EURAMET.QM-S11

Supplementary Comparison on

Determination of Elements in River Water

Final Report

Süleyman Z. Can[1], Betül Arı Engin[1], Alper İşleyen[1], Aida Jotanovic[2], Osvaldo Acosta[3], Pedro Prina[3], Mariano Schvartz[3], Milenko Savić[4], Maja Stojanović[4], Diego Alejandro Ahumada Forigua[5], Johanna P. Abella G.[5], Diego A. Ahumada F.[5], Teemu Näykki[6], Timo Sara-Aho[6], Jochen Vogl[7], Maren Koenig[7], Olaf Rienitz[8], Janine Noordmann[8], Carola Pape[8], Jessica Towara[8], Elias Kakoulides[9], Charalampos Alexopoulos[9], Rosi Ketrin[10], Eka Mardika[10], Isna Komalasari[10], Christine Elishian[10], Evaldas Naujalis[11], Birutė Knašienė[11], Christian Uribe[12], Elmer Carrasco[12], Agnieszka Zoń[13], Beata Warzywoda[13], Aleksei Stakheev[14], Vladimir Dobrovolskiy[14], Tatiana Stolboushkina[14], Anastasia Glinkova[14], Egor Sobina[15], Tatyana Tabatchikova[15], Luka Gaževi[16], Marija Paunović[16], Radojko Jaćimović[17], Tea Zuliani[17], Ramiro Pérez Zambra[18], Romina Napoli[18]

[1]TÜBİTAK UME	TÜBİTAK Ulusal Metroloji Enstitüsü	Türkiye
[2]IMBIH	Institute of Metrology of Bosnia and Herzegovina	Bosnia and Herzegovina
[3]INTI	Instituto Nacional de Tecnología Industrial	Argentina
[4]IW	Institut za vode d.o.o. Bijeljina	Bosnia and Herzegovina
[5]INM	Instituto Nacional de Metrología de Colombia	Colombia
[6]SYKE	Finnish Environment Institute	Finland
[7]BAM	Bundesanstalt für Materialforschung und –prüfung	Germany
[8]PTB	Physikalisch-Technische Bundesanstalt	Germany
[9]EXHM/GCSL-EIM	National Laboratory of Chemical Metrology/General Chemistry State Laboratories - Hellenic Institute of Metrology	Greece
[10]RCM-LIPI	Research Center for Metrology, Indonesian Institute of Sciences	Indonesia
[11]FTMC	Center For Physical Sciences And Technology	Lithuania
[12]INACAL	Instituto Nacional de Calidad	Peru
[13]GUM	Central Office of Measures	Poland
[14]VNIIFTRI	Russian Metrological Institute of Technical Physics and Radio Engineering	Russia
[15]UNIIM	Ural Research Institute for Metrology	Russia
[16]DMDM	Directorate of Measures and Precious Metals	Serbia
[17]IJS	Jožef Stefan Institute	Slovenia
[18]LATU	Laboratorio Tecnológico del Uruguay	Uruguay

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1. INTRODUCTION

The need for quality assessment of anthropogenic impact on environmental pollution is increasing due to discharge from various industries, the use of chemicals in agriculture and the consumption of fossil fuels. Diminishing resources such as natural waters used for the cultivation of agricultural products, plant and animal habitats are under severe pollution pressure and are at constant risk. The EU has stipulated the maximum allowable concentration of priority pollutants in different classes of surface water under the Water Framework Directive in Directive 2008/105/EC[1] Annex I "Environmental quality standards for priority substances and certain other pollutants", and Annex II "List of priority substances in the field of water policy". Several parameters, such as Pb, Cd, Ni, Hg were listed in the priority substances and Cd and Hg were further identified as priority hazardous substances. Arsenic is also an important contaminant for its potential toxicological and carcinogenic effects.

In the framework of the Matrix Reference Materials for Environmental Analysis (EnvCRM) project (www.envcrm.com) funded by European Metrology Programme for Innovation and Research (EMPIR) Environmental Call, an inter-comparison study between the partners is organised in order to characterise the produced candidate reference material. The candidate 'Elements in River Water' certified reference material (CRM) is one of the three reference materials targeted in the project, and serves as the test material for this study. The partners carried out measurements in order to characterise the analytes of interest: Pb, Cd, Ni and As as mandatory elements, and Se as an option.

In April 2017, the project proposal of an inter-comparison study "Determination of elements in river water was presented at the Working Group on Inorganic Analysis (IAWG) of the *Consultative Committee for Amount of Substance – Metrology in Chemistry and Biology* (CCQM) and it was decided to perform this measurement as EURAMET Supplementary Comparison EURAMET.QM-S11. The aim of the study was approved as to test the capabilities of participants in measuring the elements As, Cd, Ni, Pb and Se in river water. While the elements As, Cd, Ni and Pb are mandatory measurands, Se is an optional one. Participants were asked to perform the measurements with respect to the protocol provided (Appendix II).

In parallel to the supplementary comparison, the pilot study EURAMET 1424 was organized to give less experienced institutes as well as university laboratories the opportunity to participate.

The participants of supplementary comparison will be able to use the comparison results to support their calibration and measurement capability (CMC) claims for the elements of interest. Along with the Core Capability Tables, it may be possible to claim CMCs in related matrices as well.

2. TIMETABLE

The study has been conducted by the following timetable:

June 15, 2017	Registration deadline
June/July 2017	Distribution of samples
November 30 2017	Results submission deadline
February 2018	Presentation of results at the EURAMET TCMC-SCIA Meeting
April 2018	Presentation of results at the CCQM-IAWG Meeting

3. PARTICIPATING INSTITUTES

Initially 18 institutes were registered using the form (Appendix III) in the Supplementary Comparison EURAMET.QM-S11 but one institute, Instituto de Salud Pública de Chile (ISP) did not submit the data. Table 1 lists the participating laboratories in EURAMET.QM-S11 except ISP.

Table 1. List of participating NMIs/DIs for EURAMET.QM-S11

No	NMI/DI	Country	Contact Person	Analyst(s)	Measurand
1	INTI Instituto Nacional de Tecnología Industrial	Argentina	Osvaldo Acosta Mabel Puelles	Pedro Prina, Mariano Schvartz y Osvaldo Acosta	As, Cd, Ni, Pb
2	IMBIH Institute of Metrology of Bosnia and Herzegovina	Bosnia and Herzegovina	Aida Jotanovic	-	- (coordinating laboratory)
3	IW Institutzavoded.o.o. Bijeljina	Bosnia and Herzegovina	Milenko Savić	Maja Stojanović	As, Cd, Ni, Pb, Se
4	INM Instituto Nacional de Metrología de Colombia	Colombia	Diego Alejandro Ahumada Forigua		As, Cd, Ni, Pb
5	SYKE Finnish Environment Institute	Finland	Teemu Näykki	Timo Sara-Aho	As, Cd, Ni, Pb, Se
6	BAM Bundesanstalt für Materialforschung und Prüfung	Germany	Jochen Vogl	Maren Koenig	Cd, Ni, Pb

No	NMI/DI	Country	Contact Person	Analyst(s)	Measurand
7	PTB Physikalisch-Technische Bundesanstalt	Germany	Olaf Rienitz	Janine Noordmann, Carola Pape, Jessica Towara	As, Cd, Ni, Pb
8	EXHM/GCSL-EIM National Laboratory of Chemical Metrology/General Chemistry State Laboratories - Hellenic Institute of Metrology	Greece	Elias Kakoulides Charalampos Alexopoulos	Elias Kakoulides Charalampos Alexopoulos	As, Cd, Ni, Pb, Se
9	RCM-LIPI Research Center for Metrology, Indonesian Institute of Sciences	Indonesia	Rosi Ketrin	EkaMardika, Isna Komalasari, Christine Elishian	As, Cd, Ni, Pb
10	FTMC Center For Physical Sciences And Technology	Lithuania	Evaldas Naujalis	Birutė Knašienė	As, Cd, Ni, Pb, Se
11	INACAL Instituto Nacional de Calidad	Peru	Christian Uribe Elmer Carrasco	/	As, Cd, Ni, Pb, Se
12	GUM Central Office of Measures	Poland	Agnieszka Zoń	Agnieszka Zoń, Beata Warzywoda	As, Cd, Ni, Pb
13	UNIIM Ural Research Institute for Metrology	Russia	Egor Sobina	Tatyana Tabatchikova	As, Cd, Ni, Pb, Se
14	VNIIFTRI Russian Metrological Institute of Technical Physics and Radio Engineering	Russia	Aleksey Stakheev	Vladimir Dobrovolskiy, Aleksei Stakheev, Tatiana Stolboushkina, Anastasia Glinkova	As, Cd, Ni, Pb
15	DMDM Directorate of Measures and Precious Metals	Serbia	Luka Gažević	Marija Paunović	As, Cd, Ni, Pb, Se
16	IJS Jožef Stefan Institute	Slovenia	Radojko Jaćimović	Tea Zuliani	Cd, Ni, Pb, Se
17	TÜBİTAK UME TÜBİTAK Ulusal Metroloji Enstitüsü	Turkey	Süleyman Z. Can	Betül Ari	As, Cd, Ni, Pb, Se
18	LATU Laboratorio Tecnológico del Uruguay	Uruguay	Ramiro Pérez Zambra Romina Napoli	Ramiro Pérez Zambra / Romina Napoli	As, Cd, Ni, Pb, Se

4. TEST MATERIAL

4.1. Sample preparation

Test samples were candidate certified reference materials produced according to the requirements in ISO 17034:2016 standard[2] to certify the previously listed measurands within the EMPIR-EnvCRM project. Raw material collection area is a creek feeding an artificial lake (Darlık Dam) which is one of Istanbul's water supplies. Prior to collection, carboys (10 L, HDPE) were cleaned with deionized water and were dried at ambient temperature. Water samples were collected directly using beakers (polypropylene) and transferred to cleaned carboys. Collected samples (around 140 L in 14 carboys) were transported to TÜBİTAK UME and were acidified to have a 2 % (v/v) HNO_3. The samples were stored at +4 °C for further processing.

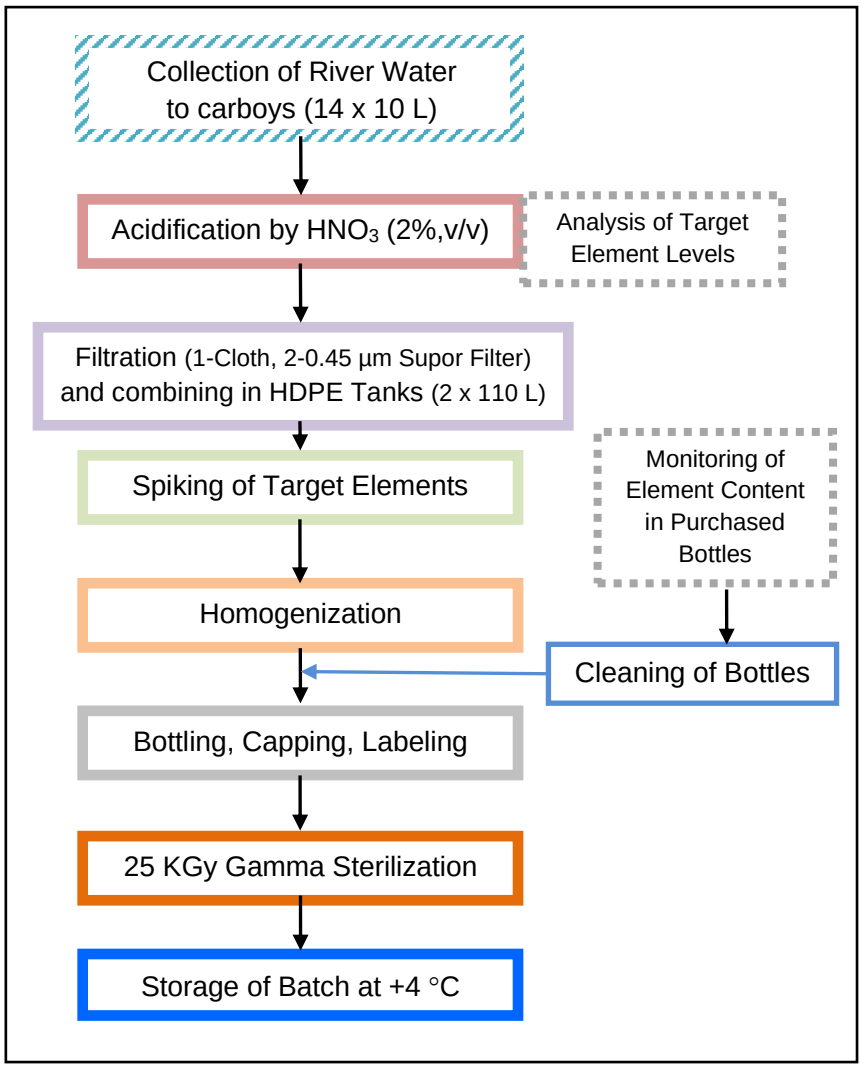
For preliminary measurements, subsamples from 10 different carboys were taken and analysed with HR-ICP-MS for five target elements. Results of this measurement are summarized in Table 2.

Table 2. Preliminary measurement result in the raw river water sample

Element	**Measured levels** (µg/kg ± Std. Dev. *n*=20)	**Target Range** (µg/kg)
As	0.99 ± 0.04	2 – 20
Cd	< 0.1	0.1 – 5
Ni	1.40 ± 0.21	2 – 20
Pb	0.29 ± 0.07	2 – 20
Se	< 0.1	2 – 20

Results showed that the raw material has too low elemental content, thus it was decided to spike this material to reach target levels for all elements. The spike mixture (250 mL) was prepared from NIST and SCP Science standards for As, Cd, Ni, Pb and Se. The material in 10-L carboys were combined in homogenization tanks (2 x 110 L, HDPE) by filtering through first a cloth filter, and then a filter with a pore size of 0.45 µm. Homogenization performed by circulating the content between the HDPE tanks for a total of 6 h. Filling and capping were done using automated filling machine. Gamma irradiation is applied to improve the shelf life of products by eliminating any present organism such as bacteria in the bottled product (^{60}Co γ-irradiation with 25 kGy dose).

The process of preparation of sample material is shown on flow diagram below:



4.2. Homogeneity tests

Homogeneity study between the units is performed to show that assigned values are valid for all units within the stated uncertainty. Homogeneity study between the units is performed with a specific number of samples representing the whole batch. Twelve units were selected by using random stratified sampling. Homogeneity tests were carried out by measuring three sub-samples under the repeatability conditions. The samples to be analysed were introduced to the instruments by random order to find out any trend arising from analytical and/or filling sequences.

The data obtained were evaluated statistically by regression analysis for the presence of any trend in analytical and filling sequence at 99 % confidence level. After evaluation of data, only analytical sequence trend was found for As and Cd.

Grubbs test (one sided) was applied to all data for the presence of outlier at 99 % confidence level and no outlier was detected.

Analysis of Variance (ANOVA) is a statistical tool used to estimate the between bottle heterogeneity (u_{bb}) and was calculated using the equation (1) in accordance with ISO Guide 35:2017 [3]:

$$s_{bb} = u_{bb} = \sqrt{\frac{MS_{between} - MS_{within}}{n}} \quad (1)$$

where $MS_{between}$: Mean of square of variance between units, MS_{within}:mean of square of variance within units, n: number of replicates per unit.

$MS_{between}$ is found to be smaller than MS_{within} in conditions for which the heterogeneity of the material is smaller than heterogeneity that can be determined by the applied analytical method or measurement fluctuations that may have occurred randomly. In these cases, since u_{bb} cannot be calculated, u^*_{bb} was calculated as heterogeneity contributing to uncertainty including method repeatability using equation (2) in accordance with ISO Guide 35:2017 [3]:

$$u^*_{bb} = \sqrt{\frac{MS_{among}}{n}} \sqrt[4]{\frac{2}{\nu\, MS_{within}}} \quad (2)$$

$\nu\, MS_{within}$: Degree of freedom of mean square of within bottles

The homogeneity study results, reported in Table 3 show that no significant heterogeneity was observed in the river sample, and the test material was considered as satisfactory for the purpose of this comparison.

Table 3. Results of Homogeneity Study

Analyte	u^*_{bb} (%)
As	0.36
Cd	0.54
Ni	0.90
Pb	0.30
Se	0.79

4.3. Stability study

The stability studies were carried out using an isochronous design. In that approach, samples are stored for a certain time at different temperature conditions. Afterwards, the samples are moved to conditions where further degradation can be assumed to be negligible ("reference conditions"), effectively "freezing" the degradation status of the materials. At the end of the isochronous storage, the samples are analysed simultaneously under repeatability conditions. Analysis of the material (after various exposure times and temperatures) under repeatability conditions greatly improves the sensitivity of the stability tests.

For the *Short-Term Stability (STS)* test, two different temperatures (18 °C and 60 °C) and four time points (0, 1, 2 and 4 weeks) were tested. Fourteen samples were randomly selected and 12 samples were subjected to the test temperatures for the specified time intervals as two of them serves as the reference samples. Test samples were moved to +4 °C (reference temperature) after completion of the test time. All samples were analysed at the same time. Three replicate measurements for each unit were performed by each laboratory under the repeatability conditions.

The data for each temperature were first examined by single Grubbs test for both 95 % and 99 % confidence intervals to find out outliers. Since no technical reason can be found to reject these data, all outliers were included in the STS calculations.

Values calculated for each time point were plotted against the time for the assessment of short-term stability. The relationship between variables were analysed in order to determine if any significant change exists with the testing time (regression analysis). It was found that the slopes were not significantly different than zero for all parameters in the 95 % and 99 % confidence intervals.

Uncertainty calculations are done using equation (3). Maximum time for transfer is chosen as one week.

$$u_{sts} = \frac{RSD}{\sqrt{\sum(t_i - \bar{t})^2}} \qquad (3)$$

where *RSD*: relative standard deviation obtained from all data in STS, t_i:time point for each replicate,
$\bar{t}$: mean of all time points.

Results obtained from short term stability tests are given in Table 4.

Table 4. Results of Short-term Stability Study

Analyte	18 °C u_{sts} (%)	60 °C u_{sts} (%)	Significant trend? (95%)		Significant trend? (99%)	
			18 °C	60 °C	18°C	60 °C
As	0.27	0.22	No	No	No	No
Cd	0.23	0.18	No	No	No	No
Ni	0.26	0.21	No	No	No	No
Pb	0.18	0.14	No	No	No	No
Se	1.0	0.8	No	No	No	No

Result of STS study showed that the river water sample can be transferred to the end users without applying any cooling elements if the ambient temperature is not exceeding 60°C, and duration is not exceeding ONE week.

Among the all participants who received samples, 11 of them returned the sample receipt forms. Most participants declared that no problem is observed in the samples received. RCM-LIPI mentioned in the formed that '*It is warm, I check the temperature in the box is 32 °C.*' Since the observation is well below the tested temperature of 60 °C, no action was taken.

The stability of the material has been monitored for longer period of time to ensure that all the measurands are stable throughout the comparison period. Randomly selected samples were subjected to the test temperature of 18 °C, targeted storage condition, for a period of 12 months, covering the whole comparison schedule. Test period followed by the isochronous ICP-MS measurements to monitor the stability. The results showed that the material showed no sign of degradation for any of the measurands at 99 % confidence interval. Thus, the material is accepted to be stable for the comparison period.

4.4. Instruction to participants

Each participant has received two sample bottles containing approximately 100 mL of the river water and was requested to perform at least three measurements of both samples. It was recommended that the bottles to be kept +4 °C until the sample preparations for measurements.

Participants were requested to report the mean value of at least three measurements on two delivered samples as the mass fraction of measurands in µg/kg for total arsenic, cadmium, nickel and lead, as mandatory elements, and selenium as optional along with additional

information on associated uncertainty with specified coverage factor with a full uncertainty budget and main sources, as well as short description of used analytical method and standard reference materials used for calibrations.

5. METHODS OF MEASUREMENT

The measurement methods were left free to be selected by the participants. The majority of the participants used measurement techniques such as Inductively Coupled Plasma Mass Spectrometry (ICPMS) and Isotope Dilution ICPMS (ID-ICPMS), and two laboratories used the Electro Thermal Atomic Absorption Spectrometry (ET-AAS) technique. One laboratory used Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method.

The method summary is shown in Table 5.

Table 5. Summary of measurement methods used by the participants

NMI/DI	Analyte	Measurement Technique	Calibration Method	Analytical Instrument	Reference Material/ Traceability
INTI	As, Cd, Ni, Pb	ICPMS	External calibration As75; Cd111;Ni62 and Pb206, 207, 208; Ge as internal standard;	ICPMS, ELAN-DRC Perkin Elmer	As NIST SRM 3103a Cd NIST SRM3108 Ni NIST SRM 3136 Pb NIST SRM 3128
IW	As, Cd, Ni, Pb, Se	ET-AAS	External calibration	AAS;6300 Shimadzu	As LGC 137075-2 Cd LGC 120958-1 Ni LGC 114351-2 Pb LGC 115275-4
INM	As, Cd, Ni, Pb	ICPMS	Standard addition; As75, Cd111, Ni60, 61; Pb206,208; Rh as internal standard	ICPMS, NexION300D Perkin Elmer	Pb NIST SRM 3128 Cd NIST SRM 3108 As SMU B03 Ni SMU B24
	Cd, Ni, Pb	ET-AAS	Cd228.8nm, Ni237.0nm, Pb283.31nm	AAS, PinAAcle 900T Perkin Elmer	
SYKE	As, Cd, Ni	ICPMS	Isotope measurement As75, Cd111, Ni60 Rh as internal standard	ICPMS, Icap Q Thermo Scientific	As NIST SRM 3103a Cd NIST SRM 3108 Ni NIST SRM 3136 Pb NIST SRM 981
	Pb	ID-ICPMS	Exact matching double ID-ICPMS		

NMI/DI	Analyte	Measurement Technique	Calibration Method	Analytical Instrument	Reference Material/ Traceability
BAM	Cd, Ni, Pb	ID-ICPMS	Exact matching double ID-ICPMS; Cd111/Cd113 Cd112/Cd111 with correction for Sn interface; Ni60/Ni61 Pb208/Pb207 Pb206/Pb207 Pb204/Pb207Pb-isotopic standard used for correction of instrumental mass discrimination	ICPMS, Element 2 Thermo	BAM back-spikes, prepared primary calibration solution from: Ni BAM RS4 Cd Johnson Matthey high purity 99.999 % Pb BAM Y004
PTB	As	ICPMS	Gravimetric standard addition; In as internal standard	ICPMS, Finnigan Element XR Thermo Fisher Scientific	As NIST SRM 3103a (*in house prepared primary solution from As metal HPS*) Cd NIST SRM 3108 Cd NIST SRM 746 Ni NIST SRM 986 Pb NIST SRM 981
	Cd, Ni, Pb	IDMS	Double IDMS, exact matching technique		
EXHM/ GCSL-EIM	As	ICPMS	Standard addition; As CRM	ICPMS, Element 2 Thermo	As NIST SRM 3103a Cd NIST SRM 3108 Ni NIST SRM 3136 Pb NIST SRM 3128 Se NIST SRM 3149
	Cd, Ni, Pb, Se	ID-ICPMS	reverse IDMS spike Cd111, Cd111 and Cd114 monitored; Ni60, Ni58 and Ni60 monitored; Pb206, Pb206, and Pb208 monitored; Se77, Se76, Se77 and Se 78 monitored		
RCM-LIPI	As, Cd, Ni, Pb	ICPMS	External calibration As75; Cd112; Ni60 and Pb207; Y as internal standard and compare with standard addition	ICPMS, ICAP Qs Thermo	CRM ERM CA011 As NIST SRM 83d Cd NIST SRM 3108 Ni NIST SRM 3136 Pb BAM Y004 (Pb 0001)
FTMC	As, Cd, Ni, Pb, Se	ICPMS	Single-point calibration method; As75, Cd110, Cd111, Cd112, Cd113, Cd114, Ni60, Pb206, Pb207, Pb208, Se77, Se78	ICPMS, Element 2 Thermo	NIST SRM 1643f

NMI/DI	Analyte	Measurement Technique	Calibration Method	Analytical Instrument	Reference Material/ Traceability
INACAL	As, Cd, Ni, Pb, Se	ET-AAS	Single point standard addition; matrix modifier- 5µl of 0.1% Pd solution	AAS, Analyst 800 Perkin Elmer	As NIST SRM 3103a Cd NIST SRM 3108 Ni NIST SRM 3136 Pb NIST SRM 3128 Se NIST SRM 3149
GUM	As, Cd, Ni, Pb	ICPOES	External calibration method; As189.0nm, Cd214.4 nm, Ni231.6nm, Pb220.3 nm	ICP-OES, ICAP 6500 duo Thermo Scientific	As SMU B03 Cd SMU B08 Ni SMU B24 Pb SMU B26
UNIIM	As, Se	ICPMS	Calibration curve	ICPMS, NexION 300D Perkin Elmer	As-GSO 7976-2001 Se-GSO 7779-2000
	Cd, Ni, Pb	ID-ICPMS	Primary measurement method ID-ICP MS		
VNIIFTRI	As, Cd, Ni, Pb	ICPMS	Gravimetric standard additions, 3 points calibration; Ni60, As75, Cd112, Cd114, Pb206, Pb207, Pb208	ICPMS, PlasmaQuant MS Analytik Jena	As Merck CRM Cd Merck CRM Ni Merck CRM Pb Merck CRM
DMDM	As, Cd, Ni, Pb, Se	ICPMS	Calibration curve, gravimetric method, multielemental standard for calibration; Internal standard-LGC VHG-LIS2-100 Multielement mix	ICPMS, ICAP Q Thermo Scientific	As, Ni, Cd, Pb ROTH single element solution
IJS	Cd, Ni, Pb, Se	ICPMS	External calibration; 60Ni, 111Cd, 208Pb, 78Se; Internal standards 103Rh, 115In, 209Bi	ICPMS, 7900x Agilent	Cd NIST SRM 3108 Ni NIST SRM 3136 Pb NIST SRM 3128 Se NIST SRM 3149
TUBITAK UME	As, Ni	ICPMS	Gravimetric standard addition with Y as the internal standard	Thermo Finnigan Element 2 HR-ICPMS and Agilent 8800 QQQ ICPMS	As NIST SRM 3103a Ni NIST SRM 3136 Cd IRMM 622 Pb NIST SRM 991, SRM 981, SRM 3128 Se NIST SRM 3149
	Cd, Pb, Se	ID-ICPMS	Exact-matching ID: $^{113}Cd/^{111}Cd$ (Single ID) $^{208}Pb/^{206}Pb$ (Double ID) $^{78}Se/^{76}Se$ (Triple ID)		
LATU	As	ICPMS	Gravimetric standard addition; Ge as internal standard;	ICPMS, Element 2 Thermo	As NIST SRM 3103a Cd NIST SRM 3108 Ni NIST SRM 3136 Pb NIST SRM 3128 Se NIST SRM 3149
	Cd, Ni, Pb, Se	ID-ICPMS	Exact-matching isotope dilution; 114Cd/111Cd, 60Ni/61Ni, 208Pb/206Pb, 78Se/77Se		

6. RESULTS AND DISCUSSION

6.1. General

The total number of registered institutes for EURAMET.QM-S11 comparison is 18, and 17 of them submitted their results. A summary of basic statistics from the submitted data are listed in Tables 6 to 10. All data are given in µg/kg.

Table 6. Reported As results in alphabetical order by country

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty, *u* (µg/kg)	Coverage Factor (k)	Expanded Uncertainty, *U* (µg/kg)
INTI	**14.128**	0.310	2	0.620
IW	**15.05**	0.653	2	1.305
INM	**15.68**	0.358	2	0.72
SYKE	**17.78**	0.843	2	1.69
PTB	**15.24**	0.11	2	0.22
EXHM/GCSL-EIM	**18.96**	0.37	2	0.74
RCM-LIPI	**15.6499**	0.5411	2	1.0822
FTMC	**15.38**	0.44	2.262	0.99
INACAL	**16.060**	0.24	2	0.48
GUM	**15.05**	0.85	2	1.69
UNIIM	**14.9**	0.65	2	1.3
VNIIFTRI	**16.23**	0.55	2	1.11
DMDM	**15.008**	0.187	2	0.374
UME	**15.07**	0.19	2	0.38
LATU	**15.32**	0.15	2	0.30

Table 7. Reported Cd results in alphabetical order by country

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty, *u* (µg/kg)	Coverage Factor (k)	Expanded Uncertainty, *U* (µg/kg)
INTI	**0.4889**	0.0130	2	0.0260
IW	**0.444**	0.014	2	0.028
INM	**0.533**	0.016	2	0.032
SYKE	**0.548**	0.0165	2	0.033
BAM	**0.5229**	0.0028	2	0.0057
PTB	**0.5099**	0.0041	2.11	0.0087
EXHM/GCSL-EIM	**0.529**	0.024	2	0.049
RCM-LIPI	**0.5220**	0.0510	2	0.1020
FTMC	**0.504**	0.014	2.262	0.032
INACAL	**0.544**	0.008	2	0.016
GUM	**0.48**	0.04	2	0.08
UNIIM	**0.47**	0.025	2	0.05
VNIIFTRI	**0.229**	0.011	2	0.022
DMDM	**0.409**	0.025	2	0.050
IJS	**0.524**	0.011	2	0.022
UME	**0.5221**	0.0035	2	0.0069
LATU	**0.527**	0.0094	2	0.019

Table 8. Reported Ni results in alphabetical order by country

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty, *u* (µg/kg)	Coverage Factor (k)	Expanded Uncertainty, *U* (µg/kg)
INTI	**14.246**	0.210	2	0.420
IW	**13.79**	0.507	2	1.014
INM	**14.96**	0.367	2	0.73
SYKE	**14.91**	0.375	2	0.75
BAM	**14.395**	0.052	2	0.104
PTB	**14.51**	0.11	2.03	0.23
EXHM/GCSL-EIM	**14.71**	0.32	2	0.63
RCM-LIPI	**14.8883**	0.7373	2	1.4747
FTMC	**14.21**	0.41	2.262	0.94
INACAL	**15.95**	0.31	2	0.62
GUM	**13.09**	0.29	2	0.58
UNIIM	**13.7**	0.55	2	1.1
VNIIFTRI	**16.16**	0.49	2	0.98
DMDM	**11.577**	0.212	2	0.424
IJS	**14.4**	0.3	2	0.6
UME	**14.44**	0.20	2	0.40
LATU	**14.49**	0.12	2	0.23

Table 9. Reported Pb results in alphabetical order by country

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty, *u* (µg/kg)	Coverage Factor (k)	Expanded Uncertainty, *U* (µg/kg)
INTI	**13.633**	0.200	2	0.400
IW	**12.55**	0.104	2	0.208
INM	**13.64**	0.42	2	0.85
SYKE	**13.62**	0.17	2	0.34
BAM	**13.547**	0.043	2	0.085
PTB	**13.48**	0.088	2	0.18
EXHM/GCSL-EIM	**12.99**	0.20	2	0.40
RCM-LIPI	**12.2642**	0.3180	2	0.6360
FTMC	**13.21**	0.252	2.262	0.57
INACAL	**14.68**	0.29	2	0.58
GUM	**12.20**	0.56	2	1.13
UNIIM	**13.3**	0.4	2	0.8
VNIIFTRI	**13.94**	0.52	2	1.04
DMDM	**11.613**	0.199	2	0.398
IJS	**13.94**	0.46	2	0.92
UME	**13.37**	0.09	2	0.17
LATU	**13.63**	0.11	2	0.22

Table 10. Reported Se results in alphabetical order by country

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty, *u* (µg/kg)	Coverage Factor (k)	Expanded Uncertainty, *U* (µg/kg)
EXHM/GCSL-EIM	**4.08**	0.22	2	0.44
FTMC	**4.95**	0.156	2.262	0.35
INACAL	**5.49**	0.08	2	0.16
UNIIM	**4.5**	0.55	2	1.1
IJS	**4.69**	0.17	2	0.34
UME	**4.953**	0.049	2	0.098
LATU	**4.96**	0.068	2	0.14

6.2. Traceability of Calibrants used by Participants

Participants were required to provide the information about the traceability of the reference materials/calibrants they used in comparison. The information provided is summarized in Table 5 - *Summary of measurement methods used by the participants*. Participants mostly used calibration standards produced by NMIs or in-house prepared materials. On the other hand, the participants IW, VNIIFTRI and DMDM used commercial standards in their measurements. Thus, the results reported by these participants were not included in reference value calculations.

INM reported Cd, Ni and Pb results as the combination of the results obtained by ICP-MS and ET-AAS techniques, in reference to Levenson (*J. Res. Natl. Inst. Stand. Technol.* 105, 571 (2000)).

6.3. Statistical Evaluation of the Participant Data

The data submitted by the participants was first evaluated using robust statistics as described by the CCQM Guidance Note [4]. The aim is to check the consistency, and observe any outlier data, i.e. outside the$(x \pm 2\sigma)$ range where x is the mean and σ is the dispersion. After performing the checks, the following observations were made:

As: SYKE and EXHM/GCSL-EIM results are outliers.

Cd: VNIIFTRI and DMDM results are outliers.

Ni: INACAL, VNIIFTRI and DMDM results are outliers.

Pb: DMDM result is outlier.

Se: No outlier data.

After sharing the data with the participants, SYKE and EXHM/GCSL-EIM reviewed their As data, and affirmed weak repeatability and method based reasons. VNIIFTRI reviewed their Cd and Ni results, and adduced technical reasons for outlying data. DMDM evaluated their Cd, Ni and Pb results, and justified technical reasons for outlying data. Since the explanations made by participants do not point any obvious reason, the data have been retained in the calculations provided that the calibrations standards used meet the traceability requirements.

6.4. Calculation of the Reference Values and Associated Uncertainties

The consensus values and their respective standard uncertainties are calculated and presented in this report, using different location estimators including arithmetic mean and median. During the SCIA meeting on 05 February 2018 it was agreed that all participants' data should be included in the calculation of Supplementary Comparison Reference Value (SCRV) unless otherwise a participant claims to be not included for a technical reason. Therefore, excepting the participants who asked for exclusion previously, all the data were retained in the calculation of SCRV and Supplementary Comparison Reference Uncertainty (SCRU). On the other hand, the participants used commercial standards in their measurements were not included in SCRV and SCRU calculations.

The statistics used for the evaluation of data to determine the SCRV and SCRU consist mainly of two approximations: The arithmetic mean and the median of the data set. The formulation used for the calculation of arithmetic mean and the median and their uncertainties are as follows:

1. Arithmetic Mean

$$x_{A.mean} = \frac{1}{n}\sum_{i=1}^{n} x_i \tag{4}$$

$$s_{A.mean} = \sqrt{\left(\frac{1}{n-1}\sum_{i=1}^{n}(x_i - x_{A.mean})^2\right)} \tag{5}$$

$$u_{A.mean} = \frac{s}{\sqrt{n}} \tag{6}$$

2. Median

$$x_{Median} = \begin{cases} \frac{1}{2}(x'_{\frac{m}{2}} + x'_{\frac{m}{2}+1}) & m \text{ is even} \\ x'_{m/2} & m \text{ is odd} \end{cases} \tag{7}$$

$$MAD = median(|x_i - x_M|) \tag{8}$$

$$MAD_E = \hat{\sigma} = MAD * 1.483 \quad (9)$$

$$u_{median} = MAD_E \sqrt{\frac{\pi}{2n}} \quad (10)$$

Values of the SCRV and respective standard uncertainties calculated are presented in bold numbers in Table 11for each measurand. All data are given in µg/kg.

Table 11.Calculated SCRV and SCRU using the arithmetic mean and median approaches

Measurand	n	Arithmetic mean	$u_{A.mean}$	Median	u_{median}
As	12	15.78	0.38	**15.35**	**0.17**
Cd	14	0.516	0.006	**0.523**	**0.006**
Ni	14	14.49	0.17	**14.47**	**0.12**
Pb	14	13.39	0.17	**13.51**	**0.09**
Se	7	**4.80**	**0.17**	4.95	0.18

Graphical presentations of the SCRV and u(SCRU) for Arithmetic mean and Median are given in Figures 1 to 10, as well as participants' results for EURAMET.QM-S11.

The error bars represent the combined standard uncertainties for the individual participant's results. The horizontal solid green line represents the SCRV, and the red dashed lines represent SCRV ± u(SCRU).

In 2nd meeting of the CCQM IAWG in 2018 (Ottawa, Canada), the results have been shared and discussed in detail. It was decided that the reference values will be calculated as the median for As, Cd, Ni and Pb. And, arithmetic mean was decided to be the reference value for Se.

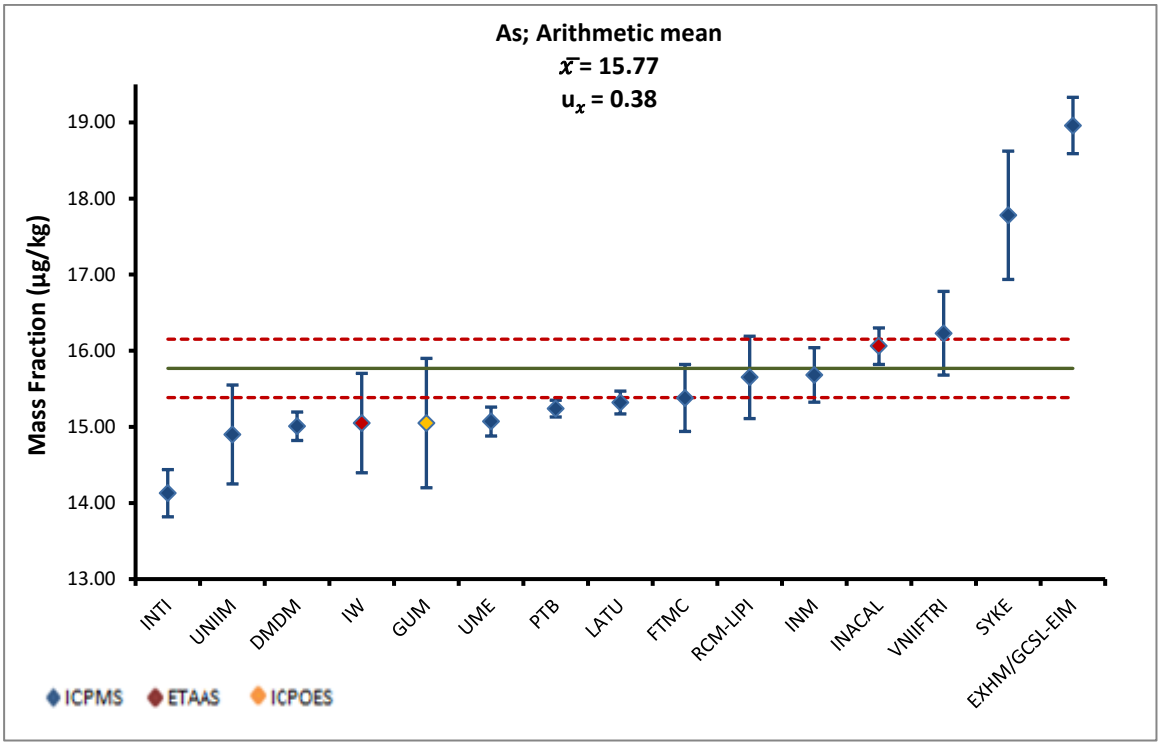


Figure 1. Participants' results and measurement uncertainties for As(arithmetic mean)

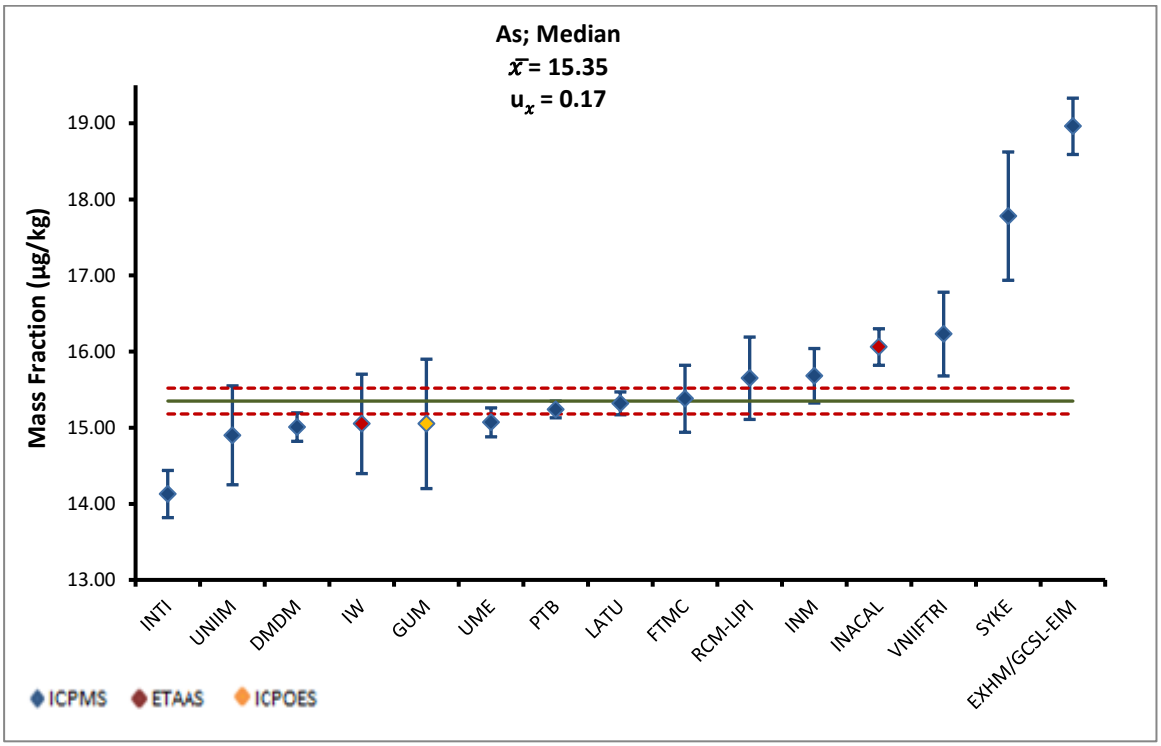


Figure 2. Participants' results and measurement uncertainties for As (median)

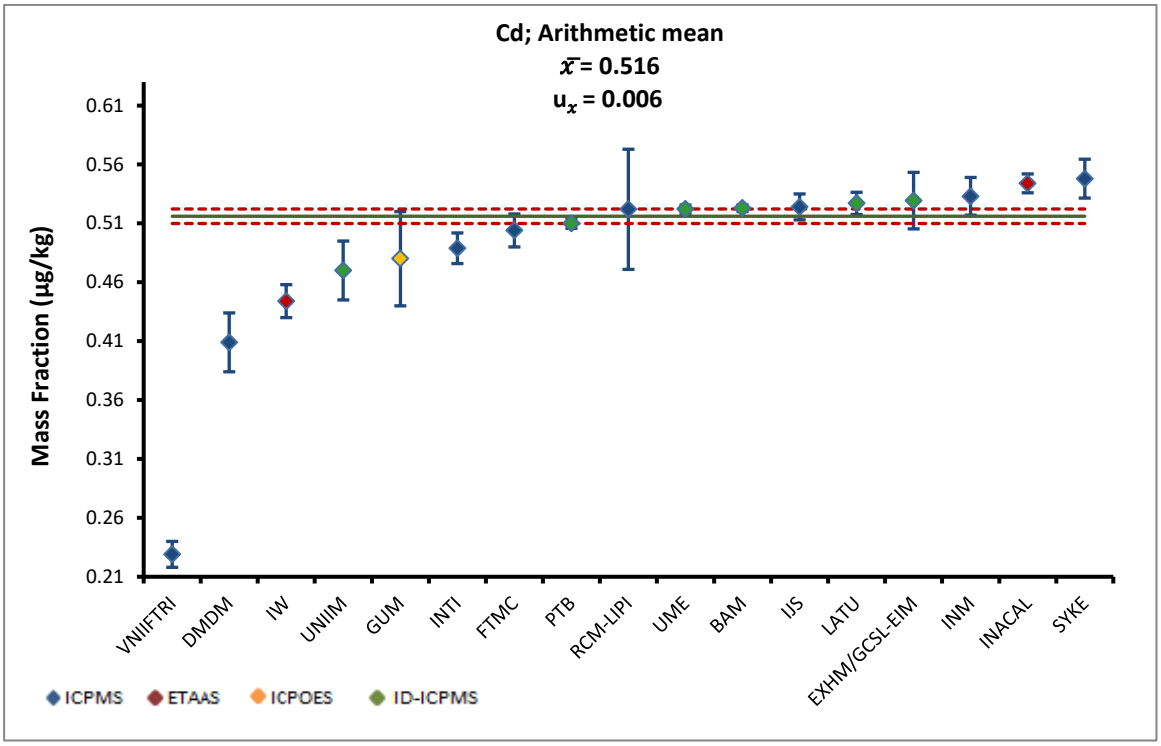


Figure 3. Participants' results and measurement uncertainties for Cd (arithmetic mean)

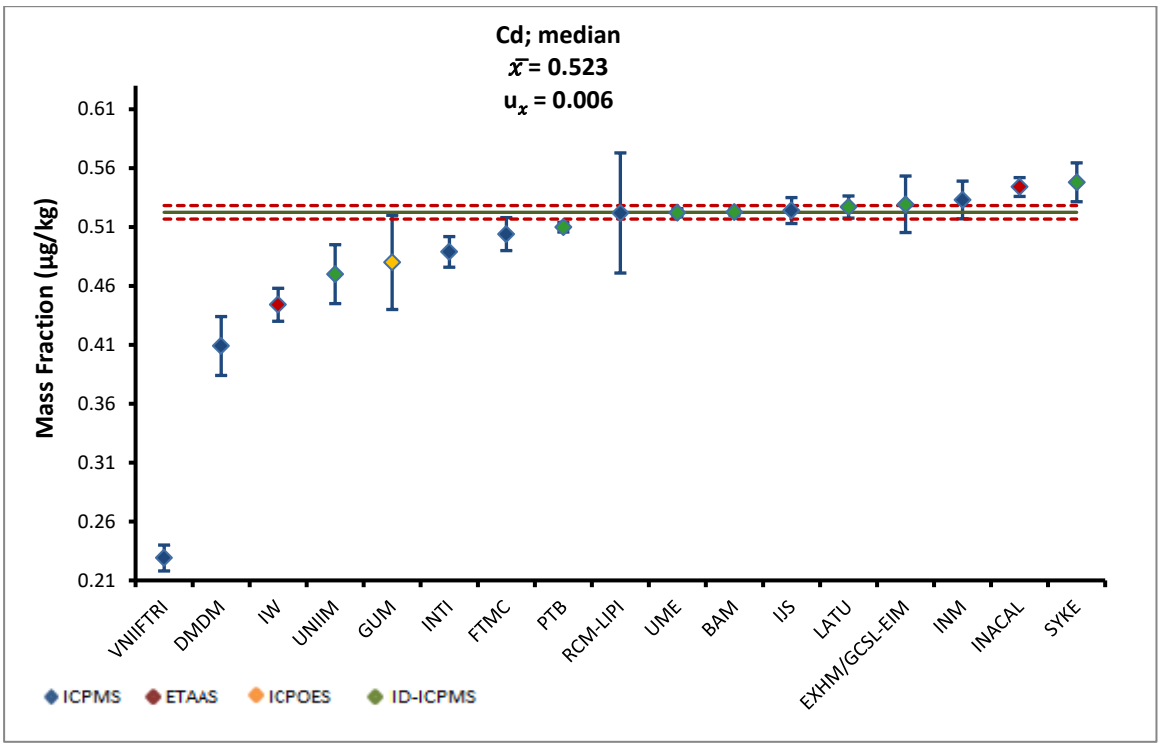


Figure 4. Participants' results and measurement uncertainties for Cd (median)

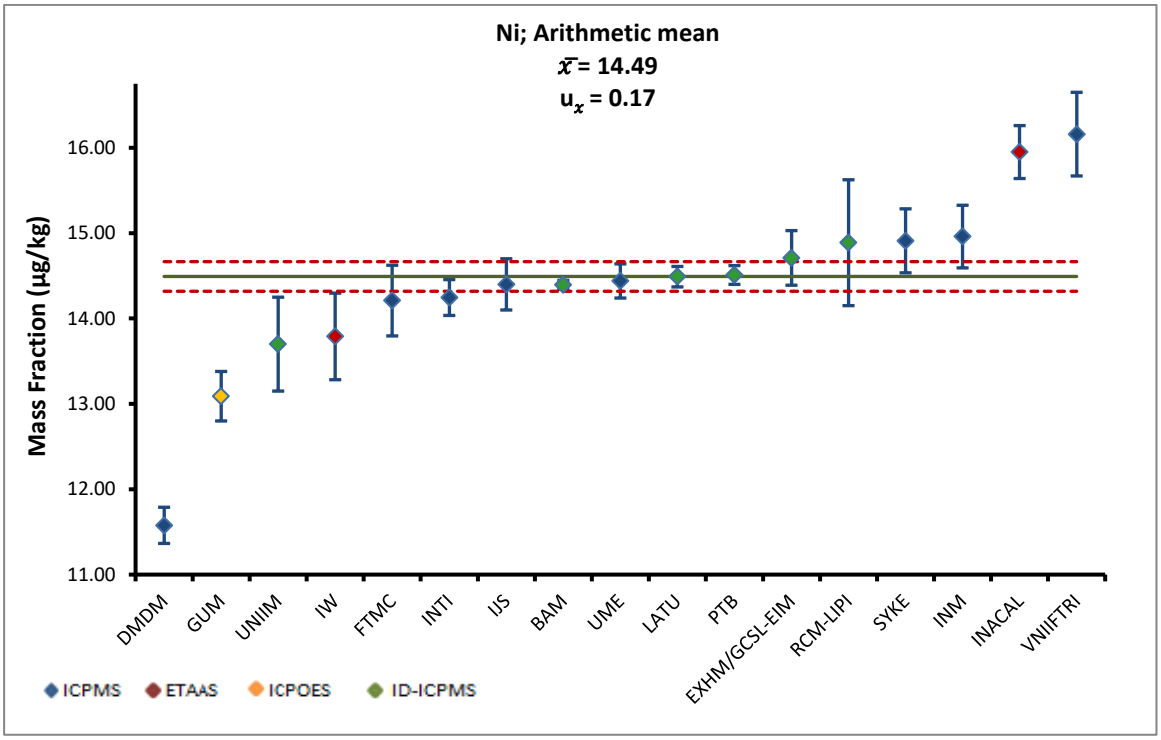


Figure 5. Participants' results and measurement uncertainties for Ni (arithmetic mean)

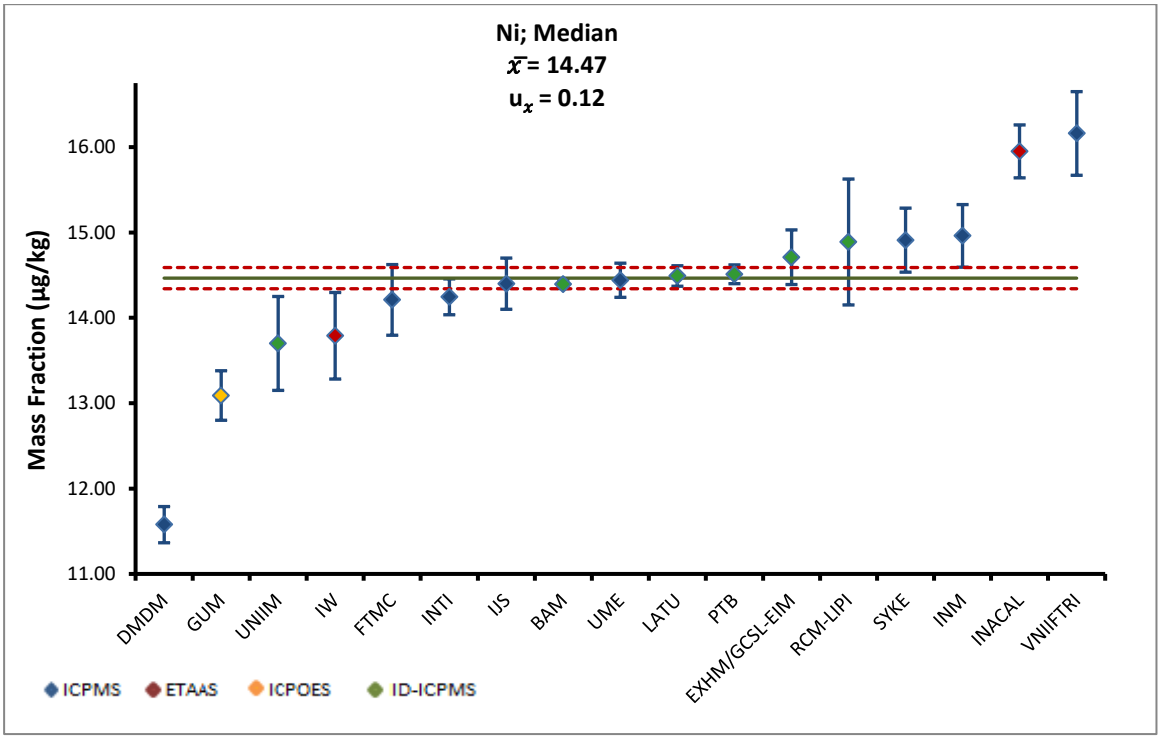


Figure 6. Participants' results and measurement uncertainties for Ni (median)

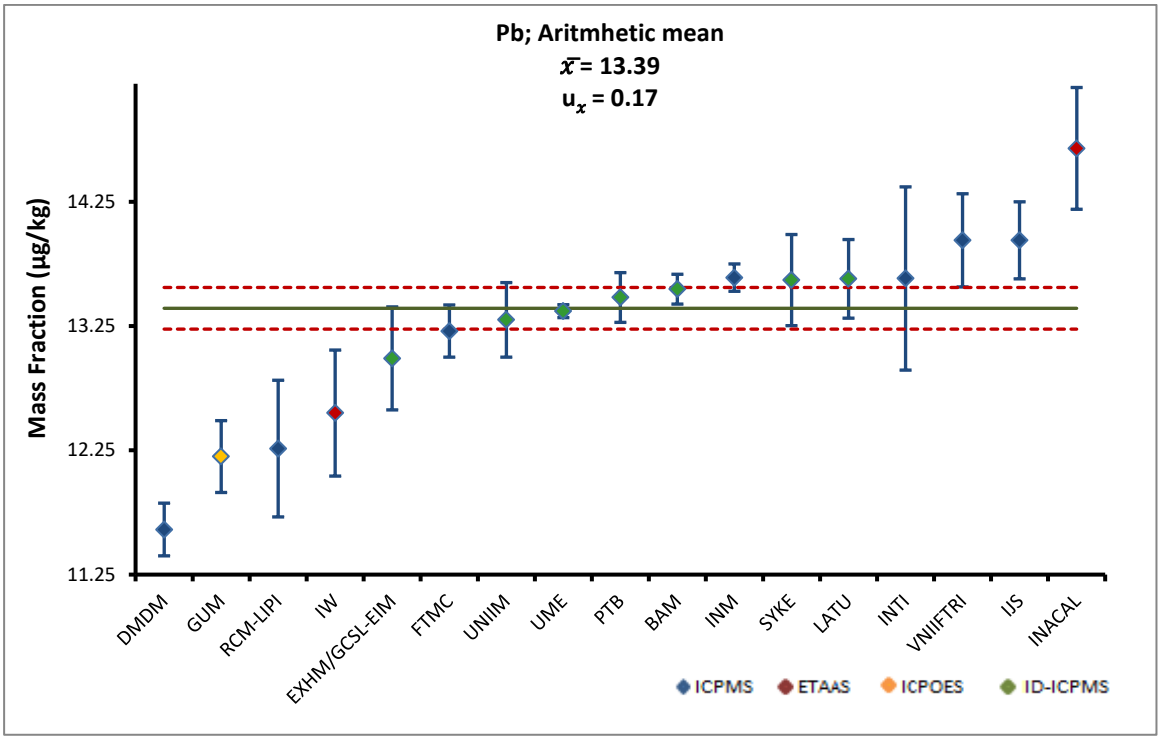


Figure 7. Participants' results and measurement uncertainties for Pb (arithmetic mean)

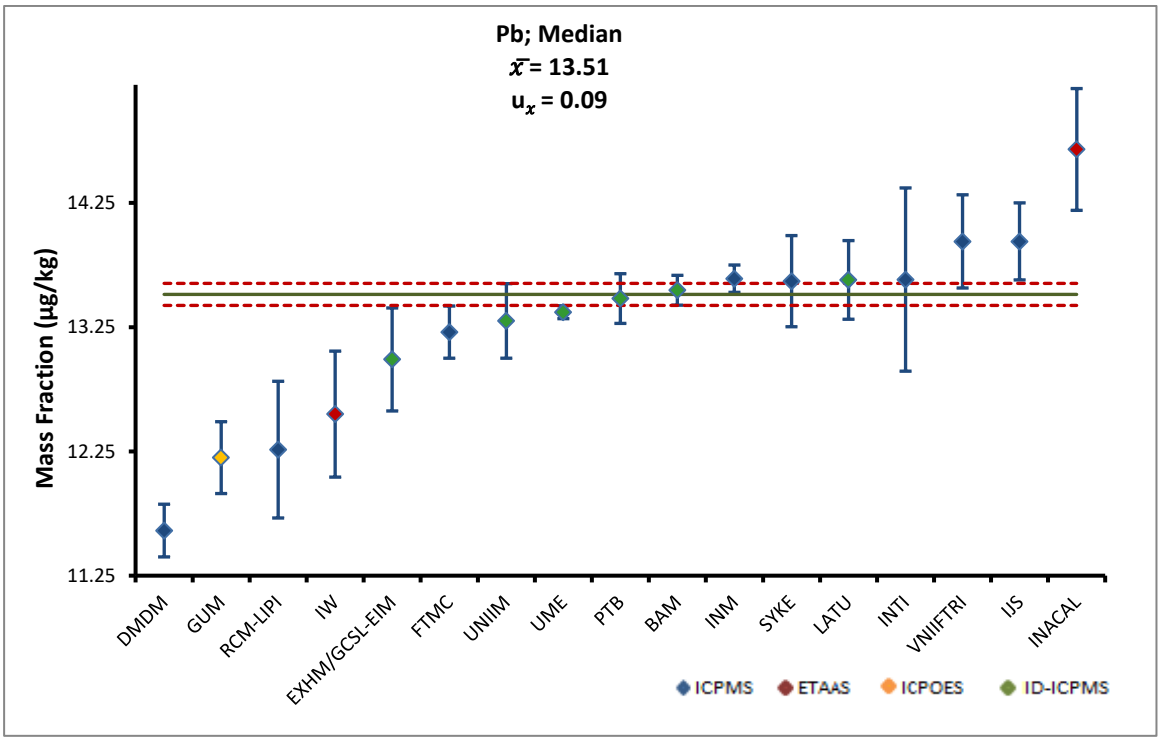


Figure 8. Participants' results and measurement uncertainties for Pb (median)

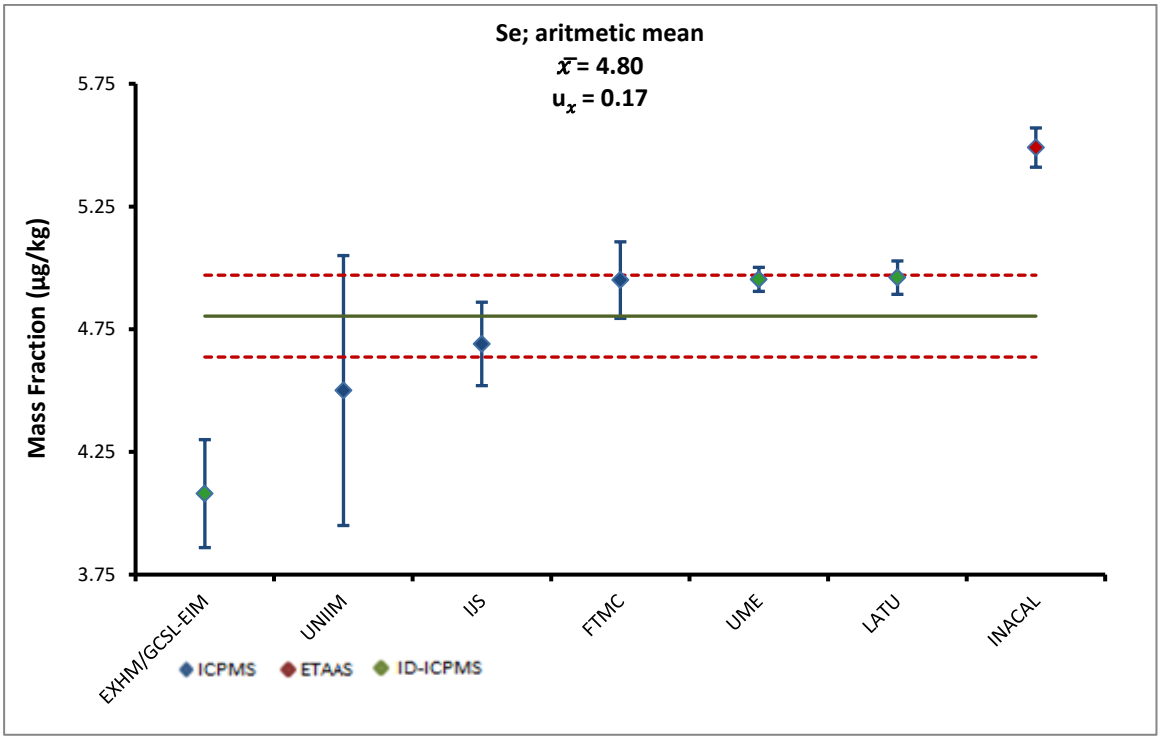


Figure 9. Participants' results and measurement uncertainties for Se (arithmetic mean)

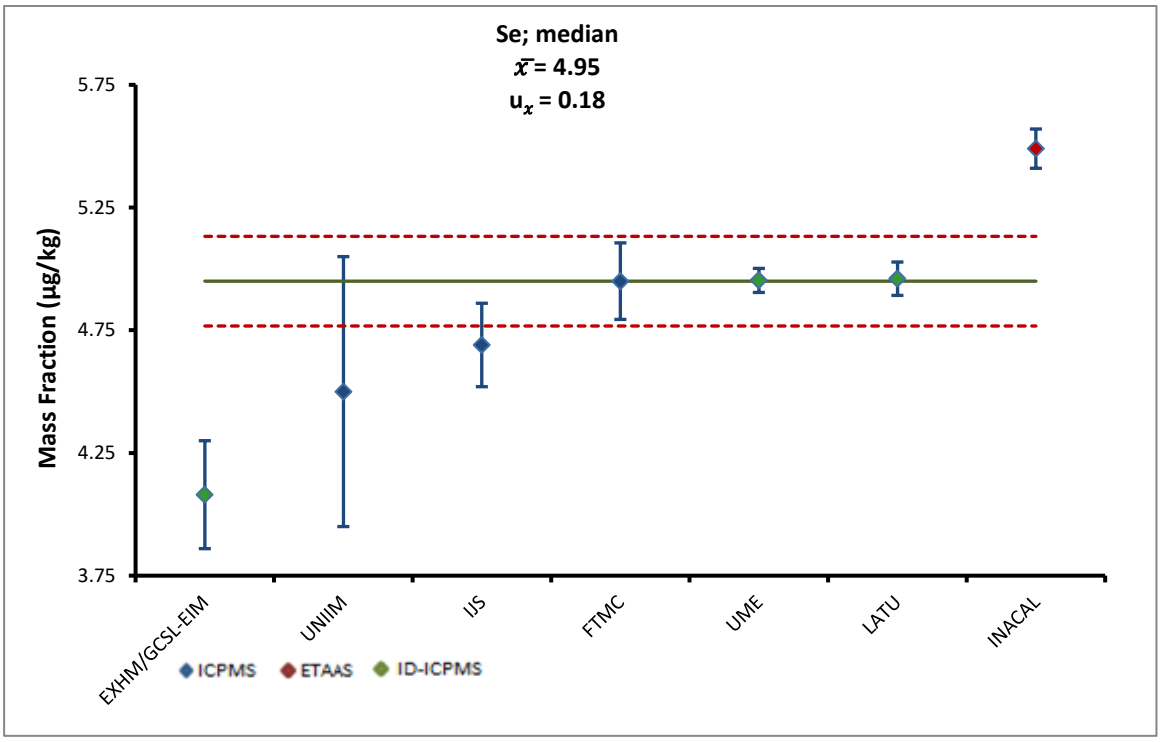


Figure 10. Participants' results and measurement uncertainties for Se (median)

6.5. Equivalence statements

The degree of equivalence and its uncertainty of a reported result by a participant compared to the SCRV were calculated using equations (11) and (12) as follows:

$$d_i = x_i - x_{SCRV} \quad (11)$$

$$U(d_i) = 2\sqrt{u(x_i)^2 + u(x_{SCRV})^2} \quad (12)$$

where:

d_i is the degree of equivalence (DoE) for participant *i* (*i* = 1,…,n),

x_i is the reported result from the *i*th participating institute (i = 1,…,n),

x_{SCRV} is the supplementary comparison reference value,

$U(d_i)$ is the uncertainty of DoE for participant *i* (*i* = 1,…,n).

Results from IW, DMDM and VNIIFTRI were not included in SCRV and SCRU calculations for using commercial calibration standards but they were involved in DoE calculations. The equivalence statements are listed in Table 12 to 16, and the graphical presentations are given in Figure 11 to 15.

Table 12. Equivalence statement for As

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty *u* (µg/kg)	d_i (µg/kg)	$U(d_i)$ (µg/kg)	$d_i / U(d_i)$
INTI	14.13	0.31	-1.22	0.71	-1.73
UNIIM	14.90	0.65	-0.45	1.34	-0.33
DMDM	15.01	0.19	-0.34	0.50	-0.68
IW	15.05	0.65	-0.30	1.35	-0.22
GUM	15.05	0.85	-0.30	1.73	-0.17
UME	15.07	0.19	-0.28	0.51	-0.55
PTB	15.24	0.11	-0.11	0.40	-0.27
LATU	15.32	0.15	-0.03	0.45	-0.07
FTMC	15.38	0.44	0.03	0.94	0.03
RCM-LIPI	15.65	0.54	0.30	1.13	0.26
INM	15.68	0.36	0.33	0.79	0.42
INACAL	16.06	0.24	0.71	0.59	1.21
VNIIFTRI	16.23	0.55	0.88	1.15	0.76
SYKE	17.78	0.84	2.43	1.72	1.41
EXHM/GCSL-EIM	18.96	0.37	3.61	0.81	4.43

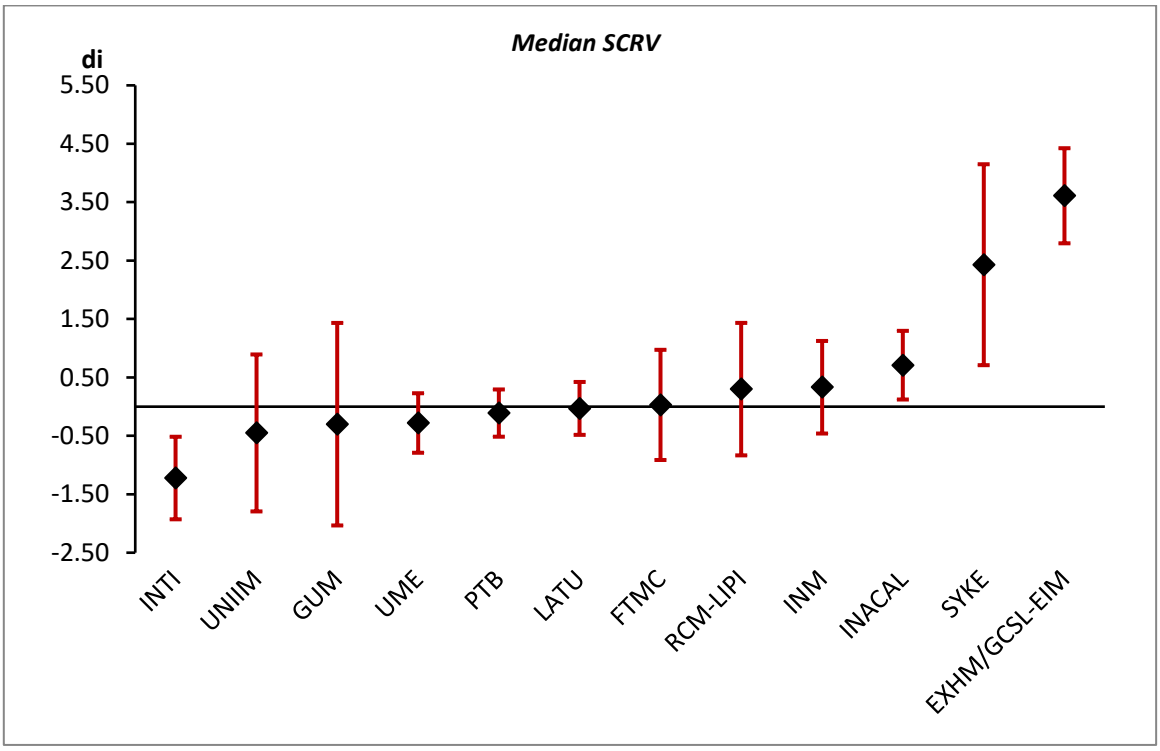


Figure 11. Equivalence statement for As

Table 13. Equivalence statement for Cd

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty *u* (µg/kg)	d_i (µg/kg)	$U(d_i)$ (µg/kg)	$d_i / U(d_i)$
VNIIFTRI	0.229	0.011	-0.294	0.025	-11.83
DMDM	0.409	0.025	-0,114	0,051	-2,21
IW	0.444	0.014	-0,079	0,030	-2,59
UNIIM	0.470	0.025	-0.053	0.051	-1.02
GUM	0.480	0.040	-0.043	0.081	-0.53
INTI	0.489	0.013	-0.034	0.028	-1.18
FTMC	0.504	0.014	-0.019	0.030	-0.61
PTB	0.510	0.004	-0.013	0.014	-0.89
RCM-LIPI	0.522	0.051	-0.001	0.103	-0.01
UME	0.522	0.004	0.000	0.013	-0.03
BAM	0.523	0.003	0.000	0.013	0.03
IJS	0.524	0.011	0.001	0.025	0.06
LATU	0.527	0.009	0.004	0.022	0.20
EXHM/GCSL-EIM	0.529	0.024	0.007	0.049	0.14
INM	0.533	0.016	0.010	0.034	0.31
INACAL	0.544	0.008	0.021	0.020	1.09
SYKE	0.548	0.017	0.025	0.035	0.73

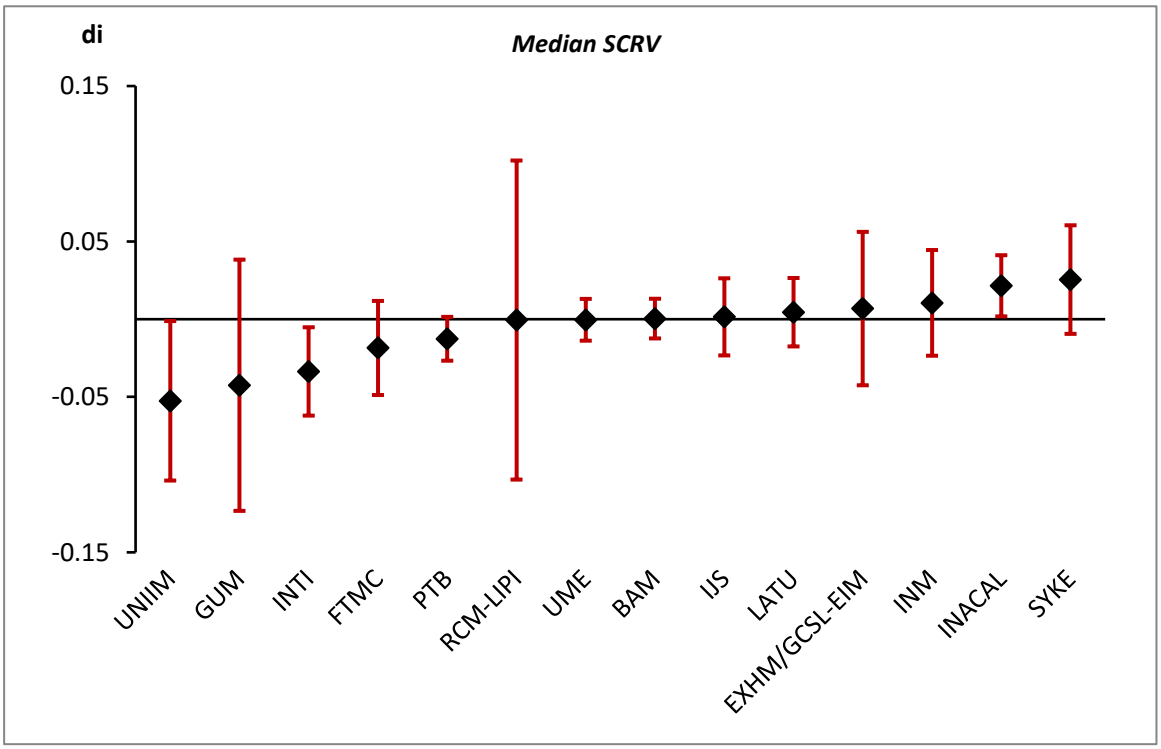


Figure 12. Equivalence statement forCd

Table 14. Equivalence statement for Ni

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty *u* (µg/kg)	d_i (µg/kg)	$U(d_i)$ (µg/kg)	$d_i / U(d_i)$
DMDM	11.58	0.21	-2.89	0.49	-5.88
GUM	13.09	0.29	-1.38	0.63	-2.18
UNIIM	13.70	0.55	-0.77	1.13	-0.68
IW	13.79	0.50	-0.68	1.03	-0.65
FTMC	14.21	0.41	-0.25	0.86	-0.29
INTI	14.25	0.21	-0.22	0.49	-0.45
IJS	14.4	0.3	-0.06	0.65	-0.10
BAM	14.40	0.05	-0.07	0.27	-0.26
UME	14.44	0.20	-0.03	0.47	-0.05
LATU	14.49	0.12	0.03	0.35	0.07
PTB	14.51	0.11	0.04	0.33	0.14
EXHM/GCSL-EIM	14.71	0.32	0.25	0.69	0.36
RCM-LIPI	14.89	0.74	0.42	1.50	0.28
SYKE	14.91	0.38	0.45	0.79	0.56
INM	14.96	0.37	0.50	0.77	0.64
INACAL	15.95	0.31	1.49	0.67	2.22
VNIIFTRI	16.16	0.49	1.70	1.01	1.68

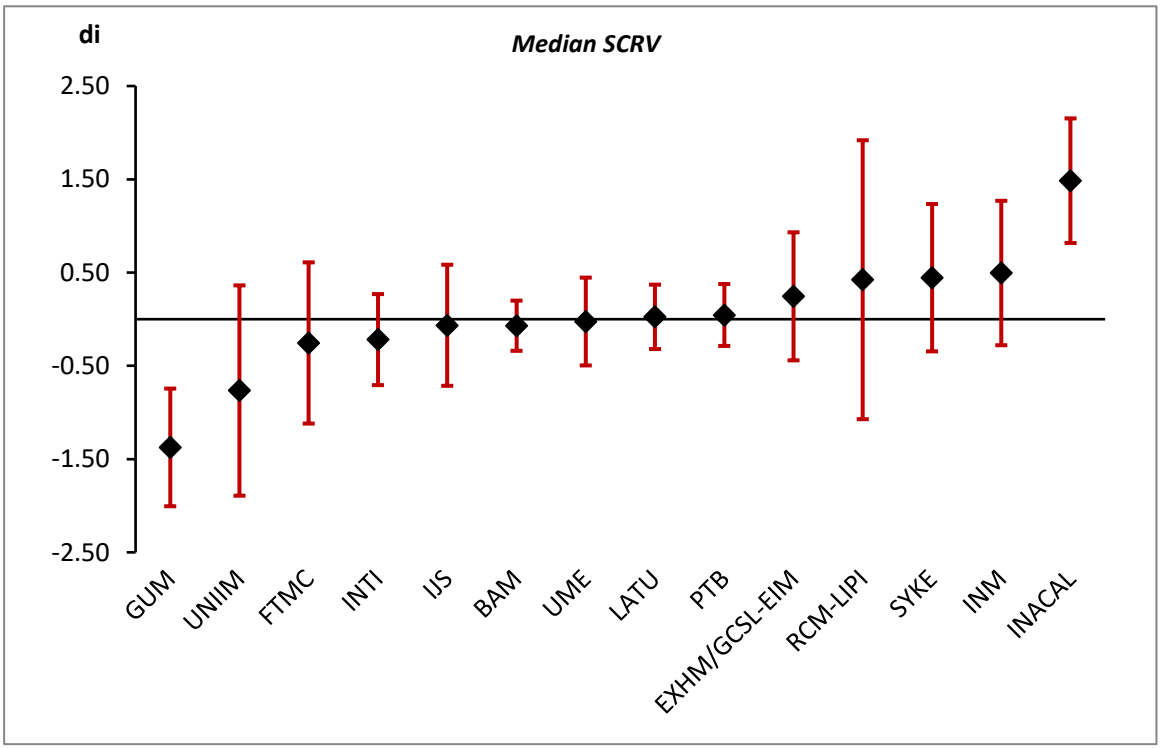


Figure 13. Equivalence statement for Ni

Table 15. Equivalence statement for Pb

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty *u* (µg/kg)	d_i (µg/kg)	$U(d_i)$ (µg/kg)	$d_i / U(d_i)$
DMDM	11.61	0.20	-1.90	0.44	-4.36
GUM	12.20	0.56	-1.31	1.13	-1.16
RCM-LIPI	12.2642	0.3180	-1.25	0.66	-1.89
IW	12.55	0.08	-0.96	0.24	-3.97
EXHM/GCSL-EIM	12.99	0.20	-0.52	0.44	-1.20
FTMC	13.21	0.25	-0.30	0.54	-0.57
UNIIM	13.3	0.4	-0.21	0.82	-0.26
UME	13.37	0.09	-0.14	0.25	-0.57
PTB	13.48	0.09	-0.03	0.25	-0.13
BAM	13.547	0.043	0.03	0.20	0.17
SYKE	13.62	0.17	0.11	0.38	0.28
LATU	13.63	0.11	0.12	0.28	0.41
INTI	13.633	0.200	0.12	0.44	0.27
INM	13.64	0.42	0.13	0.86	0.15
VNIIFTRI	13.94	0.36	0.43	0.74	0.58
IJS	13.94	0.46	0.43	0.94	0.46
INACAL	14.68	0.29	1.17	0.61	1.92

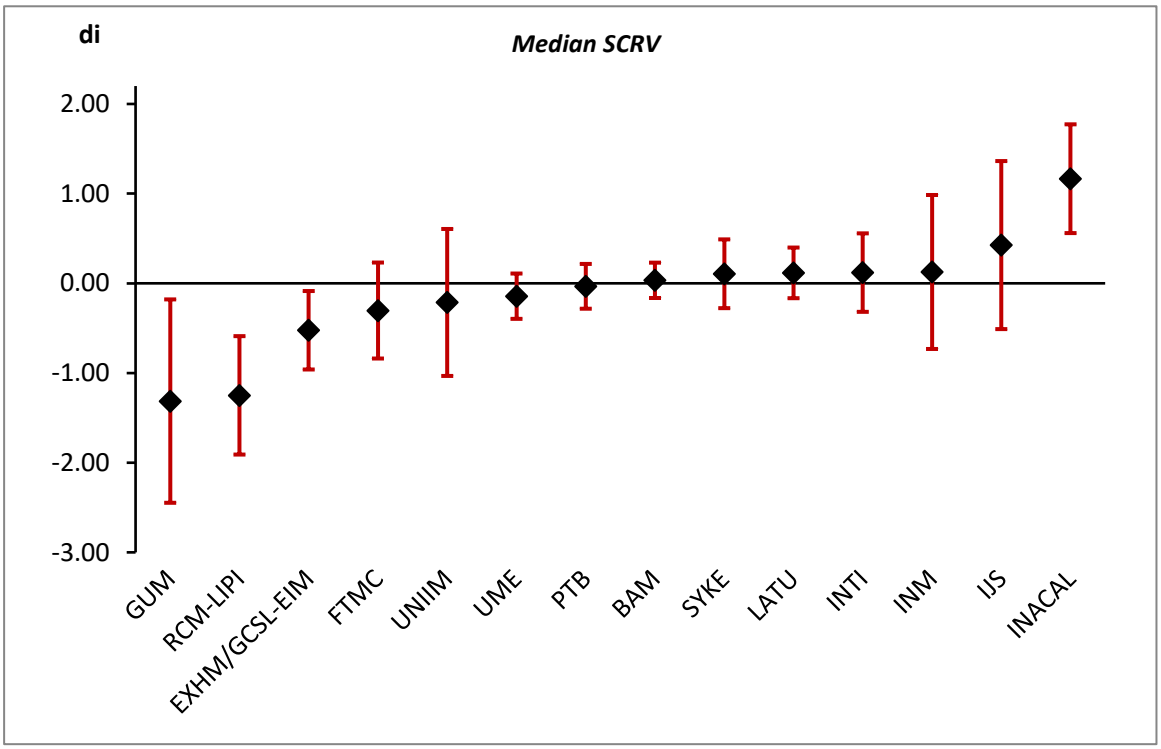


Figure 14. Equivalence statement for Pb

Table 16. Equivalence statement for Se

NMI/DI	Mass Fraction (µg/kg)	Standard Uncertainty *u* (µg/kg)	d_i (µg/kg)	$U(d_i)$ (µg/kg)	$d_i / U(d_i)$
EXHM/GCSL-EIM	4.08	0.22	-0.72	0.55	-1.31
UNIIM	4.5	0.55	-0.30	1.15	-0.26
IJS	4.69	0.17	-0.11	0.48	-0.24
FTMC	4.95	0.16	0.15	0.46	0.32
UME	4.953	0.049	0.15	0.35	0.43
LATU	4.96	0.07	0.16	0.36	0.44
INACAL	5.49	0.08	0.69	0.37	1.86

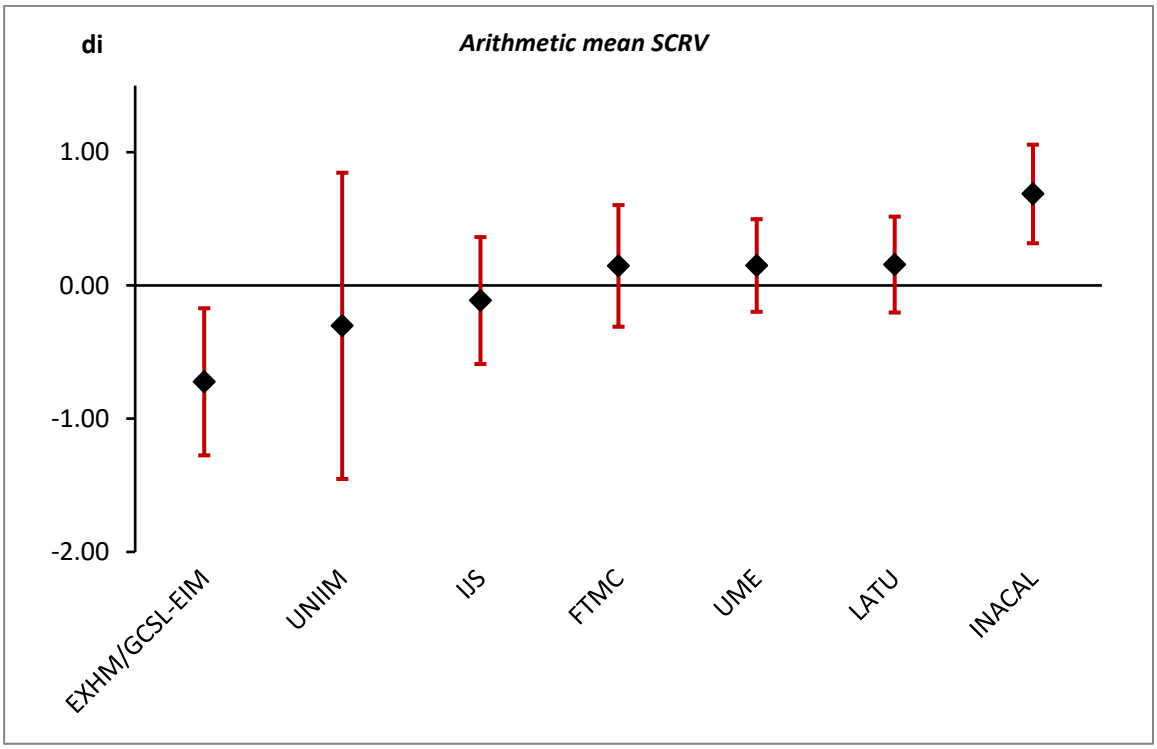


Figure 15. Equivalence statement for Se

7. CONCLUSION

EURAMET.QM-S11 supplementary comparison has been conducted in collaboration between EURAMET TC-MC and CCQM IAWG. As a comparison originated and organized within EURAMET, it also took attention of many NMIs/DIs in other regions to demonstrate their capabilities in measurement of elements in µg/kg level in river water matrix. Four elements (As, Cd, Ni and Pb) with regulated levels in drinking and surface waters were the mandatory measurands whereas Se was included as an optional measurand due its analytical challenges especially in mass spectrometric measurements.

The results show that most NMIs/DIs participated in the comparison successfully demonstrated their measurement capabilities for the elements of interest within the acceptable uncertainty limits. A small number of institutes obtained results outside the consensus values due to analytical reasons.

The most common technique used in the study is ICPMS with direct calibration and isotope dilution (IDMS) methods. Some institutes used ICP-OES and ETAAS techniques to perform their measurements.

Results of the comparison may be used to support claims for calibration measurement capabilities (CMC) based on the performance. The capabilities demonstrated should be comparable to water matrices with similar category and low difficulty. The core capability table for the comparison is given in Appendix I.

8. ACKNOWLEDGEMENTS

Coordinators of the comparison gratefully acknowledge the participants for their contribution to the study.

9. REFERENCES

1. Directive 2008/105/EC of the European Parliament and of the Council(https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:32008L0105)
2. ISO 17034:2016 - General requirements for the competence of reference material producers
3. ISO Guide 35:2017 - Reference materials - Guidance for characterization and assessment of homogeneity and stability
4. CCQM Guidance Note: Estimation of consensus KCRV and associated degrees of equivalence, Version 10, 2013v.10

Appendix I – CCQM IAWG Core Capability Table

Analyte groups	Matrix challenges						Calibration materials and solutions
	Water/aqueous	High Silica content (e.g. Soils, sediments, plants, …)	High salts content (e.g. Seawater, urine, …)	High organics content (e.g. high carbon) (e.g. Food, blood/serum, cosmetics, …)	Difficult to dissolve metals (Autocatalysts, …)	High volatile matrices (e.g. solvents, fuels, …)	
Group I and II: Alkali and Alkaline earth (Li, Na, K, Rb, Cs, Be, Mg, Ca, Sr, Ba)							
Transition elements (Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Y, Zr, Nb, Mo, Tc, Ag, Cd, Ta, W, Au, Hg, Al, Ga, In, Tl, Pb, Po)	**Cd, Ni, Pb**						
Platinum Group elements (Ru, Rh, Pd, Os, Ir, Pt)							
Metalloids / Semi-metals (B, Si, Ge, As, Sb, Te, Se)	**As, Se**						
Non-metals (P, S, C, N, O)							
Halogens (F, Cl, Br, I)							
Rare Earth Elements (Lanthanides, Actinides)							
Inorganic species (elemental, anions, cations)							
Small organo-metallics							
Proteins							
Nanoparticles							

Appendix II – Study Protocol

EURAMET.QM-S11 / EURAMET pilot

Supplementary comparison & pilot study on determination of Elements in River Water

Technical protocol

1. Introduction

The need for quality assessment of anthropogenic impact on environmental pollution is increasing due to discharge from various industries, the use of chemicals in agriculture and the consumption of fossil fuels. Diminishing resources such as natural waters used for the cultivation of agricultural products, plant and animal habitats are under severe pollution pressure and are at constant risk. The EU has stipulated the maximum allowable concentration of priority pollutants in different classes of surface water under the Water Framework Directive in Directive 2008/105/EC Annex I "Environmental quality standards for priority substances and certain other pollutants", and Annex II "List of priority substances in the field of water policy". Several parameters, such as Pb, Cd, Ni, Hg were listed in the priority substances and Cd and Hg were further identified as priority hazardous substances. Arsenic is also an important contaminant for its potential toxicological and carcinogenic effects.

The study is organized as part of the Matrix Reference Materials for Environmental Analysis (EnvCRM) project (www.envcrm.com) funded by Environmental European Metrology Programme for Innovation and Research (EMPIR). The candidate 'Elements in River Water' certified reference material (CRM) is one of the three reference materials targeted in the project, and serves as the test material for this study.

The aim of the study is to test the capabilities of participants in measuring the elements As, Cd, Ni, Pb and Se in river water. While the elements As, Cd, Ni and Pb are mandatory measurands, Se is optional. Although it was targeted first, Hg has been excluded from the list due to the stability issues.

The participants of supplementary comparison will be able to use the comparison results to support their CMC claims for the elements of interest. Along with the Core Capability Tables, it may be possible to claim CMCs in related matrices as well.

2. Test material

River water, candidate certified reference material, was collected from a creek feeding Darlık dam reservoir providing city water to Istanbul. The material was acidified to have a final concentration of 2% (v/v) HNO_3, and stored in +4 °C refrigerated room for 2 months until further processing. All the water was filtered through a coarse filter and a 0.45 um-pore size filter, respectively. After adding the elements to reach in appropriate concentrations, the whole batch (about 140 L) was homogenized for 6 hours. After this step, the water sample was filled in HDPE bottles to have a volume of 100 mL. Capped bottles were gamma irradiated for sterilization.

The homogeneity of the test material was performed by selecting 12 bottles among a batch of 1200 with random stratified sampling approach. The measurements were conducted with HR-ICPMS, and the data were evaluated with ANOVA. Between bottle homogeneity values for As, Cd, Ni, Pb and Se were determined as 0.34%, 0.43%, 0.90%, 0.30% and 0.79%, respectively.

The stability of the material was tested for 4 weeks at 40 °C and 60 °C to check the stability during the transport. The results revealed no sign of significant change for any measurand for the test period. Long term stability tests are in progress, and the tests will be continued until result submission date.

Each participant will receive two sample bottles containing approximately 100 mL of the river water. The sample receipt form is expected to be filled and returned to TUBITAK UME via e-mail once they are received. It is recommended that the bottles are kept +4 °C until the sample preparations for measurements.

3. Coordinating laboratories

The comparison is co-organized by TUBITAK UME and IMBIH. TUBITAK UME, as the pilot laboratory, will conduct the preparation of samples, and homogeneity and stability tests. Distribution of samples will be performed by TUBITAK UME. IMBIH and TUBITAK UME will evaluate the participant results, and will draft the measurement reports.

4. Measurands and methods

The participants are required to measure and report the mass fractions of the 4 mandatory

and 1 optional measurands. The measurands and their expected mass fractions are listed in Table 1.

Table 1. Measurands and respective mass fraction ranges

Measurand	Expected mass fraction (µg/kg)
Arsenic	2 – 20
Cadmium	0.1 – 5
Nickel	2 – 20
Lead	2 – 20
*Selenium**	*2 – 20*

*Optional

The participants can use the method of their choice.

5. Reporting and submission of results

A reporting form will be provided to participants after test materials are distributed. Each participant will be expected to report individual results, detailed uncertainty budget, details about the method used, etc. At least 3 results from each of the two bottles will be expected for each measurand. All analytical calibrations should be performed using metrologically traceable standards.

Reference value for each measurand will be either the mean or the median of the submitted Supplementary comparison data. If any participant submits results by multiple methods, the result with the smallest uncertainty will be chosen for the calculation of the reference value. Results from participants of pilot study will not be used for reference value determination.

All participants in EURAMET.QM-S11 are required to submit a Core Capability Table for the measurement technique they use. Forms of appropriate Core Capability Table are available at CCQM-IAWG website.

6. Schedule

June 15, 2017	Registration deadline
June/July 2017	Distribution of samples
November 30, 2017	Results submission deadline
February 2018	Presentation of results at the EURAMET TC-MC SCIA Meeting
April 2018	Presentation of results at the CCQM-IAWG Meeting

7. Participants

Participation to EURAMET Supplementary comparison is open to all interested NMIs or DIs. Any other institutions or laboratories may participate in the pilot study with approval.

8. Registration

Please complete and return the enclosed registration forms to Süleyman Z. Can (suleyman.can@tubitak.gov.tr) and Alper Isleyen (alper.isleyen@tubitak.gov.tr) no later than the registration deadline, 12 June 2017.

For enquiries, participants may contact the coordinating laboratories as follows:

Dr. Süleyman Z. CAN
TUBITAK UME
Baris Mah. Dr.Zeki Acar Cad. No:1
41470 Gebze-Kocaeli, TURKEY
suleyman.can@tubitak.gov.tr
+90 262 679 5000 Ext. 6207 (P)
+90 262 679 5001 (F)

Aida Jotanović, MSc
Institute of metrology of B&H
Augusta Brauna No. 2, 71000 Sarajevo
Bosnia and Herzegovina
aida.jotanovic@met.gov.ba
+387 (0) 33 568 925 (P)
+387 (0) 33 568 909 (F)

Appendix III – Registration Form

EURAMET.QM-S11 / EURAMET Pilot
Elements in River Water

Registration Form

Please provide the following participant information:

Name of Institute : _____

NMI/DI/Other : _____

Contact Person : _____

Email Address : _____

Phone Number : _____

Fax Number : _____

Postal Address : _____

Postal Code : _____

City/Country : _____

Date : _____

Please select the appropriate boxes (double click to check)

Measurand	**EURAMET.QM-S11**	**EURAMET Pilot**
Arsenic	☐	☐
Cadmium		
Nickel		
Lead		
Selenium ***(Optional)***	☐	☐

Non-MRA signatories can only participate in the EURAMET Pilot Study.

Please note that TUBITAK UME takes no responsibility for any import taxes or unforeseen additional charges imposed during transportation of comparison samples.

Please, return the completed from to Suleyman Z. Can (**suleyman.can@tubitak.gov.tr**) and Alper Isleyen (**alper.isleyen@tubitak.gov.tr**) by **June 15, 2017**.