►**B**

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

(Text with EEA relevance)

(OJ L 77, 16.3.2001, p. 14)

Amended by:

		Official Journal		
		No	page	date
►**M1**	Commission Decision 2001/873/EC of 4 December 2001	L 325	34	8.12.2001
►**M2**	Commission Directive 2005/4/EC of 19 January 2005	L 19	50	21.1.2005

▼B

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

(Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Directive 85/591/EEC of 20 December 1985 concerning the introduction of Community methods of sampling and analysis for the monitoring of foodstuffs intended for human consumption ([1]), and in particular Article 1 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 466/2001 of 8 March 2001 setting maximum levels for certain contaminants in foodstuffs ([3]) establishes, besides others, maximum levels for lead, cadmium, mercury and 3-monochloropropane-1,2-diol (3-MCPD) in foodstuffs and makes reference to the measures laying down the sampling and analysis methods to be used.

(3) Council Directive 89/397/EEC of 14 June 1989 on the official control of foodstuffs ([4]) lays down the general principles for the performance of control of foodstuffs. Council Directive 93/99/EEC of 29 October 1993 on the subject of additional measures concerning the official control of foodstuffs ([5]) introduces a system of quality standards for laboratories entrusted by the Member States with the official control of foodstuffs.

(4) Sampling plays a crucial role in obtaining representative results for the determination of the levels of the contaminants which may be heterogeneously distributed in a lot.

(5) Directive 85/591/EEC has fixed general criteria for methods of sampling and analysis but in certain cases more specific criteria become necessary in order to ensure that laboratories, in charge of the control, use methods of analysis with comparable levels of performance.

(6) The provisions for the sampling and methods of analysis have been drawn up on the basis of present knowledge and they may be adapted to take account of advances in scientific and technological knowledge.

(7) The measures provided for in this Directive are in accordance with the opinion of the Standing Committee for Foodstuffs,

HAS ADOPTED THIS DIRECTIVE:

Article 1

The Member States shall take all measures necessary to ensure that the sampling for the official control of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs is carried out in accordance with the methods described in Annex I to this Directive.

([1]) OJ L 372, 31.12.1985, p. 50.
([2]) OJ L 37, 13.2.1993, p. 1.
([3]) See page 1 of this Official Journal.
([4]) OJ L 186, 30.6.1989, p. 23.
([5]) OJ L 290, 24.11.1993, p. 14.

▼B

Article 2

The Member States shall take all measures necessary to ensure that sample preparation and methods of analyses used for the official control of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs comply with the criteria described in Annex II to this Directive.

Article 3

The Member States shall, not later than ►M1 5 April 2002 ◄, bring into force the laws, regulations or administrative provisions necessary to comply with the provisions of this Directive. They shall forthwith notify the Commission thereof.

When Member States adopt these provisions, the provisions shall contain a reference to this Directive or shall be accompanied by such reference at the time of their official publication. The procedure for such reference shall be adopted by Member States.

Article 4

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

This Directive is addressed to the Member States.

▼B

ANNEX I

METHODS OF SAMPLING FOR OFFICAL CONTROL OF THE LEVELS OF LEAD, CADMIUM, MERCURY AND 3-MCPD 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 Regulation (EC) No 466/2001 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 qualified person as specified by the Member States.

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 unless this conflicts with Member States' regulations on sampling. The size of the laboratory samples for enforcement shall be sufficient to allow at least for duplicate analyses.

▼B

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 following the Member States' regulations. 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

▼M2

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 does not exceed the respective maximum level as laid down in Regulation (EC) No 466/2001, taking into account the expanded measurement uncertainty and correction for recovery (1).

The lot is rejected if the mean exceeds the respective maximum level beyond reasonable doubt, taking into account the expanded measurement uncertainty and correction for recovery.

▼**M2**

The present interpretation rules are of application for the analytical result obtained on the sample for official control. In case of analysis for defence or referee purposes, the national rules apply.

▼B

ANNEX II

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 ([a]) 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 Regulation (EC) No 466/2001.

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 $r = 2{,}8 \times s_r$.

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

RSD_r = relative standard deviation, calculated from results generated under repeatability conditions $[(s_r / \bar{x}) \times 100]$, where $\bar{x}$ 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 %); $R = 2{,}8 \times s_R$.

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

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

$HORRAT_r$ = 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 ([b]).

▼B

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.

For the analysis of lead in wine, Commission Regulation (EEC) No 2676/90 ([1]) 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 Regulation (EC) No 466/2001
Detection limit	No more than one tenth of the value of the specification in Regulation (EC) No 466/2001, except if the value of the specification 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 Regulation (EC) No 466/2001, 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 ([c]).

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/kg on a dry matter basis	—
Standard deviation of the field blank signal	Less than 4 µg/kg	—

([1]) OJ L 272, 3.10.1990, p. 1.

▼B

Criterion	Recommended value	Concentration
In-house precision estimates — standard deviation of replicate measurements at different concentrations	< 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

▼M2

3.3.3. *Performance Criteria — Uncertainty Function Approach*

However, an uncertainty approach may also be used to assess the suitability of the method of analysis to be used by the laboratory. The laboratory may use a method which will produce results within a maximum standard uncertainty. The maximum standard uncertainty can be calculated using the following formula:

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

where:

Uf is the maximum standard uncertainty

LOD is the limit of detection of the method

C is the concentration of interest

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

C (µg/kg)	α
≤ 50	0,2
51-500	0,18
501-1 000	0,15
1 001-10 000	0,12
≥ 10 000	0,1

and U is the expanded uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95 %.

If an analytical method provides results with uncertainty measurements less than the maximum standard uncertainty the method will be equally suitable to one which meets the performance characteristics given above.

3.4. **Estimation of the analytical trueness, recovery calculations and reporting of results**

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

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

The analyst should note the 'European Commission Report on the relationship between analytical results, the measurement of uncertainty, recovery factors and the provisions in EU food legislation'(d).

The analytical result has to be reported as x +/– U whereby x is the analytical result and U is the measurement uncertainty.

▼B

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 Regulation (EC) No 466/2001.

▼B

REFERENCES

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

(b) W Horwitz, 'Evaluation of Analytical Methods for Regulation of Foods and Drugs', Anal. Chem., 1982, No 54, 67A-76A

(c) 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').

▼M2

(d) European Commission Report on the relationship between analytical results, the measurement of uncertainty, recovery factors and the provisions in EU food legislation, 2004

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