COMMISSION REGULATION (EU) No 836/2011

of 19 August 2011

amending Regulation (EC) No 333/2007 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 EUROPEAN COMMISSION,

Having regard to the Treaty on the Functioning of the European Union,

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) Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs [2] established, inter alia, maximum levels for the contaminant benzo(a)pyrene.

(2) The Scientific Panel on Contaminants in the Food Chain of the European Food Safety Authority (EFSA) adopted an opinion on Polycyclic Aromatic Hydrocarbons in Food on 9 June 2008 [3]. The EFSA concluded that benzo(a)pyrene is not a suitable marker for the occurrence of polycyclic aromatic hydrocarbons (PAH) in food and that a system of four specific substances or eight specific substances would be the most suitable markers of PAH in food. The EFSA also concluded that a system of eight substances would not provide much added value compared to a system of four substances.

(3) As a consequence Commission Regulation (EU) No 835/2011 [4] amended Regulation (EC) No 1881/2006 in order to set maximum levels for the sum of four polycyclic aromatic hydrocarbons (benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene).

(4) Commission Regulation (EC) No 333/2007 [5] lays down analytical performance criteria only for benzo(a)pyrene. It is therefore necessary to lay down analytical performance criteria for the other three substances for which maximum levels are now set out in Regulation (EC) No 1881/2006.

(5) The European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons (EU-RL PAH) in collaboration with the national reference laboratories carried out a survey among official control laboratories to assess which analytical performance criteria would be achievable for benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene in relevant food matrices. The outcome of this survey was summarised by the EU-RL PAH in the Report on 'Performance characteristics of analysis methods for the determination of 4 polycyclic aromatic hydrocarbons in food' [6]. The results of the survey show that the analytical performance criteria currently applicable to benzo(a)pyrene are also suitable for the other three substances.

(6) Experience acquired while implementing Regulation (EC) No 333/2007 revealed that in some cases the current sampling provisions may be impracticable or may lead to unacceptable economic damage to the sampled lot. For such cases, departure from the sampling procedures should be allowed, provided that sampling remains sufficiently representative of the sampled lot or sublot and that the procedure used is fully documented. For sampling at the retail stage, flexibility to depart from the sampling procedures existed already. The provisions for sampling at retail stage should be aligned with the general sampling procedures.

(7) More detailed provisions are needed as regards the material of sampling containers when samples are taken for PAH analysis. Plastic containers are widely used by enforcement authorities, but they are not suitable when sampling is carried out for PAH analysis, as the PAH content of the sample can be altered by these materials.

(8) Clarification is needed for some aspects of the specific requirements for analytical methods, in particular the requirements regarding the use of the performance criteria and the 'fitness-for-purpose' approach. Furthermore, the presentation of the tables with the performance criteria should be modified to appear more uniform across all analytes.

(9) Regulation (EC) No 333/2007 should therefore be amended accordingly. Since Regulation (EU) No 835/2011 and this Regulation are inter-linked, both Regulations should become applicable on the same date.

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[1] OJ L 165, 30.4.2004, p. 1.
[2] OJ L 364, 20.12.2006, p. 5.
[3] The EFSA Journal (2008) 724, p. 1.
[4] See page 4 of this Official Journal.
[5] OJ L 88, 29.3.2007, p. 29.

[6] JRC Report 59046, 2010.

(10) The measures provided for in this Regulation are in accordance with the opinion of the Standing Committee on the Food Chain and Animal Health and neither the European Parliament nor the Council have opposed them,

HAS ADOPTED THIS REGULATION:

Article 1

Regulation (EC) No 333/2007 is amended as follows:

(1) the title is replaced by the following:

'**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 polycyclic aromatic hydrocarbons in foodstuffs**';

(2) in Article 1, paragraph 1 is replaced by the following:

'1. Sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and polycyclic aromatic hydrocarbons ("PAH") 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.';

(3) the Annex is amended in accordance with the Annex to this Regulation.

Article 2

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 September 2012.

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

Done at Brussels, 19 August 2011.

For the Commission
The President
José Manuel BARROSO

ANNEX

The Annex to Regulation (EC) No 333/2007 is amended as follows:

(1) in point B.1.7 'Packaging and transmission of samples', the following second paragraph is added:

'In case of sampling for PAH analysis plastic containers shall be avoided if possible as they could alter the PAH content of the sample. Inert, PAH-free glass containers, adequately protecting the sample from light, shall be used wherever possible. Where this is practically impossible, at least direct contact of the sample with plastics shall be avoided, e.g. in case of solid samples by wrapping the sample in aluminium foil before placing it in the sampling container.';

(2) points B.2 and B.3 are replaced by the following:

'B.2. SAMPLING PLANS

B.2.1. **Division of lots into sublots**

Large lots shall be divided into sublots on condition that the sublot 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 sublot may exceed the mentioned weight by a maximum of 20 %.

B.2.2. **Number of incremental samples**

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 sublot shall be as given in Table 3.

In the case of bulk liquid products the lot or sublot 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 sublot. It is therefore sufficient to take three incremental samples from a lot or sublot to form the aggregate sample.

The incremental samples shall be of similar weight/volume. The weight/volume 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	15-30 tonnes
< 15	—

Table 3

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

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 sublot 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 sublot 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 1 package or unit
26-100	about 5 %, at least 2 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.

Where it is not possible to carry out the method of sampling set out in this chapter because of the unacceptable commercial consequences (e.g. because of packaging forms, damage to the lot, etc.) or where it is practically impossible to apply the abovementioned method of sampling, an alternative method of sampling may be applied provided that it is sufficiently representative for the sampled lot or sublot and is fully documented.

B.2.3. **Specific provisions for the sampling of large fish arriving in large lots**

In case the lot or sublot to be sampled contains large fishes (individual fishes weighing more than about 1 kg) and the lot or sublot weighs more than 500 kg, the incremental sample shall consist of the middle part of the fish. Each incremental sample shall weigh at least 100 g.

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 point B.2.2 of this Annex.

Where it is not possible to carry out the method of sampling set out in point B.2.2 because of the unacceptable commercial consequences (e.g. because of packaging forms, damage to the lot, etc.) or where it is practically impossible to apply the abovementioned method of sampling, an alternative method of sampling may be applied provided that it is sufficiently representative for the sampled lot or sublot and is fully documented.';

(3) in the first paragraph of point C.1 'Laboratory Quality Standards', footnote 1 is deleted;

(4) in point C.2.2.1 'Specific procedures for lead, cadmium, mercury and inorganic tin', the second paragraph is replaced by the following:

'There are many satisfactory specific sample preparation procedures which may be used for the products under consideration. For those aspects not specifically covered by this Regulation, the CEN Standard "Foodstuffs - Determination of trace elements – Performance criteria, general considerations and sample preparation"([1]) has been found to be satisfactory but other sample preparation methods may be equally valid.';

(5) point C.2.2.2 is replaced by the following:

'C.2.2.2. Specific procedures for polycyclic aromatic hydrocarbons

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 analytes can adsorb onto these materials.';

(6) point C.3.1 'Definitions' is amended as follows:

(a) the definition for 'HORRAT$_r$' is replaced by the following:

'HORRAT (*)$_r$ = The observed RSD_r divided by the RSD_r value estimated from the (modified) Horwitz equation (**) (cf. point C.3.3.1 ("Notes to the performance criteria")) using the assumption r = 0,66 R.

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(*) Horwitz W. and Albert, R., 2006, The Horwitz Ratio (HorRat): A useful Index of Method Performance with respect to Precision, Journal of AOAC International, Vol. 89, 1095-1109.
(**) M. Thompson, Analyst, 2000, p. 125 and 385-386.'

(b) the definition for 'HORRAT$_R$' is replaced by the following:

'HORRAT (*)$_R$ = The observed RSD_R divided by the RSD_R value estimated from the (modified) Horwitz equation (**) (cf. point C.3.3.1 ("Notes to the performance criteria")).

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(*) Horwitz W. and Albert, R., 2006, The Horwitz Ratio (HorRat): A useful Index of Method Performance with respect to Precision, Journal of AOAC International, Vol. 89, 1095-1109.
(**) M. Thompson, Analyst, 2000, p. 125 and 385-386.'

(c) the definition for 'u' is replaced by the following:

'u = Combined standard measurement uncertainty obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model (*)

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(*) International vocabulary of metrology – Basic and general concepts and associated terms (VIM), JCGM 200:2008.';

(7) point C.3.2 is replaced by the following:

'C.3.2 **General requirements**

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

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

For the analysis of lead in wine, the methods and rules established by the OIV (*) apply in accordance with Article 31 of Council Regulation (EC) No 479/2008 (**).

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(*) Organisation internationale de la vigne et du vin.
(**) Council Regulation (EC) No 479/2008 of 29 April 2008 on the common organisation of the market in wine amending Regulations (EC) No 1493/1999, (EC) No 1782/2003, (EC) No 1290/2005, (EC) No 3/2008 and repealing Regulations (EEC) No 2392/86 and (EC) No 1493/1999 (OJ L 148, 6.6.2008, p. 1).';

(8) point C.3.3.1 is replaced by the following:

'C.3.3.1. Performance criteria

Where no specific methods for the determination of contaminants in foodstuffs are prescribed at European Union level, laboratories may select any validated method of analysis for the respective matrix provided that the selected method meets the specific performance criteria set out in Tables 5, 6 and 7.

It is recommended that fully validated methods (i.e. methods validated by collaborative trial for the respective matrix) are used where appropriate and available. Other suitable validated methods (e.g. in-house validated methods for the respective matrix) may also be used provided that they fulfil the performance criteria set out in Tables 5, 6 and 7.

Where possible, the validation of in-house validated methods shall include a certified reference material.

(a) Performance criteria for methods of analysis for lead, cadmium, mercury and inorganic tin:

Table 5

Parameter	Criterion		
Applicability	Foods specified in Regulation (EC) No 1881/2006		
Specificity	Free from matrix or spectral interferences		
Repeatability (RSD$_r$)	HORRAT$_r$ less than 2		
Reproducibility (RSD$_R$)	HORRAT$_R$ less than 2		
Recovery	The provisions of point D.1.2 apply		
	Inorganic tin	Lead, cadmium, mercury	
		ML is < 0,100 mg/kg	ML is ≥ 0,100 mg/kg
LOD	≤ 5 mg/kg	≤ one fifth of the ML	≤ one tenth of the ML
LOQ	≤ 10 mg/kg	≤ two fifths of the ML	≤ one fifth of the ML

(b) Performance criteria for methods of analysis for 3-MCPD:

Table 6

Parameter	Criterion
Applicability	Foods specified in Regulation (EC) No 1881/2006
Specificity	Free from matrix or spectral interferences
Field blanks	Less than LOD
Repeatability (RSD$_r$)	0,66 times RSD$_R$ as derived from (modified) Horwitz equation
Reproducibility (RSD$_R$)	as derived from (modified) Horwitz equation
Recovery	75-110 %
LOD	≤ 5 µg/kg (on dry matter basis)
LOQ	≤ 10 µg/kg (on dry matter basis)

(c) Performance criteria for methods of analysis for polycyclic aromatic hydrocarbons:

The four polycyclic aromatic hydrocarbons to which these criteria apply are benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene.

Table 7

Parameter	Criterion
Applicability	Foods specified in Regulation (EC) No 1881/2006
Specificity	Free from matrix or spectral interferences, verification of positive detection
Repeatability (RSD$_r$)	HORRAT$_r$ less than 2
Reproducibility (RSD$_R$)	HORRAT$_R$ less than 2

Parameter	Criterion
Recovery	50-120 %
LOD	≤ 0,30 µg/kg for each of the four substances
LOQ	≤ 0,90 µg/kg for each of the four substances

(d) Notes to the performance criteria:

The Horwitz equation (*) (for concentrations $1,2 \times 10^{-7} \leq C \leq 0,138$) and the modified Horwitz equation (**) (for concentrations $C < 1,2 \times 10^{-7}$) are generalised precision equations which are independent of analyte and matrix but solely dependent on concentration for most routine methods of analysis.

Modified Horwitz equation for concentrations $C < 1,2 \times 10^{-7}$:

$RSD_R = 22 \%$

where:

— RSD_R is the relative standard deviation calculated from results generated under reproducibility conditions $[(s_R / \bar{x}) \times 100]$

— C is the concentration ratio (i.e. 1 = 100 g/100 g, 0,001 = 1 000 mg/kg). The modified Horwitz equation applies to concentrations $C < 1,2 \times 10^{-7}$.

Horwitz equation for concentrations $1,2 \times 10^{-7} \leq C \leq 0,138$:

$RSD_R = 2C^{(-0,15)}$

where:

— RSD_R is the relative standard deviation calculated from results generated under reproducibility conditions $[(s_R / \bar{x}) \times 100]$

— C is the concentration ratio (i.e. 1 = 100 g/100 g, 0,001 = 1 000 mg/kg). The Horwitz equation applies to concentrations $1,2 \times 10^{-7} \leq C \leq 0,138$.

(*) W. Horwitz, L.R. Kamps, K.W. Boyer, J.Assoc.Off.Analy.Chem.,1980, 63, 1344.
(**) M. Thompson, Analyst, 2000, p. 125 and 385-386.';

(9) point C.3.3.2 is replaced by the following:

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

For in-house validated methods, as an alternative a "fitness-for-purpose" approach (*) may be used to assess their suitability for official control. Methods suitable for official control must produce results with a combined standard measurement uncertainty (u) less than the maximum standard measurement uncertainty calculated using the formula below:

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

where:

— Uf is the maximum standard measurement uncertainty (µg/kg).

— LOD is the limit of detection of the method (µg/kg). The LOD must meet the performance criteria set in point C.3.3.1 for the concentration of interest.

— C is the concentration of interest (µg/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 (µg/kg)	α
≤ 50	0,2
51-500	0,18

C (µg/kg)	α
501-1 000	0,15
1 001-10 000	0,12
> 10 000	0,1

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

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(*) M. Thompson and R. Wood, Accred. Qual. Assur., 2006, p. 10 and 471-478.
(**) http://ec.europa.eu/food/food/chemicalsafety/contaminants/report-sampling_analysis_2004_en.pdf;

(10) in point D.1.2 'Recovery calculations', the second paragraph is replaced by the following:

'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), and thus that the method is not biased. In case the result is reported uncorrected for recovery this shall be mentioned.';

(11) in point D.1.3 'Measurement uncertainty', the second paragraph is replaced by the following:

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

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(*) http://ec.europa.eu/food/food/chemicalsafety/contaminants/report-sampling_analysis_2004_en.pdf'.