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Delegations will find attached document D034437/03 - Annex 1.

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ANNEX 1

ANNEX

to the

COMMISSION REGULATION (EU) No .../...

laying down methods of sampling and performance criteria for the methods of analysis for the official control of the levels of erucic acid in foodstuffs and repealing Commission Directive 80/891/EEC

ANNEX

PART A: DEFINITONS

For the purposes of this Annex, the following definitions shall apply:

"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].
"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; aggregate samples shall be considered as representative of the lots or sublots from which they are taken;
"laboratory sample":	a sample intended for the laboratory.

PART B: SAMPLING METHODS

B.1. GENERAL PROVISIONS

B.1.1. Personnel

Sampling shall be performed by an authorised person designated by the Member State.

B.1.2. Material to be sampled

Each lot or sublot which is to be examined shall be sampled separately.

B.1.3. Precautions to be taken

In the course of sampling, precautions shall be taken to avoid any changes which would affect the levels of erucic acid, adversely affect the analytical determination or make the aggregate samples unrepresentative.

B.1.4. Incremental samples

As far as possible incremental samples shall be taken at various places distributed throughout the lot or sublot. Departure from such procedure shall be recorded in the record provided for under point B.1.8. of this Annex

B.1.5. Preparation of the aggregate sample

The aggregate sample shall be made up combining the incremental samples.

B.1.6. Samples for enforcement, defence and referee purposes

The samples for enforcement, defence and referee purposes shall be taken from the homogenised aggregate sample unless this conflicts with the rules of the Member States as regards the rights of the food business operators.

B.1.7. Packaging and transmission of samples

Each 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 any change in composition of the sample which might arise during transportation or storage.

B.1.8. Sealing and labelling of samples

Each sample taken for official use shall be sealed at the place of sampling and identified in accordance with the rules of the Member States.

A record shall be kept of each sampling, permitting each lot or sublot from which the sample has been taken to be identified unambiguously. That record shall indicate all of the following:

(i) reference to the number of lot from which the sample has been taken;

(ii) the date and place of sampling;

(iii) any additional information likely to be of assistance to the analyst.

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. The weight or number of sublots of products traded in bulk consignments shall be as given in Table 1. The weight or number of sublots of other products shall be as given in Table 2 . 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 indicated in Tables 1 and 2 may be exceeded by a maximum of 20%.

B.2.2. NUMBER, WEIGHT AND VOLUME OF INCREMENTAL SAMPLES

The aggregate sample shall be at least 1 kg or 1 litre except where this is not possible e.g. when the sample consists of one 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 insofar as possible and insofar 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 or volume. The weight or 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 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	Number of packages or units to be taken

lot/sublot	
≤ 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

Where sampling using the method set out in this chapter B.2 would lead to unacceptable commercial consequences (e.g. because of packaging forms, damage to the lot, etc.) or would be practically impossible, an alternative method of sampling may be applied provided that it is sufficiently representative for the sampled lot or sublot and is fully documented in the report provided for under point B.1.8..

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..

Where sampling using the method set out in point B.2.2. would lead to unacceptable commercial consequences (e.g. because of packaging forms, damage to the lot, etc.) or would be practically impossible, an alternative method of sampling may be applied provided that it is sufficiently representative for the sampled lot or sublot and is fully documented in the report provided for under point B.1.8.

PART C: SAMPLE PREPARATION AND ANALYSIS

C.1. LABORATORY QUALITY STANDARDS

Laboratories shall comply with the provisions of Article 12 of Regulation (EC) No 882/2004.

Laboratories shall participate in appropriate proficiency testing schemes which comply with the 'International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories'[1] developed under the auspices of IUPAC/ISO/AOAC.

Laboratories shall 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'[2].

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

C.2. SAMPLE PREPARATION

C.2.1. Precautions and general considerations

[1] "The international harmonized protocol for the proficiency testing of analytical chemistry laboratories" by M. Thompson, S.L.R. Ellison and R. Wood, Pure Appl. Chem., 2006, 78, 145-196.

[2] Edited by M. Thompson and R. Wood, Pure Appl. Chem., 1995, 67, 649-666.

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

All of the sample material received by the laboratory shall be used for the preparation of the laboratory sample.

Compliance with maximum levels laid down in Regulation (EC) No 1881/2006 shall be established on the basis of the levels determined in the laboratory samples.

C.2.2. Treatment of the sample as received in the laboratory

The complete aggregate sample shall be finely ground (where relevant) and thoroughly mixed using a process that has been demonstrated to achieve complete homogenisation.

C.3. PERFORMANCE CRITERIA FOR THE METHODS OF ANALYSIS

C.3.1. Definitions

The following definitions shall apply:

"r" =	Repeatability the value below which the absolute difference between 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 $[(s_r / \bar{x}) \times 100]$.
"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 $[(s_R / \bar{x}) \times 100]$.
"LOD"=	Limit of detection, smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty. The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations (n>20).
"LOQ"=	Limit of quantification, lowest content of the analyte which can be measured with reasonable statistical certainty. If both accuracy and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to six or ten times the standard deviation of the mean of blank determinations (n>20).

"u" = Combined standard measurement uncertainty obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model[3].

"U" = The expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95% (U = 2u).

"Uf" = Maximum standard measurement uncertainty.

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.

C.3.3. Specific requirements

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 Table 5.

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 Table 5.

Further details are given in the Notes to the performance criteria as set out in this point.

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

Table 5: Performance criteria for methods of analysis for erucic acid

Parameter	**Criterion**
Applicability	Foods specified in Regulation (EC) No 1881/2006
Specificity	Free from matrix or spectral interferences
Repeatability (RSD_r)	0.66 times RSD_R as derived from (modified) Horwitz equation
Reproducibility (RSD_R)	2 x value derived from (modified) Horwitz equation

[3] International vocabulary of metrology – Basic and general concepts and associated terms (VIM), JCGM 200:2008.

Recovery	95 – 105 %
LOD	≤ 1 g/kg
LOQ	≤ 5 g/kg

Notes to the performance criteria:

The Horwitz equation[4] (for concentrations $1.2 \times 10^{-7} \leq C \leq 0.138$) and the modified Horwitz equation[5] (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 = 100g/100g, 0.001 = 1000 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 = 100g/100g, 0.001 = 1000 mg/kg). The Horwitz equation applies to concentrations $1.2 \times 10^{-7} \leq C \leq 0.138$.

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

For in-house validated methods, as an alternative a 'fitness-for-purpose' approach[6] may be used to assess their suitability for official control. Methods suitable for official control shall produce results with a combined standard measurement uncertainty (u) less than the maximum standard measurement uncertainty calculated using the formula below:

4 W. Horwitz, L.R. Kamps. K.W. Boyer, J.Assoc.Off.Analy.Chem.,1980, 63, 1344.
5 M. Thompson, Analyst, 2000, 125, 385-386.
6 M. Thompson and R. Wood, Accred. Qual. Assur., 2006, 10, 471-478.

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

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 out 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 6.

Table 6: 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
501-1000	0.15
1001-10000	0.12
> 10000	0.1

PART D: REPORTING AND INTERPRETATION OF RESULTS

D.1. REPORTING

D.1.1. Expression of results

The results shall be expressed in the same units and with the same number of significant figures as the maximum levels laid down in Regulation (EC) No 1881/2006.

D.1.2. Recovery calculations

If an extraction step is applied in the analytical method, the analytical result shall be corrected for recovery. In this case the level of recovery shall be reported.

In case no extraction step is applied in the analytical method, 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.

D.1.3. Measurement uncertainty

The analytical result shall be reported as x +/- U whereby x is the analytical result and U is the expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95% (U = 2u).

The analyst shall note the 'Report on the relationship between analytical results, measurement uncertainty, recovery factors and the provisions in EU food and feed legislation[7]'.

D.2. INTERPRETATION OF RESULTS

D.2.1. Acceptance of a lot or sublot

The lot or sublot is accepted if the analytical result of the laboratory sample does not exceed the respective maximum level laid down in Regulation (EC) No 1881/2006 taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.

D.2.2. Rejection of a lot or sublot

The lot or sublot is rejected if the analytical result of the laboratory sample exceeds beyond reasonable doubt the respective maximum level laid down in Regulation (EC) No 1881/2006 taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.

D.2.3. Applicability

The interpretation rules set out under points D.2.1. and D.2.2. shall apply for the analytical result obtained on the sample for enforcement. In case of analysis for defence or referee purposes, the national rules shall apply.

[7] http://ec.europa.eu/food/food/chemicalsafety/contaminants/report-sampling_analysis_2004_en.pdf