L.N. 240 of 2003

FOOD SAFETY ACT, 2002
(ACT No. XIV of 2002)

Contaminants in Food (Sampling and Analysis Methods) Regulations, 2003

IN exercise of the powers conferred by article 10 of the Food Safety Act, the Minister of Health has made the following regulationsĠ

1. The title of these regulations is the Contaminants in Food (Sampling and Analysis Methods) Regulations, 2003. Title.

2.1 The sampling for the official control of the levels of aflatoxins in foodstuffs shall be carried out in accordance with the methods described in the First Schedule to these regulations. Sampling Methods for aflatoxins in foodstuffs.

2.2 The sample preparation and methods of analyses used for the official control of the levels of aflatoxins in foodstuffs shall comply with the criteria described in the Second Schedule to these regulations.

3.1 The sampling for the official control of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs shall be carried out in accordance with the methods described in the Third Schedule to these regulations. Sampling Methods for lead, cadmium, mercury and 3-MPCD in foodstuffs.

3.2 The sample preparation and methods of analyses used for the official control of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs shall comply with the criteria described in the Fourth Schedule to these regulations.

FIRST SCHEDULE
(Equivalent to Annex I of Commission Directive 98/53/EC)

Methods of sampling for official checking control of the levels of aflatoxins in certain foodstuffs

1. Purpose and scope

Samples intended for official checking of the levels of aflatoxin content in foodstuffs shall be taken according to the methods described below. Aggregate samples thus obtained shall be considered as representative of the lots. Compliance with maximum limits laid down in Commission Regulation (EC) No 1525/98 shall be established on the basis of the levels determined in the laboratory samples.

2. Definitions

Lot: an identifiable quantity of a food commodity delivered at one time and determined by the official to have common characteristics, such as origin, variety, type of packing, packer, consignor or markings.
Sublot: designated part of a large lot in order to apply the sampling method on that designated part. Each sublot must be physically separate 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.
Laboratory sample: sample intended for the laboratory (=subsample).

3. General provisions

3.1. Personnel
Sampling shall be performed by an official analyst as specified by the Act.

3.2. Material to be sampled
Each lot which is to be examined must be sampled separately. In accordance with the specific provisions in point 5 of this Schedule, large lots should be subdivided into sublots to be sampled separately.

3.3. Precautions to be taken
In the course of sampling and preparation of the laboratory samples precautions must be taken to avoid any changes which would affect the aflatoxin content, adversely affect the analytical determination or make the aggregate samples unrepresentative.

3.4. Incremental samples

As far as possible incremental samples should be taken at various places distributed throughout the lot or sublot. Departure from this procedure must be recorded in the record provided for in 3.8.

3.5. Preparation of the aggregate sample and the laboratory samples (subsamples)

The aggregate sample is made up by uniting and sufficiently mixing the incremental samples. After mixing, the aggregate sample must be divided into equal subsamples in accordance with the specific provisions of point 5 of this Schedule. The mixing is necessary to ensure that each subsample contains portions of the whole lot or sublot.

3.6. Replicate samples

The replicate samples for enforcement, trade (defence) and referee purposes are to be taken from the homogenised laboratory sample.

3.7. Packaging and transmission of laboratory samples

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

3.8. Sealing and labelling of laboratory samples

Each sample taken for official use shall be sealed at the place of sampling and identified. A record must be kept of each sampling, permitting each lot to be identified unambiguously and giving the date and place of sampling together with any additional information likely to be of assistance to the analyst.

4. Explanatory provisions

4.1. Different types of lots

Food commodities may be traded in bulk, containers, or individual packings (sacks, bags, retail packings, etc.). The sampling procedure can be applied to all the different forms in which the commodities are put on the market. Without prejudice to the specific provisions as laid down in point 5 of this Schedule, the following formula can be used as a guide for the sampling of lots traded in individual packings (sacks, bags, retail packings, etc.):

Sampling frequency (SF) = {Weight of the lot} x {Weight of the incremental sample}/{Weight of the aggregate sample} x {Weight of individual packing}

– Weight: in kg
– Sampling frequency (SF): every nth sack or bag from which an incremental sample must be taken (decimal figures should be rounded to the nearest whole number).

4.2. Weight of the incremental sample

The weight of the incremental sample should be about 300 grams unless otherwise defined in point 5 of this Schedule and with the exception of spices in which case the weight of the incremental sample is about 100 grams. In the case of retail packings, the weight of the incremental sample depends on the weight of the retail packing.

4.3. Number of incremental samples for lots of less than 15 tonnes

The number of incremental samples to be taken depends on the weight of the lot, with a minimum of 10 and a maximum of 100, unless otherwise defined in point 5 of this Schedule. The figures in the following table may be used to determine the number of incremental samples to be taken.

Table 1: Number of incremental samples to be taken depending on the weight of the lot

Lot weight (tonnes)	No of incremental samples
≤ 0.1	10
> 0.1 - ≤ 0.2	15
> 0.2 - ≤ 0.5	20
> 0.5 - ≤ 1.0	30
> 1.0 - ≤ 2.0	40
> 2.0 - ≤ 5.0	60
> 5.0 - ≤ 10.0	80
> 10.0 - ≤ 15.0	100

5. Specific provisions

5.1. General survey of the sampling procedure for groundnuts, nuts, dried fruit, spices and cereals

Table 2: Subdivision of lots into sublots depending on product and lot weight

Commodity	Lot weight (tonnes)	Weight or number of sublots	Number of incremental samples	Aggregate sample weight (kg)
Dried figs and other dried fruit	≥ 15 < 15	15-30 tonnes -	100 10-100 (*)	30 ≤ 30
Groundnuts, pistachios, Brazil nuts and other nuts	≥ 500 > 125 & < 500 ≥ 15 & ≤ 125 < 15	100 tonnes 5 sublots 25 tonnes -	100 100 100 10-100 (*)	30 30 30 ≤ 30
Cereals	≥ 1500 > 300 & < 1500 ≥ 50 & ≤ 300 < 50	500 tonnes 3 sublots 100 tonnes -	100 100 100 10-100 (*)	30 30 30 1-10

(*) Depending on the lot weight – see point 4.3 or 5.3 of this Schedule

Commodity	Lot weight (tonnes)	Weight or number of sublots	Number of incremental samples	Aggregate sample weight (kg)
Spices	≥ 15 < 15	25 tonnes -	100 10-100 (*)	10 1-10

5.2. Groundnuts, pistachios and Brazil nuts
Dried figs
Cereals (lots ≥ 50 tonnes)
Spices

5.2.1. Sampling procedure

– On condition that the sublot can be separated physically, each lot must be subdivided into sublots following Table 2 at point 5.1. 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 %,
– each sublot must be sampled separately,
– number of incremental samples: 100. In the case of lots under 15 tonnes, the number of incremental samples to be taken depends on the weight of the lot, with a minimum of 10 and a maximum of 100 (see point 4.3),
– weight of the aggregate sample = 30 kg which has to be mixed and to be divided into three equal subsamples of 10 kg before grinding (this division into three subsamples is not necessary in the case of groundnuts, nuts and dried fruit intended for further sorting or other physical treatment, however, this will depend upon the availability of equipment which is able to homogenise a 30 kg sample). In cases where the aggregate sample weights are under 10 kg, the aggregate sample must not be divided into three subsamples. In the case of spices the aggregate sample weighs not more than 10 kg and therefore no division in subsamples is necessary.
– laboratory sample: a subsample of 10 kg (each subsample must be separately ground finely and mixed thoroughly to achieve complete homogenisation, in accordance with the provisions laid down in the Second Schedule),
– if it is not possible to carry out the method of sampling described above because of the commercial consequences resulting from damage to the lot (because of packaging forms, means of transport, etc.) an alternative method of sampling may be applied provided that it is as representative as possible and is fully described and documented.

5.2.2. Acceptance of a lot or sublot

– For groundnuts, nuts and dried fruit subjected to a sorting or other physical treatment and spices:
  – acceptance if the aggregate sample or the average of the subsamples conforms to the maximum limit,

- rejection if the aggregate sample or the average of the subsamples exceeds the maximum limit,

– for groundnuts, nuts, dried fruit and cereals intended for direct human consumption:
  - acceptance if none of the subsamples exceeds the maximum limit,
  - rejection if one or more of the subsamples exceeds the maximum limit,

– where the aggregate sample is under 10 kg:
  - acceptance if the aggregate sample conforms to the maximum limit,
  - rejection if the aggregate sample exceeds the maximum limit.

5.3. Nuts other than groundnuts, pistachios and Brazil nuts
Dried fruit other than figs
Cereals (lots under 50 tonnes)

5.3.1. Sampling procedure

For these products, the sampling procedure laid down in point 5.2.1 may be applied. However, taking into account the low incidence of contamination for these products and/or the newer forms of packaging in which products can be traded, simpler sampling methods may be applied. For cereal lots under 50 tonnes, a sampling plan consisting of, depending on the lot weight, 10 to 100 incremental samples each of 100 grams, resulting in an aggregate sample of 1 to 10 kg may be used. The figures in the following table can be used to determine the number of incremental samples to be taken.

Table 3: Number of incremental samples to be taken depending on the weight of the lot of cereals

Lot weight (tonnes)	Number of incremental samples
≤ 1	10
> 1 - ≤ 3	20
> 3 - ≤ 10	40
> 10 - ≤ 20	60
> 20 - ≤ 50	100

5.3.2. Acceptance of a lot or sublot

See point 5.2.2.

5.4. Milk

5.4.1. Sampling procedure

Sampling in accordance with Commission Decision 91/180/EEC of 14 February 1991 laying down certain methods of analysis and testing of raw milk and heat-treated milk [1] :

– number of incremental samples: minimum 5,
– weight of aggregate sample: minimum 0,5 kg or litres.

5.4.2. Acceptance of a lot or sublot

– Acceptance if the aggregate sample conforms to the maximum limit,
– rejection if the aggregate sample exceeds the maximum limit.

5.5. Derived products and compound foods

5.5.1. Milk products

5.5.1.1. Sampling procedure

Sampling in accordance with Commission Directive 87/524/EEC of 6 October 1987 laying down Community methods of sampling for chemical analysis for the monitoring of preserved milk products [2].

Number of incremental samples: minimum 5.

For the other milk products an equivalent method of sampling is used.

5.5.1.2. Acceptance of a lot or sublot

– Acceptance if the aggregate sample conforms to the maximum limit,
– rejection if the aggregate sample exceeds the maximum limit.

5.5.2. Other derived products with very small particle weight, i.e. flour, fig paste, peanut butter (homogeneous distribution of aflatoxin contamination)

5.5.2.1. Sampling procedure

– Number of incremental samples: 100. For lots of under 50 tonnes the number of incremental samples should be 10 to 100, depending on the lot weight (see Table 3 at point 5.3.1 of this Schedule),
– the weight of the incremental sample should be about 100 grams. In the case of lots in retail packing, the weight of the incremental sample depends on the weight of the retail packing,
– weight of aggregate sample = 1-10 kg sufficiently mixed.

5.5.2.2. Number of samples to be taken

– The number of aggregate samples to be taken depends on the lot weight. The division of large lots into sublots must be done as defined for cereals in Table 2 under point 5.1,
– each sublot must be sampled separately.

[1] OJ L 93, 13. 4. 1991, p. 1.

[2] OJ L 306, 28. 10. 1987, p. 24.

5.5.2.3 Acceptance of a lot or sublot

- Acceptance if the aggregate sample conforms to the maximum limit,
- rejection if the aggregate sample exceeds the maximum limit.

5.6. Other derived products with a relatively large particle size (heterogeneous distribution of aflatoxin contamination)

Sampling procedure and acceptance as defined at points 5.2 and 5.3 of this Schedule for the raw agricultural product.

6. Sampling at retail stage

Sampling of foodstuffs at the retail stage should be done where possible in accordance with the above sampling provisions. Where this is not possible, other effective sampling procedures at retail stage can be used provided that they ensure sufficient representativeness for the sampled lot.

SECOND SCHEDULE
(Equivalent to Annex II of Commission Directive 98/53/EC)

Sample preparation and criteria for methods of analysis used in official checking of the levels of aflatoxins in certain foodstuffs

1. Introduction

1.1. Precautions

Daylight should be excluded as much as possible during the procedure, since aflatoxin gradually breaks down under the influence of ultra-violet light. As the distribution of aflatoxin is extremely non-homogeneous, samples should be prepared – and especially homogenised – with extreme care. All the material received by the laboratory is to be used for the preparation of test material.

1.2. Calculation of proportion of shell/kernel of whole nuts

The limits fixed for aflatoxins in the Contaminants in Food Regulations apply to the edible part. The level of aflatoxins in the edible part can be determined by:

– shelling samples of nuts `in shell' and the level of aflatoxins is directly determined in the edible part,
– homogenise the nuts `in shell' by taking them through the sample preparation procedure. The sampling and analytical procedure must estimate the weight of nut kernel in the aggregate sample. The weight of nut kernel in the aggregate sample is estimated after establishing a suitable factor for the proportion of nut shell to nut kernel in whole nuts. This proportion is used to ascertain the amount of kernel in the bulk sample taken through the sample preparation and analysis procedure. Approximately 100 whole nuts are taken at random separately from the lot or are to be put aside from each aggregate sample. The ratio may, for each laboratory sample, be obtained by weighing the whole nuts, shelling and re-weighing the shell and kernel portions. However, the proportion of shell to kernel may be established by the laboratory from a number of samples and so can be assumed for future analytical work. But if a particular laboratory sample is found to be in contravention of any limit, the proportion should be determined for that sample using the approximately 100 nuts that have been set aside.

2. Treatment of the sample as received in the laboratory

Finely grind and mix thoroughly each laboratory sample using a process that has been demonstrated to achieve complete homogenisation.

3. Subdivision of samples for enforcement and defence purposes

The replicate samples for enforcement, trade (defence) and referee purposes shall be taken from the homogenized material.

4. Method of analysis to be used by the laboratory and laboratory control requirements

4.1. Definitions

A number of the most commonly used definitions that the laboratory will be required to use are given below:

The most commonly quoted precision parameters are repeatability and reproducibility.

r = repeatability, the value below which the absolute difference between two 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 $r = 2.8 \cdot s_r$

s_r = Standard deviation, calculated from results generated under repeatability conditions

RSD_r = relative standard deviation, calculated from results generated under repeatability conditions $[(S_r/x) \cdot 100]$, where x is the average of results over all laboratories and samples

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 %); $R = 2.8\ s_R$

s_R = standard deviation, calculated from results under reproducibility conditions

RSD_R = relative standard deviation calculated from results generated under reproducibility conditions $[(S_R/x).100]$

4.2. General requirements

Methods of analysis used for food control purposes must comply whenever possible with the provisions of points 1 and 2 of the Annex to Council Directive 85/591/EEC.

4.3. Specific requirements

Where no specific methods for the determination of aflatoxin levels in foodstuffs are prescribed at Community level, laboratories may select any method provided the selected method meets the following criteria:

Criterion	Concentration range	Recommended value	Maximum permitted value
Blanks	All	Negligible	
Recovery – Aflatoxin M1	0.01 – 0.05 µg/kg > 0.05 µg/kg	60 to120 % 70 to 110 %	
Recovery – Aflatoxins B_1, B_2, G_1, G_2	< 1.0 µg/kg 1-10 µg/kg > 10 µg/kg	50 to 120 % 70 to110 % 80 to 110 %	
Precision RSD_R	All	As derived from Horwitz equation	2 x value derived from Horwitz equation

Precision RSD_R may be calculated as 0.66 times precision RSD_R at the concentration of interest.

Notes:

– Values to apply to both B_1 and sum of $B_1+B_2+G_1+G_2$,

– if sum of individual aflatoxins $B_1+B_2+G_1+G_2$ are to be reported, then response of each to the analytical system must be either known or equivalent,

– the detection limits of the methods used are not stated as the precision values are given at the concentrations of interest,

– the precision values are calculated from the Horwitz equation, i. e.:
$RSD_R = 2^{(1-0,5 \log C)}$
where:
  – RSD_R is the relative standard deviation calculated from results generated under reproducibility conditions $[(S_R/x) .100]$
  – C is the concentration ratio (i. e. 1 = 100 g/100 g, 0,001 = 1 000 mg/kg).
This is a generalised precision equation which has been found to be independent of analyte and matrix but solely dependent on concentration for most routine methods of analysis.

4.4. Recovery calculation

The analytical result is to be reported corrected or uncorrected for recovery. The manner of reporting and the level of recovery must be reported.

4.5. Laboratory quality standards

Laboratories must comply with Council Directive 93/99/EEC.

THIRD SCHEDULE
(Equivalent to Annex I of Commission Directive 2001/22/EC)

Methods Of Sampling For Offical Control Of The Levels Of Lead, Cadmium, Mercury And 3-MPCD In Certain Foodstuffs

1. PURPOSE AND SCOPE

Samples intended for the official control of the levels of lead, cadmium, mercury and 3-MCPD contents in foodstuffs shall be taken according to the methods described below. Aggregate samples thus obtained shall be considered as representative of the lots or sublots from which they are taken. Compliance with maximum levels laid down in the Contaminants in Food Regulations shall be established on the basis of the levels determined in the laboratory samples.

2. DEFINITIONS

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.

Laboratory sample: sample intended for the laboratory

3. GENERAL PROVISIONS

3.1. **Personnel**
Sampling shall be performed by an authorised analyst as specified by the Act.

3.2. **Material to be sampled**
Each lot which is to be examined must be sampled separately.

3.3. **Precautions to be taken**
In the course of sampling and preparation of laboratory samples precautions must be taken to avoid any changes which would affect the lead, cadmium, mercury and 3-MCPD contents, adversely affect the analytical determination or make the aggregate samples unrepresentative.

3.4. **Incremental samples**

As far as possible incremental samples shall be taken at various places distributed throughout the lot or sublot. Departure from this procedure must be recorded in the record provided for under 3.8.

3.5. **Preparation of the aggregate sample**

The aggregate sample is made up by uniting all incremental samples. It shall be at least 1 kg unless not practical, e.g. when a single package has been sampled.

3.6. **Subdivision of aggregate sample in laboratory samples for enforcement, defence and referee purposes**

The laboratory samples for enforcement, trade (defence) and referee purposes shall be taken from the homogenised aggregate sample. The size of the laboratory samples for enforcement shall be sufficient to allow at least for duplicate analyses.

3.7. **Packaging and transmission of aggregate and laboratory samples**

Each aggregate and laboratory 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 change of composition of the aggregate and laboratory samples which might arise during transportation or storage.

3.8. **Sealing and labelling of aggregate and laboratory samples**

Each sample taken for official use shall be sealed at the place of sampling and identified. A record must be kept of each sampling, permitting each lot to be identified unambiguously and giving the date and place of sampling together with any additional information likely to be of assistance to the analyst.

4. SAMPLING PLANS

Sampling should ideally take place at the point where the commodity enters the food chain and a discrete lot becomes identifiable. The sampling method applied shall ensure that the aggregate sample is representative for the lot that is to be controlled.

4.1. **Number of incremental samples**

In the case of liquid products for which a homogeneous distribution of the contaminant in question can be assumed within a given lot, it is sufficient to take one incremental sample per lot which forms the aggregate sample. Reference to the lot number shall be given. Liquid products containing hydrolysed vegetable protein (HVP) or liquid soya sauce shall be shaken well, or homogenised by other suitable means, before the incremental sample is taken. For other products, the minimum number of incremental samples to be taken from the lot shall be as

given in Table 1. The incremental samples shall be of similar weight. Departure from this procedure must be recorded in the record provided for under 3.8.

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

Weight of lot (kg)	Minimum number of incremental samples to be taken
< 50	3
50 to 500	5
> 500	10

If the lot consists of individual packages, then the number of packages which shall be taken to form the aggregate sample is given in Table 2.

Table 2: Number of packages (incremental samples) which shall be taken to form the aggregate sample if the lot consists of individual packages

Number of packages or units in the lot	Number of packages or units to be taken
1 to 25	1 package or unit
26 to 100	About 5 %, at least 2 packages or units
> 100	About 5 % at maximum 10 packages or units

5. COMPLIANCE OF THE LOT OR SUBLOT WITH THE SPECIFICATION

The control laboratory shall analyse the laboratory sample for enforcement at least in two independent analyses, and calculate the mean of the results. The lot is accepted if the mean conforms to the respective maximum level as laid down in the Contaminants in Food Regulations. It is rejected if the mean exceeds the respective maximum level.

FOURTH SCHEDULE
(Equivalent to Annex II of Commission Directive 2001/22/EC)

Sample Preparation And Criteria For Methods Of Analysis Used In Offical Control Of The Levels Of Lead, Cadmium, Mercury And 3-MCPD In Certain Foodstuffs

1. INTRODUCTION

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

2. SPECIFIC SAMPLE PREPARATION PROCEDURES FOR LEAD, CADMIUM AND MERCURY

There are many satisfactory specific sample preparation procedures which may be used for the products under consideration. Those described in the draft CEN Standard 'Foodstuffs — Determination of trace elements — Performance criteria and general consideration' have been found to be satisfactory ([3]) but others may be equally valid.
The following points must be noted for any procedure used:

— bivalve molluscs, crustaceans and small fish: where these are normally eaten whole, the viscera are to be included in the material to be analysed,
— vegetables: only the edible portion of is to be tested, with note to be taken of the requirements of the Contaminants in Food Regulations.

3. METHOD OF ANALYSIS TO BE USED BY THE LABORATORY AND LABORATORY CONTROL REQUIREMENTS

3.1. **Definitions**

A number of the most commonly used definitions that the laboratory will be required to use are given below:

r = repeatability, the value below which the absolute difference between two 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 $r = 2.8 \cdot s_r$.

s_r = standard deviation, calculated from results generated under repeatability conditions.

RSD_r = relative standard deviation, calculated from results generated under repeatability conditions $[(s_r / \bar{x}) \times 100]$, where $\bar{x}$ is the average of results over all laboratories and samples.

[3] Draft Standard prEN 13804, 'Foodstuffs — Determination of Trace Elements — Performance Criteria and General Considerations', CEN, Rue de Stassart 36, B-1050 Brussels.

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 %); $R = 2.8 \cdot s_R$.

s_R = standard deviation, calculated from results under reproducibility conditions.

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

$HORRAT_r$ = the observed RSD_r divided by the RSD_r value estimated from the Horwitz equation using the assumption r = 0,66R

$HORRAT_R$ = the observed RSD_R value divided by the RSD_R value calculated from the Horwitz equation ([4]).

3.2. **General requirements**

Methods of analysis used for food control purposes must comply whenever possible with the provisions of paragraphs 1 and 2 of the Annex to Directive 85/591/EEC of the European Community. For the analysis of lead in wine, Commission Regulation (EEC) No 2676/90([5]) determining Community methods for the analysis of wines lays down the method to be used in Chapter 35 of its Annex.

3.3. **Specific requirements**

3.3.1. *Lead, cadmium and mercury analyses*

Specific methods for the determination of lead, cadmium and mercury contents are not prescribed. Laboratories shall use a validated method that fulfils the performance criteria indicated in Table 3. Where possible, the validation shall include a certified reference material in the collaborative trial test materials.

Table 3: Performance criteria of methods for lead, cadmium and mercury analyses

Parameter	Value/comment
Applicability	Foods specified in the Contaminants in Food Regulations
Detection limit	No more than one tenth of the value of the specification in the Contaminants in Food Regulations, except if the value of the specification

[4] W Horwitz, 'Evaluation of Analytical Methods for Regulation of Foods and Drugs', Anal. Chem., 1982, No 54, 67A-76A

[5] OJ L 272, 3.10.1990, p. 1.

Parameter	Value/comment
	for lead is less than 0.1 mg/kg. For the latter, no more than one fifth of the value of the specification
Limit of quantification	No more than one fifth of the value of the specification in the Contaminants in Food Regulations, except if the value of the specification for lead is less than 0.1 mg/kg. For the latter, no more than two fifths of the value of the specification
Precision	$HORRAT_r$ or $HORRAT_R$ values of less than 1.5 in the validation collaborative trial
Recovery	80-120 % (as indicated in the collaborative trial)
Specificity	Free from matrix or spectral interferences

3.3.2. *3-MCPD analysis*

Specific methods for the determination of 3-MCPD contents are not prescribed. Laboratories shall use a validated method that fulfils the performance criteria indicated in Table 4. Where possible, the validation shall include a certified reference material in the collaborative trial test materials. A specific method has been validated by collaborative trial and has been shown to meet the requirements of Table 4 ([6]).

Table 4: Performance criteria of methods for 3-MCPD analysis

Criterion	Recommended value	Concentration
Field blanks	Less than the detection limit	-
Recovery	75 – 110 %	All
Limit of quantification	10 (or less) µg on a dry matter basis	-
Standard deviation of the field blank signal	Less than 4 µg/kg	-
In-house precision estimates – standard deviation of replicate measurements at different concentrations	< 4 µg/kg < 6 µg/kg < 7 µg/kg < 8 µg/kg < 15 µg/kg	20 µg/kg 30 µg/kg 40 µg/kg 50 µg/kg 100 µg/kg

3.4. **Estimation of the analytical trueness and recovery calculations**

Wherever possible the trueness of the analysis shall be estimated by including suitable certified reference materials in the analytical run. The 'Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement' ([7])

[6] Method of Analysis to determine 3-Monochloropropane-1,2-Diol in Food and Food Ingredients using Mass Spectrometric Detection, submitted to CEN TC 275 and AOAC International (also available as 'Report of the Scientific Cooperation task 3.2.6: Provision of validated methods to support the Scientific Committee on Food's recommendations regarding 3-MCPD in hydrolysed protein and other foods').

[7] ISO/AOAC/IUPAC Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement. *Edited* Michael Thompson, Steven L R Ellison, Ales Fajgelj, Paul Willetts and Roger Wood, Pure Appl. Chem., 1999, No 71, 337-348

developed under the auspices of IUPAC/ISO/AOAC shall be taken into account. The analytical result shall be reported corrected or uncorrected. The manner of reporting and the level of recovery must be reported.

3.5. **Laboratory quality standards**

Laboratories must comply with Directive 93/99/EEC.

3.6. **Expression of results**

The results shall be expressed in the same units as the maximum levels laid down in the Contaminants in Foods Regulations.

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