Testing of trace elements in Food

食品中微量元素的檢測

23.3.2018

Lead in CAP 132V

Lead (Pb)

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)	Portion of the commodity/product to which the proposed ML applies (The proposed ML applies to the edible portion if there is no specification)
Fruiting vegetables, Cucurbits (Note 1)	6	0.05	Note 7
Fruiting vegetables, other than Cucurbits (Note 1)	6	0.05	Note 7
Preserved tomatoes †	6	0.05	
Processed tomato concentrates†	6	0.05	
Canned chestnuts and canned chestnuts puree†	6	0.05	
Milk (Note 1)	1	0.02	
Secondary milk products (Note 1)	6 [solid food] /1[liquid]	0.02	Applies to the food as consumed.
Infant formula, formula for special medical purposes intended for infants and follow-up formula (Note 1)	6	0.01	Applies to formula as consumed.

Lead in CAP 132V

Lead (Pb)

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)	Portion of the commodity/product to which the proposed ML applies (The proposed ML applies to the edible portion if there is no specification)
Fruit juices (Note 1)	1	0.03	Not apply to juices exclusively from berries and other small fruits. Applies to whole commodity (not concentrated) or commodity reconstituted to the original juice concentration, ready to drink. Applies also to nectars, ready to drink.
Fruit juices exclusively from berries and other small fruits (Note 1)	1	0.05	Applies to whole commodity (not concentrated) or commodity reconstituted to the original juice concentration, ready to drink. Applies also to nectars, ready to drink.

Mercury in CAP 132V

Mercury (Hg)

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)	Portion of the commodity/product to which the proposed ML applies (The proposed ML applies to the edible portion if there is no specification)
		Expressed in total mercury	
Vegetables	0.5 (total mercury)	0.01	Not apply to edible fungi. Notes 2-9
Rice, husked rice, polished rice, maize, maize flour, wheat, wheat flour	0.5 (total mercury)	0.02	
Milk	0.5 (total mercury)	0.01	
Secondary milk products	0.5 (total mercury)	0.01	Applies to the food as consumed.

Arsenic in CAP 132V (Existing)

Schedule 1: Maximum permitted concentration of certain metals naturally present in specified foods

Metal	Description of food	Maximum permitted concentration in parts per million
Arsenic (AS_2O_3)	Solids being fish and fish products	6
	Solids being shellfish and shellfish products	10

Schedule 2: Maximum permitted concentration of certain metals present in specified foods

Arsenic (AS_2O_3)	Solids other than-	
	(i) fish and fish products; and	
	(ii) shellfish and shellfish products	1.4
	All food in liquid form	0.14

Arsenic in Proposed Amendments

Arsenic (As)

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)	Portion of the commodity/product to which the proposed ML applies (The proposed ML applies to the edible portion if there is no specification)
			Expressed as total arsenic	
Vegetables	1.4	1.1	0.5	Notes 1-8
Cereals	1.4	1.1	0.5	Not apply to rice.
Meat of animal	1.4	1.1	0.5	Applies to whole commodity (without bones). Also applies to fat from the meat.
Meat of poultry	1.4	1.1	0.5	Applies to whole commodity (without bones). Also applies to fat from the meat.
Animal, edible offal of	1.4	1.1	0.5	
Poultry, edible offal of	1.4	1.1	0.5	
Edible fats and oils (Note 9)	1.4 [solid food]/ 0.14 [liquid food]	1.1 [solid food]/ 0.1 [liquid food]	0.1	Not apply to fish oil.*
Fat spreads and blended spreads (Note 9)	1.4	1.1	0.1	
Salt, food grade (Note 9)	1.4	1.1	0.5	
Natural mineral waters (Note 9)	0.14	0.1	0.01 (mg/L)	
Bottled/packaged drinking waters (other than natural mineral waters) (Note 9)	0.14	0.1	0.01 (mg/L)	

Arsenic in Proposed Amendments

Arsenic (As)

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)	Portion of the commodity/product to which the proposed ML applies (The proposed ML applies to the edible portion if there is no specification)
			Expressed as inorganic arsenic	
Rice, husked (Note 9)	1.4	1.1	0.35	
Rice, polished (Note 9)	1.4	1.1	0.2	
Aquatic animals	10	7.9	0.5	Not apply to fish and intestine of sea cucumber. Applies to edible portion of the crab, including the liver and gonads or parts thereof after removal of shell. Cephalopods: Applies to whole commodity after removal of shell and viscera. Scallops: Applies to whole commodity after removal of shell and viscera.
Fish	6	4.8	0.1	Whole commodity after removing the digestive tract.
Fish oil*	0.14	0.1	0.1	
Seaweed	1.4	1.1	1	

Methods for Inorganic Arsenic

Available International / National Standards:

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

CHINA (GB): GB 5009. 11-2014

中华人民共和国国家标准

GB 5009.11—2014

食品安全国家标准

食品中总砷及无机砷的测定

第二篇　食品中无机砷的测定

第一法　液相色谱-原子荧光光谱法(LC-AFS)法

第二法　液相色谱-电感耦合等离子质谱法(LC-ICP/MS)

EU: EN 16802-2016

BS EN 16802:2016

BSI Standards Publication

Foodstuffs — Determination of elements and their chemical species — Determination of inorganic arsenic in foodstuffs of marine and plant origin by anion-exchange HPLC-ICP-MS

USA: FDA EAM 4.11

U.S. Department of Health & Human Services

FDA U.S. Food and Drug Administration

Elemental Analysis Manual

for Food and Related Products

4.11 Arsenic Speciation in Rice and Rice Products Using High Performance Liquid Chromatography-Inductively Coupled Plasma-Mass Spectrometric Determination

Version Draft 1.1 (November 2012)

Methods for Inorganic Arsenic

	GB 5009.11-2014	GB 5009.11-2014	BS EN 16802-2016	FDA EAM 4.11
	Method 1	**Method 2**		
scope	rice, aquatic product, infant formula	rice, aquatic product, infant formula	foodstuff from marine and plant origin	rice and rice product
Extraction solution	0.15M HNO3	0.15M HNO3	0.1M HNO3 + 3% H2O2	0.28M HNO3
Extraction condition	90C, 150min	90C, 150min	90C, 60min	95C, 90min
Cleanup	hexane wash and C18 SPE (except for rice)	hexane wash and C18 SPE (except for rice)	Nil	Nil
Determination technique	IC AFS	IC ICPMS	IC ICPMS	IC ICPMS
Calibration	As III and As V	As III and As V	As V	As III and As V

Interlaboratory comparison for Inorg. As

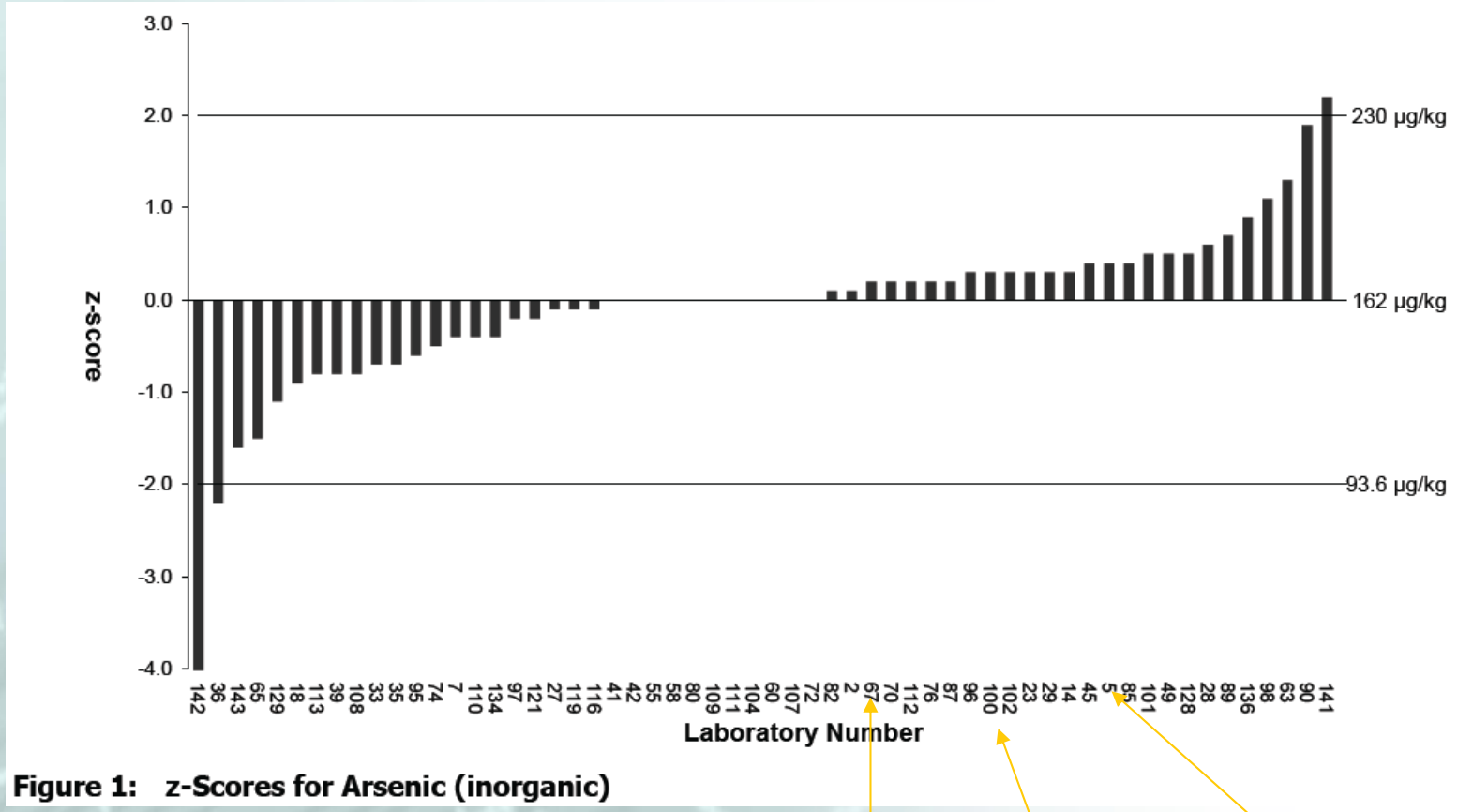

Figure 1: z-Scores for Arsenic (inorganic)

HNO3
LC-AFS

HNO3
LC-ICPMS

HNO3 +
H2O2
LC-ICPMS

Interlaboratory comparison for Inorg. As

Arsenic (inorganic)

Sample Weight (g)	laboratory number
<1	002 005 027 028 039 058 063 067 074 076 080 085 089 090 096 097 100 107 109 119 121
≥1 - <2	014 035 041 042 049 070 095 098 104 111 113 116 136
≥2 - <5	082 108 129
≥5 - <10	110

Determination	laboratory number
cold vapour / hydride generation AAS	076 136
cold vapour / hydride generation AFS	014 104 108
FIAS	089
HPLC	109 111
hydride generation AAS	035 129
IC-HPLC	041 063 097 098
ICP-MS	027 039 041 049 058 070 080 082 085 098 109 110 111 113
HPLC - ICP-MS	100
HPLC/ICP-MS	074 096 107
HPLC-AFS	067
HPLC-ICP/MS	028
HPLC-ICPMS	002 042
HPLC-ICP-MS	119 121
IC-ICP-MS	005
lc-afs	095

Sample Preparation	laboratory number
dry ashing	076 089 110
reduction As(V) - As(III)	076 089 090 111 136
solvent extraction	005 014 027 028 039 041 049 067 076 097 098 104 109 113 116 119
wet digestion	042 058 063 070 082 095 107 108 121 129
acid extraction	096
extraction with diluted nitric acid	035
microwave assisted extraction	074
Solubilised in HCl, extracted in chloroform, then back-extracted into HCl.	080
ultrasonic assisted enzymatic extraction (UAEE).	002
Water Bath	085

Sample Preparation Reagents Used	laboratory number
chloroform	049 076 080
hydrochloric acid	074 076 080 095 108 111 129 136
hydrogen peroxide	074 085 090 111 129
nitric acid	014 027 028 035 039 041 042 058 063 067 070 076 082 085 089 090 096 097 098 100 104 107 109 110 113 116 119 121 129
sulphuric acid	110
H2O2 + HNO3 in H2O	005
water and IPA	002

Mercury in CAP 132V

Schedule 2: Maximum permitted concentration of certain metals present in specified foods

Mercury (Hg)	All food in solid form	0.5
	All food in liquid form	0.5

Methylmercury in Proposed Amendments

Food items	Existing maximum permitted concentration (ppm)	Proposed maximum level (ML) (mg/kg, unless otherwise specified)	Portion of the commodity/product to which the proposed ML applies (The proposed ML applies to the edible portion if there is no specification)
		Expressed in methylmercury	
Fish (Note 1)	0.5 (total mercury)	0.5	Whole commodity after removing the digestive tract.
		Expressed in total mercury	
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	

Methods for Methylmercury

Available International / National Standards:

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

CHINA (GB): GB 5009. 17-2014

中华人民共和国国家标准

GB 5009.17—2014

食品安全国家标准

食品中总汞及有机汞的测定

第二篇　食品中甲基汞的测定

液相色谱-原子荧光光谱联用方法

EU: EN 16801-2016

BS EN 16801:2016

BSI Standards Publication

Foodstuffs — Determination of elements and their chemical species — Determination of methylmercury in foodstuffs of marine origin by isotope dilution GC-ICP-MS

USA: FDA EAM 4.8

U.S. Department of Health & Human Services

FDA U.S. Food and Drug Administration

Elemental Analysis Manual

for Food and Related Products

4.8 High Performance Liquid Chromatographic-Inductively Coupled Plasma-Mass Spectrometric Determination of Methylmercury and Total Mercury in Seafood

Version 1 (June 2008)

Methods for Methylmercury

	GB 5009.17-2014	BS EN 16801-2016	FDA EAM 4.8	
scope	Food	aquatic product	aquatic product	
Extraction solution	5M HCl	25% TMAH solution	L-cysteine solution	
Extraction condition	Ultrasonic bath 60 min neutralize with NaOH, add L-cysteine solution	Ambient, overnight	60C 120 min	
Cleanup	-	Extract by hexane	-	
Determination technique	LC-AFS	GC-ICPMS (Et4B derivatisation in hexane)	LC-ICPMS	
Calibration	External Calibration (MeHg)	IDMS	External Calibration (MeHg)	

Interlaboratory comparison for Methylmecury

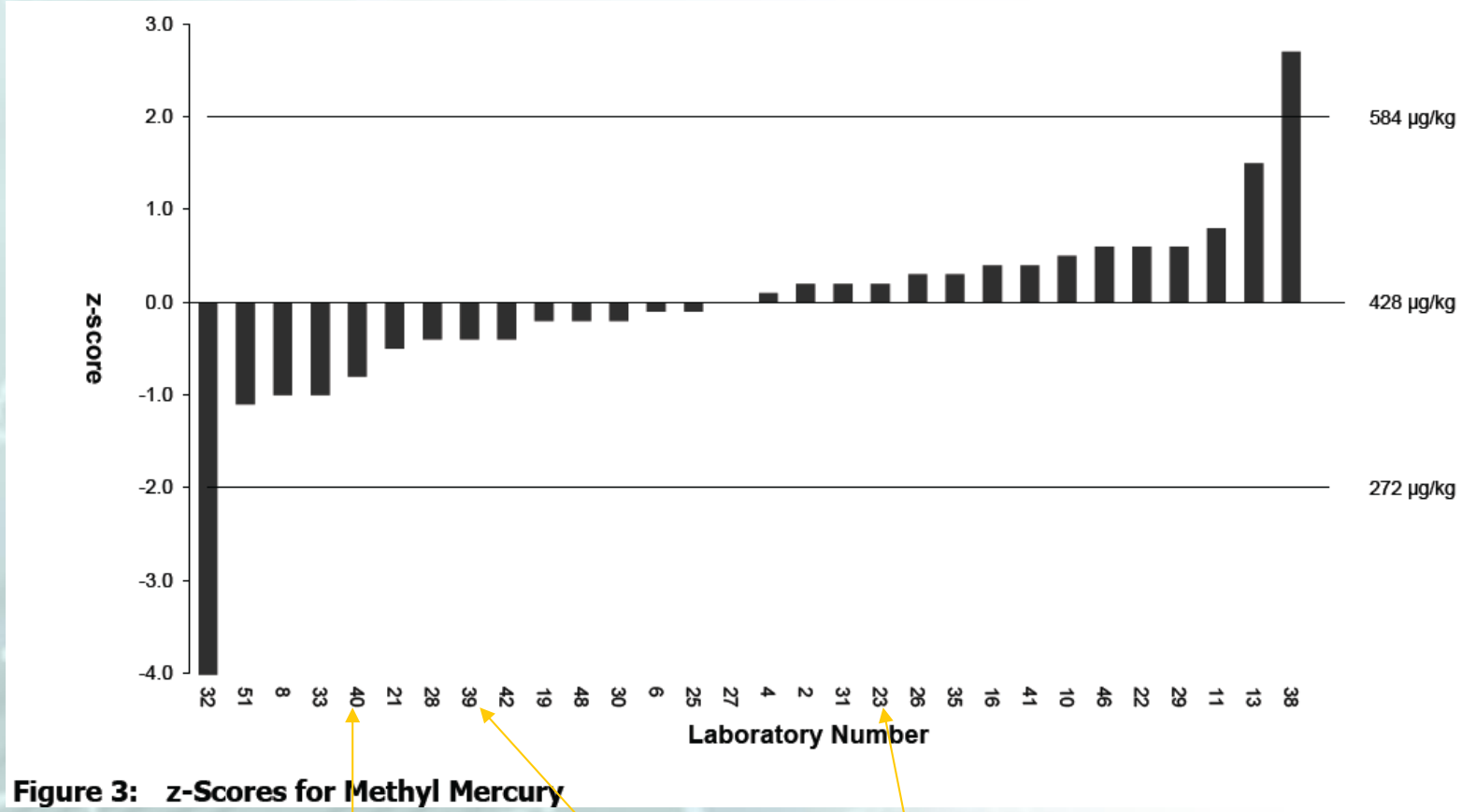

Figure 3: z-Scores for Methyl Mercury

Acid extract LC-AFS

L-cysteine LC-ICPMS

Alkaline extract GC-ICPMS

Interlaboratory comparison for Methylmecury

Methyl Mercury

Sample Weight (g)	laboratory number
<1	004 006 008 013 016 01[illegible] 031 033 035 039 040 04[illegible]
≥1 - <2	002

Sample Preparation	laboratory number
microwave digestion	028
solvent extraction	004 008 013 016 023 029 030 031 033 035 040 041 042 046 051
wet digestion	019 039
distillation, oxidation	002
extraction with HCl	021

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	002 021 040 051
2-mercaptoethanol in 5% methanol	028
acetic acid buffer	031
extraction into toluene and complexation with cysteine	033
H2O	035
HBr, Toluene, L-Cisteine	004
HBr; TOLUENE; L-CISTEINE; SODIUM ACETATE; SODIUM SULFATE	016
hydrobromic acid, cysteine, toluene	051
KOH-Methanol	019
L-cysteine	039 041
L-cysteine hydrochloride monohydrate	046
L-cysteine-HCL	042
TMAH	023
Toluene, L-Cysteine monohydrate hydrochloride, Hydrobromic acid, Sodium Sulphate anhydrous, Sodium acetate anhydrous	029

Determination	laboratory number
automated mercury analyser	004 029 051
HPLC	021 046
ICP-MS	002 021 035 041 046
AMA 254	033
gas chromatography -pyrolysis- cold vapour atomic fluorescence	019
GC/MS	008
GC-ICPMS	006
GC-MS	031
HPLC-ICP-MS	028 039 042
ICP-MS/HPLC	013
ID-GC-ICP-MS	023
LC-AFS	040
LC-ICP-MS	030
TDA-AAS	016

Thank you