TOPIC 12 – OFFICIAL CONTROLS

Subject N° 18: Sampling methodology for specific official controls

Content

This handbook is intended to set general rules about sampling techniques (constitution, execution) on the control site, to check the compliance of all types of goods with a specification, whether or not regulatory. It does not include decision rules on the lot of controlled goods.

Sampling covers all sampling operations on all types of goods. It ceases at the first operation of a physical and chemical analysis on a homogeneous portion of the sample.

Annex 1 summarises all operations for sampling goods.

1. Specific vocabulary

1.1. Lot

It is a defined quantity of goods determined, manufactured or produced in conditions that are assumed to be uniform.

The term “lot” means a lot destined to be controlled by sampling, i.e. a quantity of material or a set of items taken as a sample.

In the meaning of this handbook for sampling controls, the lot defined above must also be sold or intended for sale as it is.

1.2. Homogeneous lot

It is a lot for which no identifiable cause for variation in components and raw materials occurred during production, preparation, manufacture, transport or storage.

In terms of statistics, a lot is considered to be homogeneous in relation to a given property if the distribution of values noted for characteristic parameters of this property is approximately normal.

Where the distribution is not normal or the value of its standard deviation is high, the lot must be considered as heterogeneous.

Items in a lot can be both considered as homogeneous for a given property and heterogeneous for another.

1.3. Item

It relates to:

- either an object, a unit or a defined quantity of material where a set of observations can be made;
- or the result of previous observations, be they qualitative (the increment is qualified as compliant or non-compliant) or quantitative (measurement result).

1.4. Increment

If a lot comprises individualised units, it is an item in the lot taken to form a sample.

If the lot is bulk goods, it is a quantity of material taken once, at a point in the lot, to form a sample; this quantity is then individualised permanently in a receptacle (box, spoon, etc.) for observations or measurements.

1.5. Characteristic

Property used to distinguish the individuals of a population. A characteristic can be qualitative (attribute) or quantitative.

A quantitative characteristic is said to be continuous if, in its field of variation, it can be assimilated with a continuous variable in the sense of mathematical analysis: for example, the protein content of a food.

A quantitative characteristic which can only take integer values is called discrete: for example, the number of pre-packs in a box.

1.6. Sampling

Sampling relates to a procedure for sampling goods to form a sample.

1.7. Sample

It is a set of one or more increments taken in a lot of goods intended to provide information on these goods or the process that produced them. This information can potentially be used as a basis for a decision.

1.8. Sample size

This is the number of items or increments making up the sample.

1.9. Sampling plan

It is a plan whereby one or more samples are taken to provide information to be compiled and a decision to be made where necessary.

It sets the rules stipulating the sampling modalities and, if appropriate, these rules can be supplemented by fixing acceptance or rejection conditions of the controlled lot.

Every "Quality-Safety" sampling plan must state the following instructions and include a decision rule for the controlled lot:

- the nature or objective of the control;

- the type of sampling used (see nomenclature of samplings in 1.12);
- the type of each sample taken and prepared (see nomenclature of samples in 1.13);
- the number of increments of each sample taken.

The principal stages in the sampling procedure are mainly:

- the increment modalities;
- the modalities for preparing samples based on these increments to create the sample intended for the physical and chemical analyses;
- the modalities for identifying each lot and each sample taken;
- the acceptance or rejection conditions for the controlled lot.

1.10. Non-compliant or defective increment and non-compliant or defective item

It is a defective increment or item with one or more defects defined in the sampling plan, based on the regulations or standards or professional usage.

1.11. Defect

It is the non-compliance of an item or increment to the stipulations imposed for a characteristic by the sampling plan.

1.12. Sampling types

If appropriate, the samplings listed below can be used to further characterise the goods to be controlled.

1.12.1. Random sampling

It is a sample made up of items in a lot that all have the same chance of being part of the sample.

Where the lot is not made up of individualised parts, it is divided mentally into virtual items.

All the items in a lot (real or virtual) have two distinct numbers:

- the number of items in a sample is determined or designated randomly, using random numbers generated by a table or a calculator or statistical software;
- all possible combinations of n items in a lot of N items have therefore the same probability of being taken.

1.12.2. Stratified sampling

The strata are the different parts into which the population to be sampled is divided.

Stratified sampling involves taking a sample of items or increments from each stratum; all these samples make up the sample intended for the physical and chemical analysis.

1.12.3. Multi-stage sampling

For the sake of convenience, the items making up the goods to be controlled can be grouped into primary units (sometimes called clusters) so that every item belongs to a primary unit and only one.

The items are then secondary units. Two-stage sampling involves taking a sample of primary units at the first stage, then at the second stage, a sample of secondary units from each primary unit taken at the first stage.

The primary sample is the sample taken in a lot at the first stage of multi-stage sampling.

Consequently, the sample taken in the primary sample is called the secondary sample.

Samples with more than two stages can therefore be considered under a similar mechanism, with the final sample corresponding to the final sampling stage.

Example of three-stage sampling in a lot of pre-packs of cigarettes controlled on the manufacturing line:

- primary unit: a box;
- secondary unit: a pack;
- third unit: a cigarette.

1.13. Sample types

The samples defined below will be executed according to the instructions in the sampling plan.

1.13.1. Representative sample of the controlled lot

Sample that reflects the best the controlled goods; the sample taken at random is a representative sample.

1.13.2. Global sample

This is the quantity of goods formed by uniting all the increments.

1.13.3. Reduced sample

This is the quantity of goods coming from the reduction of the global sample or a partial sample in conditions guaranteeing the representativeness of samples; this sample reflects as closely as possible the sample intended for the physical and chemical analysis and the amount of matter it contains cannot be less than the amount required for the physical and chemical analysis.

1.13.4. Laboratory sample

This is a small quantity of goods representative of the reduced sample and intended for the physical and chemical analysis in a laboratory.

1.13.5. Reference sample

This is a sample prepared at the same time as the laboratory sample which is stored to serve as a laboratory sample, mainly in the event of a counter-expertise.

1.14. Preparing a sample

For bulk goods, this involves all physical operations such as mixing, dividing, etc., required to convert the global sample into the state of laboratory sample.

Preparing a sample for the laboratory must ensure the representativeness of the sampled lot.

2. Type of controls

These are "Quality-Safety" controls.

The aim of these controls is to ensure the quality and safety of the products and of certain services and to make sure that the claims or advertising to promote the products (labels, designations of origin, organic production, etc.) relate to reality.

It is important to inform the consumers fairly and therefore contribute to inspiring confidence in the purchase.

The quality that consumers are entitled to expect from a product or service must be ensured (rules for labelling, composition and naming of goods, control of falsification and deceit).

The aim is also to protect the safety and health of consumers by intervening in all food and industrial products and at all levels (production, import, distribution) and in the services, in all business sectors.

The increments made according to the sampling plan are used to corroborate the level 1 visual inspection by the verifying officer.

3. Means and methods

3.1. General rules

These rules aim to obtain and prepare a sample to determine one or more characteristics of the controlled product.

The sampling must be the most representative of the controlled goods.

3.1.1. Equipment used

See investigator guide in terms of quality approach sampling (annex 1).

3.1.2. Preliminary thoughts on the sampling operation

For the opportunity to sample: see investigator guide (annex 1).

Preliminary thought should be given to the following questions before starting the sampling operation:

- aim of the operation?
- gathering information on the quality and/or safety of the goods?
- noting an infringement?
- type of constraints imposed by the operation?
- suitability of the equipment?
- state of the goods (solid, liquid, gaseous), degree of homogeneity of the goods, dimension of the goods?
- storage conditions?
- nature of the characteristic(s) to be controlled?

3.1.3. Instructions of a sampling plan

Sample taking must follow the instructions in a sampling plan chosen in advance.

1. Which sampling plan should be used?

The plan is fixed:

- either by the data sheet;
- or by a standard relating to the controlled product or a sampling plan;
- or by special instructions from the laboratory which will then be included in a product sheet.

2. Standard structure of a sampling plan

Every plan to be used must include all the headings listed and expanded below. Failing that, the professionals controlled may dispute its validity.

- **The control nature or objective**
  - Based on statistical principles;

- Or failing statistical principles, established arbitrarily based on experience or opportunity, by taking account of all available information on the goods at the time of sampling.

➢ **The type of sample taken and prepared**

See nomenclature of samples in 1.12.

➢ **Size of each sample taken as increment**

See the product's data sheet.

➢ **Modalities for identifying each sample taken**

See investigator guide (annex 1).

➢ **Type of sampling used**

See nomenclature of samplings in chapter 1.

➢ **Main stages in the sampling procedure**

Quantity of goods to be sampled
See the relevant product data sheet or liaise, if necessary, with an official laboratory.

Increment modalities
See below in 3.1.4, n° 4.

Modalities for preparing samples from these increments
See specific sampling provisions in the product sheet; liaise, if necessary, with an official laboratory.

➢ **Acceptance or rejection conditions for the controlled lot**

Liaise with an official laboratory.

3.1.4. Sampling procedure

The following sampling procedure must be followed when taking samples.

1. Identifying the lot before any samples are taken

Note:

- the size of the lot (volume, weight, number of items included in it, etc.)
- the inscriptions on the labels or commercial documents.

2. Examining the lot before any samples are taken

Note:

- the characteristics of its environment, mainly the temperature;
- the state of the goods (damaged parts, heterogeneous parts, etc.).

3. Heterogeneous lots

It is essential to have information on all the different heterogeneous parts in a heterogeneous lot; these are processed as separate lots and subjects for increments:

- on the damaged parts;
- on the fractions of the lot obtained by dividing them virtually (mentally) into fractions with more similar properties.

Nevertheless, these operations are not mandatory if information is available on all the heterogeneous parts of the lot; this information is sent to the analysis laboratory.

4. Taking increments

➢ Precautions to be taken

Protecting sampling operations (controlled products and control equipment) from any contamination such as rain, heat, dust or modification to receptacles in which the increments are placed.

➢ Choosing increments at random

Whenever possible, to avoid any challenge to the representativeness of the sample increment, increments should be chosen at random without fail. Proceed as follows:

- if the lot is not formed of individualised parts, for example milk in a tank, wheat stored in a hopper or wine stored in a barrel, the lot is divided mentally into virtual items;
- all the items in a lot (real or virtual) have two distinct numbers;
- all the parts of the controlled lot, whether or not individualised virtually, must have the same chance of being taken; this requirement is ensured by using random sampling. This involves making up a sample with items in a lot (real or virtual) that all have the same chance of being part of the sample.
- the number of items (real or virtual) taken and included in a sample is determined by chance, using random numbers generated by a table or a calculator or statistical software.

When it is impossible to take items at random (for example, in a huge warehouse where the products are badly stored), the following precautions must be taken:

- avoid choosing the most accessible items systematically;
- do not systematically choose items that stand out with a clear characteristic;
- where there is a periodic phenomenon that can give highly biased results and which can potentially distort the validity and representativeness of the results, for example an out-of-adjustment dosing machine so that every x seconds the product conditioned by this machine shows defects, or a food contaminant that is distributed selectively at the bottom of a hopper, avoid taking every k second or every k^{th} packet or every k^{th} centimetre or taking a unit every "n" pallet, carton or pre-pack, etc.

A periodic phenomenon is assessed, where appropriate, by:

- examining manufacturing control charts for the goods;
- information to be compiled on the product storage from a laboratory, control networks or professional organisations.

Avoid choosing items resembling each other, for example those corresponding to a short manufacturing time or which are contained in the same pallet or carton.

➢ **Goods in the form of individualised objects**

For example, pre-packs or manufactured objects.

Case of instruments or manufactured objects and pre-packs where the content is less than the minimum quantity to be sampled for physical and chemical analyses

It is preferable to keep the goods in their original packaging when controlling the average value of a characteristic, in order to maintain the initial state:

- take sufficient numbers of pre-packs, instruments or manufactured objects to reach the quantity set by the instructions from the official laboratory to constitute an increment;
- the sample intended for the physical and chemical analysis is then made up of all increments set by the sampling plan.

Case of instruments or manufactured goods and pre-packs where the content is more than the minimum quantity of goods to be sampled for physical and chemical analyses

Where necessary, it is preferable to keep the goods in their original packaging in order to maintain their initial state. Unless specified otherwise in the sampling plan, the sample size is made up of a pre-pack of instruments or manufactured goods.

Given the inaccuracy of investigations based on a single sample, the sample size and analytical determinations must be increased to obtain a significant result.

➢ **Bulk goods**

Quantity of goods to be sampled

- where the goods are covered by a data sheet, take the amount fixed by the sheet;
- where the goods are not covered by a data sheet, liaise with an official laboratory.

Operating procedure

It is recommended to carry out the sampling when loading or unloading the goods from the means of transport, or during the storing process, when the goods are being moved.

Goods are taken randomly. They are no longer goods that participate in a draw, but virtual units (quantity of goods unloaded in x seconds, content of a receptacle, etc.).

For example, if unloading takes four hours, samples are taken at the 10^{th}, 21^{st} and 37^{th} minute; the range of minutes chosen was determined by random prior to the sampling operation.

A firm operating technique is required in a lot of moving goods; this means cutting through the entire thickness of the stream so that the global sample corresponds to a full section of the stream, which can be heterogeneous given the different densities and particle

dimensions, or possess heterogeneity factors generated by the goods' manufacturing, storage or circulation techniques.

This type of operation can only be envisaged if automatic sampling tools are available.

General case

Wherever possible, to offset any effects of a selective distribution of components through the density, the samples should be taken in the entire depth of the bulk goods.

Where no information on the distribution of components or contaminants in the bulk goods is available, the increments should be multiplied in the bulk space.

The sampling probe must be plunged in several directions using a turning movement, except for liquids and moving goods.

Powdered goods

The sampling equipment must have a large enough opening to be able to take the largest particles in the goods.

Failing suitable automatic sampling equipment, probes with a large enough opening should be used. Wherever possible, they penetrate the goods in several directions at a regular speed until they reach the bottom.

Liquid or semi-liquid products stored in drums or barrels

The product must be mixed carefully, for example with a stirring rod long enough to reach the bottom and stir all the product, and light enough to be handled quickly.

Avoid creating a foam. The walls and bottom should be scraped if necessary.

A dipping receptacle of a size relating to the minimum quantity is then plunged into the liquid.

The constant plunging speed must be estimated so that the receptacle fills up as it descends and is completely full when it reaches the bottom.

Liquid products stored in large receptacles and tanks

Where it is impossible to stir the contents mechanically to mix them up, samples are taken at several points.

Pastes and semi-solid products, depending on the consistency, form and weight of the goods to be sampled

One of the following techniques is to be used, depending on the consistency of the goods:

- hard consistency: sampling using a knife or saw; making several cuts which direction depends on the nature of the product;
- other cases: sampling using a probe as indicated in p. 12 ("General case").

Gases

Consult an official laboratory before sampling.

5. Constituting samples from increments

Follow the instructions in the sampling plan (see 1.9).

6. Sampling report

All samplings must have a written sampling report according to defined modalities.

3.2. Rules specific to the products

3.2.1. Existence of data sheets

See the sheets established by each of the authorising offices.

3.2.2. Lack of data sheets

If appropriate, follow the general rules of this handbook and liaise with an official laboratory for any specific additional sampling modalities.

4. Minimum quantities to be taken for the sample intended for the laboratory, and special packaging and transport conditions

4.1. Purpose

The aim of the data sheet, included in this handbook, is to define the minimum quantities to be taken for the samples intended for the laboratories.

It does not deal with the sampling modalities (number of units per sample, sampling modalities) intended to ensure the representativeness of the sample in the lot (see regulations or product data sheets when they exist).

4.2. Scope

This handbook covers the samplings taken under the "Quality and Safety" controls, whether they be intended for the laboratory or set aside for an expertise, if necessary. It states the weight (or volume) of the sample to be sent to the laboratory so that it can make all routine determinations in a given product.

4.3. Specific vocabulary

4.3.1. Sampling, sample, unit

A sampling can be made up of one or more samples (example: three samples for the PO3, one sample for the PE1); each sample can be made up of one or more units (example: one PO3 of ham, made up of three samples each comprising eleven units).

4.3.2. Sales unit

Sales units are normally lots of two to six (or more) product units (e.g. a six-pack of yoghourt pots).

4.3.3. Minimum quantity

Amount of product used by the laboratory to carry out all routine determinations.

NB: the minimum quantity must be respected for each unit in the sample.

4.4. Quantities to be taken

The quantities to be taken, shown in the headings of this handbook, are per sample (and per unit). They have been assessed as the minimum required by the laboratory to carry out the routine determinations on the products in question.

For the products not listed in the headings, the quantity will be assessed by comparison, or even better, after having obtained the information from the concerned laboratory. This is

particularly true, wherever possible, for cosmetics, maintenance products and industrial chemical substances, where counting has deliberately been limited and which require multiple determinations, given their complex composition. The same applies to industrial equipment and products.

In case of doubt, especially when assessing the contamination rate of pollutants, contact must be made with the competent laboratory to determine the exact quantity to be taken, or a lesser quantity than planned when the analysis is focused on determining a reduced number of criteria.

For goods conditioned in small units (butter, cheese and other dairy products, biscuits, sugar, detergents, etc.), the sampling must gather enough unit packs to obtain the quantity indicated.

4.5. Special conditions for transporting and storing samples

The investigators may usefully refer to the procedures of the investigator guide (see annex 1) or product sheets (when they exist).

4.5.1. Special transport conditions

The product must be left in the original packaging whenever possible. This is especially true for olive oils, coffees, chicory coffees, teas, household or agricultural pesticides, cosmetic and hygiene products and distilled or demineralised waters.

Additional protection must nevertheless be guaranteed to maintain the characteristics of the goods, for example:

- to prevent increasing the proportion of fragments in coffee;
- to prevent some compounds like formaldehyde from evaporating (floors, materials in contact with foodstuffs, etc.); contact should be made with the competent laboratory to determine how to proceed (type of overpack to be used, etc.);
- to prevent cross-contamination during the transport of samples intended to find materials that are in contact with food or pesticide residues and samples for microbiological analysis;
- to prevent contamination through samples of fruit and vegetables intended to seek out pesticide residues coming into contact with other processed products, ventilated bags must be used (mesh nets are not suitable);
- to prevent the degradation of mycotoxins, the samples must be transported, wherever possible, in opaque packaging;
- to prevent the degradation of specific components contained in foods intended for particular nutritional use (e.g. most vitamins), it is important to protect them from potential degradation as much as possible.

If fractioning takes place, the label or original packaging must be sent, whenever possible, to the laboratory with its intended sample.

It is mandatory to store samples of perishable products using the cold chain, in accordance with the instructions of the investigator guide (see annex 1).

4.5.2. Sample storage conditions

All samples must be stored in the best conditions, if necessary away from light, heat or humidity, depending on the nature and the indications marked on their packaging.

Only two chemical preservatives – potassium dichromate and salicylic acid – can be used in the following conditions with the added preservatives stated on the label:

- potassium dichromate used for bulk raw milk at a rate of 0.25 g per 250 ml bottle,
- salicylic acid used for vinegary wines at a rate of 1 g per bottle. A sample without the addition of this preservative is sent to the laboratory at the same time.

4.6. Food products

4.6.1. Food additives and processing aids

Flavouring agents	200 g
Other additives (emulsifiers, thickeners, texturising agents, nutritive value improvers, etc.):	
- pure (purity criteria)	50 g
- dosage in food products	200 g
- in formulation (composition)	200 g
Oenological products:	
- all products except enzymes	500 g
- enzymes	50 g

4.6.2. Animal feed

Mineral compounds, simple or compound feeds	>= 500 g to be adapted to the number of determinations requested
Additives	250 g minimum
Dioxin, PCBs	See 4.11 "Pollutants and contaminants"

4.6.3. Drinks

Table, spring and mineral waters	1 l (excluding microbiological analysis)

		+ 1 l if micro-pollutants
Fruit and vegetable juices, nectars, non-alcoholic drinks		1 l
Concentrated fruit juices		250 ml
Cordials		500 ml
Lemonades and fizzy drinks	1 sample for CO2 dosing and 1 sample for the other determinations + 1 sample for volume measurement	2 x 500 ml (3 if greater volume)
Preparation for drinks		100 g
Wines	General case	750 ml
	NMR analysis: 1 additional sample	750 ml
	Fresh grapes for micro-vinification for NMR	10 kg min
	Search for ochratoxin A	See Mycotoxins
Ciders	1 sample for NMR analysis and 1 sample for the other determinations	2 x 750 ml
Aperitifs		700 ml
Spirits		700 ml
Natural flavours of cordials		750 ml

4.6.4. Fruit, vegetables, mushrooms

1. Pesticide residues

Refer to the laboratory guidelines for the quantities to be taken.

2. Nitrates

Lettuce and spinach	Nitrate dosing	10 lettuce or 1 kg of spinach

3. Heavy metals, mycotoxins, radioactivity

See relevant chapters.

4. Other determinations not specified elsewhere

Dried fruit and vegetables (nuts, prunes, apricots, peanuts, etc.)	Humidity, varietal identification, rancidification, calibre, preservatives	1 kg (at least 5 units for coconuts)

Fruit and vegetable preserves (including stewed fruit)			250 g (or 1 sales unit)
Potatoes		varietal identification	at least 30 tubers
Mushrooms	fresh	varietal identification	500 g
	dried	varietal identification	30 g
Truffles	Whole or in bits	varietal identification	a number of pieces or bits representative of the lot representing 25 g or 1 sales unit
	fragments	varietal identification	50 g or 1 sales unit
	peel	varietal identification	25 g or 1 sales unit
	juice	varietal identification	100 ml

4.7. Milk, milk products, eggs and egg products

Natural milks:		
- milk from all animal species, in raw state, pasteurised, sterilised, flavoured		250 ml (or 1 sales unit)
- milk from all animal species, for a limited control seeking antibiotics or identification of the producing species		150 ml
Processed milks:		
- concentrated milks		150 g (or 1 sales unit)
- powdered milks		100 g (or 1 sales unit)
- fermented, gelled, renneted		120 ml (or 1 sales unit)
- creams and cream desserts		100 g (or 1 sales unit)
- milk desserts		100 g (or 1 sales unit)
- cheese	general case	150 g
	cheese: dosing of the natamycin	200 cm^2 surface crust
Eggs		6 units
All types of egg products		250 g

4.8. Fats

Butter	more than 100 g or 1 sales unit
Other fats (all types: fats, oils) (*)	250 g (100 ml minimum for frying oils)

(*) Conditioned olive oil is always kept in its original packaging.

4.9. Microbiology

Refer to the product data sheets or laboratory guidelines for the quantities to be taken.

4.10. Genetically modified organisms (GMO)

Seeds	oilseed rape	200 g minimum
	corn and soy bean	1 kg minimum
Raw products (grain, etc.)		10000 grains or their equivalent weight with a maximum of 3 kg
Products of first-stage processing (semolinas, flour, grits, meals, etc.)		1 kg
Liquid products		500 ml
Paste and viscous products		500 g
Finished products (conditioned)		2 individual packages or 100 g

4.11. Pollutants and contaminants

4.11.1. Asbestos

Asbestos detection	pre-packed products	as is
Asbestos detection	materials (sheets of fibre cement, etc.)	5 x 5 cm surface area

4.11.2. Polycyclic Aromatic Hydrocarbons (PAHs)

PAHs	Human and animal food	500 g

4.11.3. Heavy metals

<table>
<tr><td rowspan="10">Pb, Cd, Hg</td><td colspan="2" rowspan="2">pre-packed products</td><td>liquids: 1 unit</td></tr>
<tr><td>solids: 1 to 10 packs or units depending on the lot volume</td></tr>
<tr><td rowspan="8">bulk products</td><td>minimum weight</td><td>1 kg made up of three to ten increments according to the weight of the lot</td></tr>
<tr><td colspan="2">However, the rules below can be applied to the following products. For the others, contact the laboratory (additives, herbs and spices, etc.).</td></tr>
<tr><td>cereals as grain or flour</td><td>300 g</td></tr>
<tr><td>fruit, vegetables, mushrooms, potatoes</td><td>300 g if unit weight < 25 g
1 kg if unit weight > 25 g</td></tr>
<tr><td>fish</td><td>3 units (500 g minimum)</td></tr>
<tr><td>shellfish, crustaceans, molluscs</td><td>1 kg</td></tr>
<tr><td>animal feed</td><td>500 g minimum</td></tr>
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4.11.4. Mycotoxins

Mycotoxins in food intended for human consumption	
Ochratoxin: cereals and raisins	1 to 10 kg depending on lot weight
Ochratoxin: wine and grape juice	1 litre
Patulin: juice, stewed fruit	1 to 10 packs or units
Alfatoxins B and G: dried fruit, oil crops, cereals	3 to 30 kg depending on lot weight
Aflatoxins B and G: spices	1 to 10 kg depending on lot weight
Aflatoxin M1: dairy products	500 g minimum or 1 litre minimum
Other mycotoxins, other processed products	500 g minimum
Mycotoxins in products intended for animal feed	
Aflatoxin B1	500 g minimum per global sample (the number of global samples depends on the size of the lot)

4.11.5. Nitrates in plants

Nitrates in plants	See products

4.11.6. Polychlorinated biphenyls (PCBs) and dioxins

PCBs and dioxins	human food	1 kg (for the PE1, a 500 g sample is deemed sufficient, even 200 g for high-fat products)
PCBs and dioxins	animal feed	500 g
Detecting MCPD (monochloropropane diol)		1 sales unit of 100 ml minimum (liquids) or 200 g (solids)

4.11.7. Radioactivity

Fruit and vegetables	detecting radioactivity	1 kg
Cereals and legumes	detecting radioactivity	1 kg
Animal foodstuffs	detecting radioactivity	1 kg
Milk	detecting radioactivity	1 l
By-products	detecting radioactivity	1 kg (or 1 l)
Aromatic plants and dried mushrooms	detecting radioactivity	200 g

4.11.8. Pesticide residues

Pesticide residues	See products

4.12. Special food products and nutritional labelling

4.12.1. Minimum required for a full analysis

Basic analysis and other determinations	200 g or 400 ml
Vitamins and polyphenols	300 g or 400 ml
Minerals and trace elements	50 g

4.12.2. Quantities by type of determination

Caloric value and dietary fibres	100 g or 200 ml
Amino-acids, sugars, polyols, sweetening agents, preservatives	100 g or 200 ml
Miscellaneous nutritional additives (creatine, carnitine, etc.)	100 g or 200 ml
Common vitamins: C, B1, B2, B5, PP, B6, H, B9, A, E, β.carotene	150 g or 250 ml
Vitamin D, K, nucleotides, choline, carotenoids	200 g or 300 ml
Polyphenols (flavonoids, isoflavones, OPCs)	50 g or 100 ml
Minerals - trace element: amount to meet most requests	50 g
Sodium, potassium, calcium, magnesium	10 to 20 g
Phosphorous	10 to 20 g
Zinc, copper, manganese, iron, chromium	10 to 20 g
Iodine, selenium	20 g minimum
Food supplements based on plants and released medicinal plants: macro- and microscopic examination, dosing of main active ingredients	50 g
Protein allergens	20 g per type of allergen

The quantities taken must be accumulated for a full analysis.

4.13. Aromatic products, soups and seasonings

4.13.1. Flavourings, spices and essential oils

Flavourings and spices:	
orange blossom water	300 ml
vanilla pods or powder	100 g
vanilla extract	100 ml
peppers (all types)	25 g
saffrons	5 g for basic analysis
other flavourings or spices	50 ml or 50 g

Flavouring agents:	
base product (... flavouring, ... extract), concentrated or otherwise	25 ml
compound flavourings, aromatic compositions, floral waters	250 g (or 250 ml)
natural flavourings	See products

4.13.2. Infusion plants

Infusion plants, fresh medicinal plants	dosage of pesticide residues	200 g
Infusion plants, fresh or dried medicinal plants	identification	25 g

4.13.3. Coffees, teas, chicory

Green coffees	identification	350 g
Roasted coffees, whether or not decaffeinated, as beans or ground	identification	250 g
Coffee substitutes	identification	100 g preferably
Chicory coffees	identification	100 g packed
Teas	identification	100 g
Soluble extracts or liquids of coffee, chicory, tea or coffee/chicory	identification	100 g (or 100 ml)

4.13.4. Stocks and soups

Liquid or paste products	200 g (or 200 ml)
Powdered products	100 g

4.13.5. Vinegars

All types of vinegar	500 ml

4.13.6. Other seasonings (condiments and salt)

Mustards, condiments, sauces	100 g
Salts (all types)	100 g
Brines (fresh preparations before any use)	250 ml

4.14. Meat-based products

4.14.1. Cooked meats, meat- and poultry-based products

Meats and offal (including minced meat)	200 g
Meat-based products (to determine the species)	50 g lean meat (no bard, rind, crust, etc.)
Meat-based products (for the histological examination)	50 g
Cooked meat preparations in bulk, canned or semi-preserved (cassoulet, choucroute, etc.)	one sales unit (if physical and chemical analysis on meat content, 200 g minimum of meat content)
Cooked meats (all preparations, including hams)	300 g (so that 200 g remain after removing the fat, rind, bard, jelly, crust, etc.)
Coated cooked meats (barded, with crust, in jelly, etc.)	600 g (so that 200 g remain after removing the fat, rind, bard, jelly, crust, etc.)
Truffled preparations (to determine the proportion of truffles)	600 g
Foie gras and (chemical-)based preparations	200 g
Foie gras and foie gras-based preparations (% of bits)	1 sales unit
Foie gras and foie gras-based preparations (for histological examination only)	50 g (or 1 sales unit)

4.14.2. Fishery and aquaculture products

Whole fish (including species identification)	400 g or several specimens
Bits of fish, fillets, etc. (including species identification)	250 g
Fresh, frozen and deep-frozen pre-shelled crustaceans	250 g
Fresh, frozen and deep-frozen unshelled crustaceans	400 g
Scallops	300 g
Frozen shellfish	250 g of the edible part
Canned fish, crustaceans and miscellaneous pre-packs	1 sales unit
Canned food for histamine determination	9 units from the same lot
Canned and deep-frozen food for metrological control (net	20 units from the same lot

weight, glazing)	
Miscellaneous	200 g
Polyphosphates	200 g

4.14.3. Gasteropods, amphibians

Gasteropods		12 to 24
Amphibians	Ionisation detection	200 g minimum

4.15. Cereal products

Cereals (for human consumption)	500 g
Flours and dusting agents	200 g
Breadmaking flours (to determine the W value)	1 kg
Breads (all types, including toasts) - Crispbreads (all types, must be in original packaging)	200 g
Dietary pasta (all types)	200 g
Fresh pastries and industrial pastries, biscuits, gingerbreads	200 g
Semolinas, tapiocas	200 g

4.16. Sugar-based products

Sugars	150 g
Vanilla sugars	100 g
Honeys and hive products	150 g
Royal jelly	20 g
Confectionery products (including candied fruit)	250 g
Natural flavourings	400 g
Jams, marmalades, jellies	250 g
Cocoas, chocolate products and breakfasts	150 g
Chocolates and confectionery products containing inclusions (e.g. hazelnut chocolates, nougats with dried fruit, etc.)	300 g

Ices, ice creams, sorbets	200 g
Preparations for making ices, ice creams and sorbets	200 g

4.17. Industrial materials and industrial products

4.17.1. Fibres, textiles and leather

Materials, in lengths or samples	1/3 of a 20 cm strip across the whole width
Patterned materials in lengths or samples	as above and with at least two full patterns
Items for the feet (stockings, socks, etc.)	1 pair
Clothing	1 complete item if it has different parts and colours
Detecting azo dyes in the textiles and leathers	1 complete item or a representative part of all colours present, minimum 5 g per colour
Flammability test	1 unit per maintenance mode stipulated on the labelling

4.17.2. Personal care, cosmetics

Aerosol products	general case	2 generators
	control of the regulation under pressure	6 generators
Perfumery and grooming products containing alcohol		25 ml or 25 g
Products not necessarily containing alcohol:		
Products intended for contact with the mucous membranes:		
- dental and mouth care products		50 ml or 50 g
Products intended for other uses:		
- beauty products (mask, cream, emulsion, gel, oils, foundations, powders); products presented as affecting the skin (sun, sun tanning, anti-wrinkle products); deodorants, anti-perspirants; products containing surface agents (shampoo, bath product, toilet soaps, etc.)		100 ml or 100 g
- products for hair (all types)		2 packs
- products for the nails (varnish, removers)		25 ml

- tattooing inks		2 x 50 ml
- PPE	for optical control	1
- Sunglasses	for control against standard	5

4.17.3. Hardware products

Aerosol products (general case)	2 generators
Control of the regulation under pressure	6 generators
Other products:	
- cleaning products for materials entering into contact with foodstuffs (disinfectants not subject to authorisation, dishwashing rinsing products, etc.)	250 ml or 250 g
- disinfectant or anti-pest products not subject to authorisation (household products, products for industries, descaling agents, bleach, etc.)	250 ml or 250 g
- soap (all types), detergents, softeners, scouring products, cleaning products (waxes, greases, polishes), strippers, products for windows, wood varnishes, glosses, waterproofing agents, primers, stain removers, solvents (trichloroethylene, turpentine, benzene, etc.), paints, varnishes, driers, adhesives, linseed oil, inks and any hazardous or poisonous substances or preparations (phosphoric, hydrochloric or sulphuric acid, soda, potassium, ammonium, etc.)	100 ml or 100 g
Distilled or demineralised waters	1 pack
Methylated spirits, denatured alcohols	1 pack

4.17.4. Fuels, petroleum products

Petroleum fuels (unleaded and leaded petrol, engine mixes)	2 l

4.17.5. Toys

Soft toys with removable clothes	European safety standard	2 units
Toys	request for tests relating to toy standards	1 unit
	for electric toys, when tests under standard EN 50088 are also requested	1 additional unit
Percussion primers for priming		minimum 100 primers

pistol		

4.17.6. Conditional material for food contact

Wood for PCP analysis	see product data sheets or the laboratory	200 cm²
Rubber		10 g
Ceramics, glass		4 units
Metal materials		3 units and at least 60 cm²/unit
Plastics		12 units
Plastics in the form of film		12 units of A4 format size

4.17.7. Metals and alloys

Metals and alloys	all types excluding precious metals	100 g
Jewels	to detect allergenic metals, false precious stones	1 jewel 2 pearls

4.17.8. Furniture, home decoration

Furniture	safety, identification of the wood	1 unit
Garland	constructive analysis and IP index	1 unit

4.17.9. Tools

Electric tools		1 (2 if EMC)
Thermal motor tools		1

4.17.10. Small household goods and domestic equipment

Electrical goods	e.g. robots, hairdryers, diffusers, light fittings, etc.	1 (2 if EMC)
Domestic equipment	e.g. kitchen utensils	to be specified according to the type of equipment

4.18. Agricultural and assimilated products

4.18.1. Plant health products

Agriculture pest control and assimilated products	
- pre-packed products	1 sales unit
- bulk products	500 g

4.18.2. Seeds (excluding GMO)

Cereals	
- corn - basic seed of inbred lines	250 g
- rice	500 g
- buckwheat	600 g
- oats, wheats (durum and soft), corn (basic seed apart from inbred lines and certified seed), barley, rye, sorghum, triticales	1 kg

Vegetables NB: - for F1 hybrids, the minimum weight can be reduced to 1/4 of the fixed weight, without dropping below 5 g and include at least 400 grains - for lots packed in small packages, the sample can be reduced to 2 bags without the number of grains dropping below 400 per sample.	
- celery, watercress, marjoram, purslane, savory, thyme	5 g
- dill, basil, carrot, lettuce, sorrel, parsley, dandelion	10 g
- chicories (curly endive, scarole, witlof and wild, large leaved)	15 g
- orach, aubergine, chervil, Chinese leaves, lamb's tongue, turnip (root), parsnip, leek, roquette, tomatoes	20 g
- cabbage (including broccoli and cauliflower), Welsh onions, chives, cucumber-gherkin, garden cress, fennel, onion	25 g
- salsify, black salsify	30 g
- hot pepper-pepper	40 g
- cardoon, radishes	50 g
- spinach, rhubarb	75 g
- asparagus, garden beetroot, melon, Swiss chard	100 g

- artichoke, courgette	150 g
- water melon, New Zealand spinach, pumpkin-gourd	250 g
- squashes, marrow	350 g
- lentils, peas	500 g
- beans	700 g
- broad beans, Spanish beans, sweetcorn	1 kg
Industrial beet and chicories	
- industrial chicory	50 g
- beet	500 g
Oil and fibre plants	
- poppy seed	50 g
- mustards (brown and black)	100 g
- oilseed rape, common turnip	200 g
- flax (oil and textile)	300 g
- white mustard	400 g
- hemp	600 g
- soy bean, sunflower	1 kg
Forage plants	
- yarrow, crested dog's tail	25 g
- bentgrass (all species), yellow oat grass, Bermuda grass, timothy (bulbous, field), oxeye daisy, bluegrass (all species)	50 g
- Brome (other than ceratochlea), foxtail millet	90 g
- orchard grass, fescue (tall, sheep's, meadow or red), Harding grass, plantain, soapwort, meadow foxtail	100 g
- kidney vetch, ceratochlea brome, swede, kale, festulolium, tall oat grass, bird's-foot trefoil, rye grass (all species), white clover, hybrid clover, Persian clover, rutabaga	200 g
- crown vetch	250 g
- alfalfa, black medick, forage radish, *phaceliata nacetifolia*, purple clover	300 g

- sainfoin grain, Spanish sainfoin grain, Alexandria clover	400 g
- brachypodium	450 g
- fenugreek, crimson clover	500 g
- sainfoin fruit	600 g
- burnet, subterranean clover	650 g
- forage sorghum	900 g
- field beans, lupin (all species), forage and protein peas, Spanish sainfoin fruit, vetch (all species)	1 kg
- potatoes (seed potatoes only, do not confuse with table potatoes)	0.5 kg

4.18.3. Other industrial materials

Baby soothers	15
Imitation weapons (ball pistols, not toys)	1 unit
Articles imitating foodstuffs	1 unit
Natural and artificial Christmas trees	1 unit
Decorative candle arrangements	1 unit
Other industrial material	see with the laboratory

4.18.4. Other industrial products

Cements	measurement of the hexavalent chromium rate	5 kg
Other		see with the laboratory

5. Appendices

Annex 1: Investigator guide in terms of quality approach sampling

Sheet 1: Necessary preparations prior to field surveying

1. List of equipment

- Sampling labels;
- Sampling report;
- Reimbursement form;
- Sampling equipment: thermometer, sterile bags, sampling pouches, seals, wax, sealing clamp, single-use gloves, disinfectant wipes, gas jet, etc.;
- Review any laboratory instructions about the products.

2. Prepare the insulated boxes

1. Clean and disinfect the box if necessary.
2. Insert the eutectic plates according to size (capacity) of the box and the desired temperature.

Capacity of the insulated box **for example**	**Used to transport fresh products above 0°C**		**Used to transport deep-frozen and frozen products**	
	Number of small plates (-3°C)	**Number of large plates (-3°C)**	**Number of small plates (-21°C)**	**Number of large plates (-21°C)**
12 litres	2 placed on the side	1 placed in the slide of the box lid	2 placed on the side	1 placed in the slide of the box lid
30 litres	4 placed on the side	1 placed in the slide of the box lid	4 placed on the side	1 placed in the slide of the box lid

These minimum rules must be applied strictly, except for samples without temperature restrictions.

Advice

- It is advisable to add extra plates in extreme heat.
- The use of an additional icebox can limit opening times when repeated samples are taken in several companies.

When using a thermo button:

A numbered thermo button is placed in the insulated boxes (do not put the thermo button in a plastic food bag to avoid disrupting it).

Indicate the exact position of thermo buttons.

Sheet 2: Sampling in the company

Preamble

The sampling is a extremely important administrative act.

A contentious procedure, even the administrative closure of a company can depend on it.

The sampling technique and conditions, the type, quantity and quality of samples and their storage and transport conditions must therefore comply with precise, defined and encoded procedures that are listed below.

The sampling will be rejected by the laboratory if there is a failure to comply with these guidelines.

Therefore, at any time in the "inspection-laboratory" sequence, do not hesitate to seek advice from the laboratory technician so that you take the samples under conditions that comply with regulation.

The inspectors must have in their possession the contact details of the Control and Test Body that governs their control structure

1. Samplings

- Carry out the samplings according to the laboratory or administrative instructions (quantities to be sampled).
- It is essential to note the time of each sampling on the label(s) and repeat it in the relevant sampling report.
- Seal them (to make the products inaccessible) with the labels, checking that they have been signed by the inspector and the holder (see § 2).
- **Caution:** for microbiological sampling, the sample is placed firstly in a sterile bag or container and then in a second bag. Only the second bag is sealed with the sampling label.
- Refrigerated and frozen products must not be placed in the same container.
- Hot-sampled products must cool without fail on the sampling site before being placed in a box.
- Load the samples (if appropriate, change the thermo button between the samples) and close the insulated box.
- In the summer and/or extreme heat, it is preferable to group the samplings in the same locality if possible or to use several iceboxes.

2. Labels

Caution: fill in all the headings!

- **origin of samples:** sampling location and origin of products making up the sample; it is advisable to be as precise as possible on the manufacturing conditions of the sampled product;
- **name and brand:** where appropriate;
- **sampling date**;
- **date of manufacture**;
- **sampling number:** in both the log and the DCW;
- **analysis to be made:** to be specified or otherwise in physico-chemistry (in microbiology, the non-specification of the determination leads to the application of a pre-established search table).

Do not forget to have the labels signed by the professional and the investigator(s).

3. Sampling report

Write the report filling in all the headings, deleting as appropriate, indicating the number of deleted words and lines and paginating the document.

- The sampling time written on the label is repeated by the inspector in the body of the sampling report.
- For samplings with no value (frying oil, calf urine, etc.), the statement will be "Amount of the value declared by him: nil" (delete the statement "estimated by the inspector").

WHERE THE REGULATIONS MAKE PROVISION FOR THIS:

4. Reimbursement slip

All units sampled are reimbursed, including those left in the professional's care.

Fill in the reimbursement slip requesting supporting documentation for the value of the sample. The value of samples to be reimbursed must be indicated before signing the report (no deferred value statement). If the professional does not declare a value, the inspector estimates it.

The purchase price or cost price including VAT should be indicated.

Sheet 3: Operations to be carried out when returning to the department

For samplings under controlled temperature

1. Tranship the samplings into the refrigerator or freezer

For temporary storage in the department.

2. When using a thermo button: read it

- For the iceboxes, the data recorded by the thermo button must be read upon your return to the department, on the specific PC.
- The information recorded every five minutes on the thermo button is saved for seven days, then deleted. It is therefore essential to read the temperature and print the corresponding curve when returning from each survey.
- **Caution:** temperatures taken into account are those included in the time interval between the sampling and the transfer of samples into the cold cabinet in the department (adjust the time based on the programming and the time of year - summer or winter).
- If the reading is non-compliant, contact the laboratory to find out what should be done with the samplings.

For all the samplings

3. Record the sampling in the department's administrative log

- The number allocated to the sampling must be included in the report or the sample taking report which is then filed.
- This number must also be shown on the label(s) of sampling(s) sent to the laboratory and those stored in the department.
- Make sure that data agree between the sampling labels, the report and the entries in the administrative log in the department.

4. Classifying samples (where there are no guidelines)

- Each department states here how to identify samples when this information is given in their instructions (example: coloured stickers).
- Each department states how to store samples as described in their instructions.

Sheet 4: Sending the sample to the laboratory

Important: Who prepares and who sends? See instructions.

1. Meet the deadlines for the shipment to the laboratory

- The samples must be sent as quickly as possible to the laboratory.
- For the samples with a limited lifetime, a maximum time must be fixed between the sampling date and the shipment date; by the regulations:
  As a guide (shipment time normally applied in Europe):
  - **For the microbiological analysis:**
    < 48 hours for fresh produce
    < 10 days for frozen produce
  - **For the physico-chemical analysis:**
    < 36 hours to 72 hours for prepared products (ready-to-use products, trimmed beans, etc.) and for fruit and vegetables
    < 15 days for other fresh produce and frozen produce.
- Shipment must be made respecting the fixed temperature and in sufficient time for the laboratory to commence its analysis before the best before date.

- The shipment day in the week must take account of the shipment time, working days and the possibility of being taken in charge until the laboratory deposit phase.
- For products with no limited lifetime, the maximum time between the sampling date and the shipment date to the laboratory is thirty days.

2. Checking the sampling

Check the indications on the back of the label:

- either **"routine analysis"** or **"standard analysis";**
- or precise **"analytical research"** requested by the inspector.

Attach any documents that may be useful to the analysis (manufacturing sheet, etc.).

3. Prepare and ship the package

- Make sure the contents will not move before you ship.
- Special case of samplings subjected to temperature constraints: do everything possible to ensure transport that complies strictly with the temperature constraints (additional eutectic plates, etc.).

4. Checking the acknowledgement of receipt of the package

- Acknowledgement of receipt at the return from the laboratory: to be filed in the department.
- If the laboratory states a non-conformity on the acknowledgement of receipt, draw up a non-conformity sheet.

Sheet 5: Monitoring the sample on its return from the laboratory analysis

1. Confirm that the test report agrees with the record in the department (contact the laboratory if they do not match).
2. Inform the professional holder and/or person in charge of the first marketing immediately of the test report conclusions.
3. Where appropriate, recover and file the third sample.
4. File the documents (test report, letter to the professional, etc.).

Annex 2: Control flow chart per sampling

Stages	
Preliminary reflection on the sampling operation to be carried out	See instructions given in 3.1.2
Follow the instructions in the sampling plan	See instructions given in 3.1.3
Identifying the lot before any sampling	See instructions given in 3.1.4, n° 1
Examining the lot before any sampling	See instructions given in 3.1.4, n° 2
Essential homogeneity of increments taken in heterogeneous lots	See instructions given in 3.1.4, n° 3
Taking increments	See instructions given in 3.1.4, n° 4
Constituting samples from increments	See instructions given in 3.1.4, n° 5
Sampling report	See investigator guide (see annex 1)

Annex 3

Example of random sampling of large quantities of pre-packed products: 50 bottles of fizzy drinks in a warehouse

Lot composition:

- 25 pallets, each holding 100 boxes
- 6 bottles in each box

1. Application of the sampling plan if one exists.
2. Otherwise:

Constitution of a first level sample	Note for each pallet: - the lot number, - the date and time of manufacture, - give each pallet a virtual number if there is no lot number, - put them in an urn and pick five numbers at random, - identify and mark the five pallets.
Constitution of a second level sample	Choose ten boxes from each pallet thus designated, i.e. a total of fifty boxes.

	Choice of boxes: front, rear, top, bottom, left and right. Note all the indications applied to each box.
Constitution of a third level sample (final sample)	Take one bottle of fizzy drink from each box thus defined. Choice of bottles: front, rear, middle, left and right. Note the lot numbers, date and time of manufacture and all markings that identify the sample to be analysed.

The sample of fifty bottles is constituted this way.