

**VETERINARY SERVICES ACT
(CAP. 437)**

**Sampling Methods and the methods of analysis for the official
control of the levels of lead, cadmium, mercury and 3-MCPD in
Foodstuffs Rules, 2005**

IN exercise of the powers conferred under article 25 of the Veterinary Services Act, the Minister for Rural Affairs and the Environment, in agreement with the Minister of Health, the Elderly and Community Care, has made the following rules:-

Title and scope.

1. (1) The title of these rules is the Sampling Methods and the methods of analysis for the official control of the levels of lead, cadmium, mercury and 3-MCPD in Foodstuffs Rules, 2005.

(2) The scope of these rules is to implement the rules found under European Union Council Directive 2001/22/EC on the sampling methods and the methods of analysis for the official control of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs.

Definitions.

2. For the purposes of these rules, and unless context otherwise requires -

“aggregate sample” means the combined total of all the incremental samples taken from the lot or subplot;

“the Commission” means the European Commission;

“the Community” means the European Community as established under the Treaty establishing the European Community;

“the competent authority” means the Veterinary Services within Malta as provided under article 2 of the Veterinary Services Act;

“incremental sample” means a quantity of material taken from a single place in the lot or subplot;

“laboratory sample” means the sample intended for the laboratory;

“lot” means an identifiable quantity of food delivered at one time and determined by the veterinary 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;

“Member State” means a State which is a Member within the European Community;

“sublot” means a 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;

“third country” means a State which is not a Member within the European Community;

“Veterinary Services” means the competent authority within the territory of Malta as established under article 2 of the Veterinary Services Act.

3. Malta shall take all measures necessary to ensure that the sampling for the official controls of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs is carried out in accordance with the methods described in Schedule I to these rules.

Sampling for official controls.

4. Malta 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 Schedule II to these rules.

Sample preparation and methods of analyses.

5. When Malta adopts these provisions, the provisions shall contain a reference to these rules or shall be accompanied by such reference at the time of their official publication. The procedure for such reference shall be that applicable in Malta.

Reference of procedure and applicability.

SCHEDULE I
METHODS OF SAMPLING FOR OFFICIAL 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. GENERAL PROVISIONS

2.1. Personnel

Sampling shall be performed by an authorised qualified person as specified by Malta.

2.2. Material to be sampled

Each lot which is to be examined must be sampled separately.

2.3. 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.

2.4. Incremental samples

As far as possible incremental samples shall be taken at various places distributed throughout the lot or subplot. Departure from this procedure must be recorded in the record provided for under 2.8.

2.5. 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.

2.6. 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 Veterinary Services' regulations on sampling. The size of the laboratory samples for enforcement shall be sufficient to allow at least for duplicate analyses.

2.7. 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.

2.8. 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 Veterinary Services' 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.

3. 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.

3.1. 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 2.8.

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

4. 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 Regulation (EC) No 466/2001. It is rejected if the mean exceeds the respective maximum level.

SCHEDULE 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 $r = 2,8 \times sr$.

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

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

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

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

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

$HORRATR$ = the observed $RSDR$ value divided by the $RSDR$ value calculated from the Horwitz equation (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 0,1mg/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	HORRATr or HORRATR 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	—
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

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’ (d) 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 Regulation (EC) No 466/2001.

