Commission Directive 2005/10/EC of 4 February 2005 laying down the sampling methods and the methods of analysis for the official control of the levels of benzo(a)pyrene in foodstuffs (Text with EEA relevance) (repealed)

ANNEX II

SAMPLE PREPARATION AND CRITERIA FOR METHODS OF ANALYSIS USED IN OFFICIAL CHECKING OF THE LEVELS OF BENZO(A)PYRENE IN FOODSTUFFS

1. Precautions and general considerations for benzo(a)pyrene in food samples

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. Containers should be rinsed with high purity acetone or hexane (p.A., HLPC grade or equivalent) before use to minimise the risk of contamination. Wherever possible, apparatus coming into contact with the sample should be made of inert materials e.g. aluminium, glass or polished stainless steel. Plastics such as polypropylene, PTFE etc. should be avoided because the analyte can adsorb onto these materials.

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.

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 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
$[(s_r / \bar{x}) \times 100]$
.

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

$[(s_R / \bar{x}) \times 100]$

, where

$\bar{x}$

is the average of results over all laboratories and samples.

$HORRAT_r$ = the observed RSD_r divided by the RSD_r value estimated from the Horwitz equation (1) using the assumption r = 0.66R.

$HORRAT_R$ = the observed RSD_R value divided by the RSD_R value calculated from the Horwitz equation.

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 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 benzo(a)pyrene in food are prescribed at Community level, laboratories may select any validated method provided the selected method meets the performance criteria indicated in the Table. The validation should ideally include a certified reference material.

TABLE

Performance criteria for methods of analysis for benzo(a)pyrene

Parameter	**Value/comment**
Applicability	Food specified in Regulation (EC) No …/2005
Detection limit	No more than 0,3 µg/kg
Limit of quantification	No more than 0,9 µg/kg
Precision	$HORRAT_r$ or $HORRAT_R$ values of less than 1.5 in the validation collaborative trial
Recovery	50 %-120 %
Specificity	Free from matrix or spectral interferences, verification of positive detection

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:

$$Uf = \sqrt{\left[(LOD / 2)^2 + (0.2C)^2\right]}$$

where:

Uf	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 the Table.

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 'European Commission Report on the relationship between analytical results, the measurement of uncertainty, recovery factors and the provisions in EU food legislation' (2).

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

4.5. Laboratory quality standards

Laboratories must comply with Directive 93/99/EEC.

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' (3) developed under the auspices of IUPAC/ISO/AOAC.

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' (4).

REFERENCES

1. W. Horwitz, 'Evaluation of Analytical Methods for Regulation of Foods and Drugs', Anal. Chem., 1982, 54, 67A-76A.

2. 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/index_en.htm).

3. 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).

4. 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.