



**Development work related to  
the Food Adulteration (Metallic  
Contamination) (Amendment)  
Regulation 2018**

*5.3.2019*

























# Method development for CAP132V amendment

Calculate the concentration of analytes in a food sample as follows:

$$\underline{M} \text{ (mg/kg)} = (A \times D \times V) / W$$

- M** = concentration of analyte in the sample, mg/kg  
**A** = concentration of analyte in the sample solution,  $\mu\text{g/L}$   
**D** = dilution factor of the sample solution, if any  
**V** = volume of sample solution made up after digestion, L  
**W** = weight of the sample taken for analysis, g

decrease in A or increase in W will make M decrease



# Arsenic in CAP 132V amendment

## Current Reporting Limit requirement in GL contract: 0.1 ppm

Food items, Existing maximum permitted concentration (expressed as (As<sub>2</sub>O<sub>3</sub>)) (ppm), Existing maximum permitted concentration (expressed as inorganic arsenic)(ppm), Proposed maximum level (ML)(Expressed as total arsenic, mg/kg, unless otherwise specified)

Vegetables,	1.4,	1.1,	0.5			
Cereals,	1.4,	1.1,	0.5			
Meat of animal,	1.4,	1.1,	0.5			
Edible fats and oils,	1.4,					
Fat spreads and blended spreads,	1.4,	1.1,	0.1			
Salt, food grade,	1.4,	1.1,	0.5			
Natural mineral waters,	0.14,	0.1,	0.01 (mg/L)			
Bottled/package drinking waters (other than natural mineral waters),	0.14,	0.1,	0.01 (mg/L)			



# Arsenic in CAP 132V amendment

Food items, Existing maximum permitted concentration (expressed as  $\text{As}_2\text{O}_3$ ) (ppm), Existing maximum permitted concentration (expressed as inorganic arsenic)(ppm), Proposed maximum level (ML)(Expressed as inorganic arsenic, mg/kg, unless otherwise specified)

Rice husked,	1.4,	1.1,	0.35	
Rice polished,		1.4,	1.1,	0.2
Aquatic animals,		10,	7.2,	0.5
Fish,	6,	4.5,	0.1	
Fish oil,	0.14,	0.1,	0.1	
Seaweed,	1.4,	1.1,	1	



# Mercury in CAP 132V amendment

Current Reporting Limit requirement in GL contract: 0.03 ppm

Food items, Existing maximum permitted concentration(ppm), otherwise specified)	Proposed maximum level (ML)(mg/kg, unless otherwise specified)
Fish, 0.5 (total mercury),	0.5 (Expressed in methylmercury)
Vegetables, 0.5 (total mercury),	0.01 (total mercury)
Edible fungi, 0.5 (total mercury),	0.1 (total mercury)
Rice, husked rice, polished rice, maize, maize flour, wheat, wheat flour,	0.5 (total mercury) 0.02 (total mercury)
Meat of animal, 0.5 (total mercury),	0.05 (total mercury)
Animal, edible offal of, 0.5 (total mercury),	0.05 (total mercury)
Aquatic animals, 0.5 (total mercury),	0.5 (total mercury)
Milk, 0.5 (total mercury),	0.01 (total mercury)
Secondary milk products, 0.5 (total mercury),	0.01 (total mercury)





# Speciation: IUPAC-Definition

## (IUPAC – International Union for Pure and Applied Chemistry)

- ***Chemical species.*** Chemical element: specific form of an element defined as to isotopic composition, electronic or oxidation state, and/or complex or molecular structure.
- ***Speciation analysis.*** Analytical chemistry: analytical activities of identifying and/or measuring the quantities of one or more individual chemical species in a sample.



# Speciation Analysis: Problems to be Solved

- How do we take samples of the materials and isolate the species without changing its chemical form?
  - Appropriate procedures for sampling and processing
- Can we measure very small amounts of the isolated species, which may be only a minute fraction of the total?
  - Sensitive measuring methods and devices
- How do we validate methods of elemental analysis?
  - Calibration standards, Certified Reference Materials



# Speciation Analysis: Problems to be Solved

- Key requirement: preservation of the species information during the whole analytical process
- Principal strategy:
  - Keep the chemical species of interest unchanged or changed as designed during all critical steps of the analysis.



# Speciation Analysis

## Preparation / Pre-treatment: Solid Samples

- Solid-liquid extraction (leaching)

- Mechanical shaking
- Microwave assisted extraction
- Ultrasound assisted extraction
- Pressurized liquid extraction
- Sequential extraction
  - extractions with different solvents in a successive way



# Tools for elemental speciation analysis

Different separation techniques have been successfully coupled with sensitive detection techniques:

## Major separation techniques:

- Liquid Chromatography (LC, e.g. HPLC)
- Gas Chromatography (GC)

## Robust excitation source—Plasma (ICP):

- Continuous mode operation for easy hyphenation with chromatographic techniques
- High sensitivity for the elements
- Simplified calibration, relying on single standards, due to complete dissociation of compounds; linear behavior over many orders of magnitude
- Simultaneous sampling for multi-elements



# Methods for Methylmercury

## Available International / National Standards:

- China (GB): GB 5009.17-2014
- EU: EN 16801-2016
- USA: FDA EAM 4.8



# Methods for Methylmercury

GB 5009.17-2014

scope Food

Extraction solution 5M HCl

Extraction condition Ultrasonic bath 60 min

neutralize with NaOH, add L-cysteine solution

Cleanup -

Determination technique LC-AFS

Calibration External Calibration (MeHg)

BS EN 16801-2016

scope aquatic product

Extraction solution 25% TMAH solution

Extraction condition Ambient, overnight

Cleanup Extract by hexane

Determination technique GC-ICPMS

(Et4B derivatisation in hexane)

Calibration IDMS



# Methods for Methylmercury

FDA EAM 4.8

scope aquatic product

Extraction solution L-cysteine solution

Extraction condition 60C 120 min

Cleanup -

Determination technique LC-ICPMS

Calibration External Calibration (MeHg)

GL Method

scope Fish

Extraction solution

Mercaptoethanol solution

Extraction condition 60C 15 min

Cleanup

Determination technique LC-ICPMS

Calibration External Calibration (MeHg)





# Methods for Inorganic Arsenic

## Available International / National Standards:

- China (GB): GB 5009.11-2014
- EU: EN 16802-2016
- USA: FDA EAM 4.11



# Methods for Inorganic Arsenic

GB 5009.11-2014                      Method 1  
Scope    rice, aquatic product, infant formula  
Extraction solution                      0.15M HNO<sub>3</sub>  
Extraction condition                      90C, 150min  
Cleanup hexane wash and C18 SPE (except for rice)  
Determination technique    IC AFS  
Calibration                      As III and As V

GB 5009.11-2014                      Method 2  
Scope    rice, aquatic product, infant formula  
Extraction solution                      0.15M HNO<sub>3</sub>  
Extraction condition                      90C, 150min  
Cleanup hexane wash and C18 SPE (except for rice)  
Determination technique    IC ICPMS  
Calibration                      As III and As V



# Methods for Inorganic Arsenic

BS EN 16802-2016

Scope foodstuff from marine and plant origin

Extraction solution 0.1M HNO<sub>3</sub> + 3% H<sub>2</sub>O<sub>2</sub>

Extraction condition 90C, 60min

Cleanup Nil

Determination technique ICICPMS

Calibration As V

FDA EAM 4.11

Scope rice and rice product

Extraction solution 0.28M HNO<sub>3</sub>

Extraction condition 95C, 90min

Cleanup Nil

Determination technique ICICPMS

Calibration As III and As V



# Methods for Inorganic Arsenic

GL Method

Scope rice, aquatic product

Extraction solution 0.15M / 0.28M HNO<sub>3</sub> + 3% H<sub>2</sub>O<sub>2</sub>

Extraction condition 90C, 180min (rice); 100C, 180min  
(aquatic product)

Cleanup C18 powder (except for rice)

Determination technique IC ICPMS

Calibration As V



# GLHK

## Proficiency Testing Scheme Provider

- Since 1987, Government Laboratory has organized PT.
- Over 50 proficiency testing programmes were organized, and about 30 of them are related to food testing parameters such as, food additives, contaminants, residues of pesticides and veterinary drugs as well as metals.
- GLHK was accredited as a proficiency testing scheme provider according to requirements of the international standards ISO/IEC 17043:2010 in 2009.



# GLHK

## Proficiency Testing Scheme Provider

Programme - No. of Participants - Completion Date

Food Safety Testing – Determination of Elements (Calcium and Cadmium) in Drinking Water (APLAC T095) – 99 - Jul-2016

Food Safety Testing – Essential and toxic elements in seafood (APMP PT 11-01) -14- Sep-2013 Food Safety Testing – Essential and trace elements in seafood (APLAC T082) -71- Aug-2013

Food Safety Testing – Essential and trace elements in bovine liver (APLAC T077) -38- Jun-2011 Food

Safety Testing – Heavy metals in crustacean seafood (GLHK 1002) -68- Oct-2010

Chinese Medicine Testing – Heavy metal in herb (APLAC T065) -109- Oct-2008

Food Safety Testing – Toxic elements in seawater shrimp (APLAC T057) -102- Jul-2007



# Workflow of PT Programmes

Organisation of Proficiency Testing Programmes in accordance with ISO/IEC 17043:2010

## 1 Feasibility Study

Sourcing bulk material and standards

Analytical methods development

## 2 Material Preparation

Drying, homogenization, particle size reduction, sieving, irradiation

## 3 Homogeneity Study

## 4 Stability Study

Short-term (transportation)

Long-term (period of validity)

## 5 Determine assigned value

## 6 Measurement by participants

## 7 Stability Study (Long-term) after dispatch and results submitted

## 8 Result evaluation

Assigned value

z scores

Robust analysis

## 9 Preparation and issue of reports



# Workflow of PT Programmes

Homogenize the aquatic products purchased in market

Dry the sample with freeze dryer

Fix the particle size range of sample by suitable sieves

Mix the sample in a 3-D mixer for 5 days

Each sample about 20g and stored under inert atmosphere

Sample ready for homogeneity and stability study





# **GLHK**

## **Proficiency Testing Scheme Provider**

### **Inorganic arsenic in aquatic product**

#### **Event Period**

<b>Call for Participation</b>	<b>4 October 2018</b>
<b>Deadline of Registration</b>	<b>26 October 2018</b>
<b>Sample Collection</b>	<b>5-7 November 2018</b>
<b>Deadline of Result Submission</b>	<b>31 December 2018</b>
<b>Statistical Analysis of Results</b>	<b>January 2019</b>
<b>Distribution of Final Report</b>	<b>February 2019</b>



# GLHK

## Proficiency Testing Scheme Provider

**Inorganic arsenic in aquatic product**

- **Total 10 laboratories registered**
- **9 Laboratories reported results**
- **1 Laboratory reported result for total arsenic**
- **7 Laboratory got satisfactory results**



# Experience Sharing / Knowledge Transfer

## ➤ **Government Laboratory**

- Technical Briefings to testing laboratories (13 October 2017, 19 October 2018)

## ➤ **Centre of Food Safety**

- Technical Meeting to food trade and testing laboratories (23 March 2018)

## ➤ **The Hong Kong Council for Testing and Certification**

- Technical Seminar to food trade and testing laboratories (25 May 2018)



*Thank you*

