

## A.L. 107 ta' l-2006

**ATT DWAR IS-SIGURTÀ TA' L-IKEL  
(KAP. 449)**

**Regolamenti ta' l-2006 li jemendaw ir-Regolamenti  
dwar Sustanzi li Jikkontaminaw fl-Ikel  
(Metodi kif jittiehdu l-Kampjuni u kif issir l-Analizi)  
(Emenda) (Nru. 2)**

BIS-SAHHA tas-setghat moghtija mill-artikolu 10 ta' l-Att dwar is-Sigurtà ta' l-Ikel, il-Ministru tas-Sahha, l-Anzjani u l-Kura fil-Komunità ghamel dawn ir-regolamenti li ġejjin:-

1. It-titolu ta' dawn ir-regolamenti hu Regolamenti ta' l-2006 li jemendaw ir-Regolamenti dwar Sustanzi li Jikkontaminaw fl-Ikel (Metodi kif jittiehdu l-Kampjuni u kif issir l-Analizi) (Emenda) (Nru. 2), u ghandhom jinqraw u jkunu interpretati bhala haġa wahda mar-Regolamenti ta' l-2004 dwar Sustanzi li Jikkontaminaw fl-Ikel (Metodi kif jittiehdu l-Kampjuni u kif issir l-Analizi), minn issa 'l quddiem imsejhin "ir-regolamenti prinċipali". Titolu.  
A.L. 488 ta' l-2004.
2. Dawn ir-regolamenti jimplimentaw id-dispożizzjonijiet tad-Direttiva tal-Kummissjoni 2005/10/KE ta' l-4 ta' Frar, 2005. Skop.
3. Minflok ir-regolament 7 tar-regolamenti prinċipali ghandu jidhol dan li ġej- Jemenda regolament  
7 tar-regolamenti  
prinċipali.
  - 7.1. It-tehid ta' kampjuni ghal kontroll ufficjali tal-livelli ta' benzo(a)pirin f'oġġetti ta' l-ikel ghandu jsir skond il-metodi deskritti fil-hdax-il Skeda.
  - 7.2. Il-preparazzjoni ghat-tehid ta' kampjun u metodi ta' analizi użati ghall-kontroll ufficjali tal-livelli ta' benzo(a)pirin f'oġġetti ta' l-ikel ghandu jikkonforma mal-kriterji deskritti fit-Tnax-il Skeda."
4. Minnufih wara l-Ghaxar Skeda li tinsab mar-regolamenti prinċipali ghandu tizdied din il-hdax-il Skeda li ġejja:- Izid il-hdax-il Skeda  
mar-regolamenti  
prinċipali.

“IL-HDAX-IL SKEDA

(Ekwivalenti għal Anness I tad-Direttiva tal-Kummissjoni  
2005/10/KE)

**METODI TA' TEHID TA' KAMPJUNI GĦAL KONTROLL  
UFFIĊJALI TAL-LIVELLI TA' BENZO(A)PIRIN  
F'OĠĠETTI TA' L-IKEL**

1. *Fini u Skop*

Kampjuni mahsubin għal iċċekkjar uffiċjali tal-livelli ta' benzo(a)pirin f'oġġetti ta' l-ikel għandhom jittiehdu skond il-metodi deskritti hawn taht. Kampjuni aggregati mehudin b'dan il-mod għandhom jitqiesu bħala rappreżentattivi tal-lottijiet. Konformità mal-limiti massimi preskritti fir-Regolament tal-Kummissjoni (KE) Nru 466/2001 għandha tkun stabbilita fuq il-bażi tal-livelli determinati fil-kampjuni tal-laboratorju.

2. *Definizzjonijiet*

‘Lott’: kwantità identifikabbli ta' oġġett ta' l-ikel konsenjata f'okkażjoni waħda u determinata mill-uffiċjal li għandha karatteristiċi komuni, bħalma huma oriġini, varjetà, tip ta' ppakkjar, min ippakkjaha, min għamel il-konsenja, jew marki.

‘Sublott’: parti speċifikata minn lott biex ikun applikat il-metodu ta' tehid ta' kampjuni fuq dik il-parti speċifikata; kull sublott għandu jkun separat fiżikament u identifikabbli.

‘Kampjun inkriminali’: kwantità ta' materjal mehuda minn post wiehed fil-lott jew sublott.

‘Kampjun aggregat’: it-total mehud flimkien tal-kampjuni inkriminali kollha mehudin mil-lott jew sublott.

‘Kampjun tal-laboratorju’: kampjun mahsub għal laboratorju.

3. *Dispożizzjonijiet ġenerali*

3.1. Personnel

L-analiżi tal-kampjuni għandha ssir minn analista uffiċjali kif speċifikat mill-Kummissjoni għas-Sigurtà fl-Ikel.

### 3.2. Materjal li se jkun analizzat

Kull lott li se jkun eżaminat għandhom jittiehdu minnu kampjuni separatament.

### 3.3. Prekawzjonijiet li għandhom jittiehdu

Fil-kors tat-tehid tal-kampjuni u l-preparazzjoni tal-kampjuni tal-laboratorju għandhom jittiehdu prekawzjonijiet biex ikunu evitati kull xorta ta' bidliet li jaffettwaw il-kontenut ta' benzo(a)pirin, jaffettwaw hażin id-determinazzjoni analitika jew jirrendu l-kampjuni aggregati mhux rappreżentattivi.

### 3.4. Kampjuni inkriminali

Safejn hu possibbli, kampjuni inkriminali għandhom jittiehdu minn diversi partijiet imqassmin mal-lott jew sublott kollu. Jekk din il-proċedura ma tintużax, dan irid jitniżżel fir-rekord.

### 3.5. Preparazzjoni tal-kampjun aggregat

Il-kampjun aggregat hu magħmul bit-tagħqid tal-kampjuni inkriminali kollha. Dan il-kampjun aggregat hu omogeneizzat fil-laboratorju sakemm dan ma jkunx kompatibbli ma' l-implimentazzjoni ta' punt 3.6.

### 3.6. Kampjuni tal-laboratorju replikati

Il-kampjuni tal-laboratorju replikati għall-finijiet ta' infurzar tal-liġi, kummerċ (difiza) u referenza għandhom jittiehdu mill-kampjun aggregat omogeneizzat sakemm dan ma jmurx kontra r-regoli nazzjonali dwar tehid ta' kampjuni.

### 3.7. Ippakkjar u ġarr tal-kampjuni

Kull kampjun tal-laboratorju għandu jitqiegħed f'kontenitur nadif u inerti li joffri protezzjoni biżżejjed minn kontaminazzjoni u kontra hsarat waqt il-ġarr. Kull prekawzjoni neċessarja għandha tittiehed biex tkun evitata bidla fil-kompożizzjoni tal-kampjun tal-laboratorju li tista' tokkorri waqt il-ġarr jew il-ħżin.

### 3.8. Issiġillar u tikkettjar ta' kampjuni tal-laboratorju

Kull kampjun mehud għal użu uffiċjali għandu jkun issiġillat fil-post fejn issir l-analiżi tal-kampjun u jkun identifikat skont ir-regoli nazzjonali.

Ghandu jinżamm rekord ta' kull tehid ta' kampjun li jippermetti li kull lott ikun identifikat minghajr ambigwià u li jaghti d-data u l-post minn fejn ittieded flimkien ma' kull informazzjoni addizzjonali li x'aktarx tista' tkun ta' ghajnuna għall-analista.

#### 4. *Pjanijiet kif jittieded kampjun*

Il-metodu kif jittieded kampjun ghandu jiżgura li l-kampjun aggregat hu rappreżentattiv tal-lott li jrid ikun ikkontrollat.

##### 4.1. Numru ta' kampjuni inkrimentali

Fil-każ ta' żjut, li għalihom wiehed jista' jassumi li hemm distribuzzjoni omogenea ta' benzo(a)pirin f'lott speċifikat, hu biżżejjed li jittieddu tliet kampjuni inkrimentali minn kull lott biex ikun iffurmat il-kampjun aggregat. Ghandha tinghata referenza lin-numru tal-lott. Fil-każ ta' żejt taż-żebbuġ u żejt tal-ghadma taż-żebbuġa, aktar informazzjoni qed tinghata fir-Regolament tal-Kummissjoni (KE) Nru 1989/2003.

Fil-każ ta' prodotti ohra, in-numru minimu ta' kampjuni inkrimentali li jrid jittieded ghandu jkun kif jidher f'Tabella 1. Il-kampjuni inkrimentali ghandhom ikunu ta' piż simili, mhux inqas minn 100g kull wiehed, u jirriżultaw f'kampjun aggregat ta' mhux inqas minn 300g (ara punt 3.5).

TABELLA 1

#### **Numru minimu ta' kampjuni inkrimentali li ghandu jittieded mil-lott**

Piż tal-lott (f'kg)	Numru minimu ta' kampjuni inkrimentali li ghandu jittieded
< 50	3
50 sa 500	5
> 500	10

Jekk il-lott jikkonsisti f'pakki individwali, allura n-numru ta' pakki li ghandu jittieded biex jiffurma kampjun aggregat qed jidher f'Tabella 2.

TABELLA 2

**Numru ta' pakki (kampjuni inkriminali) li ghandu jittiehed biex jifforma l-kampjun aggregat jekk il-lott jikkonsisti f'pakki individwali**

Numru ta' pakki jew unitajiet fil-lott jew sublott	Numru ta' pakki jew unitajiet li ghandu jittiehed
1 sa 25	1 pakk jew unita
26 sa 100	Xi 5%, mill-inqas 2 pakki jew unitajiet
> 100	Xi 5%, massimu ta' 10 pakki jew unitajiet

**4.2. Tehid ta' kampjuni fl-istadju tal-bejgh bl-imnut**

Tehid ta' kampjuni ta' oggetti ta' l-ikel fl-istadju tal-bejgh bl-imnut ghandu jsir fejn hu possibbli skond id-dispozizzjonijiet dwar tehid ta' kampjuni moghtija hawn fuq. Meta dan m'hux possibbli, jistghu jintuzaw proceduri effettivi ohra fl-istadju tal-bejgh bl-imnut sakemm ikun zgurat li jkunu rappreżentattivi biżżejjed tal-lott li minnu tiehdu l-kampjuni.

**5. *Konformità tal-lot jew sublott ma' l-ispeċifikazzjoni***

Il-laboratorju li jaghmel il-kontroll ghandu janalizza l-kampjun tal-laboratorju ghall-infurzar tal-liġi f'zewg analizi fil-każ li r-rizultat mehud fl-ewwel analizi hu inqas minn 20% taht jew fuq il-livell massimu, u f'dawn il-każijiet ghandu jikkalkola l-medja tar-rizultati.

Il-lott hu accettat jekk ir-rizultat ta' l-ewwel analizi jew, meta analizi duplikata hija mehtiega, jekk il-medja ma taqbiżx il-livell massimu (kif stipulat fir-Regolament (KE) Nru 466/2001) waqt li jittiehed każ ta' l-incertezza tal-kejl u korrezzjoni ghal irkupru.

Il-lott ma jikkonformax mal-livell massimu (kif imnizzel fir-Regolament (KE) Nru 466/2001) jekk ir-rizultat ta' l-ewwel analizi jew, meta analizi duplikata hija mehtiega, il-medja taqbeż il-livell massimu bla ma jkun hemm dubju ragunevoli waqt li jittiehed każ ta' l-incertezza tal-kejl u korrezzjoni ghal irkupru.”

5. Minnufih wara l-hdax-il Skeda ġdida li tinsab mar-regolamenti prinċipali, għandha tizzied din it-Tnax-il Skeda li ġejja:-

“IT-TNAX-IL SKEDA

(Ekwivalenti għal Anness II tad-Direttiva tal-Kummissjoni  
2005/10/KE)

**PREPARAZZJONI TA' KAMPJUN U KRITERJI  
GĦAL METODI TA' ANALIŻI UŻATI FL-IĊĊEKKJAR  
UFFIĊJALI TAL-LIVELLI TA' BENZO(A)PIRIN  
F'OĠĠETTI TA' L-IKEL**

1. *Prekawzjonijiet u konsiderazzjonijiet generali fil-każ ta' benzo(a)pirin f'kampjuni ta' l-ikel*

Ir-rekwizit bażiku hu li jittiehed kampjun tal-laboratorju rappreżentattiv u omogenju bla ma' tiddaħhal kontaminazzjoni sekondarja.

L-analista għandu jiżgura li kampjuni ma jkunux kontaminati waqt il-preparazzjoni tal-kampjun. Kontenituri għandhom jitlaha l-hu b'acetone jew hexane mill-aktar safi (p.A., grad HPLC jew ekwivalenti) qabel jintużaw biex ir-riskju ta' kontaminazzjoni jitnaqqas kemm jista' jkun. Kulfejn hu possibbli, apparat li jiġi f'kuntatt mal-kampjun għandu jkun ta' materjal inerti, eż. aluminju, hġieg jew azzar li ma jissaddadx illustrat. Oġġetti tal-plastik bħal polypropylene, PTFE eċċ. għandhom ikunu evitati għaliex l-analit jista' jaqbad ma' dawn il-materjali.

Il-materjal kollu tal-kampjun li l-laboratorju jirċievi għandu jintuża għall-preparazzjoni tal-materjal li se jkun ittestjat. Huma biss kampjuni omogeneizzati b'reqqa li jagħtu riżultati riproducibbli.

Hemm hafna proċeduri speċifiċi sodisfaċenti għall-preparazzjoni ta' kampjuni li jistghu jintużaw.

2. *Trattament tal-kampjun kif jasal fil-laboratorju*

Ithan irqiq (fejn hu rilevanti) u hawwad sewwa l-kampjun aggregat shih billi tuża proċess li jkun intwera li jwassal għal omogeneizzazzjoni shiha.

3. *Suddiviżjoni ta' kampjuni għal finijiet ta' infurzar tal-liġi u difiża*

Il-kampjuni replikati għal infurzar tal-liġi, kummerç (difiza) u referenza għandhom jittiehdu mill-materjal omogenezzat sakemm dan ma jmurx kontra r-regoli nazzjonali dwar tehid ta' kampjuni.

4. *Metodu ta' analiżi li għandu jintuża mil-laboratorju u htigiet ta' kontroll fil-laboratorju*

#### 4.1. Definizzjonijiet

Numru ta' definizzjonijiet użati l-aktar komunement li l-laboratorju jkun mehtieġ juża qed jingħataw hawn taht:

$r$  = Ripetibilità, il-valur li meta ma jintlahaqx id-differenza assoluta bejn ir-rizultati ta' żewġ testijiet singoli mehudin f'kondizzjonijiet ripetibbli (jigifieri, l-istess kampjun, l-istess operatur, l-istess apparat, l-istess laboratorju, u wara intervall qasir ta' hin) tista' tistenna li jkun fil-medda ta' probabbiltà speċifika (tipikament 95%) u għalhekk  $r = 2.8 \times s_r$

$s_r$  = Devjazzjoni standard, ikkalkulata minn rizultati generati taht kondizzjonijiet ripetibbli

$RSD_r$  = Devjazzjoni standard relattiva, ikkalkulata minn rizultati generati taht kondizzjonijiet ripetibbli  $[(s_r/\bar{x}) \times 100]$ .

$R$  = Riproduċibilità, il-valur li meta ma jintlahaqx id-differenza assoluta bejn ir-rizultati ta' żewġ testijiet singoli mehudin f'kondizzjonijiet riproduċibbli (jigifieri fuq materjal identiku mehudin minn operatori f'laboratorji differenti u li jużaw il-metodu ta' ttestjar standardizzat) tista' tistenna li jkun fil-medda ta' ċerta probabbiltà (tipikament 95%);  $R = 2.8 \times s_R$ .

$s_R$  = Devjazzjoni standard, ikkalkulata minn rizultati generati taht kondizzjonijiet riproduċibbli.

$RSD_R$  = Devjazzjoni standard relattiva, ikkalkulata minn rizultati generati taht kondizzjonijiet riproduċibbli  $[(s_R/\bar{x}) \times 100]$ , fejn  $\bar{x}$  hija l-medja tar-rizultati mogħtija mil-laboratorji u kampjuni kollha.

$HORRAT_r$  = ir- $RSD_r$  osservat, diviż bir- $RSD_r$  valur stmat mill-ekwazzjoni Horwitz (1) bl-użu ta' l-assunzjoni  $r = 0.66R$ .

$HORRAT_R$  = il-valur  $RSD_R$  osservat diviż bir- $RSD_R$  valur ikkalkulat mill-ekwazzjoni Horwitz.

U = l-inċertezza mwassgħa, bl-użu ta' fattur ta' kopertura ta' 2 li jagħti livell ta' fiduċja approssimattiv ta' 95%.

#### 4.2. Rekwiżiti ġenerali

Metodi ta' analiżi użati għall-finijiet ta' kontroll ta' l-ikel għandhom jikkonformaw mal-punti 1 u 2 ta' l-Anness mad-Direttiva tal-Kunsill 85/591/KEE.

#### 4.3. Rekwiżiti speċifiċi

Meta ebda metodi speċifiċi għad-determinazzjoni ta' livelli ta' benzo(a)pirin fl-ikel m'huma preskritti fuq livell Komunitarju, laboratorji jistgħu jagħzlu kull metodu validat sakemm il-metodu magħżul jilhaq il-kriterji ta' operat indikati fit-Tabella. Il-validazzjoni idealment għandha tinkludi materjal ta' referenza ċertifikat.

### TABELLA

#### **Kriterji ta' operat għal metodi ta' analiżi għal benzo(a)pirin**

Parametru	Valur/kumment
Applikabilità	Ikel speċifikat f'Regolament (KE) Nru 208/2005
Limitu ta' Tisjib	Mhux aktar minn 0,3 $\mu\text{g}/\text{kg}$
Limitu ta' kwantifikazzjoni	Mhux aktar minn 0,9 $\mu\text{g}/\text{kg}$
Preciżjoni	Valuri $\text{HORRAT}_r$ jew $\text{HORRAT}_R$ inqas minn 1.5 fil-prova kollaborattiva tal-validazzjoni
Rkupru	50 % – 120%
Speċifiċità	Hieles minn interferenzi matrici jew spettrali, verifikazzjoni ta' tisjib pożittiv

#### 4.3.1. Kriterji ta' Operat — Approċċ skond Funzjoni ta' Inċertezza

Izda approċċ ta' inċertezza jista' jintuża wkoll biex iqis l-addattabilità tal-metodu ta' analiżi li għandu jintuża mil-laboratorju. Il-

laboratorju jista' juża metodu li jaghti riżultati fl-ambitu ta' incertezza standard massima. L-incertezza standard massima tista' tkun ikkalkulata bl-użu tal-formola li ġejja:

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

Fejn:

Uf hu l-incertezza standard massima  
 LOD hu l-limitu tat-tisjib li għandu l-metodu  
 C hu l-konċentrament ta' interess

Jekk metodu analitiku jaghti riżultati b'qisien ta' incertezza inqas mill-incertezza standard massima, il-metodu jkun addattat ugwalment għal wiehed li jilhaq il-karatteristiċi ta' operat mogħtija fit-Tabella.

#### 4.4. Kalkolu ta' rkupru u rappurtar ta' riżultati

Ir-riżultat analitiku għandu jkun irrappurtat korrett jew mhux korrett għall-irkupru. Il-mod ta' l-irrappurtar u l-livell ta' rkupru għandhom ikuni rrappurtati. Ir-riżultat analitiku korrett għal irkupru jintuża għall-iċċekkjar tal-konformità (ara l-ħdax-il Skeda, punt 5).

L-analista għandu jinnota l-‘European Commission Report on the relationship between analytical results, the measurement of uncertainty, recovery factors and the provisions in EU food legislation’ (2).

Ir-riżultat analitiku għandu jkun irrappurtat bhala  $x \pm U$  fejn  $x$  hi r-riżultat analitiku u  $U$  hi l-incertezza tal-kejl.

#### 4.5. ‘Standards’ ta' kwalità tal-laboratorju

Laboratorji għandhom jikkonformaw mad-Direttiva 93/99/KEE.

#### 4.6. Konsiderazzjonijiet ohra għall-analiżi

Ittestjar ta' profiċjenza

Parteċipazzjoni fi skemi ta' ttestjar ta' profiċjenza addattati li jikkonformaw ma' l-‘International Harmonised Protocol for the

Proficiency Testing of (Chemical) Analytical Laboratories' (3) żviluppati taht l-awspiċi ta' l-IUPAC/ ISO/AOAC.

#### Kontroll intern tal-kwalità

Laboratorji għandhom ikunu jistgħu juru li għandhom diġà proċeduri ta' kontroll intern ta' kwalità. Eżempji ta' dawn huma l- 'ISO/AOAC/IUPAC Guidelines on Internal Quality Control in Analytical Chemistry Laboratories' (4).

#### REFERENZI

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.
3. ISO/AOAC/IUPAC International Harmonised Protocol for Proficiency Testing of (Chemical) Analytical Laboratories, Editjat minn M. Thompson u R. Wood, *Pure Appl. Chem.*, 1993, 65, 2123-2144 (Ippubblikat ukoll f'J. AOAC International, 1993, 76, 926).
4. ISO/AOAC/IUPAC International Harmonised Guidelines for Internal Quality Control in Analytical Chemistry Laboratories, Editjat minn M. Thompson u R. Wood, *Pure Appl. Chem.*, 1995, 67, 649-666."

L.N. 107 of 2006

**FOOD SAFETY ACT  
(CAP. 449)**

**Contaminants in Food (Sampling and Analysis Methods)  
(Amendment) (No. 2) Regulations, 2006**

IN exercise of the powers conferred by article 10 of the Food Safety Act, the Minister of Health, the Elderly and Community Care has made the following regulations:-

**1.** The title of these regulations is the Contaminants in Food (Sampling and Analysis Methods) (Amendment) (No. 2) Regulations, 2006, and they shall be read and construed as one with the Contaminants in Food (Sampling and Analysis Methods) Regulations, 2004, hereinafter referred to as “the principal regulations”.

Citation.

L.N. 488 of 2004.

**2.** These regulations implement the provisions of Commission Directive 2005/10/EC of 4 February, 2005.

Scope.

**3.** For regulation 7 of the principal regulations there shall be substituted by the following:-

Amends regulation 7 of the principal regulations.

“7.1. The sampling for the official control of the levels of benzo(a)pyrene in foodstuffs shall be carried out in accordance with the methods described in the Eleventh Schedule.

7.2. The sampling preparation and methods of analysis used for the official control of the levels of benzo(a)pyrene in foodstuffs shall comply with the criteria described in the Twelfth Schedule.”.

**4.** Immediately after the Tenth Schedule to the principal regulations there shall be added the following Eleventh Schedule:-

Adds Eleventh Schedule to the principal regulations.

**“ELEVENTH SCHEDULE**

(Equivalent to Annex I of Commission Directive 2005/10/EC)

**METHODS OF SAMPLING FOR OFFICIAL CONTROL OF  
THE LEVELS OF BENZO(A)PYRENE IN FOODSTUFFS**

**1. Purpose and Scope**

Samples intended for official checking of the levels of benzo(a)pyrene in 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 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 Food Safety Commission.

### 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 benzo(a)pyrene 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 subplot. 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 unless this is incompatible with implementation of point 3.6.

### 3.6. Replicate laboratory samples

Replicate laboratory samples for enforcement, trade (defence) and referee purposes shall be taken from the homogenized aggregate sample unless this conflicts with national 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 national rules.

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

In the case of oils, for which a homogeneous distribution of benzo(a)pyrene can be assumed within a given lot, it is sufficient to take three incremental samples per lot to form the aggregate sample. Reference to the lot number shall be given. For olive oil and olive pomace

oil further information on sampling is given in Commission Regulation (EC) No 1989/2003.

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, no less than 100g each, resulting in an aggregate sample of no less than 300g (see point 3.5).

TABLE 1

**Minimum number of incremental samples to be taken from the lot**

Weight of lot (in 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 or subplot	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

#### 4.2. 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 duplicate analyses in cases where the obtained result of the first analysis is less than 20 % below or above the maximum level, and in these cases shall calculate the mean of the results.

The lot is accepted if the result of the first analysis or, where duplicate analysis is necessary, 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 result of the first analysis or, where duplicate analysis is necessary, if the mean exceeds the maximum level beyond reasonable doubt taking into account the measurement uncertainty and correction for recovery.”

5. Immediately after the new Eleventh Schedule to the principal regulations, there shall be added the following Twelfth Schedule:-

Adds Twelfth Schedule to the principal regulations.

#### “TWELFTH SCHEDULE

(Equivalent to Annex II of Commission Directive 2005/10/EC)

#### **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., HPLC grade or equivalent) before use to minimize 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 homogenized material unless this conflicts with national 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 208/2005
Detection limit	Not more than 0,3 µg/kg
Limit of quantification	Not more than 0,9 µg/kg
Precision	HORRAT <sub>r</sub> or HORRAT <sub>R</sub> 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:

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

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 the Eleventh Schedule, 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  $x \pm U$  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.
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  
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