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Profiling Trace Metals in Food: LC-ICP-MS Speciation of Arsenic in Rice

Rima Juskelis, Katie Banaszewski, Jack Cappozzo

Institute for Food Safety and Health
Illinois Institute of Technology, Chicago, IL

October 9, 2012

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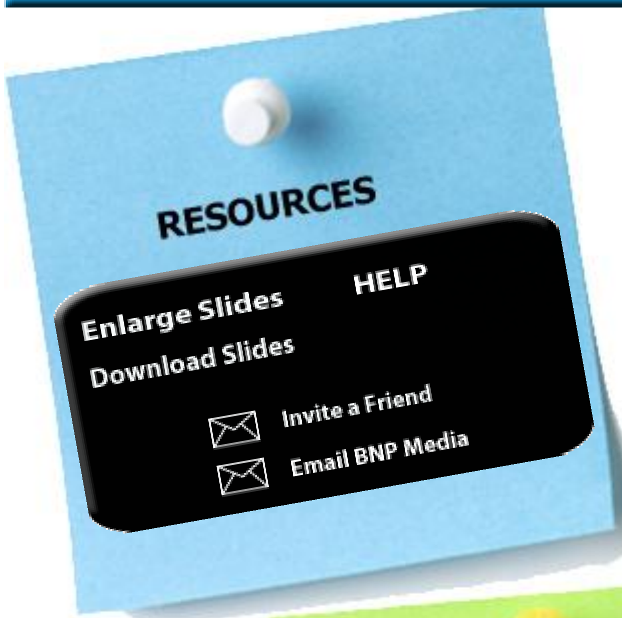

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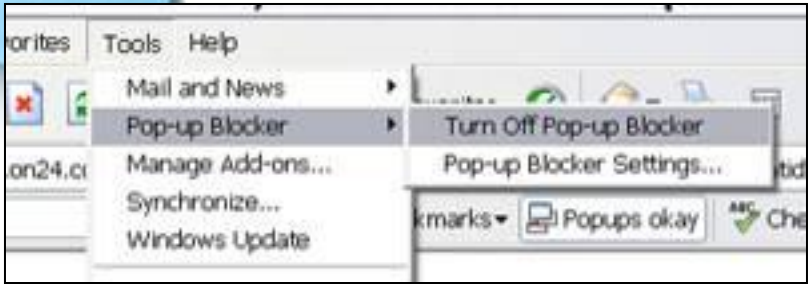

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Today's Outline

- Background – Why is this important?
- Analytical Method
- As Speciation
- Total As

Background

Food safety has become a major focus for the general public, food industry and government agencies. Government agencies have taken steps to increase food safety by requiring HACCP a few years back and recently passing the Food Safety Modernization Act (FSMA). Arsenic in food has come to light because of the attention of media but also as a real concern especially for imported foods and ingredients into the United States. Rice is currently being studied because of the lack of information to gain information for risk assessment.

Toxic Metals in Foods and Water in the Media

- Hexavalent Cr in drinking water – the Erin Brokovich Story
- Mercury in canned tuna (and other fish)
- Arsenic in chicken feed
- Arsenic in Apple juice
- Arsenic in rice and brown rice syrup

- What is safe, what isn't, and how do we know?

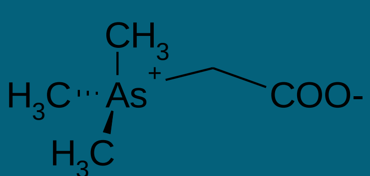
Why analyze concentration of species instead of total elemental concentrations?

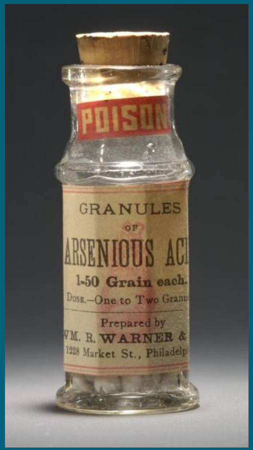
- The toxicity, bioavailability, and mobility of a given element depends on its form.
  - •e.g., inorganic arsenic is far more toxic than organic arsenic, but inorganic mercury is far less toxic than organic mercury

- Lethal dose of organic arsenic
  - LD_{50} of DMA 200-1000 mg per kg body weight
  - LD_{50} of Arsenobetaine >10,000 mg per kg body weight

Lethal dose of inorganic arsenic

- LD_{50} of As_2O_3 10 mg per kg body weight
- LD_{50} of As_2O_5 30 mg per kg body weight





Arsenic in rice – Is it toxic, or not…

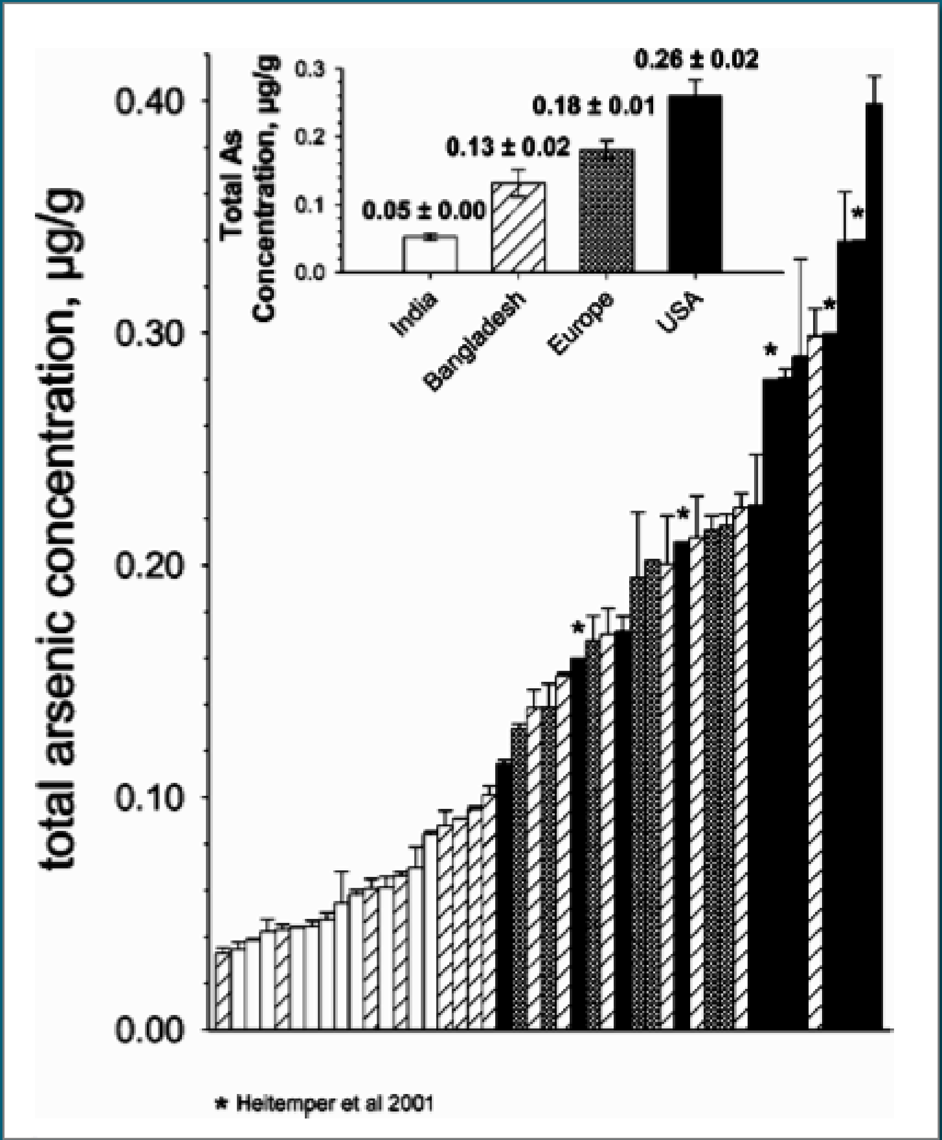
Variation in Arsenic Speciation and Concentration in Paddy Rice Related to Dietary Exposure

P. N. WILLIAMS,[†] A. H. PRICE,[†]
A. RAAB,[‡] S. A. HOSSAIN,[†,§]
J. FELDMANN,[‡] AND A. A. MEHARG*,[†]
School of Biological Sciences, University of Aberdeen, Aberdeen, AB24 3UU, UK, and Department of Chemistry, University of Aberdeen, Aberdeen, AB24 3UE, UK

- "USA long grain rice had the highest mean arsenic level in the grain at 0.26 μg As g^{-1}"

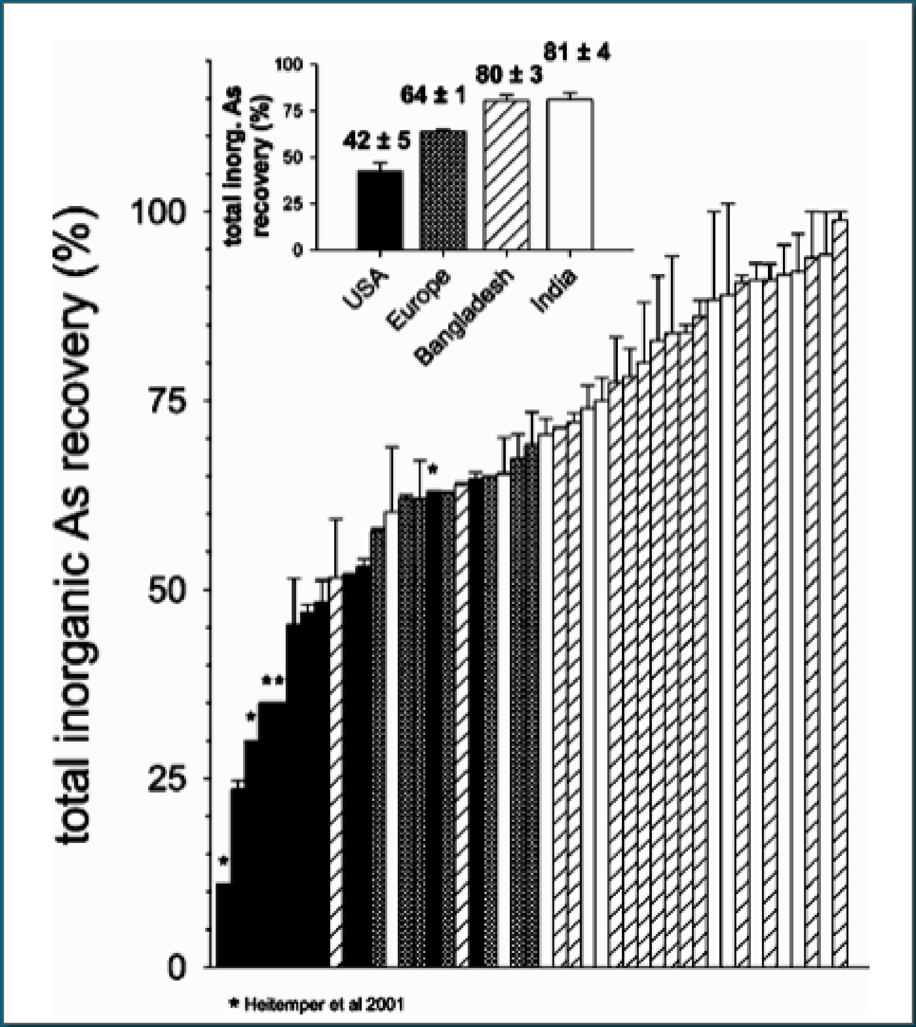
- "arsenic in rice contributes considerably to arsenic ingestion in subsistence rice diets"

- Williams et al ES&T 39: 2005



Arsenic speciation in rice

- Percentage inorganic As in market rice.



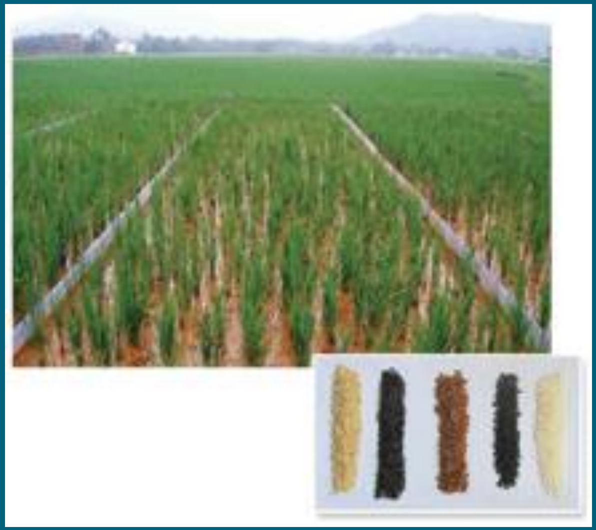
- Arsenic grown on the same soil
- 'genetic variation accounts for differences in uptake and speciation' speciation in the grain differs between rice cultivars

- Is this legacy arsenic from DMA use as a pesticide?
- DMA is main form of arsenic in US rice:

- Williams et al ES&T 39: 2005

Arsenic and Paddy Rice: A Neglected Cancer Risk?

- Three sets of findings reporting elevated As in rice and products such as rice bran and rice crackers
- Speciation: inorganic – oxides As^{V} and As^{III}
- China regulates As levels in food: recently reduced 'safe' level from 700 to 150 μg kg^{-1}
- US and EU have not set limits for As in food
- 1993 US Food & Drug Administration Guidance document on shellfish consumption: tolerable daily intake 130 μg day^{-1}
- Rice contain 10 fold more As than wheat or other cereals



- Field trials of low-As rice:
- Zhu Yong-Guan's group.
- Grain levels ranged from 63-421 μg kg^{-1}

- Science 11 July 2008 Volume 321

UK food agency 2009 advisory

Food Standards Agency

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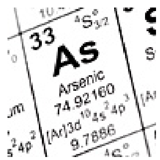
Arsenic in rice research published

Thursday 21 May 2009



The Agency has today published results from two studies: arsenic levels in rice drinks and one on cooking methods to reduce arsenic levels in rice. As a result of the rice drink study, the Agency recommends that toddlers and young children should not have rice drinks, often known as rice milk, as a replacement for cows' milk, breast milk or infant formula.

The rice drink study followed concerns about results from a study published last year that measured arsenic levels in these types of drinks. The research published today examined 60 samples of rice drinks and found low levels of arsenic in all of them (see The science behind the story section below).

The level of total arsenic ranged from 0.010 - 0.034 milligram/kilogram and the levels of inorganic – the more harmful – form of arsenic ranged from 0.005 - 0.020 milligram/kilogram. The proportion of inorganic arsenic in the rice drink samples ranged from 48 - 63%. None of the results were over the current legal limit (but see the Current regulations section below).

In the second study, researchers looked at the effect of cooking methods on arsenic content of rice. The Agency is not advising anyone to change the way they cook rice as a result of this study as the impact on the overall dietary intake of arsenic from different cooking methods is minimal.

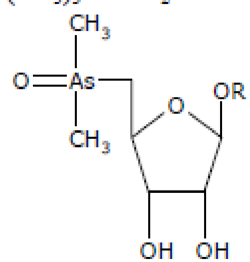
What the Agency advises

As a precaution, toddlers and young children between 1 and 4.5 years old should not have rice drinks as a replacement for cows' milk, breast milk, or infant formula. This is because they will then drink a relatively large amount of it, and their intake of arsenic will be greater than that of older children and adults relative to their bodyweight. This is both on nutritional grounds and because such substitution can increase their intake of inorganic arsenic, which should be kept as low as possible. A daily half pint or 280 millilitres of rice drink could double the amount of the more harmful form of arsenic they consume each day.

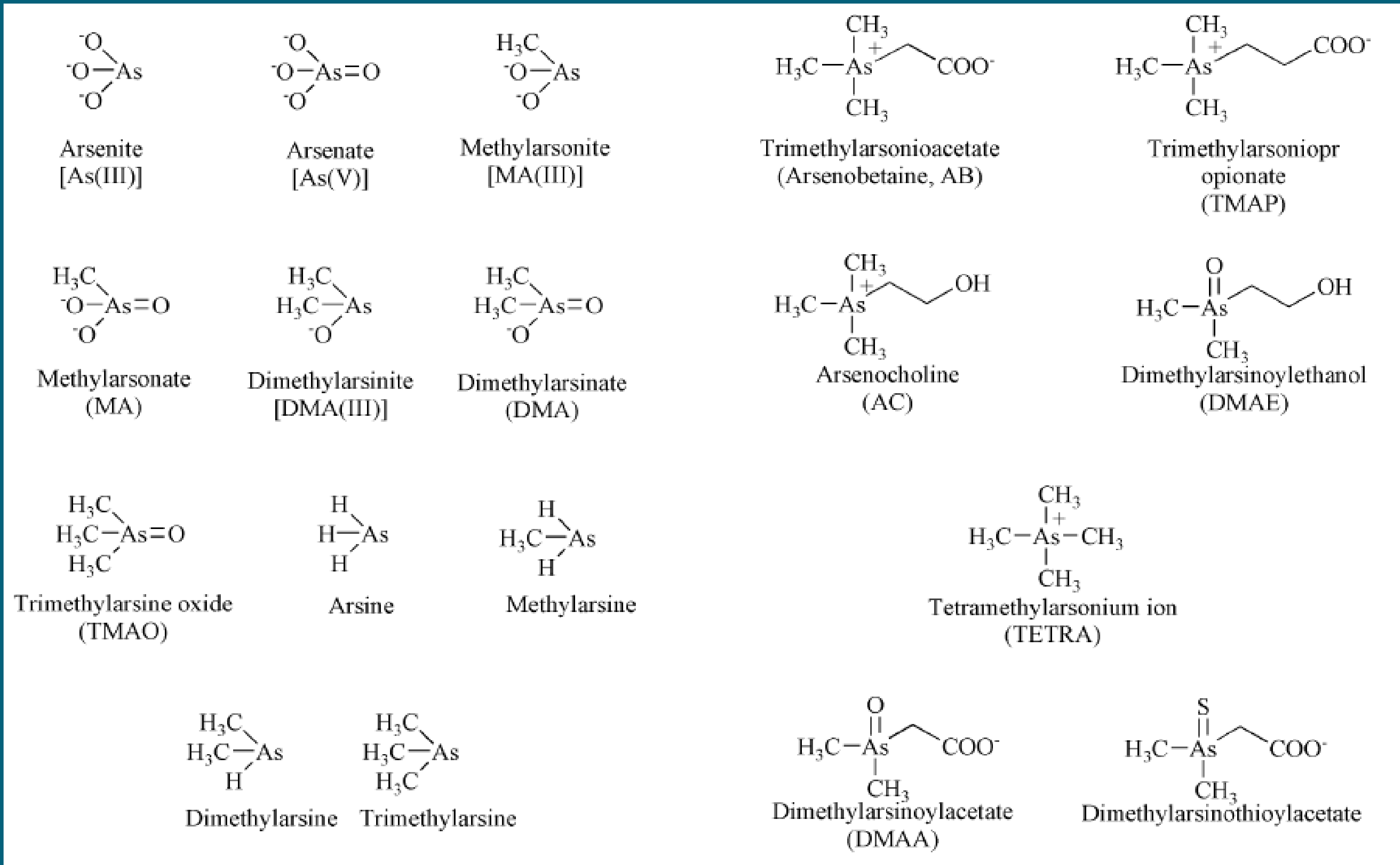
There is no immediate risk to children who have been consuming rice drinks and it is unlikely that there would have been any long-term harmful effects but to reduce further exposure to arsenic parents should stop giving these drinks to toddlers and young children.

Toxicity of Arsenic

Table 1: Names, abbreviations, and chemical structures for arsenic species referred to in this report (from EFSA Scientific Opinion on Arsenic in Food)

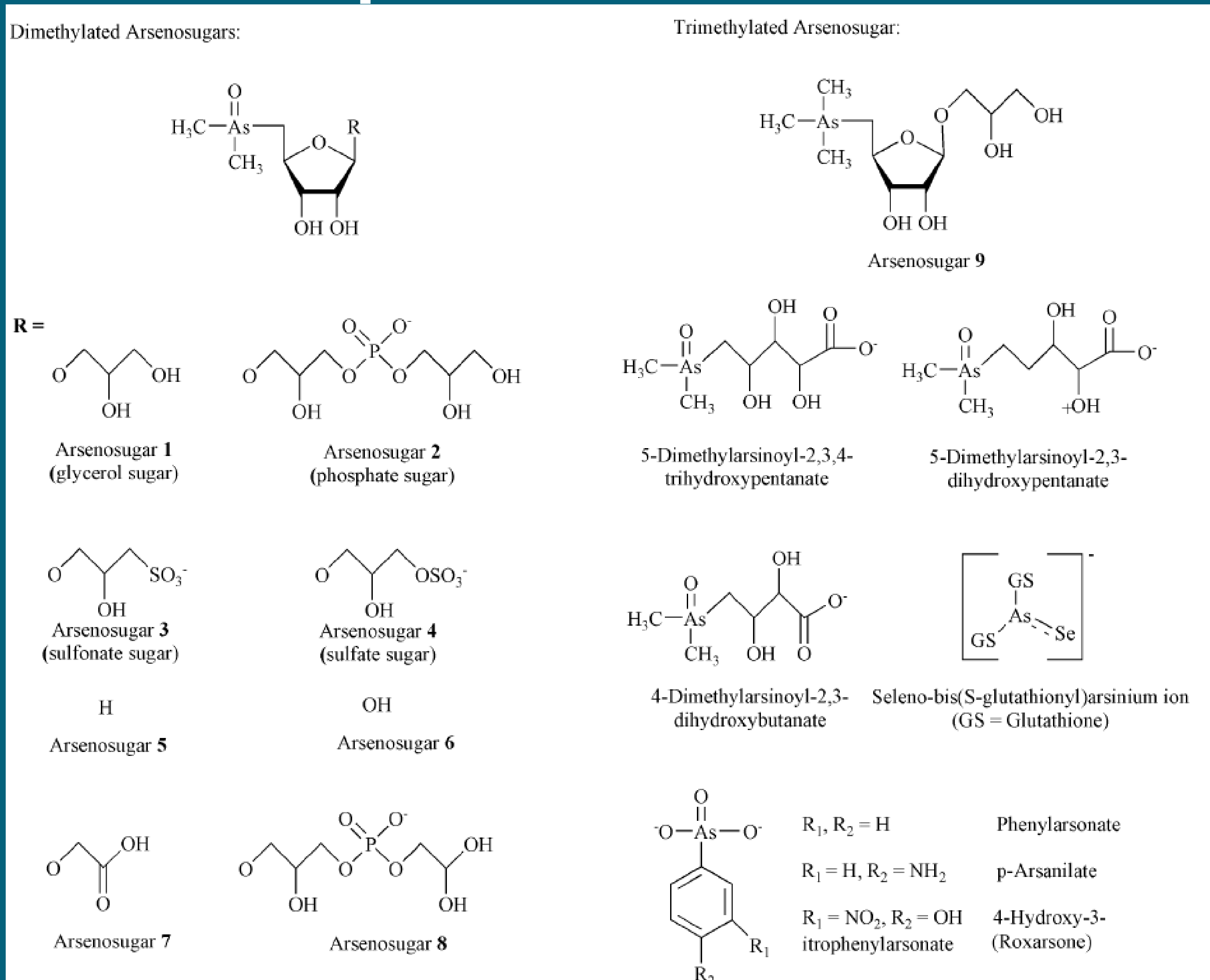
Name	**Abbreviation**	**Chemical structure**[(a)]	**Relevance/comment**
Inorganic arsenic	iAs		Sum of As(III) and As(V).
Arsenite	As(III)	$As(O^-)_3$	Trace to low levels in most foods; highly toxic.
Arsenate	As(V)	$O{=}As(O^-)_3$	Trace to low levels in most foods; a major form in water; highly toxic.
Arsenobetaine	AB	$(CH_3)_3As^+CH_2COO^-$	Major arsenic species in most seafoods; non-toxic.
Arsenosugars[(b)]			Major (edible algae) or significant (molluscs) arsenic species in many seafoods.
Arsenolipids[(c)]		e.g. 	Newly discovered arsenic species present in fish oils and fatty fish; likely to be present in other seafoods as well.
Trimethylarsonio propionate	TMAP	$(CH_3)_3As^+CH_2CH_2COO^-$	Minor arsenic species present in most seafoods.
Methylarsonate	MA	$CH_3AsO(O^-)_2$	Trace arsenic species of some seafoods and terrestrial foods; a significant human urine metabolite of iAs.
Methylarsonite	MA(III)	$CH_3As(O^-)_2$	Not usually detected in foods; detected in some human urine samples as a metabolite of iAs; a toxic species thought to be important for arsenic's mode of toxic action.
Dimethylarsinate	DMA	$(CH_3)_2AsO(O^-)$	Minor arsenic species in seafoods and some terrestrial foods; the major human urine metabolite of iAs, arsenosugars and arsenolipids.
Thio-dimethylarsinate	Thio-DMA	$(CH_3)_2AsS(O^-)$	A minor human urine metabolite of inorganic arsenic and arsenosugars.

There are a LOT of arsenic species!



Analyst, 2004, 129, 373–395

More arsenic species



Analyst, 2004, 129, 373–395

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Introduction to Chromatography for Speciation

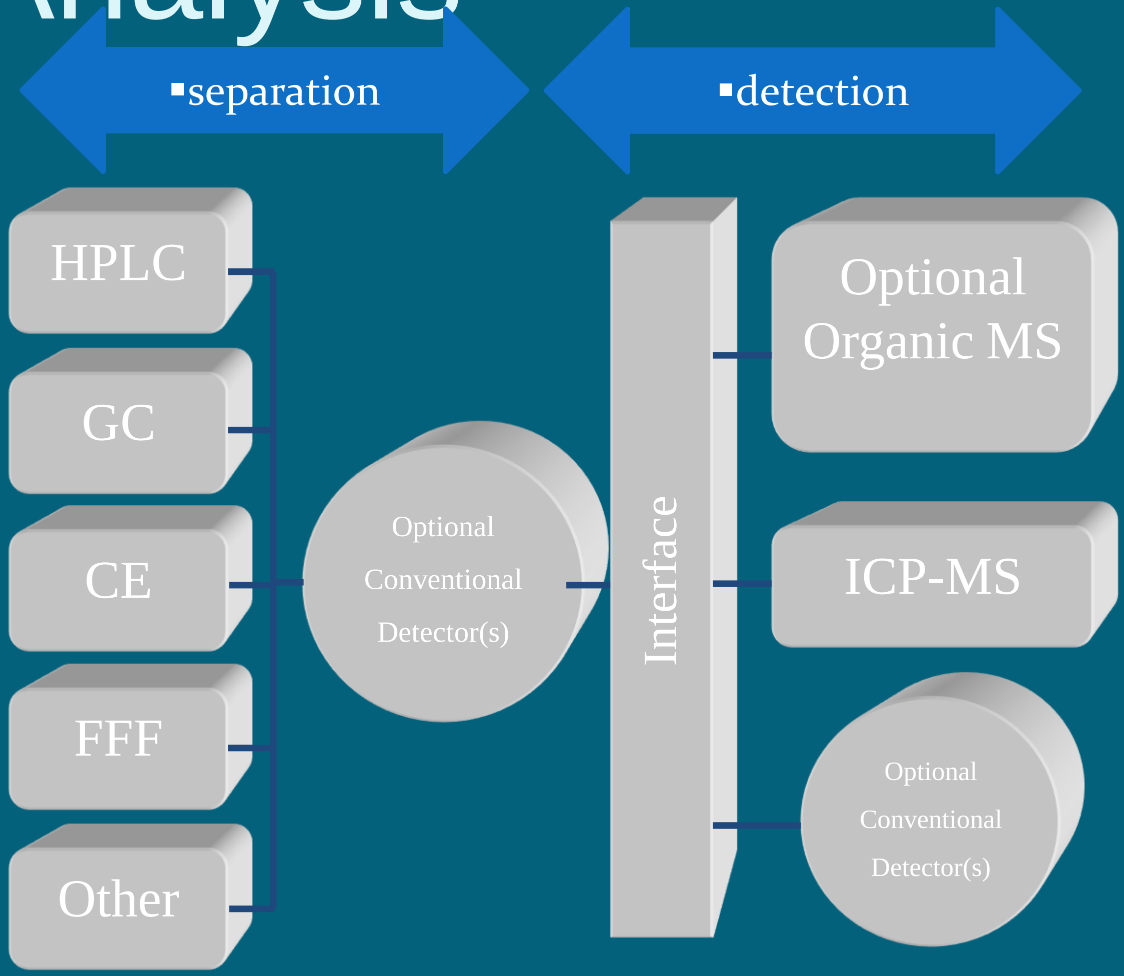
- Instrumentation
- Method Specific Parameters
  - For Arsenic (As) speciation

Characteristics of LC-ICP MS

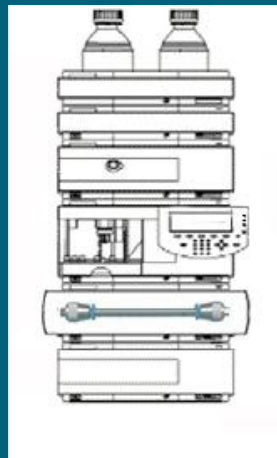
- Eluting peaks contains the monitored element with and no molecular information is directly obtained
- Peak identity is based on retention time matching with standards, i.e. identification is only as good as your standards
- Unknown peaks are common and can be extremely difficult to identify
- Mobile phases are limited, use only low organic (<15% or so)
- pH range of MP is limited to the plumbing of the LC system

Speciation Analysis

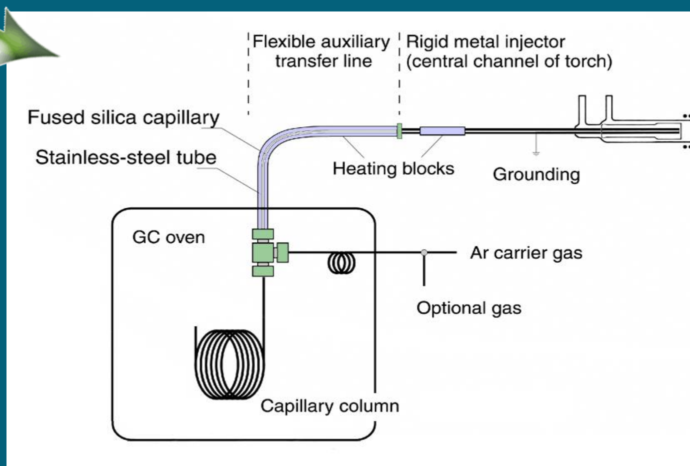
- Elemental speciation requires 2 steps
1. Separate the species
2. Identify and quantify the elemental composition of the species
- The separation is done chromatographically or electrophoretically.
- The elemental identification and quantification is performed using ICP-MS coupled to the chromatograph
- Most common techniques are GC-ICP-MS and HPLC-ICP-MS

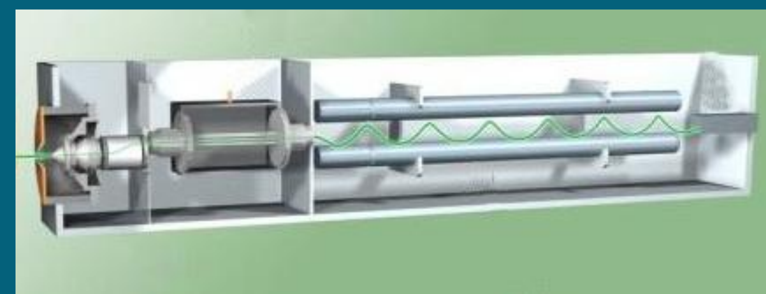


Coupling HPLC or GC to ICP-MS



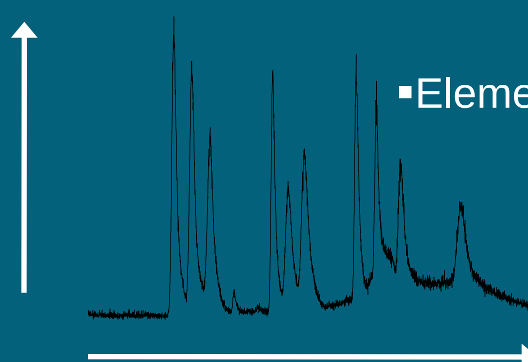
- **Sample**
- **OR**





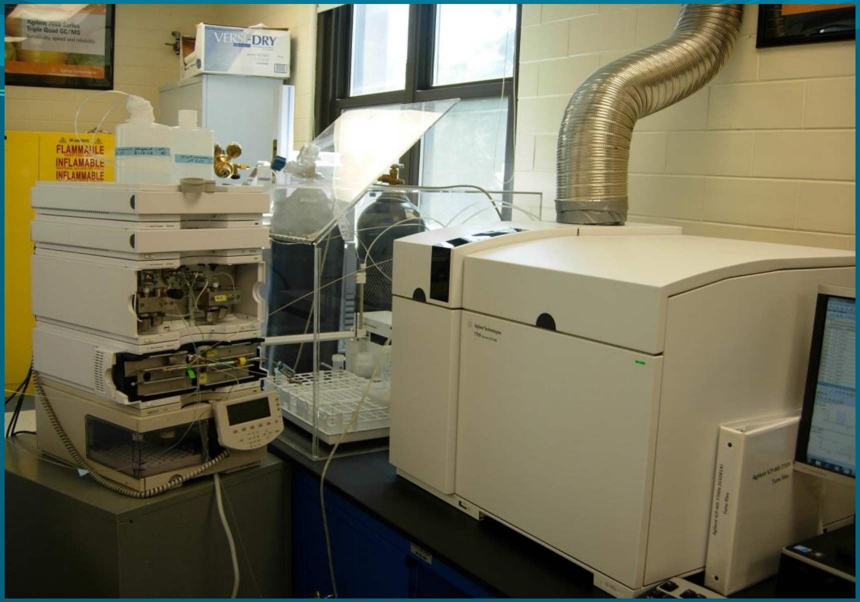
- ICP-MS for element specific detection

- Instrument Response

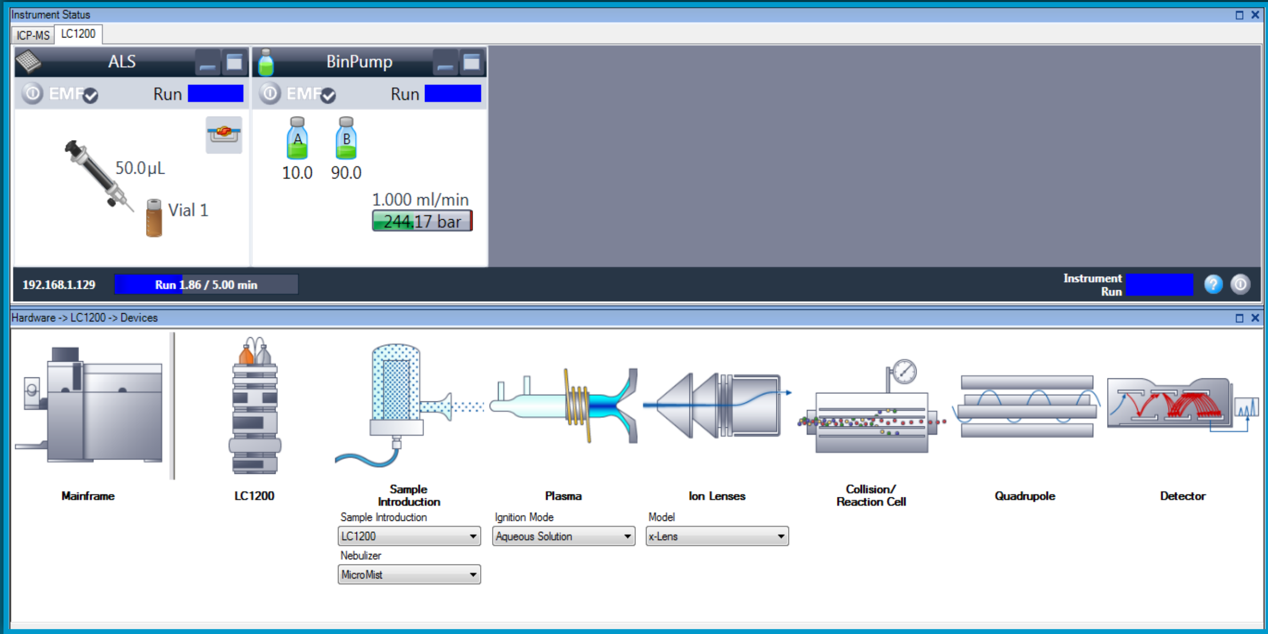


- Elemental Speciation
- Time

HPLC-ICP-MS



- Simple set-up
- ICP-MS MassHunter software controls LC and ICP-MS as a fully integrated analytical system
- Agilent LC connection kit provides complete hardware connectivity



ICP-MS MassHunter software screenshot showing details of the HPLC-ICP-MS configuration

ICP-MS as an elemental detector for speciation analysis

- Strengths:
- Extremely sensitive (ppt or better detection limits)
- Nearly universal elemental coverage
- Capable of isotope dilution quantification
- Compound independent response
- Excellent matrix tolerance
- Very wide linear dynamic range (9 orders of magnitude)

ICP-MS as an elemental detector for speciation analysis

- Limitations:
- No molecular (structural) information*
- Relatively expensive

- *Molecular identification needs to be established by another means:
  - Retention time matching
  - In parallel with molecular MS

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Method

- Extract sample w/ 0.28M HNO_3 @ 95°C for 90 minutes
- Dilute, centrifuge, filter, adjust pH
- Analyze by LC-ICP-MS
- Determine concentrations of AsIII and AsV *(AsIII + AsV = iAs)* as well as DMA and MMA.

Equipment

- ICP-MS, Agilent 7500ce or 7700
- HPLC, Agilent 1100, 1200, 1260 or 1290
  - A 6-port switching valve (integrated or external)
  - PRP X100 column, w/ guard column
- Tubes, bottles, syringes, other typical lab equipment
  - Block digestion system
  - pH meter
  - Centrifuge
  - Vortex mixer
  - *When in doubt acid wash appropriate supplies*
  - Grinder
  - Desiccators
  - Oven

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Reagents

- Arsenic standards
  - MMA, DMA, As(III), and As(V)
- All other reagents should be ultra high purity is possible, if not, then lack of detectable arsenic should be verified
- SRM 1568a, SRM 1568, CRM 7503-a, SRM 1643e

SRMs or CRMs

- SRM 1568 – f/ NIST - Rice Flour – replaced by 1568a, verified for Total As and other elements
- SRM 1568a – f/ NIST - Rice Flour –verified for Total As and other elements, currently in the process of verifying for As speciation
- CRM 7503-a – f/ NMIJ (National Metrology Institute of Japan) – White Rice Flour - verified for total As and As species.

SRM/CRM	Total As (µg/kg)	AsIII (µg/kg)	AsV (µg/kg)	iAs (µg/kg)	DMA (µg/kg)	MMA (µg/kg)
1568a	290 ± 30*	60 ± 12**	39 ± 8**	100 ± 20**	171 ± 34**	11 ± 2**
1568	410 ± 50*	85 ± 17**	31 ± 6**	116 ± 23**	285 ± 57**	22 ± 4**
7503-a	98 ± 7*	71.1 ± 2.9*	13.0 ± 0.9*	84 ± 17**	13.3 ± 0.9*	nr

- *Certified Value with Uncertainty expressed as a 95% Confidence Interval or 95% Confidence Interval plus an allowance for systematic error.
- **Uncertainty expressed as ± 20% of the average value from the best available data, nr = not reported

•SRM 1643e – f/ NIST – Trace Elements in Water, verified for Total As (58.98±0.7ng/g) and other elements.

Sample Preparation

- Sample Extraction:
  - Get the analytes out of the solid sample into a liquid phase
  - Need to preserve the species integrity
  - Extract with high efficiency
  - 0.28M HNO_3 and heat will do this
    - Validated by Huang et. al.

- Huang, J.-H., Ilgen, G., and Fecher, P., *J. Anal. At. Spectrom.* (2010) 25, 800-802.

ICP-MS conditions

ICP-MS Conditions	
RF power	1500 W
Plasma gas flow	15 L/min
Auxilliary (makeup) gas flow	0.15 L/min
Nebulizer (carrier) gas flow	0.95 L/min
Nebulizer type	Glass concentric
Sampling depth	8.0 mm
Peristaltic pump speed	0.4 rps
Spray chamber temp.	2°C
Collision cell	He @ 2.0 mL/min (dependent on instrument sensitivity)
Data acquisition mode	Time-resolved, m/z 75 for $^{75}As^+$, m/z and m/z 77 for $^{40}Ar^{37}Cl^+$
Dwell time	0.8 s (m/z 75), 0.2 s (m/z 77)

LC conditions

HPLC Conditions - Agilent 1200	
Column	PRP-X100, 4.1x20 mm, stainless steel, 10µm particle size
Mobile phase composition	10 mM $(NH_4)_2HPO_4$, pH 8.25, isocratic
Mobile phase flow rate	1 mL/min
Injection volume	100 µL
Acquisition time	1200 s (20 min)

Analytes of interest

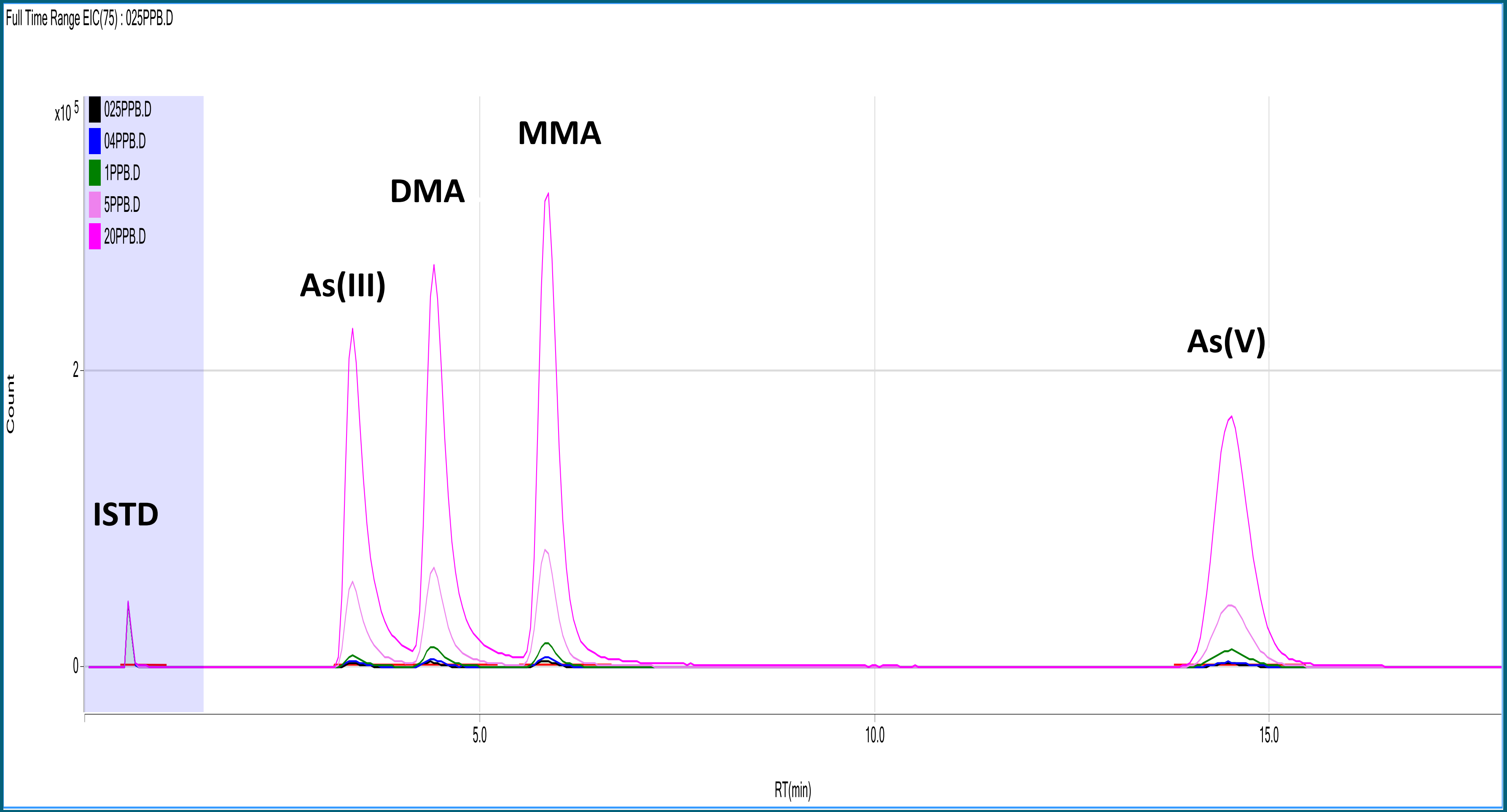
As Species	Formula	pK_a[1]	Charge @ pH 8.25
As(III)	O=As(OH)	9.3	~ 0
Dimethylarsenic acid (DMA)	$(CH_3)_2AsO(OH)$	6.2	~ -1
Monomethylarsonic acid (MMA)	$CH_3AsO(OH)_2$	2.6, 8.2	~ -1.5
As(V)	$O{=}As(OH)_3$	2.3, 6.9, 11.4	~ -2

- [1] *F. Kitagawa, K. Shiomi, K. Otsuka, Electrophoresis 27 (2006) 2233*

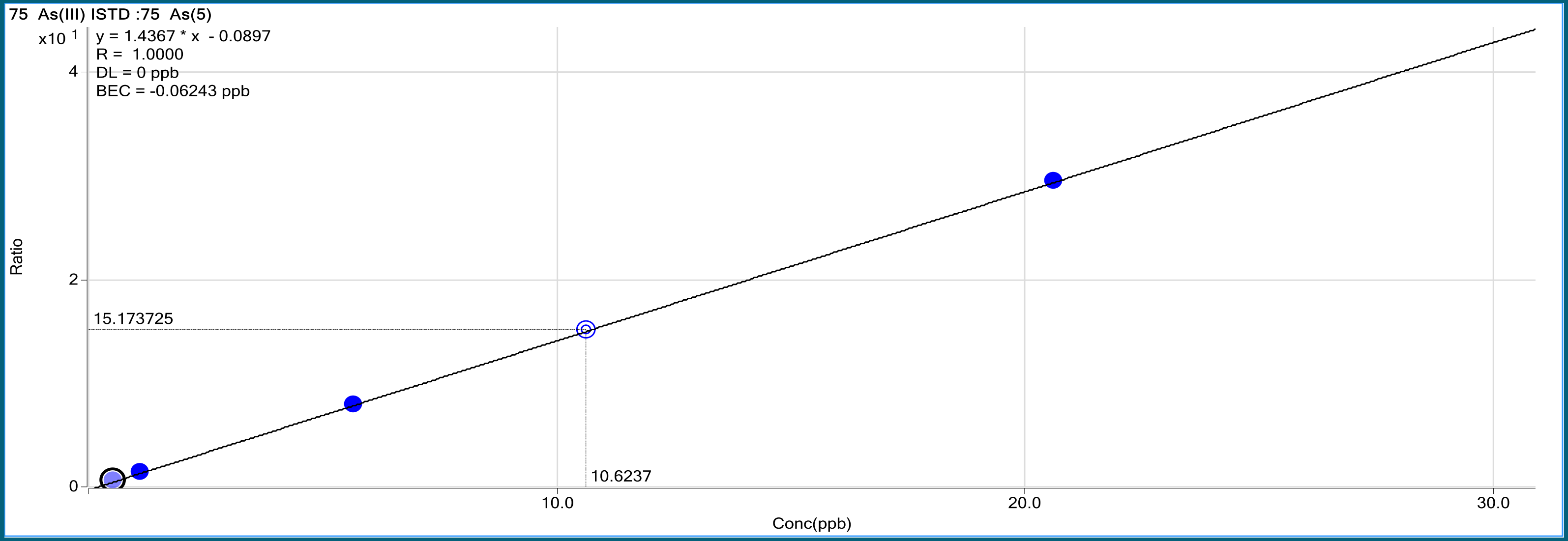
Instrument Calibration Data for As standards

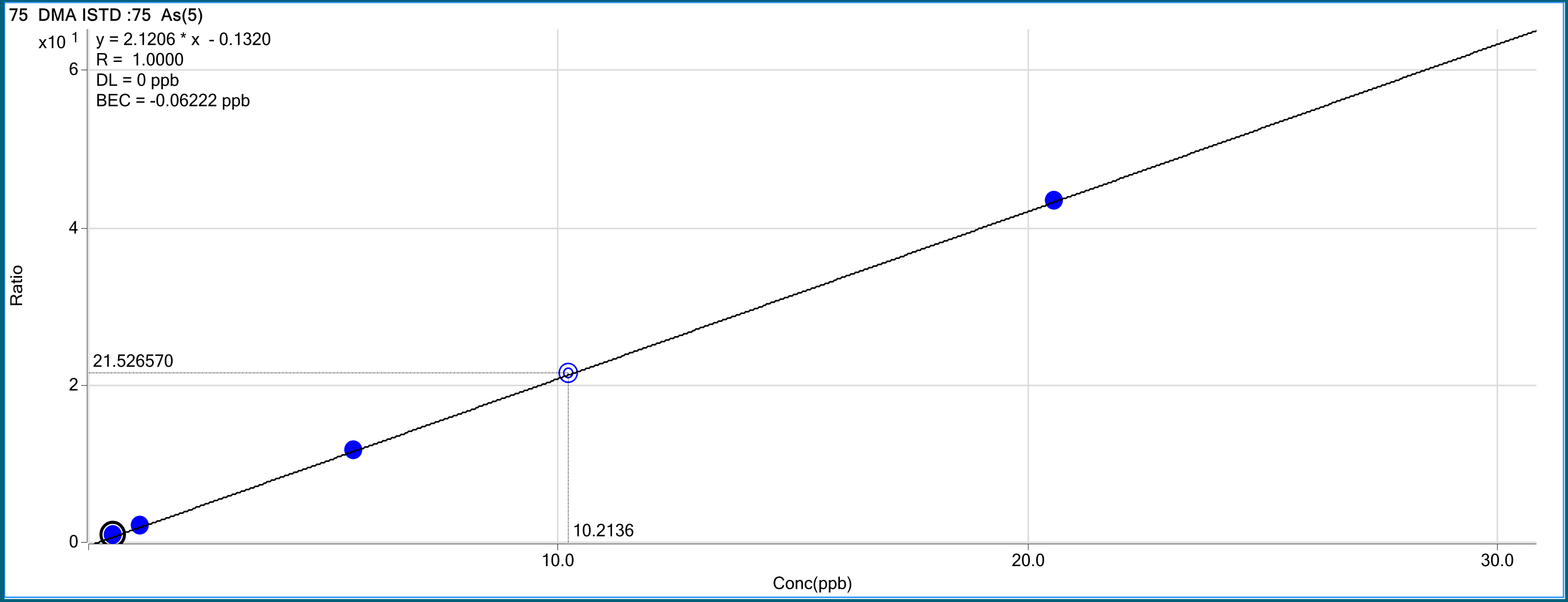
- Calibration standards were prepared in the range of 0.25 ng/g to 20 µg/g .
- All calibration curves were linear over the entire concentration range ($R^2 = 1.00$). Calibration check (10 ng/g of each: AsIII, DMA, MMA, and AsV) percent recovery was 96 -104% with % RSD < 3%.
- Standard Reference Materials (NIST 1568 rice flour and CRM 7503-a from NMIJ) were used as a quality control for both - total As and iAs.
- The LOD values for As species ranged from 0.9 ng/g to 1.7 ng/g, and LOQ 7 µg/kg to 14 µg/kg .
- % RSD for representative sample analyzed in replicate (n=3) was < 4%.

Overlaid chromatograms for As calibration standards:
0.25, 0.4, 1.0, 5.0, and 20 ppb As

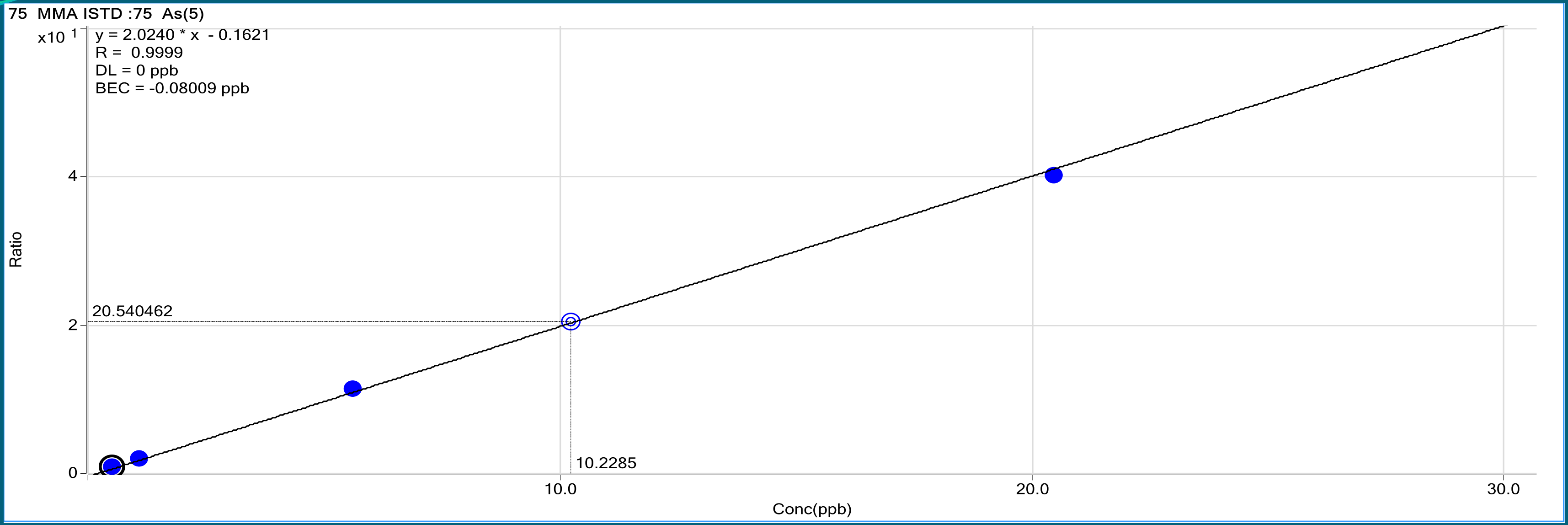


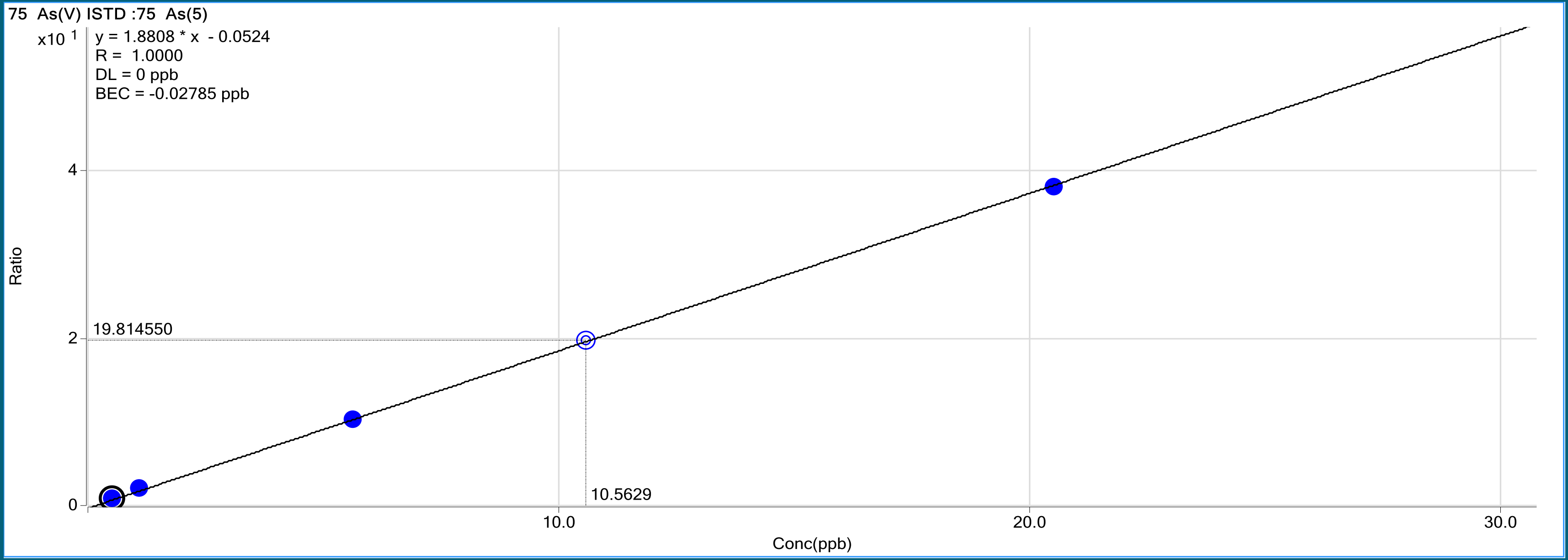
Calibration curves for As(III) & DMA standards





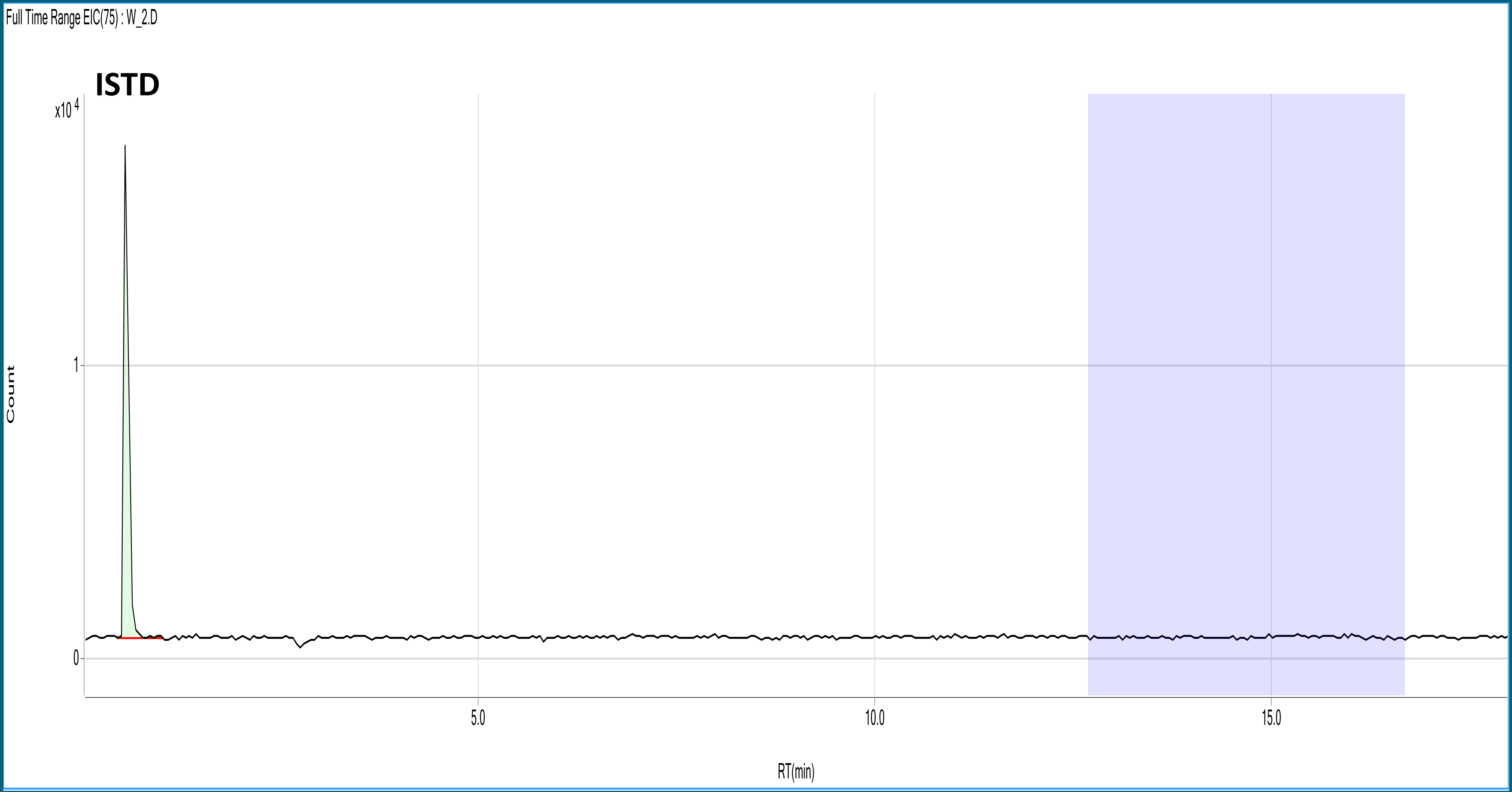
Calibration curves for MMA & As(V) standards



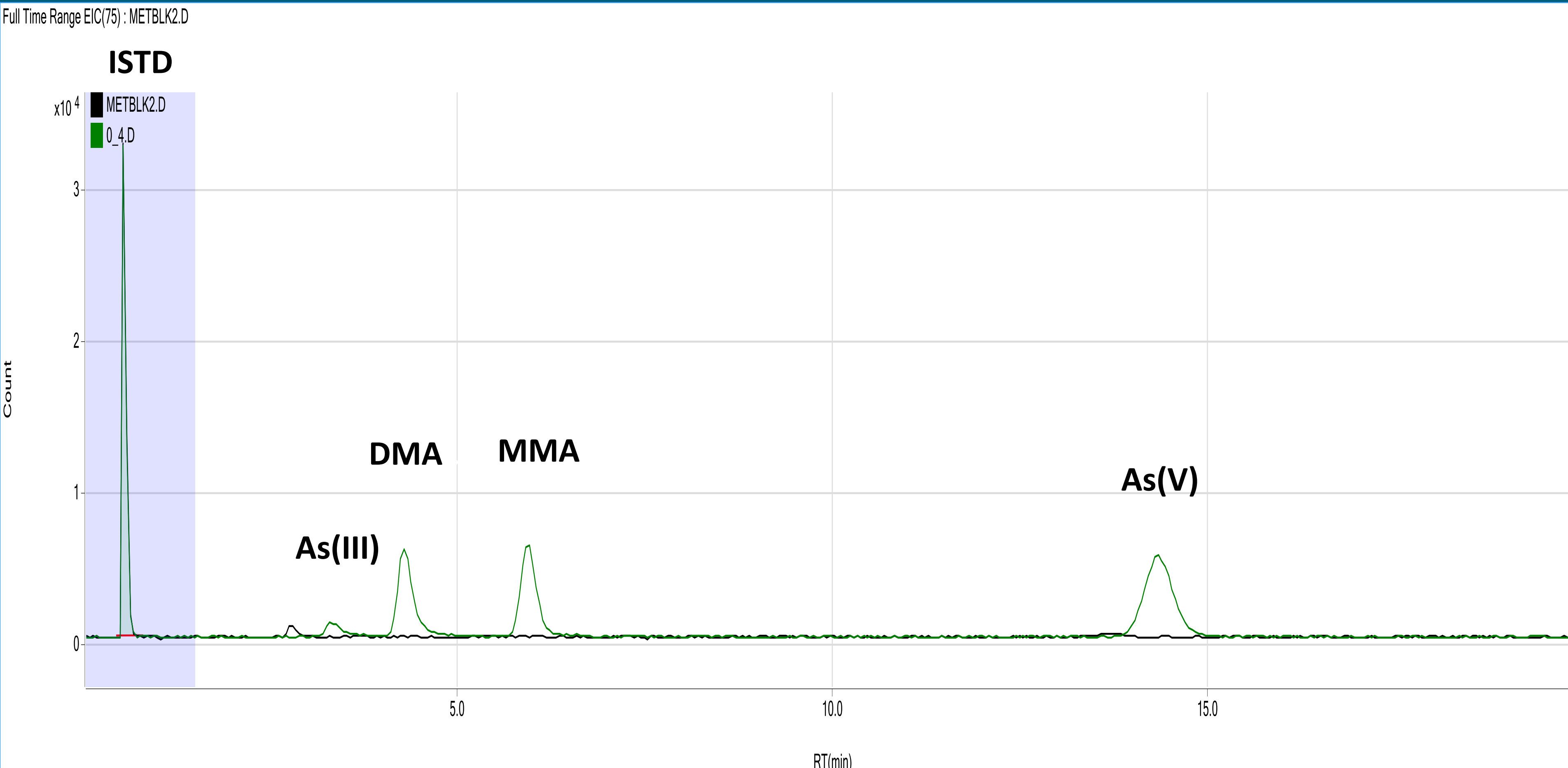


De-ionized water

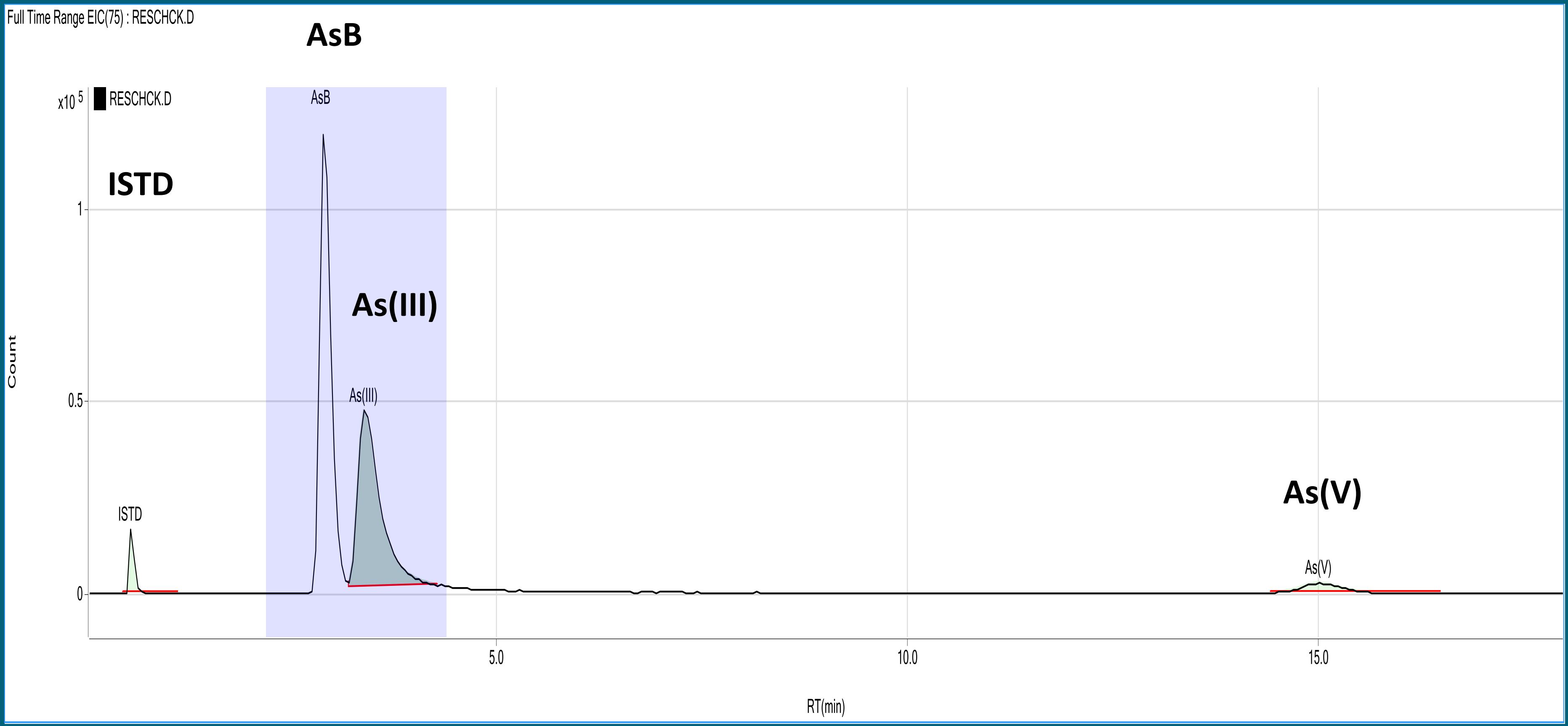
- System check



Method Blank and 0.4 ppb STD



Resolution check standard containing 5ng/g As(III) and AsB



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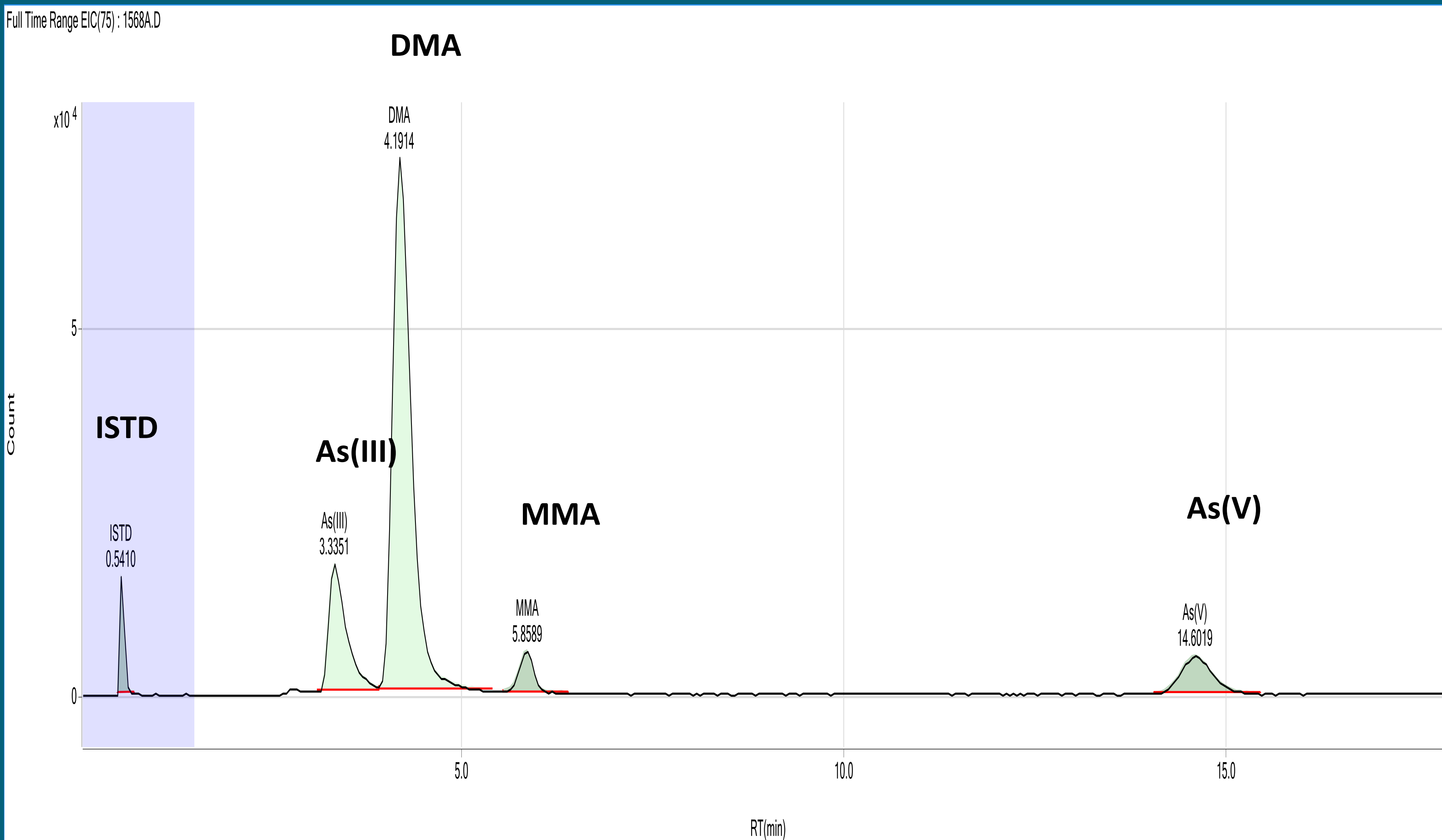
LOD/LOQ for As Species in Rice

Species	RT (min)	Peak area	ASDL (ng/g)	ASQL (ng/g)	Method LOD µg/kg	Method LOQ µg/kg
75 As(III)	3.6	48162	0.03	0.26	1.7	13
75 DMA	4.4	69264	0.02	0.14	0.9	7
75 MMA	6.1	71551	0.02	0.18	1.2	9
75 As(V)	14.1	84219	0.04	0.27	1.8	14

SRM 1568a Rice Flour diluted extract



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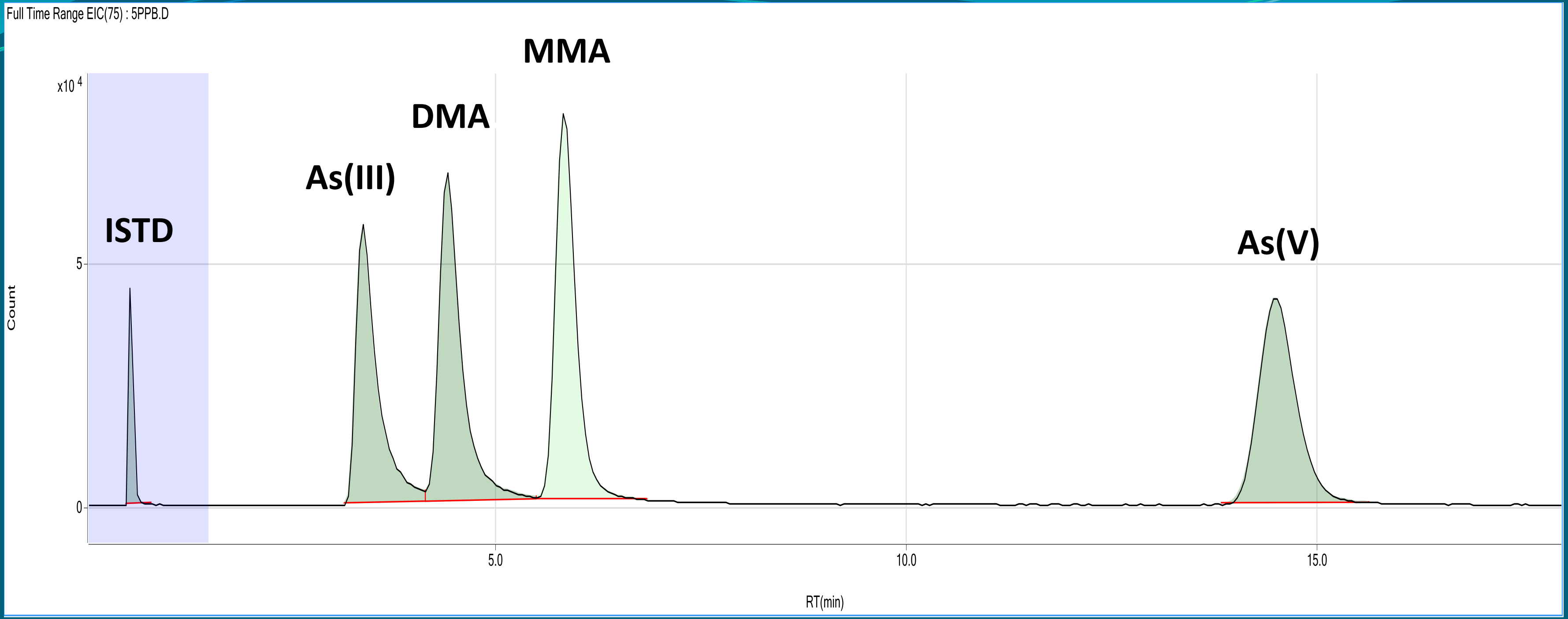
Example Chromatogram

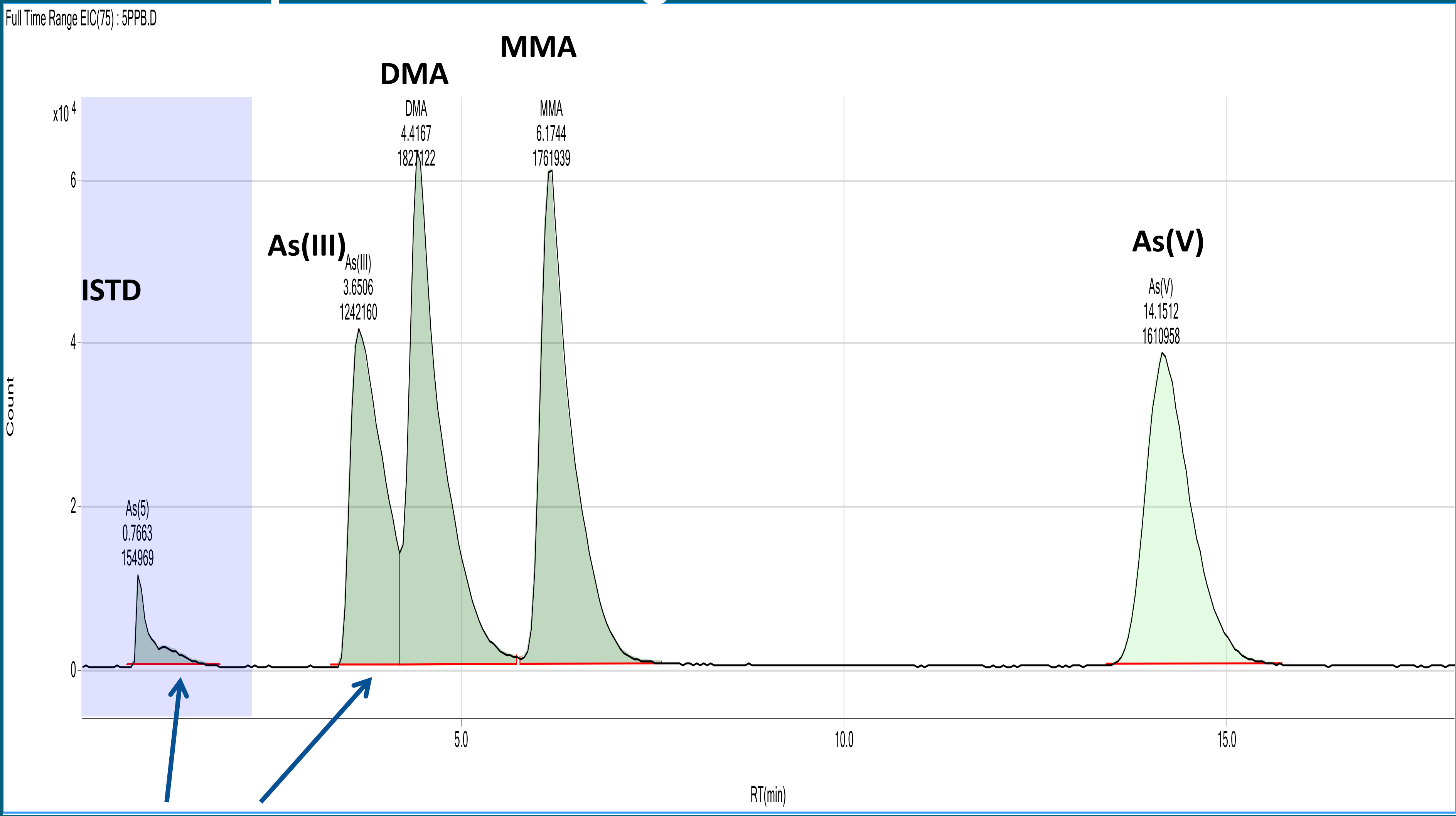


Chromatographic separation of arsenic standards at 5 ng/g.

- **Things that can affect the chromatography**…
- Composition of mobile phase – pH, buffer strength, ionic strength, etc
- Composition of sample - pH, ionic strength, other components
- Column length, pore size, packing material
- Column age (column contamination, loss of active sites)
- HPLC-ICP-MS Interface peek tubing diameter
- Flow Rate

Things that can affect the chromatography

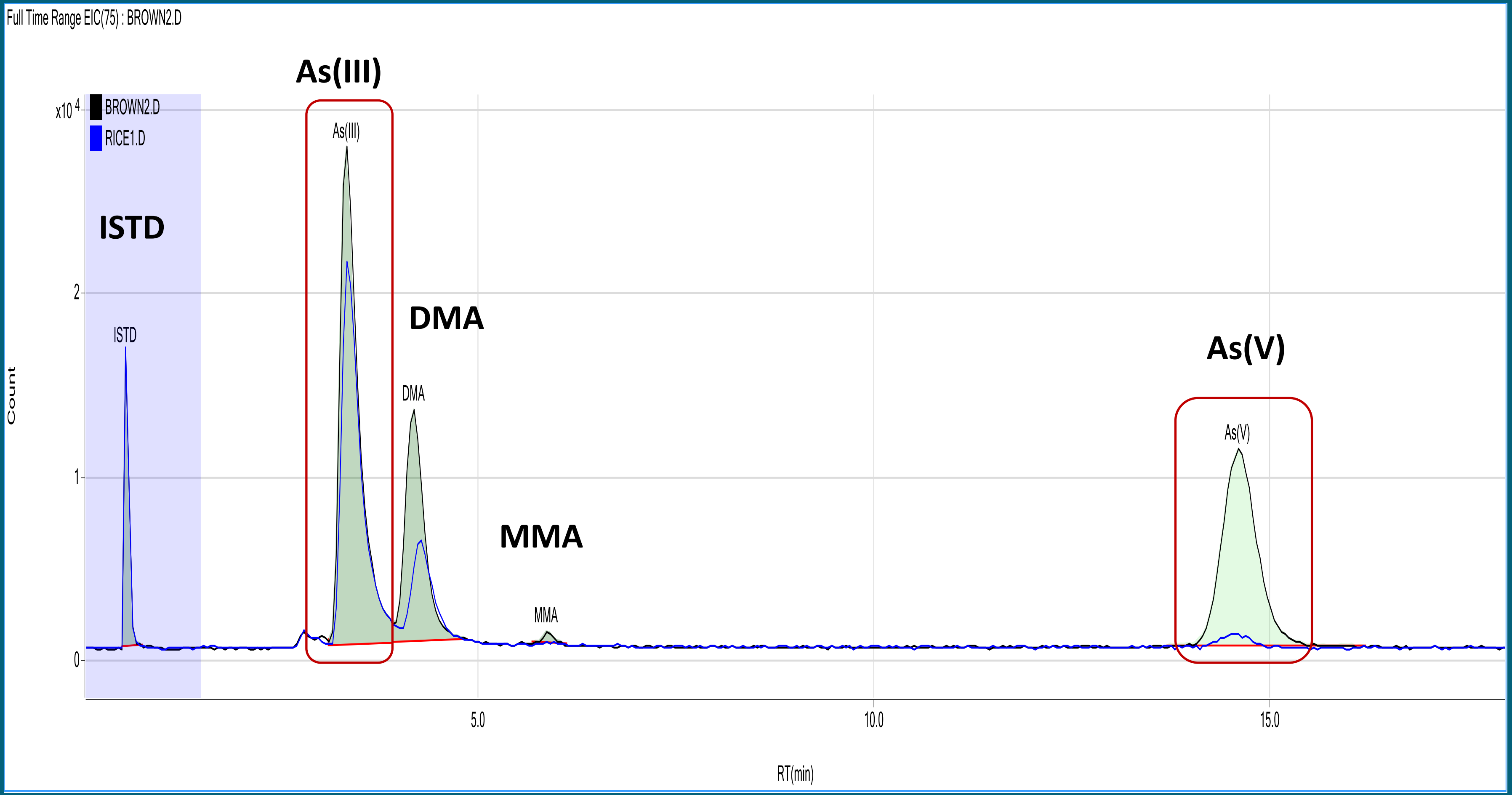
Example Chromatogram



Peek tubing diameter is too large (it should be 1/16 x 0.02 x 6 in; O.D x I.D. x length)

Arsenic in Rice: Example Data

Brown Rice and White Rice flour comparison



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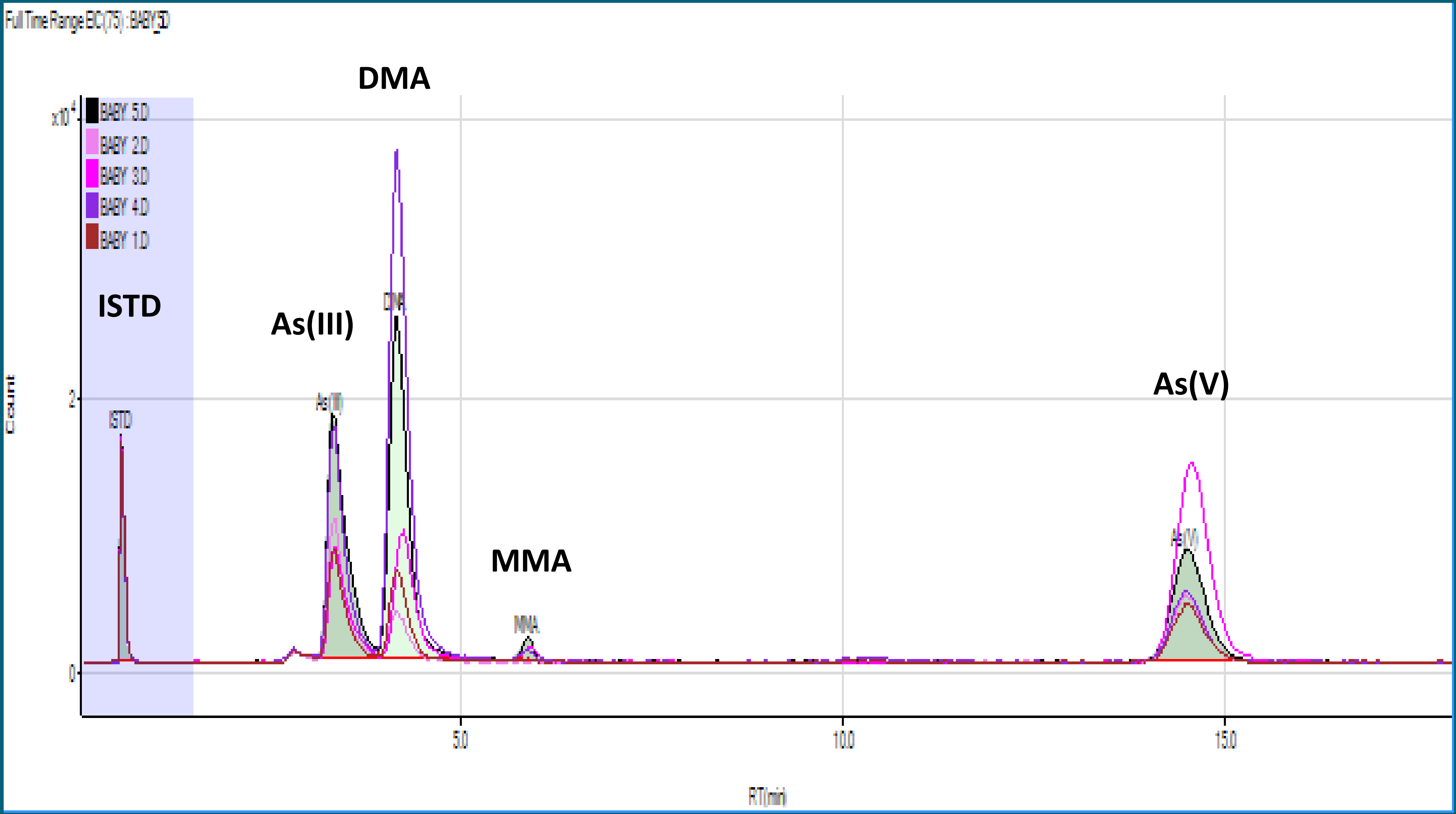
Example Chromatogram



A representative chromatogram of arsenic speciation in infant rice cereal.

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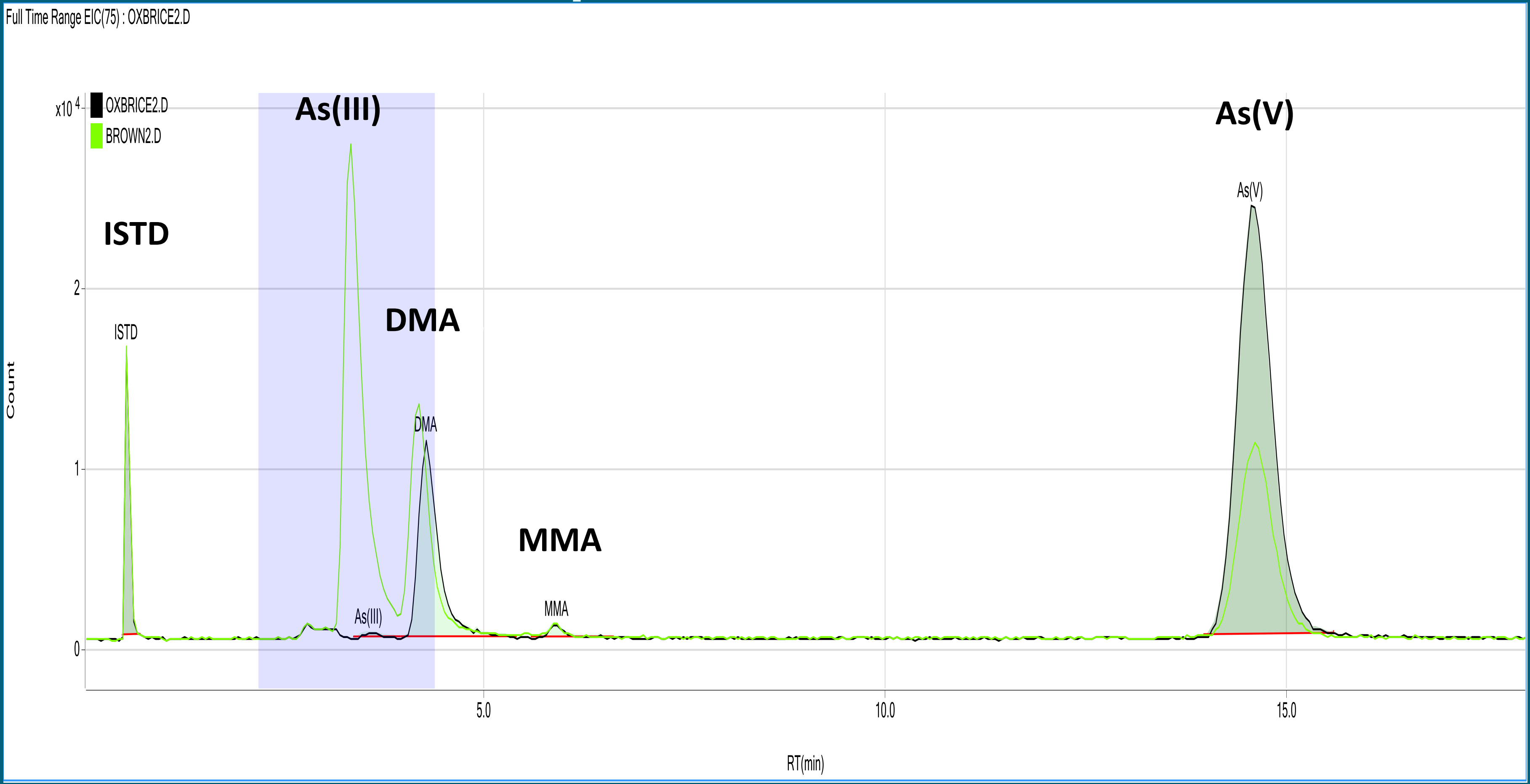
Brown rice flour sample treated with H_2O_2

- To investigate possible interferences on the AsIII determination, sample extract was oxidized to AsV



iAs in Brown rice cereal – 202ng/g and in treated sample – 165 ng/g

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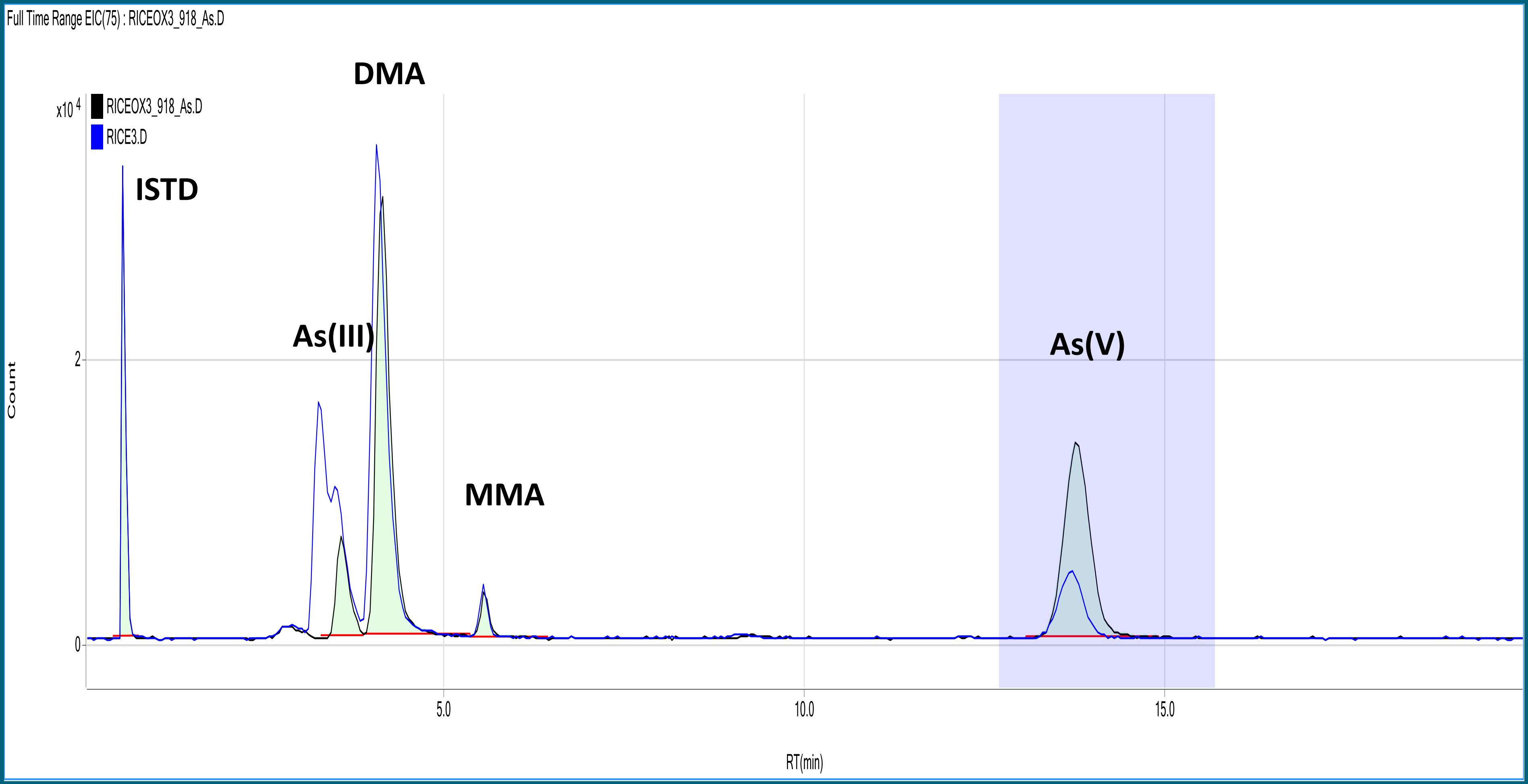
Rice flour sample treated with H_2O_2

- To investigate possible interferences on the AsIII determination, sample extract was oxidized to AsV



Arsenic Speciation in Rice Flour and Rice Cereal for Infants Using High Performance Liquid Chromatography-Inductively Coupled Plasma-Mass Spectrometric Determination

Speciated and total arsenic was determined in *25 infant rice cereals* (seven different brands) from various US supermarkets and *14 rice flours* from four different countries.

Arsenics levels in white and brown rice flour were compared .

The mixed grain rice cereal contained the least total (81 ng/g) and inorganic arsenics (51 ng/g).

Levels of inorganic arsenics (iAs) greatly varied among all rice cereals. There was no statistical difference [0.051 = P(0.05)] in iAs levels between organic and non-organic rice cereals (an average of 96 ng/g in organic white rice cereal versus 81 ng/g in non-organic white rice cereal).

Concentration of As (ng/g) in infant rice

		75 As(III)	75 Unk	75 DMA	75 MMA	75 As(V)	iAs	Sum As
Sample name	Brand	Conc. [ng/g]	Estimated Conc. [ng/g]	Conc. [ng/g]	Conc. [ng/g]	Conc. [ng/g]	Conc. [ng/g]	Sum As Species (ng/g) (AsIII + AsV + DMA +MMA+ UNK)
Organic, rice cereal for babies_4	C	73.56	NA	145.4	TR (2.6)	32.82	106.4	254.4
Organic, rice cereal for babies_6	C	49.8	NA	42.02	ND	36.62	86.41	129.4
Organic, whole grain Infant rice cereal _3	B	38.72	NA	40.16	TR (3.0)	0	38.72	81.92
Organic brown Infant rice cereal _2	A	44.48	NA	14.94	ND	30.06	74.54	89.77
Organic brown Infant rice cereal _9	A	31.74	NA	21.45	ND	23.51	55.25	76.71
Organic brown Infant rice cereal _10	B	ND*	13.51	162.1	TR (4.2)	37.97	37.97	204.4
Organic brown Infant rice cereal _11	B	48.81	NA	35.4	TR (2.0)	76.3	125.1	162.5
Organic brown Infant rice cereal _12	A	43.84	NA	78.1	ND	33.95	77.79	156.6
Organic brown Infant rice cereal _24	G	56.61	NA	24.29	ND	37.46	94.07	119.3
Oganic brown Infant rice cereal _25	G	58.99	NA	24.72	ND	41.6	100.6	126.2

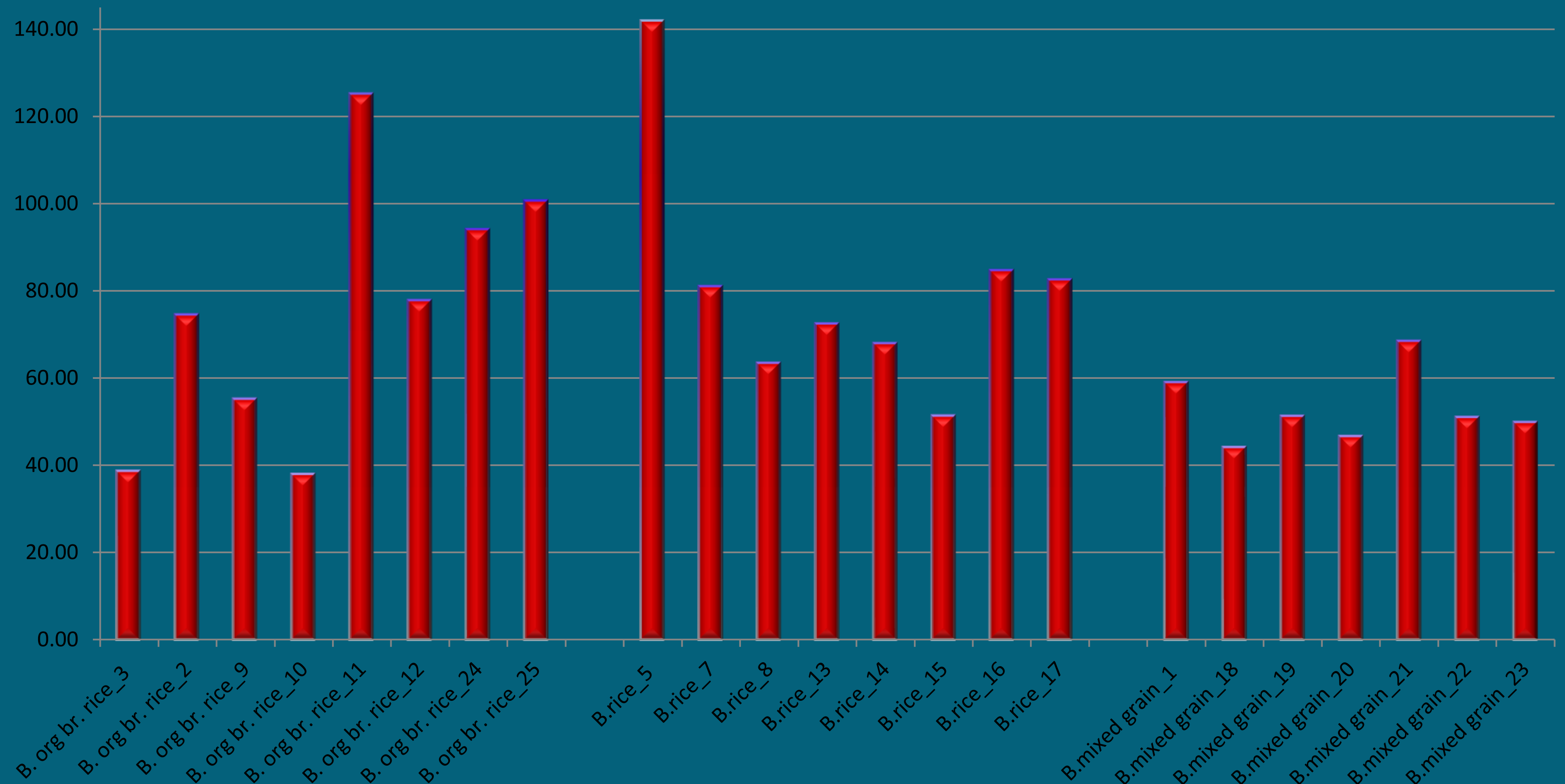
Note: 1) ND - samples were oxidized with H2O2.*
2) Seven different brands of baby cereal were analyzed (A-H).

Concentration of As (ng/g) in infant rice cereal (cont.)

		75 As(III)	75 Unk	75 DMA	75 MMA	75 As(V)	iAs	Sum As
Sample name	Brand	Conc. [ng/g]	Estimated Conc. [ng/g]	Conc. [ng/g]	Conc. [ng/g]	Conc. [ng/g]	Conc. [ng/g]	Sum As Species (ng/g) (AsIII + AsV + DMA +MMA+ UNK)
Rice single grain Infant cereal _8	E	ND*	7.611	71.8	TR (1.7)	63.44	63.44	137
Rice single grain Infant cereal _13	F	42.98	NA	32.36	ND	29.5	72.49	105.2
Rice single grain Infant cereal _14	F	40.49	NA	27.82	ND	27.47	67.96	96.02
Rice single grain Infant cereal _15	F	27.4	NA	24.54	ND	23.99	51.39	76.18
Rice single grain Infant cereal _16	A	ND*	8.507	84.17	TR (2.3)	84.66	84.66	171.1
Rice single grain Infant cereal _17	F	41.5	NA	30.41	ND	41.05	82.55	113.8
Mixed Grain Infant cereal _1	A	33.42	NA	24.37	ND	25.62	59.04	83.7
Mixed Grain Infant cereal _18	A	ND*	3.355	26.02	ND	44.16	44.16	70.69
Mixed Grain Infant cereal _19	A	18.5	NA	20.16	ND	32.82	51.32	71.66
Mixed Grain Infant cereal _20	A	ND*	3.419	39.81	ND	46.69	46.69	87.64
Mixed Grain Infant cereal _21	A	20.49	NA	43.89	ND	47.99	68.47	113.5
Mixed Grain Infant cereal _22	A	ND*	3.347	38.96	ND	51.06	51.06	90.9
Mixed Grain Infant cereal _23	A	ND*	2.073	37.89	ND	49.91	49.91	88.88

Note: 1) ND - samples were oxidized with H2O2.*
2) Seven different brands of baby cereal were analyzed (A-H).

iAs Concentration (ng/g) in baby rice cereal



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Arsenic Speciation in Infants Rice Cereal (cont.)

In twenty five baby rice cereals, the total arsenic concentration ranged from 70 to 254 ng/g.

The iAs as a percent of total arsenic ranged from 19 to 80%.

The major detected organic arsenic specie was DMA. MMA was not detected or only trace levels were found.

The LOD values for As species ranged from 0.9 ng/g to 1.7 ng/g, and LOQ 7 µg/kg to 14 µg/kg.

% RSD for representative sample analyzed in replicate (n=3) was < 4%. Spiked sample iAs % recovery at three levels (50, 100 and 150 ng/g) were 110% ± 5.7, 96% ± 4.9 and 104% ± 9.9, respectively.

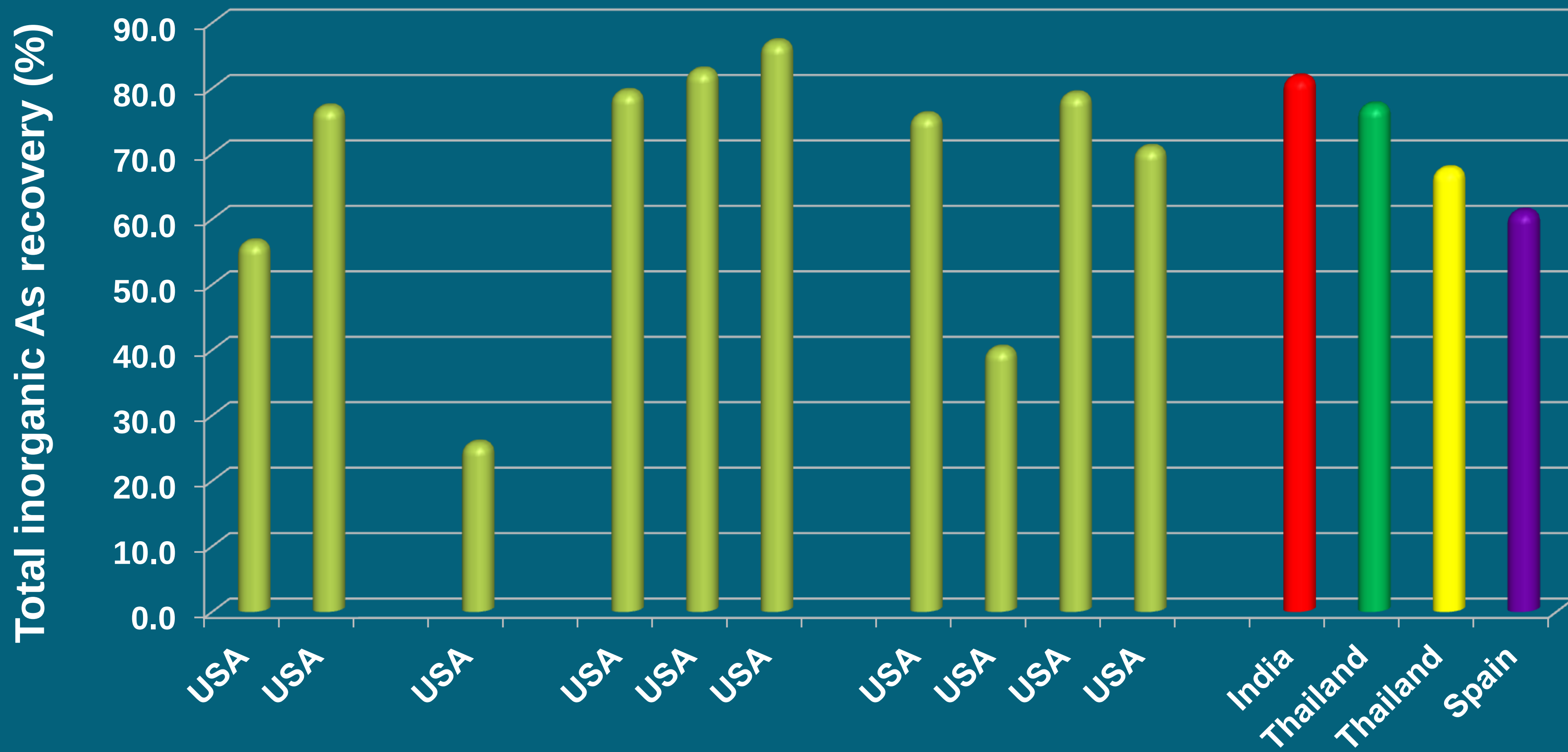
Arsenic Speciation in Rice Flour

Fourteen rice flour samples were obtained from local stores. There were ten from the USA (white and brown rice, both organic and non-organic), and four white rice flour samples from Thailand, India, and Spain.

Quantitative Results for As Species in Rice flour

Sample name	Origin	iAs conc. [ng/g]	Sum As Species (ng/g) (AsIII + AsV + DMA +MMA+ UNK)	Total iAs recovery (%)
Organic Brown rice flour-3	USA	160.75	284.92	56.4
Organic Brown Rice flour-10	USA	140.22	182.01	77.0
Organic White Rice flour-4	USA	65.57	255.49	25.7
Brown Rice flour-1	USA	200.07	251.98	79.4
Brown Rice flour-12	USA	172.39	208.49	82.7
Brown Rice flour-14	USA	186.05	213.84	87.0
White Rice flour-2	USA	99.17	130.76	75.8
White Rice flour-5	USA	65.51	163.10	40.2
White Rice flour-11	USA	113.67	143.82	79.0
White Rice flour-13	USA	120.28	169.75	70.9
White Rice flour-6	India	81.26	99.57	81.6
White Rice flour-7	Thailand	113.50	146.73	77.4
White Rice flour-8	Thailand	53.04	78.47	67.6
White Rice flour-9	Spain	182.58	298.78	61.1

Percentage inorganic rice in Market rice



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Arsenic Speciation in Rice Flour (cont.)

In fourteen rice flours, the total arsenic concentration ranged from 78 to 299 ng/g and iAs ranged from 53-200ng/g.

The iAs as a percent of total arsenic ranged from 26% to 87%.

The major detected organic arsenic specie was DMA. MMA was not detected or only trace levels were found.

The highest concentration of total As was in rice flour sample from Spain (299ng/g)

% RSD for representative samples analyzed in replicate (n=3) was < 3%.
Spiked sample iAs % recovery at three levels (50, 100 and 150 ng/g) were 116% ± 0.0, 88% ± 1.3 and 116% ± 0.2, respectively.
Spiked sample iAs % recovery at three levels (75, 150 and 225 ng/g) were 118% ± 1.8, 117% ± 4.1 and 115% ± 4.2, respectively.

Conclusion

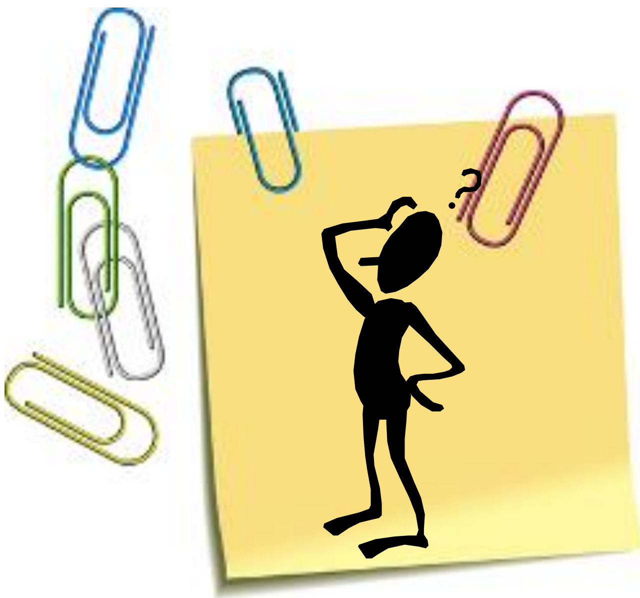
- **This study determined levels of different arsenic species in infant cereals and rice flour.**

- **These results for iAs in US rice cereal did not exceed the Chinese maximum allowed concentration of 150 ng/g iAs in rice. Brown rice flour did exceed the maximum allowed concentration.**

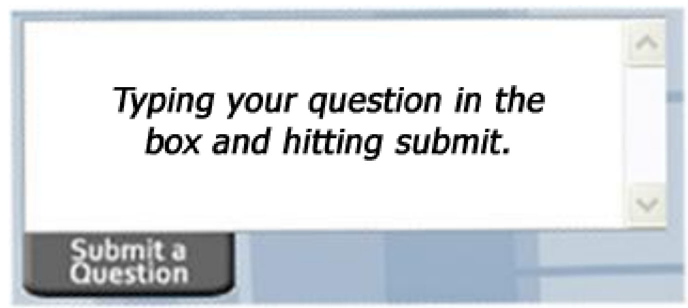
- **Speciation of arsenic in rice products is necessary because of the growing consumer and regulatory concern and the lack of information or regulation on arsenic levels in foods.**

- **This study provides new and much needed information on arsenic levels in rice and rice based infant cereals.**

Questions?



Please Submit your questions by:



Thank You!

We apologize for this brief interruption but we are experiencing technical difficulties and will resume shortly.