (multi-ingredient) of plant origin</b> , type 15, including products with ingredients of animal origin where the ingredient(s) of plant origin predominate(s), and group 078, breads			
5.1 <b>Products of high unit value</b>		packages or units taken with a sampling device	<b>0.1 kg*</b>
5.2 <b>Solid products of low bulk density</b>	hops tea	packaged units, or units taken with a sampling device	<b>0.2 kg</b>

Commodities are classified according to the Codex Alimentarius<sup>6</sup>  
Refer to Table 1 to determine the number of primary samples required.



Commodity classification	Examples	Nature of primary samples to be taken	Minimum size of each laboratory sample
5.3 <b>Other solid products</b>	bread flour apple pomace dried fruit	packages or other whole units, or units taken with a sampling device	<b>0.5 kg</b>
5.4 <b>Liquid products</b>	vegetable oils juices	packaged units, or units taken with a sampling device	<b>0.5 l or 0.5 kg</b>

\* 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 5. Egg and dairy products: description of primary samples and minimum size of laboratory samples**

Commodity classification	Examples	Nature of primary samples to be taken	Minimum size of each laboratory sample
<b>Class B, primary food commodities of animal origin</b>			
1. Poultry eggs, type 7, group 039			
1.1 Eggs, except quail and similar		whole eggs	<b>12 whole chicken eggs, 6 whole goose or duck eggs</b>
1.2 Eggs, quail and similar		whole eggs	<b>24 whole eggs</b>
2. Milks, type 6, group 033		whole unit(s), or unit(s) taken with a sampling device	<b>0.5 l</b>
<b>Class E, processed foods of animal origin</b>			
3. Secondary food commodities of animal origin, type 16, group 082 skimmed milks, evaporated milks and milk powders Derived edible products of animal origin, type 17, group 086 milkfats, group 087 butters, butteroils, creams, cream powders, caseins, etc. Manufactured food (single ingredient) of animal origin, type 18, group 090 Manufactured food (multi-ingredient) of animal origin, type 19, group 092 (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	<b>0.5 l (liquid) or 0.5 kg (solid)</b>
<i>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.</i>			
<i>(ii) Milk powders in bulk should be sampled aseptically, passing a dry borer tube through the powder at an even rate.</i>			
<i>(iii) Creams in bulk should be mixed thoroughly with a plunger before sampling but foaming, whipping and churning must be avoided.</i>			
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	<b>0.2 kg or 0.2 l</b>
3.3 Cheeses, including processed cheeses		Whole unit(s) or unit(s) cut with a sampling device	<b>0.5 kg</b>
units 0.3 kg or greater			
units < 0.3 kg		whole unit(s), or unit(s) cut with a sampling device	<b>0.3 kg</b>
<i>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.</i>			
3.4 Liquid, frozen or dried egg products		unit(s) taken aseptically with a sampling device	<b>0.5 kg</b>

Commodities are classified according to the Codex Alimentarius<sup>6</sup>  
Refer to Table 1 to determine the number of primary samples required.

## ANNEX I. DEFINITION OF TERMS

### **Analytical portion**

A representative quantity of material removed from the analytical sample, of proper size for measurement of the residue concentration.

*Note.* A sampling device may be used to withdraw the analytical portion.

### **Analytical sample**

The material prepared for analysis from the laboratory sample, by separation of the portion of the product to be analysed<sup>5,6</sup> and then by mixing, grinding, fine chopping, etc., for the removal of analytical portions with minimal sampling error.

*Note.* Preparation of the analytical sample must reflect the procedure used in setting Codex MRLs and thus the portion of the product to be analysed may include parts that are not normally consumed.

### **Bulk sample**

For products other than meat and poultry, the combined and well mixed aggregate of the primary samples taken from a lot. For meat and poultry, the primary sample is considered to be equivalent to the bulk sample.

*Notes.* (a) The primary samples must contribute sufficient material to enable all laboratory samples to be withdrawn from the bulk sample.

(b) Where separate laboratory samples are prepared during collection of the primary sample(s), the bulk sample is the conceptual sum of the laboratory samples, at the time of taking the samples from the lot.

### **Laboratory sample**

The sample sent to, or received by, the laboratory. A representative quantity of material removed from the bulk sample.

*Notes.* (a) The laboratory sample may be the whole or a part of the bulk sample.

(b) Units should not be cut or broken to produce the laboratory sample(s), except where subdivision of units is specified in Table 3.

(c) Replicate laboratory samples may be prepared.

### **Lot**

A quantity of a food material delivered at one time and known, or presumed, by the sampling officer to have uniform characteristics such as origin, producer, variety, packer, type of packing, markings, consignor, etc. A suspect lot is one which, for any reason, is suspected to contain an excessive residue. A non-suspect lot is one for which there is no reason to suspect that it may contain an excessive residue.

*Notes.* (a) Where a consignment is comprised of lots which can be identified as originating from different growers, etc., each lot should be considered separately.

(b) A consignment may consist of one or more lots.

(c) Where the size or boundary of each lot in a large consignment is not readily established, each one of a series of wagons, lorries, ship's bays, etc., may be considered to be a separate lot.

(d) A lot may be mixed by grading or manufacturing processes, for example.

### **Primary sample**

One or more units taken from one position in a lot.

*Notes.* (a) The position from which a primary sample is taken in the lot should preferably be chosen randomly but, where this is physically impractical, it should be from a random position in the accessible parts of the lot.

(b) The number of units required for a primary sample should be determined by the minimum size and number of laboratory samples required.

- (c) For plant, egg and dairy products, where more than one primary sample is taken from a lot, each should contribute an approximately similar proportion to the bulk sample.
- (d) Units may be allocated randomly to replicate laboratory samples at the time of collecting the primary sample(s), in cases where the units are of medium or large size and mixing the bulk sample would not make the laboratory sample(s) more representative, or where the units (e.g. eggs, soft fruit) could be damaged by mixing.
- (e) Where primary samples are taken at intervals during loading or unloading of a lot, the sampling “position” is a point in time.
- (f) Units should not be cut or broken to produce the primary sample(s), except where sub-division of units is specified in Table 3.

### **Sample**

One or more units selected from a population of units, or a portion of material selected from a larger quantity of material. For the purposes of these recommendations, a representative sample is intended to be representative of the lot, the bulk sample, the animal, etc., in respect of its pesticide residue content and not necessarily in respect of other attributes.

### **Sampling**

The procedure used to draw and constitute a sample.

### **Sampling device**

(i) A tool such as a scoop, dipper, borer, knife or spear, used to remove a unit from bulk material, from packages (such as drums, large cheeses) or from units of meat or poultry which are too large to be taken as primary samples. (ii) A tool such as a riffle box, used to prepare a laboratory sample from a bulk sample, or to prepare an analytical portion from an analytical sample.

*Notes.* (a) Specific sampling devices are described by ISO<sup>1,2,3</sup> and IDF<sup>4</sup> standards.

- (b) For materials such as loose straw or leaves, the hand of the sampling officer may be considered to be a sampling device.

### **Sampling officer**

A person trained in sampling procedures and, where required, authorised by the appropriate authorities to take samples.

*Note.* The sampling officer is responsible for all procedures leading to and including preparation, packing and shipping of the laboratory sample(s). The officer must understand that consistent adherence to the specified sampling procedures is necessary, must provide complete documentation for samples, and should collaborate closely with the laboratory.

### **Sample size**

The number of units, or quantity of material, constituting the sample.

### **Unit**

The smallest discrete portion in a lot, which should be withdrawn to form the whole or part of a primary sample.

*Note.* Units should be identified as follows.

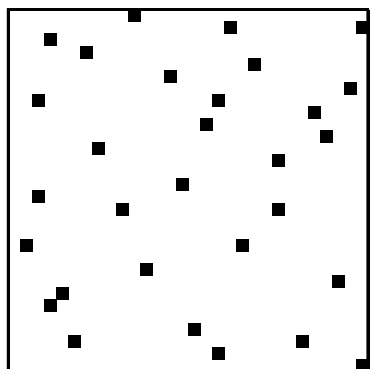
- (a) **Fresh fruit and vegetables.** Each whole fruit, vegetable or natural bunch of them (e.g. grapes) should form a unit, except where these are small. Units of packaged small products may be identified as in (d), below. Where a sampling device may be used without damaging the material, units may be created by this means. Individual eggs, fresh fruit or vegetables must not be cut or broken to produce units.
- (b) **Large animals or parts or organs of them.** A portion, or the whole, of a specified part or organ should form a unit. Parts or organs may be cut to form units.

- (c) **Small animals or parts or organs of them.** Each whole animal or complete animal part or organ present may form a unit. Where packaged, units may be identified as in (d), below. Where a sampling device may be used without affecting residues, units may be created by this means.
- (d) **Packaged materials.** The smallest discrete packages should be taken as units. Where the smallest packages are very large, they should be sampled as bulk, as in (e), below. Where the smallest packages are very small, a pack of packages may form the unit.
- (e) **Bulk materials and large packages** (such as drums, cheeses, etc.) which are individually too large to be taken as primary samples. The units are created with a sampling device.

**ANNEX II.A SCHEMATIC REPRESENTATION OF SAMPLING: MEAT AND POULTRY**

**Lot and primary samples of suspect meat or poultry:**

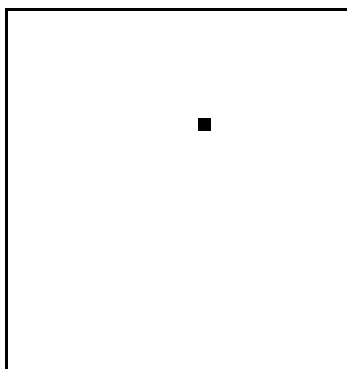
primary samples taken from a number of randomly chosen positions (see Tables 1, 2 and 3)



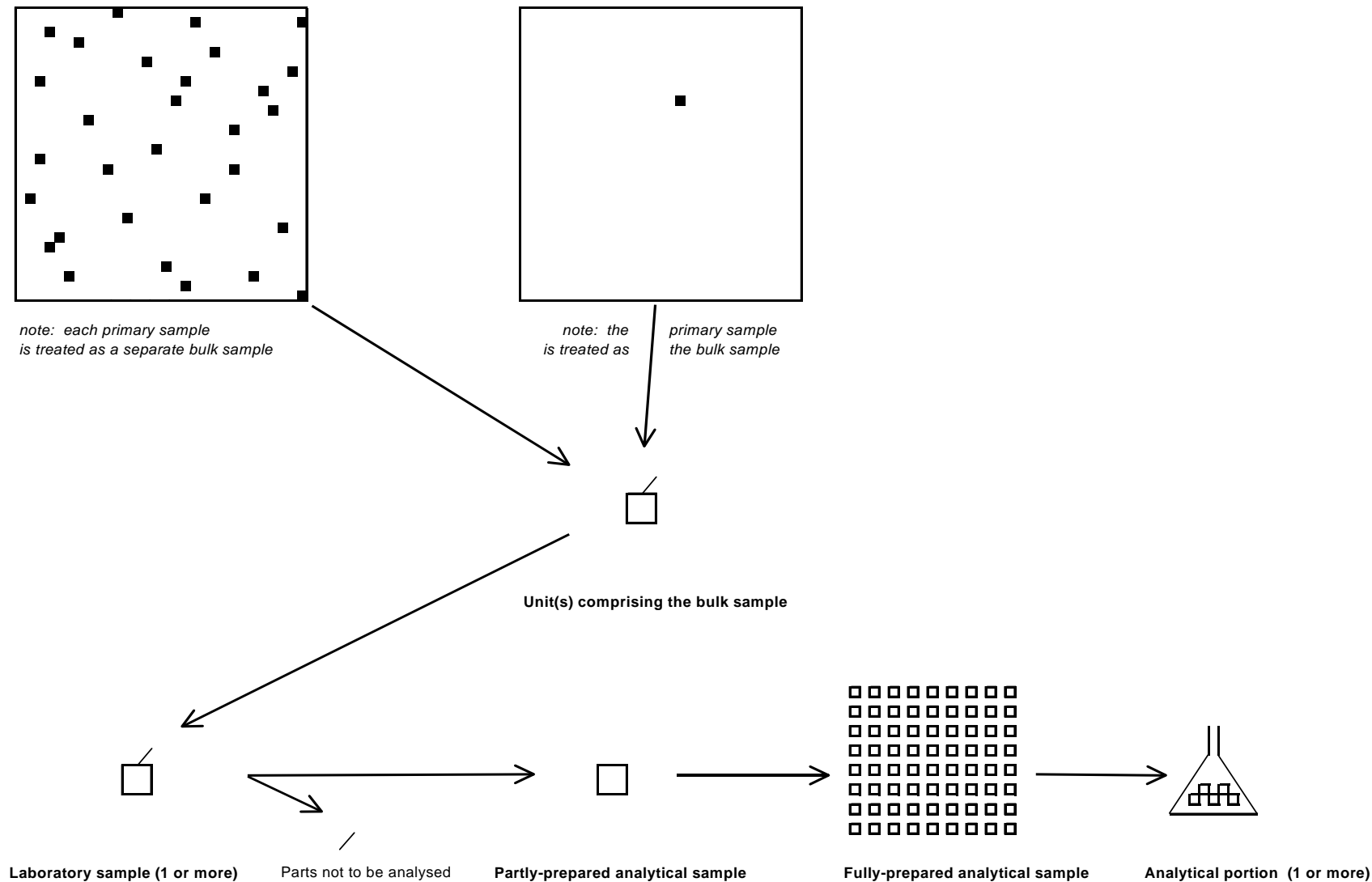
*note: each primary sample is treated as a separate bulk sample*

**Lot and primary samples of non-suspect meat or poultry**

1 primary sample taken from a randomly chosen position (see Tables 1 and 3)

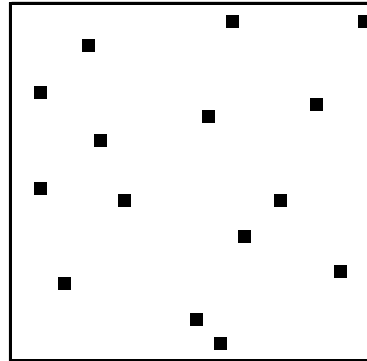


*note: the is treated as primary sample the bulk sample*

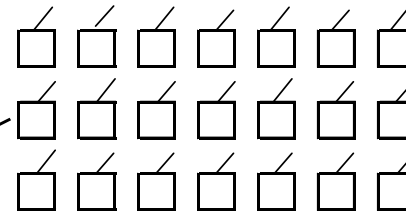


**ANNEX II.B SCHEMATIC REPRESENTATION OF SAMPLING: PRODUCTS OTHER THAN MEAT AND POULTRY**

**Lot and primary samples of any other product**  
 1, 3, 5, 10 or 15 primary samples taken from  
 an equal number of randomly chosen positions  
 (see Tables 1, 4 and 5)

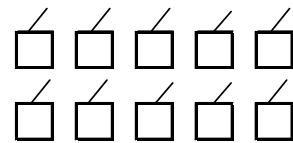


*note: primary samples are combined  
 to form the bulk sample*

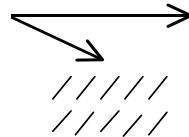


**Units comprising the bulk sample**

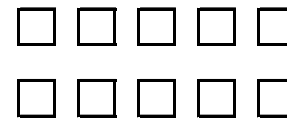
*note: where laboratory samples are prepared directly from the lot,  
 the bulk sample is the conceptual sum of the laboratory samples*



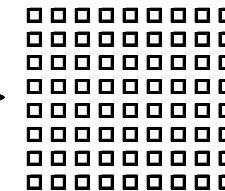
**Laboratory sample (1 or more)**



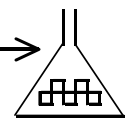
**Parts not to be analysed**



**Partly-prepared analytical sample**



**Fully-prepared analytical sample**



**Analytical portion (1 or more)**

**ANNEX III. EXAMPLES**

Notes. (i) These examples are provided only as illustrations, they do not form part of the recommendations.

(ii) Decisions as to whether or not an MRL is exceeded should be based on the analytical data available but decisions on the consequent action are a matter for the authorities involved.

**Example A.**

The assumed facts:

1. A 500 t consignment of imported frozen animal carcasses, 300 t labelled as producer A and 200 t labelled as producer B, is to be checked for residues.
2. The carcasses are from an exporter whose products have recently been associated with excessive residues of permethrin (fat-soluble) and diflubenzuron (non-fat-soluble).
3. Carcasses in lot A have trimmable fat, whereas those in lot B do not.
4. The sampling plan is to provide a 95% probability of detection if 10% of the carcasses contain excessive residues.
5. There is no legal requirement to prepare replicate laboratory samples.
6. Sampling records are in hard copy form.
7. Rendering of fat tissue for extraction of lipid is acceptable under national law.

Consequent actions and decisions:

1. The consignment is sampled as 2 separate, suspect lots, A and B.
2. Table 2 shows that 29 laboratory samples should be taken and therefore, as far as practicable, 29 carcasses are selected at random from each lot.
3. From each selected carcass in lot A, a minimum of 0.5 kg of adhering fat tissue is taken as a (primary) laboratory sample and a minimum of 0.5 kg of meat (meat does not include bone) is taken as a separate (primary) laboratory sample.
4. The carcasses in lot B have no trimmable fat and 29 samples of 2 kg meat are taken.
5. As each laboratory sample is taken, it is placed in a new polythene bag, securely labelled and sealed, and the sample record completed. The samples are sent to the laboratory, ensuring that they do not thaw. Copies of the sample records are given to the owner/custodian of the consignment. Copies are sent with the samples and also retained by the sampling officer.
6. Fat tissue laboratory samples from lot A are rendered, the lipid collected and aliquots (analytical portions) analyzed for permethrin residues. The results are expressed on a whole fat tissue basis.
7. Bones, if any, are removed from the meat laboratory samples, which are minced before the determination of diflubenzuron residues in analytical portions. The results are expressed on the basis of whole meat without bone.
8. If meat samples from both lots contain diflubenzuron  $\leq 0.05$  mg/kg and all samples from lot A contain  $< 1$  mg/kg permethrin, lot B is acceptable and lot A is acceptable with respect to diflubenzuron residues.
9. If 3 of the 29 fat samples of lot A contain permethrin  $> 1$  mg/kg, replicate analytical portions of fat from these 3 laboratory samples are analyzed. Taking into account the analytical uncertainty, if the results confirm that the MRL is exceeded, the 3 carcasses do not comply with the MRL, whereas the other 26 do comply with the MRL.
10. If the entire lot is not to be rejected on this basis, laboratory samples of fat tissue from the remaining carcasses in lot A may be taken for analysis, in order to separate the acceptable carcasses from those that are unacceptable.



**Example B.**

The assumed facts:

1. A consignment of 60 t of apples in 12 kg cartons (each containing approximately 100 apples) is to be checked for residues.
2. All cartons have the same grower code and date marks.
3. Triplicate laboratory samples are required by national law.
4. The sampling officer is unsure of the degree of mixing that has occurred during packing and grading.
5. Sampling records are in hard copy form.
6. A replicate laboratory sample is held by the monitoring laboratory, until required for analysis by the referee laboratory.

Consequent actions and decisions:

1. The consignment is sampled as a single lot.
2. As far as practicable, 10 cartons are selected at random and 3 new polythene bags provided for the laboratory samples.
3. From each carton, apples are taken and placed in each of the bags (1-2 in each), ensuring that in each bag there is a minimum of 10 apples, weighing a total of  $\geq 1$  kg. The bags are then securely labelled and sealed, and the sample records completed and attached.
4. Two of the laboratory samples are sent to the monitoring laboratory and the third laboratory sample is given to the owner/custodian of the lot.
5. At the monitoring laboratory, the first laboratory sample is prepared and processed and an analytical portion analyzed. The second laboratory sample is retained without further processing.
6. If the results show the confirmed presence of iprodione in excess of the MRL of 10 mg/kg, one or more replicate analytical portion are analyzed.
7. If the results indicate that the MRL is exceeded, the authorities notify the owner/custodian of the consignment (who may arrange independent analysis of the laboratory sample provided) and send the remaining sealed laboratory sample to a reference laboratory.
8. Taking into account the analytical uncertainty at both laboratories, if the results from the reference laboratory indicate residues of iprodione  $\geq 10$  mg/kg, the MRL is considered to be exceeded.

**REFERENCES**

1. **International Organisation for Standardization**, 1979. International Standard ISO 950: Cereals – Sampling (as grain).
2. **International Organisation for Standardization**, 1979. International Standard ISO 951: Pulses in bags – Sampling.
3. **International Organisation for Standardization**, 1980. International Standard ISO 1839: Sampling – Tea.
4. **International Dairy Federation**, 1995. International IDF Standard 50C: Milk and milk products – methods of sampling.
5. **Joint FAO/WHO Food Standards Programme**. Guidelines on portion of commodities to which Codex maximum residue limits apply and which is analyzed (CAC/GL 41-1993).
6. **Joint FAO/WHO Food Standards Programme**. Codex classification of foods and animal feeds (CAC/MISC 4-1993).