

18th Meeting of the Directors of NMIs and Member States Representatives
at the BIPM on 25th October 2016

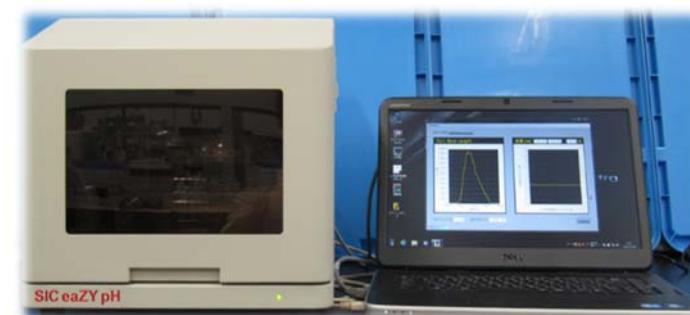
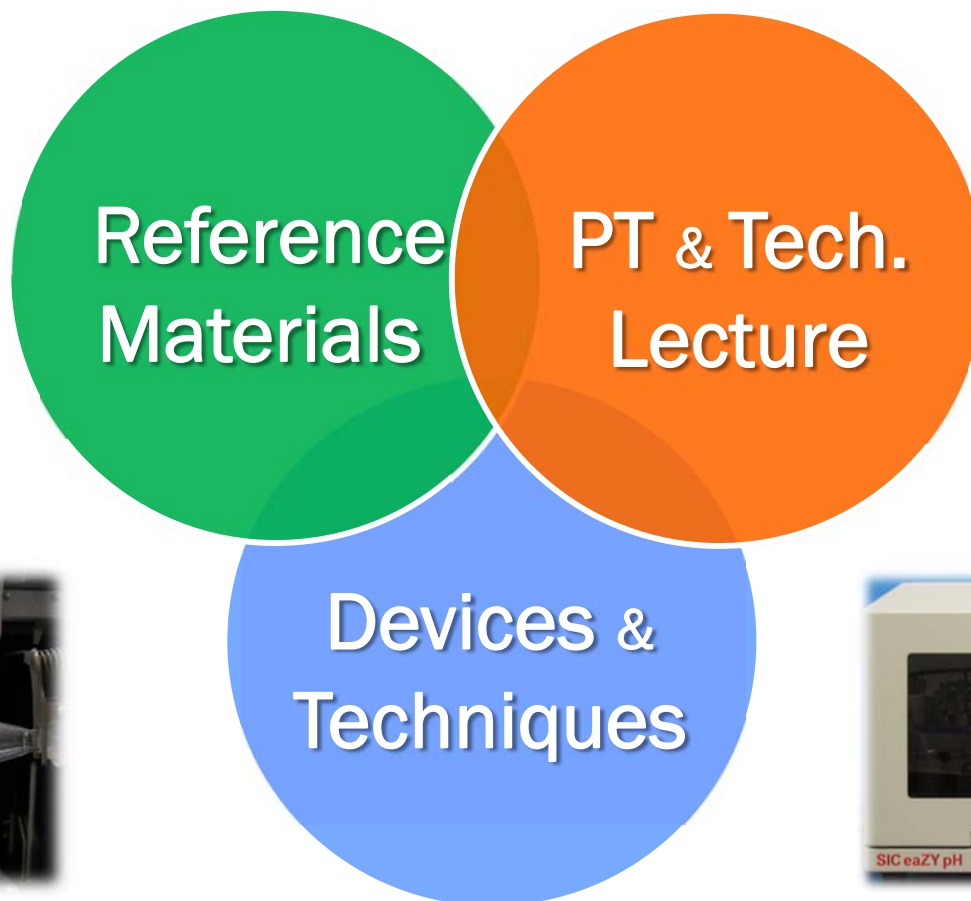
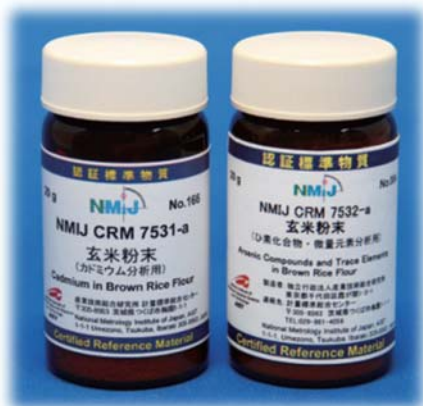
Trace Element Analysis in Food

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Supporting for Ensuring Reliability in Food Analysis

NMIJ/AIST provides
 “Tools” and “Knowledge”



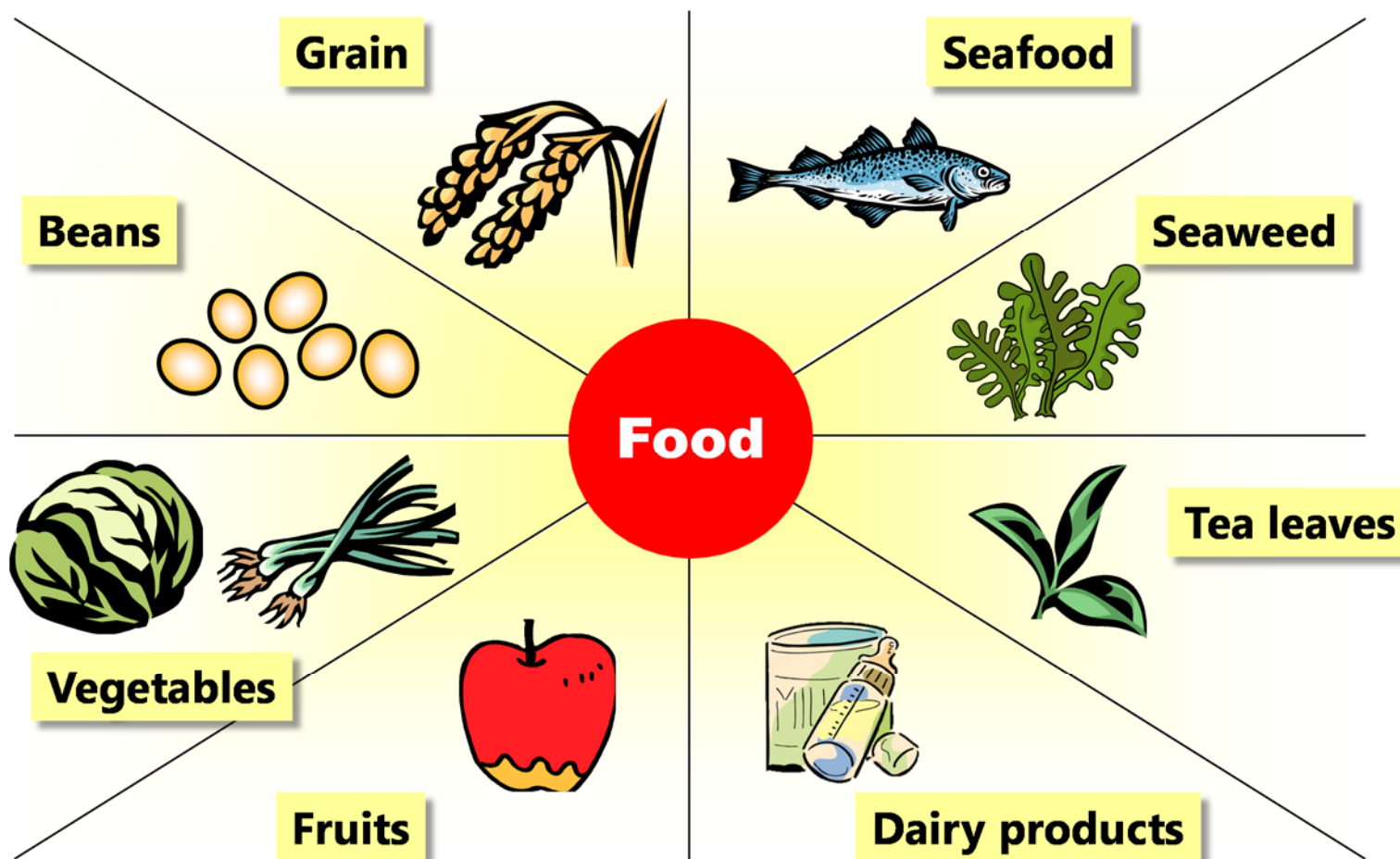
Certified Reference Material (CRM) for chemical analysis

“Measuring Scale” & “Self-checking Tool”
in Chemical Analysis



NMIJ's Matrix Type CRMs for Food Analysis

- Trace elements including toxic metals and their chemical species
- Organic pollutants such as PCBs, Dioxin
- Pesticide residues



Matrix-type CRM / Food for Inorganic Analysis

7402-a	Cod fish Tissue (13 Elements, AsB, Methyl-Hg)	
7403-a	Swordfish Tissue (14 Elements, AsB, Methyl-Hg)	← CCQM-K43.1
7405-a	Seaweed (Hijiki) (18 Elements, Arsenate)	
7501-a	White Rice Flour: Cd Level I (11 Elements)	
7502-a	White Rice Flour: Cd Level II (18 Elements)	
7503-a	White Rice Flour (6 Elements, As(III)+As(V), DMAA)	
7505-a	Tea Leaf Powder (18 Elements)	← PT (Japan)
7511-a	Soybean (8 Elements)	
7512-a	Milk Powder (13 Elements)	
7531-a	Brown Rice Flour (6 Elements)	← PT (Japan)
7532-a	Brown Rice Flour (8 Elements, Inorg. As, DMAA)	← KC + PT (Japan/Mexico)
7533-a	Brown Rice Flour (8 Elements, Inorg. As, DMAA)	← KC + PT (Japan/Thailand)

Matrix-type CRM / Food for Organic Analysis

4214-a	Dogfish liver oil (OCPs)
7504-a	Unpolished rice (Pesticide residues)
7404-a	Japanese sea bass tissue (PCBs, OCPs)
7507-a	Green onion (Pesticide residues)
7508-a	Cabbage (Pesticide residues)
7510-a	Apple (Pesticide residues)
7509-a	Soybean (Pesticide residues)

Game changer for calibration: quantitative NMR

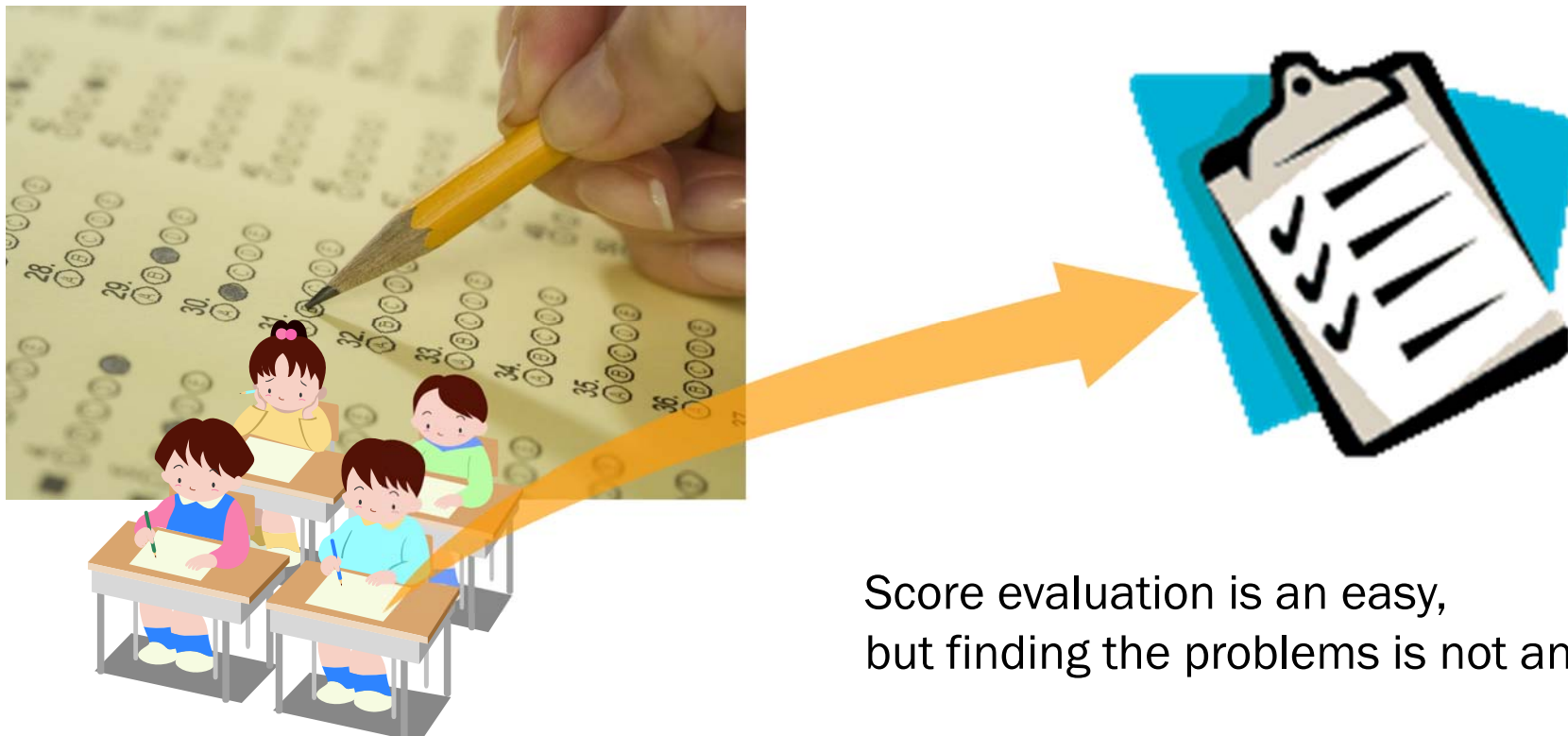


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Usual Proficiency Testing: for External Quality Assessment

Reported values are evaluated by comparing with an assigned value such as z-score evaluation.

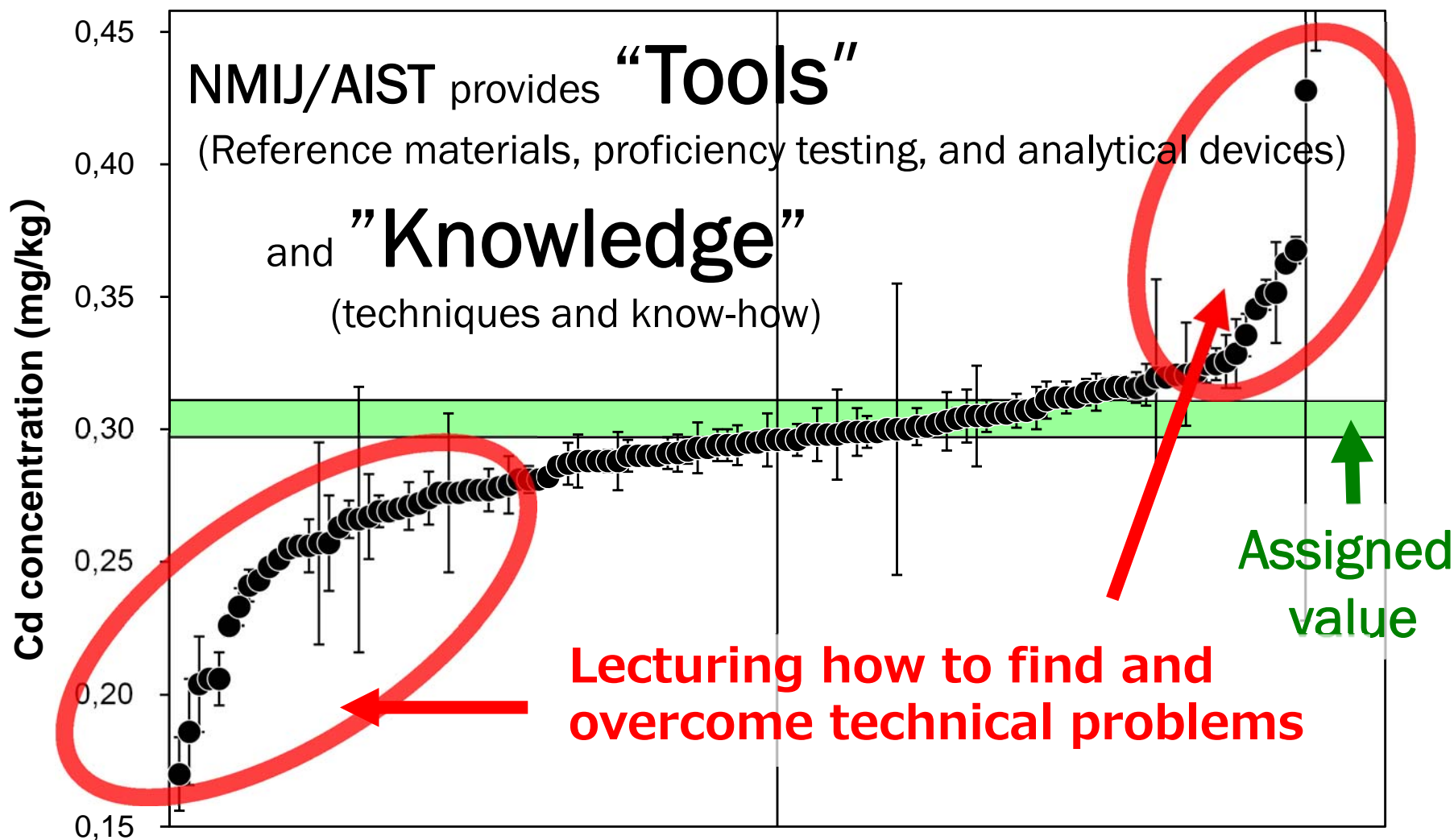
Participants should find and overcome technical problems by themselves, but those are not an easy task.



Score evaluation is an easy,
but finding the problems is not an easy !

NMIJ/AIST Support for Overcoming Technical Problems

Proficiency Testing in Japan: Cadmium in brown rice



Number of participants : 133

Overview of Certification: ex Rice Flour CRM

Property values were determined by 5 analysts

ICP-QMS, ICP-SFMS

Microwave digestion

Dry ashing with acid

ICP-OES, FP

GFAAS, FAAS



Determination of Property values

Ex. Cd concentrations in white rice flour CRMs

Property value was calculated as the weighted mean of analytical results.

The weight is the inverse of the combined standard u of the analytical results.

	NMIJ CRM7501-a	NMIJ CRM7502-a
MW digestion/ ID-ICP-MS	0.0516±0.0020	0.551 ± 0.010
Dry-ash/ ID-ICP-MS	0.0514±0.0011	0.552 ± 0.010
GFAAS	0.0524±0.0016	0.545 ± 0.014
ICP-MS	0.0517±0.0018	0.546 ± 0.006
Property Value	0.0517 mg/kg	0.548 mg/kg

World First CRM of Rice for **inorganic As** analysis

(since 2008)

NMIJ CRM 7501-a: White Rice Flour

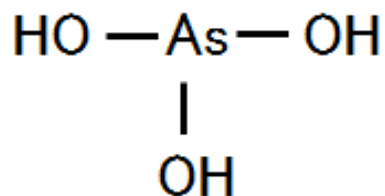


- Prepared from natural products.
- Certified for the concentrations of Cd, As and other elements, corresponding to the CODEX regulatory standard.
- Mutually recognized in the international framework.

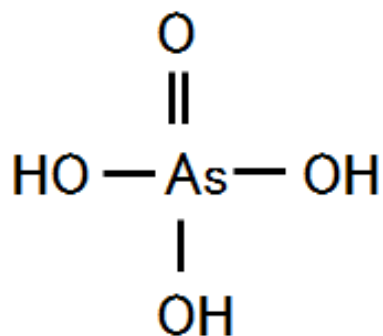


The quality of the certified values is important.

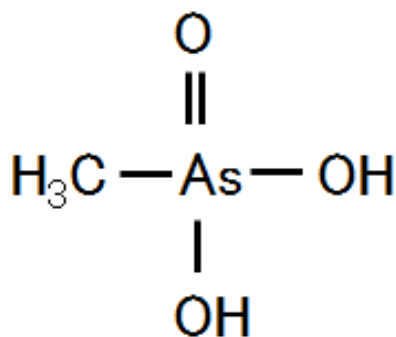
Some As species



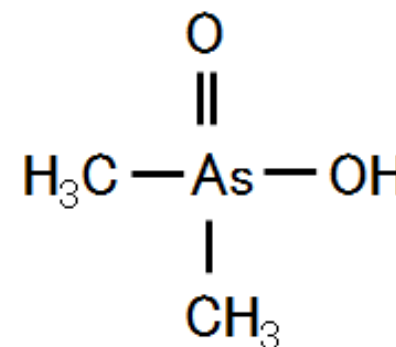
arsenious acid [As(III)]



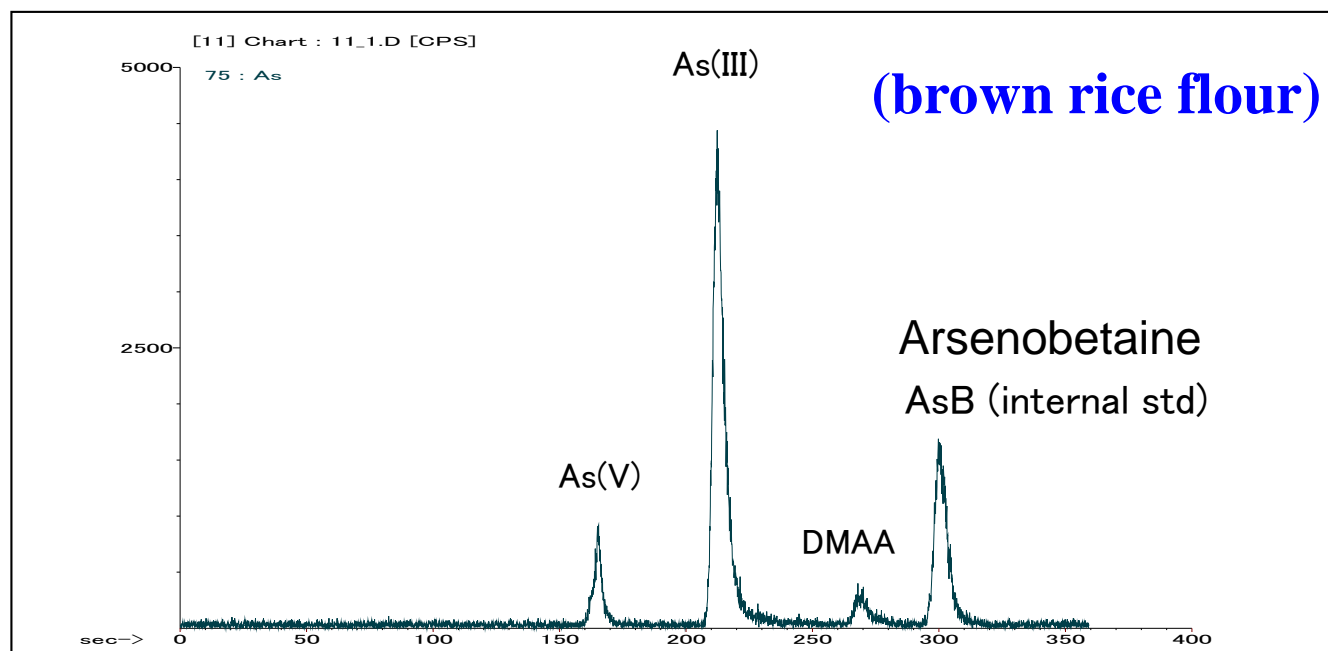
arsenic acid [As(V)]



monomethylarsonic acid (MMAA)

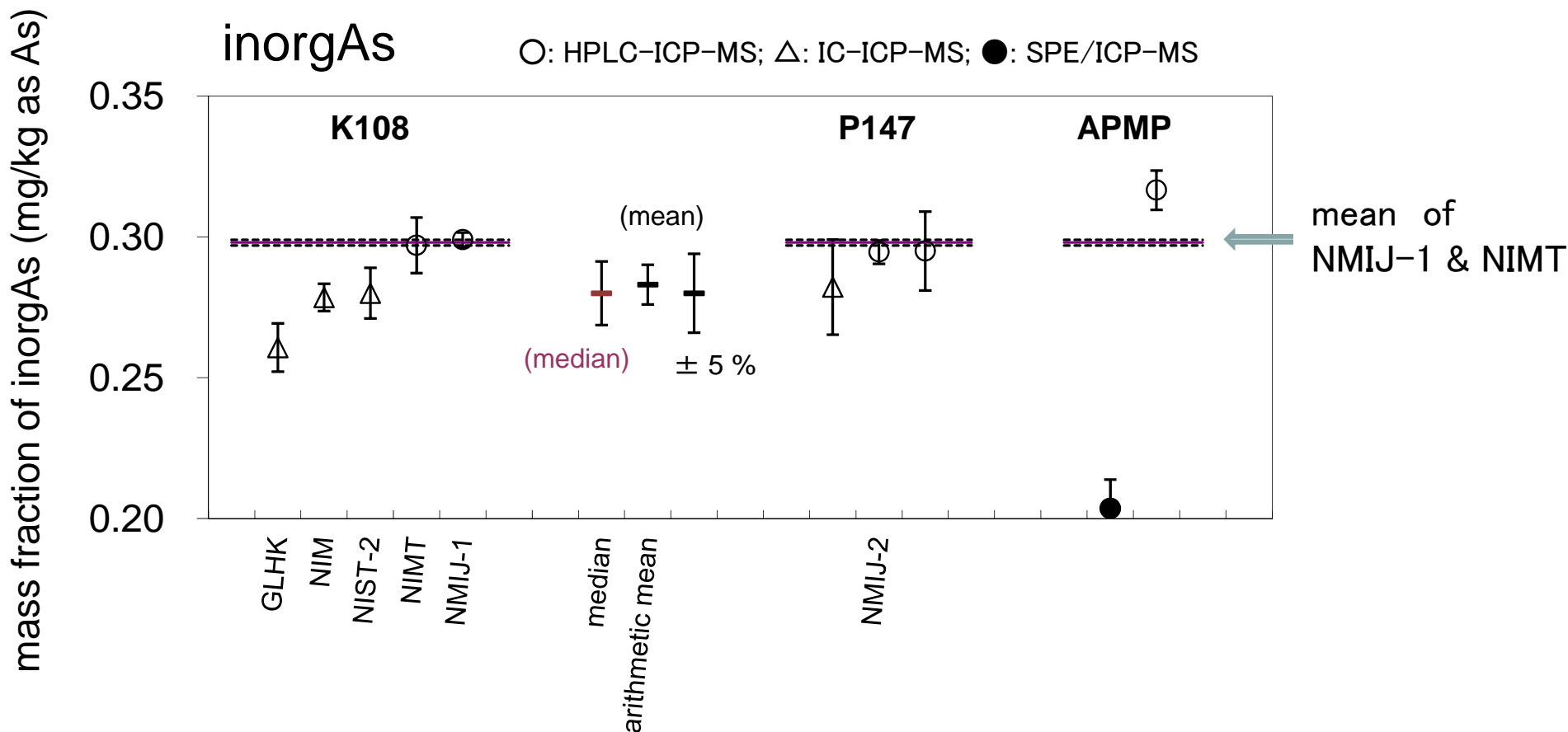


dimethylarsinic acid (DMAA)



CCQM-K108/P147+APMP.QM-P21 **inorgAs**

coordinated by NMIJ (2013 - 2014)



Results for inorgAs of CCQM-K108/P147+APMP.QM-P21
 The half of each bar indicates the combined standard uncertainty ($k=1$).

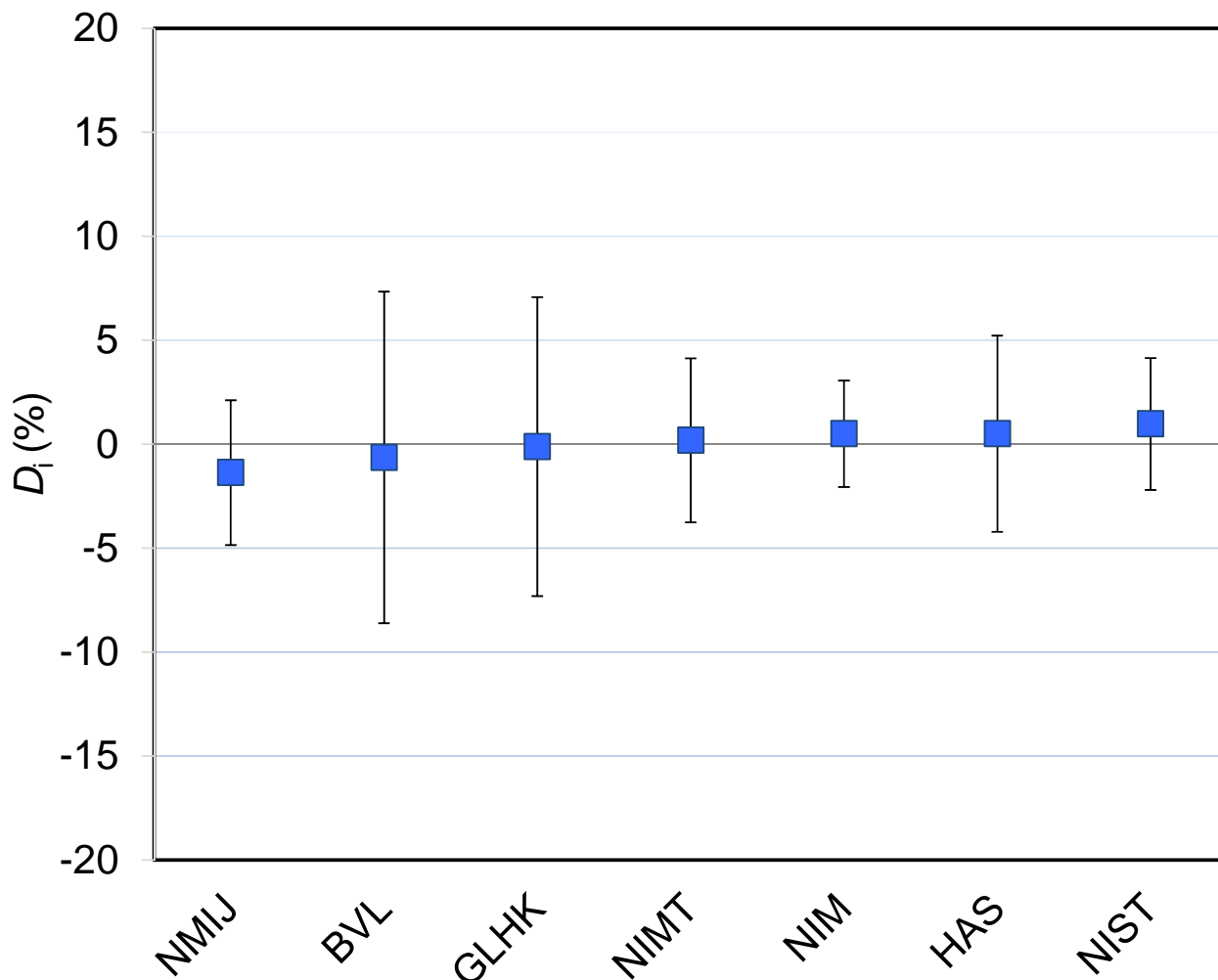
Determination of arsenic species and total arsenic in brown rice flour

CCQM-K108.2014 coordinated by NMIJ

Determination of arsenic species and total arsenic in brown rice flour

(2015 - 2016)

Equivalent Statement: DoE for **total As**



	Proposal KCRV	Expanded uncertainty, $k=2$
Median	0.6430	0.0028

D_i : relative values of degree of equivalence

Half of the error bars bar indicates the expanded uncertainty ($k=2$).

Median was used for the DoE calculation.

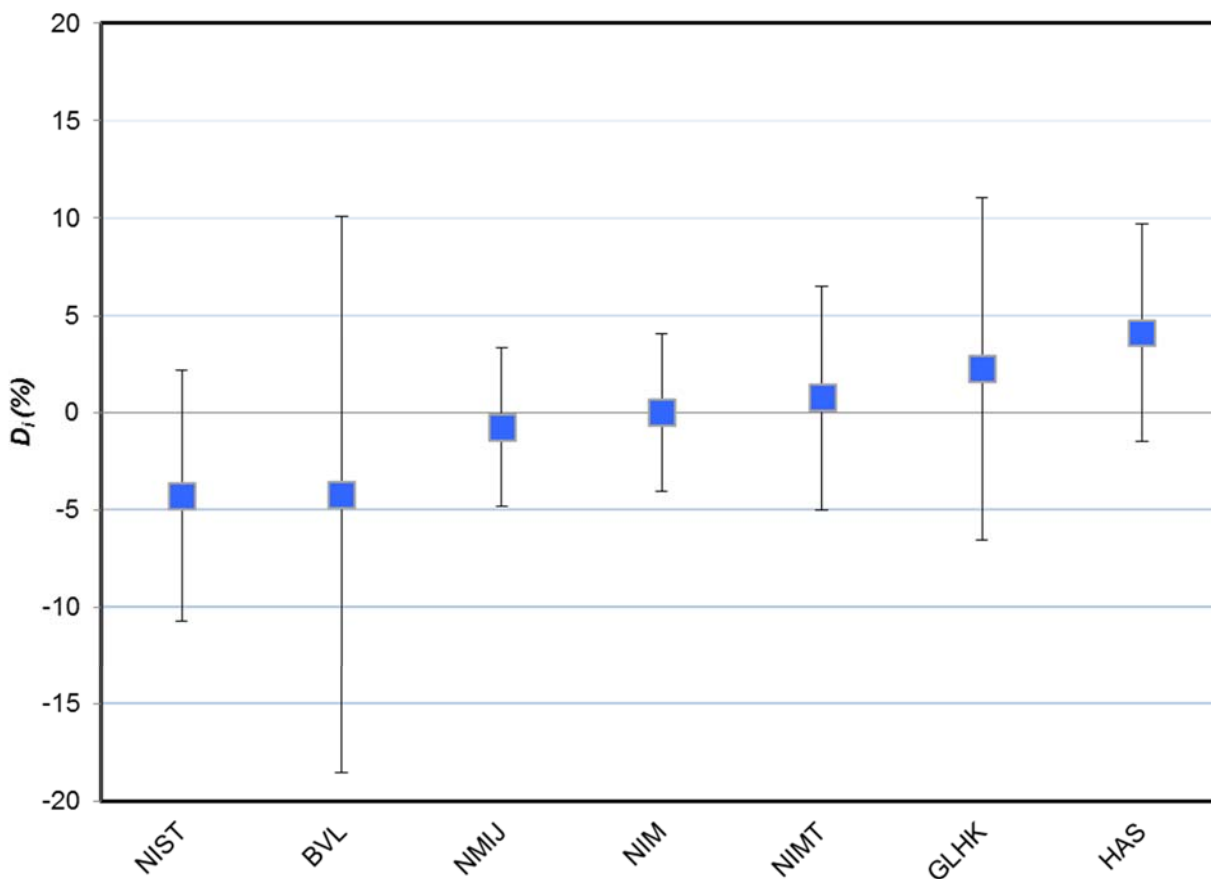
CCQM-K108.2014 coordinated by NMIJ

Determination of arsenic species and total arsenic in brown rice flour

(2015 - 2016)

Equivalent Statement: DoE for Inorg. As

**As(III) + As(V)
Highly toxic**



	Proposal KCRV	Expanded uncertainty, $k=2$
Median	0.5360	0.0084

D_i : relative values of degree of equivalence

Half of the error bars bar indicates the expanded uncertainty ($k=2$).

Median was used for the DoE calculation.

Brown rice CRM for ^{134}Cs and ^{137}Cs measurement

NMIJ and NARO (National Agriculture and Food Research Organization) developed a brown rice certified reference material (CRM) for ^{134}Cs and ^{137}Cs measurement.

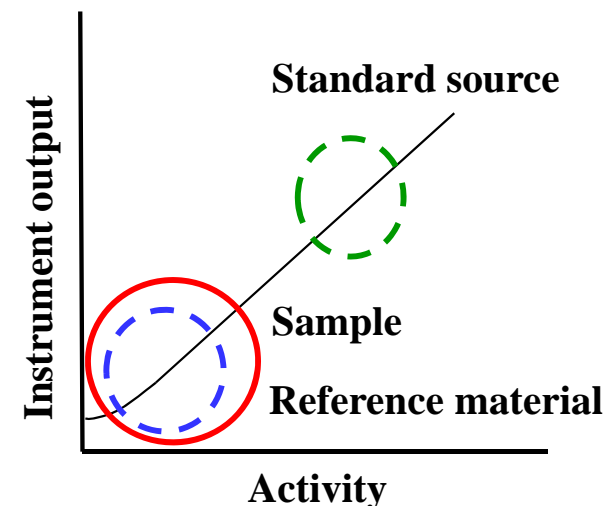
Specification of the CRM

- CRM number: **NMIJ CRM 7541-a**
- Matrix: **brown rice**
- Amount: **81 g**
- Activity (reference date: August 1st, 2012):

	Activity concentration [Bq/kg]	Relative expanded uncertainty ($k=2$) [Bq/kg]
Cs-134	33.6	2.6
Cs-137	51.8	4.6



*Standard sources are used for instrument calibration.
This CRM can be used to validate activity measurements.*



APMP.RI(II)-S3 (Cs-134.Cs-137) was conducted in 2013-2014, using a similar sample.
CCRI(II) supplementary comparison is planned in 2016-2017, using a wheat sample.

Thank you for your attention !

Please visit the website on NMIJ Reference Materials:
<https://www.nmij.jp/english/service/C/>