

COMMISSION REGULATION (EC) No 333/2007

of 28 March 2007

laying down the methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs

(Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules ⁽¹⁾, in particular Article 11(4) thereof,

Whereas:

(1) Council Regulation (EEC) No 315/93 of 8 February 1993 laying down Community procedures for contaminants in food ⁽²⁾ provides that maximum levels must be set for certain contaminants in foodstuffs in order to protect public health.

(2) Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs ⁽³⁾ establishes maximum levels for lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in certain foodstuffs.

(3) Regulation (EC) No 882/2004 lays down general principles for the official control of foodstuffs. However, in certain cases more specific provisions are necessary to ensure that official controls are performed in a harmonised manner in the Community.

(4) The methods of sampling and analysis to be used for the official control of levels of lead, cadmium, mercury, 3-MCPD, inorganic tin and benzo(a)pyrene in certain foodstuffs are established in Commission Directive 2001/22/EC of 8 March 2001 laying down the sampling methods and the methods of analysis for the

official control of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs ⁽⁴⁾, Commission Directive 2004/16/EC of 12 February 2004 laying down the sampling methods and the methods of analysis for the official control of the levels of tin in canned foods ⁽⁵⁾ and Commission Directive 2005/10/EC of 4 February 2005 laying down the sampling methods and the methods of analysis for the official control of the levels of benzo(a)pyrene in foodstuffs ⁽⁶⁾, respectively.

(5) Numerous provisions on sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs are similar. Therefore, in the interest of clarity of legislation, it is appropriate to merge those provisions in one single legislative act.

(6) Directives 2001/22/EC, 2004/16/EC and 2005/10/EC should therefore be repealed and replaced by a new Regulation.

(7) The measures provided for in this Regulation are in accordance with the opinion of the Standing Committee for the Food Chain and Animal Health,

HAS ADOPTED THIS REGULATION:

Article 1

1. Sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene listed in sections 3, 4 and 6 of the Annex to Regulation (EC) No 1881/2006 shall be carried out in accordance with the Annex to this Regulation.

2. Paragraph 1 shall apply without prejudice to the provisions of Regulation (EC) No 882/2004.

⁽¹⁾ OJ L 165, 30.4.2004, p. 1, corrected by OJ L 191, 28.5.2004, p. 1. Regulation as amended by Commission Regulation (EC) No 1791/2006 (OJ L 363, 20.12.2006, p. 1).

⁽²⁾ OJ L 37, 13.2.1993, p. 1. Regulation as amended by Regulation (EC) No 1882/2003 of the European Parliament and of the Council (OJ L 284, 31.10.2003, p. 1).

⁽³⁾ OJ L 364, 20.12.2006, p. 5.

⁽⁴⁾ OJ L 77, 16.3.2001, p. 14. Directive as last amended by Directive 2005/4/EC (OJ L 19, 21.1.2005, p. 50).

⁽⁵⁾ OJ L 42, 13.2.2004, p. 16.

⁽⁶⁾ OJ L 34, 8.2.2005, p. 15.

Article 2

Directives 2001/22/EC, 2004/16/EC and 2005/10/EC are hereby repealed.

References to the repealed Directives shall be construed as references to this Regulation.

Article 3

This Regulation shall enter into force on the 20th day following its publication in the *Official Journal of the European Union*.

It shall apply from 1 June 2007.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 28 March 2007.

For the Commission
Markos KYPRIANOU
Member of the Commission

ANNEX

PART A

DEFINITIONS

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

- 'lot': an identifiable quantity of food delivered at one time and determined by the official to have common characteristics, (such as origin, variety, type of packing, packer, consignor or markings). In the case of fish, also the size of fish shall be comparable;
- 'sublot': designated part of a large lot in order to apply the sampling method on that designated part. Each sublot must be physically separated and identifiable;
- 'incremental sample': a quantity of material taken from a single place in the lot or sublot;
- 'aggregate sample': the combined total of all the incremental samples taken from the lot or sublot; aggregate samples shall be considered as representative of the lots or sublots from which they are taken;
- 'laboratory sample': a sample intended for the laboratory.

PART B

SAMPLING METHODS**B.1. GENERAL PROVISIONS****B.1.1. Personnel**

Sampling shall be performed by an authorised person as designated by the Member State.

B.1.2. Material to be sampled

Each lot or sublot which is to be examined shall be sampled separately.

B.1.3. Precautions to be taken

In the course of sampling, precautions shall be taken to avoid any changes which would affect the levels of contaminants, adversely affect the analytical determination or make the aggregate samples unrepresentative.

B.1.4. Incremental samples

As far as possible, incremental samples shall be taken at various places distributed throughout the lot or sublot. Departure from such procedure shall be recorded in the record provided for under point B.1.8. of this Annex.

B.1.5. Preparation of the aggregate sample

The aggregate sample shall be made up by combining the incremental samples.

B.1.6. Samples for enforcement, defence and referee purposes

The samples for enforcement, defence and referee purposes shall be taken from the homogenised aggregate sample unless this conflicts with the rules of the Member States as regards the rights of the food business operator.

B.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 to 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.

B.1.8. Sealing and labelling of samples

Each sample taken for official use shall be sealed at the place of sampling and identified following the rules of the Member States.

A record shall be kept of each sampling, permitting each lot or subplot to be identified unambiguously (reference to the lot number shall be given) and giving the date and place of sampling together with any additional information likely to be of assistance to the analyst.

B.2. SAMPLING PLANS

Large lots shall be divided into sublots on condition that the subplot may be separated physically. For products traded in bulk consignments (e.g. cereals), Table 1 shall apply. For other products Table 2 shall apply. Taking into account that the weight of the lot is not always an exact multiple of the weight of the sublots, the weight of the subplot may exceed the mentioned weight by a maximum of 20 %.

The aggregate sample shall be at least 1 kg or 1 litre except where it is not possible e.g. when the sample consists of 1 package or unit.

The minimum number of incremental samples to be taken from the lot or subplot shall be as given in Table 3.

In the case of bulk liquid products the lot or subplot shall be thoroughly mixed in so far as possible and in so far it does not affect the quality of the product, by either manual or mechanical means immediately prior to sampling. In this case, a homogeneous distribution of contaminants is assumed within a given lot or subplot. It is therefore sufficient to take three incremental samples from a lot or subplot to form the aggregate sample.

The incremental samples shall be of similar weight. The weight of an incremental sample shall be at least 100 grams or 100 millilitres, resulting in an aggregate sample of at least about 1 kg or 1 litre. Departure from this method shall be recorded in the record provided for under point B.1.8. of this Annex.

Table 1

Subdivision of lots into sublots for products traded in bulk consignments

Lot weight (ton)	Weight or number of sublots
≥ 1 500	500 tonnes
> 300 and < 1 500	3 sublots
≥ 100 and ≤ 300	100 tonnes
< 100	—

Table 2

Subdivision of lots into sublots for other products

Lot weight (ton)	Weight or number of sublots
≥ 15	15 to 30 tonnes
< 15	—

Table 3

Minimum number of incremental samples to be taken from the lot or subplot

Weight or volume of lot/sublot (in kg or litre)	Minimum number of incremental samples to be taken
< 50	3
≥ 50 and ≤ 500	5
> 500	10

If the lot or subplot consists of individual packages or units, then the number of packages or units which shall be taken to form the aggregate sample is given in Table 4.

Table 4

Number of packages or units (incremental samples) which shall be taken to form the aggregate sample if the lot or subplot consists of individual packages or units

Number of packages or units in the lot/sublot	Number of packages or units to be taken
≤ 25	at least one package or unit
26 to 100	about 5 %, at least two packages or units
> 100	about 5 %, at maximum 10 packages or units

The maximum levels for inorganic tin apply to the contents of each can, but for practical reasons it is necessary to use an aggregate sampling approach. If the result of the test for an aggregate sample of cans is less than, but close to, the maximum level of inorganic tin and if it is suspected that individual cans might exceed the maximum level, then it might be necessary to conduct further investigations.

B.3. SAMPLING AT RETAIL STAGE

Sampling of foodstuffs at retail stage shall be done where possible in accordance with the sampling provisions set out in points B.1. and B.2. of this Annex.

Where this is not possible, an alternative method of sampling at retail stage may be used provided that it ensures sufficient representativeness for the sampled lot or subplot.

PART C**SAMPLE PREPARATION AND ANALYSIS****C.1. LABORATORY QUALITY STANDARDS**

Laboratories shall comply with the provisions of Article 12 of Regulation (EC) No 882/2004 ⁽¹⁾.

Laboratories shall participate in appropriate proficiency testing schemes which comply with the 'International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories' ⁽²⁾ developed under the auspices of IUPAC/ISO/AOAC.

Laboratories shall be able to demonstrate that they have internal quality control procedures in place. Examples of these are the 'ISO/AOAC/IUPAC Guidelines on Internal Quality Control in Analytical Chemistry Laboratories' ⁽³⁾.

⁽¹⁾ As amended by Article 18 of Commission Regulation (EC) No 2076/2005 (OJ L 338, 22.12.2005, p. 83).

⁽²⁾ The international harmonized protocol for the proficiency testing of analytical chemistry laboratories' by M. Thompson, S.L.R. Ellison and R. Wood, Pure Appl. Chem., 2006, 78, 145-96.

⁽³⁾ Edited by M. Thompson and R. Wood, Pure Appl. Chem., 1995, 67, 649-666.

Wherever possible the trueness of analysis shall be estimated by including suitable certified reference materials in the analysis.

C.2. SAMPLE PREPARATION

C.2.1. Precautions and general considerations

The basic requirement is to obtain a representative and homogeneous laboratory sample without introducing secondary contamination.

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

Compliance with maximum levels laid down in Regulation (EC) No 1881/2006 shall be established on the basis of the levels determined in the laboratory samples.

C.2.2. Specific sample preparation procedures

C.2.2.1. Specific procedures for lead, cadmium, mercury and inorganic tin

The analyst shall ensure that samples do not become contaminated during sample preparation. Wherever possible, apparatus and equipment coming into contact with the sample shall not contain those metals to be determined and be made of inert materials e.g. plastics such as polypropylene, polytetrafluoroethylene (PTFE) etc. These should be acid cleaned to minimise the risk of contamination. High quality stainless steel may be used for cutting edges.

There are many satisfactory specific sample preparation procedures which may be used for the products under consideration. Those described in the CEN Standard 'Foodstuffs — Determination of trace elements — Performance criteria, general considerations and sample preparation' ⁽¹⁾ have been found to be satisfactory but others may be equally valid.

In the case of inorganic tin, care shall be taken to ensure that all the material is taken into solution as losses are known to occur readily, particularly because of hydrolysis to insoluble hydrated Sn(IV) oxide species.

C.2.2.2. Specific procedures for benzo(a)pyrene

The analyst shall ensure that samples do not become contaminated during sample preparation. Containers shall be rinsed with high purity acetone or hexane before use to minimise the risk of contamination. Wherever possible, apparatus and equipment coming into contact with the sample shall be made of inert materials such as aluminium, glass or polished stainless steel. Plastics such as polypropylene or PTFE shall be avoided because the analyte can adsorb onto these materials.

C.2.3. Treatment of the sample as received in the laboratory

The complete aggregate sample shall be finely ground (where relevant) and thoroughly mixed using a process that has been demonstrated to achieve complete homogenisation.

C.2.4. Samples for enforcement, defence and referee purposes

The samples for enforcement, defence and referee purposes shall be taken from the homogenised material unless this conflicts with the rules of the Member States on sampling as regards the rights of the food business operator.

⁽¹⁾ Standard EN 13804:2002, 'Foodstuffs — Determination of trace elements — Performance criteria, general considerations and sample preparation', CEN, Rue de Stassart 36, B-1050 Brussels.

C.3. METHODS OF ANALYSIS

C.3.1. Definitions

The following definitions shall apply:

- 'r' = Repeatability the value below which the absolute difference between single test results obtained under repeatability conditions (i.e., same sample, same operator, same apparatus, same laboratory, and short interval of time) may be expected to lie within a specific probability (typically 95 %) and hence $r = 2,8 \times s_r$.
- 's_r' = Standard deviation calculated from results generated under repeatability conditions.
- 'RSD_r' = Relative standard deviation calculated from results generated under repeatability conditions $[(s_r/\bar{x}) \times 100]$.
- 'R' = Reproducibility the value below which the absolute difference between single test results obtained under reproducibility conditions (i.e., on identical material obtained by operators in different laboratories, using the standardised test method), may be expected to lie within a certain probability (typically 95 %); $R = 2,8 \times s_R$.
- 's_R' = Standard deviation, calculated from results under reproducibility conditions.
- 'RSD_R' = Relative standard deviation calculated from results generated under reproducibility conditions $[(s_R/\bar{x}) \times 100]$.
- 'LOD' = Limit of detection, smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty. The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations ($n > 20$).
- 'LOQ' = Limit of quantification, lowest content of the analyte which can be measured with reasonable statistical certainty. If both accuracy and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to six or 10 times the standard deviation of the mean of blank determinations ($n > 20$).
- 'HORRAT_r' = The observed RSD_r divided by the RSD_r value estimated from the Horwitz equation ⁽¹⁾ using the assumption $r = 0,66R$.
- 'HORRAT_R' = The observed RSD_R value divided by the RSD_R value calculated from the Horwitz equation.
- 'u' = Standard measurement uncertainty.
- 'U' = The expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95 % ($U = 2u$).
- 'U_f' = Maximum standard measurement uncertainty.

C.3.2. General requirements

Methods of analysis used for food control purposes shall comply with the provisions of points 1 and 2 of Annex III to Regulation (EC) No 882/2004.

Methods of analysis for total tin are appropriate for official control on inorganic tin levels.

For the analysis of lead in wine, Commission Regulation (EEC) No 2676/90 ⁽²⁾ lays down the method to be used in chapter 35 of its Annex.

C.3.3. Specific requirements

C.3.3.1. Performance criteria

Where no specific methods for the determination of contaminants in foodstuffs are prescribed at Community level, laboratories may select any validated method of analysis (where possible, the validation shall include a certified reference material) provided the selected method meets the specific performance criteria set out in Tables 5 to 7.

⁽¹⁾ M. Thompson, Analyst, 2000, 125, 385-386.

⁽²⁾ OJ L 272, 3.10.1990, p. 1. Regulation as last amended by Regulation (EC) No 1293/2005 (OJ L 205, 6.8.2005, p. 12).

Table 5

Performance criteria for methods of analysis for lead, cadmium, mercury and inorganic tin

Parameter	Value/Comment
Applicability	Foods specified in Regulation (EC) No 1881/2006
LOD	For inorganic tin less than 5 mg/kg. For other elements less than one tenth of the maximum level in Regulation (EC) No 1881/2006, except if the maximum level for lead is less than 100 µg/kg. For the latter, less than one fifth of the maximum level
LOQ	For inorganic tin less than 10 mg/kg. For other elements less than one fifth of the maximum level in Regulation (EC) No 1881/2006, except if the maximum level for lead is less than 100 µg/kg. For the latter, less than two fifth of the maximum level
Precision	HORRAT _r or HORRAT _R values of less than 2
Recovery	The provisions of point D.1.2. apply
Specificity	Free from matrix or spectral interferences

Table 6

Performance criteria for methods of analysis for 3-MCPD

Criterion	Recommended Value	Concentration
Field blanks	Less than the LOD	—
Recovery	75 to 110 %	all
LOD	5 µg/kg (or less) on a dry matter basis	
LOQ	10 µg/kg (or less) on a dry matter basis	—
Precision	< 4 µg/kg	20 µg/kg
	< 6 µg/kg	30 µg/kg
	< 7 µg/kg	40 µg/kg
	< 8 µg/kg	50 µg/kg
	< 15 µg/kg	100 µg/kg

Table 7

Performance criteria for methods of analysis for benzo(a)pyrene

Parameter	Value/Comment
Applicability	Foods specified in Regulation (EC) No 1881/2006
LOD	Less than 0,3 µg/kg
LOQ	Less than 0,9 µg/kg
Precision	HORRAT _r or HORRAT _R values of less than 2
Recovery	50 to 120 %
Specificity	Free from matrix or spectral interferences, verification of positive detection

C.3.3.2. 'Fitness-for-purpose' approach

Where a limited number of fully validated methods of analysis exist, alternatively, a 'fitness-for-purpose' approach may be used to assess the suitability of the method of analysis. Methods suitable for official control must produce results with standard measurement uncertainties less than the maximum standard measurement uncertainty calculated using the formula below:

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

where:

U_f is the maximum standard measurement uncertainty ($\mu\text{g}/\text{kg}$);

LOD is the limit of detection of the method ($\mu\text{g}/\text{kg}$);

C is the concentration of interest ($\mu\text{g}/\text{kg}$);

α is a numeric factor to be used depending on the value of C . The values to be used are given in Table 8.

Table 8

Numeric values to be used for α as constant in formula set out in this point, depending on the concentration of interest

C ($\mu\text{g}/\text{kg}$)	α
≤ 50	0,2
51 to 500	0,18
501 to 1 000	0,15
1 001 to 10 000	0,12
$> 10\ 000$	0,1

PART D

REPORTING AND INTERPRETATION OF RESULTS

D.1. REPORTING

D.1.1. Expression of results

The results shall be expressed in the same units and with the same number of significant figures as the maximum levels laid down in Regulation (EC) No 1881/2006.

D.1.2. Recovery calculations

If an extraction step is applied in the analytical method, the analytical result shall be corrected for recovery. In this case the level of recovery must be reported.

In case no extraction step is applied in the analytical method (e.g. in case of metals), the result may be reported uncorrected for recovery if evidence is provided by ideally making use of suitable certified reference material that the certified concentration allowing for the measurement uncertainty is achieved (i.e. high accuracy of the measurement). In case the result is reported uncorrected for recovery this shall be mentioned.

D.1.3. Measurement uncertainty

The analytical result shall be reported as $x \pm U$ whereby x is the analytical result and U is the expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95 % ($U = 2u$).

The analyst shall note the 'Report on the relationship between analytical results, measurement uncertainty, recovery factors and the provisions in EU food and feed legislation' ⁽¹⁾.

D.2. INTERPRETATION OF RESULTS**D.2.1. Acceptance of a lot/sublot**

The lot or subplot is accepted if the analytical result of the laboratory sample does not exceed the respective maximum level as laid down in Regulation (EC) No 1881/2006 taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.

D.2.2. Rejection of a lot/sublot

The lot or subplot is rejected if the analytical result of the laboratory sample exceeds beyond reasonable doubt the respective maximum level as laid down in Regulation (EC) No 1881/2006 taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.

D.2.3. Applicability

The present interpretation rules shall apply for the analytical result obtained on the sample for enforcement. In case of analysis for defence or reference purposes, the national rules shall apply.

⁽¹⁾ http://europa.eu.int/comm/food/food/chemicalsafety/contaminants/sampling_en.htm