

THIS REPORT CONTAINS ASSESSMENTS OF COMMODITY AND TRADE ISSUES MADE BY USDA STAFF AND NOT NECESSARILY STATEMENTS OF OFFICIAL U.S. GOVERNMENT POLICY

Voluntary - Public

Date: 12/15/2018

GAIN Report Number: TS1824

Tunisia

Post: Tunis

Maximum Limits for Contaminants in Foodstuffs

Report Categories:

Sanitary/Phytosanitary/Food Safety

SP2 - Prevent or Resolve Barriers to Trade that Hinder U.S. Food and Agricultural Exports

FAIRS Subject Report

Grain and Feed

Approved By:

Morgan Haas

Prepared By:

FAS/Tunis

Report Highlights:

This report contains an unofficial translation of Tunisia's maximum limits and the methods of sampling and analysis for official control of contaminants in foodstuffs, including for metals, mycotoxins, dioxins and PCBs, PAHs, nitrates, radionuclides, acrylonitrile, VCM, melamine, and 3-MCPD. Key limits, including for deoxynivalenol in wheat, have been set at levels more restrictive than Codex Alimentarius. Tunisian imports of targeted foodstuffs are valued at \$2 billion, of which 10% comes from the United States. This measure was not notified to the WTO.

Republic of Tunisia

Official Gazette

Annex of Republic of Tunisia Official Gazette No. 44 dated May 31, 2013

Order of the Minister of Health, the Minister of Industry, the Minister of Commerce and Handicrafts, the Minister of Agriculture and the Minister of Equipment and Environment of May 13, 2013, fixing the list of maximum limits for some contaminants in foodstuffs and the methods of taking samples and analysis for the official control.

Order of the Minister of Health, the Minister of Industry, the Minister of Trade and Handicrafts, the Minister of Agriculture and the Minister of Equipment and Environment dated 13 May 2013 fixing the list of the maximum limits for some contaminants in foods and the methods of taking samples and analysis for the official control

The Minister of Health, the Minister of Industry, the Minister of Trade and Handicrafts, the Minister of Agriculture and the Minister of Equipment and Environment,

Considering [Constituent Act No. 2011-6](#) of December 16, 2011, providing for the provisional organization of public authorities,

Considering the [Law No. 92-117](#) of December 7, 1992, on the consumer protection,

Considering the [Law No. 2009-38](#) of June 30, 2009, on the National Standardization System,

Considering the [Decree No. 74-1064](#) of November 28, 1974, concerning the definition of the duties and responsibilities of the Ministry of Public Health,

Considering the [Decree No. 95-916](#) of May 22, 1995, concerning the definition of the tasks and responsibilities of the Ministry of Industry, as amended and supplemented by [Decree No. 2010-3215](#) of December 13, 2010,

Considering the [Decree No. 2001-419](#) of February 13, 2001, regarding the laying down the responsibilities of the Ministry of Agriculture

Considering the [Decree No. 2001-2965](#) of December 20, 2001, the laying down the responsibilities of the Ministry of Commerce,

Considering the [Decree No. 2005-2933](#) of November 1, 2005, laying down the responsibilities of the Ministry of the Environment and Sustainable Development,

Considering the [Decree No. 2013-1372](#) of March 15, 2013, appointing members of the Government,

Considering the [Order](#) of the Minister for the National Economy of September 18, 1993 laying down the procedures for the collection of samples provided for by [Law No. 92-117](#) of December 7, 1992 on consumer protection, as amended and supplemented by the [Order](#) of July 21, 2003.

Order:

Article 1 - The provisions of this Order shall apply to foodstuffs intended for human consumption.

Art. 2 - The purpose of this Order is to fix the maximum tolerated limits of contaminants in foodstuffs intended for human consumption, with the exception of mineral waters and drinking water.

The following substances are excluded from the scope of this Order:

- contaminants not having an impact on public health, but only on food quality,

- pesticide residues,
- residues of veterinary medicinal products,
- microbial toxins, such as botulinum toxin and staphylococcus enterotoxin,
- technological additives.

Art. 3 - For the purposes of this Order:

Foodstuffs: Any processed, partially processed or raw material intended for human consumption including beverages, chewing gum and all substances used in the manufacture, preparation or treatment of food, excluding substances used only as medicines, cosmetics or tobacco.

Contaminants: Any substance not intentionally added to the food, but which is however present in the latter as a residue of the production, manufacture, processing, preparation, processing, packaging, transport or storage of the foodstuff, or as a result of environmental contamination. The term does not apply to insect debris, rodent hairs and other foreign substances.

Maximum limit: The maximum limit (ML) for a contaminant in a food is the maximum concentration of that substance that must be legally permitted for that product.

Art. 4 - The methods of sampling and the methods of analysis for the official control of the limits of certain contaminants in foodstuffs are set out in Annex II of this Order.

Art. 5 - It is prohibited to place on the market the foodstuffs referred to in Annex I of this Order where they contain contaminants that exceed the maximum limit.

Art. 6- The maximum limits referred to in Annex I to this Order shall apply to the edible part of the concerned foodstuffs, unless otherwise specified in that Annex.

Art. 7 - It is prohibited to use foodstuffs which have levels exceeding the maximum limits laid down in Annex I of this Order as food ingredients

Art. 8- Any violation of the provisions of this order is liable to prosecution in accordance with the laws and regulations in force.

Art. 9 - Any previous provisions contrary to this Order are repealed and in particular the Tunisian standard NT 117.02 (1983) approved by the decree of January 25 1986.

Art. 10 - This Order will be published in the Official Gazette of the Republic of Tunisia. Tunis, May 13, 2013.

Cleared by

Head of Government **Ali Larayedh**

Minister of Health **Abdellatif Mekki**

Minister of Commerce and Handicraft **Abdelwahab Maater**

Minister of Agriculture **Mohamed Ben Salem**

Minister of Equipment and Environment **Mohamed Salmane**

Minister of Industry **Mehdi Jomaa**

ANNEX I
Maximum Limits for Some Contaminants
in Foodstuffs

1. METALS

Definition: Heavy metals are minerals that are classed as heavy because of their high density. They can be absorbed directly through the food chain, causing chronic or acute effects. The main heavy metals are lead, cadmium, mercury, arsenic.

Sampling and analysis methods Annex II.1: Methods of sampling and analysis for official control of levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo (a) pyrene in foodstuffs.

Foodstuffs		Maximum Limits (mg/kg in weight in fresh state)	Remarks / observations
1.1	Lead (Pb)		
1.1.1	Raw milk, heat treated milk and milk for producing dairy products.	0.020	
1.1.2	Formulas for infants and follow-on formulas	0.020	The maximum limits in products to be used (placed on the market as such or after reconstitution in accordance with the manufacturer's instructions).
1.1.3	Bovine meat, mutton, pork and poultry (excluding offal)	0.10	
1.1.4	Bovine meat, mutton, pork and poultry	0.50	
1.1.5	Muscle meat of fish.	0.30	When the fish is to be consumed whole, the maximum level applies to the whole fish.
1.1.6	Crustaceans: muscle flesh appendages and the abdomen (at the exclusion of cephalothorax from crustaceans). In the case of crabs and crustaceans of crab type (Brachyura et Anomura), muscle flesh of the appendages.	0.50	
1.1.7	Bivalve molluscs.	1.5	
1.1.8	Cephalopods (without viscera).	1.0	

Foodstuffs		Maximum Limits (mg/kg in weight in fresh state)	Remarks / observations
1.1.9	Vegetable legumes, cereals and legumes	0.020	For vegetable legumes the maximum limit applies once they are washed and the edible part is separated.
1.1.10	Vegetables, excluding brassicas, leafy vegetables, herbs, mushrooms and seaweed. In the case of potatoes, the maximum limit applies to peeled products.	0.010	The maximum limit applies once the fruit or vegetables have been washed and the edible portion is separated.
1.1.11	Brassicas, leafy vegetables and mushrooms: Agaricus bisporus (Paris mushroom), Pleurotus ostreatus (oyster mushroom), Lentinula edodes (Shiitake).	0.30	- The maximum limit for leafy vegetables does not apply to herbs - The maximum limit for mushrooms applies once they are washed and the edible part is separated.
1.1.12	Fruits, excluding berries and small fruits.	0.10	The maximum limit applies once the fruit has been washed and the edible part is separated.
1.1.13	Berries and small fruits.	0.20	The maximum limit applies once the fruit has been washed and the edible part is separated.
1.1.14	Oils and fats, including milk fat.	0.10	
1.1.15	Fruit juices, reconstituted concentrated fruit juices and fruit nectars.	0.050	
1.1.16	Wines (including sparkling wines, but excluding liqueur wines), ciders, perry and fruit wines.	0.20	
1.1.17	Aromatized wines, aromatized wine-based drinks and aromatized wine-product cocktails	0.20	
1.1.18	Food supplements	3.0	The maximum level relates to the food supplement as offered for sale
1.2	Cadmium		
1.2.1	Meat of beef, mutton, pork and poultry (excluding offal).	0.050	
1.2.2	Horse Meat, excluding offal.	0.20	

Foodstuffs		Maximum Limits (mg/kg in weight in fresh state)	Remarks / observations
1.2.3	Livers of beef, sheep, pigs, Poultry and horse.	0.50	
1.2.4	Kidneys of beef, mutton, pig, poultry and horse.	1.0	
1.2.5	Muscular flesh of fish, excluding species listed in Points 1.2.6, 12.7 and 1.2.8.	0.050	When the fish is to be consumed whole, the maximum level applies to the whole fish.
1.2.6	Muscular flesh of the following fishes: Bonito (<i>Sarda sarda</i>) sar a blackhead (<i>Diplodus vulgaris</i>) eel (<i>Anguilla anguilla</i>) thick-lipped mullet (<i>Mugil labrosus labrosus</i>) horse mackerel (<i>Trachurus</i> species) louvereau (<i>Luvarus imperialis</i>) mackerel (<i>Scomber</i> species) sardine (<i>Sardina pilchardus</i>) sardinops (<i>Sardinops</i> species) than (<i>Thunnus</i> species, <i>Euthynnus</i> species, <i>Katsuwonus pelamis</i>) Céteau or language of lawyer (<i>Dicologlossa cuneata</i>)	0.10	When the fish is to be consumed whole, the maximum level applies to the whole fish.
1.2.7	Muscular flesh of the following fishes: Bonitou (<i>Auxis</i> species)	0.20	When the fish is to be consumed whole, the maximum level applies to the whole fish.
1.2.8	Muscular flesh of the following fishes: Anchovies (<i>Engraulis</i> species) swordfish (<i>Xiphias gladius</i>)	0.30	When the fish is to be consumed whole, the maximum level applies to the whole fish.
1.2.9	Crustaceans: muscular flesh of appendages and abdomen (excluding cephalothorax of crustaceans). In the case of crabs (<i>Brachyura</i> and <i>Anomura</i>), muscular flesh of the appendages.	0.50	
1.2.10	Bivalve mollusks.	1.0	
1.2.11	Cephalopods (without viscera).	1.0	
1.2.12	Cereals, excluding bran, germ, wheat and rice	0.10	
1.2.13	Bran, germ, wheat and rice	0.20	
1.2.14	Soybeans	0.20	
1.2.15	Vegetables and fruit, excluding leafy vegetables, herbs, cabbages, mushrooms, stem-vegetables, root and tuber vegetables, and seaweeds	0.050	The maximum limit applies once the vegetables have been washed and the edible portion is separated

1.2.16	Root vegetables, root vegetables and tuber vegetables, excluding celeriac. In the case of potatoes, the maximum limit applies to the peeled products	0.10	The maximum limit applies once the vegetables have been washed and the edible portion is separated
1.2.17	Leafy vegetables, herbs, cabbages, celeriac and mushrooms: Agaricus bisporus (Paris mushroom), Pleurotus ostreatus (oyster mushroom), Lentinula edodes (Shiitake).	0.20	The maximum limit applies once the vegetables have been washed and the edible portion is separated
1.2.18	Mushrooms, excluding those listed in 1.2.17	1.0	The maximum limit applies once the vegetables have been washed and the edible portion is separated
1.2.19	Food supplements, excluding those listed in point 1.2.20	1.0	The maximum level relates to the food supplement as offered for sale
1.2.20	Food supplements composed exclusively or mainly of dried seaweed, seaweed products, or dried bivalve mollusks	3.0	
1.3	Mercury		
1.3.1	Fishery products (26) and muscular flesh of fish (24) (25), excluding the species listed in point 1.3.2. The maximum level for crustaceans applies to the muscular flesh of appendages and abdomen (excluding cephalothorax from crustaceans). In the case of Crab crabs (Bachyura and Anomura), it applies to the muscular flesh of the appendages.	0.50	When the fish is to be consumed whole, the maximum level applies to the whole fish.
1.3.2	Muscular flesh of the following fish: Anglerfish (Lophus species) Wolf (Anarhichas lupus) Bonito (Sarda sarda) eel (Anguilla species) emperor, orange roughy, Mediterranean roughy (Hoplostethus species) Grenadier of rock (Coryphaenoides rupestris) Halibut (Hippoglossus hippoglossus) Cape abapse (Genypterus capensis) Marlin (Makaira Species) Cardin (Lepidorhombus species) mullet (Mullus species) Rose (Genypterus blacodes) Pike (Esox Lucius) Palomite (Orcynopsis unicolor) Mediterranean Capelin (Tricopterus minutes) Common pailona (Centroscymnes coelolepis) Rays (Raja species) Large redfish (Sebastes marinus, S. mentella, S. viviparus) Sailboat (Istiphorus platypterus) Sabers (Lepidopus caudatus, Aphanopus carbo) Dorado, pageotus (Pagellus species) Sharks (all species))	1.0	When the fish is to be consumed whole, the maximum level applies to the whole fish.

Foodstuffs		Maximum Limits (mg/kg in weight in fresh state)	Remarks / observations
	escolier serpent (Lepidocybium flavobrunneum, Ruvettus pretiosus, Gempylus serpens) Sturgeon (Acipenser species) Swordfish (Xiphias gladius) Tuna (Thunnus species, Euthynnus species, Katswonus pelamis).		
1.3.3	Food supplements.	0.10	The maximum level relates to the food supplement as offered for sale
1.4	Tin (inorganic)		
1.4.1	Canned food other than beverages.	200	
1.4.2	Canned beverages, including fruit and vegetable juices.	100	
1.4.3	Canned baby foods and canned cereal preparations for infants and young children, excluding dried and powdered products.	50	The maximum limit applies to the product as it is placed on the market.
1.4.4	Infant formula and canned follow-on preparation (including infant milk and follow-on milk), excluding dried and powdered products.	50	The maximum limit applies to the product as it is placed on the market
1.4.5	Canned dietetic foods for special medical purposes specifically for infants, excluding dried and powdered products.	50	The maximum limit applies to the product as it is placed on the market
1.5	Arsenic		
1.5.1	Fat and edible oils.	0.1	
1.5.2	Margarine.	0.1	
1.5.3	Minarine	0.1	
1.5.4	Animal fats with a specific name (Fat, melted pork fat, primary juice and edible tallow).	0.1	
1.5.5	Olive oil, refined	0.1	
1.5.6	Olive oil, virgin	0.1	
1.5.7	Olive oil, pomace oil	0.1	
1.5.8	Vegetable oils, raw peanut, babassu, coconut, cotton seed, grape seed, corn, mustard seed, palm kernel, palm tree, rapeseed, safflower, sesame seed, soybean, and sunflower seeds, and palm olein, stearin and palm superolein.	0.1	
1.5.9	Vegetable oils, edible peanuts, babassu, coconut, cottonseed, grape seed, corn, mustard seed, palm kernel, palm tree, rapeseed, safflower, sesame seed, soybean, and sunflower seeds, and olein, stearin and palm superolein.	0.1	
1.5.10	Food grade salt	0.5	

2. MYCOTOXINS

Definition: The term mycotoxin comes from the Greek "mycos" which means fungus. It refers to the toxic chemicals produced by certain molds that develop on certain foods. These substances are very toxic to humans. They can cause cancerous tumors, but also lesions on the kidney, liver, genes, the immune system, the genitals, and the nervous system. About 20 families of mycotoxins are health concerns such as aflatoxins; Ochratoxins, zearalenone, fumonisin, patulin... Aflatoxins are the best known of mycotoxins. They are produced by the fungi of the *Aspergillus* species. The four main aflatoxins detected in the contaminated vegetal products are B 1, B 2, G 1 and G 2. In addition, aflatoxin derivatives can be found in milk and dairy products (aflatoxins M1 and M2). These derivatives are produced by ruminants fed with contaminated foodstuffs.

Sampling and analysis methods Annex II.2: Methods of sampling and analysis for official control of levels of mycotoxin in foodstuffs;

Foodstuffs		Maximum Limits (µg/kg)			Remarks / observations
2.1	Aflatoxins	B1	B1+B2+G1+G2	M1	
2.1.1	Peanuts and other oilseeds intended for a sorting treatment or other physical methods prior to human consumption or use as food ingredients: Except - peanuts and other oilseeds intended for crushing for the production of refined vegetable oil	8.0	15.0	-	The maximum limits refer to the part of peanuts and nuts intended to be consumed. If peanuts and "whole" nuts are analyzed, it is assumed, when calculating the aflatoxin content, that all of the contamination is on the part intended to be consumed, except for Brazil nuts
2.1.2	Almonds, pistachios and apricot kernels intended for sorting or other physical methods prior to human consumption or for use as food ingredients	12.0	15.0	-	
2.1.3	Hazelnuts and Brazil nuts intended for sorting or other physical methods prior to human consumption or use as food ingredients	8.0	15.0		
2.1.4	Nuts, with the exception of nuts listed in points 2.1.2 and 2.1.3, intended for sorting	5.0	10.0	-	

Foodstuffs		Maximum Limits (µg/kg)			Remarks / observations
	or other physical methods prior to human consumption or use as food ingredients				
2.1.5	Peanuts and other oilseeds and products derived from their processing, intended for direct human consumption or for use as food ingredients except: - raw vegetable oils intended for refining - refined vegetable oils.	2.0	4.0	-	The maximum limits refer to the part of peanuts and nuts intended to be consumed. If peanuts and "whole" nuts are analyzed, it is assumed, when calculating the aflatoxin content, that all of the contamination is on the part intended to be consumed, except for Brazil nuts
2.1.6	Almonds, pistachios and kernels of apricot intended for direct human consumption or use as food ingredients.	8.0	10.0	-	
2.1.7	Hazelnuts and Brazil nuts intended for direct human consumption or for use as food ingredients.	5.0	10.0		
2.1.8	Nuts, with the exception of the nuts listed in points 2.1.6. And 2.1.7, and products derived from their processing intended for direct human consumption or for use as an ingredient in foodstuffs	2.0	4.0	-	
2.1.9	Dried fruits intended for sorting or other physical methods prior to human consumption or use as food ingredients	5.0	10.0	-	
2.1.10	Dried fruit and products derived from their processing, intended for direct human consumption or for use as an ingredient in foodstuffs	2.0	4.0.	-	
2.1.11	All cereals and all products derived from cereals, including processed cereal products, with the exception of the foodstuffs listed in points 2.1.12, 2.1.15 and 2.1.17	2.0	4.0	-	

Foodstuffs		Maximum Limits (µg/kg)			Remarks / observations
2.1.12	Corn and rice intended for sorting or other physical methods prior to human consumption or for use as food ingredients	5.0	10.0	-	
2.1.13	Raw milk, heat-treated milk and milk intended for the processing of milk-based products	-	-	0.050	
2.1.14	Following spice categories: Capsicum spp. (derived dried fruit, whole or in powder form, including peppers, chili powder, cayenne pepper and paprika) Piper spp. (Fruit derived, including white pepper and black pepper) Myristica fragrans (nutmeg) Zingiber officinale (ginger) Curcuma longa (Indian saffron) Mixtures of spices containing one or more of the above mentioned spices	5.0	10.0	-	
2.1.15	Preparations with cereals and baby foods for infants and young children.	0.10	-	-	The maximum limit applies to dry matter
2.1.16	Infant formulas and follow-on formulas, including infant milk and follow-on milk.	-	-	0.025	The maximum limits are for ready-to-use products (placed on the market as such or after reconstitution in accordance with the manufacturer's instructions).
2.1.17	Dietetic foods for special medical purposes specifically for infants	0.10	-	0.025	The maximum limits refer to in the case of milk and milk products, products ready for use (placed on the market as such or reconstituted in accordance with the manufacturer's instructions) and, in the case of products other than milk and dairy products, to the dry matter.

Foodstuffs		Maximum Limits (µg/kg)	Remarks / observations
2.2	Ochratoxin A (OTA)		
2.2.1	Raw cereals	5.0	
2.2.2	All products derived from raw cereals, including processed cereal products and cereals intended for direct human consumption, except for foodstuffs listed in points 2.2.9 and 2.2.10	3.0	-
2.2.3	Raisins (currants, sultanas and other raisins)	10.0	
2.2.4	Roasted coffee beans and ground roasted coffee, except for instant coffee	5.0	
2.2.5	Soluble coffee (instant coffee)	10.0	
2.2.6	Wines (including sparkling wines but excluding liqueur wines and wines with a minimum alcoholic strength by volume of 15%) and fruit wines.	2.0	
2.2.7	Aromatized wines, aromatized wine-based drinks and aromatized wine-product cocktails.	2.0	The maximum OTA limit applicable to these beverages depends on the proportion of wine and / or grape must present in the finished product
2.2.8	Grape juice, reconstituted concentrated grape juice, grape nectar, grape must and reconstituted concentrated grape must, intended for direct human consumption.	2.0	
2.2.9	Preparations of cereal-based and baby foods for infants and young children.	0.50	The maximum limit applies to dry matter
2.2.10	Dietetic foods for special medical purposes specifically for infants.	0.50	The maximum limits refer to in the case of milk and milk products, products ready for use (placed on the market as such or reconstituted in accordance with the manufacturer's instructions) and, in the case of products other than milk and dairy products, to the dry matter.
2.2.11	Spices Capsicum spp. (derived dried fruit, whole or in powder, including chillies, chili powder, cayenne pepper and paprika) Piper spp. (Derived fruits, including white pepper and black pepper) Myristica fragrans (nutmeg) Zingiber officinale (ginger) Curcuma longa (Indian saffron) Mixtures of spices containing one or more of the above mentioned spices	15	

Foodstuffs		Maximum Limits (µg/kg)	Remarks / observations
2.2.12	Licorice (<i>Glycyrrhiza glabra</i> , <i>Glycyrrhiza inflata</i> and other species)		The maximum limit applies to the pure and undiluted extract when 1 kg of extract is obtained from 3 to 4 kg of licorice wood
2.2.12.1	Licorice wood, ingredient for infusion	20	
2.2.12.2	Licorice extract for use in food products, in particular beverages and confectionery	80	
2.3	Patuline		
2.3.1	Fruit juices, reconstituted concentrated fruit juices and fruit nectars.	50	
2.3.2	Spirits, cider and other fermented beverages produced from apples or containing apple juice	50	
2.3.2	Products made from apple pieces, such as applesauce and apple puree, intended for direct consumption, with the exception of foodstuffs referred to in points 2.3.4 and 2.3.5.	25	
2.3.4	Apple juice and products from apple pieces, such as applesauce and apple puree, intended for infants and young children, and labeled and sold as such.	10.0	The maximum levels relate to the products ready for use (placed on the market as such or after reconstitution in accordance with the manufacturer's instructions).
2.3.5	Foods for babies, other than cereal preparations, for infants and young children.	10.0	The maximum limits are for ready-to-use products (placed on the market as such or after reconstitution in accordance with the manufacturer's instructions).
2.4	Deoxynivalenol		For the application of the maximum deoxynivalenol limits, rice is excluded from 'cereals' and rice products are excluded from 'cereal products'.
2.4.1	Raw cereals other than durum wheat, oats and corn.	1250	The maximum limits apply to raw cereals placed on the market for primary processing. "Primary processing" means any physical or thermal treatment applied to the grain, other than drying. Cleaning, sorting and drying operations are not considered to be "Primary processing" because no physical action is exerted on the grain itself and the grain remains completely intact after cleaning and sorting. In integrated production and processing systems, the maximum level applies to raw cereals provided that they are intended for primary processing.
2.4.2	Durum wheat and raw oats	1750	
2.4.3	Raw corn with the exception of raw corn intended for processing by wet milling.	1750	

Foodstuffs		Maximum Limits (µg/kg)	Remarks / observations
2.4.4	Cereals intended for direct human consumption, cereal flour, bran and germ as a finished product marketed for direct human consumption, with the exception of the foodstuffs listed in points 2.4.7, 2.4.8 and 2.4.9	750	
2.4.5	Pasta (dried)	750	Pasta (dry) is pasta with a water content of about 12%.
2.4.6	Bread (including small baked goods), pastries, biscuits, cereal snacks and breakfast cereals	500	
2.4.7	Preparations of cereals and baby foods for infants and young children.	200	The maximum limit applies to dry matter.
2.4.8	Milling fractions of corn with particle size > 500 microns and other corn milling products with particle sizes > 500 microns not intended for direct human consumption.	750	
2.4.9	Milling fractions of corn with a particle size ≤500 microns and other corn milling products with a particle sizes ≤500 microns not intended for direct human consumption.	1250	
2.5	Zearalenone		
2.5.1	Raw cereals other than corn	100	The maximum limits apply to raw cereals placed on the market for primary processing. "Primary processing" means any physical or thermal treatment applied to the grain, other than drying. Cleaning, sorting and drying operations are not considered to be a "primary processing" because no physical action is exerted on the grain itself and the grain remains completely intact after cleaning and sorting. In integrated production and processing systems, the maximum level applies to raw cereals provided that they are intended for primary processing.
2.5.2	Raw corn with the exception of corn intended to be processed by wet grinding.	350	
2.5.3	Cereals intended for direct human consumption, cereal flour, bran and germ as a finished product marketed for direct human consumption, with the exception of the foodstuffs listed in 2.5.6, 2.5.7, 2.5.8, 2.5.9, and 2.5.10	75	
2.5.4	Refined corn oil	400	
2.5.5	Bread (including small bakery products), pastries, biscuits, cereal snacks and breakfast cereals, excluding corn snacks and corn breakfast cereals	50	

Foodstuffs		Maximum Limits (µg/kg)	Remarks / observations
2.5.6	Corn for direct human consumption, corn-based snacks and breakfast cereals based on corn	100	
2.5.7	Preparations of cereals (excluding corn preparations) and baby foods for infants and young children.	20	The maximum limit applies to dry matter
2.5.8	Corn preparations for infants and young children.	20	The maximum limit applies to dry matter
2.5.9	Milling fractions of corn with a particle size > 500 microns and other corn milling products with a particle size > 500 microns not intended for direct human consumption.	200	
2.5.10	Milling fractions of corn with a particle size ≤500 microns and other maize milling products with a particle size ≤500 microns not intended for direct human consumption.	300	
2.6	Fumonisin	SumB1+B2	
2.6.1	Raw maize with the exception of raw maize intended for processing by wet milling.	4000	The maximum limits apply to raw cereals placed on the market for primary processing. "Primary processing" means any physical or thermal treatment applied to the grain, other than drying. Cleaning, sorting and drying operations are not considered to be a "primary processing" because no physical action is exerted on the grain itself and the grain remains totally intact after cleaning and sorting. In integrated production and processing systems, the maximum level applies to raw cereals provided that they are intended for primary processing.
2.6.2	Corn intended for direct human consumption, corn-based foodstuffs intended for direct human consumption, with the exception of the foodstuffs listed in points 2.6.3 and 2.6.4.	1000	
2.6.3	Corn breakfast cereals and corn snacks	800	
2.6.4	Corn preparations and baby foods for infants and young children.	200	The maximum limit applies to dry matter
2.6.5	Milling fractions of corn with a particle size > 500 microns and other corn grinding products with a particle size > 500 microns not intended for direct human consumption.	1400	

Foodstuffs		Maximum Limits (µg/kg)	Remarks / observations
2.6.6	Corn milling fractions with particle size ≤500 microns and other maize milling products with a particle size ≤ 500 microns not intended for direct human consumption.	2000	

3. DIOXINS AND DIOXIN-LIKE PCBs

Definition: The term "dioxin" refers to a group of 75 congeners of the family of polychlorinated dibenzo p-dioxins ("PCDDs") and 135 congeners of the polychlorinated dibenzofuran ("PCDF") family, 17 of which are toxicologically important. The most toxic congener is 2, 3, 7, 8-tetrachlorodibenzo-p dioxin (TCDD) has been listed as having carcinogenicity on humans.

Polychlorinated biphenyls (PCBs) constitute a group of 209 different congeners that can be classified into two categories according to their toxicological properties: 12 of them have toxicological properties similar to those of dioxins and are therefore often referred to as "PCBs" Of dioxin type ". Other PCBs, which do not exhibit this dioxin-like toxicity, have a different toxicological profile.

Sampling and analysis methods Annex II.3: Methods of sampling and analysis for official control of levels of dioxins (PCDD/PCDF) and dioxin-like PCBs in certain foodstuffs.

Foodstuffs		Maximum Limits		Remarks / observations
		Sum of Dioxins and Furans WHO-PCDD / F-TEQ)	Sum of dioxins, furans and dioxin-like PCBs WHO-PCDD / F-PCB- TEQ)	The higher concentrations are calculated assuming that all values of the different congeners below the quantification limit are equal to the limit of quantification.
3.1	Meat and meat products (excluding edible offal) derived from the following animals: - beef and sheep - poultry - pork	3.0 pg / g of fats (*) 2.0 pg / g of fats (*) 1.0 pg / g of fats (*)	4.5 pg / g of fats(*) 4.0 pg / g of fats(*) 1.5 pg / g of fats(*)	Maximum levels of dioxins [sum of polychlorinated dibenzo-para-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), expressed as World Health Organization (WHO) toxic equivalent, by applying WHO-TEF (Toxic Equivalency Factors , 1997)] and limits
3.2	Terrestrial animal livers referred to in point 3.1 and products derived from these livers	6.0 pg/g of fats (*)	12.0 pg/g of fats(*)	

Foodstuffs		Maximum Limits		Remarks / observations
3.3	Muscle flesh of fish and fishery products and derived products, excluding eels (**). The maximum limit for crustaceans applies to the muscle flesh of appendages and abdomen (excluding crustacean cephalothorax). In the case of crabs and crabs-like crustaceans (Brachyura and Anomura), applies to the muscular flesh of the appendages.	4.0 pg/g fresh weight	8.0 pg/g weight fresh	<p>Maximum sum of dioxins and PCBs of the fishery and dioxin type [sum of polychlorinated dibenzo-para-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polychlorinated biphenyls (PCBs) expressed as toxic equivalents of the World Health Organization), Applying the TEF-WHO (toxic equivalence factors, 1997 (*)]</p> <p>- (*): The maximum levels do not apply to foodstuffs containing <1% fat.</p> <p>- (**): When the fish is to be consumed whole the maximum level applies to whole fish.</p> <p>- (***) : For canned fish liver, the maximum level shall apply to all the contents of the preserves intended for consumption.</p>
3.4	Muscle meat of eel (<i>Anguilla anguilla</i>) and derived products	4.0 pg/g fresh weight	12.0 pg/g fresh weight	
3.5	Raw milk and dairy products including butterfat	3.0 pg/g of fats (*)	6.0 pg/g of fats (*)	
3.6	Chicken eggs and egg products	3.0 pg/g of fats (*)	6.0 pg/g of fats (*)	
3.7	Fats of the following animals: -cattle and sheep -poultry -pork	3.0 pg/g of fats 2.0 pg/g of fats 1.0 pg/g of fats	4.5 pg/g of fats 4.0 pg/g of fats 1.5 pg/g of fats	
3.8	Mixed animal fats	2.0 pg/g of fats	3.0 pg/g of fats	
3.9	Vegetable oils and fats	0.75 pg/g of fats	1.5 pg/g of fats	
3.10	Marine oils (fish body oil, fish liver oil and oils of other marine organisms intended for human consumption)	2.0 pg/g of fats	10.0 pg/g of fats	
3.11	Fish liver and fish products derived for its processing, excluding marine oils referred to in point 3.10		25.0 pg/g fresh weight (***)	

4. POLYCYCLIC AROMATIC HYDROCARBONS (PAH)

Definition: PAHs represent a family of more than one hundred organic molecules with at least two aromatic cycles. They are divided into two categories: low molecular weight compounds with less than 4 aromatic cycles and high molecular weight compounds having 4 or more cycles. Some molecules have a carcinogenic effect: 1,2-benzanthracene, chrysene, fluranthene and especially benzo [a] pyrene or (3,4-benzopyrene). Benzo (a) pyrene can be used as a marker for the presence and effect of carcinogenic PAHs in food.

Sampling and analysis methods Annex II.1: Methods of sampling and analysis for official control of levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo (a) pyrene in foodstuffs.

Foodstuffs		Maximum Limits (µg/kg of weight at fresh state)	Remarks / observations
4.1	Benzo (a) pyrene		
4.1.1	Oils and fats (excluding cocoa butter) intended for direct human consumption or for use as food ingredients.	2.0	
4.1.2	Smoked meat and smoked meat products	5.0	
4.1.3	Muscular flesh of smoked fish and smoked fishery products, excluding bivalve mollusks. The maximum limit for smoked crustaceans applies to the muscle flesh of appendages and abdomen (excluding crustacean cephalothorax). In the case of smoked crab and crab crustaceans (Brachyura and Anomura), it applies to the muscular flesh of the appendages.	5.0	- When the fish is to be consumed whole, the maximum limit applies to the whole fish.
4.1.4	Muscle flesh of non-smoked fish	2.0	-When the fish is to be consumed whole, the maximum limit applies to the whole fish.
4.1.5	Non-smoked crustaceans and cephalopods. The maximum limit for crustaceans applies to the muscle flesh of appendages and abdomen (excluding cephalothorax of crustaceans). In the case of crabs and crustaceans and crabs of (Brachyura and Anomura) type, it applies to the muscular flesh of the appendages.	5.0	
4.1.6	Bivalve mollusks	10.0	

Foodstuffs		Maximum Limits ($\mu\text{g}/\text{kg}$ of weight at fresh state)	Remarks / observations
4.1.7	Cereal preparations and baby foods for infants and young children.	1.0	-The maximum limit applies to the product as it is placed on the market.
4.1.8	Infant formulas and follow-on formulas, including infant milk and follow-on milk.	1.0	-The maximum limit applies to the product as it is placed on the market.
4.1.9	Dietetic foods for special medical purposes specifically for infants.	1.0	-The maximum limit applies to the product as it is placed on the market.

5. NITRATES

Definition: Nitrates are substances that are not toxic, but their metabolism is composed of N-nitro (nitrites, nitrosamines) makes them hazardous. In humans, nitrites are responsible for the risks of acute methemoglobinemia, which is predominantly observed in infants. The risks associated with the formation of nitrosamines, namely the occurrence of cancers, are currently less well established.

Sampling and analysis methods Annex II.4: Methods of sampling and analysis for official control of levels of nitrate in certain foodstuffs.

Foodstuffs		Maximum Limits ($\text{mg NO}_3/\text{kg}$)		Remarks / observations
5.1	Fresh spinach (<i>Spinacia oleracea</i>).	Harvest from October 1st to March 31 st	3000	
		Harvest from April 1st to September 30 th	2500	
5.2	Spinach, preserved, frozen		2000	
5.3	Fresh lettuce (<i>Lactuca sativa</i> L.) (lettuce grown under cover and cultivated lettuce in the field with the exception of lettuce in point 5.4)	Harvest from October 1st to March 31 st :		
		- Lettuce grown under cover	4500	
		- Lettuces grown outdoors	4000	
5.3		Harvest from April 1st to September 30 th :		
		- Lettuce grown under cover	3500	
		- Lettuces grown outdoors	2500	
5.4	"Iceberg" type lettuces	- Lettuce grown under cover	2500	
		- Lettuces grown outdoors	2000	
5.5	Cereal preparations and baby foods for infants and young children.		200	The maximum limits for ready-to-use products (placed on the market as such or after reconstitution in accordance with the manufacturer's instructions).

6. RADIONUCLIDES

Definition: Most of the ionizing radiation in our environment comes from radionuclides. Radionuclides may be naturally occurring or derived from industrial activity. Most radionuclides are naturally present at very low concentrations, but incidents resulting from accidents or malicious acts have shown how easy it is to contaminate a very large area.

Sampling and analysis methods

Foodstuffs		Representative Radionuclides	Maximum Limits (Bq/kg)	Remarks / observations
6.1	Foods for infants*	^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Am	1	*: When they are intended for this purpose **: This corresponds to the value of the sulfide (organically bound) ***: This corresponds to the value of tritium (organically bound)
		^{90}Sr , ^{106}Ru , ^{129}I , ^{131}I , ^{235}U	100	
		$^{35}\text{S}^{**}$, ^{60}Co , ^{89}Sr , ^{103}Ru ,	1000	
		^{134}Cs , ^{137}Cs , ^{144}Ce , ^{192}Ir , $^3\text{H}^{***}$, ^{14}C , ^{99}Tc	1000	
6.2	Food, except infant food	^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Am	10	*: When they are intended for this purpose **: This corresponds to the value of the sulfide (organically bound) ***: This corresponds to the value of tritium (organically bound)
		^{90}Sr , ^{106}Ru , ^{129}I , ^{131}I , ^{235}U	100	
		$^{35}\text{S}^{**}$, ^{60}Co , ^{89}Sr , ^{103}Ru ,	1000	
		^{134}Cs , ^{137}Cs , ^{144}Ce , ^{192}Ir , $^3\text{H}^{***}$, ^{14}C , ^{99}Tc	10000	

7. ACRYLONITRILE

Definition: The acrylonitrile monomer is the starting material for the manufacture of polymers used as fibers, resins, rubber and also as packaging materials for foods rich in oxalic acid. Acrylonitrile does not exist naturally. Acrylonitrile is classified as a possible human carcinogen (group 2B). Polymers derived from acrylonitrile may also contain small amounts of free monomer.

Sampling and analysis methods

Foodstuffs	Maximum Limits (mg/kg)	Remarks / observations
7.1 Food for human consumption.	0.02	

8. CHLORIDE OF VINYL MONOMER

Definition: Vinyl Chloride Monomer (VCM) is the main substance from which polymers, used as resins, are made as packaging materials for food. Vinyl chloride does not exist naturally. VCM residues may be present in the polymer. Vinyl chloride is considered a human carcinogen.

Sampling and analysis methods

	Foodstuffs	Maximum Limits (mg/kg)	Remarks / observations
8.1	Food for human consumption.	0.01	

9. MELAMINE

Definition: Melamine is a chemical used to produce resins, usually by reaction with formaldehyde. It has many industrial uses such as the production of laminated panels, glues, adhesives, molding powders, coatings and flame retardants. It is a permitted substance for the manufacture of synthetic materials which may be in contact with foodstuffs. C'est aussi un métabolite d'un pesticide (Ia cyromazin). La mélamine est aussi utilisée comme engrais.

Sampling and analysis methods

	Foodstuffs	Maximum Limits (mg/kg)	Remarks / observations
9.1	Food for human consumption (other than preparations for infant).	2.5	-The maximum limit applies to levels of melamine that are unintentionally and unavoidably present in foods for human consumption. -The maximum limit does not apply to foods for which it is possible to demonstrate that the melamine limit exceeding 2.5 mg / kg is due to:
9.2	Preparations for infants.	1.0	<ul style="list-style-type: none"> ○ The authorized use of cyromazine as an insecticide. The limit of melamine will not exceed the limit of cyromazine. ○ Migration from food contact materials taking into account any allowable migration limits at the national level. <p>- The maximum limit does not apply to melamine which may be present after processing in the ingredients / additives of the following animal feedstuffs: guanidino acetic acid (GAA), urea and biuret following normal processing.</p>

10. MONOCHLORO-PROPANE-1,2-DIOL (3-MCPD)

Definition: 3-Monochloropropane-1,2-diol (3-MCPD) belongs to the range of chemicals known as chloropropanols. These substances are contaminants that form during the processing and manufacture of certain foods and ingredients. They were initially discovered in acid-hydrolyzed vegetable proteins (HVP). They may be present in soy sauces where HVPs are used as ingredients.

Sampling and analysis methods Annex II.1: Methods of sampling and analysis for official control of levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo (a) pyrene in foodstuffs.

Foodstuffs		Maximum Limits ($\mu\text{g}/\text{kg}$)	Remarks / observations
10.1	Hydrolyzed vegetable protein.	20	The maximum level is given for the liquid product containing 40% of dry matter, corresponding to a maximum level of 50 $\mu\text{g} / \text{kg}$ in dry matter. This figure shall be adjusted in proportion to the dry matter content in the product.
10.2	Soy sauce	20	

ANNEX II

Methods of sampling and analysis for official control of levels of contaminants in foodstuffs

- **ANNEX II.1:** METHODS OF SAMPLING AND ANALYSIS FOR OFFICIAL CONTROL OF LEVELS OF LEAD, CADMIUM, MERCURY, INORGANIC TIN, 3-MCPD AND BENZO (A) PYRENE IN FOODSTUFFS ;
- **ANNEX II.2:** METHODS OF SAMPLING AND ANALYSIS FOR OFFICIAL CONTROL OF MYCOTOXIN LEVELS IN SOME FOODSTUFFS;
- **ANNEX II.3:** METHODS OF SAMPLING AND ANALYSIS FOR OFFICIAL CONTROL OF LEVELS OF DIOXIN (PCDD / PCDF) AND DIOXIN-LIKE PCBS IN SOME FOODSTUFFS;
- **ANNEX II.4:** SAMPLING AND ANALYSIS METHODS FOR OFFICIAL CONTROL OF NITRATE LEVELS IN SOME FOODSTUFFS.

ANNEX II.1
METHODS OF SAMPLING AND ANALYSIS FOR OFFICIAL CONTROL
OF LEVELS OF LEAD, CADMIUM, MERCURY, INORGANIC TIN, 3-
MCPD AND BENZO (A) PYRENE IN FOODSTUFFS

1. DEFINITIONS

For the purposes of this annex, the following definitions shall apply:

“lot”: a quantity of identifiable foodstuff, delivered at one time, delivered at one time, and determined by the official to have common characteristics (such as origin, variety, packaging type, packaging, sender or marker). In the case of fish, the size of the animal must also be comparable;

“sub-lot”: the part of the lot with a large size selected for sampling. Each sub-lot shall be separate and identifiable;

“incremental sample”: a quantity of material taken at one single point of the lot or sub-lot;

“aggregate sample”: All the incremental samples taken from the lot or sub-lot; the aggregate samples are considered representatives of the lots or sub-lots from which they are taken;

“laboratory sample”: a sample that dedicated for a laboratory.

2. METHODS OF SAMPLING

2.1 GENERAL PROVISIONS

2.1.1. Personnel

Sampling is made by an authorized person in compliance with applicable regulations.

2.1.2. Material for sampling

Any lot or sub-lot, which is to be analyzed, shall be sampled separately.

2.1.3. Precautions

In the course of sampling and preparation of the samples, precautions shall be taken to avoid any alteration that could impact the contaminant levels or affect the analysis or representativeness of the aggregate samples.

In addition, all necessary measures should be taken to ensure the safety of the persons taking the samples.

2.1.4. Incremental Samples

If possible, the samples are taken at different points distributed throughout the lot or sub-lot.

2.1.5. Preparation of the aggregate sample

The aggregate sample is made up by combining all the incremental samples.

2.1.6. Samples dedicated for control, defense or reference purposes

The samples dedicated for control, defense or reference purposes are taken from the homogenized aggregate sample.

2.1.7. Packaging and transmission of samples

Each sample is placed in a clean inert container, with adequate protection against contamination risks, the loss of substance by absorption through the internal wall of the container and possible damage in transit. All necessary precautions shall be taken to avoid any modification in the composition of the sample during transportation or storage.

2.1.8. Sealing and labeling of samples

Each sample shall be officially sealed at the place of sampling and identified in compliance with applicable regulations.

2.2. SAMPLING PLANS

Big size lots are subdivided into sub-lots, provided that the sub-lots can be physically separated. For products marketed in bulk (cereals, for example), **Table 1** is applied. For other products, **Table 2** is applied. Since the weight of a lot is not always an exact multiple of the weight of the sub-lots, the weight of the sub-lots may exceed the specified weight by up to 20%.

The global sample must be at least 1 kilo or 1 liter, except where this is not possible, for example when the sample consists of a single package or a single unit.

The minimum number of incremental samples to be taken from the lot or sub-lot is shown in **Table 3**.

In the case of bulk liquid products, wherever possible and provided that the quality of the product does not dissolve, the lot or sub-lot is carefully mixed or homogenized, either by a manual process or by a mechanical process just before sampling. In this case, it is possible to assume a homogeneous distribution of the concerned contaminants within a given lot or sub-lot. It is therefore sufficient to take three incremental samples per lot or sub-lot to constitute the aggregate sample.

All incremental samples have a similar weight. Each incremental sample weighs at least 100 grams or 100 milliliters forming an aggregate sample of at least 1 kilogram or 1 liter.

Table 1: Subdivision of lots into sub-lots for products marketed in bulk

Weight of the lot (in tons)	Weight or number of sub-lots
≥1500	500 tons
>300 and <1500	3 sub-lots
≥100 and ≤300	100 tons
<100	-

Table 2: Subdivision of lots into sub-lots for other products

Weight of the lot (in tons)	Weight or number of sub-lots
≥15	15-30 tons
≥15	-

Table 3: Minimum number of elementary samples to be taken from the lot or sub-lot

Weight or volume of the lot-sub-lot (in kilos or liters)	Minimum number of elementary samples to collect
<50	3
≥50 and ≤500	5
>500	10

If the lot or sub-lot is in separate packages or units, the number of packages or units to be taken to form the aggregate sample is shown in **Table 4**.

Table 4: Number of packages or units (incremental samples) to be collected to form the aggregate sample if the lot or sub-lot consists of separate packages or units

Number of packages or units in the lot/sub-lot	Number of packages or units to be collected
≤25	at least one package or unit
26-100	About 5%, at least two packages or units
>100	About 5%, ten packs or units at the maximum

Maximum limits for inorganic tin apply to the contents of each box. It is nevertheless necessary to use an aggregate sample for practical reasons. If the result of the test done on the boxes of an aggregate sample is only slightly below the maximum allowable limit for inorganic tin, and if it is assumed that some boxes are likely to exceed this maximum limit, further analysis may be necessary.

2.3. SAMPLING AT THE STAGE OF RETAIL

Sampling (or "sampling") of foodstuffs at the retail stage shall, as far as possible, be carried out in accordance with the relevant provisions of points 2.1 and 2.2 of this Annex.

If this is not feasible, another method of collection at the retail stage may be used, provided that it ensures sufficient representativeness of the sampled lot or sub-lot.

3. PREPARATION AND ANALYSIS OF SAMPLES

3.1 Quality standards applied at the laboratory

Analytical laboratories shall comply with the following requirements:

3.1.1 Materials' Requirement:

- a. The staff performing the analysis shall be impartial and shall not have any conflict of interest with regard to the exercise of the tasks delegated to them.
- b. The laboratories must have adequate capacity to carry out the examinations and sufficiently qualified and experienced staff in sufficient numbers to carry out the analysis and the monitoring requirements effectively and efficiently.

- c. They must have appropriate and properly maintained facilities and equipment that enable staff to carry out the analyzes in an efficient and effective manner;

3.1.2 Moral Requirement: Transparency and Confidentiality:

- a. Analytical laboratories ensure that their activities are conducted with a high level of transparency. To this end, the relevant information shall be made available to the competent authority as soon as possible.
- b. The analytical laboratory shall adopt the necessary measures to ensure that its staff members are required not to disclose information obtained in the exercise of their analytical tasks and which are by their nature covered by professional secrecy.

3.2 Sample processing and preparation:

3.2.1. Treatment of the sample received in the laboratory:

The aggregate sample is finely ground (if necessary) and thoroughly mixed according to an approved method guaranteeing complete homogenization.

Samples must be handled and labeled in such a way to ensure both legal and analytical validity.

3.2.2. Samples dedicated for control, defense or reference purposes

Samples dedicated for control, defense or reference purposes are taken from homogenized material.

3.2.3 Preparation of the sample

It essentially consists of obtaining a sample from the representative and homogeneous laboratory without introducing secondary contamination.

The total sample received by the laboratory must be used for the preparation of the laboratory sample.

Compliance with the maximum limits laid down in this Order shall be established on the basis of the levels determined in the laboratory's samples.

3.2.4. Specific Sample Preparation Procedures for TMA Analysis

The analyst must ensure that the samples are not contaminated during preparation. Wherever possible, the appliances and equipment that are in contact with the sample must not contain the researched metals and must be made of inert materials, for example plastics such as polypropylene, polytetrafluoroethylene (PTFE), etc. They must be cleaned with acid to minimize the risk of contamination.

High quality stainless steel or plastic can be used for cutting edges.

Many specific specimen preparation procedures can be used satisfactorily for the products under consideration.

3.3. Methods of analysis:

3.3.1 General Requirement

1. Analysis methods used for official control are standardized methods in conformity with:
 - a) if such standards do not exist, to internationally acknowledged protocols or those adopted in national legislation, or
 - b) In the absence of this, to the appropriate methods for this end, or elaborated according to scientific protocols.

2. If paragraph 1 is not applicable, analysis methods may be validated within the laboratory following a protocol, which is internationally accepted.

3. The analysis methods, including confirmatory or reference methods needed in case of protest are, to the best possible, characterized by the following appropriate criteria:
 - a) accuracy
 - b) application field (analyte, matrix and concentration range);
 - c) detection level;
 - d) determination level;
 - e) repeatability;
 - f) reproducibility;
 - g) recuperation rate;
 - h) selectivity;
 - i) sensitivity;
 - j) linearity;
 - k) error margin and measurement uncertainty;
 - l) other criteria following need.

4. The competent authority is entitled to check the performance criteria, the analysis parameters, considerations related to uncertainty of the validation measures and procedures of the methods.

3.3.2. Specific criteria for EMT analysis

3.3.2.1. Definitions

The following definitions are applied:

- «**r**»= **repeatability:** value below which we can expect that the absolute difference between the results of individual test obtained in repeatability conditions (i.e. the same sample, the same operator, the same equipment, even the laboratory and a short period of time), is situated in a certain probability level (in principle 95%); Where $r = 2.8 \times S_r$.
- «**S_r**»= **Standard deviation:** calculated from results obtained in repeatability conditions.
- «**RSD_r**»= **Relative standard deviation:** calculated from results obtained in repeatability conditions.

$$RSD_r = (S_r / \bar{x}) \times 100$$

- «**R**»= **reproducibility:** value below which we can expect that the absolute difference between the results of individual tests obtained

inreproducibility conditions (i.e. for the same product executed by different operators in different laboratories using the method of standardized test) is situated in a certain probability level (in principle 95%); $R = 2.8 \times S_r$.

«**sR**»= **Standard deviation:** calculated from results obtained in repeatability conditions.

«**RSDR**»= **Relative standard deviation:** calculated from the results obtained in repeatability conditions.

$$RSD_R = (S_R/x) \times 100$$

«**LOD**»= **Detection level:** the lowest detected level (in analyte), from which we may deduce the presence of analyte with a reasonable statistical certainty. The detection level is numerically equal to three times the standard deviation of average blank tests ($n > 20$).

«**LOQ**»= **Quantification level:** the lowest level of analyte, measurable with a reasonable statistical certainty. If accuracy and precision are both at a concentration oscillating around the detection level, the quantification level is numerically equal to ten times the standard deviation of average blank test ($n > 20$).

«**HORRAT_r**»= The observed RSD_r divided by the estimated RSD_r from Horwitz equation (1) postulating that $r = 0,66R$.

«**HORRATR**»= The observed value of $RSDR$ divided by the $RSDR$ value, which is calculated from Horwitz equation.

«**u**»= Compound measurement uncertainty.

«**U**»= Widened measurement uncertainty.

The expanded uncertainty is obtained by multiplying the compound-type certainty by the coverage factor k , with $k=2$ for a confidence level of 95%.

$$U = 2 \times u$$

«**Uf**» = Maximum standard measurement uncertainty.

3.3.2.2. Performance Criteria for Analysis Methods

Any method used for the official control of contaminants in foodstuffs referred to in 3.3.1 of this Decree must comply with the specific performance criteria indicated in **Table 5**.

Table 5: Performance Criteria for Analytical Methods for Lead, Cadmium, Mercury and Inorganic Tin

Parameter	Value / comment
Applicability	Foodstuffs listed in this Order
LOD	For inorganic tin, less than 5 mg / kg hands. For the other elements, less than one tenth of the maximum limit set in this Order, unless the maximum lead content is less than 100µg/kg. In the latter case, less than one-fifth of the maximum limit.
LOQ	For inorganic tin, less than 10 mg / kg hands. For the other elements, less than one-fifth of the maximum content set in this Order, unless the maximum lead content is less than 100µg/kg. In the latter case, less than two fifths of the maximum limit.
Fidelity (repeatability and reproducibility)	HORRAT _r or HORRATR less than 2.
Recovery	The dispositions in points 4.1.2
Specificity	No interferences due to matrix or spectral.

3.3.2.3. Approach to Uncertainty Function

Where there are a limited number of fully validated methods of analysis, we can choose to adopt an approach based on the uncertainty function approach to assess the adequacy of the analytical method. Appropriate methods for official controls should produce results with an uncertainty measure less than the maximum standard uncertainty calculated using the following formula:

$$U_f = \sqrt{(\text{LOD} / 2)^2 + (\alpha C)^2}$$

in which :

U_f is the maximum standardized measurement uncertainty (µg/kg);

LOD is the detection limit of the method (µg/kg);

C is the concentration of interest (µg/kg);

α is a numerical factor dependent on the value of C.

The values to be used are given in **Table 8**.

Table 8: Numerical values corresponding to the constant α in the formula given under this point, as a function of the concentration of interest

C ($\mu\text{g}/\text{kg}$);	α
≤ 50	0.2
51-500	0.18
501-1000	0.15
1001-10000	0.12
>10000	0.1

4. REGISTRATION AND INTERPRETATION OF RESULTS

4.1. REGISTRATION

4.1.1. Expression of results

The results shall be expressed in the same units and with the same number of significant digits as the maximum limits set out in this Order.

We then obtain as expression of the final result:

$$\text{Result} = C \pm U \text{ with } (k=2)$$

4.1.2. Recovery rate calculations

If the analysis method includes an extraction phase, the analysis result is corrected for retrieval. In this case, the recovery rate must be mentioned.

If the analytical method does not include any extraction phase (for metals, for example), the result may be recorded uncorrected for recovery if it is established, ideally with a material The certified concentration taking into account the measurement uncertainty is reached (ie high accuracy of the measurement). It should be noted that the result is shown not corrected for recovery.

4.1.3. Uncertainty of measurement

The analytical result must be recorded using the formula $x \pm U$ in which x is the result of analysis and U the measurement uncertainty widened and by using an enlargement factor of 2 which gives a confidence level of About 95% ($U = 2u$).

The analyst shall take due account of the report on the relationship between analytical results, measurement uncertainty, recovery factors and provisions of Community legislation on food and feed.

4.2. RESULTS INTERPRETATION

4.2.1. Acceptance of a lot or sub-lot

The lot or sub-lot is accepted if the laboratory test result does not exceed the applicable maximum limit set by this Order, taking into account the increased measurement uncertainty and the correction of the result under the recovery when the method of analysis used comprises an extraction phase.

4.2.2. Rejection of a lot or sub-lot

The lot or sub-lot is refused if the laboratory test result clearly exceeds the applicable maximum limit set by this Order, taking into account the increased measurement uncertainty and the correction of the result under the recovery when the analytical method used comprises a phase of extraction.

ANNEX II.2

SAMPLING METHODS AND ANALYSIS FOR OFFICIAL CONTROL OF MYCOTOXIN LEVELS IN SOME FOODSTUFFS

1. DEFINITIONS:

Within the objectives of the following Annex, the following definitions apply:

“Lot”: a quantity of identifiable foodstuff, delivered at one time, and determined by the official to have common characteristics (such as origin, variety, packaging type, packaging, sender or marker).

“sub-lot”: the part of the lot with a large size selected for sampling. Each sub-lot shall be separate and identifiable;

“incremental sample”: a quantity of material taken at one single point of the lot or the sub-lot;

“aggregate sample”: All the incremental samples taken from the lot or sub-lot.

“laboratory sample”: a sample dedicated for the laboratory.

2. METHODS OF SAMPLING

2.1.GENERAL PROVISIONS

2.1.1 Personnel

Sampling is made by an authorized person in compliance with applicable regulations.

2.1.2 Material for sampling

Any lot or sub-lot, which is to be analyzed, shall be sampled separately. In compliance with specific sampling regulations applicable to different mycotoxins, the large lots are subdivided into sub-lots and sampled separately.

2.1.3 Precautions

In the course of sampling and preparation of the samples, precautions shall be taken to avoid any alteration that could impact:

- Mycotoxin contents, the analysis or make the aggregate sample unrepresentative
- Food security of the samples.

Furthermore, all necessary measures shall be taken to ensure the security of the agents performing the sampling.

2.1.4 Incremental samples

If possible, the samples are taken at different points distributed throughout the lot or sub-lot.

2.1.5 Preparation of the aggregate sample

The aggregate sample is made up by combining all the incremental samples.

2.1.6 Samples dedicated for control, defense or reference purposes

The samples dedicated for control, defense or reference purposes are taken from the homogenized aggregate sample.

2.1.7 Packaging and transmission of samples

Each sample is placed in a clean inert container, with adequate protection against contamination risks, the loss of substance by absorption through the internal wall of the container and possible damage in transit. All necessary precautions shall be taken to avoid any modification in the composition of the sample during transportation or storage.

2.1.8 Sealing and labeling the samples

Each sample shall be officially sealed at the place of sampling and identified in compliance with applicable regulations.

2.2 DIFFERENT TYPES OF LOTS

Foodstuffs may be traded in bulk, in containers or in individual packs, such as bags for retail sale. The sampling method may be used for all the different forms of product commercialization.

Without prejudice to the specific conditions stated in other parts of the present Annex, the following formula may be used as a guide for sampling lots traded in individual packs, such as bags for retail sale:

$$\text{Sampling frequency } n = \frac{\text{Weight of lot} \times \text{weight of incremental sample}}{\text{Weight of aggregate sample} \times \text{weight of retail sale unit}}$$

Weight: expressed in kilograms

Sampling frequency: number of individual packs between two samplings, each sampling is carried out after a given number of bags (decimal numbers are rounded to the nearest whole number)

2.3 Sampling method for cereals and cereal products

This method of sampling is used for official control of maximum levels of aflatoxins B1, total aflatoxins, ochratoxin A and Fusarium toxin in cereals and cereal products.

2.3.1. Weight of the incremental sample

The weight of the incremental sample is about 100 grams, unless differently specified in 2.3.

Concerning lots packaged for retail sale, the weight of the incremental sample depends on the weight of the retail sale unit.

For retail sale units weighing over 100 grams, the aggregate sample shall weigh over 10 kilograms.

If the weight of the retail sale unit highly exceeds 100grams, it is advised to take 100grams from each unit to form the incremental sample. This operation may be carried out at the place of sampling or in the laboratory. However, if this sampling method reveals commercially risky and unacceptable, due to the deterioration of the lot (due to packaging or to transit, etc.), an alternative sampling method may be used.

For example, if a high-value product is commercialized in retail sale units of 500 grams or 1 kg, we can have the aggregate sample by combining fewer incremental samples than indicated in **Tables 1 and 2**, provided its weight is the same as the required weight for the aggregate sample as indicated in the said tables.

If the retail sale unit weighs below 100 grams and if this difference is negligible, we consider that one unit equals one incremental sample, which gives an aggregate sample of less than 10 kilograms. If retail sale unit weighs a lot less than 100 grams, the incremental sample is made up of two units or more so that its nears as much as possible 100 grams.

2.3.2. General summary of the sampling method for cereals and cereal products

Table 1: Subdivision of the lots into sub-lots based on the product and the lot weight

Product	Lot weight (in tons)	Weight or number of sub- lots	Number of incremental samples	Weight of aggregate sample (in kilograms)
Cereals and cereal products	≥ 1500	500 tons	100	10
	>300 and <1500	3 sub-batches	100	10
	≥ 50 and \leq	100 tons	100	10
	<50	-	3 – 100(*)	1 - 10

(*) According to the weight of the lot – See Table 2.

2.3.3. Sampling method for cereals and cereal products for lots weighing 50 tons or more

- Provided the sub-lots can be physically separated, each lot is subdivided into sub-lots as per **Table 1**. Insofar as the weight of a lot is not always an exact multiple of the weight of the sub-lot, the weight of a sub-lot may exceed the indicated weight by up to 20%. If the lot is not or cannot be physically divided into sub-lots, a minimum of one hundred samples are taken from it.

- Each sub-lot shall be sampled separately.

- Number of incremental samples: one hundred. Weight of aggregate sample= 10 kg.

- If it is not possible to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), an alternative method, which is thoroughly described and documented, may be used insofar that it is as representative as possible. We may use an alternative sampling method in case it is materially impossible to use the method described above, for example, for cereals when they are stocked in silos.

2.3.4. Sampling method for cereals and cereal products for lots weighing below 50 tons

For lots of cereals and cereal products weighing below 50 tons, between ten and one hundred incremental samples are taken, according to the weight of the lot; this gives an aggregate sample of 1 to 10 kg. For smaller lots (≤ 0.50 ton) of cereals and cereal products, fewer incremental samples are needed, however, the weight of the aggregate sample, combining all the incremental samples, shall be at least 1 kg.

The figures in **Table 2** may be helpful to determine the number of incremental samples to take.

Table 2: Number of incremental samples to take according to the weight of the lot of cereals and cereal products

Lot weight (in tons)	Number of incremental samples	Weight of aggregate sample (in kg)
≤ 0.05	3	1
$> 0.05 \leq 0.5$	5	1
$> 0.5 \leq 1$	10	1
$> 1 \leq 3$	20	2
$> 3 \leq 10$	40	4
$> 10 \leq 20$	60	6
$> 20 \leq 50$	100	10

2.3.5. Sampling at retail sale level

Sampling foodstuffs at retail sale level is, if possible, realized in compliance with the provisions in paragraph 2.3

If this reveals impossible, an alternative sampling method may be used, which shall be thoroughly described and documented and shall ensure a representative aggregate sample. In any case, the aggregate sample shall be at least 1 kg (if the portion to sample is too small, it may weigh below 1 kg).

2.3.6. Acceptance of a lot or a sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.4. SAMPLING METHOD FOR DRIED FRUITS INCLUDING DRY RAISINS AND THEIR DERIVATIVES, YET EXCLUDING DRIED FIGS

This sampling method is used for official control of maximum levels in:

- Aflatoxin B1 and total aflatoxin in dried fruits, excepting dried figs.
- Ochratoxin A in dried raisins (Corinth dried raisins, “dried raisins” and sultanas).

2.4.1 Weight of the incremental sample

The weight of the incremental sample is about 100 grams, unless it is defined differently in paragraph 2.4.

For the lots packaged for retail sale, the weight of the incremental sample depends on the weight of the retail sale unit.

For retail sale units weighing over 100 grams, the weight of the aggregate sample shall be over 10 kg.

If the weight of the retail sale unit highly exceeds 100 grams, it is advised to take 100 grams from each unit to form the aggregate sample. This operation may be carried out at the place of sampling or in the laboratory. However, if this sampling method presents commercial risks due to the deterioration of the lot (packaging type, transport, etc.), it is possible to use an alternative sampling method. For example, if a high-value product is commercialized in retail sale units of 500 grams or 1 kg, the aggregate sample shall combine fewer incremental samples than indicated in **Tables 1 and 2**, on condition that its weight is the same as the required weight for the aggregate sample as indicated in the said tables.

If the retail sale unit weighs less than 100 grams and if this difference is negligible, we consider that one unit equals one incremental sample, which gives an aggregate sample of less than 10 kilograms. If retail sale unit weighs a lot less than 100 grams, the incremental sample shall include two units or more so that it nears as much as possible 100 grams

2.4.2 General summary of sampling method for dried fruits excepting dried figs

Table 1: Subdivision of the lots and sub-lots according to the product and weight of the lot

Product	Lot weight (in tons)	Weight or number of sub-lots	Number of incremental samples	Weight of aggregate sample (in Kg)
Dried fruits	≥ 15	15 – 30 tons	100	10
	<15	-	10 – 100 (*)	1 - 10

(*) according to the weight of the lot - See table 2 in this of present Annex.

2.4.3 Sampling method for dried fruits (lots ≥ 15 tons), excepting figs.

- Provided the sub-lots can be physically separated, each lot is subdivided into sub-lots as per **Table 1**. Insofar as the weight of a lot is not always an exact multiple of the weight of the sub-lots, the weight of a sub-lot may exceed the indicated weight by 20% maximum.
- Each sub-lot is sampled separately.
- Number of incremental samples: one hundred. Weight of aggregate sample= 10 kg.
- If it is not possible to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), an alternative method may be used, which shall be thoroughly described and documented, insofar as the aggregate sample is as representative as possible.

2.4.4 Sampling method for dried fruits (lots < 15 tons), excepting dried figs

For lots below 15 tons of dried fruits, excepting figs, sampling is realized using between 10 and 100 incremental samples according to the weight of the lot, so that an aggregate sample shall be 1 to 10 kg.

The figures in the table below may be helpful to determine the number of incremental samples.

Table 2: Number of incremental samples according to the weight of the lot of dried fruits

Lot weight (in tons)	Number of incremental samples	Weight of aggregate sample (in kg)
≤ 0.1	10	1
$> 0.1 \leq 0.2$	15	1.5
$> 0.2 \leq 0.5$	20	2
$> 0.5 \leq 1.0$	30	3
$> 1.0 \leq 2.0$	40	4
$> 2.0 \leq 5.0$	60	6
$> 5.0 \leq 10$	80	8
$> 10 \leq 15$	100	10

2.4.5 Sampling at retail sale level

Sampling foodstuffs at retail sale level is, if possible, realized in compliance with the provisions in paragraph 2.4.

If this reveals impossible, an alternative sampling method may be used, which shall be thoroughly described and documented and shall ensure a representative aggregate sample. In any case, the aggregate sample must weigh at least 1 kg (if the portion to sample is too small, it may weigh below 1 kg).

2.4.6 Specific rules for sampling dried fruits, excepting dried figs, sold vacuum-packed

For lots weighing 15 tons or more, at least 25 incremental samples are needed to make an aggregate sample of 10 kg and for lots below 15 tons, 25% of the weight of the incremental samples mentioned in **Table 2** are needed to have an aggregate sample as per the same table.

2.4.7 Acceptance of a lot or a sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.5 SAMPLING METHOD FOR DRIED FIGS

This method of sampling is used for official control of maximum levels of aflatoxins B1, total aflatoxins fixed in dried figs.

2.5.1 Weight of the incremental sample

The weight of the incremental sample is about 300 grams, unless stated differently in 2.5.

In the case of lots packaged for retail sale, the weight of the incremental sample depends on the weight of retail sale unit.

For retail sale units weighing over 300 grams, the weight of the aggregate sample shall be over 30 kg. If the unit weight highly exceeds 300 grams, it is advised to take 300grams from each unit to form the incremental sample. If this operation presents commercial risks due to the deterioration of the lot (packaging type, transport means, etc.), an alternative sampling method may be used. For example, if a high-value product is commercialized in retail sale units of 500 grams or 1 kg, we may have the aggregate sample by combining fewer elementary samples than indicated in **Tables 1, 2 and 3**, provided its weight is the same as the required weight for the aggregate sample as indicated in the said tables.

If the retail sale unit weighs less than 300 grams and if this difference is negligible, we consider that one unit equals one incremental sample, which gives an aggregate sample of less than 30 kilograms. If retail sale unit weighs a lot less than 300 grams, the incremental sample is made up of two units or more so that its weight nears as much as possible 300 grams.

2.5.2 General summary of sampling method for dried figs

Table 1: Subdivision of the lots and sub-lots according to the product and weight of the lot

Product	Lot weight (in tons)	Weight or number of sub-lots	Number of incremental samples	Weight of aggregate sample (in Kg)
Dried figs	≥ 15	15 – 30 tons	100	30
	<15	-	10 – 100 (*)	≤ 30

(*) according to the weight of the lot - See table 2 in part 5.

2.5.3 Sampling method for dried figs (lots of 15 tons)

- Insofar as the sub-lots can be physically separated, each lot is subdivided into sub-lots as per **Table 1**. Insofar as the weight of a lot is not always an exact multiple of the weight of the sub-lots, the weight of the sub-lots may exceed the indicated weight by a maximum of 20%.

- Each sub-lot is sampled separately.
- Number of incremental samples: one hundred.
- Weight of aggregate sample= 30 kg; roughly mixed, this latter must be divided into three equal laboratory samples of 10 kg each before being crushed (this operation of dividing into three laboratory samples is not necessary if the dried figs are destined for sorting out operation or any other physical processing and if there is an equipment for homogenizing 30 kg).

- Each laboratory sample of 10 kg is finely ground and carefully mixed for full homogenization as per part 2 of the present Annex.
- If it is impossible to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), an alternative method may be used, which shall be well described and documented, insofar as the aggregate sample is as representative as possible.

2.5.4 Sampling method for dried figs (lots < 15 tons)

The number of incremental samples, a minimum of 10 and a maximum of 100 depends on the weight of the lot.

The figures in **Table 2** below may be used to determine the number of incremental samples as well as the subsequent division of the aggregate sample.

Table 2: Number of incremental samples according to the lot weight and the number of subdivisions in the aggregate sample

Lot weight (in tons)	Number of incremental samples	Weight of aggregate sample (in kg) (for products packaged for retail sale, the weight of the aggregate sample may vary – see part 2.5.1)	Number of laboratory samples made from the aggregate sample
≤ 0.1	10	3	1 (no division)
>0.1 ≤ 0.2	15	4.5	1 (no division)
> 0.2 ≤ 0.5	20	6	1 (no division)
> 0,5 ≤ 1,0	30	9 (< 12kg)	1 (no division)
> 1.0 ≤ 2.0	40	12	2
> 2.0 ≤ 5.0	60	18 (< 24kg)	2
> 5.0 ≤ 10	80	24	3
> 10 ≤ 15	100	30	3

- Weight of aggregate sample= 30 kg; roughly mixed, this latter shall be divided into two or three equal laboratory samples of 10 kg each before being crushed (this operation of dividing into two or three laboratory samples is not necessary if the dried figs are destined for sorting out operation or any other physical processing and if there is an equipment for homogenizing 30 kg).

If it weighs below 30 kg, the aggregate sample is divided into laboratory samples according to the following instructions:

- < 12 kg: no division into laboratory samples
- ≥ 12 kg < 24 kg: division into two laboratory samples

- ≥ 24 kg: division into three laboratory samples
- If it is not practical to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), an alternative method may be used, which is fully described and well documented, insofar as the sample is as representative as possible.

2.5.5 Sampling method for derivatives and compound foodstuffs

2.5.5.1 Derived products with fine particles (homogeneous distribution of contamination by the aflatoxins)

Number of incremental samples: 100, for lots weighing below 50 tons, the number of incremental samples varies between ten and one hundred according to the weight of the lot (see Table 3).

Table 3: Number of incremental samples according to the weight of the lot

Lot weight (in tons)	Number of incremental samples	Weight of aggregate sample (in kg)
≤ 1	10	1
$> 1 \leq 3$	20	2
$> 3 \leq 10$	40	4
$> 10 \leq 20$	60	6
$> 20 \leq 50$	100	10

- The weight of the incremental sample shall be about 100 grams. If the lot is packaged for retail sale, the weight of the incremental sample depends on the weight of retail sale unit.
- The weight of the aggregate sample, carefully mixed up is between 1 and 10 kg.

2.5.5.2 Other derived products with relatively rough particles (homogeneous distribution of contamination by the aflatoxins)

The sampling method and the compliance criteria are the same as those used for dried figs (2.5.3 and 2.5.4)

2.5.6 Sampling at retail sale stage

Sampling foodstuffs at retail sale stage is realized, to the best possible, according to the provisions in this part.

If this reveals not practical, an alternative sampling method may be used, which is thoroughly described and documented and shall ensure a representative aggregate sample. In any case, the aggregate sample shall weigh at least 1 kg (if the portion to sample is too small, it may weigh below 1 kg).

2.5.7 Specific sampling method for dried figs and derivatives sold vacuum-packed

2.5.7.1 Dried figs

For lots weighing 15 tons or more, at least 50 incremental samples are needed to form the aggregate sample of 30 kg and for lots weighing below 15 tons, 50% of the weight of the incremental samples mentioned in **Table 2** are needed to have an aggregate sample as per the same table.

2.5.7.2 Products with fine particles derived from dried figs

For lots weighing 15 tons or more, at least 25 incremental samples are needed to form the aggregate sample of 10 kg and for lots weighing below 50 tons, 25% of the weight of the incremental samples mentioned in **Table 3** are needed to form the aggregate sample as per the same table.

2.5.8 Acceptance of the lot or sub-lot

Dried figs to be processed by sorting out or any other physical process:

- Acceptance if the aggregate sample or the average laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the aggregate sample or the average laboratory sample exceeds maximum levels beyond reasonable doubt, taking into consideration the correction for recovery and the measurement uncertainty.

Dried figs dedicated for direct human consumption:

- Acceptance if no one of the laboratory samples exceeds maximum levels, taking into consideration the correction according to correction for recovery and the measurement uncertainty.
- Rejection if one or more of the laboratory samples exceed beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

In case the aggregate sample weighs 12 kg or less:

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measure incertitude.

2.6. SAMPLING METHOD FOR GROUNDNUTS, THE OTHER OLEAGINOUS GRAINS, APRICOT STONES AND SHELL FRUITS

This sampling method is used for official control of maximum levels of Aflatoxins B1 and total Aflatoxins fixed in groundnuts, the other oleaginous grains, apricot stones and shell fruits

2.6.1 Weight of the incremental sample

The weight of the incremental sample is about 200 grams, unless stated differently in 2.6.

In the case of lots packaged for retail sale, the weight of the incremental sample depends on the weight of retail sale unit.

For retail sale units weighing over 200 grams, the weight of the aggregate sample shall be over 20 kg. If the unit weight highly exceeds 200 grams, it is advised to take 200grams from each unit to constitute the incremental sample. This operation may be realized at the place of sampling or in the laboratory. If this presents commercial risks due to the deterioration of the lot (packaging type, transport means, etc.), it is possible to use another sampling method. For example, if a high-value product is sold in units of 500 grams or 1 kg, we can have the aggregate sample by combining less incremental samples than indicated in **Tables 1, 2 and 3**, insofar as the aggregate sample has the required weight as indicated in the said tables.

If the retail sale unit weighs less than 200 grams and if this difference is negligible, we consider that one unit equals one incremental sample, which gives an aggregate sample of less than 20 kilograms. If retail sale unit weighs a lot less than 200 grams, the incremental sample is made up of two units or more so that its weight nears as much as possible 200 grams.

2.6.2 General summary of the sampling method for groundnuts, the other oleaginous grains, apricot stones and shell fruits

Table 1: Division of the lot or sub-lot according to the lot weight

Product	Lot weight (in tons)	Weight or number of sub-lots	Number of incremental samples	Weight of aggregate sample (in Kg)
Groundnuts, other oleaginous grains, apricot stones and shell fruits	500	100 tons	100	20
	>125 and <500	5 sub-batches	100	20
	15 and 125	25 tons	100	20
	<15	-	10 – 100 (*)	20

(*) According to the weight of the lot – See Table 2 in part 2.6 of the present Annex

2.6.3 Sampling method for groundnuts, the other oleaginous grains, apricot stones and shell fruits (lots \geq 15 tons)

- Insofar as the sub-lots can be physically separated, each lot is subdivided into sub-lots as per **Table 1**. Insofar as the weight of a lot is not always an exact multiple of the weight of the sub-lots, the weight of a sub-lot may exceed the indicated weight by 20% maximum.
- Each sub-lot shall be sampled separately.
- Number of incremental samples: one hundred.
- Weight of aggregate sample= 20 kg; roughly mixed, this latter must be divided into two equal laboratory samples of 10 kg each before being crushed (this operation of dividing into two laboratory samples is not necessary if the groundnuts, other oleaginous grains, apricot stones and shell fruits are destined for sorting out operation or any other physical processing and if there is an equipment for homogenizing 20 kg).
- Each laboratory sample of 10 kg is finely ground and carefully mixed for full homogenization as per part 3 of the present Annex.
- If it is not practical to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), an alternative method may be used provided it is as representative as possible and it should be fully described and well documented.

2.6.4. Sampling method for groundnuts, the other oleaginous grains, apricot stones and shell fruits (lots < 15 tons)

The number of incremental samples, a minimum of 10 and a maximum of 100 depends on the weight of the lot.

The figures in **Table 2** below may be used to determine the number of incremental samples as well as the subsequent division of the aggregate sample.

Table 2: Number of incremental samples according to the lot weight and the number of subdivisions of the aggregate sample

Lot weight (in tons)	Number of incremental samples	Weight of aggregate sample (in kg) (for products packaged for retail sale, the weight of the aggregate sample may vary – see part 2.6.1)	Number of laboratory samples made up from the aggregate sample
≤ 0.1	10	2	1 (no division)
> 0.1 ≤ 0.2	15	3	1 (no division)
> 0.2 ≤ 0.5	20	4	1 (no division)
> 0.5 ≤ 1.0	30	6	1 (no division)
> 1.0 ≤ 2.0	40	8 (< 12 kg)	1 (no division)
> 2.0 ≤ 5.0	60	12	2
> 5.0 ≤ 10	80	16	2
> 10 ≤ 15	100	20	2

- Weight of aggregate sample ≤ 20 kg; roughly mixed, this latter must be divided into two equal laboratory samples of up to 10 kg each before being crushed (this operation of dividing into two laboratory samples is not necessary if the groundnuts, the other oleaginous grains, apricot stones and shell fruits are destined for sorting out operation or any other physical processing and if there is an equipment for homogenizing 20 kg).

If it weighs below 20 kg, the aggregate sample is divided into laboratory samples according to the following instructions:

- < 12 kg: no division into laboratory samples
- ≥ 12 kg: division into two laboratory samples
- Each laboratory sample shall be finely ground and carefully mixed for full homogenization as per part 3 of the present Annex.
- If it is not practical to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), we may use another method provided it is as representative as possible and it should be fully described and well documented.

2.6.5 Sampling method for derivatives, excepting vegetal oils and compound foodstuffs

2.6.5.1 Derived products (other than vegetal oil) with fine particles, such as groundnut flour or pastry (homogeneous distribution of contamination by the aflatoxins)

Number of samples: one hundred; for lots weighing below 50 tons, the number of incremental samples varies between ten and one hundred according to the lot weight (see Table 3).

Table 3: Number of incremental samples according to the lot weight

Lot weight (in tons)	Number of incremental samples	Weight of aggregate sample (in kg)
≤ 1	10	1
$> 1 \leq 3$	20	2
$> 3 \leq 10$	40	4
$> 10 \leq 20$	60	6
$> 20 \leq 50$	100	10

- The weight of the incremental sample must be about 100 grams. If the lot is packaged for retail sale, the weight of the incremental sample depends on the weight of retail sale unit.
- The weight of the aggregate sample, carefully mixed is between 1 and 10 kg.

2.6.5.2 Derived products with relatively rough particles (homogeneous distribution of contamination by the aflatoxins)

Sampling method and compliance criteria are the same as those used for groundnuts, the other oleaginous grains, apricot stones and shell fruits (2.6.3 and 2.6.4)

2.6.6 Sampling at retail sale stage

Sampling foodstuffs at retail stage is, to the best possible, carried out in compliance with the provisions mentioned in the present part 2.6.

If this reveals not practical, an alternative sampling method may be used, which is thoroughly described and documented and shall ensure a representative aggregate sample. In any case, the aggregate sample shall weigh at least 1 kg (if the portion to sample is too small, it may weigh below 1 kg).

2.6.7 Specific sampling method for groundnuts, the other oleaginous grains, apricot stones and shell fruits and the derived products sold vacuum-packed

2.6.7.1 Pistachio, groundnut, Brazil nut

For lots weighing 15 tons or more, at least fifty incremental samples are needed to form an aggregate sample of 20 kg and for lots weighing below 15 tons, 50% of the number of incremental samples mentioned in **Table 2** are needed to have an aggregate sample as per the same table.

2.6.7.2 Apricot stones, shell fruits other than pistachio and Brazil nut, other oleaginous grains

For lots weighing 15 tons or more, at least twenty-five incremental samples are needed to form an aggregate sample of 20 kg and for lots weighing below 15 tons, 25% of the number of

incremental samples mentioned in **Table 2** are needed to have aggregate sample as per the same table.

2.6.7.3 Fine particle products derived from shell fruits, apricot stones and groundnut

For lots weighing 50 tons or more, at least twenty-five incremental samples are needed to form an aggregate sample of 20 kg and for lots weighing below 50 tons, 25% of the number of incremental samples mentioned in **Table 3** are needed to have an aggregate sample as per the same table.

2.6.8 Acceptance of the lot or sub-lot

For groundnuts, the other oleaginous grains, apricot stones and shell fruits to be sorted out or processed through any other physical process:

- Acceptance if the aggregate sample or the average laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the aggregate sample or the average laboratory sample clearly exceeds maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

For groundnuts, the other oleaginous grains, apricot stones and shell fruits dedicated for direct human consumption:

- Acceptance if no one of the laboratory samples exceeds maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if one or both laboratory samples exceed beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

In case the aggregate sample weighs 12 kg or less:

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.7 SAMPLING METHOD FOR SPICES

This sampling method is to be used for official control of maximum levels of Ochratoxin, Aflatoxin B1 and total Aflatoxins, laid down for spices

2.7.1 Weight of the incremental sample

The weight of the incremental sample is about 100 grams, unless stated differently in the present part 2.7.

In the case of lots packaged for retail sale, the weight of the incremental sample depends on the weight of the retail sale unit.

For retail sale units weighing over 100 grams, the weight of the aggregate sample shall be over 10 kg. If the unit weight highly exceeds 100 grams, it is advised to take 100 grams from each unit to constitute the incremental sample. This operation may be realized at the place of sampling or in the laboratory. If this presents commercial risks due to the deterioration of the lot (packaging type, transport means, etc.), it is possible to use another sampling method. For example, if a high-value product is sold in retail sale units of 500 grams or 1 kg, we may form the aggregate sample by combining fewer incremental samples than indicated in **Tables 1 and 2**, provided its weight is the same as the required weight for the aggregate sample as indicated in the said tables.

If the retail sale unit weighs less than 100 grams and if this difference is negligible, we consider that one unit equals one incremental sample, which gives an aggregate sample of less than 10 kilograms. If retail sale unit weighs a lot less than 100 grams, the incremental sample is made up of two units or more so that its weight nears as much as possible 100 grams.

2.7.2 General summary of sampling method for spices

Table 1: Division of the lot into sub-lots according to the lot weight

Product	Lot weight (in tons)	Weight or number of sub-lots	Number of incremental samples	Weight of aggregate sample (in Kg)
Spices	≥ 15	-25 tons	100	10
	< 15	-	5 – 100 (*)	0,5 - 10

(*) According to the weight of the lot – See Table 2 in this part 2.7

2.7.3 Sampling method for spices (≥ 15 tons)

- Insofar as the sub-lots can be physically separated, each lot is subdivided into sub-lots as per **Table 1**. Insofar as the weight of a lot is not always an exact multiple of the weight of the sub-lots, the weight of a sub-lot may exceed the indicated weight by 20% maximum.
- Each sub-lot shall be sampled separately.
- Number of incremental samples: one hundred.

- Weight of aggregate sample= 10 kg.
- If it is not practical to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), an alternative sampling method may be used, which is fully described and well documented and shall be as representative as possible.

2.7.4 Sampling method for spices (< 15 tons)

For spice lots below 15 tons, the sampling plan is realized with a number of incremental samples between 5 and 100 according to the weight of the lot; this gives an aggregate sample of 0,5 to 10 kg.

The figures in the table below may be helpful to determine the number of incremental samples.

Table 2: Number of incremental samples according to the weight of spice lots

Weight of lot (in tons)	Number of incremental samples	Weight of aggregate sample (in kg)
< 0,01	5	0.5
> 0.01 ≤ 0.1	10	1.0
> 0.1 ≤ 0.2	15	1.5
> 0.2 ≤ 0.5	20	2
> 0.5 ≤ 1.0	30	3
> 1.0 ≤ 2.0	40	4
> 2.0 ≤ 5.0	60	6
> 5.0 ≤ 10.0	80	8
> 10 ≤ 15	100	10

2.7.5 Sampling at retail sale stage

Sampling foodstuffs at retail sale stage is realized, to the best possible, according to the provisions in part 2.7.

If this reveals unpractical, an alternative sampling method may be used, which is thoroughly described and documented and shall ensure a representative aggregate sample. In any case, the aggregate sample shall weigh at least 0.5 kg (if the portion to sample is too small, it may weigh below 0.5 kg).

2.7.6 Specific sampling method for vacuum-packed spices

For lots weighing 15 tons or more, at least twenty-five incremental samples are needed to form an aggregate sample of 10 kg and for lots weighing below 15 tons, 25% of the number of incremental samples mentioned in **Table 2** are needed to have an aggregate sample as per the same table.

2.7.7 Acceptance of a lot or sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.8 SAMPLING METHOD FOR MILK AND DAIRY PRODUCTS, FORMULAE FOR INFANTS AND FOLLOW ON FORMULAE, INCLUDING MILK FOR INFANTS AND FOLLOW ON MILK

This sampling method is to be used in official control of maximum levels of Aflatoxin M1 laid down for milk and dairy products, formulae for infants and follow on formulae, including milk for infants and follow on milk, follow on milk and dietetic products for infants (milk and dairy products) dedicated for specific medical purposes.

2.8.1 Sampling method for milk and dairy products, formulae for infants and follow on formulae, including milk for infants and follow on milk

The aggregate sample shall weigh at least 1 kg or 1 liter, unless unpractical, for example when the sample includes just one bottle.

The minimum number of incremental samples taken in the lot is indicated in Table 1. The number of incremental samples depends on the usual form under which the product is traded. If it concerns liquid products traded in bulk, to the best possible and without damaging the quality of the product, the lot is carefully mixed, either manually or by means of a mechanical process, prior to sampling. As the distribution of Aflatoxin M1 in a given lot is assumed to be homogeneous, we just need to take three incremental samples by lot to constitute the aggregate sample.

All the incremental samples, probably often bottles or bricks have a similar weight. Each incremental sample weighs at least 100 grams so that the aggregate sample should reach 1 kg or 1 liter.

Table 1: Minimum number of incremental samples to take from the lot

Form of commercialization	Volume or weight of the lot (in liter or kg)	Minimum number of samples	Minimum volume or weight of the aggregate sample (in liter or kg)
In bulk	-	3 – 5	1
Bottle/brick	≤ 50	3	1
Bottle/brick	From 50 to 500	5	1
Bottle/brick	> 500	10	1

2.8.2 Sampling at retail sale stage

Sampling foodstuffs at retail sale stage is, to the best possible, realized in compliance with the provisions in the present part 2.8.

If this reveals unpractical, an alternative sampling method may be used, which should be thoroughly described and documented and shall ensure a sufficiently representative aggregate sample.

2.8.3 Acceptance of a lot or sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty (or the decision level – see part 3 of the present Annex).

- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty (or the decision level – see part 3 of the present Annex).

2.9 SAMPLING METHOD FOR COFFEE, COFFEE PRODUCTS, LIQUORICE WOOD AND LIQUORICE EXTRACT

This sampling method is to be used in official control of maximum levels of Ochratoxin A in roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract.

2.9.1 Weight of the incremental sample

The weight of the incremental sample is about 100 grams, unless defined differently in the present part 2.9.

For lots packaged for retail sale, the sample weight depends on the weight of retail sale unit.

For retail sale units weighing over 100 grams, the aggregate sample shall weigh over 10 kg. If the weight of retail sale units highly exceeds 100 grams, it is advised to take 100 grams from each unit to constitute the incremental sample.

This operation may be carried out at the place of sampling or in the laboratory. However, if this presents commercial risks due to the deterioration of the lot (packaging, transport, etc.), another alternative sampling method may be used. For example, if a high-value product is sold in retail sale units of 500 grams or 1 kg, the aggregate sample may be formed by combining fewer incremental samples than indicated in **Tables 1 and 2**, insofar as its weight is the same as the required weight for the aggregate sample as indicated in the said tables.

If the retail sale unit weighs less than 100 grams and if this difference is negligible, we consider that one unit equals one incremental sample, which gives an aggregate sample of less than 10

kilograms. If retail sale unit weighs a lot less than 100 grams, the incremental sample is made up of two units or more so that its weight nears as much as possible 100 grams.

2.9.2 General summary of sampling method for roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract

Table 1: Subdivision of lots into sub-lots according to the product and the lot weight

Product	Lot weight (in tons)	Weight or number of sub-lots	Number of incremental samples	Weight of aggregate sample (in kg)
roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract	≥ 15	15 – 30 tons	100	10
	< 15		10 to 100 (*)	1 to 10

(*) according to the weight of the lot – See Table 2 of the present part 2.9

2.9.3 Sampling method for roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract (lots ≥ 15)

- Insofar as the sub-lots can be physically separated, each lot is subdivided into sub-lots as per **Table 1**. Insofar as the weight of a lot is not always an exact multiple of the weight of the sub-lots, the weight of a sub-lot may exceed the indicated weight by 20% maximum.
- Each sub-lot shall be sampled separately.
- Number of incremental samples: one hundred.
- Weight of aggregate sample= 10 kg.
- If it is not practical to use the sampling method described in this paragraph due to possible deterioration of the lot (because of packaging or transport, etc.), an alternative sampling method may be used, which is thoroughly described and documented, and shall ensure an aggregate sample as representative as possible.

2.9.4 Sampling method for roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract (lot < 15 tons)

For the lots of roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract weighing less than 15 tons, the sampling plan is realized with a number of incremental samples between ten and one hundred according to the weight of the lot; this gives an aggregate sample of 1 to 10 kg.

The figures in the table below may be helpful to determine the number of incremental samples.

Table 2: Number of incremental samples according to the weight of the lot of roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract (lot < 15 tons)

Lot Weight (in tons)	Number of incremental samples	Weight of aggregate sample (in kg)
< 0.1	10	1
> 0.1 ≤ 0.2	55	1.5
> 0.2 ≤ 0.5	20	2
> 0.5 ≤ 1.0	30	3
> 1.0 ≤ 2.0	40	4
> 2.0 ≤ 5.0	60	6
> 5.0 ≤ 10.0	80	8
> 10.0 ≤ 15.0	100	10

2.9.5 Sampling method for roasted coffee grains, roasted ground coffee, instant coffee, liquorice wood and liquorice extract vacuum-packed

For lots weighing 15 tons or more, at least twenty-five incremental samples are needed to form an aggregate sample of 10 kg and for lots weighing below 15 tons, 25% of the number of incremental samples mentioned in **Table 2** are needed to have an aggregate sample as per the same table.

2.9.6 Sampling at retail sale stage

Sampling foodstuffs at retail sale level is realized, to the best possible, according to the provisions in this part 2.9.

If this reveals unpractical, an alternative sampling method may be used, which is thoroughly described and documented and shall ensure a representative aggregate sample. In any case, the aggregate sample shall weigh at least 1 kg (if the portion to sample is too small, it may weigh below 1 kg).

2.9.7 Acceptance of a lot or sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.10 SAMPLING METHOD FOR FRUIT JUICE, INCLUDING RAISIN MUST OR JUICE, CIDER AND WINE

This sampling method is to be used in official control of maximum levels of:

- Ochratoxin A in wine, raisin must and juice
- Patulin in fruit juice, nectars, liquors, cider and other fermented apple products or products containing apple juice.

2.10.1 Sampling method and samples

The aggregate sample is at least 1 liter, for example when the sample includes one single bottle.

The minimum number of samples to realize is indicated in **Table 1**.

The minimum number of incremental samples depends on the usual form under which the product is traded. If it concerns liquid products in bulk, to the best possible and without damaging the quality of the product, the lot is carefully mixed, either manually or by means of a mechanical process prior to sampling. Insofar as the distribution of Ochratoxin A and Patulin in a given lot is assumed to be homogeneous, just three incremental samples are needed to constitute the aggregate sample.

All the incremental samples, which shall probably be bottles or bricks, have a similar weight. Each incremental sample weighs at least 100 grams so that the aggregate sample shall reach 1 liter.

Table 1: Minimum number of samples to take from the lots

Form of commercialization	Volume of the lot (in liter)	Minimum number of incremental samples to realize	Aggregate sample minimum volume (in liter)
Bulk (fruit juice, cider, wine)	-	3	1
Bottles/bricks (fruit juice, liquors, cider)	<50	3	1
Bottles/bricks (fruit juice, liquors, cider)	From 50 to 500	5	1
Bottles/bricks (fruit juice, liquors, cider)	>500	10	1
Bottles/bricks wine	≤50	1	1
Bottles/bricks wine	From 50 to 500	2	1
Bottles/bricks wine	>500	3	1

2.10.2 Sampling at retail sale level

Sampling foodstuffs at retail sale level is, to the best possible, realized in compliance with the provisions in this part (if the portion to sample is too small to have a global sample of 1 liter, the volume of this latter may be below the liter.

If this reveals impossible, an alternative sampling method may be used, it shall be thoroughly described and documented and shall ensures a sufficiently representative aggregate sample.

2.10.3 Acceptance of the lot or the sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.11. SAMPLING METHOD FOR SOLID APPLE PRODUCTS, INCLUDING THOSE DEDICATED FOR INFANTS AND YOUNG CHILDREN AND APPLE JUICES

This sampling method is to be used for official control of maximum levels of Patulin laid down for solid apple products, including those dedicated for infants and young children and apple juices.

2.11.1 Sampling method

The aggregate sample must weigh at least 1 kg, unless impossible, for example when sampling one single conditioned unit.

The minimum number of incremental samples is to be realized from a lot is determined in **Table 1**. If this concerns liquid products, the lot is, to the best possible, mixed either manually or by means of a mechanical process, just before sampling. Insofar as the distribution of Patulin in a given lot is assumed to be homogeneous, then three incremental samples are taken from each lot to constitute the aggregate sample.

All the incremental samples have a similar weight. Each incremental sample weighs at least 100 grams so that the aggregate sample shall be 1 kg.

Table 1: Minimum number of samples to realize from the lot

Lot weight (in kg)	Number of incremental samples	Weight of aggregate sample (in kg)
< 50	3	1
From 50 to 500	5	1
> 500	10	1

If the lot is made of distinct packaged units, the number of units needed to constitute the aggregate sample is indicated in **Table 2**

Table 2: number of units (incremental samples) needed to constitute the aggregate sample if the lot is made of distinct units.

Number of packaged units in the lot	Number of conditioned units to take	Weight of aggregate sample (in kg)
From 1 to 25	1 unit	1
From 26 to 100	About 5%, 2 units at least	1
> 100	About 5%, 10 units maximum	1

2.11.2 Sampling at retail sale stage

Sampling foodstuffs at retail sale stage is realized, to the best possible, in compliance with the provision in the present part.

If this reveals impossible, another sampling method can be used, which is thoroughly described and documented and shall ensure a representative aggregate sample. In any case, the aggregate sample must weigh at least 1 kg (if the portion to sample is too small, it may weigh below 1 kg).

2.11.3 Acceptance of the lot or sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.12. SAMPLING METHOD FOR FOODS FOR INFANTS AND PROCESSED CEREAL FOODS DEDICATED FOR INFANTS AND YOUNG CHILDREN

This sampling method is to be used in official control of maximum levels related to:

- Aflatoxins, Ochratoxins A and Fusarium Toxines in foods for infants and processed cereal foods dedicated for infants and young children.
- Aflatoxins, Ochratoxins A in dietetic foods for infants (other than milk and dairy products) dedicated for special medical purposes.
- Patulin in foods for infants, other than processed cereal foods, dedicated for infants and young children. For the control of maximum levels of Patulin in fruit juices and solid apple products dedicated for infants and young children, it is advised to use the sampling method described in 2.11 of the present Annex.

2.12.1 Sampling method

- The sampling method for cereals and cereal products described in paragraph 2.3.4 of the present Annex is to be used for foodstuffs dedicated for infants and young children.

Consequently, the number of incremental samples to take depends on the weight of the lot and is between ten and one hundred, in compliance with the rules provided for in Table 2, paragraph 2.3.4 of the present Annex.

For very small lots (≤ 0.5 tons), fewer incremental samples may be taken, however, the aggregate sample combining all the incremental samples must weigh at least 1 kg.

- The weight of the incremental sample is about 100 grams. In the case of lots packaged for retail sale, the weight of the incremental sample depends on the weight of the retail sale unit, and, in the case of very small lots (≤ 0.5 tons), this weight is such that when all the incremental samples are added, we obtain an aggregate sample of at least 1 kg.
- The weight of the aggregate sample, adequately mixed, is between 1 to 10 kg.

2.12.2 Sampling method at retail sale stage

Sampling foodstuffs at retail sale stage is realized, to the best possible, in compliance with the rules mentioned in this part 2.12.

If this reveals impossible, an alternative sampling method may be used, which is thoroughly described and documented and shall ensure a representative aggregate sample (if the portion to sample is too small, it may weigh below 1 kg).

2.12.3 Acceptance of a lot or sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

2.13. SAMPLING METHOD FOR VEGETAL OILS

This sampling method is to be used for official control of maximum levels of mycotoxins, mainly aflatoxins B1, total aflatoxins and zearalenone, laid down for vegetal oils.

2.13.1 Sampling method for vegetal oils

- Each incremental sample must weigh about 100 grams (ml) minimum (according to the nature of the lot, for example for vegetal oil in bulk, at least three incremental samples of about 350 ml minimum are necessary), to form an aggregate sample of 1 kg (1 liter).

The minimum number of incremental samples to take is indicated in Table 1. The lot is carefully mixed, either manually or by a technical process, prior to sampling. Insofar as we assume a homogeneous distribution of aflatoxins within a given lot; then, we just need to take three incremental samples by lot to constitute the aggregate sample.

Table 1: Minimum number of incremental samples to take by lot

Commercialization form	Lot weight (in kg) volume of lot (in liter)	Number of incremental samples to take
In bulk (*)	-	3
Bricks	≤ 50	3
Bricks	> 50 to 500	5
Bricks	> 500	10

(*) provided the sub-lot can be physically separated, the large lots/bulk of vegetable oil is subdivided into sub-lots as per Table 2.

Table 2: Subdividing lots into sub-lots according to the lot weight

Product	Lot weigh (in tons)	Weight or number of sub-lots	Number of incremental samples	Minimum weight of aggregate sample (in kg)
Vegetal oils	≥ 1500	500 tons	3	1
	> 300 and < 1500	3 sub-batch	3	1
	≥ 50 and ≤ 300	100 tons	3	1
	< 50	-	3	1

2.13.2 Sampling method for vegetal oils at retail sale level

Sampling foodstuffs at retail sale level is realized, to the best possible, in compliance with the provisions mentioned in this part 2.13.

If this reveals impossible, an alternative sampling method may be used, which is thoroughly described and documented and shall ensure a representative aggregate sample. Aggregate sample must weigh 1 kg at least (if the portion to sample is too small, it may weigh below 1 kg).

2.13.3 Acceptance of the lot or sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.
- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

3. PREPARATION AND ANALYSIS OF THE SAMPLES

3.1 Quality standard applicable to the laboratory

Laboratories shall comply with the following:

3.1.1 Material requirements

- a) Staff realizing the analyses for official control must be impartial without any interest conflict relating to the tasks assigned to them.
- b) The laboratories must have the required capacity to carry out the analyses as well as duly qualified and experienced personnel in sufficient number so that they can execute their analyses and tasks efficiently and effectively
- c) The laboratories must have adequate installations and equipment with appropriate maintenance to allow personnel to carry out their tasks efficiently and effectively.

3.1.2 Moral requirements: transparency and confidentiality

- a) Laboratories are keen to execute their activities at a high level of transparency. For this end, they must make available to competent authorities any pertinent information they hold as quick as possible.
- b) Laboratories must take the necessary measure so that their personnel should not reveal any information they obtain during their activities within official control; this information is, by nature, covered by the professional secret

3.2 PREPARATION OF THE SAMPLE

3.2.1 Processing the sample received by the laboratory

The full aggregate sample is finely crushed (if necessary) and carefully mixed by means of a method that ensures complete homogenization.

Samples must be processed and labeled in such a way that their analytic and legal validity should be guaranteed.

If maximum level applies to the dry substance, the level of the product in dry substance is determined on a part of the homogenized sample by means of a precise and secure method.

3.2.2 Samples dedicated for control, defense or reference purposes

Samples dedicated for control, defense or reference purposes are taken from the homogenized substance.

3.2.3 Preparation of the sample

The aim is to have a laboratory sample that is representative and homogeneous without any introduced external contamination.

All the samples received by the laboratory shall be used for the preparation of the laboratory sample.

Compliance with maximum levels fixed in the present decision is based on the levels determined in laboratory samples.

3.2.1 Specific procedure for the preparation of samples to analyze mycotoxin

Mycotoxins are generally distributed in a heterogeneous way; samples are prepared and often homogenized with greatest care.

When homogenization is realized in the laboratory, all the samples received shall be homogenized.

During aflatoxin analysis daylight should be avoided as much as possible, because aflatoxin disintegrates progressively through ultraviolet light.

3.2.2 Calculation of the proportion shell/almond for whole shell fruits

The levels laid down for aflatoxins in the present decision are applicable to the edible part. The level of aflatoxin in the edible part may be determined as follows:

- The samples of shell fruits may be shelled to determine the level of aflatoxin in the edible part.
- The procedure to prepare the sample may be applied to the fruit in its shell. The sampling mode and the analysis method shall include an estimation of the weight of almonds in the aggregate sample. This latter may be estimated by setting an appropriate factor characterizing the proportion between the shell and the almond in the whole fruits. This proportion is used to determine the quantity of almonds in the aggregate sample used for the preparation of the method to analyze the sample.

About one hundred shelled fruits are taken at random from the lot or in the aggregate sample and set apart. For each laboratory sample, we may have the researched ratio by weighing the whole fruits, then shelling them and weighing the almond shells.

Once the proportion between the shell and the almond is determined from a certain number of samples, it may be taken into consideration in subsequent analyses. However, if a laboratory sample exceeds the fixed level, the proportion is determined by means of the hundred fruits set apart.

3.2.4 Replicate samples

The replicate samples are taken from the homogenized products.

3.3. METHOD TO BE USED BY THE LABORATORY AND CONTROL REQUIREMENTS

3.3.1 General requirements

1 - Analysis methods used for official control are standardized and compliant methods or:

- a) If such standards do not exist, to internationally acknowledged protocols or those adopted in national legislation, or
- b) In the absence of this, to the appropriate methods for this end, or elaborated according to scientific protocols.

2 – If paragraph 1 is not applicable, analysis methods may be validated within the laboratory following a protocol, which is internationally accepted (International, European or National standards).

3 – The analysis methods, including confirmatory or reference methods needed in case of protest are, to the best possible, characterized by the following appropriate criteria:

- a) Accuracy
- b) Application field (analyte, matrix and concentration range)
- c) Detection level
- d) Determination level
- e) Repeatability
- f) Reproducibility
- g) Recuperation rate
- h) Selectivity
- i) Precision
- j) Linearity
- k) Error margin and measurement uncertainty
- l) Other criteria following need

4 The competent authority is entitled to check the performance criteria, the analysis parameters, considerations related to uncertainty of the validation measures and procedures of the methods.

3.3.1 Definition

3.3.2 Specific criteria for EMT analysis

3.3.2.1 Definition

The following definitions are applied:

“r”: **repeatability:** value below which we can expect that the absolute difference between the results of individual tests obtained in repeatability conditions (i.e. same sample, same operator, same equipment, same laboratory and a short period of time) is situated in a certain probability level (in principle 95%); so $r = 2,8 \times S_r$.

“S_r”: **Standard deviation** calculated from results obtained in repeatability conditions.

“RSD_r”: **Relative standard deviation** calculated from results obtained in repeatability conditions

$$RSD_r = (S_r/x) \times 100$$

“R”: **Reproducibility:** value below which we can expect that the absolute difference between the results of individual tests obtained in reproducibility conditions (i.e. for the same product executed by different operators in different laboratories using the method of standardized test), is situated in a certain probability level (in principle 95%); $R = 2,8 \times S_r$.

“s_r”: **standard deviation** calculated from results obtained in reproducibility conditions.

“RSD_R”: **Relative standard deviation,** calculated from results obtained in reproducibility conditions

$$RSD_R = (S_R/x) \times 100$$

“LOD”: **Detection level:** the lowest detected level (in analyte), from which we may deduce the presence of analyte with a reasonable statistical certainty. The detection level is numerically equal to three times the standard deviation of average blank test ($n > 20$).

“LOQ”: **Quantification level:** the lowest level of analyte, measurable with a reasonable statistical certainty. If accuracy and precision are both at a concentration oscillating around the detection level, the quantification level is numerically equal to ten times the standard deviation of average blank test ($n > 20$).

“HORRAT_r”: The observed RSD_r divided by the estimated RSD_r from HORWITZ equation (1) postulating that $r = 0,66R$.

“HORRAT_R”: The observed value of RSD_R divided by the RSD_R value, which is calculated from HORWITZ equation.

“u”: Compound measurement uncertainty

“U”: Widened measurement uncertainty

The expanded uncertainty is obtained by multiplying the compound-type certainty by the coverage factor k , with $k=2$ for a confidence level of 95%.

$$U = 2 \times u$$

“Uf”: Maximum standard measurement uncertainty.

4.3.1 Performance criteria of the analysis methods

Insofar as local legislation imposes no specific method for the determination of mycotoxin levels in foodstuffs, laboratories are free to choose the method they prefer, insofar as it complies with the following criteria:

a) Performance criteria for aflatoxins

Criterion	Concentration range	Recommended level	Maximum level allowed
Blank levels	All	Negligible	-
Recovery – Aflatoxin M1	0.01–0.05 µg/kg	From 60 to 120%	
	> 0.05 µg/kg	From 70 to 110%	
Recovery – Aflatoxin B1, B2, G1, G2	< 1µg/kg	From 50 to 120%	
	1 – 10µg/kg	From 70 to 110%	
	> 10µg/kg	From 80 to 110%	
Reliability RSD _R	All	Derived from HORWITZ equation	2xvalue derived from HORWITZ equation

We may calculate precision RSD_R by multiplying by 0.66 reliability RSD_R with observed concentration.

Note

- Value to apply to B1 as well as the sum of B1+B2+G1+G2.
- If we must record the sum of different aflatoxins B1+B2+G1+G2, the response of each of them to the analysis system must be either known or equivalent.

b) Performance criteria for Ochratoxin A

Level of µg/kg	Ochratoxin A		
	RSD _r %	RSD _R %	Recovery%
< 1	≤ 40	≤ 60	From 50 to 120
1 - 10	≤ 20	≤ 30	From 70 to 110

c) Performance criteria for Patulin

Level of µg/kg	Patulin		
	RSD _r %	RSD _R %	Recovery%
< 20	≤ 30	≤ 40	From 50 to 120
20 – 50	≤ 20	≤ 30	From 70 to 105
> 50	≤ 10	≤ 25	From 75 to 105

d) Performance criteria for desoxynivalenol

Level of µg/kg	desoxynivalenol		
	RSD _r %	RSD _R %	Recovery%
> 100 – ≤500	≤ 20	≤ 40	From 60 to 110
> 500	≤ 20	≤ 40	From 70 to 120

e) Performance criteria for zearalenone

Level of µg/kg	zearalenone		
	RSD _r %	RSD _R %	Recovery%
≤ 50	≤ 40	≤ 50	From 60 to 120
> 50	≤ 25	≤ 40	From 70 to 120

f) Performance criteria for Fumonisin B1 and B2

Level of %g/kg	Fumonisin B1 or B2		
	RSD _r %	RSD _R %	Recovery%
≤ 500	≤ 30	≤ 60	From 60 to 120
> 500	≤ 20	≤ 30	From 70 to 110

g) Performance criteria for toxins T-2 and HT-2

Level of µg/kg	Toxin T-2		
	RSD _r %	RSD _R %	Recovery%
50 – 250	≤ 40	≤ 60	From 60 to 130
>250	≤ 30	≤ 50	From 60 to 130

Level of µg/kg	Toxin HT-2		
	RSD _r %	RSD _R %	Recovery%
100 – 200	≤ 40	≤ 60	From 60 to 130
>200	≤ 30	≤ 50	From 60 to 130

h) Note relating to performance criteria for Mycotoxins

- Detection limits of the methods used are not indicated, as the values related to precision are given at the concentrations of interest.
- Values relating to precision are calculated on the basis of HORWITZ equation, i.e.:

$$RSD_R = 2^{(1-0.5 \log C)}$$

In which equation:

- RSD_R is the standard deviation calculated from the results obtained in reproducibility conditions
- It is the rate of concentration (i.e. 1= 100g/100g, 0,001= 1000mg/kg)

This is generalized reliability equation, which has been found to be independent from analyte and matrix and only dependent on concentration for most routine methods of analysis.

4.3.2 ‘Adaptation-to-purpose’ approach

When a limited number of fully validated analysis methods are available, we may choose to adopt an approach based on ‘adaptation-to-purpose’, which defines one single parameter, the function of adaptation-to-purpose approach, to evaluate the analysis method acceptability. An adaptation function is a function of uncertainty, which specifies maximum uncertainty levels, below which the chosen adaptation-to-purpose is considered as secure.

Due to the limited number of fully validated analysis methods through a collaborative study, specifically for the determination of the level of Toxins T-2 and HT-2, the approach based on uncertainty function, which specifies maximum acceptable uncertainty, may also be used to evaluate the adequacy (adaptation-to-purpose) of the analysis method to be used by the laboratory. The laboratory may apply a method that produces results within maximum standardized uncertainty levels. Maximum standardized uncertainty may be calculated by means of the following formula:

Or:

$$Uf = \sqrt{(LOD / 2)^2 + (\alpha C)^2}$$

- Uf is the maximum standardized uncertainty (µg/kg)
- LOD is the method detection level (µg/kg)
- α is a constant numerical factor dependent on the value of C. The values to use are indicated in the table below;
- It is the concentration of interest (µg/kg).

If the analysis method gives results with measurement uncertainty below maximum standardized uncertainty, the method is recognized as valid as a method meeting the performance criteria in 4.3.1.

Numerical values corresponding to the constant α in the formula stated in this paragraph according to the observed concentration.

C (µg/kg)	α
≤ 50	0.2
51 – 500	0.18
5001 – 1000	0.15
1001 – 10000	0.12
> 10000	0.1

4.4 Evaluation of the measurement uncertainty, calculation of the recovery rate and recording the results

The result of the analysis must be recorded corrected or uncorrected for recovery. Recording method and recovery rate shall be indicated. The analytic result corrected for recovery is used to check compliance.

The analysis result shall be consigned under the form $x \pm U$, x representing the analysis result and U the expanded measurement uncertainty.

U is the expanded measurement uncertainty using an expanding coefficient 2, which gives a level of confidence of about 95%.

The present rules for the interpretation of the results in view of compliance or non-compliance of the lot are applicable to the results of the sample analysis dedicated for official control or for defense and reference purposes.

ANNEX 3.3
SAMPLING AND ANALYSIS METHOD FOR OFFICIAL CONTROL OF
LEVELS OF DIOXINES (PCDD/PCDF) AND DIOXINE-LIKE PCB IN
SOME FOODSTUFFS

1. DEFINITIONS

Lot: a quantity of identifiable foodstuff, delivered at one time, determined by the official to have common characteristics such as origin, variety, packaging type, packaging, sender or marker. Concerning fish and fishing products, the size of the fish must be comparable. Even if the size and/or the weight of the fish is not comparable in a lot, this latter is considered a lot, however, a specific sampling procedure shall be applied.

Sub-lot: the part of the lot with a large size selected for sampling. Each sub-lot shall be separate and identifiable;

Incremental sample: a quantity of material taken at one single point of the lot or the sub-lot;

Aggregate sample: All the incremental samples taken from the lot or sub-lot.

Laboratory sample: representative part or quantity of the aggregate sample dedicated for the laboratory.

2. METHOD OF SAMPLING

2.1. General provisions

2.1.1 Personnel

Sampling is performed by an authorized person in compliance with applicable regulations

2.1.2 Material to sample

Any lot or sub-lot to be analyzed is sampled separately

2.1.3 Precautions

In the course of sampling and the preparation of the samples, precautions shall be taken to avoid any alteration liable to modify the level of Dioxin and Dioxin-like PCB, impact the analysis or make the aggregate samples unrepresentative.

Furthermore, it is advised to take all necessary measures to ensure the security of the persons performing the sampling.

2.1.4 Incremental samples

To the best possible, the samples shall be taken at different points distributed throughout the lot or the sub-lot.

2.1.5 Preparation of the aggregate sample

The aggregate sample shall be made up by combining the incremental samples. It shall be at least 1 kg, unless unpractical, for example when one single pack represents a sample.

2.1.6 Replicate samples

Replicate samples dedicated for control, defense and reference purposes are taken from the homogenized aggregate sample. The size of the laboratory samples for enforcement shall be sufficient to allow at least for duplicate analyses.

2.1.7 Packaging and transmission of samples

Each sample shall be placed in a clean, inert container offering adequate protection from contamination, from loss of analytes by adsorption through the internal wall of the container and against damage in transit. All necessary precautions shall be taken to avoid any change in composition of the sample, which might arise during transportation or storage.

2.1.8 Sealing and labeling the samples

Each sample is officially sealed at the place of sampling and identified in compliance with applicable regulations.

2.2. SAMPLING METHOD

The sampling method applied shall ensure that the aggregate sample is representative for the lot or sub-lot to be analyzed.

2.2.1. Division of the lots into sub-lots

The large lots are subdivided into sub-lots, insofar as the sub-lot can be separated physically. **Table 1** concerns large lots of products traded in bulk (for example, vegetal oils). **Table 2** concerns the other products. Insofar as the weight of the lot is not always an exact multiple of the weight of the sub-lots, the weight of the sub-lots may exceed the indicated weight by 20% maximum.

Table 1: Subdivision of the lots of products traded in bulk into sub-lots

Lot weight (in tons)	Weight or number of sub-lots
≥ 1500	500 tons
> 300 and < 1500	3 sub-batches
≥ 50 and ≤ 300	100 tons
< 50	-

Table 2: Subdivision of the lots into sub-lots for the other products

Lot weight (in tons)	Weight or number of sub-lots
≥ 15	15 – 30 tons
< 15	-

2.2.2 Number of incremental samples

The aggregate sample combining the incremental samples weighs at least 1 kg (see paragraph 2.1.5 of the present Annex).

The minimum number of incremental samples to take from the lot or sub-lot is indicated in Table 3 and 4. In the case of liquid products in bulk, the lot or sub-lot is carefully mixed as much as possible insofar as this does not alter the quality of the product, manually or by means of a mechanical process prior to sampling. In this case we assume the distribution of contaminants within the lot or the sub-lot is homogeneous. Hence, three incremental samples are taken from the lot or the sub-lot to form the aggregate sample.

Incremental samples have similar weight. Each incremental sample weighs at least 100 grams. The size of the aggregate sample for hens is of at least twelve eggs (either in bulk or packed, see tables 3 and 4).

Table 3: Minimum number of incremental samples to take from the lot or sub-lot

Weight or volume of the lot or sub-lot (in kg or liter)	Minimum number of incremental samples to realize
< 50	3
From 50 to 500	5
> 500	10

If the lot or sub-lot includes distinct units or packs, the number of units or packs to form the sample is indicated in **Table 4**.

Table 4: Number of units or packs (incremental samples) to constitute the aggregate sample if the lot or sub-lot includes distinct units or packs

Number of packs or units in the lot or sub-lot	Number of packs or units to take
From 1 to 25	At least one pack or unit
From 26 to 100	About 5%, at least 2 units or packs
> 100	About 5%, at least 10 units or packs maximum

2.2.3 Specific provisions for sampling lots containing whole fish of comparable size

Fish are deemed to have comparable size and weight when the difference in size and weight do not exceed 50%.

The number of incremental samples to take is determined in Table 3. The aggregate sample, combining the incremental samples weighs at least 1 kg (see paragraph 2.1.5).

If the lot to sample contains small size fish (individual weight below 1 kg), the whole fish is taken as incremental sample in order to constitute the aggregate sample. If the resulting aggregate sample weighs over 1 kg, the incremental samples may be taken in the middle part with a weight of 100 grams each. The whole part on which maximum level is applied is used for homogenizing the sample.

The middle part of the fish is the part where there is its gravity center. This latter is situated in most cases at the level of the dorsal fin (when the fish has one) or at half-distance from the gill and the anus.

If the lot to sample contains larger fish (individual weight over 1 kg), the incremental sample is made up of the middle part of the fish. Each incremental sample weighs at least 100 grams. In the case of fish of intermediary size (about 1 to 6 kg), the incremental sample consists in a slice cut between the backbone and the abdomen.

In the case of very large fish (> about 6 kg), the incremental sample is made up of meat taken from the right dorsal-lateral muscle (frontal view) in the middle part of the fish.

In case taking such a piece in the middle part of the fish causes an important economic loss, or taking 3 incremental samples of 350 grams each is sufficient, whatever the size of the lot, or two equal pieces of meat may be taken, one in the muscle near the fishtail and the other in the muscle near the head to have an incremental sample, which is representative of Dioxin level in the whole fish.

2.2.4 Sampling lots of fish containing whole fish with different size and/or weight.

Provisions in paragraph 2.2.3 to constitute the sample are applicable.

If a class/category of weight or size is predominant (about 80% or more), the sample is taken from this class/category. This sample may be considered as representative of the whole lot.

If no class/category predominates in size or weight, it is advisable to select fish that is representative of the lot to constitute the sample.

2.2.5 Sampling at retail sale level

Sampling foodstuffs at retail sale level is performed, to the best possible, following the related provisions in 2.2.2 of the present Annex. If this is not possible, an alternative sampling method may be used, insofar as it ensures that the samples are sufficiently representative of the sampled lots or sub-lots.

2.3 Compliance of the lot or sub-lot with specifications

The lot is compliant if the result of only one analysis does not exceed the corresponding maximum level in Dioxins and Dioxin-like PCB, laid down in the present decision, taking into consideration the correction for recovery and the measurement uncertainty.

We consider that the lot does not comply with maximum level laid down in the present decision if the result analysis related to the higher estimation, confirmed by a duplicate analysis, exceeds with quasi certainty the maximum level laid down in the present decision, taking into consideration the correction for recovery and the measurement uncertainty.

The measurement uncertainty may be considered in the following two ways:

-calculating the expanded uncertainty by means of a coverage factor of 2, which gives a level of confidence of about 95%. A lot or sub-lot does not comply if the measured value U exceeds the maximum level allowed. In case of distinct proportioning of the Dioxins and the Dioxin-like PCB, the estimated sum of the expanded uncertainty of the distinct analysis results of Dioxin and Dioxin-like PCB shall be used for the sum of Dioxin and Dioxin-like PCB,

The present interpretation rules apply to the analysis results of the samples dedicated for official control. In case of analysis for defense or reference purposes, the national rules are applicable.

2.4 PREPARATION OF THE SAMPLES AND PROVISIONS RELATED TO ANALYSIS METHODS USED FOR OFFICIAL CONTROL OF THE LEVEL OF DIOXINE (PCDD/PCDF) AND DIOXINE-LIKE PCB IN SOME FOODSTUFFS

3.1 Quality standards applicable to the laboratory

Laboratories shall meet the following provisions:

3.1.1 Material requirements

- a) Personnel working on the analysis for official control should be impartial without any interest conflicts relating to the duties assigned to them.
- b) The laboratories must have the required capacity to carry out the analyses as well as duly qualified and experienced personnel in sufficient number so that they can perform their analyses and tasks efficiently and effectively
- c) They must have adequate installations and equipment with appropriate maintenance to allow personnel to carry out their tasks in an efficient and effective way.

3.1.2 Moral requirements

- a) Laboratories endeavor to perform their activities at a high level of transparency. For this end, they shall make available to competent authorities any pertinent information they hold as quickly as possible.
- b) The laboratories must take the necessary measure so that their personnel should not reveal any information they obtain during their activities within official control; this information is, by nature, covered by the professional secret

3.2 Processing and preparing the samples

3.2.1 Method of sampling in the laboratory

The full aggregate sample is finely crushed (if necessary) and carefully mixed by means of a method that ensures complete homogenization.

Samples shall be processed and labeled in such a way that their analytic and legal validity should be ensured.

3.2.2 Samples dedicated for control, defense or reference purposes

Samples dedicated for control, defense or reference purposes are taken from the homogenized substance.

3.2.3 Preparation of the sample

The aim is to have a laboratory sample that is representative and homogeneous without any introduced external contamination.

All the samples received by the laboratory shall be used for the preparation of the laboratory sample.

3.2.4 Specific procedures for the preparation of the samples for Dioxin analysis

2.4.1 Application scope

The provisions in the present Annex apply to the analysis of foodstuffs realized for official control of the level of Dioxins [polychlorodibenzo-p-dioxin (PCDD) and polychlorodibenzofurans (PCDF) and dioxin-like PCB.

In order to control the presence of Dioxins in foodstuffs, we may use a strategy based on screening, so that we can select the samples with Dioxin and Dioxin-like PCB levels below 25% or over maximum level. Dioxin level and the sum of Dioxins and Dioxin-like PCB in the samples having high levels must be determined/confirmed by means of a confirmatory method.

Screening methods aim at detecting the presence of Dioxin and Dioxin-like PCB at the level under consideration. They have a great capacity to process samples, which allows for screening many samples in order to detect those that could reveal positive. They are specifically designed to avoid false negative results.

Confirmatory methods provide complete or complementary information, which allow for doubtless identification and quantification of Dioxin and Dioxin-like PCB at the level under consideration.

2.4.2 Background

The concentrations of each substance in a given sample are multiplied by their respective toxic equivalent factors (TEF), as laid down by the World Health Organization mentioned in the appendix of the present Annex, then they are added so that they give total concentration in Dioxin-like compounds expressed in toxic equivalents (TEQ).

With regard to the purpose of the present decision, the accepted specific level of a congener quantification is the concentration of analyte in a sample extract, which produces an instrumental response to the two different ions that are controlled by a ratio S/N (Signal/Noise) of 3:1 for the less precise signal and meets the basic conditions (for example, retention time, isotopic report according to the determination procedure described in the method EPA 1613, revision B).

2.4.3 Provisions of quality warranty for the preparation of the samples

- Measurements are needed in order to avoid any cross contamination at each step of the sampling and analysis process.
- The samples shall be preserved and transported in containers made of glass, aluminum, polypropylene or polyethylene. The sample container shall be cleaned from any paper dust. Glass shall be washed with a certified solvent exempt from Dioxin or previously controlled for Dioxin detection.

- The preservation and transport of the sample shall be done in such a way that the integrity of the foodstuff sample should be preserved.
- If necessary, each laboratory sample shall be finely crushed and carefully mixed with a method that ensures full homogenization (for example, sifting them through a 1 mm sieve); the samples shall be dried before being crushed if they have a high moisture level.
- A blank test shall be performed, carrying out all the analytic procedure and omitting the sample.
- The extract weight shall be high enough to meet precision requirements.
- The specific procedures for the preparation of the samples used for the products under consideration are validated according to internationally acknowledged guidelines.
- In the case of fish, the skin must be removed as maximum levels apply to the muscle meat. However, it is necessary that all the remains of muscle meat and the fat on the internal side of the flesh should be carefully and completely scraped and added to the sample to analyze.

2.4.4 Provisions applicable to the laboratory

- Laboratories shall demonstrate the method validity in a variety around the level involved, for example, at levels equal to 0.5, 1 time and twice this level with an acceptable variation coefficient for repeated analyses. For more details on validity criteria, see paragraph 5.
- The level of quantification for a confirmatory method should not exceed about a fifth of the level involved.
- Laboratory proficiency shall be proven by the continuous successful participation in inter-laboratory studies for the determination of PCDD/Fs and dioxin-like PCBs in relevant food matrices and concentration ranges.
- Laboratories shall meet quality system requirements and apply standardized or validated methods.

2.4.5 Provisions for analysis procedures of Dioxin and Dioxin-like PCB

Basic provisions for analysis procedure validity

- Low range and limits of quantification. For the PCDDs and PCDFs, quantification range must be in pictogram TEQ (10-12 g), due to the very high toxicity of these compounds. It is proved that there are a lot more PCBs than PCDDs and PCDFs. For most congeners of PCB group, the limit of quantification in the nanogram is sufficient. However, for the measurement of the more toxic toxin-like congeners (particularly non-ortho substituted congeners), the lower end of the range must reach the pictogram like the PCDDs and PCDFs.

- High selectivity (specificity). A distinction is necessary between the PCDDs and PCDFs and dioxin-like PCBs from a multitude of other compounds extracted with the sample, which are liable to interfere and which are present in much higher concentrations than those in the analytes to be quantified. For gas chromatography/mass spectrometry (GC-MS) methods, a differentiation among various congeners is necessary (e.g. The seventeen PCDDs and PCDFs substituted by 2,3,7,8 and the twelve dioxin-like PCBs) and other congeners. Bio-analytical tests should allow for the determination of TEQ values as the sum of the PCDDs, PCDFs and Dioxin-like PCBs.

- High accuracy (trueness and precision)

The analysis should allow for a true evaluation of the effective concentration in a sample. A high accuracy (accurate measurement: closeness between measurement and the real or assigned value of the measured) is necessary to avoid the rejection of a sample on the basis of poor reliability of the TEQ level. Accuracy is expressed as trueness (difference between the average value measured for an analyte in a certified material and its certified value, expressed as percentage of this value) and precision (RSD_R relative standard deviation calculated from results generated under reproducibility conditions).

Screening methods may include bio-analytical methods and CG/SM methods; gas chromatography/mass spectrometry methods with high resolution/mass spectrometry (CGHR/SMHR). The criteria below shall be met for total TEQ value:

	Screening method	Confirmatory methods
Negative false rate	< 1%	
accuracy		- 20% + 20%
Reliability (RSD _R)	< 30%	< 15%

2.4.6 Specific provisions for CG/SM methods used for screening and confirmation

Adding 2,3,7,8 substituted PCDD/Fs internal standard, labeled 13C and internal dioxin-like PCB standards labeled 13C, shall be carried out at the beginning of the analytical method, for example just before extraction to validate the analytical procedure. We must add at least one congener for each of the tetra- to octa-chlorinated homologous groups for PCDD/Fs and at least one congener for each of the homologous groups for dioxin-like PCBs (another method consists in adding at least one congener to each mass spectrometric selected ion recording function used for controlling the PCDD/Fs and the dioxin-like PCBs. It is recommended, in the case of confirmatory methods to use all the seventeen 2,3,7,8 substituted internal PCDD/F standards labeled 13C as well as all the twelve internal dioxin-like PCBs labeled 13C.

Relative response factors shall also be determined in the case of the congeners for which no 13C-labelled analogue is added by using appropriate calibration solutions.

- For foodstuffs of plant origin and foodstuffs of animal origin containing less than 10 % fat, the addition of the internal standards is mandatory prior to extraction. For foodstuffs of animal origin containing more than 10 % fat, the internal standards may be added either before or after fat

extraction. An appropriate validation of the extraction efficiency shall be carried out, depending on the stage at which internal standards are introduced and on whether results are reported (on product or fat basis).

- Prior to the CG/SM analysis, one or two substitution standards shall be added.

- A recovery control is necessary. In case of confirmatory methods, internal standards recovery rates shall be situated in a range 60 to 120%. For individual congeners, specifically for some hepta- and octa- chlorinated dibenzo-p-dioxins and dibenzofurans, lower or higher recovery rates are acceptable, on the condition that their contribution to TEQ value does not exceed 10% of total TEQ value (based on PCDD/F and dioxin-like PCBs sum). For GC-MS screening methods, the recoveries shall be in the range of 30 to 140 %.

- Separation of PCDD/Fs from interfering chlorinated compounds such as non-dioxin-like PCBs and chlorinated diphenyl by means of the appropriate chromatographic techniques (preferably with a florisil, alumina and/or carbon column).

- Gas-chromatographic separation of isomers shall be sufficient (< 25 % peak to peak between 1,2,3,4,7,8-HxCDF and 1,2,3,6,7,8-HxCDF).

- Calibration shall be carried out according to method EPA 1613, revision B, called “Tetra-Trough-Octa-chlorinated dioxin and furans by isotope dilution HRGC/HRMS”, of US Agency of Environment Protection, or any other method with the same efficiency criteria.

- The difference between lower and upper levels shall not exceed 20% for foodstuffs whose contamination with dioxins is about 1pg OMS-TEQ/g fat (based on the sum of PCDD/F and dioxin-like PCB). The same provisions apply to foodstuffs with low fat levels, whose contamination is about 1pg OMS-TEQ/g of product. For lower contamination levels, for example 0,50 pg OMS-TEQ/g of product, the difference between upper and lower levels may be situated in a range between 25 to 40%.

2.4.7 Analytical screening method

2.4.7.1 Introduction

Different approaches may be used for screening method: a screening approach is a quantitative approach.

Screening approach:

The response of the samples is compared to a reference sample, at the level under consideration. Samples with response below the reference are declared negative and those with response above the reference are considered positive.

Provisions:

- In the course of every series of tests, a blank sample and a reference sample shall be extracted at the same time and in the same conditions. The response of the reference sample shall be clearly higher than that of the blank sample;
- Additional reference samples, with a concentration equal to 0.5 times and 2 times the level under consideration shall be included to demonstrate the efficiency of the test in the pertinent range for controlling the level under consideration;
- In case we proceed to test other matrices, the validity of the reference sample or samples shall be proved, preferably by using samples with TEQ value, set by CGHR/SMHR, nearing the value of the reference sample or, if not, an enriched blank to reach this level;
- Insofar as no internal standard can be used in the bio-analytical tests, repeatability tests are carried out to have information about standard deviation within a series of tests. Variation coefficient shall be below 30%;
- For bio-analytical tests, target compounds, potential interference and maximum tolerated value for the blank, are defined.

Quantitative approach:

Quantitative approach includes necessarily typical dilution series, double or triple measurement and cleaning process as well as blank tests and recovery tests. The result may be expressed in TEQ, which assumes that the compounds at the origin of the signal meet the TEQ principle. For this end, we may use TCDD (or a typical dioxin/furan/dioxin-like PCB mix) to have a standard curve, which helps calculate TEQ value in the extract and, consequently, for the sample. The result is then corrected of TEQ value calculated by means of a blank sample in order to take into consideration any impurity coming from the solvents or the chemical substances used) and for a recovery (this further quantity is calculated from TEQ value in a quality control sample with a concentration close to the level under consideration). We should bear in mind that a part of the apparent recovery loss may be due to matrix and/or TEF value variations effects for bio-analytical tests and TEF official values laid down by the WHO.

2.4.7.2 Requirements related to analytical screening methods

- Screening may be carried out by means of CG/SM or bio-analytical methods. Provisions in point 6 shall be used for CG/SM methods. Specific provisions are set at point 2.4.7.3 of the present Annex for cellular bio-analytical tests and in point 4.7 of the present Annex for bio-analytical tests carried out by means of kits.
- Information shall be provided relating to the number of false positive and false negative of a large number of samples below or above maximum level or the cut-off value, in comparison with TEQ value determined by a confirmatory analytical method. The real rates of false negative must

be below 1%. The rate of false positive sample must remain sufficiently low for beneficial use of the screening method.

- Positive results must always be confirmed by a confirmatory analytical method (CGHR/SMHR). Furthermore, a wide range of TEQ samples must be confirmed by CGHR/SMHR (about 2% of the negative samples). Information relating to conformity between bio-analytical results and CGHR/SMHR results must be provided.

2.4.7.3 Specific requirements for cellular bio-analytical tests

- For bio-analytical methods, a series of TCDD concentrations or a dioxin/furan/dioxin-like PCB mix (response curve with $R^2 > 0.95$ for a full dose) is necessary in each test. However, for screening the lower working range, we may use a more detailed curve to analyze samples with low levels.

- For bio-analytical results in a constant time interval TCDD concentration shall be used (about 3 times the quantification level) on a quality control form. We may also rely on the relative response of a reference sample compared to TCDD standard curve, insofar as the cell response may be dependent on many factors.

- Quality control graphs shall be realized and checked for each type of reference material, in order to ensure the result compliance with the guidelines provided.

- The sample dilution induction used shall be situated in the linear part of the reference curve, particularly for quantitative calculations. Samples situated beyond this linear part shall be diluted and tested again. That's why it is advised to test at least three dilutions simultaneously.

- Standard variation shall neither exceed 15% when a triple measurement is performed for each sample dilution, nor exceed 30% for three independent experiments.

- We may choose for the detection level equal to three times the standard variation of the blank solvent or of the effective response. Another method consists in taking a concentration, which corresponds to a much higher response than the effective response on the standard curve of the day (induction factor 5 times higher than the blank solvent). It is possible to take for quantification level a value, which is five to six times above standard variation of solvent blank or to the effective response or take a concentration, which corresponds to a response above the effective response on the standard curve of the day (induction factor 10 times above solvent blank).

2.4.7.4 Specific requirements for bio-analytical test carried out by means of kits

- ✓ Bio-analytical test by means of kits shall display sufficient reliability and precision in order to be applied to foodstuffs.
- ✓ It is advisable to follow the manufacturer's instructions relating to the preparation of the samples and the analyses.

- ✓ Kits with expired dates cannot be used.
- ✓ Use the appropriate kit for the material or the component.
- ✓ Kit preservation temperature must be within specified temperature range and the operation temperature must comply with the specified value.
- ✓ Detection level for immune-tests is determined by multiplying by three the standard variation, based on ten blank analyses, and by dividing the product obtained by the curve value in the linear regression equation.
- ✓ Reference standards shall be used for laboratory tests, to ensure that response to standard is within acceptable range.

2.4.8 Results

As far as possible, the results shall include PCDD/PCDF/PCB individual congeners and indicated in lower, upper and intermediary values, in order to include maximum information, which allows for interpreting the results according to the specific requirements.

The report shall also include fat level in the sample as well as the method used to extract it.

Recovery rate of individual internal standards shall be provided in case they are situated outside the range mentioned in point 6 or exceed maximum level. In all the other cases, they shall be provided at request.

Measurement uncertainty shall also be mentioned, as this parameter is taken into consideration to determine the sample compliance. Consequently, the analysis result shall be recorded in the form “x +/- U”, with x as the analysis result and U the expanded measurement uncertainty calculated by means of a coverage factor of 2, which gives a confidence level about 95%.

In case of distinct dioxin and dioxin-like PCB proportioning, the sum of expanded uncertainty estimations of the distinct dioxin and dioxin-like PCB analysis results shall be used for the sum dioxin and dioxin-like PCB.

The results are expressed in the same units and by (at least) the same number of significant figures as maximum levels laid down in the present Decision.

Table of Toxic equivalency factors (TEF) of World Health Organization (WHO), for the evaluation of risks for humans, based on the conclusions of WHO meeting, held in Stockholm (Sweden), 15 – 18 June 1997 [Van den Berg et al.(1998), Toxic Equivalency Factors (TEFs) for PCBs, PCDDs, PCDFs, for Humans and for Wildlife, Environmental Health Perspectives, 106 (12), 775]

Congener	TEF value	Congener	TEF value
Dibenzo-p-dioxin		“dioxin-like, non-ortho PCB	
2,3,7,8-TCDD	1	+ mono-ortho PCB	
1,2,3,7,8-PeCDD	1	Non-ortho PCB	
1,2,3,4,7,8-HxCDD	0,1	PCB 77	0,0001
1,2,3,6,7,8-HxCDD	0,1	PCB 81	0,0001
1,2,3,7,8,9-HxCDD	0,1	PCB 126	0,1
1,2,3,4,6,7,8-HpCDD	0,01	PCB 169	0,01
OCDD	0,0001		
Dibenzofuran (PCDF)		Mono-ortho PCB	
2,3,7,8-TCDF	0,1		0,0001
1,2,3,7,8-PeCDF	0,05	PCB 105	0,0005
2,3,4,7,8-PeCDF	0,5	PCB114	0,0001
1,2,3,4,7,8-HxCDF	0,1	PCB 118	0,0001
1,2,3,6,7,8-HxCDF	0,1	PCB 123	0,0005
1,2,3,7,8,9-HxCDF	0,1	PCB 156	0,0005
2,3,4,6,7,8-HxCDF	0,1	PCB 157	0,00001
1,2,3,4,6,7,8-HpCDF	0,01	PCB 167	0,0001
1,2,3,4,7,8,9-HpCDF	0,01	PCB 189	
OCDF	0,0001		

Abbreviations: « T »= Tetra, « Pe »= Penta, « Hx »= Hexa, « Hp »= Hepta, « O »= Octa,
« CDD »= Chlorinated dibenzodioxin
« CDF »= chlorinated dibenzofuran, « CB »= chlorinated biphenyl

ANNEX II. 4
SAMPLING AND ANALYSIS METHODS FOR OFFICIAL CONTROL OF NITRATE
LEVELS IN SOME FOODSTUFFS

1. DEFINITIONS

For the purpose of this Annex, the following definitions apply:

“Lot”: a quantity of identifiable foodstuff, harvested at the same time or delivered at one time, and determined by the official to have common characteristics such as origin, variety, type of land within a maximum of two hectares, packaging type, type packaging, packager, dispatcher or marker;

“sub-lot”: the part of a large lot on which to apply the sampling method designated for this effect. Each sub-lot must be separate and identifiable;

“sample or incremental unit”: a quantity of material taken at one single point of the lot or the sub-lot. It concerns, here, one single head of lettuce or spinach, a handful of leaves or one sachet of cut leaves;

“aggregate sample”: All the incremental samples taken from the lot or sub-lot;

“laboratory sample”: a sample dedicated for the laboratory;

“field”: a given piece of land belonging to the same type of land and growing the same crops, having one single variety of lettuce or spinach at the same stage of growth;

“sheltered surface”: a given piece of land covered with a glass greenhouse or a polytunnel (tunnel or greenhouse in plastic or polyethylene) containing one single variety of lettuce or spinach at the same stage of growth and to be harvested at the same time. The “sheltered surface” may also be designated by the word “lot” in the sampling method.

2. METHOD OF SAMPLING

2.1 GENERAL PROVISIONS

2.1.1 Personnel

Sampling is performed by an authorized person in compliance with applicable regulations.

2.1.2 Material to be sampled

Any lot or sub-lot, which is to be analyzed shall be sampled separately. Large lots weighing over 30 tons or having a surface area over 3 hectares) are subdivided into sub-lots and sampled separately.

2.1.3 Precautions

In the course of sampling and preparation of the samples, precautions are needed to avoid any alteration that could impact:

- Nitrate levels, the analysis or the representativeness of the aggregate sample (for example presence of earth on the lettuce or spinach during the preparation of the sample),
- Food security and integrity of the samples.

Furthermore, all necessary measures should be taken to ensure the security of the agents performing the sampling.

2.1.4 Incremental samples

If possible, the samples are taken at different points distributed throughout the lot or sub-lot.

2.1.5 Preparation of the aggregate sample

The aggregate sample is made up by combining all the incremental samples.

2.1.6 Replicate samples

Replicate samples are taken from the homogenized aggregate sample

2.1.7 Packaging and transmission of the samples

Each sample is placed in an inert plastic opaque bag, clean and hermetically sealed to avoid losses in moisture and offer adequate protection against any damage or contamination.

The sample shall be transmitted to the laboratory in the 24 hours following sampling and kept in a cool place during transit. If this is not possible, the sample is deep-frozen in the 24 hours and kept frozen (six weeks maximum).

All other necessary precautions are taken to avoid any modification in the composition of the sample that may occur during transport or storage.

2.1.8 Sealing and labeling the samples

Each sample is sealed at the place of sampling and identified in compliance with applicable regulations.

2.2 DIFFERENT TYPES OF LOTS

Foodstuffs may be traded in bulk, in containers including bags or boxes, or in individual packages dedicated for retail sale. The method of sampling may be used for all types of lots.

2.3. METHOD OF SAMPLING

As much as possible, samples are taken at different points throughout the lot or the sub-lot.

2.3.1 Taking samples from the field

When the competent authorities decide to take the samples in the field of lettuce or spinach, sampling is carried out in the following way:

The incremental samples are not taken from areas that are not representative of the field or the sheltered surface. The areas with different types of land and different types of crops or containing different varieties of lettuce or spinach or to be harvested at different times are considered as different lots or distinct fields. If the field has a surface area over 3 hectares, it is sub-divided into sub-lots and each sub-lot is sampled separately.

The incremental samples are taken along a line making a “W” or an “X” in the field. Incremental samples taken on narrow boards or in sheltered surfaces are taken along a line making a “W” or an “X” on many boards and they are united to constitute the aggregate sample.

Plants shall be cut at soil level

The sample shall include at least ten plants and the ten-plant aggregate sample shall weigh a minimum of 1 kg. Only units with marketable size shall be sampled.

The earth and the external damaged and inedible leaves are removed from each unit.

2.3.2 Sampling lots of spinach, lettuce, foods for infants and processed cereal foods dedicated for infants and young children

Sampling method is applicable to lots weighing 25 tons or less.

In the case of a lot weighing over 30 tons, it is subdivided into sub-lots of 25 tons, on condition that they can be physically separated. Insofar as the lot is not always an exact multiple of 25 tons, the weight of the sub-lot may exceed the indicated weight by a maximum of 20%. This means that the sub-lots may weigh between 25 and 30 tons. If a lot cannot be divided into sub-lots, the sample is taken from the lot.

The aggregate sample weighs 1 kg minimum, unless not practical, for example when sampling one head of lettuce or spinach or one single packaged unit.

The minimum number of incremental samples to take from the lot is indicated in **Table 1**.

Table 1: Minimum number of incremental samples to take from the lot

Lot weight (in kg)	Minimum number of incremental samples	Minimum weight of aggregate sample (in kg)
< 50	3	1
From 50 to 500	5	1
> 500	10	1

If the lot includes distinct packages, the number of packages to make the aggregate sample is indicated in **Table 2**.

Table 2: Number of packages (incremental samples) to take to form the aggregate sample when the lot includes distinct packages

Number of packs in the lot	Number of packs to take	Minimum weight of global sample (in kg)
From 1 to 25	1 unit or pack	1
From 26 to 100	About 5%, 2 units at least	1
> 100	About 5%, ten units or packs at least	1

Each lot or sub-lot to be analyzed for compliance shall be sampled separately. However, if this sampling method may have negative economic impacts due to possible deterioration of the lot (because of packaging or transport, etc.), another alternative method may be used, which is thoroughly described and documented, and shall ensure the aggregate sample is as representative as possible. The place of the lot to sample is chosen at random, if unpractical, the place is chosen at random in the accessible parts of the lot.

2.3.3 Sampling at retail sale stage

Sampling foodstuffs at retail stage is realized, to the best possible, in compliance with the provisions in point 2.3.2.

If this reveals impossible, an alternative sampling method, which is thoroughly described and documented, may be used, insofar as it ensures that the aggregate sample is sufficiently representative of the sampled lot.

2.3.4 Acceptance of a lot or a sub-lot

- Acceptance if the laboratory sample does not exceed maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

- Rejection if the laboratory sample exceeds beyond reasonable doubt maximum levels, taking into consideration the correction for recovery and the measurement uncertainty.

3. PREPARATION AND ANALYSIS OF THE SAMPLES

3.1 Quality standards applicable to the laboratory

Laboratories shall meet the following requirements:

3.1.1 Material requirements

- a) Personnel working on the analysis for official control should be impartial without any interest conflicts relating to the duties assigned to them.
- b) The laboratories must have the required capacity to carry out the analyses as well as duly qualified and experienced personnel in sufficient number so that they can perform their analyses and tasks efficiently and effectively
- c) They must have adequate installations and equipment with appropriate maintenance to allow personnel to carry out their tasks efficiently and effectively.

3.1.2 Moral requirements: transparency and confidentiality

- a) Laboratories endeavor to perform their activities at a high level of transparency. For this end, they must make available to competent authorities any pertinent information they hold as quickly as possible.
- b) Laboratories must take the necessary measure so that their personnel should not reveal any information they obtain during their activities within official control; this information is, by nature, covered by the professional secret.

3.2 PREPARATION OF THE SAMPLES IN THE LABORATORIES

3.2.1 Processing the samples in the laboratory

The full aggregate sample is finely crushed (if necessary) and carefully mixed by means of a process that ensures complete homogenization.

Samples must be processed and labeled in such a way that ensures their analytic and legal validity.

3.2.2 Samples dedicated for control, defense and reference purposes

Samples dedicated for control, defense and reference purposes are taken from the homogenized aggregate sample.

3.2.3 Preparation of the sample

The aim is to have a laboratory sample that is representative and homogeneous without any introduced external contamination.

All the samples received by the laboratory shall be used for the preparation of the laboratory sample.

Compliance with maximum levels fixed in the present Decision is based on the levels determined in laboratory samples.

3.2.4 Specific procedures for the preparation of the sample for nitrate analysis

- When sampling concerns fresh products, the samples are prepared, if possible, in the 24 hours following sampling. If unpractical, the samples are kept deep-frozen (six weeks maximum).

- The earth and dirty leaves as well as the external inedible or damaged leaves are removed from each unit. It is prohibited to wash the samples insofar as this may cause a decrease in the nitrate level they contain.

- All the samples shall be homogenized (adding water is optional). According to the size of the mixer, grinder or mincer, one or more units are combined for homogenization. The fact of freezing and mincing the products before proceeding to homogenization, may allow for better mixing. Homogenization process must ensure total homogenization. It is crucial that homogenization is total in order to maximize extraction and recovery of nitrate. In this concern, the samples are processed in the same way no matter the place of sampling (field or retail sale).

- One or more samples are taken from the resulting mash in order to be analyzed.

3.3 ANALYSIS METHOD, RECORDING THE RESULTS AND REQUIREMENTS IN TERMS OF LABORATORY CONTROL

3.3.1 General requirements

1. Analysis methods used for official control are standardized and compliant methods or:

- a) If such standards are lacking, to internationally acknowledged protocols or those adopted in national legislation, or
- b) In the absence of this, to the appropriate methods for this end, or elaborated according to scientific protocols.

2 – If paragraph 1 is not applicable, analysis methods may be validated within the laboratory following a protocol, which is internationally accepted (International, European or National standards).

3 – The analysis methods, including confirmatory or reference methods needed in case of protest are, to the best possible, characterized by the following appropriate criteria:

- a) Accuracy
 - b) Application field (analyte, matrix and concentration range)
 - c) Detection level
 - d) Determination level
 - e) Repeatability
 - f) Reproducibility
 - g) Recovery rate
 - h) Selectivity
 - i) Precision
 - j) Linearity
 - k) Error margin and measurement uncertainty
 - l) Other criteria following need
4. The competent authority is entitled to check the performance criteria, the analysis parameters, considerations related to uncertainty of the validation measures and procedures of the methods.

3.3.2 Specific criteria for nitrate analysis

3.3.2.1. Definition

The following definitions are applied:

“r”: **repeatability:** value below which we can expect that the absolute variation between the results of individual tests obtained in repeatability conditions (i.e. same sample, same operator, same equipment, same laboratory and a short period of time) is situated in a certain probability level (in principle 95%); so $r = 2,8 \times Sr$.

“Sr”: **Standard variation** calculated from results obtained in repeatability conditions.

“RSD_r”: **Relative standard variation** calculated from results obtained in repeatability conditions

$$RSD_r = (Sr/x) \times 100$$

“R”: **Reproducibility:** value below which we can expect that the absolute variation between the results of individual tests obtained in reproducibility conditions (i.e. for the same product executed by different operators in different laboratories using the method of standardized test), is situated in a certain probability level (in principle 95%); $R = 2,8 \times Sr$.

“sr”: **standard variation** calculated from results obtained in reproducibility conditions.

“RSD_R”: **Relative standard variation**, calculated from results obtained in reproducibility conditions

$$RSDR = (SR/x) \times 100$$

“LOD”: **Detection level:** the lowest detected level (in analyte), from which we may deduce the presence of analyte with a reasonable statistical certainty. The detection level is numerically equal to three times the standard variation of average of blank tests ($n > 20$).

“LOQ”: **Quantification level:** the lowest level of analyte, measurable with a reasonable statistical certainty. If accuracy and precision are both at a concentration oscillating around the detection level, the quantification level is numerically equal to ten times the standard variation of average blank tests ($n > 20$).

“HORRAT_r”: The observed RSD_r divided by the estimated RSD_r from HORWITZ equation (1) postulating that $r = 0,66R$.

“HORRAT_R”: The observed value of RSD_R divided by the RSD_R value, which is calculated from HORWITZ equation.

“u”: Compound measurement uncertainty

“U”: Expanded measurement uncertainty

We find the expanded uncertainty by multiplying the compound-type certainty by the coverage factor k, with $k=2$ for a confidence level of 95%.

$$U = 2 \times u$$

“U_f” = Maximum standard uncertainty measurement

3.2.3.1 Extraction procedure

A special attention shall be granted to the extraction procedure adopted. Many extraction procedures, such as extraction with hot water or with methanol and water (30/70), ensure efficient extraction of nitrate. Extraction with cold water shall be used only in case the sample has been deep-frozen before extraction.

3.2.3.2 Performance criteria

The specific criteria applicable to the analysis method for the control of nitrate level are the following:

Criterion	Concentration range	Recommended value	Maximum permitted value
Recovery	< 500 mg/kg	60 – 120%	
	≥ 500 mg/kg	90 – 110%	
Reliability	ALL	Derived from HORWITZ equation	2 x the value derived from HORWITZ equation

Reliability RSD_r may be calculated by multiplying by 0.66

Reliability RSD_R at the concentration of interest.

Note relating to performance criteria:

- Concentration ranges are not indicated, insofar as the values relating to reliability are calculated at the concentrations of interest.
- Values related to reliability are calculated based on HORWITZ equation, i.e.:

$$RSD_R = 2^{(1-0.5 \log C)}$$

In which equation:

- RSD_R is the relative variation-like calculated based on the results obtained in reproducibility conditions
- C is the concentration rate

3.2.4. Recording the results, estimation of the measurement uncertainty and calculation of the recovery rate

The result of the analysis shall be recorded either corrected or uncorrected for recovery. Recording method and recovery rate shall be indicated. The analytical result corrected for recovery is used to check compliance.

The result of the analysis shall be recorded under the form $x \pm U$, with x representing the analysis result and U the expanded measurement uncertainty.

U is the expanded measurement uncertainty, using a coverage coefficient 2, which gives a confidence level of about 95%.

The present interpretation rules of the analysis result in view of compliance or non-compliance of the lot are applicable to the analysis result of the sample dedicated for official control, defense or reference purposes.