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

5.3.2019

Elements in CAP 132V amendment

Barium

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Natural mineral waters	Not available	0.7 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)		1.3 (mg/L)

Boron

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Natural mineral waters	Not available	5 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)		2.4 (mg/L)

Elements in CAP 132V amendment

Copper

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Natural mineral waters	Not available	1 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)		2 (mg/L)

Manganese

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Natural mineral waters	Not available	0.4 (mg/L)

Elements in CAP 132V amendment

Nickel

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Natural mineral waters	Not available	0.02 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)		0.07 (mg/L)

Selenium

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Natural mineral waters	Not available	0.01 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)		0.04 (mg/L)

Uranium

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Bottled/packaged drinking waters (other than natural mineral waters)	Not available	0.03 (mg/L)

Antimony in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.2 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Vegetables	1	1
Cereals	1	1
Meat of animal	1	1
Meat of poultry	1	1
Fish	1	1
Crabs, prawns and shrimps	1	1
Oysters	1	1
Natural mineral waters	Not available	0.005 mg/L
Bottled/packaged drinking waters (other than natural mineral waters)	Not available	0.02 mg/L

Food items	Recommendations from Codex: Portion of the commodity to which the ML applies (and which is analysed)
Bulb vegetables	Bulb onions: whole commodity after removal of roots and adhering soil and whatever parchment skin is easily detached. Green onions: whole vegetables after removal of roots and adhering soil.
Brassica vegetables, other than Brassica leafy vegetables	Head cabbages: whole commodity as marketed, after removal of obviously decomposed or withered leaves. Cauliflower and broccoli: flower heads (immature inflorescence only). Brussels sprouts: "buttons" only. Kohlrabi: "tuber-like enlargement of the stem" only.
Fruiting vegetables, Cucurbits and Fruiting vegetables, other than Cucurbits	Whole commodity after removal of stems.
Leafy vegetables (including Brassica leafy vegetables)	Whole commodity as usually marketed, after removal of obviously decomposed or withered leaves.
Legume vegetables	Whole commodity, unless otherwise specified.
Pulses	Whole commodity.
Root and tuber vegetables	Whole commodity after removing tops. Remove adhering soil (e.g. by rinsing in running water or by gentle brushing of the dry commodity). Potato: peeled potato.
Stalk and stem vegetables	Whole commodity as marketed after removal of obviously decomposed or withered leaves. Rhubarb: leafy stem only. Globe artichoke: flower head only. Celery and asparagus: removing adhering soil.

Chromium in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.2 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Vegetables	1	0.5
Pulses	1	1
Cereals	1	1
Meat of animal	1	1
Meat of poultry	1	1
Fish	1	1
Crabs, prawns and shrimps	1	1
Oysters	1	1
Natural mineral waters	Not available	0.05 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)	Not available	0.05 (mg/L)

Cadmium in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.02 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Bulb vegetables	0.1	0.05
Brassica vegetables	0.1	0.05
Fruiting vegetables, Cucurbits	0.1	0.05
Fruiting vegetables, other than Cucurbits	0.1	0.05
Leafy vegetables (including Brassica leafy vegetables)	0.1	0.2
Legume vegetables	0.1	0.1
Pulses	0.1	0.1
Root and tuber vegetables	0.1	0.1
Stalk and stem vegetables	0.1	0.1
Vegetables unless otherwise specified	0.1	0.1
Cereals	0.1	0.1
Rice, husked	0.1	0.2
Rice, polished	0.1	0.2
Meat of cattle, pigs and sheep	0.2	0.05
Liver of cattle, pigs and sheep	Not available	0.5

Cadmium in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.02 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Kidney of poultry	Not available	1
Fish	2	0.1
Bivalve molluscs	2 [oysters] / Not available	2
Cephalopods	Not available	2
Crustaceans	2 [crab-meat, prawns and shrimps] / Not available	2
Gastropods	Not available	2
Salt, food grade	Not available	0.5
Natural mineral waters	Not available	0.003 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)	Not available	0.003 (mg/L)

Tin in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 10 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Canned foods	230	250
Canned beverages	230	150

Lead in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.1 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Fruits	6	0.1
Cranberry	6	0.2
Currants	6	0.2
Elderberry	6	0.2
Bulb vegetables	6	0.1
Brassica vegetables	6	0.1
Fruiting vegetables, Cucurbits	6	0.05
Fruiting vegetables, other than Cucurbits	6	0.05
Leafy vegetables (Brassica leafy vegetables)	6	0.3
Legume vegetables	6	0.1

Lead in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.1 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Processed tomato concentrates	6	0.05
Table olives	6	0.4
Pickled cucumbers (cucumber pickles)	6	0.1
Canned chestnuts and canned chestnuts puree	6	0.05
Cereal grains	6	0.2
Meat of cattle, pigs and sheep	6	0.1
Meat of poultry	6	0.1
Cattle, edible offal of	6	0.5
Pig, edible offal of	6	0.5
Poultry, edible offal of	6	0.5
Aquatic animals	6	1
Fish	6	0.3
Bivalve molluscs	6	1.5

Lead in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.1 ppm

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
Milk	1	0.02
Secondary milk products	6 [solid food] /1[liquid]	0.02
Infant formula, formula for special medical purposes intended for infants and follow-up formula	6	0.01
Eggs	6	0.2
Lime preserved eggs	6	0.5
Edible fats and oils	6 [solid food] /1[liquid]	0.1
Fat spreads and blended spreads	6	0.1
Salt, food grade	6	2
Natural mineral waters	1	0.01 (mg/L)
Bottled/packaged drinking waters (other than natural mineral waters)	1	0.01 (mg/L)
Fruit juices	1	0.03

Method development for CAP132V amendment

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

$$M\ (mg/kg) = (A \times D \times V) / W$$

M = **concentration of analyte in the sample, mg/kg**

A = **concentration of analyte in the sample solution, μg/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**

e.g. A 2↓ => M 2↓ ; W 2↑ => M 2↓

Lowest calibrated level; instrument sensitivity

Amount of sample used

Arsenic in CAP 132V amendment

Current Reporting Limit requirement in GL contract : 0.1 ppm

Food items	Existing maximum permitted concentration (expressed as (As_2O_3)) (ppm)	Existing maximum permitted concentration (expressed as inorganic arsenic) (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)	
			Expressed as total arsenic	
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 [solid food]/ 0.14 [liquid food]	1.1 [solid food]/ 0.1 [liquid food]	0.1	
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/packaged 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 (As_2O_3)) (ppm)	Existing maximum permitted concentration (expressed as inorganic arsenic) (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)	
			Expressed as inorganic arsenic	
Rice, husked	1.4	1.1	0.35	
Rice, polished	1.4	1.1	0.2	
Aquatic animals	10	7.9	0.5	
Fish	6	4.8	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)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)
		Expressed in methylmercury
Fish	0.5 (total mercury)	0.5
		Expressed in total mercury
Vegetables	0.5 (total mercury)	0.01
Edible fungi	0.5 (total mercury)	0.1
Rice, husked rice, polished rice, maize, maize flour, wheat, wheat flour	0.5 (total mercury)	0.02
Meat of animal	0.5 (total mercury)	0.05
Animal, edible offal of	0.5 (total mercury)	0.05
Aquatic animals	0.5 (total mercury)	0.5
Milk	0.5 (total mercury)	0.01
Secondary milk products	0.5 (total mercury)	0.01

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.

Chemical species: Structural Aspects

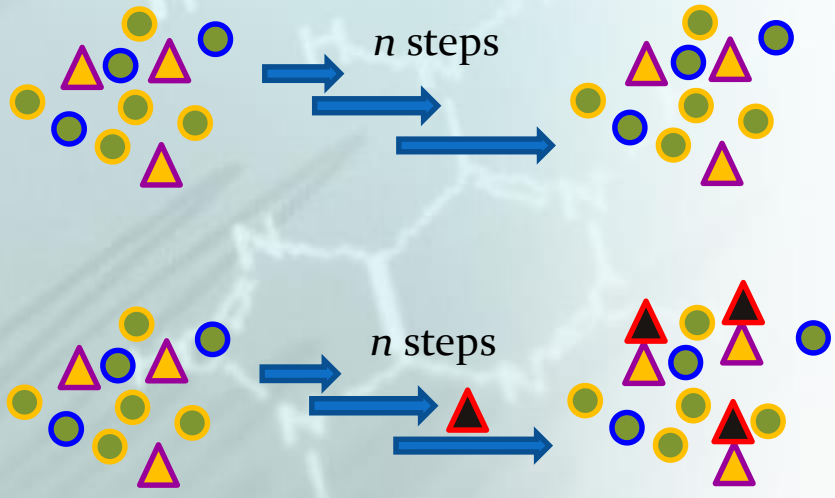
Low Molecular Mass	**Isotopic Elements and Compounds** ^{127}I, ^{129}I D_2O, $H_2{}^{18}O$	**Electronic and Oxidation States Compounds** $As^{III}{}_2O_3$, $As^{V}{}_2O_5$ $Cr^{III}{}_2O_3$, $[Cr^{VI}O_4]^{2-}$
Medium Molecular Mass	**Inorganic Complexes** Cisplatin cis-$[Cl_2Pt(NH_3)_2]$ Gadopentetate $[Gd(DTPA)(H_2O)]^{2-}$	**Organometallic species** Methylmercury $(CH_3)Hg^+$ Methylarsonic acid $(CH_3)AsO(OH)_2$ Arsenobetaine $[(CH_3)_3As^+CH_2COO^-]$ Tributyltin $[(C_4H_9)_3Sn]^+$ (TBT)
High Molecular Mass	**Macromolecular Compounds** Vitamin B_{12} (methylcobalamin) Metallo-proteins	

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

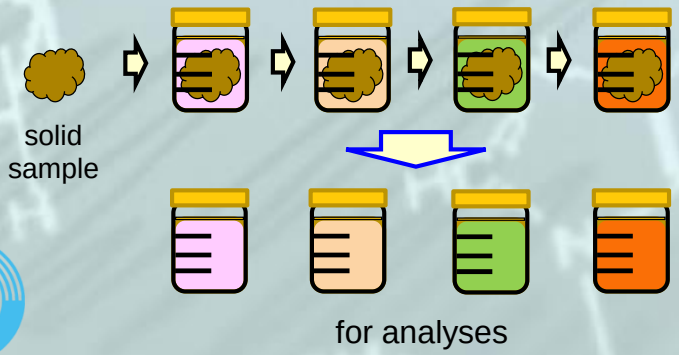
Speciation Analysis: Problems to be Solved

- *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.



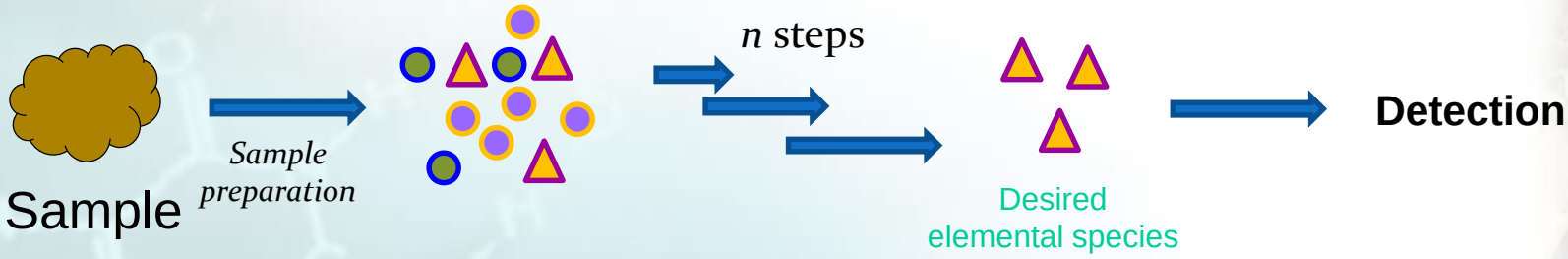
Speciation Analysis
Preparation / Pre-treatment: Solid Samples

- 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

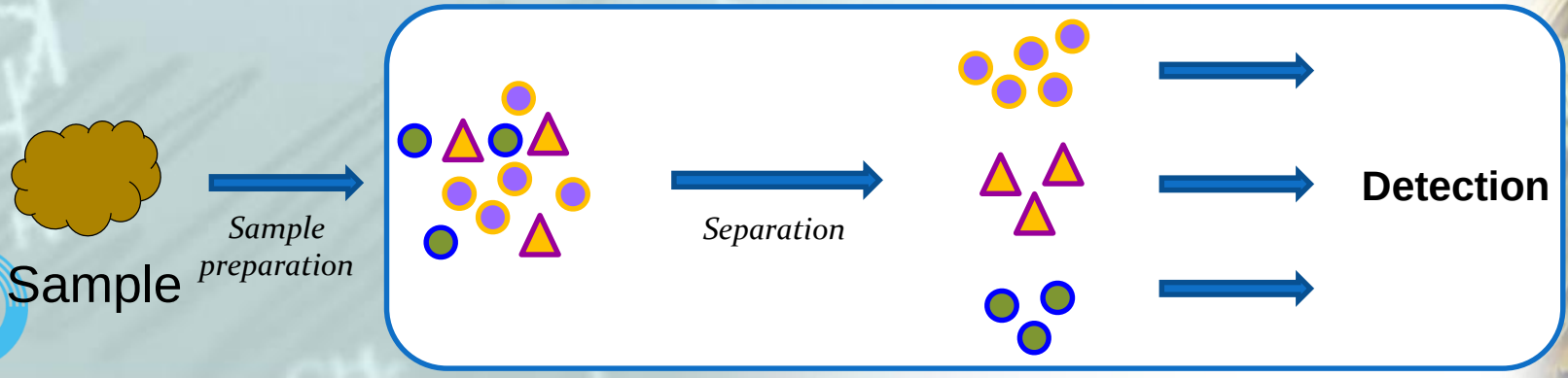


Tools for elemental speciation analysis

- Early day: a special sample preparation done OFFLINE followed by the detection step.



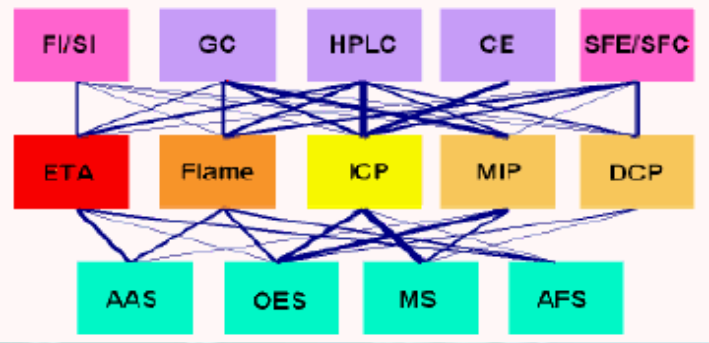
- Today: hyphenation connects the separation technique ONLINE with the detection technique.



ONLINE, one step !!!

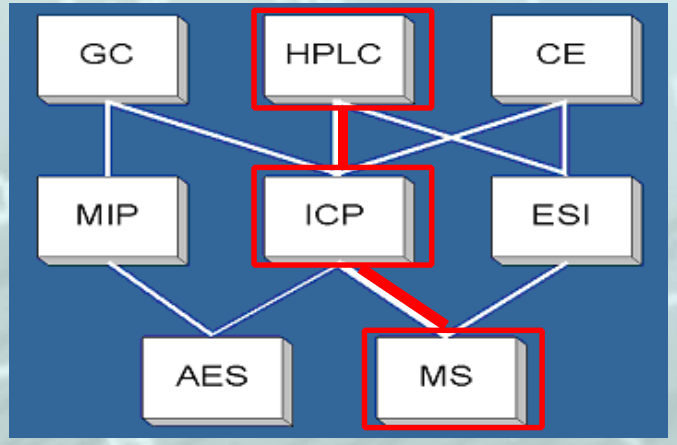
Tools for elemental speciation analysis

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	BS EN 16801-2016	FDA EAM 4.8	GL Method
scope	Food	aquatic product	aquatic product	Fish
Extraction solution	5M HCl	25% TMAH solution	L-cysteine solution	Mercaptoethanol solution
Extraction condition	Ultrasonic bath 60 min neutralize with NaOH, add L-cysteine solution	Ambient, overnight	60C 120 min	60C 15 min
Cleanup	-	Extract by hexane	-	
Determination technique	LC-AFS	GC-ICPMS (Et4B derivatisation in hexane)	LC-ICPMS	LC-ICPMS
Calibration	External Calibration (MeHg)	IDMS	External Calibration (MeHg)	External Calibration (MeHg)

Interlaboratory comparison for Methylmecury

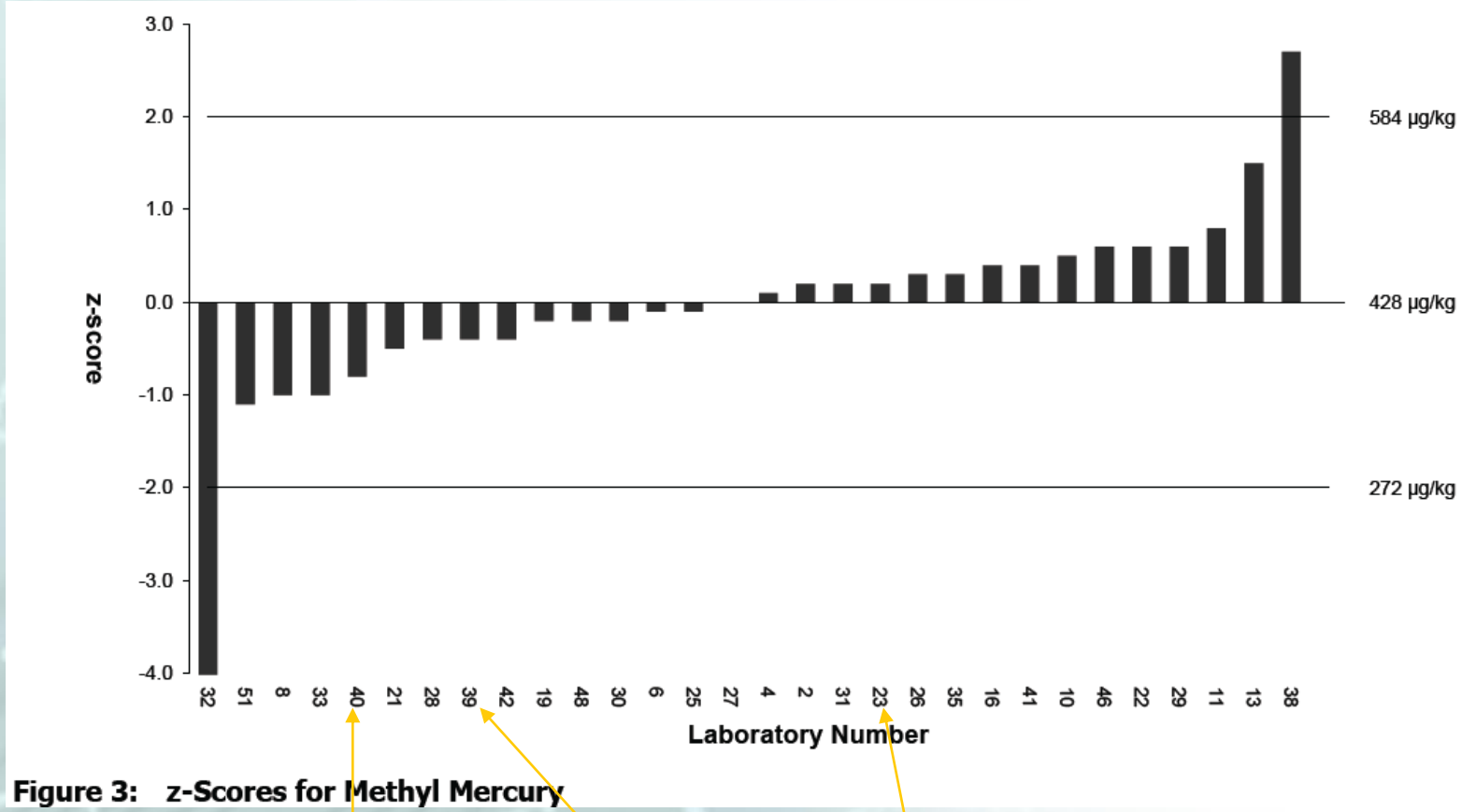


Figure 3: z-Scores for Methyl Mercury

Acid extract LC-AFS

L-cysteine LC-ICPMS

Alkaline extract GC-ICPMS

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	GB 5009.11-2014	BS EN 16802-2016	FDA EAM 4.11	GL Method
	Method 1	**Method 2**			
Scope	rice, aquatic product, infant forumla	rice, aquatic product, infant forumla	foodstuff from marine and plant origin	rice and rice product	rice, aquatic product
Extraction solution	0.15M HNO_3	0.15M HNO_3	0.1M HNO_3 + 3% H_2O_2	0.28M HNO_3	0.15M / 0.28M HNO_3 + 3% H_2O_2
Extraction condition	90C, 150min	90C, 150min	90C, 60min	95C, 90min	90C, 180min (rice); 100C, 180min (aquatic product)
Cleanup	hexane wash and C18 SPE (except for rice)	hexane wash and C18 SPE (except for rice)	Nil	Nil	C18 powder (except for rice)
Determination technique	IC AFS	IC ICPMS	IC ICPMS	IC ICPMS	IC ICPMS
Calibration	As III and As V	As III and As V	As V	As III and As V	As V

Interlaboratory comparison for Inorg. As

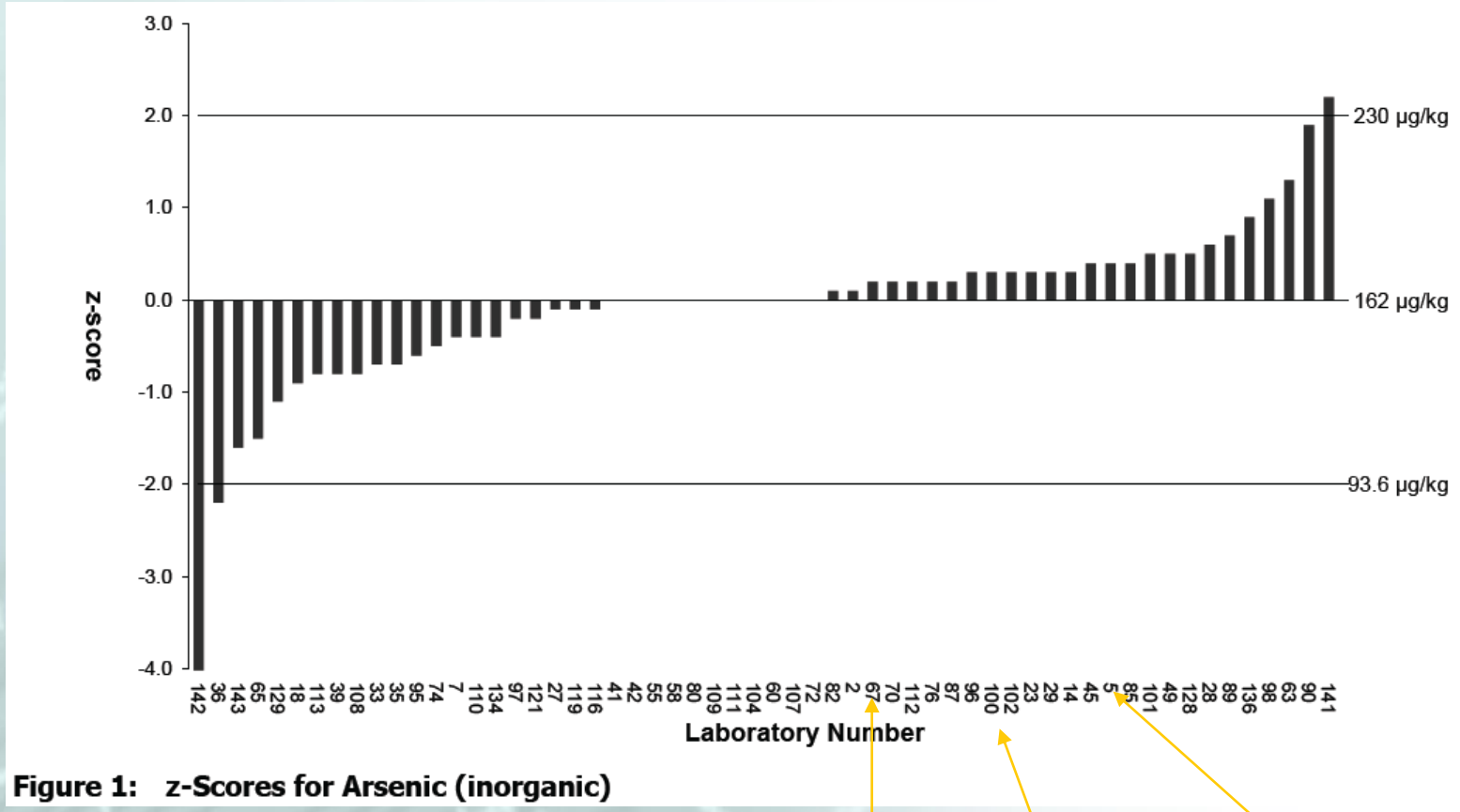


Figure 1: z-Scores for Arsenic (inorganic)

HNO3
LC-AFS

HNO3
LC-ICPMS

HNO3 +
H2O2
LC-ICPMS

Interlaboratory comparison for Inorg. As

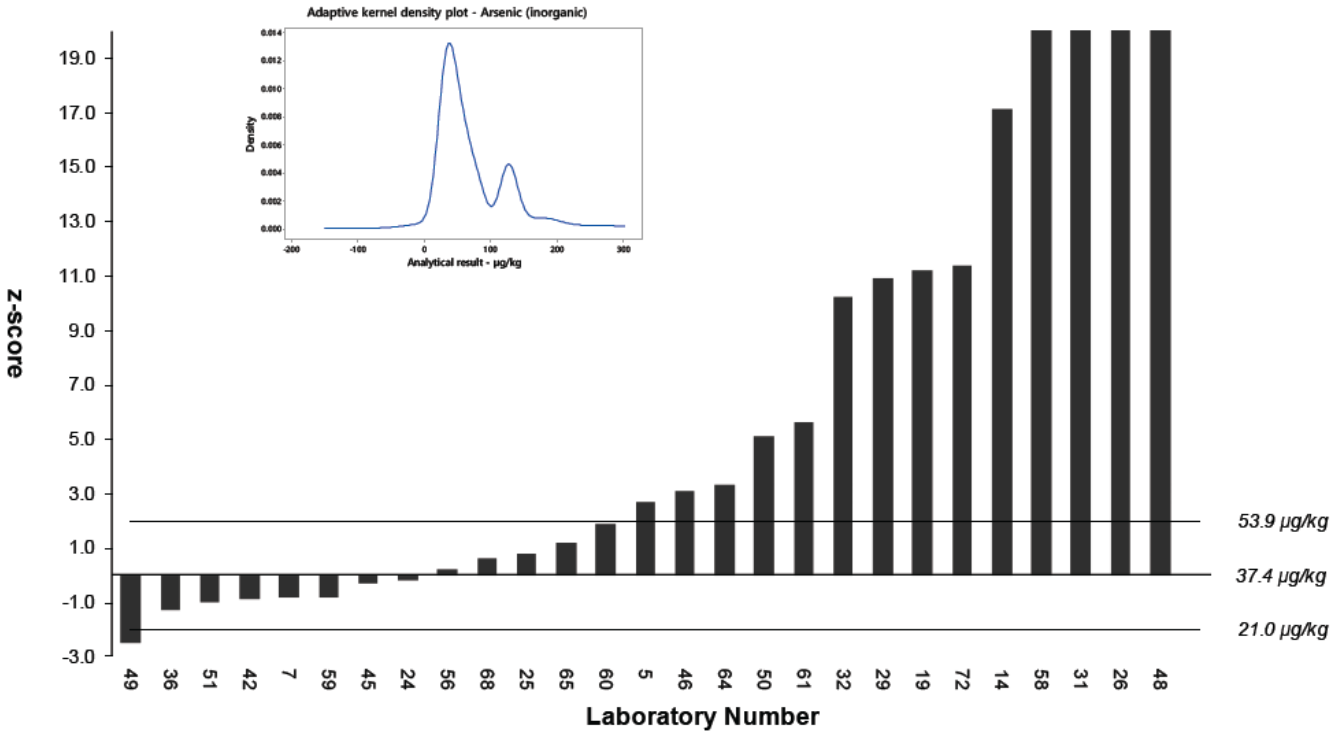


Figure 2: z-Scores for Arsenic (inorganic)

this histogram is given for information only

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.

Hong Kong Accreditation Service
香港認可處

Certificate of Accreditation
認可證書

This is to certify that
特此證明

GOVERNMENT LABORATORY - Analytical and Advisory Services Division

政府化驗所 - 分析及諮詢事務部

7/F., Homantin Government Offices, 88 Chung Hau Street, Kowloon, Hong Kong
香港九龍何文田忠孝街八十八號何文田政府合署七樓

has been accepted by the HKAS Executive, on the recommendation of the Accreditation Advisory Board, as a
在認可諮詢委員會的建議下獲香港認可處執行機關接受為

HOKLAS Accredited Proficiency Testing Provider
「香港實驗所認可計劃」認可能力驗證提供者

This Proficiency Testing Provider meets the requirements of ISO/IEC 17043:2010
and it has been accredited for providing specific proficiency testing schemes as listed in the scope of accreditation
此能力驗證提供者符合ISO/IEC 17043:2010所訂的要求
並獲認可提供載於認可範圍的指定能力驗證計劃

The common seal of the Hong Kong Accreditation Service is affixed hereto by the authority of the HKAS Executive
現經香港認可處執行機關授權在此書上香港認可處的印章

WONG Wang-wah, Executive Administrator
執行幹事 黃宏華
Issue Date : 26 February 2015
簽發日期：二零一五年二月二十六日

Registration Number : HOKLAS 001
註冊號碼：

Date of First Registration : 14 March 1986
首次註冊日期：一九八六年三月十四日

This certificate is issued subject to the terms and conditions laid down by HKAS
本證書按照香港認可處訂立的條款及條件發出

L001334

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

Feasibility Study
- Sourcing bulk material and standards
- Analytical methods development

Material Preparation
- Drying, homogenization, particle size reduction, sieving, irradiation

Homogeneity Study

Stability Study
- Short-term (transportation)
- Long-term (period of validity)

Determine assigned value

Measurement by participants

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

Result evaluation
- Assigned value
- z scores
- Robust analysis

Preparation and issue of reports

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

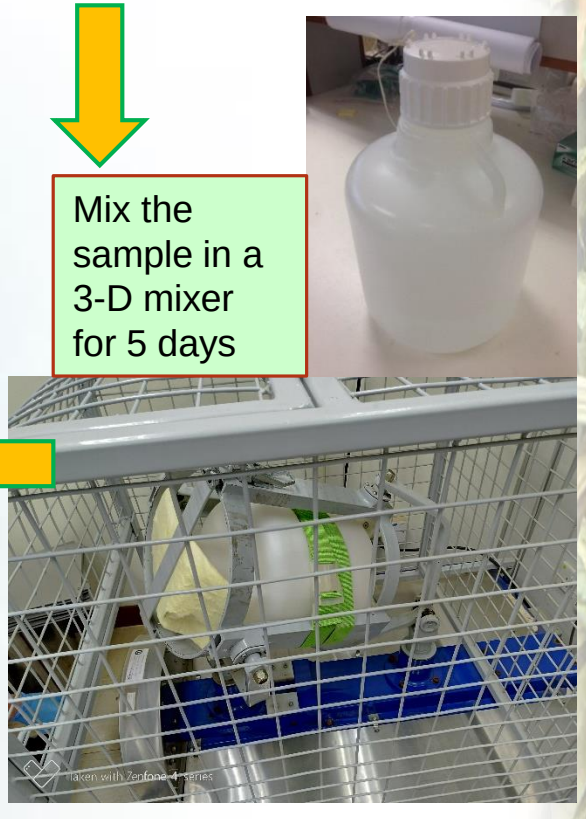
Workflow of PT Programmes

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

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

z-Score		Number of participants (Percentage)
$\|z\| \leq 2.0$		7 (87.5%)
$2.0 < \|z\| < 3.0$		(0%)
$\|z\| \geq 3.0$		1 (12.5%)
Total :		8 (100%)

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