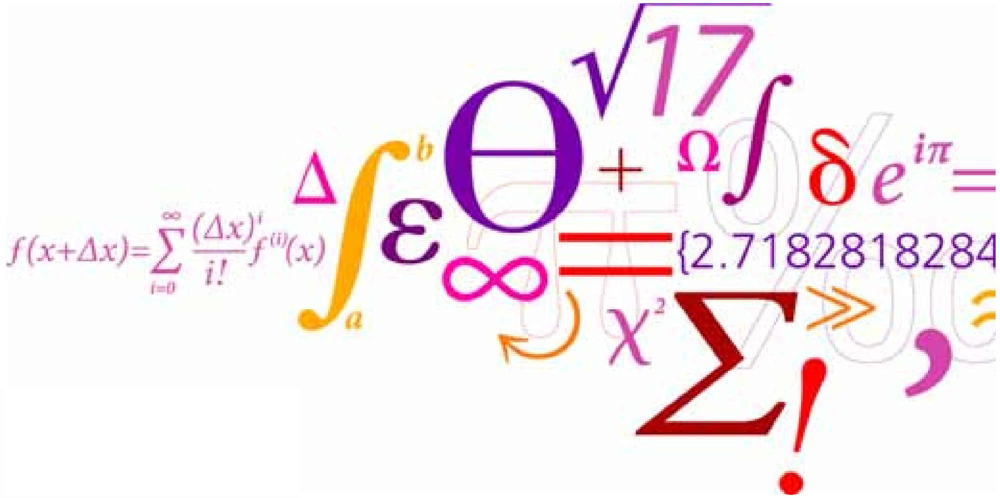
Novel developments and future trends in methods of analysis for trace element species in food and feed control

Jens J. Sloth

National Food Institute (DTU Food)

Technical University of Denmark



DTU Food

National Food Institute

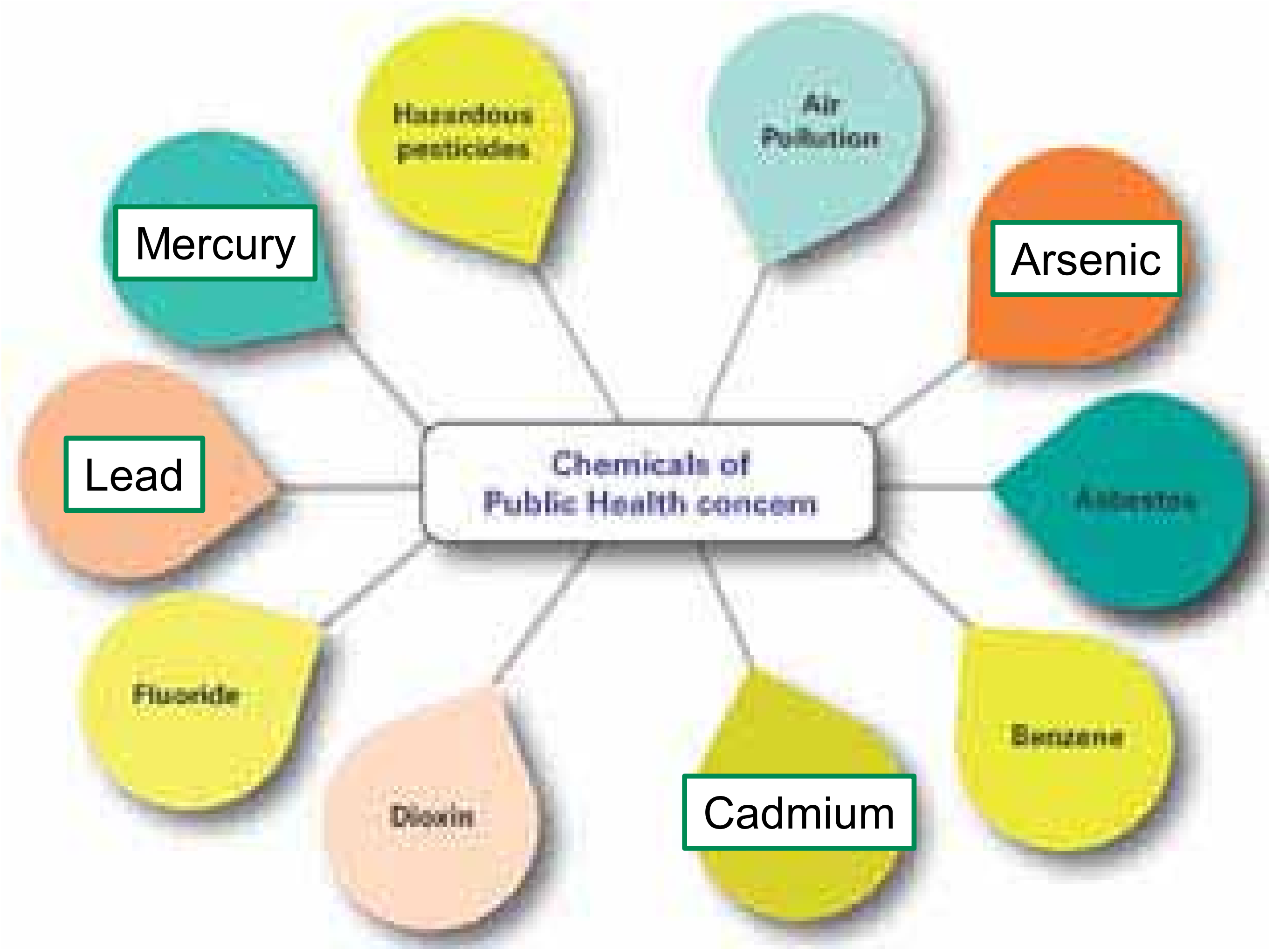
Agenda

- Speciation in relation to food and feed safety – where are we now??
- Current legislation and standardisation issues
- Selected examples
  - Arsenic speciation analysis (importance of inorganic arsenic)
  - Organotin speciation analysis (food contact materials)
  - Selenium speciation analysis (food fraud!)
- Future developments and needs

The International Programme on Chemical Safety (IPCS)

WHO ILO UNEP

DTU



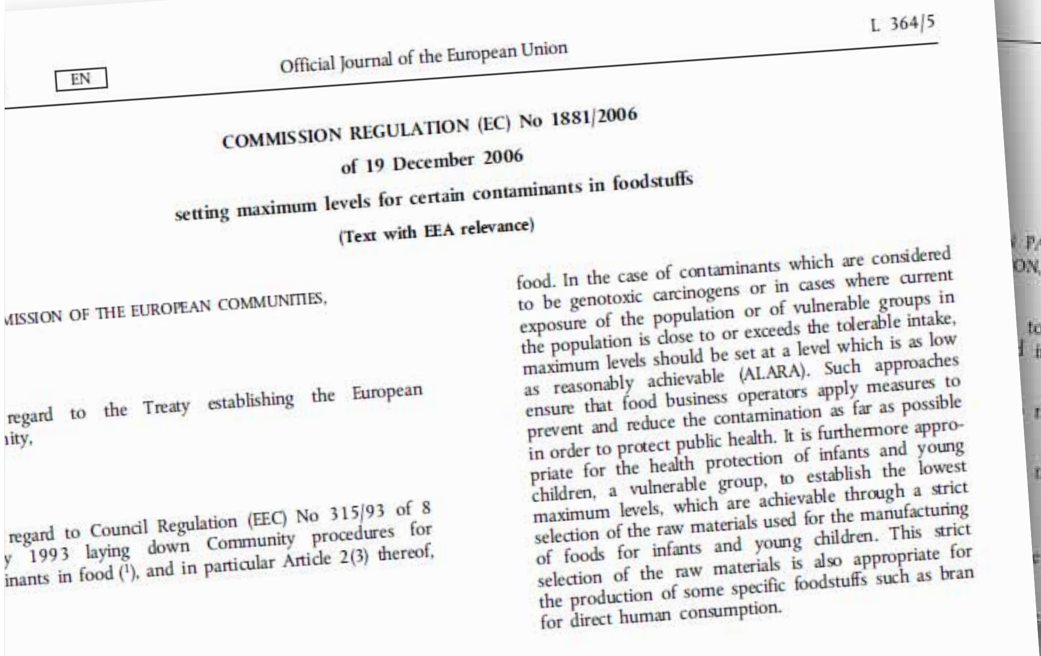
http://www.who.int/ipcs/

Current situation in EU legislation:

Foodstuffs
MLs for Pb, Cd, Hg and Sn
EU directive 2006/1881/EC

Animal feedingstuffs
MLs for As, Pb, Cd and Hg
EU directive 2002/32/EC

Only maximum levels for <u>total concentration</u> of the metals



EN Official Journal of the European Union L 364/5

COMMISSION REGULATION (EC) No 1881/2006

of 19 December 2006

setting maximum levels for certain contaminants in foodstuffs

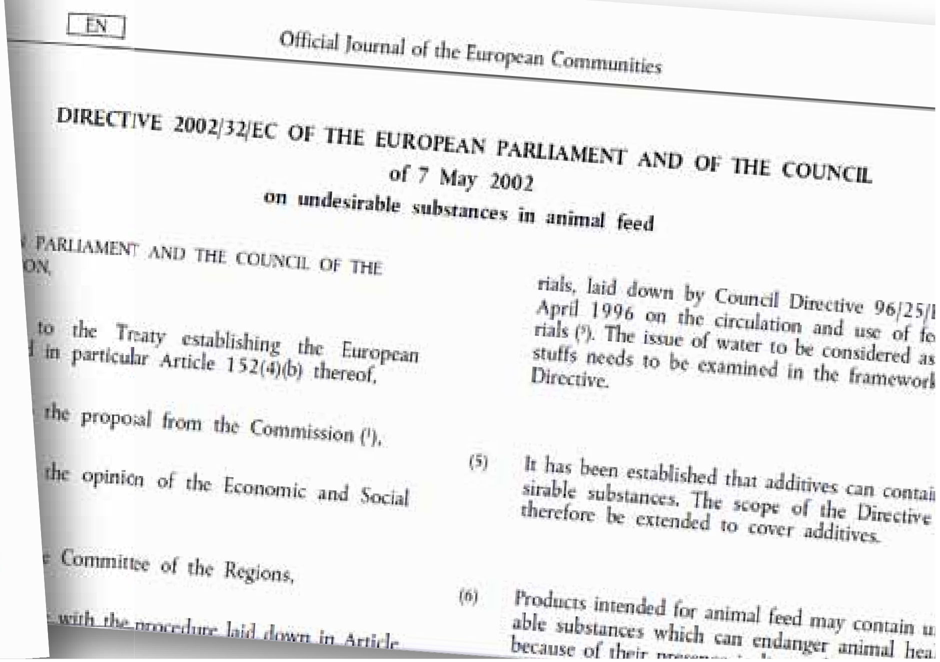
(Text with EEA relevance)

...MISSION OF THE EUROPEAN COMMUNITIES,

...regard to the Treaty establishing the European ...ity,

...regard to Council Regulation (EEC) No 315/93 of 8 ...y 1993 laying down Community procedures for ...inants in food (1), and in particular Article 2(3) thereof,

food. In the case of contaminants which are considered to be genotoxic carcinogens or in cases where current exposure of the population or of vulnerable groups in the population is close to or exceeds the tolerable intake, maximum levels should be set at a level which is as low as reasonably achievable (ALARA). Such approaches ensure that food business operators apply measures to prevent and reduce the contamination as far as possible in order to protect public health. It is furthermore appropriate for the health protection of infants and young children, a vulnerable group, to establish the lowest maximum levels, which are achievable through a strict selection of the raw materials used for the manufacturing of foods for infants and young children. This strict selection of the raw materials is also appropriate for the production of some specific foodstuffs such as bran for direct human consumption.



EN Official Journal of the European Communities

DIRECTIVE 2002/32/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL

of 7 May 2002

on undesirable substances in animal feed

...PARLIAMENT AND THE COUNCIL OF THE ...ON,

...to the Treaty establishing the European ...in particular Article 152(4)(b) thereof,

...the proposal from the Commission (1),

...the opinion of the Economic and Social

...Committee of the Regions,

...with the procedure laid down in Article

rials, laid down by Council Directive 96/25/... April 1996 on the circulation and use of fe... rials (5). The issue of water to be considered as ... stuffs needs to be examined in the framework ... Directive.

(5) It has been established that additives can contai... sirable substances. The scope of the Directive ... therefore be extended to cover additives.

(6) Products intended for animal feed may contain u... able substances which can endanger animal hea... because of their ...

Speciation and regulation - some historical viewpoints

1998

ELSEVIER

Spectrochimica Acta Part B 53 (1998) 169–175

SPECTROCHIMICA ACTA PART B

Speciation analysis: where is it going? An attempt at a forecast[1]

Bernhard Welz

Department of Applied Research, Bodenseewerk Perkin-Elmer GmbH, D-

Fresenius J Anal Chem (1999) 363 : 431–434 © Springer-Verlag 1999

CONFERENCE CONTRIBUTION

1999

Torsten Berg · Erik H. Larsen

Speciation and legislation – Where are we today and what do we need for tomorrow?

Handbook of Elemental Speciation II

Species in the Environment, Food, Medicine and Occupational Health

Editor-in-Chief Rita Cornelis

Associate Editors Joe Caruso, Helen Crews, Klaus Heumann

Speciation and the Emerging Legislation

Nicole Proust

THALES Research and Technology France, Orsay, France

Wolfgang Buscher

University of Münster, Münster, Germany

Michael Sperling

University of Münster, Münster, Germany

Wiley 2005

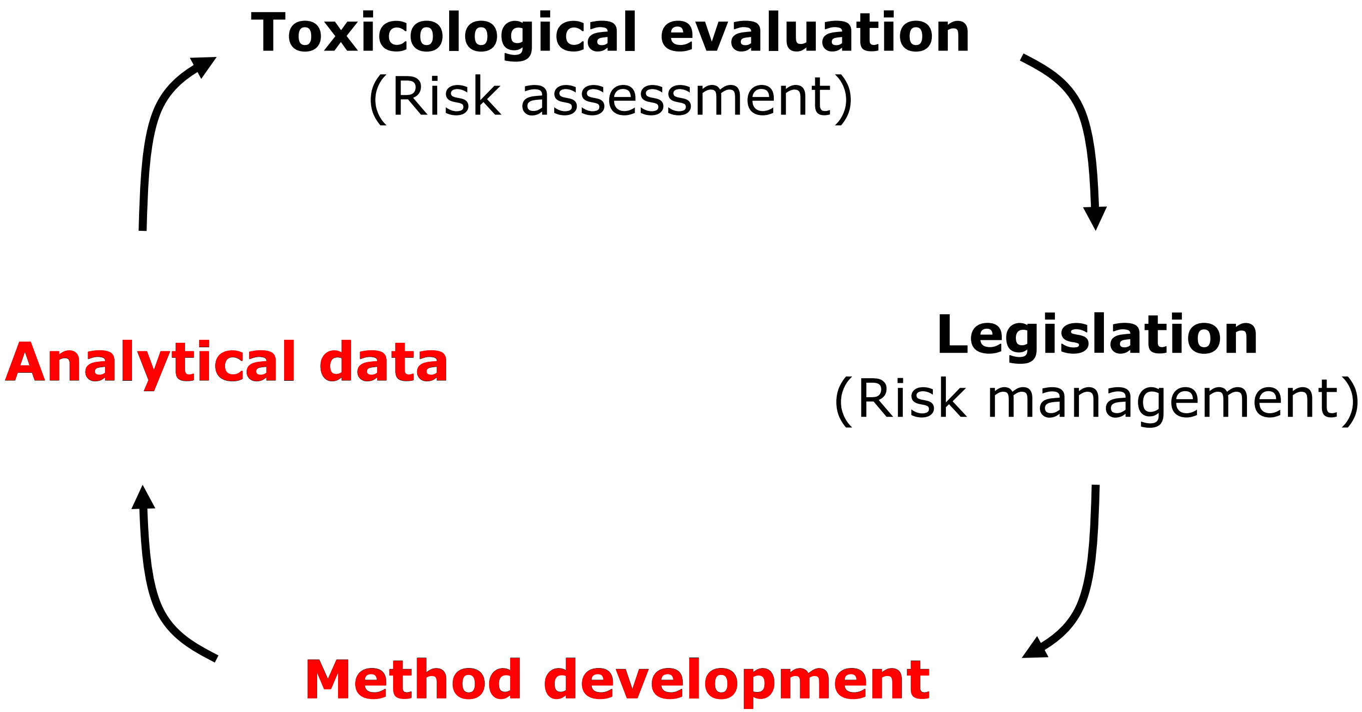
SECTION: FORUM www.rsc.org/analyst | The Analyst

Toxic metal species and food regulations—making a healthy choice

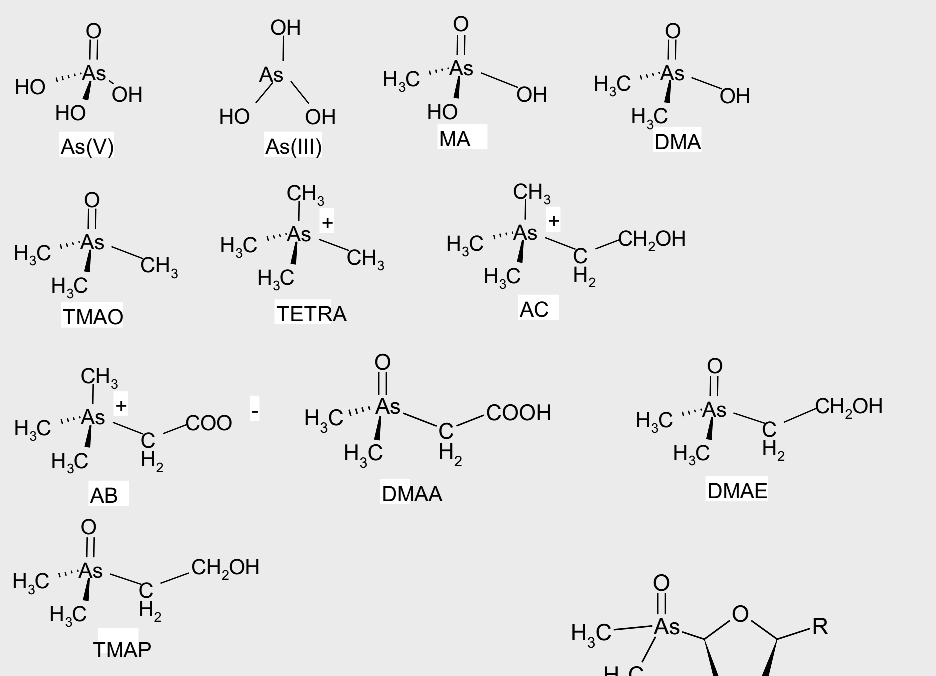
2007

Kevin A. Francesconi

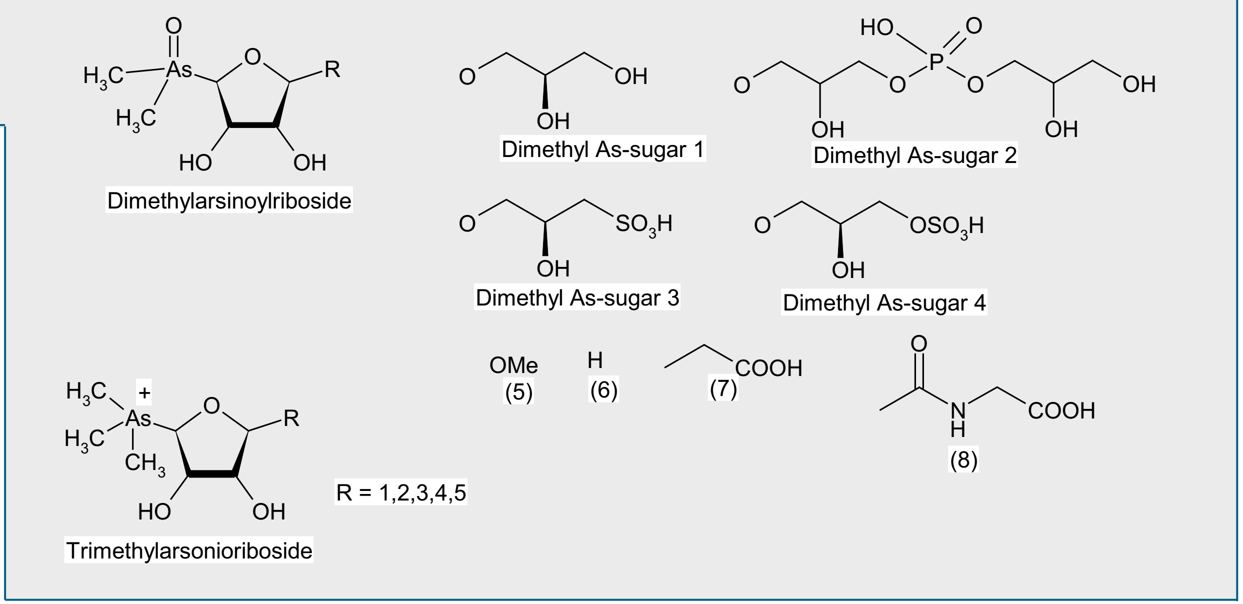
Vicious circle of progress



Arsenic compounds in the marine environment



More than **50** different arsenic species have been found in the marine environment – incl. lipid-soluble As compounds (*arsenolipids*).



Example – arsenic speciation
Important for correct risk assessment

1 kg rice => 50-300 µg As **1 kg fish => 3000-10000 µg As**

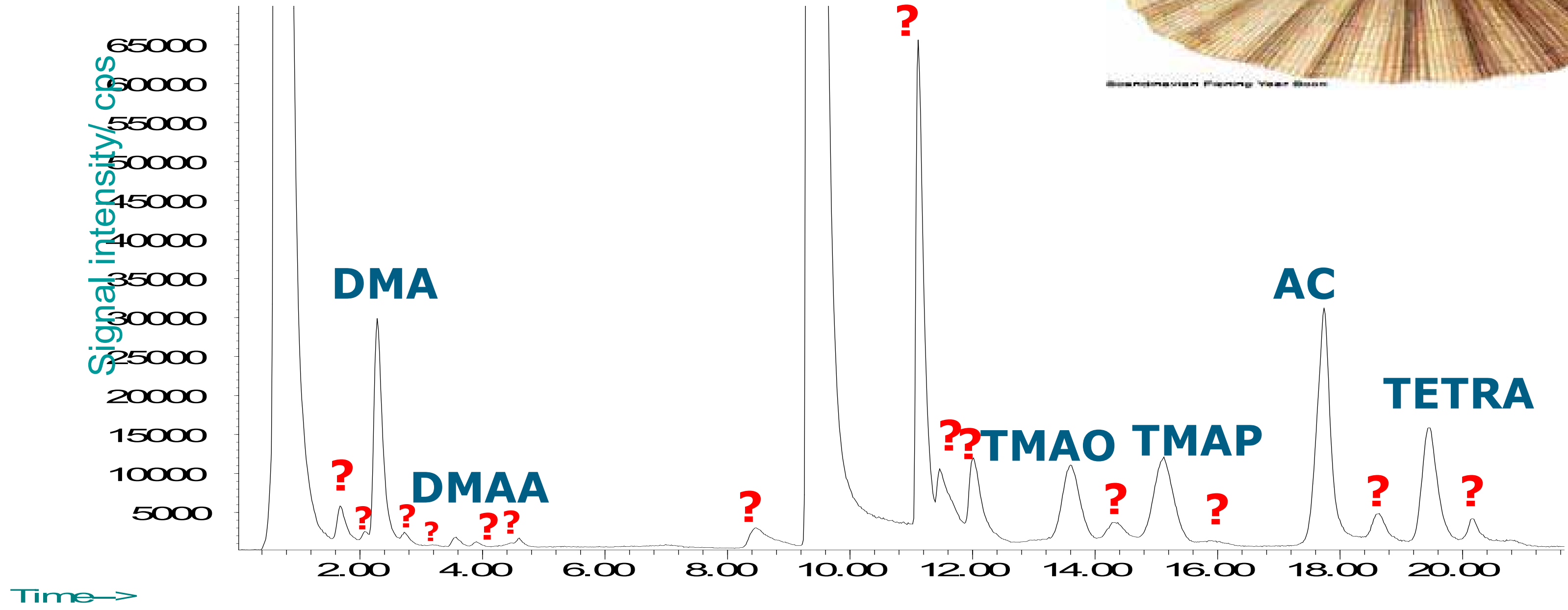
There is most focus on rice from a food safety point of view – why???

The chemical form of arsenic is important
=> Arsenic speciation

Speciation analysis of arsenic of scallop kidney

Cation-exchange with gradient elution – extraction with aqueous methanol
Column: Chrompack Ionospher 5C; Mobile phase: Pyridine; pH = 2.7

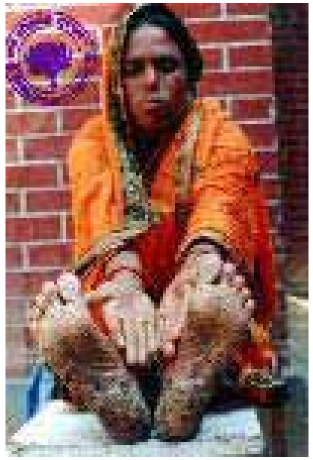
- seven compounds identified by coelution with available standards
- **16** non-identified peaks



Sloth et al, J.Anal.At.Spectrom., 2003, 18, 452-459

Food safety and arsenic – toxicity

Focus on inorganic arsenic; As(III) and As(V)



Long term exposure => skin diseases

- Keratosis, gangrene, melatosis
- Skin cancer

… and also

- lung, kidney, liver, bladder cancers

Cancer slope factor: 1.5 (mg kg^{-1} day^{-1})$^{-1}$ (for inorganic As) (US EPA 2005)

WHO (1988) PTWI for inorganic arsenic: 15 µg/kg bw/week
(Provisional Tolerable Weekly Intake)
For a 70 kg person => 150 µg / day

No longer appropriate

European Food Safety Authority

EFSA (2009) and JECFA (2010) opinions on arsenic in food

- Old PTWI value (WHO, 1988) was withdrawn

- **NEW!** $BMDL_{1.0}$ = 0.3 – 8 µg/kg bw per day for inorganic arsenic
- => EU dietary exposures within this range
- => Risk to some consumers cannot be excluded

- **NEW!** $BMDL_{0.5}$ = **3 µg/kg bw per day** for inorganic arsenic
  => 0.5% increased incidence of lung cancer for 12 y exposure

- *"…there is a need to produce speciation data for different food commodities to support dietary exposure assessment…"*

- *"…more accurate information on the inorganic arsenic content of foods is needed to improve assessments of dietary exposures to inorganic arsenic"*

- *"…need for validated methods for selective determination of inorganic arsenic in food matrices"*

Sloth et al, J.Agric.Food Chem., 2005, 53, 6011-6018

Sample identification	Inorganic arsenic	Total arsenic
Salmon (*Salmo salar*)	< 0.0006	1.9 0.2
Cod (*Gadus morhua*)	< 0.0006	17 2
Cod (*Gadus morhua*)	< 0.0006	15 2
Wolffish (*Anarhichas lupus*)	< 0.0006	4.1 0.5
Wolffish (*Anarhichas lupus*)	< 0.0006	31 4
Anglerfish (*Lophius piscatorius*)	< 0.0006	15 2
Anglerfish (*Lophius piscatorius*)	< 0.0006	44 6
Atlantic halibut (*Hippoglossus hippoglossus*)	< 0.0006	12 1
Mackerel (*Scomber scombrus*)	< 0.0006	1.7 0.2
Mackerel (*Scomber scombrus*)	< 0.0006	2.8 0.4
Herring (*Clupea harengus*)	< 0.0006	1.5 0.2
Herring (*Clupea harengus*)	< 0.0006	1.7 0.2
Herring (*Clupea harengus*)	< 0.0006	1.7 0.2
Tuna fish (Thunnus alalunga)	0.008 0.001	0.9 0.1
Lobster, tail meat (*Homarus gammarus*)	< 0.0006	14 2
Lobster, head and thorax meat (*Homarus gammarus*)	[illegible]	22 3
Crab, white meat (*Cancer pagurus*)	0.016 0.002	[illegible]
Crab, head and thorax meat (*Cancer pagurus*)	[illegible] 0.009	26 3
King crab, white meat (*Paralithodes camschaticus*)	[illegible]	26 3
Norway lobster (*Nephrops norvegicus*)	0.020 0.003	21 [illegible]
Shrimp (*Pandalus borealis*)	< 0.0006	3.8 0.5
Shrimp (*Pandalus borealis*)	< 0.0006	60 8
Shrimp (*Pandalus borealis*)	< 0.0006	67 8
Horse mussel (*Modilous modiolus*)	0.0012 0.002	3.4 0.4
Scallop muscle (*Pecten maximus*)	0.008 0.001	3.1 0.3
Oyster (*Ostrea edulis*)	0.014 0.002	1.8 0.2
Mink whale (*Balaenoptera Acutorostrata*)	< 0.0006	0.61 0.08
Harp seal (*Pagophilus groenlandicus*)		0.9 0.1
Hooded seal (*Cystophora cristata*)		0.22 0.03

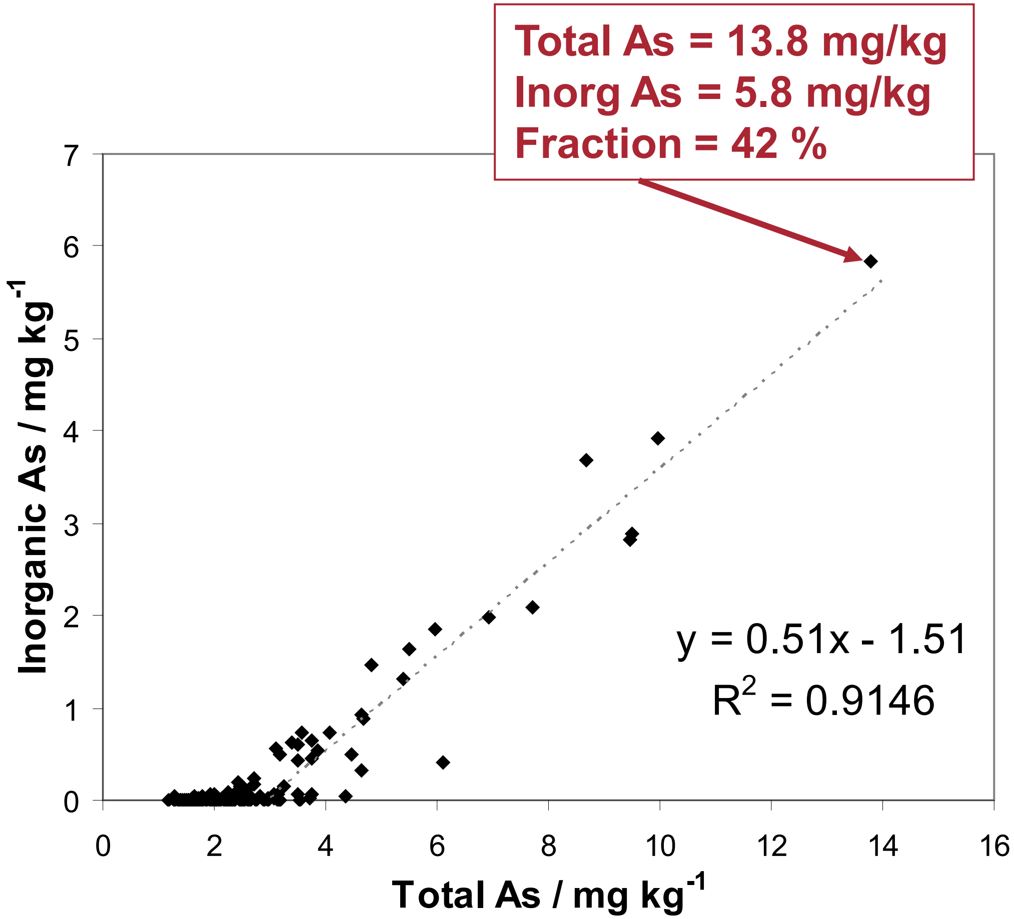
Fish muscle

Crustaceans & bivalves

Marine mammals

For all samples inorganic arsenic constitutes less than 1% of total arsenic

...but unusual high contents in some samples...



Data from 175 blue mussel (*Mytilus edulis*) samples collected along the Norwegian Coastline.

Sloth and Julshamn , 2008, J. Agri.Food Chem., 56, 1269-1273

Arsenic in rice – an emerging health issue?

Environmental Pollution 152 (2008) 746–749

Rapid communication

Inorganic arsenic levels in baby rice are of concern

Andrew A. Meharg [a,*], Guoxin Sun [b], Paul N. Williams [a,b], Eureka Adomako [a], Claire Deacon [a], Yong-Guan Zhu [b], Joerg Feldmann [c], Andrea Raab [c]

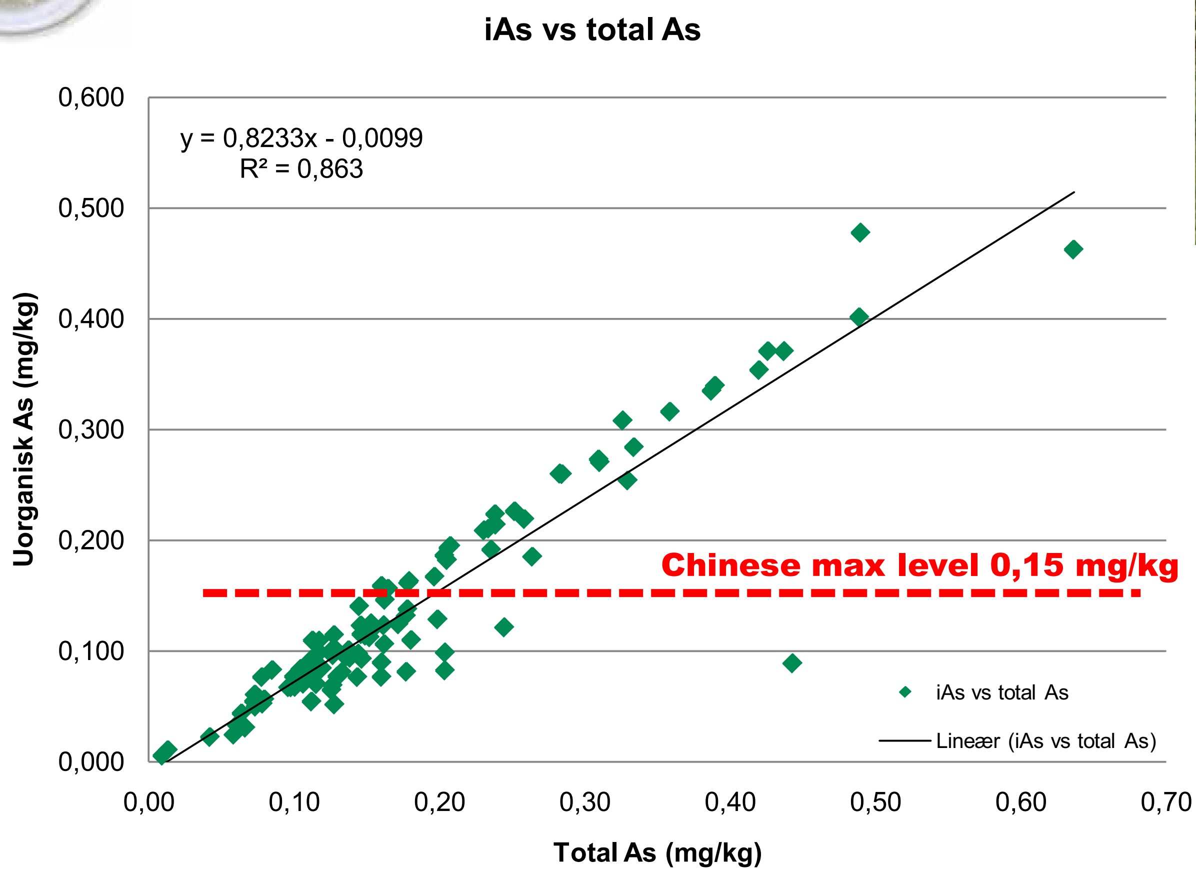
[a] School of Biological Sciences, University of Aberdeen, Cruickshank Building, St. Machar Drive, Aberdeen AB24 3UU, UK
[b] Research Centre for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, China

Median consumption of organic arsenic levels for UK babies from baby rice is above threshold considered safe.

- **17 samples from supermarkets in Aberdeen**
- **Total arsenic levels: 0.12 – 0.47 mg/kg**
- **Inorganic arsenic: 0.06 – 0.16 mg/kg (33 – 69 % of tAs)**
- **35% above Chinese max level of 0.15 mg/kg iAs**
- **No regulation on As in food in EU (yet!)**

Comment on cereals Cubadda

Arsenic in rice products



105 prøver i alt

-Hvide ris (white rice)
-Brune ris (brown rice)
-Røde ris (red rice)
-Sorte ris (black rice)
-Ris kiks (rice crackers)

33 prøver > 0,15 mg/kg

-2 parboiled (20%)
-4 brune (50%)
-4 røde (50%)
-5 sorte (71%)
-1 Basmati (10%)
-1 Grød (9%)
-1 vilde (20%)
-15 ris kiks (100%)

Rice cracker mean: 0,31 mg/kg – intake50 g/dag => 15 µg iAs (~1µg/k

> EFSA $BMDL_{01}$ på 0,3-8 µg/kg bw/dag

Inorganic arsenic in chinese food supplements

Name of Food supplement	Total Arsenic (µg/g)	Inorganic arsenic (µg/g)
Xiao Yao Wan	0.82	0.85
Bu Zhong Yi Qi Wan	0.62	0.50
Da Bu Yin Wan	0.59	0.55
Six Flavor teapills	0.72	N.D.
Golden Book Teapills	0.58	0.57
Xiang Sha Liu Jun Zi Wan	0.94	0.80
Gan Mao Ling	1.24	1.01
Chuan Xin Lian	5.00	3.17
Bi Yan Pian	0.70	0.58
Arouse power	1.12	1.02
Bio Chlorella	0.62	0.21
Unik Spirulina Kapsler	2.59	0.13
Chlorella	0.58	0.03
Ez-Biloba	0.63	0.67
Qvinde Dong Quai	0.68	0.48

Gan Mao Ling
Rec dose: 18 pills per day
⇒ iAs ~ 13 µg/day
⇒ 0.22 µg/kg bw/day (60 kg)
Close to EFSA BMDL !!

Hedegaard and Sloth, in prep

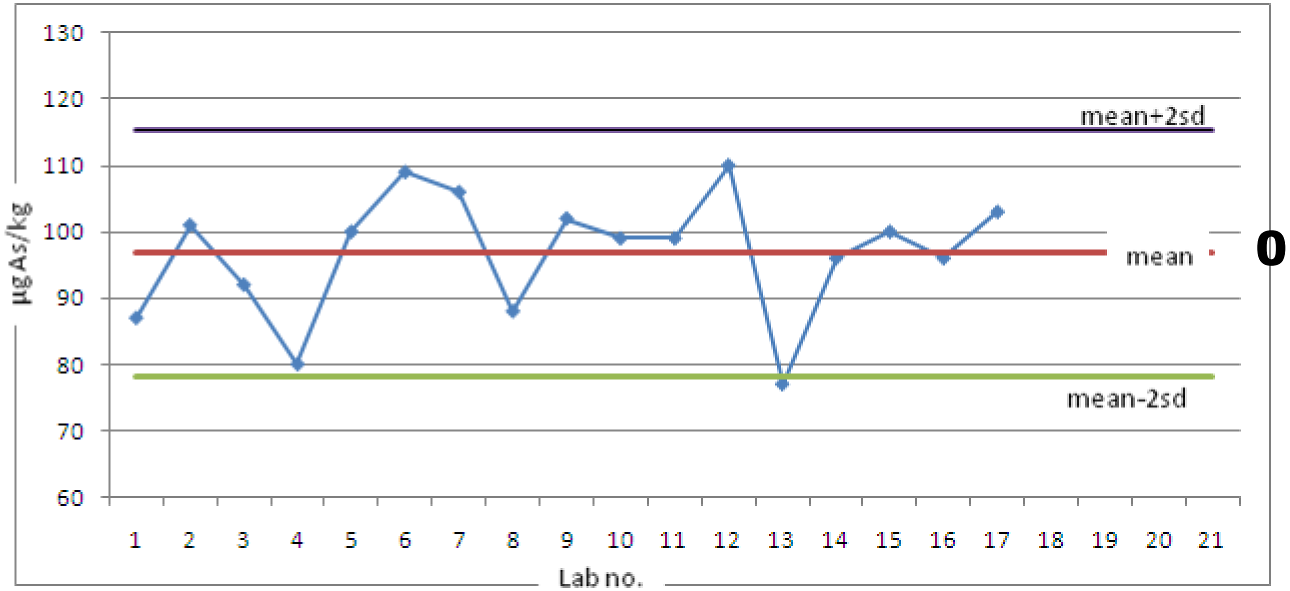
Recent meeting: **the Expert Committee “Industrial and Environmental Contaminants”**

“Based on the data processed so far, a maximum level of 0.2 mg/kg inorganic arsenic for rice as a category seems achievable”

“MSs are asked to reflect on the need for a maximum level for cereals other than rice”

“MSs are encouraged to concentrate on levels for inorganic arsenic in rice (collecting information on country of origin and rice variety), food supplements (algae) and infant food (rice based)”

Inorganic As in SRM NIST1586a

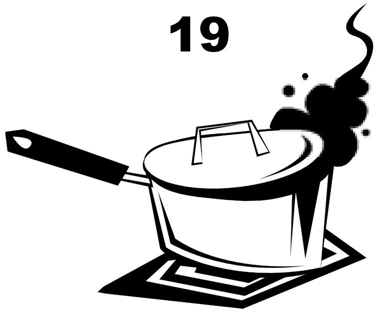


0.097 mg/kg

	Extraction solution	Detection	iAs (µg/kg)			Reference
1	2M TFA	LC-ICPMS	87	+/-	9	Ackermann (2005)
2	Enzymatic, pepsin and pancreatin	LC-ICPMS	101	+/-	7	Ackermann (2005)
3	2M TFA	LC-ICPMS	92	+/-	2	Heitkemper (2001)
4	2M TFA	LC-ICPMS	80	+/-	16	Williams (2005)
5	2M TFA	LC-ICPMS	100	+/-	10	Williams (2006)
6	MeOH:H2O	LC-ICPMS	109	+/-	3	D'Amato (2004)
7	Enzymatic, alfa-amylase	LC-ICPMS	106	+/-	7	Kohlmeyer (2003)
8	Enzymatic, protease and alfa-amylase	LC-ICPMS	88	+/-	6	Sanz (2005)
9	1M H3PO4	HG-AFS	102	+/-	2	Matosreyes (2007)
10	1% HNO3	LC-ICPMS	99	+/-	4	Raab (2009)
11	1% HNO3	LC-ICPMS	99	+/-	-	Sun (2008)
12	1% HNO3	LC-ICPMS	110	+/-	10	Sun (2009)
13	0,5 M TFA	LC-ICPMS	77	+/-	-	Heitkemper (2009)
14	enzymatic, proteas and alfa-amylase	LC-ICPMS	96	+/-	9	Mar (2009)
15	2M TFA	LC-ICPMS	100	+/-	12	Meharg (2008)
16	water	LC-ICPMS	96	+/-	3	Narukawa (2008)
17	0,07M HCL and 10%H2O2	LC-ICPMS	103	+/-	15	DTU Food (2009)

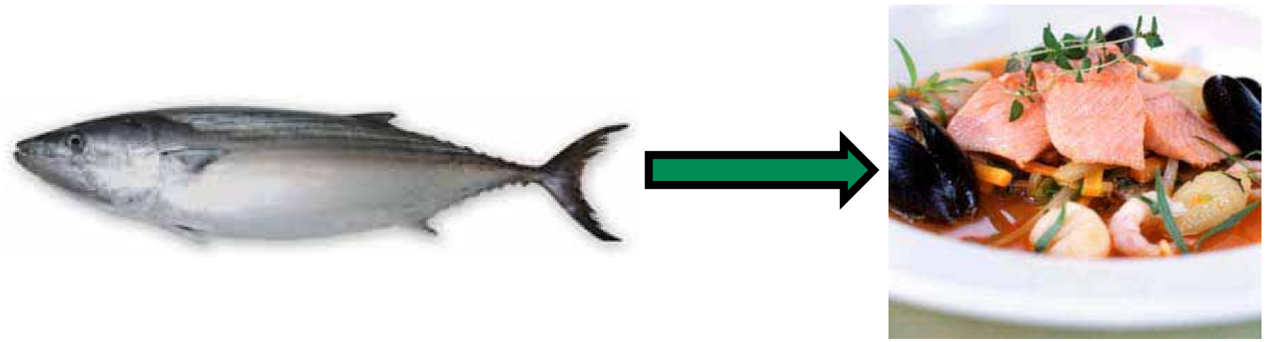
Good agreement between labs and methods

+ lecture by M. de la Calle



Processing

- Processing or storage may alter the arsenic species pattern
- No transformation of organoarsenic compounds to inorganic arsenic by normal cooking procedures (IMPORTANT!)
- Loss/uptake to/from boiling water possible
- Blue mussels => loss of AB after storage by freezing



Ref: Devesa et al, 2008, Food.Chem.Tox + Dahl et al, 2010, Food Chem. (accept

Commission directive 2009/114/EC (amendment)

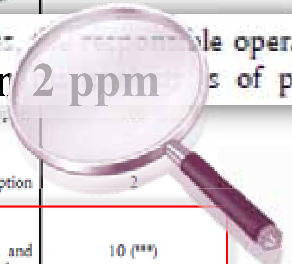
Undesirable substances	Products intended for animal feed	Maximum content in mg/kg (ppm) relative to a feedingstuff with a moisture content of 12 %
(1)	(2)	(3)
'1. Arsenic (*) (**)	Feed materials with the exception of:	2
	— meal made from grass, from dried lucerne and from dried clover, and dried sugar beet pulp and dried molasses sugar beet pulp,	4
	— palm kernel expeller,	4 (***)
	— phosphates and calcareous marine algae,	10
	— calcium carbonate,	15
	— magnesium oxide,	20
	— feedingstuffs obtained from the processing of fish or other marine animals, including fish,	25 (***)
	— seaweed meal and feed materials derived from seaweed,	40 (***)
	Iron particles used as tracer.	50
	Additives belonging to the functional group of compounds of trace elements except:	30
	— [illegible] oxide,	[illegible]
	Complete feedingstuffs with the exception of:	2
	— complete feedingstuffs for fish and complete feedingstuffs for fur animals,	10 (***)
	Complementary feedingstuffs with the exception of:	4
	— mineral feedingstuffs.	12

Only max levels for total arsenic

FOOTNOTE

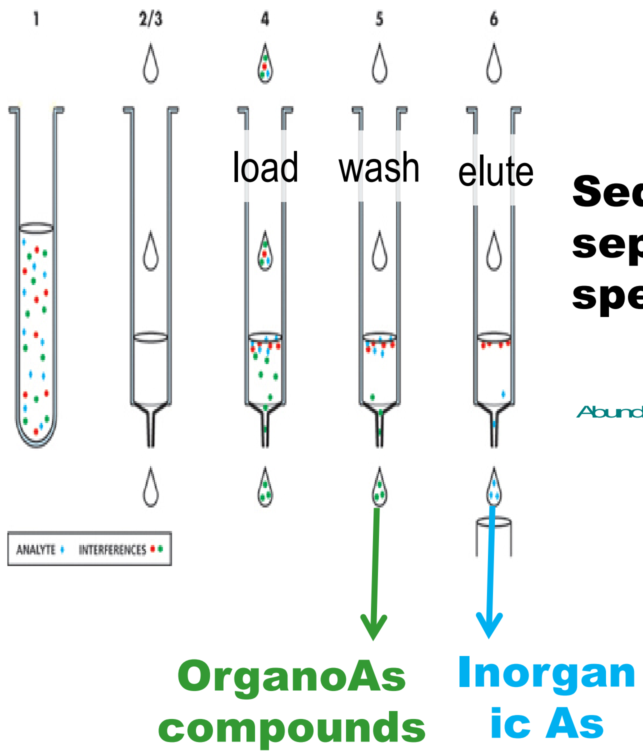
(***) Upon request of the competent authorities, the responsible operator must perform an analysis to demonstrate that the content of ... s of particul... eaweed species *Hizikia fusiforme*.'

Inorganic arsenic lower than 2 ppm

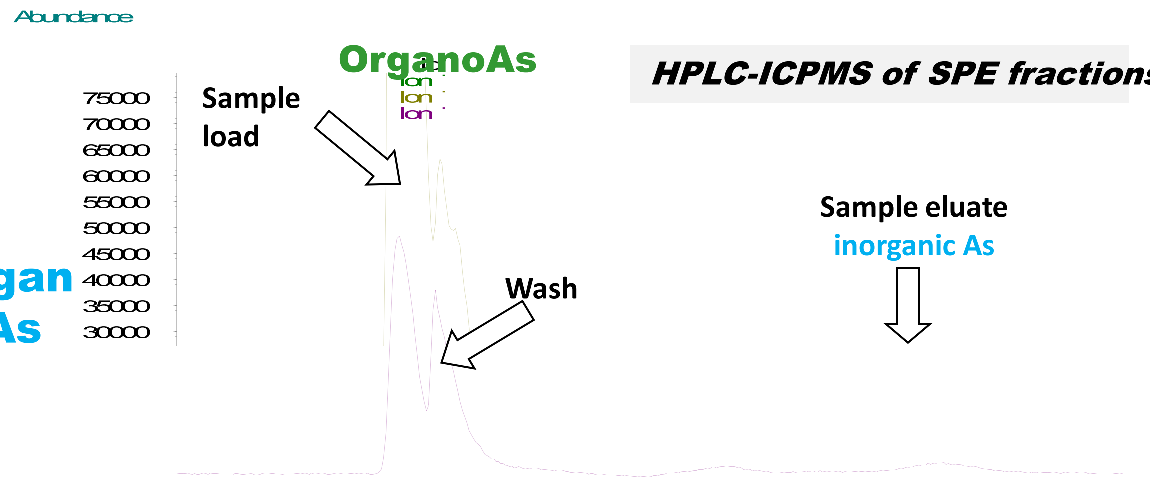


Speciation analysis is required !!

SPE-HG-AAS – a speciation alternative…

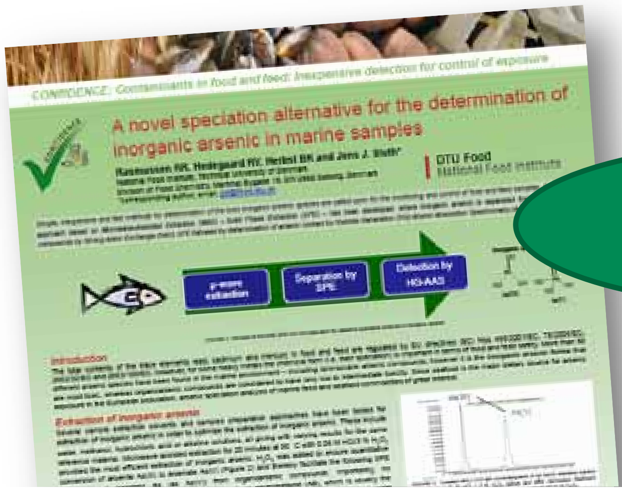


Sequential elution for selective off-line separation of inorg As from organo As species by SPE



Performance characteristics from in-house validation

Parameter	Result
Analysis time	2 x 7 h for 24 samples
LoD (mg/kg)	0.08
LoQ (mg/kg)	0.16
Repeatability (%RSD)	3 - 7
Accuracy (%)	90 - 104



Further details on poster and handouts

Samples for collaborative trial

Sample no	Sample type	Spiked / unspiked	Mean concentration (mg Kg^{-1})
IMEP32-1	Complete fish feedingstuff	Unspiked	0.07
IMEP32-2	Complete fish feedingstuff	Spiked	0.71
IMEP32-3	Fish meal	Unspiked	0.19
IMEP32-4	Fish meal	Spiked	1.06
IMEP32-5	Fish fillet	Spiked	2.64
IMEP32-7	Fish meal	Spiked	0.43
IMEP32 Control sample	CRM TORT-2 Lobster Hepatopancreas	Unspiked	0.54

Concentration range: 0.07 – 2.64 mg Kg^{-1}

Control sample

National Research Council Canada
Conseil national de recherches Canada

TORT-2

Lobster Hepatopancreas Reference Material for Trace Metals

Trace Metals
(milligrams/kilogram)

Arsenic (g,h,m) 21.6 ± 1.8

- **Total As**
- Not certified for iAs

Results - overview

Matrix	Units	IMEP 32-1	IMEP 32-2	IMEP 32-3	IMEP 32-4	IMEP 32-5	IMEP 32-7	IMEP 32 Control Sample
N° of participating laboratories		10	10	10	10	10	10	10
Remaining data after outlier elimination		29	35	28	36	36	30	34
N° of remaining laboratories		9	10	9	10	10	9	10
Outliers	%	12.1	2.8	12.5	0.0	0.0	11.8	0.0
Overall mean $X_{obs} \pm u_{obs}$	mg Kg^{-1}	0.071 ± 0.041	0.713 ± 0.117	0.189 ± 0.060	1.062 ± 0.140	2.643 ± 0.506	0.432 ± 0.066	0.544 ± 0.162
S_r	mg Kg^{-1}	0.016	0.054	0.014	0.105	0.277	0.023	0.095
RSD_r	**%**	**22.8**	**7.6**	**7.5**	**9.9**	**10.8**	**5.4**	**17.5**
r_L	mg Kg^{-1}	0.046	0.153	0.040	0.294	0.776	0.065	0.266
S_R	mg Kg^{-1}	0.041	0.117	0.060	0.140	0.506	0.066	0.162
RSD_R	**%**	**57.6**	**16.4**	**31.9**	**13.2**	**19.1**	**15.3**	**29.7**
R_L	mg Kg^{-1}	0.115	0.327	0.169	0.391	1.416	0.185	0.453
σ_H	mg Kg^{-1}	0.017	0.120	0.039	0.168	0.365	0.078	0.095
HorRat		**2.4**	**1.0**	**1.6**	**0.8**	**1.4**	**0.8**	**1.7**

HorRat >2
Low concentration!!

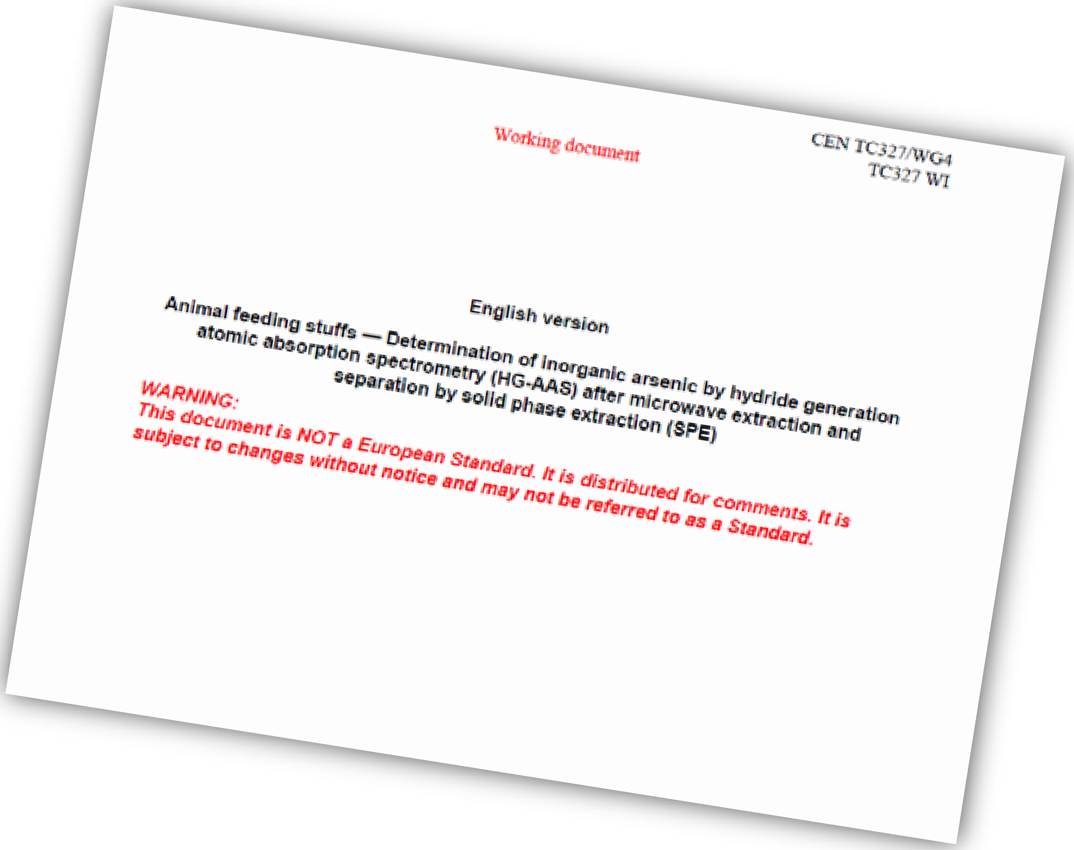
All HorRat values < 2

Conclusions - I

- **A method for determination of iAs in feed of marine origin based on SPE-HG-AAS has been developed**
- **homogene and stable test samples were prepared**
- **a collaborative trial was conducted in Oct-Nov 2010**
- **Ten laboratories were evaluated as compliant**
- **Max 1 outlier lab was identified per sample**
- **HorRat values < 2 (0.8 – 1.7) for samples in the concentration range 0.19 – 2.6 mg Kg^{-1}**
- **HorRat value > 2 (2.4) for S01 at concentration 0.07 mg Kg^{-1}**
- **-Accuracy from control sample, mean = 0.544±0.162 mg Kg^{-1}**
  -Assigned value from HPLC-ICPMS determinations = 0.599±0.07 mg Kg^{-1}
  -0.544/0.599 = 91 % (no significant difference)

Conclusions - II

- **EU directive ”guideline” maximum level is 2 ppm (EU directive 2009/141/EC on animal feed)**
- **Method working range tested in ILC: 0.1 – 2.6 mg Kg^{-1}**
- **HorRat values <2 in the working range tested**
- **The method is fit for purpose**



Working document

CEN TC327/WG4
TC327 WI

English version

Animal feeding stuffs — Determination of inorganic arsenic by hydride generation atomic absorption spectrometry (HG-AAS) after microwave extraction and separation by solid phase extraction (SPE)

WARNING:
This document is NOT a European Standard. It is distributed for comments. It is subject to changes without notice and may not be referred to as a Standard.

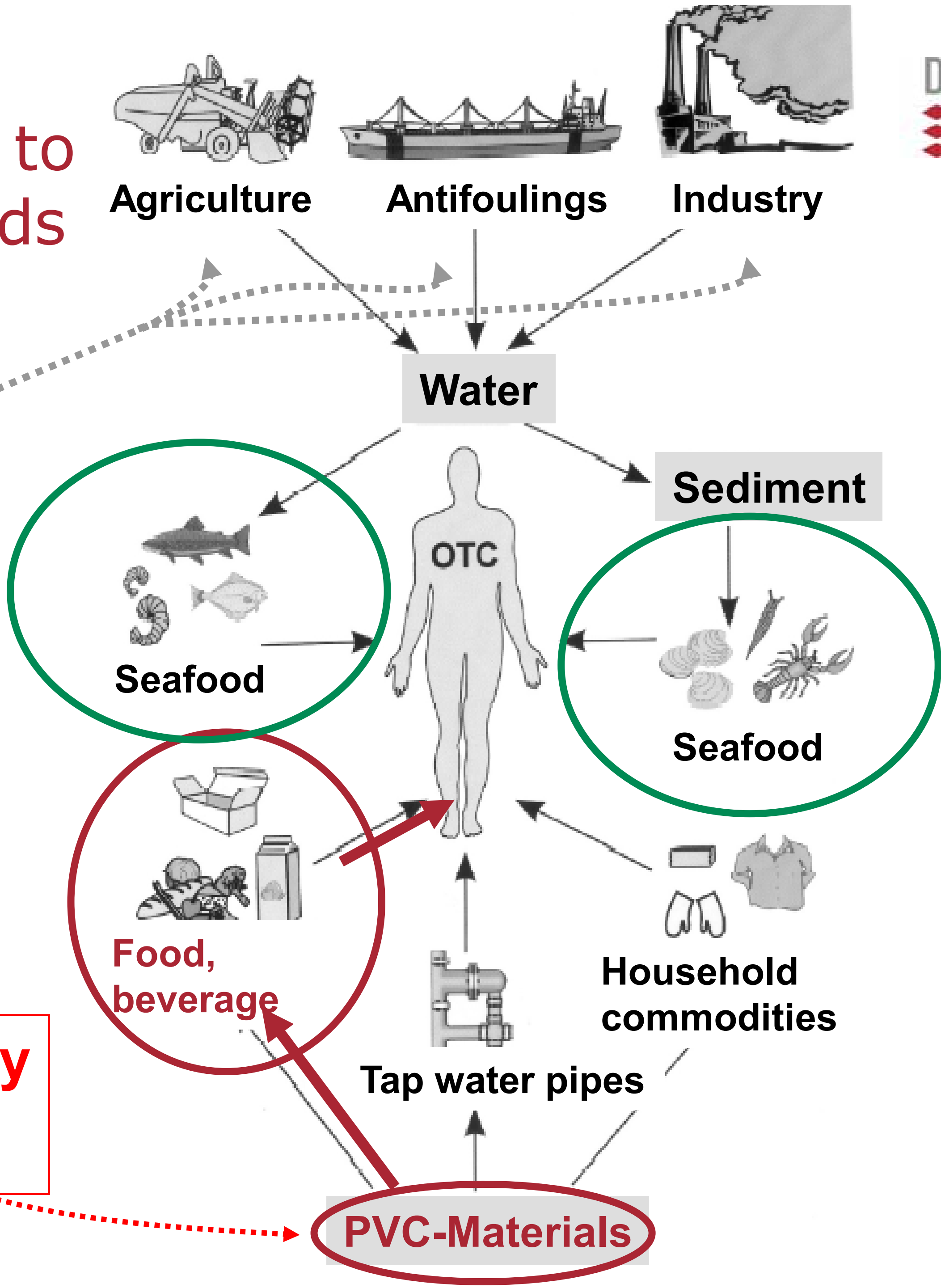
Routes of exposure to organotin compounds

Used in
- **Agriculture**
- **Antifoulings**
- **Industry**

- **PVC-Materials**

TDI: 0.25 µg/kg bw/day

∑TBT, DBT, TPhT and DOT



Hoch, Appl. Geochem., 2001, 719-43

Legislation on OTCs in Food Contact Materials

Compounds	Maximum level (µg Sn/kg foodstuff)
$\sum$ DBT, TBT, TPhT and DOT	40 (6)
$\sum$ MMT, DMT	180
MOT	1200
MDDT	12000 (50)
DDDT	24000 (50)

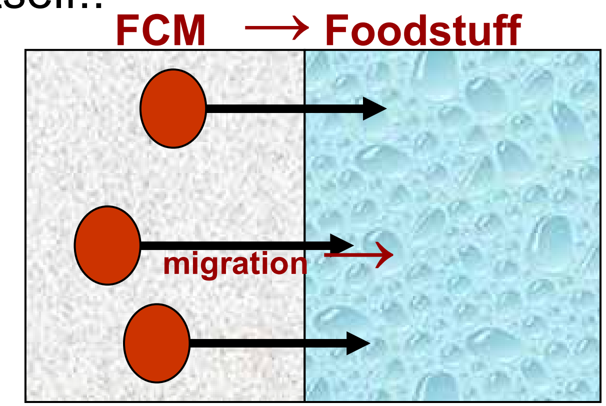
Ref: EFSA (2005); proposed EFSA values in parenthesis

Assumptions:

- 1 kg food per 6 dm^2

- 100 mL in contact with 0.6 dm^2

- Max levels on organotin migrating from the packaging material
- Testing by the use of food simulators (water, acid, oil, alcohol etc)
- BUT no maximum levels on organotins in the foodstuff itself!!

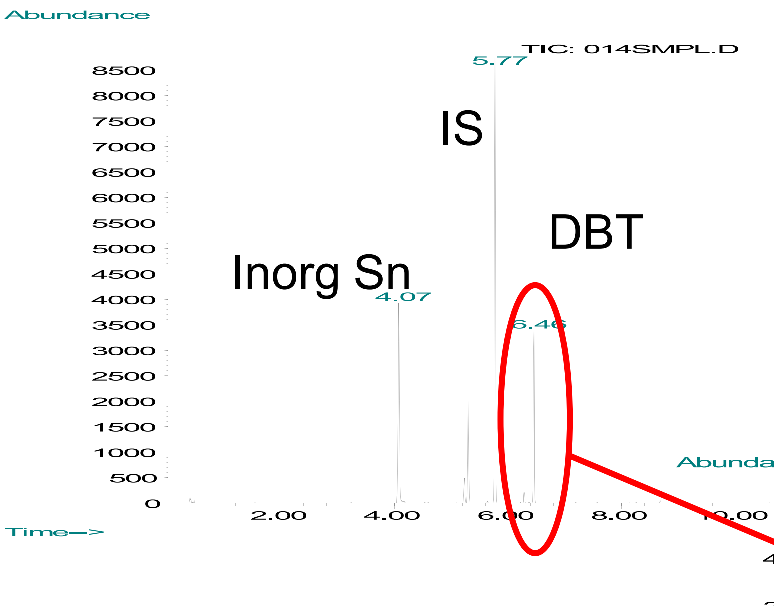


Organotin migration from Food Contact Materials II

Small scale survey on 33 FCMs

Baking paper, PVC cling films, silicone baking forms, lids with PVC gaskets

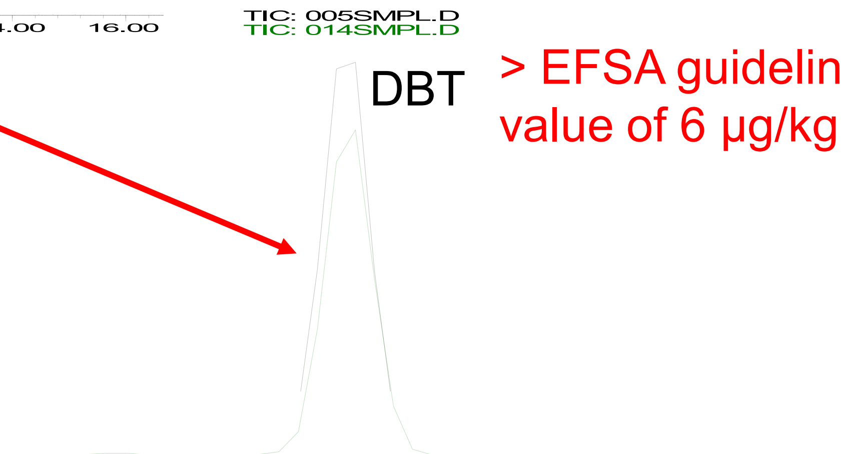
PUR-agglomerated cork wine stoppers



- •PVC lid
- •3% acetic acid

Overlaid standard and sample

➢ DBT concentration: 9.9 µg/kg

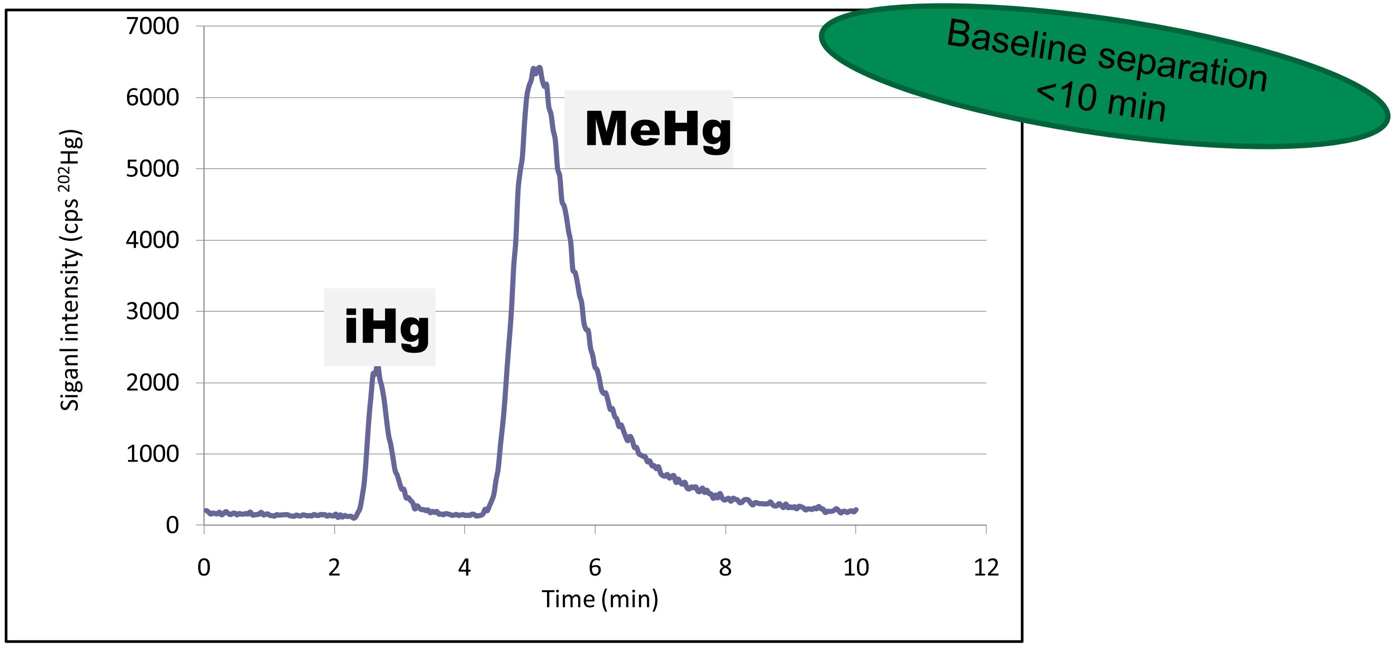


> EFSA guideline value of 6 µg/kg

Output of DK survey:
-33 samples
-11 contained OT (mainly DBT)
-3 exceeded EFSA guideline limit

Sloth et al, in prep

Speciation analysis of Mercury by HPLC-ICPMS



HPLC-ICPMS chromatogram of DORM-3 (Dogfish muscle)

CRM	Certified (mg/kg)	Result (mg/kg)
DORM-2 (dogfish muscle)	4.47 +/- 0.32	4.21
DORM-3 (dogfish muscle)	0.355 +/- 0.056	0.35
TORT-2 (Lobster hepatopancreas)	0.152 +/- 0.013	0.16

Relevance for regulation ?? But Codex opinion

Selenium in commercial food supplements

Organic bound Se?

Se yeast?

Selenomethionine?

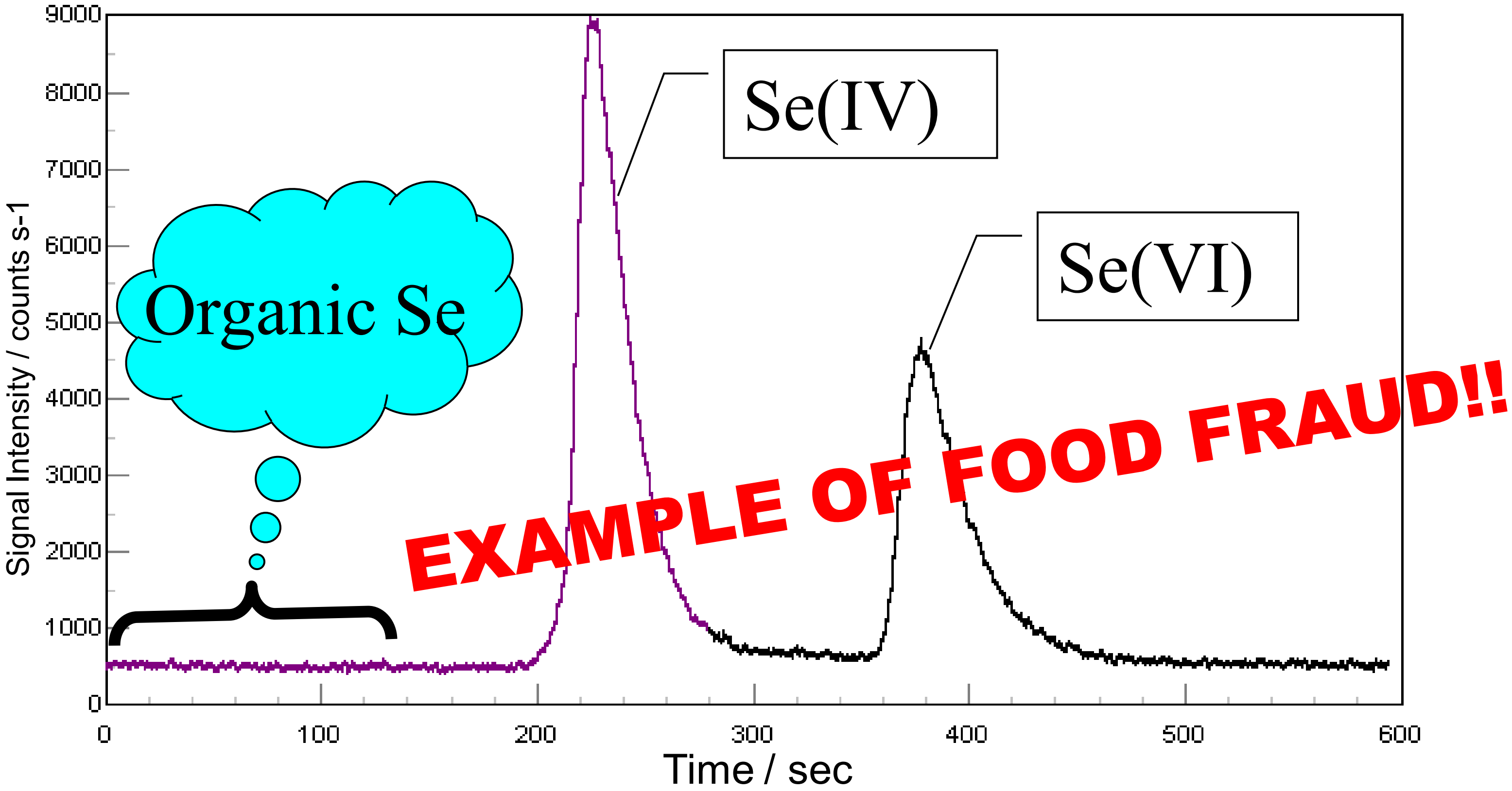
Selenite?

Amino chelated Se ?

Selenate?

Se speciation by HPLC-ICPMS

Supplement declared as *organic bound* Selenium (125 µg/tablet)



Larsen, unpublished data.

Identification number of the additive	Name of the holder of authorisation	Additive	Composition, chemical formula, description, analytical method	Species or category of animal	Maximum age	Minimum content	Maximum content	Other provisions
						Maximum content of element (Se) in mg/kg of complete feedingstuff with a moisture content of 12 %		
Category of nutritional additives. Functional group: compounds of trace elements								
3b8.12	—	Selenomethionine Selenomethionine produced by *Saccharomyces cerevisiae* CNCM I-3399 (Selenised yeast inactivated)	Characterization of the additive: Organic selenium mainly selenomethionine (63 %) content of 2 000-2 400 mg Se/kg (97-99 % of organic selenium) Characterisation of the active substance: Selenomethionine produced by *Saccharomyces cerevisiae* CNCM I-3399 (Selenised yeast inactivated) Analytical method (1): Zeeman graphite furnace atomic absorption spectrometry (AAS) or hydride AAS	All species	—		0,50 (total)	1. The additive shall be incorporated in to feed in form of a premixture. 2. For user safety: breathing protection, safety glasses and gloves should be worn during handling.

(1) Details of the analytical methods are available at the following address of the Community Reference Laboratory: www.irmm.jrc.be/crl-feed-additives

Selenium in feed
Incurred content vs added

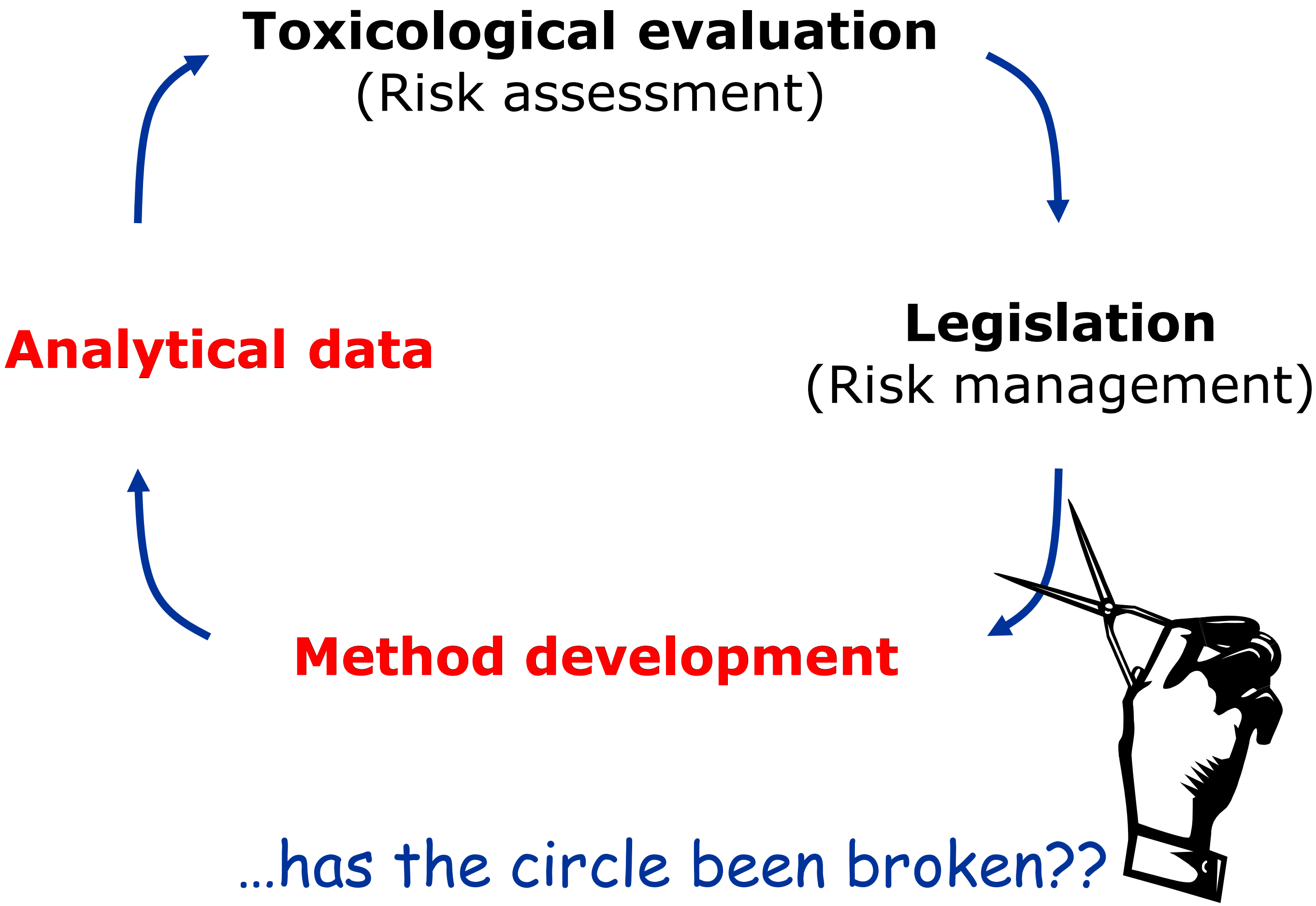
Organic bound minerals vs inorganic minerals
Feed additives and food supplements
EFSA opinions

Speciation summary

- ✓ speciation methods are more and more commonly used
- ✓ instrumentation is widely available
- ✓ legislation on species has started
- ✓ …and more is expected in the future!

- ✓ standardised methods are not ready!
- ✓ …but the need is known by authorities
- ✓ legislation is still behind!
- ✓ Lack of CRMs (e.g. for iAs)

...how to proceed?



Perspectives I

- Maximum levels in the legislation are needed
- Standardised methods for official control is needed
- On-site screening methods for fast answers

  - biosensor based => answer: result>ML?

  - can a batch of food/feed be released for trade or is further analysis required?
- Fraud cases in food supplements / feed additives

  - do you get what you pay for?

Perspectives II – trace elements in nanoform

AEROSIL® ***Evonik***

silica nanoparticles as food additive for powdered food (e.g. soup, coffee creamer)

http://www.aerosil.com/

silver nanoparticles as food supplement

www.fairvital.com

nanoclay in PET beer bottles

www.honeywell.com

NanoLyse Project

"Nanoparticles in Food: Analytical methods for detection and characterisation"

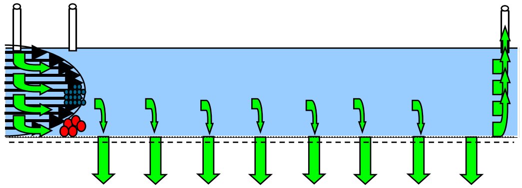
Validated methods for the determination of inorganic ENP in food extracts, based on size separation, size determination and specific detection

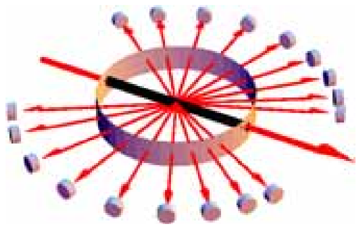
silver nanoparticles in lean meat

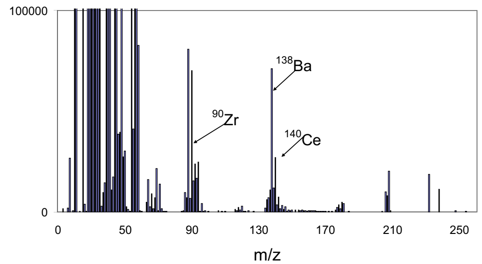
silica nanoparticles in tomato soup

http://www.nanolyse.eu

Our platform: AF4-MALS/DLS-ICP-MS







asymmetric flow field flow fractionation

(AF4)

optical detection multi angle (MALS) and dynamic light scattering (DLS), UV-vis absorption and fluorescence

inductively coupled plasma mass spectrometry

(ICP-MS)

particle separation according to their size (1nm – few µm)

particle detection

size determination

elemental detection chemical identity

quantification

Schmidt, B.; Loeschner, K.; Hadrup, N.; Mortensen, A.; Sloth, J.J.; Koch, C.B. and Larsen, E.H.: "Quantitative Characterization of Gold Nanoparticles by Field-Flow Fractionation Coupled Online with Light Scattering Detection and Inductively Coupled Plasma Mass Spectrometry", Analytical Chemistry, vol. 83 (7), 2461-2468 (2011).

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CONFIDENCE

NANOLYSE

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Ministry of Food,
Agriculture and Fisheries
Danish Veterinary and Food Administration

Thanks for your attention