

A.L. 91 ta' l-2005

**ATT DWAR IS-SIGURTÀ FL-IKEL
(KAP. 449)**

**Regolamenti ta' l-2005 li jemendaw ir-Regolamenti dwar
Sustanzi li Jikkontaminaw fl-Ikel
(Metodi kif jittiehdu l-Kampjuni u kif issir l-Analizi)**

BIS-SAHHA tas-setghat moghtija bl-artikolu 10 ta' l-Att dwar is-Sigurtà fl-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-2005 li jemendaw ir-Regolamenti dwar Sustanzi li Jikkontaminaw fl-Ikel (Metodi kif jittiehdu l-Kampjuni u kif issir l-Analizi), u ghandhom jinqraw u jftiehmha waħda mar-Regolamenti ta' l-2004 dwar Sustanzi li Jikkontaminaw fl-Ikel (Metodi kif jittiehdu l-Kampjuni u kif issir l-Analizi), hawn iktar 'l quddiem imsejha "ir-regolamenti prinċipali".

Titolu.

A.L. 488 ta' l-2004.

2. Dawn ir-regolamenti jimplementaw id-disposizzjonijiet tad-Direttiva tal-Kummissjoni 2004/16/KE tal-Komunità Ewropea.

Skop.

3. Dan li ġej għandu jżied bhala s-subparagrafu (1) tas-subregolament 1.2 tar-regolamenti prinċipali:

Jemenda regolament 1 tar-regolamenti prinċipali.

“(1) Direttiva tal-Kummissjoni 2004/16/KE tat-12 ta' Frar 2004 li tistipula l-metodi ta' tehid ta' kampjuni u l-metodi uffiċjali ta' analizi għall-kontroll tal-livelli ta' landa f'oggetti ta' l-ikel fil-laned.”.

4. Ir-regolament 7.1 tar-regolamenti prinċipali għandu jiġi enumerat mill-ġdid bhala r-regolament 8.1.

Jenumera mill-ġdid ir-regolament 7.1 tar-regolamenti prinċipali.

5. Minnufih wara r-regolament 6 tar-regolamenti prinċipali għandu jżied dan ir-regolament ġdid li ġej:-

Iżid regolament 7 ġdid mar-regolamenti prinċipali.

“Metodi kif jittiehdu l-kampjuni u kif issir l-analizi ta' landa f'oggetti ta' l-ikel fil-laned.

7.1 It-tehid ta' kampjuni għall-kontroll uffiċjali tal-livelli ta' landa f'oggetti ta' l-ikel fil-laned għandu jsir skond il-metodi murijin fil-Hdax-il Skeda li tinsab ma' dawn ir-regolamenti.

7.2 Il-preparazzjoni ta' kampjuni u metodi ta' analiżi użati għall-kontroll uffiċjali tal-livelli ta' landa f'ogġetti ta' l-ikel fil-laned għandhom ikunu jikkonformaw mal-kriterji deskritti fit-Tnax-il Skeda li tinsab ma' dawn ir-regolamenti.”

Iżid Il-Hdax-il Skeda mar-regolamenti prinċipali.

6. L-Ewwel Skeda li tinsab ma' dawn ir-regolamenti qeghda tizzied bhala l-Hdax-il Skeda mar-regolamenti prinċipali.

Iżid It-Tnax-il Skeda mar-regolamenti prinċipali.

7. It-Tieni Skeda li tinsab ma' dawn ir-regolamenti qeghda tizzied bhala t-Tnax-il Skeda mar-regolamenti prinċipali.

L-EWWEL SKEDA

“IL-HDAX-IL SKEDA

Metodi ta' Tehid ta' Kampjuni ghal Kontroll Uffiċjali tal-Livelli ta' Landa f'Ogġetti ta' l-Ikel fil-Laned**1. Fini u skop**

Kampjuni mahsubin ghal l-iċċekkjar uffiċjali tal-livelli ta' landa f'ogġetti ta' l-ikel fil-laned ghandhom jittiehdu skond il-metodi deskritti hawn taht. Kampjuni aggregati mehudin b'dan il-mod ghandhom jitqiesu bhala rappreżentattivi tal-lottijiet.

Konformità mal-limiti massimi preskritti fir-Regolament tal-Kummissjoni (KE) Nru 446/2001 ghandha tkun stabbilita fuq il-bażi tal-livelli determinati fil-kampjuni tal-laboratorju.

2. Tifsiriet

Lott: kwantità identifikabbli ta' ogġett ta' l-ikel konsenjata f'okkażjoni wahda u determinata mill-uffiċjal li jkollha karatteristiċi komuni, bhalma huma oriġini, varjetà, tip ta' ppakkjar, min ippakkjaha, min ghamel 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 ghandu jkun separat fizikament 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 ghal-laboratorju.

3. Disposizzjonijiet ġenerali**3.1. Persunal**

L-analiżi tal-kampjuni ghandha ssir minn persuna awtorizzata kif speċifikat mill-Kummissjoni ghas-Sigurtà ta' l-Ikel.

3.2. Materjal ta' kampjun li se jkun analizzat

Kull lott li se jkun eżaminat ghandu jittiehed minnu kampjuni separatament.

3.3. *Prekawzjonijiet li għandhom jittieħdu*

Fil-kors tat-teħid tal-kampjuni u l-preparazzjoni tal-kampjuni tal-laboratorju għandhom jittieħdu prekawzjonijiet biex ikunu evitati kull xorta ta' bidliet li jaffettwaw il-kontenut ta' landa, jaffettwaw hażin id-determinazzjoni analitika jew jirrendu l-kampjuni aggregati mhux rappreżentattivi.

3.4. *Kampjuni inkrimentali*

Safejn ikun possibbli, kampjuni inkrimentali għandhom jittieħdu minn diversi partijiet imqassmin mal-lott jew sublott kollu. Jekk din il-proċedura ma tintużax dan għandu jkollha fir-rekord.

3.5. *Preparazzjoni tal-kampjun aggregat*

Il-kampjun aggregat hu magħmul bit-tagħqid tal-kampjuni inkrimentali kollha. Dan il-kampjun aggregat għandu jkun omogenezzat fil-laboratorju.

3.6. *Kampjuni replikati fil-laboratorju*

Il-kampjuni replikati fil-laboratorju għall-finijiet ta' infurzar tal-liġi, kummerċ (difiza) u referenza għandhom jittieħdu mill-kampjun aggregat omogenezzat sakemm dan ma jmurx kontra regoli ohra dwar teħid ta' kampjuni.

3.7. *Ippakkjar u ġarr ta' kampjuni*

Kull kampjun għandu jkollha f'kontenitur nadif u inert li joffri protezzjoni adegwata minn kontaminazzjoni u kontra ħsarat waqt il-ġarr. Kull prekawzjoni neċessarja għandha tittieħed biex tkun evitata bidla fil-kompożizzjoni tal-kampjun li tista' tokkorri waqt il-ġarr jew il-ħżin.

3.8. *Issiġillar u tikkettjar ta' kampjuni*

Kull kampjun meħud għal użu uffiċjali għandu jkun issiġillat fil-post fejn issir l-analiżi u identifikat skond preċeduri stabbiliti mill-Kummissjoni għas-Sigurtà ta' l-Ikel. Għandu jkollha record ta' kull teħid ta' kampjun li jippermetti li kull lott ikun identifikat mingħajr ambigwiżità u li jagħti d-data u l-post minn fejn ikun ittieħed il-kampjun flimkien ma' kull informazzjoni addizzjonali li x'aktarx tista' tkun ta' għajjnuna għall-analista.

4. **Pjanijiet ta' teħid ta' kampjuni**

Il-metodu ta' teħid ta' kampjun applikat għandu jkollha li l-kampjun aggregat ikun rappreżentattiv tal-lott li jkun se jkollha kontrollat.

4.1. *Għadd ta' kampjuni inkrimentali*

L-inqas għadd ta' kampjuni inkrimentali li għandhom jittiehdu mil-laned f'lott għandu jkun kif mogħti f'Tabella 1. Il-kampjuni inkrimentali mehudin minn kull landa għandhom ikunu ta' piż simili, u jirrizultaw f'kampjun aggregat (ara punt 3.5).

Tabella 1

Għadd ta' laned (kampjuni inkrimentali) li għandhom jittiehdu biex jiffurmaw il-kampjun aggregat

Għadd ta' laned fil-lott jew sublott	Għadd ta' laned li għandhom jittiehdu
1 sa 25	mill-inqas landa waħda
26 sa 100	mill-inqas 2 laned
> 100	5 laned

Innota li l-livelli massimi japplikaw għall-kontenut ta' kull landa, iżda biex l-ittestjar ikun jista' jsir ikun meħtieġ li jintuza approċċ ta' teħid tal-kampjuni aggregat. Jekk ir-riżultat tal-kampjun aggregat ikun inqas minn, iżda qrib, il-livell massimu u jekk ikun hemm suspett li laned individwali jistgħu jaqbu l-livell massimu, allura jista' jkun meħtieġ li jsiru investigazzjonijiet ulterjuri.

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

Tehid ta' kampjuni ta' oġġetti ta' l-ikel fl-istadju tal-bejgħ bl-imnut għandu jsir, meta possibbli, skond id-disposizzjonijiet ta' hawn qabel għat-teħid ta' kampjuni. Meta dan ma jkunx possibbli, proċeduri effettivi ohra ta' teħid ta' kampjuni fl-istadju tal-bejgħ bl-imnut jistgħu jintużaw sakemm jiżguraw rappreżentattività biżżejjed għal-lott li jkun qed jittiehed bhala kampjun.

5. Konformità tal-lott jew sublott ma' l-ispeċifikazzjoni

Il-laboratorju li jagħmel il-kontroll għandu janalizza l-kampjun tal-laboratorju għall-infurzar tal-liġi mill-inqas f'żewġ analiżi indipendenti u jikkalkula l-medja tar-riżultati. Il-lott hu aċċettat jekk il-medja ma taqbiżx il-livell massimu rispettiv (kif imniżżel fir-Regolament (KE) Nru 466/2001) waqt li jingħata każ ta' l-inċertezza fil-kejl u l-korrezzjoni għal irkuprar. Il-lott ma jikkonformax mal-livell massimu (kif imniżżel fir-Regolament (KE) 466/2001) jekk il-medja tkun taqbeż bla ebda dubju l-livell massimu waqt li jingħata każ ta' l-inċertezza fil-kejl u l-korrezzjoni għal irkuprar.”.

IT-TIENI SKEDA

“IT-TNAX-IL SKEDA

Preparazzjoni ta' Kampjuni u Kriterji għal Metodi ta' Analizi Uzati fil-Kontroll Uffiċjali tal-Livelli ta' Landa f'Ogġetti ta' l-Ikel fil-Laned**1. Prekawzjonijiet u konsiderazzjonijiet ġenerali fil-każ tal-landa**

Ir-rekwizit bażiku hu li jittiehed kampjun tal-laboratorju rappreżentattiv u omogeneu bla ma tiddahhal kontaminazzjoni sekondarja. L-analista għandu jiżgura li l-kampjuni ma jiġux ikkontaminati waqt il-preparazzjoni tal-kampjun. Kull meta hu possibbli, apparat li jiġi f'kuntatt mal-kampjun għandu jkun magħmul minn materjali inert, bħalma huma materjali plastiċi bħall-polipropileni, PTFE eċċ., u dawn għandhom ikunu mnaddfin minn aċidi biex inaqqsu kemm jista' jkun ir-riskju tal-kontaminazzjoni. *Stainless steel* ta' kwalità għolja jista' jintuża għal għub li jaqtgħu. Il-materjal kollu tal-kampjuni li jasal fil-laboratorju għandu jintuża għall-preparazzjoni tal-materjal li se jkun ittestjat. Riżultati riproducibbli jistgħu jinkisbu biss minn kampjuni omogeneizzati b'mod fin hafna. Hemm hafna proċeduri speċifiċi sodisfaċenti ta' preparazzjoni ta' kampjuni x'jistgħu jintużaw. Dawk deskritti f'CEN Standard “*Determination of trace elements – Performance criteria and general consideration*” nstabu li huma sodisfaċenti (1) imma ohrajn jistgħu jkunu daqstant iehor validi.

2. Trattament ta' kampjun kif jidhol fil-laboratorju

Ishaq tajjeb hafna (meta rilevanti) u hawwad tajjeb hafna l-kampjun aggregat shih billi tuża proċess li diġà ntweru li jwassal għal omogeneizzazzjoni shiha.

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

Il-kampjuni replikati għal skopijiet ta' infurzar tal-liġi, kummerċ (difiża) u referenza għandhom jittiehdu mill-materjal omogeneizzat sakemm dan ma jkunx imur kontra regoli ohra dwar tehid ta' kampjuni.

4. Metodu ta' analiżi li għandu jintuża mill-laboratorju u rekwiżiti ta' kontroll tal-laboratorju**4.1. Tifsiriet**

Numru ta' tifsiriet uzati 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-riżultati ta' żewġ testijiet singoli mehudin f'kondizzjonijiet ripetibbli (jiġifieri, 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 ghalhekk $r = 2.8 \times s_r$

s_r = Devjazzjoni *standard*, ikkalkulata minn riżultati ġenerati taht kondizzjonijiet ripetibbli.

RSD_r = Devjazzjoni standard relattiva, ikkalkulata minn riżultati ġenerati taht kondizzjonijiet ripetibbli $[(s_r/\bar{x}) \times 100]$, fejn \bar{x} hija l-medja tar-riżultati mogħtija mill-laboratorji u kampjuni kollha.

R = Riproduċibilità, il-valur li, meta ma jintlahaqx, id-differenza assoluta bejn ir-riżultati ta' testijiet singoli meħudin f'kondizzjonijiet riprodubbli (jiġifieri fuq materjal identiku meħud minn operatori f'laboratorji differenti u li jużaw il-metodu ta' ittestjar standardizzat) tista' tistenna li jkun fil-medda ta' ċerta probabbiltà (tipikament 95 %); $R = 2,8 \times s_R$.

s_R = Devjazzjoni *standard*, ikkalkulata minn riżultati ġenerati taht kondizzjonijiet ripetibbli.

RSD_R = Devjazzjoni standard relattiva, ikkalkulata minn riżultati ġenerati taht kondizzjonijiet ripetibbli $[(s_R/\bar{x}) \times 100]$.

$HORRAT_r$ = il-valur RSD_r osservat diviż bil-valur RSD_r stmat mill-ekwazzjoni Horwitz bl-użu ta' l-assunzjoni $r = 0,66R$.

$HORRAT_R$ = il-valur RSD_R osservat diviż bil-valur RSD_R ikkalkulat mill-ekwazzjoni Horwitz (2).

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

4.2. *Rekwiżiti ġenerali*

Metodi ta' analiżi użati għall-finijiet ta' kontroll ta' l-ikel għandhom jikkonformaw mad-disposizzjonijiet ta' punti 1 u 2 ta' l-Anness tad-Direttiva tal-Kunsill 85/591/KEE ta' l-20 ta' Diċembru 1985 dwar l-introduzzjoni ta' metodi Komunitarji fuq teħid ta' kampjuni u analiżi għall-monitoraġġ ta' oġġetti ta' l-ikel maħsubin għall-konsum mill-bniedem.

4.3. *Rekwiżiti speċifiċi*

Meta ebda metodu speċifiku għad-determinazzjoni ta' landa f'oġġetti ta' l-ikel fil-laned ma jkun preskritt f'livell Komunitarju, laboratorji jistghu jagħzlu kull metodu validat sakemm il-metodu magħżul jilhaq il-kriterji indikati fit-Tabella 2. Il-validazzjoni għandha idealment tinkludi materjal ta' referenza ċertifikat.

Tabella 2

Kriterji ta' operat ghal metodi ta' analiżi ghal-landa

Parametri	Valur/Kumment
Applikabilità	Ikel speċifikat fir-Regolament (KE) Nru 242/2004
Limitu ta' tkejjil	Mhux aktar minn 5 mg/kg
Limitu ta' kwantifikazzjoni	Mhux aktar minn 10 mg/kg
Preċiżjoni	Valuri HORRAT _r jew HORRAT _R ta' inqas minn 1,5 fit-test kollaborattiv tal-validazzjoni
Rkupru	80 % sa 105 % (kif indikat fit-test kollaborattiv)
Speċifiċità	hieles minn interferenzi ta' matriċi jew spettrali

4.3.1. Kriterji ta' Operat — Approċċ għall-Funzjoni ta' Inċertezza

Iżda approċċ għall-inċertezza jista' jintuża biex jitkejjel kemm ikun adatt il-metodu ta' analiżi li għandu jintuża mil-laboratorju. Il-laboratorju jista' juża metodu li jagħti riżultati sa inċertezza massima *standard*. L-inċertezza massima *standard* tista' tkun ikkalkulata billi tintuża l-formola li ġejja:

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

fejn:

U_f hi inċertezza massima *standard*

LOD hi l-limitu ta' tkejjil tal-metodu

C hi l-konċentrazzjoni ta' interess

Jekk metodu analitiku jagħti riżultati b'kejl inċert li jkun inqas mill-inċertezza massima *standard*, il-metodu ikun adatt ugwalment għal wiehed li jilhaq il-karatteristiċi ta' operat mogħtija f'Tabella 2.

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

Ir-riżultat analitiku għandu jkun irrappurtat ikkoregut jew mhux ikkoregut għal irkupru. Il-mod kif ikun irrappurtat u l-livell ta' rkupru għandhom ikunu rrappurtati. Ir-riżultat analitiku ikkoregut għal irkupru għandu jintuża biex tkun iċċekkjata l-konformità (ara

Anness I, punt 5). L-analista ghandu jaghti każ tal-“*Harmonised Guidelines for the Use of recovery Information in Analytical Measurement*” (3) żviluppati taht l-IUPAC/ISO/AOAC. Dawn il-Linji Gwida jghinu fid-determinazzjoni ta' fatturi ta' rkupru. Ir-rizultat analitiku ghandu jkun irrappurtat bhala $x \pm U$, fejn x hija r-rizultat analitiku u U hija l-inċertezza tal-kejl..

4.5. *Standards ta' kwalità tal-laboratorju*

Laboratorji ghandhom jikkonformaw mad-Direttiva tal-Kunsill 93/99/KEE tad-29 ta' Ottubru 1993 fuq is-sugġett ta' miżuri addizzjonali dwar il-kontroll ufficjali ta' oġġetti ta' l-ikel.

4.6. *Konsiderazzjonijiet oħra għall-analiżi*

Ittestjar ta' profiċjenza

Parteċipazzjoni fi skemi adatti ta' testjar ta' profiċjenza li jikkonformaw mal-“*International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories*” (4) żviluppati taht l-awspicji tal-IUPAC/ISO/AOAC. Uħud minn dawn l-iskemi jinkludu speċifikament id-determinazzjoni ta' landa fl-ikel, u parteċipazzjoni fi skemi ta' dix-xorta hija rikmandata aktar minn skema ġenerali għad-determinazzjoni ta' metalli fl-ikel.

Kontroll ta' kwalità intern

Laboratorji ghandhom ikunu kapaċi juru li ghandhom lesti proċeduri ta' kontroll ta' kwalità intern. Eżempji ta' dan huma “*ISO/AOAC/IUPAC Guidelines on Internal Quality Control in Analytical Chemistry Laboratories*” (5).

Preparazzjoni ta' kampjun

Ghandu tinghata attenzjoni biex ikun żgurat li l-landa kollha fil-kampjun tittiehed fis-soluzzjoni għall-analiżi. B'mod partikolari hu rikonoxxut li l-proċedura tad-dissoluzzjoni tal-kampjun għandha tkun tali li ebda speċi ta' Sn(IV) idrolizzat m'hi preċipitata (jiġifieri, speċi bħal ossidu stanniku SnO_2 , Sn(OH)_4 , $\text{SnO}_2 \cdot \text{H}_2\text{O}$). homm kampjuni lesti f'5 mol/l HCl. Izda SnCl_4 jiġi faċilment volatilizzat u għalhekk is-soluzzjonijiet m'għandhomx jitghallew.

REFERENZI

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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.”.

L.N. 91 of 2005

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

**Contaminants in Food (Sampling and Analysis Methods)
(Amendment) Regulations, 2005**

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) Regulations, 2005, 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 2004/16/EC of the European Community. Scope.

3. The following shall be added as sub-paragraph (1) of subregulation 1.2 of the principal regulations: Amends regulation 1 of the principal regulations.

“(1) Commission Directive 2004/16/EC of 12 February, 2004 laying down the sampling methods and the official methods of control for the levels of tin in canned foods.”.

4. Regulation 7.1 of the principal regulations shall be renumbered as regulation 8.1 thereof. Renumbers regulation 7.1 of the principal regulations.

5. Immediately after regulation 6 of the principal regulations there shall be added the following new regulation:- Adds new regulation 7 to the principal regulations.

“Sampling and analytical methods for Tin in canned foods. **7.1** The sampling for the official control of the levels of tin in canned foods shall be carried out in accordance with the methods described in the Eleventh Schedule to these regulations.

7.2 The sample preparation and methods of analyses used for the official control of the levels of tin in canned foods shall comply with the criteria described in the Twelfth Schedule to these regulations.”.

Adds Eleventh
Schedule to the
principal
regulations.

6. The First Schedule to these regulations is being added as the Eleventh Schedule to the principal regulations.

Adds Twelfth
Schedule to the
principal
regulations.

7. The Second Schedule to these regulations is added as the Twelfth Schedule to the principal regulations.

FIRST SCHEDULE

“ELEVENTH SCHEDULE

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 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 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 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.

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 other 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 procedures established by the Food Safety Commission. 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.”.

SECOND SCHEDULE

“TWELFTH SCHEDULE

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 other 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 Sn(IV) 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.

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