

**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

(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⁾, as amended by Regulation (EC) No 1882/2003 of the European Parliament and of the Council⁽²⁾, and in particular Article 1 thereof,

Whereas:

(1) Commission Regulation (EC) No 466/2001 of 8 March 2001 setting maximum levels for certain contaminants in foodstuffs⁽³⁾, as last amended by Regulation (EC) No 242/2004⁽⁴⁾ fixes maximum limits for inorganic tin in canned foodstuffs and makes reference to measures laying down the sampling and analysis methods to be used.

(2) Council Directive 93/99/EEC of 29 October 1993 on the subject of additional measures concerning the official control of foodstuffs⁽⁵⁾, as amended by Regulation (EC) No 1882/2003, introduces a system of quality standards for laboratories entrusted by the Member States with the official control of foodstuffs.

(3) It seems necessary to fix general criteria, which the method of analysis has to comply with in order to ensure that laboratories, in charge of the control, use methods of analysis with comparable levels of performance. It is also of major importance that analytical results are reported and interpreted in a uniform way in order to ensure a harmonised enforcement approach across the European Union. These 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.

(4) 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. Methods to analyse for total tin are appropriate for controls on inorganic tin. The possible presence of organic forms of tin are considered to be negligible in relation to the maximum levels set for inorganic tin.

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

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 tin in foodstuffs is carried out in accordance with the methods described in Annex I to this Directive.

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 tin in foodstuffs comply with the criteria described in Annex II to this Directive.

Article 3

The Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with the provisions of this Directive by 31 December 2004. They shall forthwith communicate to the Commission the text of those provisions and a correlation table between those provisions and this Directive.

When Member States adopt those provisions, their provisions shall contain a reference to this Directive or shall be accompanied by such a reference on the occasion of their official publication. Member States shall determine how such reference is to be made.

⁽¹⁾ OJ L 372, 31.12.1985, p. 50.

⁽²⁾ OJ L 284, 31.10.2003, p. 1.

⁽³⁾ OJ L 77, 16.3.2001, p. 1.

⁽⁴⁾ See page 3 of this Official Journal.

⁽⁵⁾ OJ L 290, 24.11.1993, p. 14.

Article 4

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

This Directive is addressed to the Member States.

Done at Brussels, 12 February 2004.

For the Commission
David BYRNE
Member of the Commission

ANNEX I

METHODS OF SAMPLING FOR OFFICIAL CONTROL OF THE LEVELS OF TIN IN CANNED FOODSTUFFS**1. Purpose and scope**

Samples intended for official checking of the levels of tin in canned 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 levels laid down in Commission 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 a food commodity delivered at one time and having been determined by the official to have common characteristics, such as origin, variety, type of packing, packer, consignor or markings.

Sublot: designated part of a 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.

3. General provisions**3.1. Personnel**

Sampling shall be performed by an authorised 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 the samples precautions must be taken to avoid any changes, which would affect the tin 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.

3.5. Preparation of the aggregate sample

The aggregate sample is made up by uniting all incremental samples. This aggregate sample is homogenised in the laboratory.

3.6. Replicate laboratory samples

Replicate laboratory samples for enforcement, trade (defence) and referee purposes shall be taken from the homogenised aggregate sample unless this conflicts with Member States' rules on sampling.

3.7. Packaging and transmission of samples

Each 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 sample, which might arise during transportation or storage.

3.8. *Sealing and labelling of samples*

Each sample taken for official use shall be sealed at the place of sampling and identified following the Member State's 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**

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*

The minimum number of incremental samples to be taken from cans within a lot shall be as given in Table 1. The incremental samples taken from each can shall be of similar weight, resulting in an aggregate sample (see point 3.5).

Table 1

Number of cans (incremental samples) which shall be taken to form the aggregate sample

Number of cans in the lot or subplot	Number of cans to be taken
1 to 25	at least 1 can
26 to 100	at least 2 cans
> 100	5 cans

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

4.2. *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.

5. **Compliance of the lot or subplot with the specification**

The control laboratory shall analyse the laboratory sample for enforcement in at least 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 measurement uncertainty and correction for recovery.

The lot is non-compliant with the maximum level (as laid down in Regulation (EC) 466/2001) if the mean exceeds the maximum level beyond reasonable doubt taking into account the measurement uncertainty and correction for recovery.

ANNEX II

SAMPLE PREPARATION AND CRITERIA FOR METHODS OF ANALYSIS USED IN OFFICIAL CHECKING OF THE LEVELS OF TIN IN CANNED FOODSTUFFS**1. Precautions and general considerations for tin**

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

The analyst should ensure that samples do not become contaminated during sample preparation. Wherever possible, apparatus coming into contact with the sample should be made of inert materials e.g. plastics such as polypropylene, PTFE etc., and these should be acid cleaned to minimise the risk of contamination. High quality stainless steel can be used for cutting edges.

All of the sample material received by the laboratory is to be used for the preparation of test material. Only very finely homogenised samples give reproducible results.

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

2. Treatment of the sample as received in the laboratory

Finely grind (where relevant) and mix thoroughly the complete aggregate 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 homogenised material unless this conflicts with Member States' rules on sampling.

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:

r = Repeatability, the value below which the absolute difference between 2 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 (2).

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

4.2. *General requirements*

Methods of analysis used for food control purposes must comply with the provisions of items 1 and 2 of the Annex 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.

4.3. *Specific requirements*

Where no specific methods for the determination of tin in canned foodstuffs are prescribed at Community level, laboratories may select any validated method provided the selected method meets the performance criteria indicated in Table 2. The validation should ideally include a certified reference material.

Table 2

Performance criteria for methods of analysis for tin

Parameter	Value/comment
Applicability	Foods specified in Regulation (EC) No 242/2004
Detection limit	No more than 5 mg/kg
Limit of quantification	No more than 10 mg/kg
Precision	HORRAT _r or HORRAT _R values of less than 1,5 in the validation collaborative trial
Recovery	80 % to 105 % (as indicated in the collaborative trial)
Specificity	Free from matrix or spectral interferences

4.3.1. *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:

$$U_f = \sqrt{(LOD/2)^2 + (0,1C)^2}$$

where:

U_f is the maximum standard uncertainty

LOD is the limit of detection of the method

C is the concentration of interest

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 in Table 2.

4.4. *Recovery calculation and reporting of results*

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 analytical result corrected for recovery is used for checking compliance (see Annex I, point 5).

The analyst should note the 'Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement' (3) developed under IUPAC/ISO/AOAC. These Guidelines assist when determining recovery factors.

The analytical result has to be reported as $x \pm U$ whereby x is the analytical result and U is the measurement uncertainty.

4.5. *Laboratory quality standards*

Laboratories must comply with Council Directive 93/99/EEC of 29 October 1993 on the subject of additional measures concerning the official control of foodstuffs.

4.6. Other considerations for the analysis

Proficiency testing

Participation in appropriate proficiency testing schemes which comply with the 'International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories' (4) developed under the auspices of IUPAC/ISO/AOAC.

Some of these schemes specifically include the determination of tin in foods, and participation in such a scheme is recommended rather than a general scheme for the determination of metals in foods.

Internal quality control

Laboratories should 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' (5).

Sample preparation

Care must be taken to ensure that all the tin in the sample is taken into solution for analysis. In particular it is recognised that the sample dissolution procedure must be such that no hydrolysed SnIV species is precipitated (i.e. species such as stannic oxide SnO_2 , $\text{Sn}(\text{OH})_4$, $\text{SnO}_2 \cdot \text{H}_2\text{O}$).

Keep prepared samples in 5 mol/l HCl. However, SnCl_4 is easily volatilised and so solutions should not be boiled.

REFERENCES

1. BS EN 13804:2002: Foodstuffs — Determination of trace elements — Performance criteria, general considerations and sample preparation, CEN, Rue de Stassart 36, B-1050 Brussels.
 2. W Horwitz, 'Evaluation of Analytical Methods for Regulation of Foods and Drugs', *Anal. Chem.*, 1982, 54, 67A — 76A.
 3. 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, 71, 337 — 348.
 4. ISO/AOAC/IUPAC International Harmonised Protocol for Proficiency Testing of (Chemical) Analytical Laboratories, Edited by M Thompson and R Wood, *Pure Appl. Chem.*, 1993, 65, 2123 — 2144 (Also published in *J. AOAC International*, 1993, 76, 926).
 5. ISO/AOAC/IUPAC International Harmonised Guidelines for Internal Quality Control in Analytical Chemistry Laboratories, Edited by M Thompson and R Wood, *Pure Appl. Chem.*, 1995, 67, 649 — 666.
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