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The International Measurement Evaluation Programme

IMEP-19
Trace Elements in Rice
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Report to Participants

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The Mission of IRMM is to promote a common European measurement system in support of EU policies, especially internal market, environment, health and consumer protection standards.

IMEP®

provides reference values with demonstrated traceability and demonstrated uncertainty, independent of the participants' results

enables result-oriented rather than procedure oriented evaluation of performance

demonstrates a degree of equivalence in measurement results on an international scene

Contents

Summary

The International Measurement Evaluation Programme (IMEP®) was established in order to shed light on the current state of practice in chemical amount measurements. IMEP® runs in support of EU policies (e.g. Consumer Protection and Public Health, Single Market, Environment, Research and Technology, External Trade and Economic Policy).
The aim of this interlaboratory comparison programme was to picture objectively the degree of equivalence and the quality of chemical measurements by comparing participant's measurement results with the external reference values. These reference values are required to demonstrate traceability and they should have a demonstrated and adequately small uncertainty, as evaluated according to international guidelines. Hence, these reference values are completely independent from the participants' result. In IMEP® participating laboratories receive a Certified Test Sample (with undisclosed concentration values), which they can analyse using their routine analytical procedures. Participants in IMEP® can compare, on an international forum, their values to the IMEP® reference values and in this way assess the quality of their results.
The European Commission has identified food safety as one of its top priorities. The White Paper on Food Safety of January 12th, 2000 sets out the plans for a proactive new food policy. Measurements of contaminants in foodstuff play a key role in modernising legislation into a coherent and transparent set of rules.

Rice is the main foodstuff for about half of the world's population. In order to protect public health it is essential to keep contaminants at levels, which are toxicologically acceptable. Cadmium and lead may induce dysfunctions in humans. Within the European Union (EU) maximum levels of cadmium and lead in foodstuff are set in the Commission Regulation (EC) No. 466/2001. Zinc and copper are well known as essential trace elements for humans. Recently the European Commission (EC) has requested the Scientific Committee on Food (SCF) to review the upper level on the daily intake of zinc and copper SCF/CS/NUT/UPPLEV/11 Final, Nov/2000.

Participants in IMEP-19 "trace elements in rice" were offered to measure the content of *Cd, Cu, Pb* and *Zn.* Measurement results were reported by 267 participants from 43 countries in 5 continents. This report is presented in a graphical form, the results of all participants are shown together with the reference value. The results were sorted according to the different criteria e.g. degree of experience, self-declared status of accreditation, authorisation for the element under investigation, analytical technique used, applied water content determination, the quality management system in use in the laboratory, the guidelines used for quantifying measurement uncertainty and by geographical location.

1 Introduction

Since 1988, IMEP® comparisons have mainly concentrated on amount contents of minor and trace elements in various matrices such as water [1, 2, 3, 4, 5], polyethylene [6, 7], serum [8, 9, 10, 11], sediments [12], car catalysts [13] and wine [14]. One comparison addressed isotope ratio measurements in CO_2 [15]. Information concerning previous IMEP® interlaboratory comparisons can be found on the IMEP® web-site http://www.imep.ws.
IMEP-19 interlaboratory comparison focuses on measurements of Trace Elements in Rice aiming to support the European Regulation (EC) No. 466/2001 [16].

1.1 What is IMEP®

IMEP® is a tool with which field laboratories can compare their results against independent reference values (SI-traceable, where possible). Certified Test Samples (CTS) with undisclosed values are sent to interested participants. The participants are asked to return values for the parameters under investigation together with uncertainty statements claiming to contain the so-called "true values". The SI-traceable reference values, are obtained by measurement procedures based on a primary method of measurement (PMM, CCQM definition [17]). The underlying philosophy is that the best possible values will serve as reference and these are obtained from well-understood measurement processes rather than via a mere consensus approach.

Further characteristics of the IMEP® programme are:

- IMEP® demonstrates the degree of equivalence in results on chemical measurements for individual participating laboratories on a global scale.
- IMEP® participating laboratories work under normal conditions of their own choice with respect to techniques, procedures and instrumentation (preferably routine conditions) and are requested to report their results with a best estimate of combined uncertainty according to ISO/BIPM [18] or other international guidelines.
- IMEP® is open to all laboratories, IMEP® guarantees the confidentiality to the identity of its participants.
- IMEP® graphically displays reference values and results from participating laboratories. E_n-numbers [19] are issued to those participants who reported measurement results with uncertainties, that were calculated according to the Guides for Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) [18] and/or EURACHEM/CITAC (2000). IRMM could offer training activities to participants who request additional support after the completion of the respective IMEP® comparison [20].

2 IMEP-19: Trace Elements in Rice

The European Commission (EC) has identified food safety as one of its top priorities. The White Paper on Food Safety of January 12th, 2000 sets out the plans for a proactive new food policy. Measurements of contaminants in foodstuff play a key role in modernising legislation into a coherent and transparent set of rules. This reinforces controls from the farm to the table and increasing the capability of the scientific advice system, so as to guarantee a high level of human health and consumer protection [21]. Rice seems to be the oldest cereal cultivated. It is the main foodstuff for about half of the world's population. The vast majority of the world's rice is grown and consumed in Asia. In Latin America and Africa rice is also among the major nutrients. In recent decades rice consumption has been expanding beyond the traditional rice-grown areas, particularly in Europe. In order to protect public health it is essential to keep contaminants at levels which are toxicologically acceptable, thus surveillance measures have been taken regarding the presence of contaminants in foodstuff, including rice. Cadmium may induce dysfunctions and reproductive deficiencies in humans and is suspected to act as a human carcinogen. Lead may induce reduced cognitive development in children and increased blood pressure and cardiovascular diseases in adults. Therefore maximum levels of lead and cadmium in foodstuff, which is the main source of human intake of cadmium, are set in the relevant Commission Regulation (EC) [16]. Zinc and copper are well known as essential trace elements for humans. Zinc is proven to be an essential factor in over 100 enzymes. Copper is a constituent in the encym Coeruloplasmin, this is essential for the proper conduction of the cellular metabolism. Recently the European Commission (EC) has requested the Scientific Committee on Food (SCF) to review the upper level on the daily intake of zinc and copper [22].

Participants in IMEP-19 were offered to measure the amount content of *Cd*, *Cu*, *Pb* and *Zn*. This report presents all results (in graphical form) from participants in IMEP-19 "T*race Elements in Rice*". In addition, the applied water content determination and dry-mass correction in IMEP-19 are summarised in Annex 2 of this report.

The same Certified Test Sample was also offered for a key comparison and a pilot study to the Consultative Committee of Amount of Substance of the CIPM, (CCQM-K24 and CCQM-P29) and to EUROMET as a supplementary comparison (EUROMET project 565). 17 national metrology institutes, signatories to the Mutual Recognition Agreement (MRA), participated in CCQM-K24 and CCQM-P29 [23, 24]. Results of these comparisons are accessible via the BIPM key comparison database [25]. 12 members of EUROMET (including renowned National Measurement Institutes) from 12 European countries measured the IMEP-19 CTS. A separate report (EUROMET 565) was prepared for this exercise and can be found on the EUROMET web-site [26]. Ultimately, the IMEP-19 participants can compare their results with the results of laboratories that represent their country at EUROMET and CCQM level and vice versa.

3 Preparation of the Certified Test Samples in IMEP-19 and the establishment of the IMEP-19 reference values

3.1 The rice samples

The rice powder used in IMEP-19 was provided by NMIJ-Japan and originates from rice grown in cadmium contaminated water. The cadmium amount content in this rice material was envisaged to be close to the upper limit as stated in Japanese regulations. At IRMM the rice was reprocessed into units of 15g and bottled in glass containers. "Within-bottle homogeneity" tests were carried out on 20 sub-samples of 8 bottles using solid sample Zeemann Atomic Absorption Spectrometry (SS-ZAAS). "Between-bottle homogeneity" tests were performed applying Isotope Dilution Mass Spectrometry (IDMS) by analysing 2 sub-samples from 5 bottles [27]. Results from both measurements were evaluated accordingly and compared to the procedures established in ISO 35, for the certification of reference materials and based on analysis of variance ANOVA [28, 29]. No significant differences were observed.

3.2 IMEP-19 Reference laboratories

Reference laboratories in IMEP® are selected for the establishment of certified values for trace metals they have to have expertise and a proven record (e.g. publications) on application of IDMS. They have to be able to demonstrate their measurement capability for the specific measurand in the matrix to be certified. In the past IRMM and BAM have already successfully proven their measurement capabilities to measure trace elements in various matrices. Both reference laboratories participated previous to the establishment of the certified IMEP-19 reference values to the CCQM-K24, CCQM-P29 and EUROMET comparisons, the same CTS was used in IMEP-19. Their measurement capability, to measure trace elements in rice has been confirmed by comparison to 15 other National Metrology Institutes world-wide [25]. Both IMEP-19 reference laboratories (Table 1) provided a complete uncertainty budget according to the Guide to the Expression of Uncertainty in Measurement (GUM) [18].

Table 1. IMEP-19 Reference laboratories

Institution	**Country of origin**
IRMM Institute for Reference Materials and Measurements – Geel	**European Commission**
BAM Bundesanstalt für Materialforschung und –prüfung – Berlin	**Germany**

3.3 Analytical methods used

The reference measurements for *Cd*, *Cu* and *Pb* were carried out at IRMM using IDMS as a Primary Method of Measurement on a quadrupole inductively coupled plasma mass spectrometer (ICP-MS) [30]. The reference measurements for *Zn* were carried out at BAM using IDMS as a Primary Method of Measurement on a multi collector thermal ionisation mass spectrometer (TIMS).

3.4 IMEP-19 Reference values

Certified values were established for *Cd*, *Cu*, *Pb* and *Zn*. A relevant certificate was issued (see Annex 3) and distributed to all participants. The certified values for the dry-mass corrected amount contents are summarised in Table 2. The stated uncertainties are expanded uncertainties ($U = k \cdot u_c$) with a coverage factor k equal to 2.

Table 2. Certified reference values for IMEP-19.

Element	***Certified value*** $mg \cdot kg^{-1}$ ***(dry-mass)***	***Expanded uncertainty U, k=2*** $mg \cdot kg^{-1}$ ***(dry-mass)***
Cadmium	***1.618***	***0.028***
Copper	***2.816***	***0.034***
Lead	***0.415 9***	***0.007 1***
Zinc	***22.99***	***0.44***

4 Organisation & administration

4.1 Timing & deadlines

IMEP-19 was initiated in 2002. The planning of the comparison was performed in spring 2002. The public announcement (invitation to participate) of IMEP-19 was dated 15th April 2002, this letter, together with a registration form were dispatched. After the collection of the registration forms the CTS were distributed with accompanying documents to the participants during the month of June 2002. The initial deadline for the participants to report their results was the 30th September 2002. Subsequently result reporting was extended to the 11th October 2002. Each participant received a personal key-code in order to report their results and questionnaire information electronically through the IMEP® web-site. All announcements and guideline information were accessible from the IMEP® web-site [31]. The first feedback to the participants contained the reference value certificate, which was distributed by e-mail in October 2002. The IMEP-19 certificate is also accessible via the IMEP® web-site. In addition an individual certificate was distributed by post to each IMEP-19 participant in November 2002 (see Annex 4).

4.2 Registration of participants

IMEP® is currently co-ordinated in a "network" approach. Laboratories can register for participation through different communication channels. The most common are via the relevant "*regional co-ordinator*" (RCs) or directly with IRMM.

The RCs are very valuable partners for IMEP®. RCs are typically people or institutions directly involved in chemical measurements and preferably experienced and competent in metrological matters, with profound knowledge of the measurement systems of their country or region. The tasks of the RCs are to act on behalf of IRMM in order to liase with participants and administer locally in each comparison, while bridging linguistic, cultural differences and taking into account any local particularities.

About 78% of the 267 participants who reported results in IMEP-19 registered through the 32 RCs, which are listed in Table 3. A further 6% of the participants of IMEP-19 registered directly with IRMM. The remaining 16% were laboratories nominated by the European Co-operation for Accreditation (EA) in the framework of the IRMM-EA collaboration agreement.

Table 3. List of Regional Co-ordinators for IMEP-19.

Institution / Organisation	**Origin**
Instituto Nacional de Tecnologia	Buenos Aires, **Argentina**
National Association of Testing Authorities	Rhodes, **Australia**
Interuniversitäres Forschungsinstitut für Agrarbiotechnology	Tulln, **Austria**
INMETRO	Xerem Duque de Caxias,**Brazil**
National Centre of Metrology	Sofia, **Bulgaria**
State Office for Standardisation and Metrology	Zagreb, **Croatia**
State General Laboratory	Nicosia, **Cyprus**
Czech Metrology Institute	Prague, **Czech Republic**
University of Tartu	Tartu, **Estonia**
Bureau National de Métrologie	Paris, **France**
Aristotle University of Thessaloniki	Thessaloniki, **Greece**
National Office of Measures	Budapest, **Hungary**
National Metrology Institute of Japan	Tsukuba, **Japan**
Latvian National Accreditation Bureau	Riga, **Latvia**
Semiconductor Physics Institute	Vilnius, **Lithuania**
Centro Nacional de Metrología	Querétaro, **Mexico**
National Research Centre for Environmental Analysis and Measurements	Beijing, **P.R. China**
University of Warsaw	Warsaw, **Poland**
RELACRE - Associação dos Laboratórios Acreditados de Portugal	Lisbon, **Portugal**
Korea Research Institute for Standards and Science	Taejon, **Rep. of Korea**
PSB Corporation	Singapore, **Rep.of Singapore**
National Institute of Metrology	Bucharest, **Rumania**
Slovak Institute of Metrology	Bratislava, **Slovak Republic**
Metrology Institute of the Republic of Slovenia	Celje, **Slovenia**
CSIR National Metrology Laboratory	Pretoria, **South Africa**
Centro Español de Metrologia	Madrid, **Spain**
Swedish National Testing and Research Institute	Borås, **Sweden**
Eidgenössische Materialprüfungs- und Forschungsanstalt	St. Gallen, **Switzerland**
Institute of Nutrition	Nakhon Pathom, **Thailand**
NMi -Van Swinden Laboratorium	Delft, **The Netherlands**
Turkish Accreditation Agency	Ankara, **Turkey**
Laboratory of the Government Chemist	Teddington, **United Kingdom**

4.3 Collaboration with European Co-operation for Accreditation

Accreditation is a very useful tool for laboratories to demonstrate technical competence to their customers. In order to further improve the efficiency of accreditation in chemistry with respect to the evaluation and demonstration of the performance of laboratories, the European Co-operation for Accreditation (EA) and IRMM agreed to intensify their ongoing co-operation.

To officialise this co-operation, a formal “letter of intent for co-operation” was signed by the Chairman of the EA, Dr. D. Pièrre and the former director of IRMM, Prof. M. Grasserbauer in the beginning of 2001. [32].

The co-operation focuses on chemical measurements and aims to improve the metrological basis of accreditation in chemistry. This will be mainly achieved by the organisation of interlaboratory comparisons using traceable reference values obtained in terms of high quality measurements applying the principles of metrology. The National Accreditation Bodies (NAB) may nominate laboratories to participate in such activities, evaluate their performance against independent reliable reference values and request the laboratories to take appropriate corrective actions if needed.

In the framework of this collaboration IRMM offered a number of IMEP-19 CTS to the National Accreditation Bodies (NABs) in Europe (max. 5 samples per NAB) in order to include the EA accredited laboratories in IMEP-19.

This operation was co-ordinated by Mrs. Nicole Meuree-Vanlaethem from BELAC (Belgium Accreditation), with 42 EA laboratories participating from 13 countries. The data processing of the results of these laboratories was done by IRMM in the same way as the results from the other IMEP-19 participants.

4.4 Sample distribution and accompanying documents

The CTS were sent using express mail to all participants. Enclosed were a few accompanying documents (see Annex 4):

- An accompanying letter to the participants giving information relevant to the comparison, pointing out the deadlines and giving instructions on how to report their results via the IMEP® web-site. A result report form was provided to give a consistent way of reporting results by all IMEP-19 participants. A questionnaire form to collect further information concerning the IMEP-19 participants was initiated. This information is used for statistical purposes and helps group the results more efficiently for the graphical presentation.
- A “sample receipt form”, this acknowledgement verifies that the CTS arrived at its destination in good order.

4.5 Collection, data processing and confirmation of results

The IMEP-19 participants reported their measurement results through the IMEP® web-site. After submission of their results and questionnaire information, the participants received an e-mail as a confirmation of their reported data. Any discrepancies were then corrected, no more amendments could be accepted once the reference value certificate was accessible via the IMEP® web-site. From this point on all reported data was transferred to the IMEP-19 database.

4.6 Individual IMEP-19 certificate, evaluation of measurement performance

IRMM has issued individual certificates to each participant in IMEP-19. This certificate includes the reported measurement value for the IMEP-19 Certified Test Sample, the IMEP-19 Certified reference values and the deviation of the reported value from the certified value by percentage. Furthermore E_n-numbers [19] have been calculated for those participants in IMEP-19 who reported measurement results with uncertainties which were calculated according to the Guides for Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) [18] and/or EURACHEM/CITAC (2000). Maximum levels of metals in foodstuff are set in the Commission Regulation (EC) 466/2001. In the absence of performance characteristics for the uncertainty of the measured value for measurements of metals in rice in this regulation, IRMM selected as performance characteristic a range of ±10% from the reference value as extended uncertainty (U_{ext}) to calculate the E_n-numbers. It can be assumed to represent an uncertainty range that is "fit for purpose" for measurements of trace metals in foodstuff. The E_n-numbers issued to the IMEP-19 participants are based on a single performance statistic, taking into account U_{ext} of the reference value as well as the reported uncertainty of the participant's measurement result.

For participants who did not state that they calculated the reported uncertainty according to the (ISO, 1995) [18] and/or EURACHEM/CITAC (2000) guides no E_n-numbers were issued. The IMEP-19 reference value certificate together with the individual IMEP-19 certificate were distributed to the relevant IMEP-19 participant in November 2002 (see also Annex 4)

5 Participation in IMEP-19

Samples were distributed to 293 laboratories in 44 countries. Measurement results were reported by 267 participants, from 43 countries in 5 continents. Table 4 summarises the distribution of participants per country.

Table 4. IMEP-19 participants per country.

Country	Samples sent	Results received	Country	Samples sent	Results received
ARGENTINA	3	3	MALTA	1	0
AUSTRALIA	3	3	MEXICO	7	6
AUSTRIA	13	12	NORWAY	3	3
BELGIUM	4	4	P.R. CHINA	1	1
BRAZIL	5	5	POLAND	40	36
BULGARIA	3	3	REP. OF KOREA	3	3
CROATIA	4	4	RUMANIA	3	2
CYPRUS	6	5	RUSSIA	2	2
CZECH REPUBLIC	10	10	SINGAPORE	4	4
ESTONIA	4	3	SLOVAKIA	45	41
FINLAND	3	3	SLOVENIA	6	5
FRANCE	8	6	SOUTH AFRICA	2	2
GERMANY	9	9	SPAIN	6	6
GREECE	1	1	SWEDEN	2	1
HUNGARY	14	14	SWITZERLAND	6	5
INDIA	3	2	THAILAND	10	10
IRELAND	3	3	THE NETHERLANDS	6	5
ITALY	2	2	TURKEY	18	15
JAPAN	8	8	UNITED KINGDOM	5	5
LATVIA	3	3	USA	2	2
LITHUANIA	5	4	VIETNAM	2	2
MALAYSIA	2	2	YUGOSLAVIA	3	2
			TOTAL	293	267

6 Data evaluation

Several conclusions can be drawn taking into account the information provided via the questionnaires, without quoting the identity of the laboratories. In IMEP-19, all except two participants completed the questionnaire. Figure 1 shows by percentage how many samples were analysed per year by element.

Figure 1 – Number of rice samples analysed per year by element

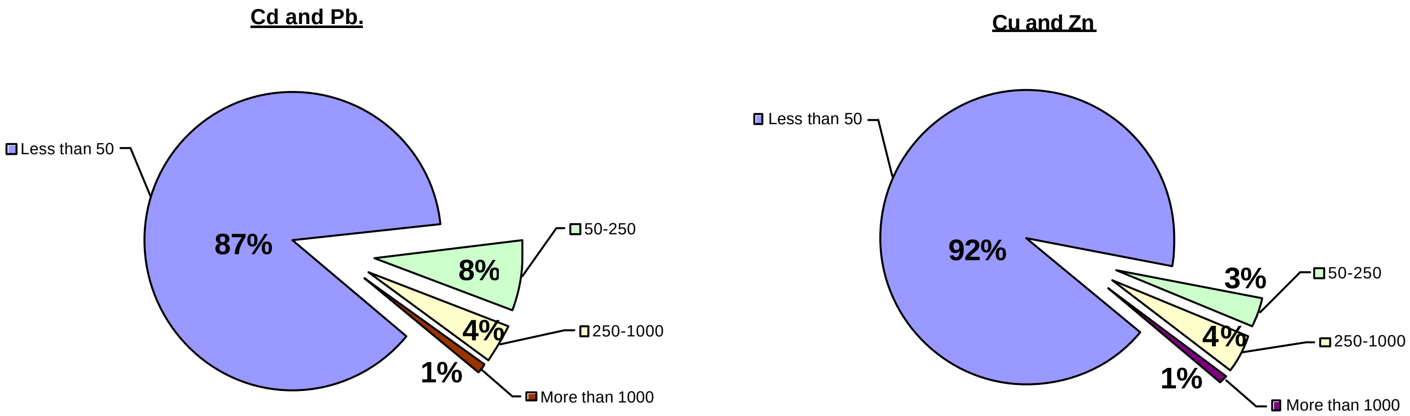


Figure 2 shows by percentage whether the CTS was analysed under routine conditions and according to (national) standards, this relates to questions 2 and 3 from the questionnaire.

Figure 2 - CTS treated according to the routine analytical procedure and national standards

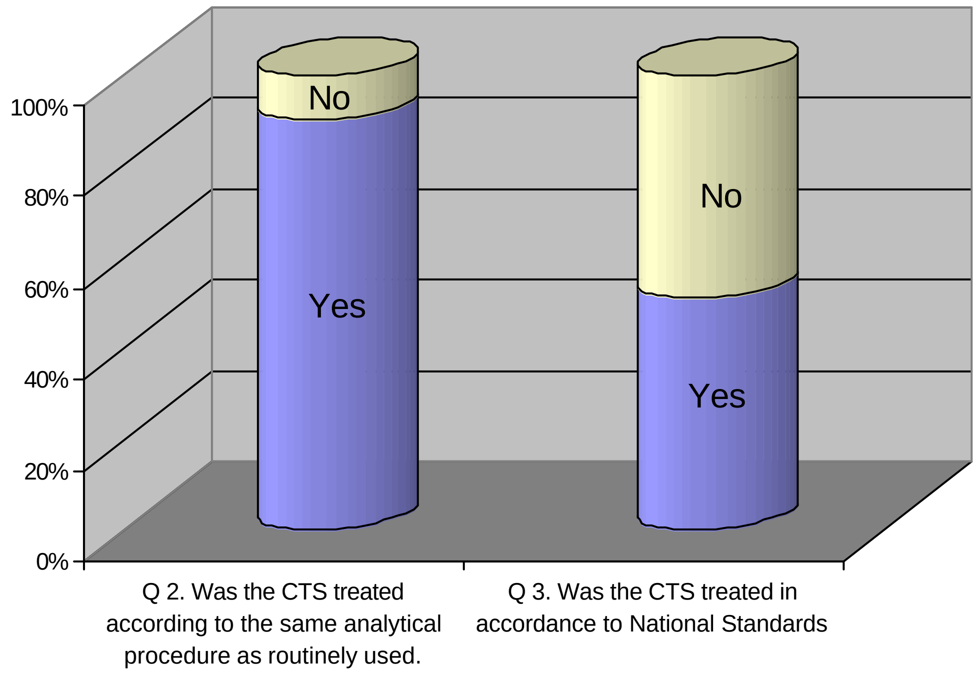
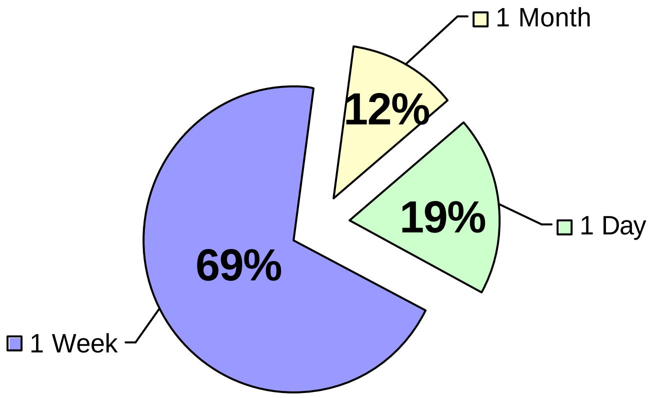


Figure 3 illustrates how much time was spent by the participants to carry out the measurements on the IMEP-19 CTS.

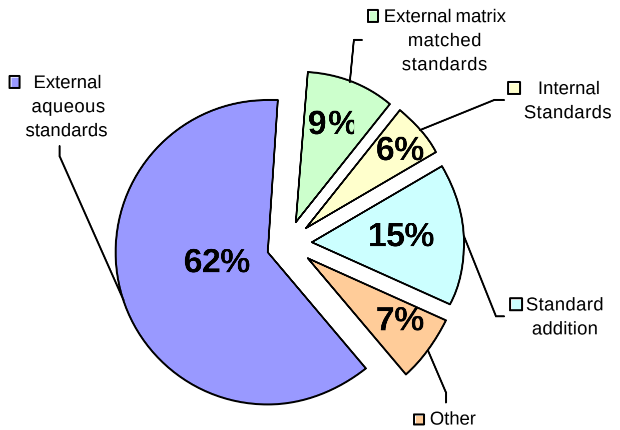
Figure 3 - Total time spent on the measurement



For all 4 elements under investigation in this interlaboratory comparison, the participants were free to measure the amount content of those elements that were of interest to their laboratory. 87% of participants in IMEP-19 reported measurement results for all 4 elements in the CTS. The most popular element was cadmium measured by 97% of the participants. Copper and zinc were measured by 95%, whereas lead was measured by 93% of the participants.

Figure 4 shows which calibration strategy was used by the IMEP-19 partcipants.

Figure 4 - Calibration Strategy



63% of laboratories participate regularly in proficiency-testing schemes (PTs) in order to assess performance for this type of analyses, but only 15% routinely use rice Certified Reference Materials (CRMs) for quality assurance.

Only 40% of laboratories routinely report uncertainties on chemical measurements to their customers. In addition 47% of IMEP-19 participants reported their measurement uncertainties calculated according to the "Guides for Quantifying Measurement Uncertainty" [18] issued by the International Organisation for Standardisation (ISO, 1995) [18] and/or EURACHEM/CITAC (2000).
Table 5 gives the number of participants per element and by which analytical techniques. For the graphical presentation, all analytical techniques have been grouped as shown in Table 6

Table 5. Number of IMEP-19 participants reported results
per analytical techniques and by element

ANALYTICAL TECHNIQUES	Cd	Cu	Pb	Zn
ETAAS & AAS	87	74	84	65
ICP-OES, ICP-AES & ICP	23	33	13	41
ICP-MS	35	33	37	27
GF-AAS & FAAS	88	91	87	95
OTHER	34	36	46	39

Table 6.Grouping the analytical techniques used by the IMEP-19 participants

Analytical technique	**Acronym**	**Group**
Anodic stripping voltammetry	ASV	OTHER
Atomic absorption spectroscopy	AAS	ETAAS & AAS
Cathodic-stripping voltammetry	CSV	OTHER
Cold Vapour-atomic absorption spectroscopy	CV-AAS	OTHER
Direct current plasma	DCP	OTHER
Electrothermal atomic absorption spectroscopy	ETAAS	ETAAS & AAS
Flame atomic absorption spectroscopy	FAAS	GF-AAS & FAAS
Flame atomic emission spectroscopy	FAES	OTHER
Flame atomic fluorescence spectroscopy	FAFS	OTHER
Flow injection analysis system-atomic absorption spectroscopy	FIAS-AAS	OTHER
Graphite furnace atomic absorption spectroscopy	GF-AAS	GF-AAS & FAAS
High resolution-inductively coupled plasma-mass spectrometry	HR-ICP-MS	OTHER
Hydride generation	HG	OTHER
Hydride generation-atomic absorption spectroscopy	HG-AAS	OTHER
Hydride-atomic absorption spectroscopy	H-AAS	OTHER
Inductively coupled plasma	ICP	ICP-OES, ICP-AES & ICP
Inductively coupled plasma-atomic emission spectrometry	ICP-AES	ICP-OES, ICP-AES & ICP
Inductively coupled plasma-mass spectrometry	ICP-MS	ICP-MS
Inductively coupled plasma-optical emission spectrometry	ICP-OES	ICP-OES, ICP-AES & ICP
Infrared spectrometry	IR	OTHER
Ion chromatography	IC	OTHER
Metal hydride system	MHS	OTHER
No statement	NONE GIVEN	OTHER
Other	OTHER	OTHER
Potentiometric stripping analysis	PSA	OTHER
Spectrophotometry	SPECTROPH.	OTHER
Xray flourescence	XRF	OTHER

6.1 Water content determination

Via the IMEP-19 questionnaire the IMEP-19 participants provided more detailed information concerning determination of the water content and the correction for dry-mass of the rice CTS. The vast majority of IMEP-19 participants applied the drying-oven method for the determination of the water content in the rice CTS. None of the IMEP-19 participants used Karl-Fischer titration to determine the water content in the CTS. A survey on the water content determination and the correction for dry-mass is summarised in Annex 2 of this report.

7 Graphical presentation of IMEP-19 results

The IMEP® results are traditionally presented graphically. Based on general information of the laboratory (e.g. country of origin) and the answers given in the questionnaire, the results obtained for each element are grouped in sets of data. For each set of data, the results are plotted together with the reference value. All results are included in the graphs. The scale of the graphs is ± 50 % deviation from the reference value. This scale was chosen for convenience. The number of results outside this range is presented in textboxes on each graph. The IMEP-19 graphs are presented by element and can be found in Annex 1 and Annex 2 of this report.

Table 7 gives a summary of all the general IMEP-19 graphs. For each element the results of all participants are plotted without any grouping (all participants). The other graphs are grouped according to the analytical technique used.

Table 8 contains all the special interest graphs for IMEP-19. The Regional graphs show all the results from the EU and EU candidate countries participants. In addition graphs have been prepared, which compare both of them. Graphs have been prepared grouping the participants results according to the different criteria from specific questionnaire information. Included in this report is a graph from participants in Thailand who measured in addition the total iron amount content in the CTS. Please note that the CTS in IMEP-19 had not been certified for the Fe amount content. Therefore no certified value can be given as a reference, the average value of the participants reported values for iron is given as additional information in the graph. Located at the end of Annex 1 are the measurement performance graphs, these show the results from participants who estimated their uncertainties according to (ISO, 1995) [18] and/or EURACHEM/CITAC (2000). The results are sorted according to the E_n-numbers. Graphs concerning the water content determination and the correction for dry-mass can be found in Annex 2 of this report.

Table 7. IMEP-19 general result graphs

criterion	*Cd*	*Cu*	*Pb*	*Zn*
all participants	✓	✓	✓	✓
analytical techniques used	✓	✓	✓	✓

Table 8. IMEP-19 special interest graphs

	Cd	*Cu*	*Pb*	*Zn*
Regional graphs				
EU countries	✓	✓	✓	✓
EU candidate countries	✓	✓	✓	✓
EU countries vs. EU candidate countries	✓	✓	✓	✓
Quality system graphs				
ISO 17025 vs. other	✓	✓	✓	✓
All continents graphs				
Geographical Europe	✓	✓	✓	✓
Americas & South Africa	✓	✓	✓	✓
Asia Pacific	✓	✓	✓	✓
Questionnaire graphs				
According to experience/less experience	✓	✓	✓	✓
No. of samples analysed per year	✓	✓	✓	✓
Time spent on the measurement	✓	✓	✓	✓
Calibration Strategy	✓	✓	✓	✓
Use of rice CRMs	✓	✓	✓	✓
Participation in PTs	✓	✓	✓	✓
Accredited/Authorised vs. Non-accredited	✓	✓	✓	✓
Uncertainties calculated according to guidelines	✓	✓	✓	✓
Report uncertainties to customers	✓	✓	✓	✓
Measurement performance graphs				
E_n-numbers for participants who estimated uncertainty according to (ISO, 1995) and/or EURACHEM/CITAC (2000)	✓	✓	✓	✓
Iron measurement graph				
Participants from Thailand	**Fe results**			

8 Acknowledgements

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9 List of abbreviations

BAM	Bundesanstalt für Materialforschung und –prüfung (Berlin, Germany)
BELAC	Belgium Accreditation
BIPM	Bureau International des Poids et Mesures (Paris, France)
CCQM	Comité Consultatif pour la Quantité de Matière
CIPM	International Committee for Weights and Measure
CITAC	Co-operation for International Traceability in Analytical Chemistry
CRMs	Certified Reference Materials
CTS	Certified Test Samples
EA	European Co-operation for Accreditation
EC	European Commission
EU	European Union
EURACHEM	A focus for Analytical Chemistry in Europe
EUROMET	Association of European Institutes for Metrology
GUM	Guide for expression for Uncertainty in Measurement
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
IDMS	Isotope Dilution Mass Spectrometry
IMEP®	International Measurement Evaluation Programme
IRMM	Institute for Reference Materials and Measurements (EC, Geel, Belgium)
ISO	International Organisation for Standardisation
IUPAC	International Union for Pure and Applied Chemistry
JRC	Joint Research Centre
MRA	Mutual Recognition Agreement
NABs	National Accreditation Bodies
NMIJ	National Measurement Institute of Japan (Tsukuba, Japan)
PMM	Primary Method of Measurement
PTs	Proficiency Testing
RCs	Regional Co-ordinators
SCF	Scientific Committee on Food
SSZAAS	Solid Sample Zeemann Atomic Absorption Spectrometry
TIMS	Thermal Ionosation Mass Spectrometry

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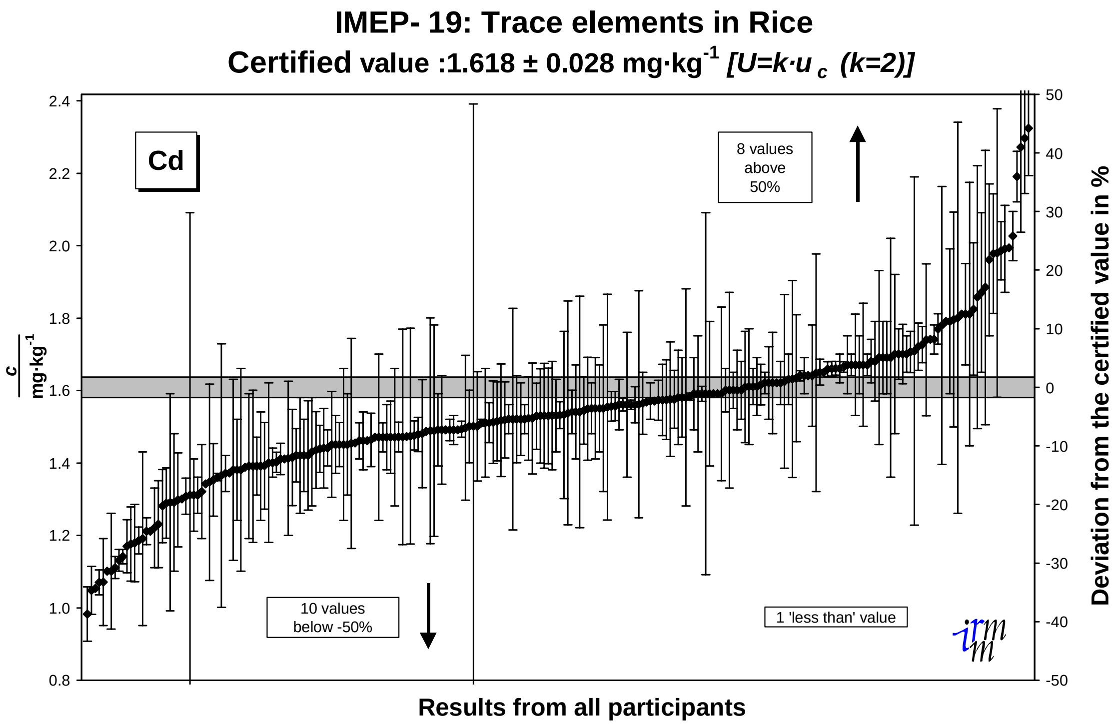
EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC
JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP-19: Trace Elements in Rice
Annex 1-Graphical presentation contents table

IMEP-19

participants' results- General graphs by element

Figure 1



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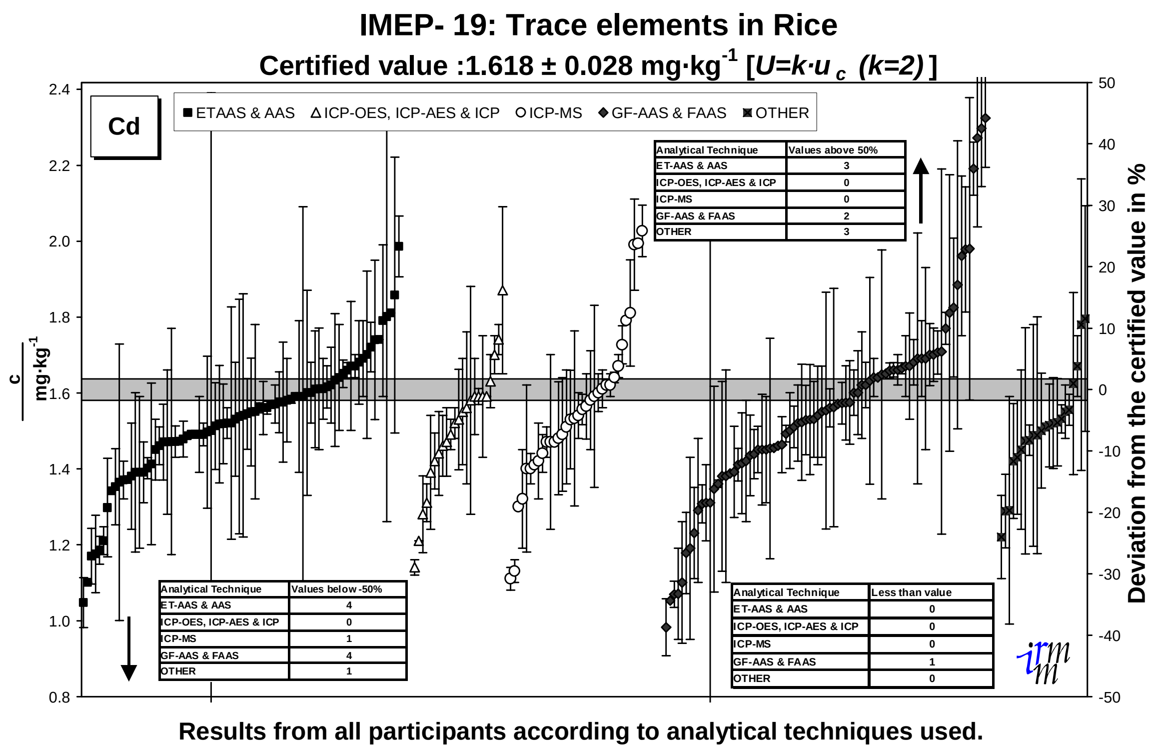

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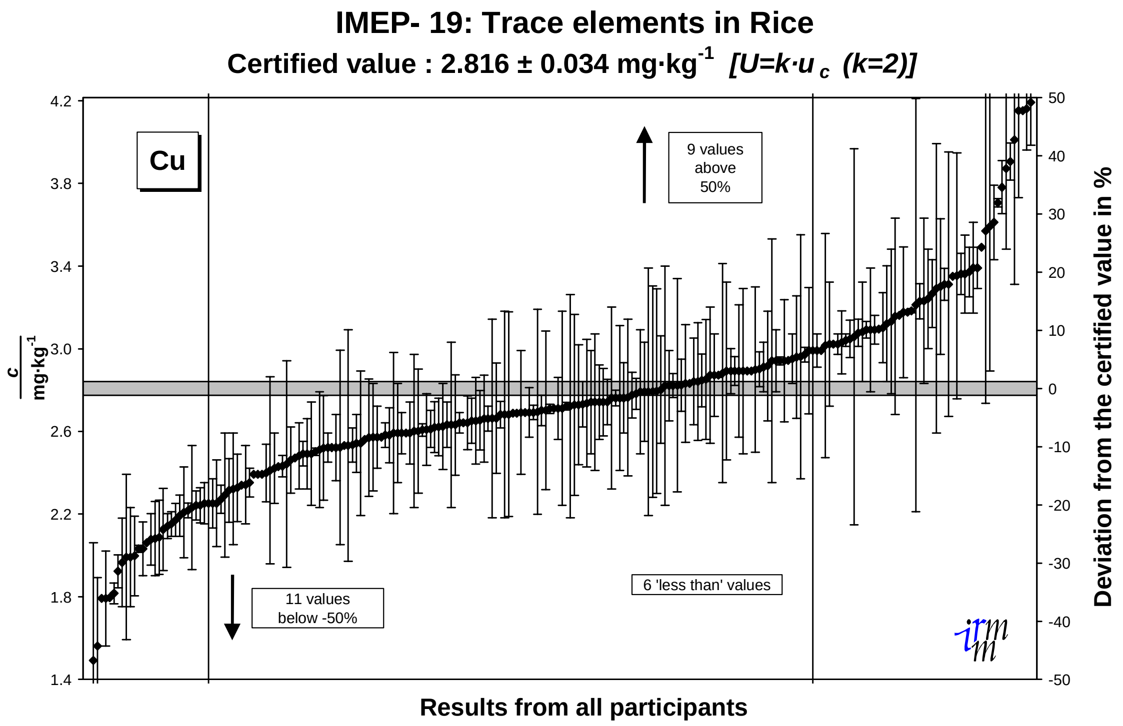

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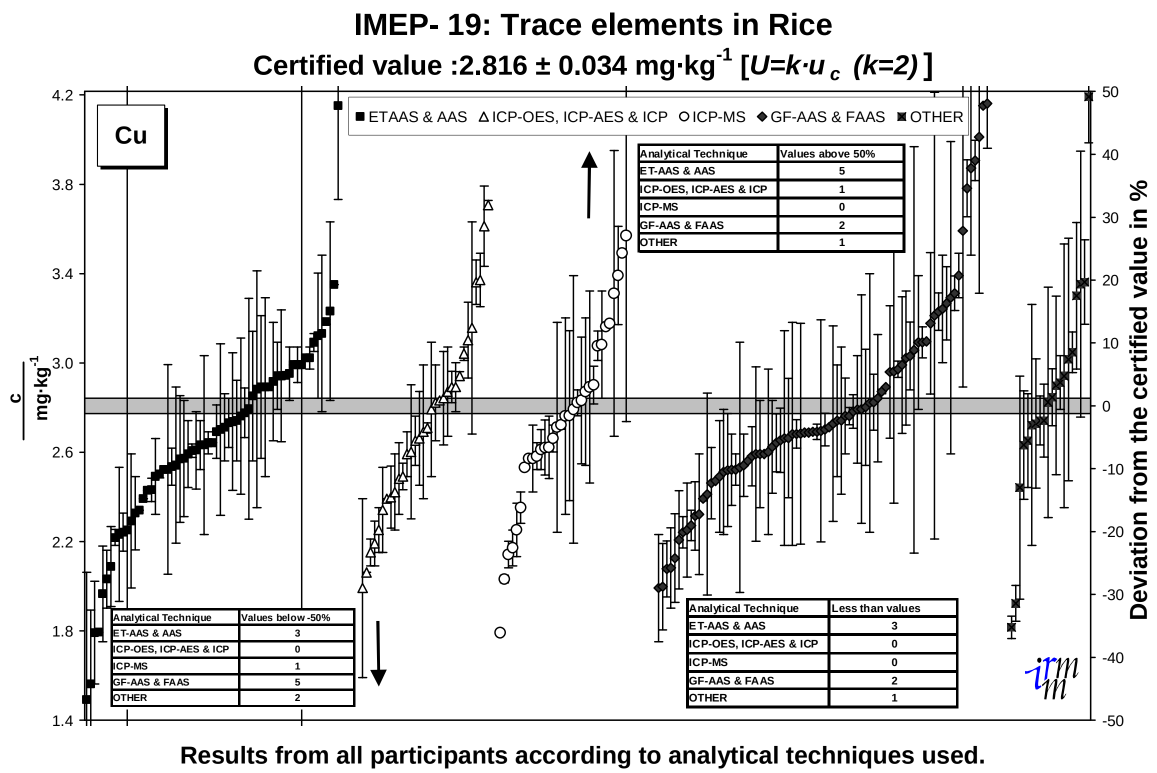

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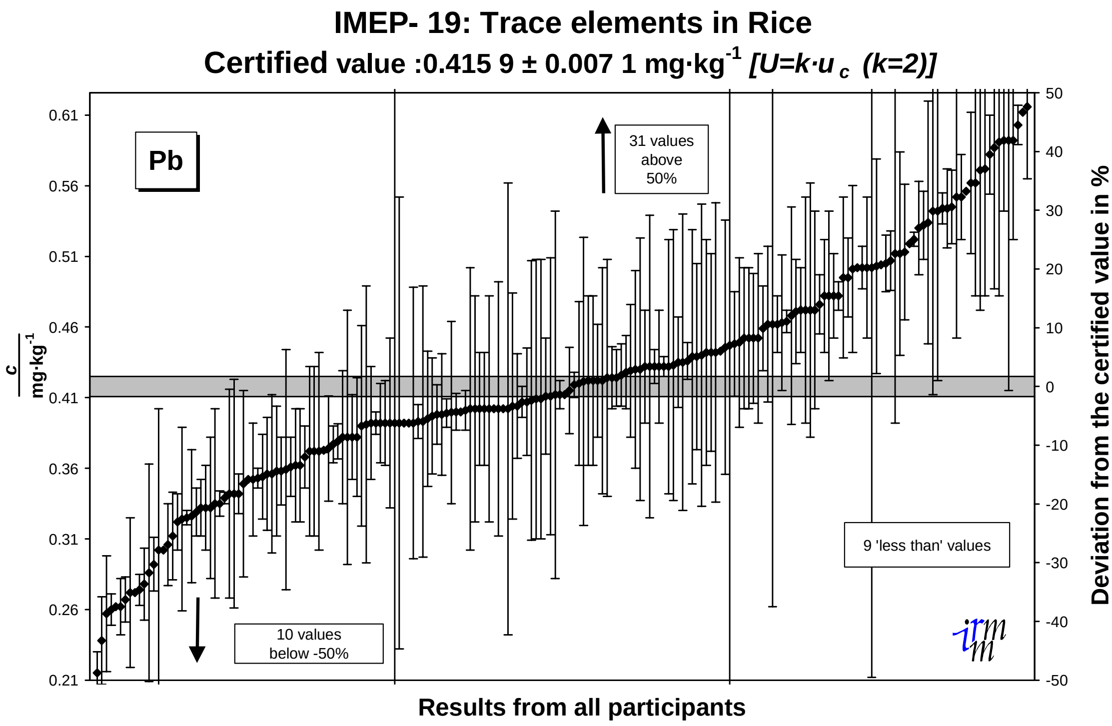

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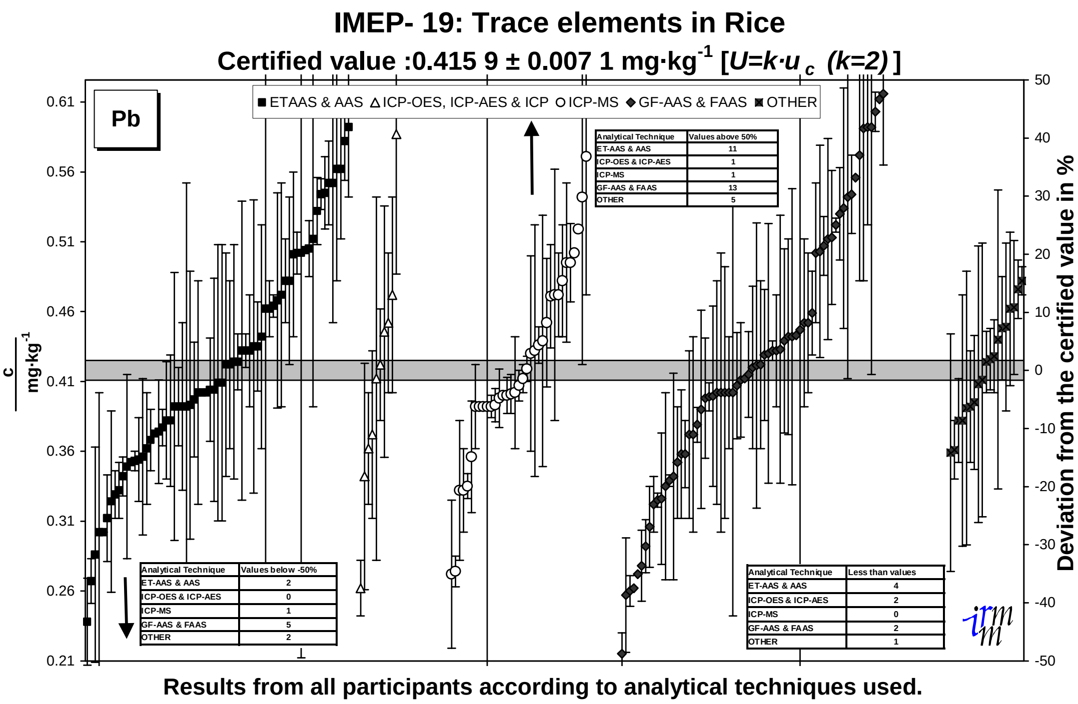

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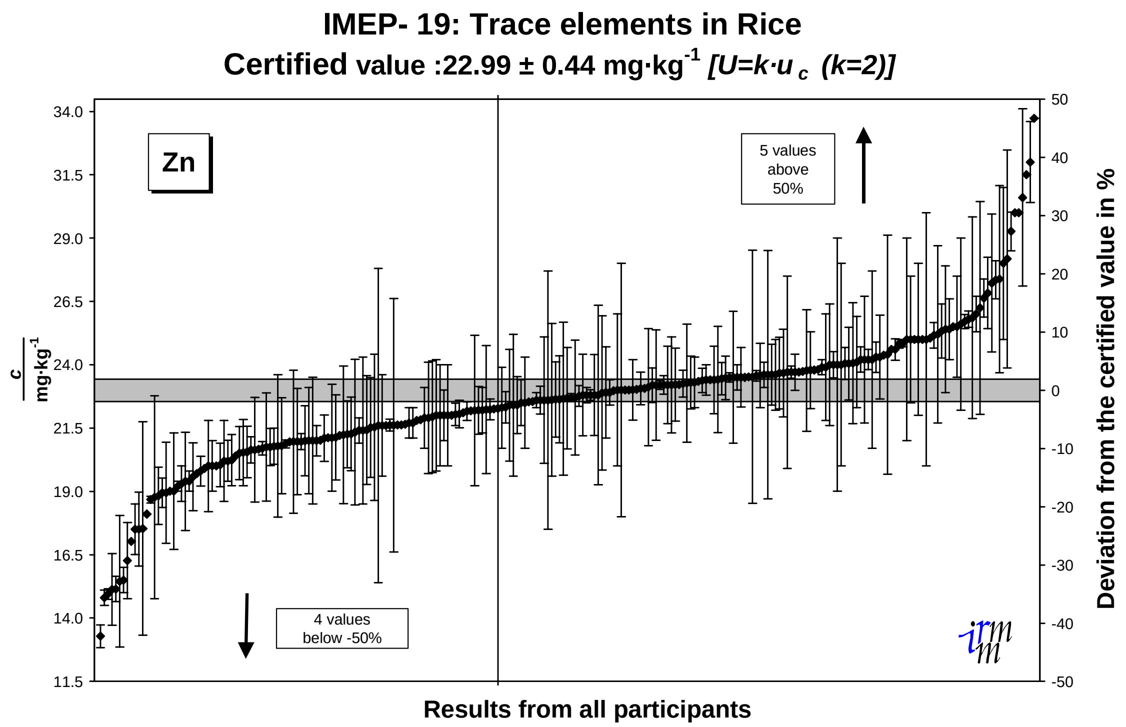

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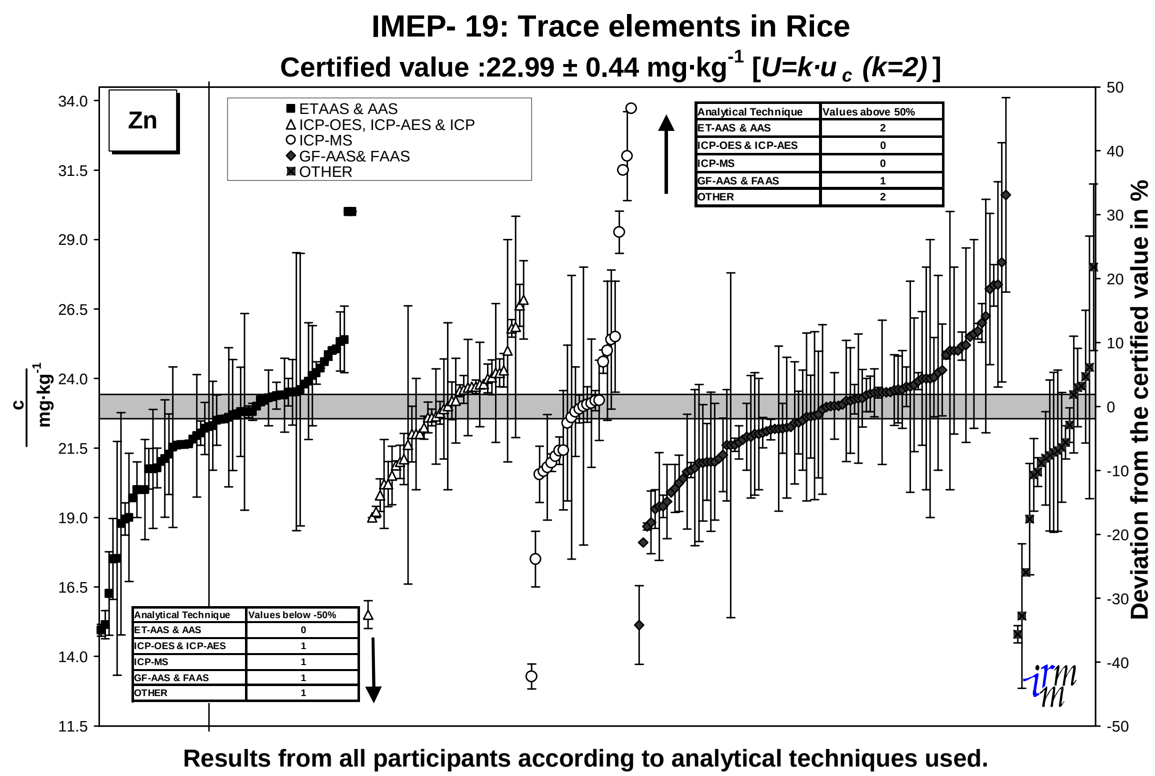
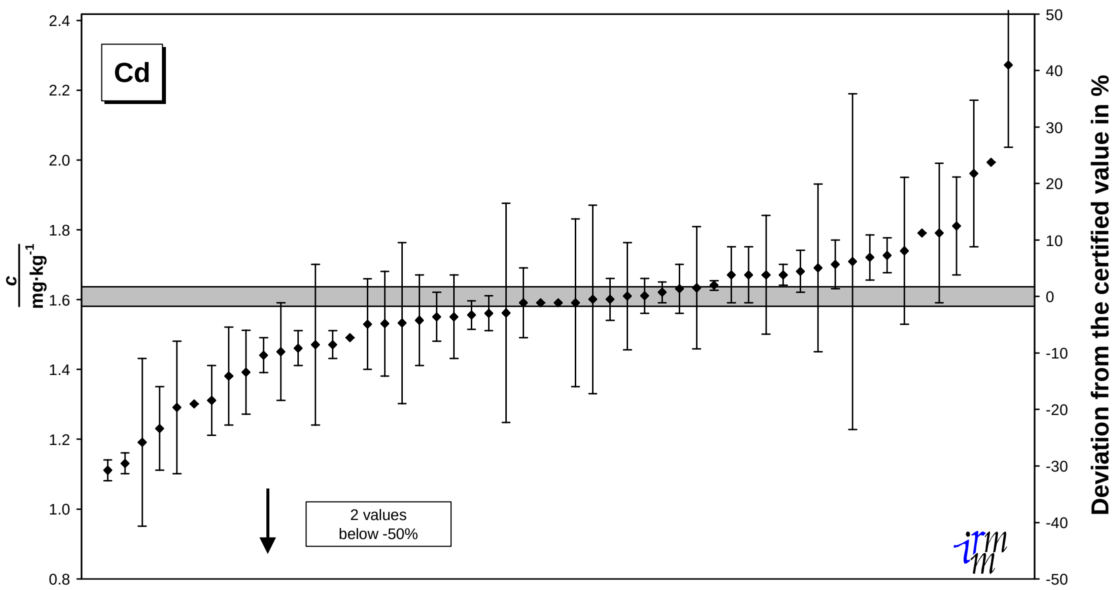


IMEP-19

participants' results - Regional graphs by element

Figure 9

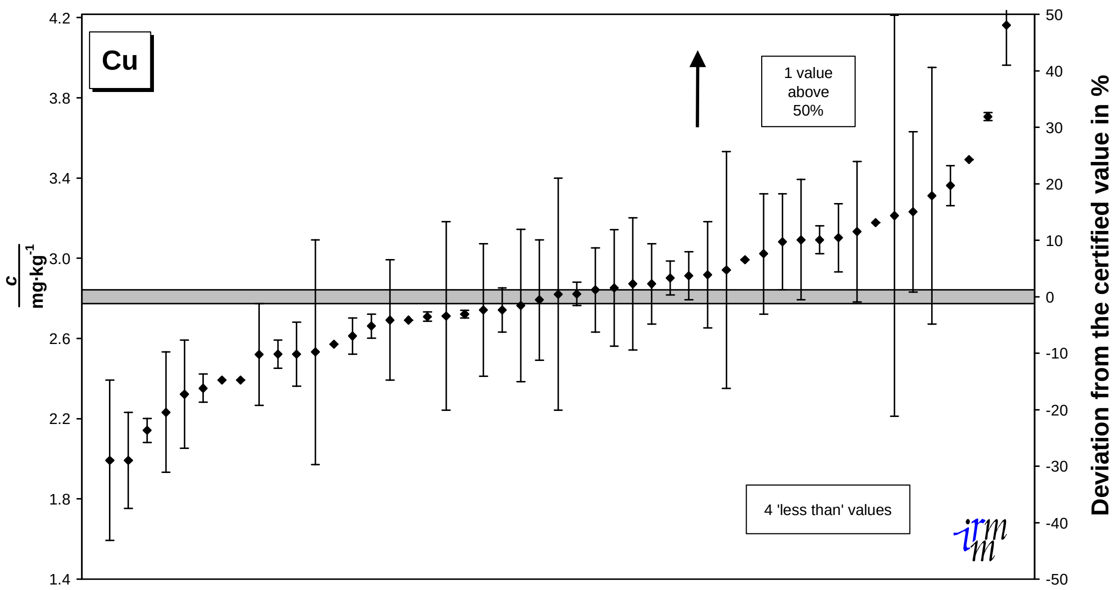
IMEP- 19: Trace elements in Rice
Certified value :1.618 ± 0.028 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from participants from EU countries (Austria, Belgium, Finland, France, Germany, Greece, Ireland, Italy, Spain, Sweden, The Netherlands and United Kingdom)

Figure 10

IMEP- 19: Trace elements in Rice
Certified value :2.816 ± 0.034 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from participants from EU countries (Austria, Belgium, Finland, France, Germany, Greece, Ireland, Italy, Spain, Sweden, The Netherlands and United Kingdom)

Figure 11

IMEP- 19: Trace elements in Rice
Certified value :0.415 9 ± 0.007 1 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*

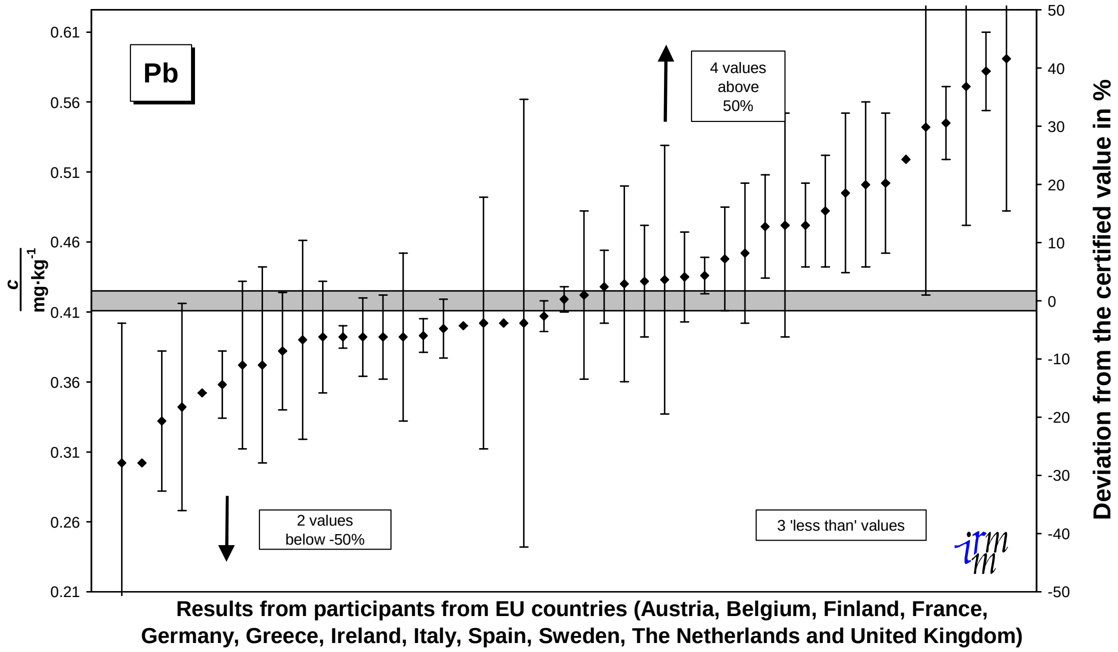
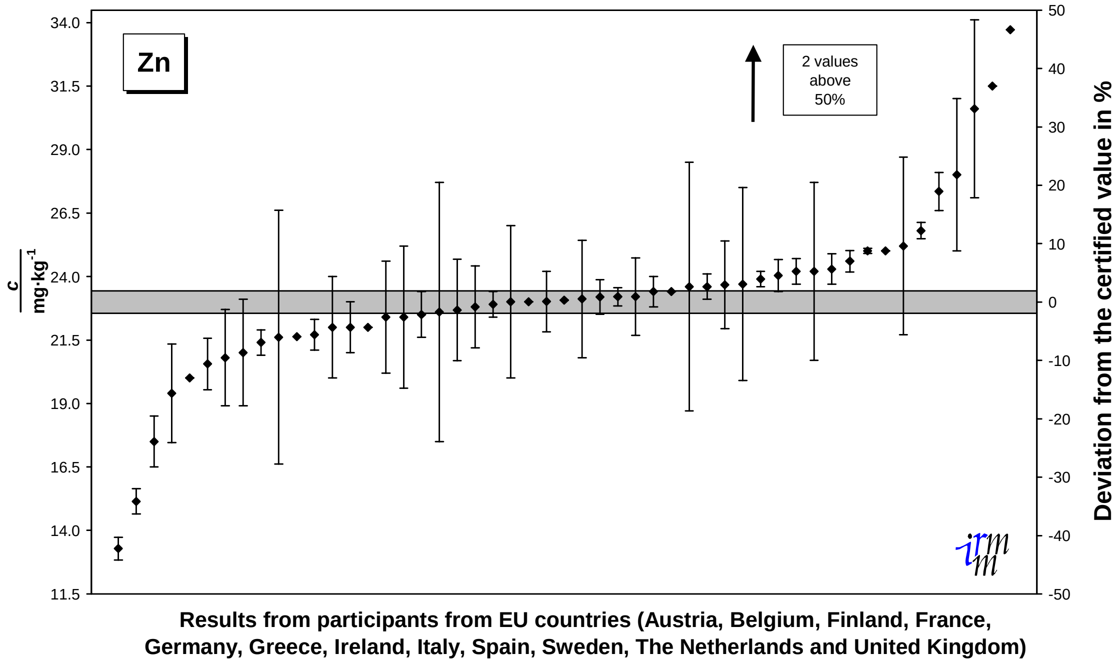


Figure 12

IMEP- 19: Trace elements in Rice
Certified value :22.99 ± 0.44 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



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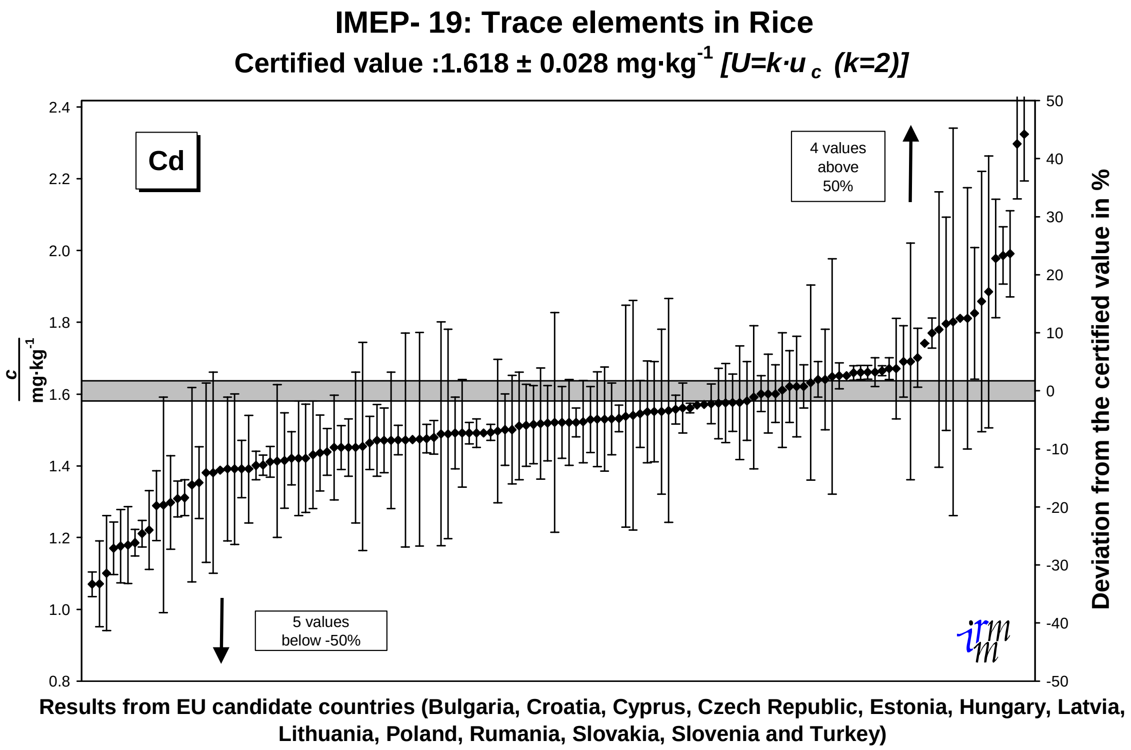

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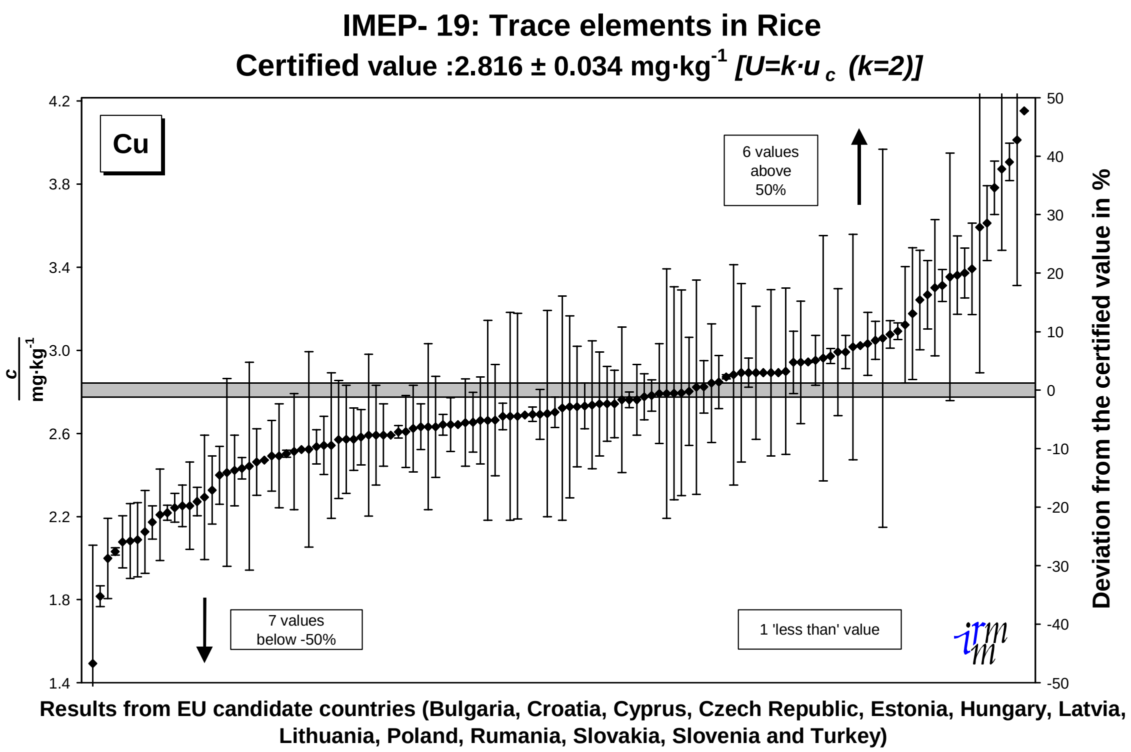

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IMEP- 19: Trace elements in Rice
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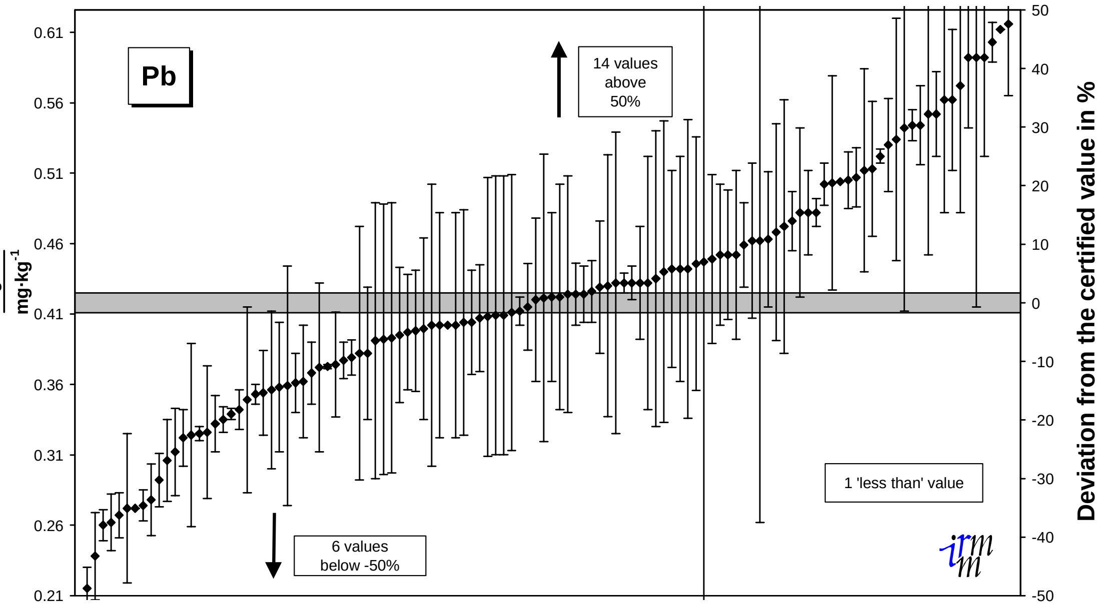
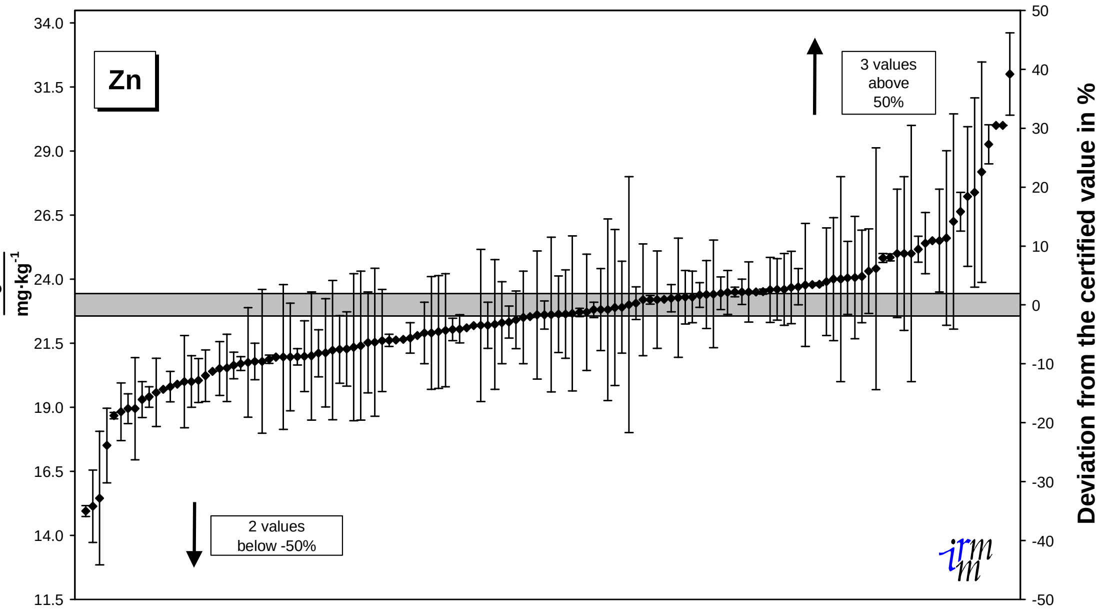


Results from EU candidate countries (Bulgaria, Croatia, Cyprus, Czech Republic, Estonia, Hungary, Latvia, Lithuania, Poland, Rumania, Slovakia, Slovenia and Turkey)

Figure 16

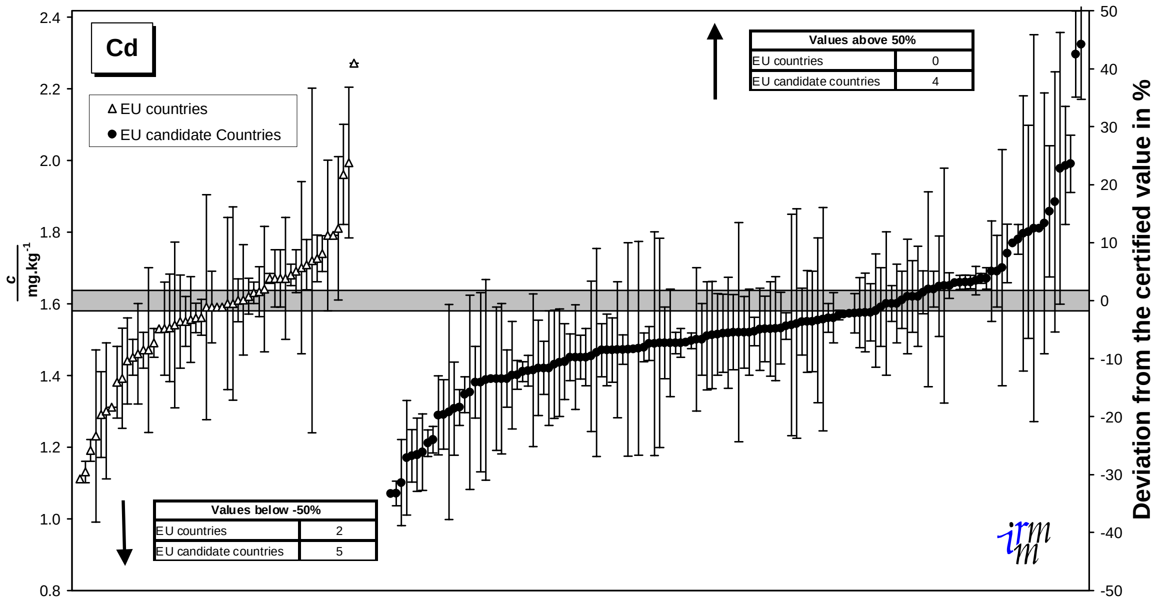
IMEP- 19: Trace elements in Rice
Certified value :22.99 ± 0.44 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from EU candidate countries (Bulgaria, Croatia, Cyprus, Czech Republic, Estonia, Hungary, Latvia, Lithuania, Poland, Rumania, Slovakia, Slovenia and Turkey)

Figure 17

IMEP- 19: Trace elements in Rice
Certified value : 1.618 ± 0.028 mg·kg⁻¹ *[U=k·u_c (k=2)]*



Results from EU countries compared with EU candidate countries.

Figure 18

IMEP- 19: Trace elements in Rice
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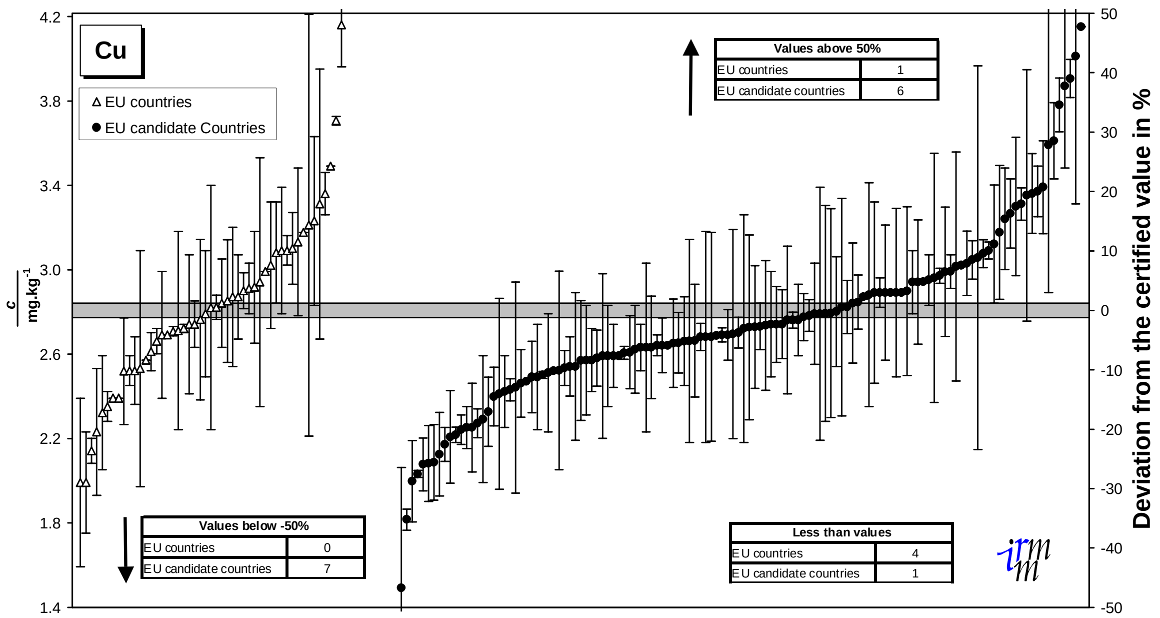

Results from EU countries compared with EU candidate countries.

Figure 19

IMEP- 19: Trace elements in Rice
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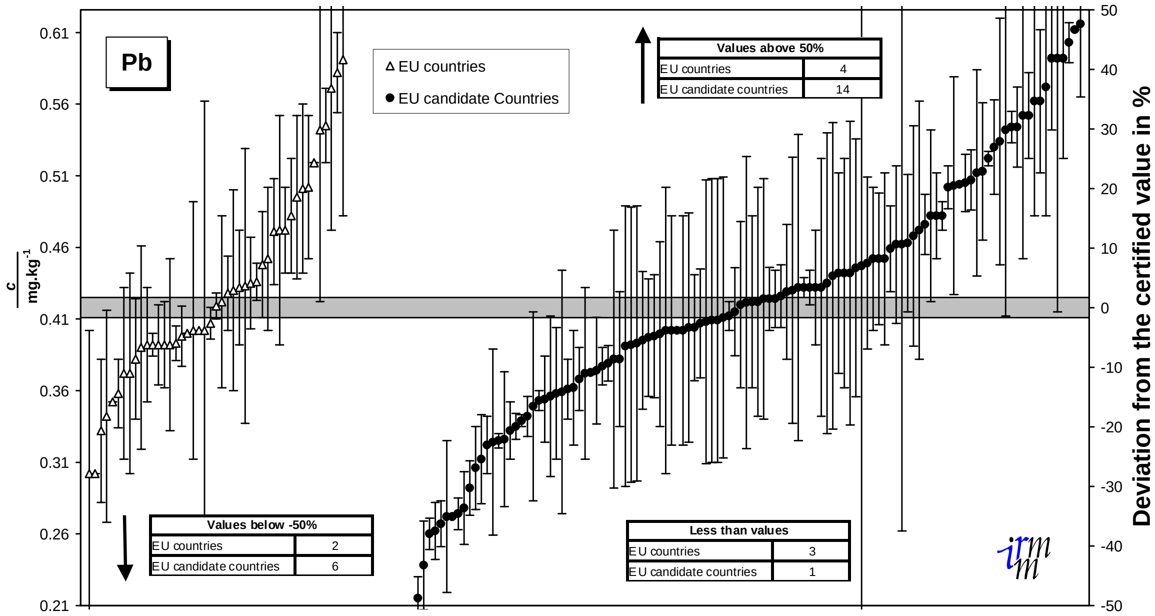

Results from EU countries compared with EU candidate countries.

Figure 20

IMEP- 19: Trace elements in Rice
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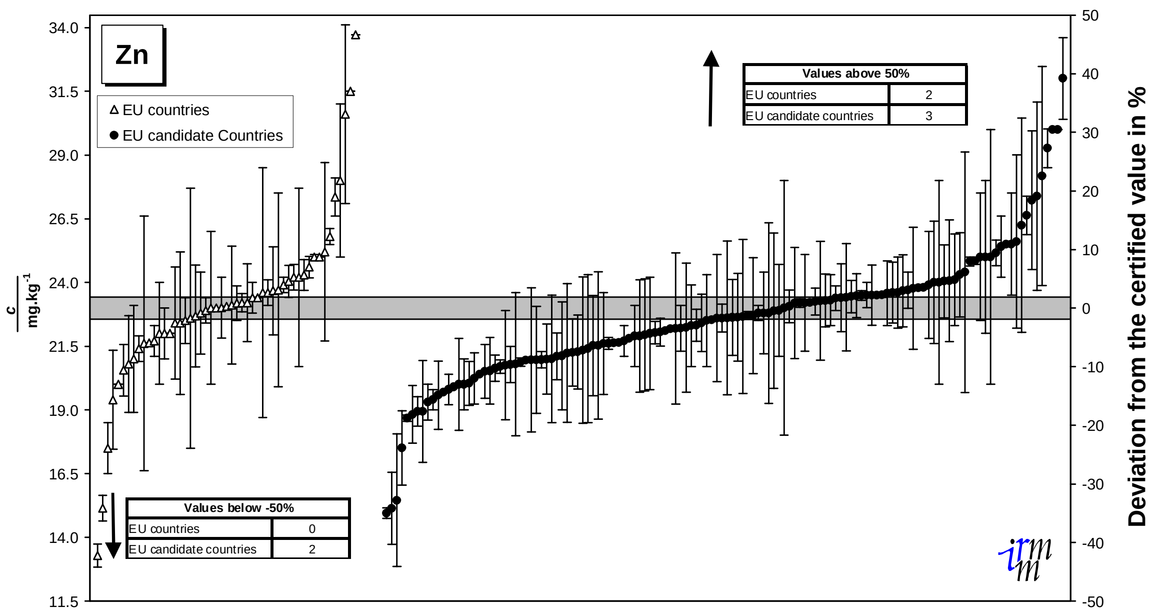

Results from EU countries compared with EU candidate countries.

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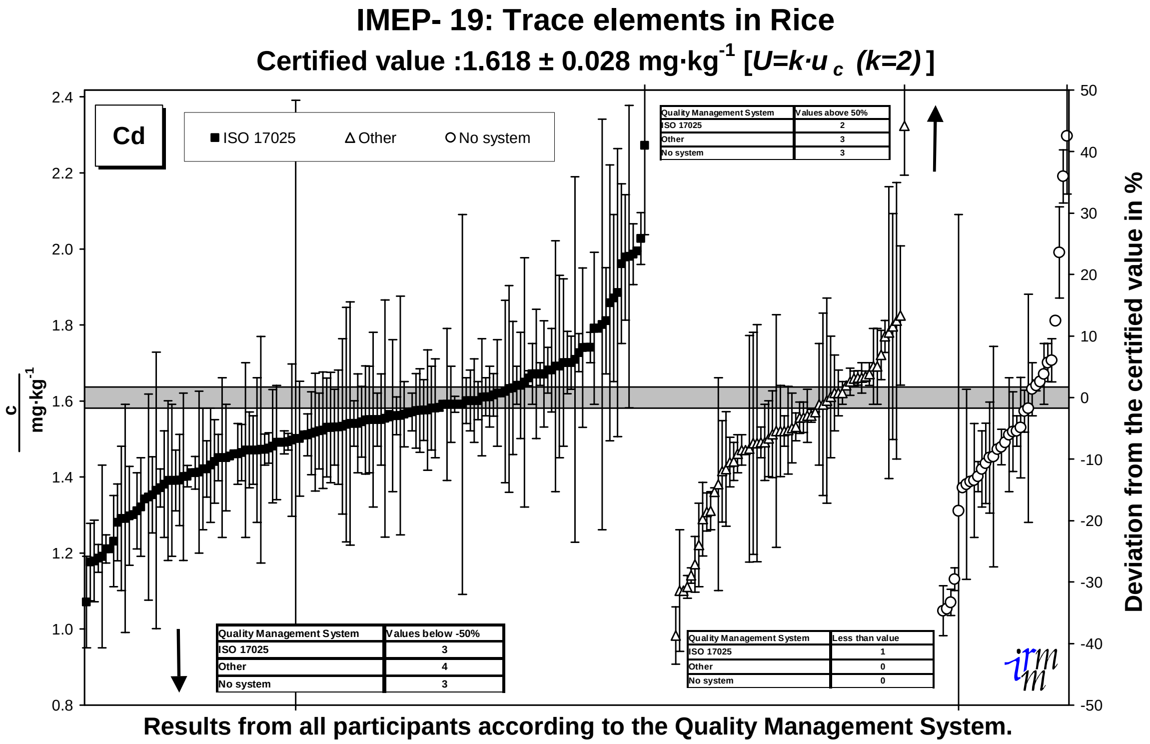
participants' results - Quality Management System graphs by element

Figure 21



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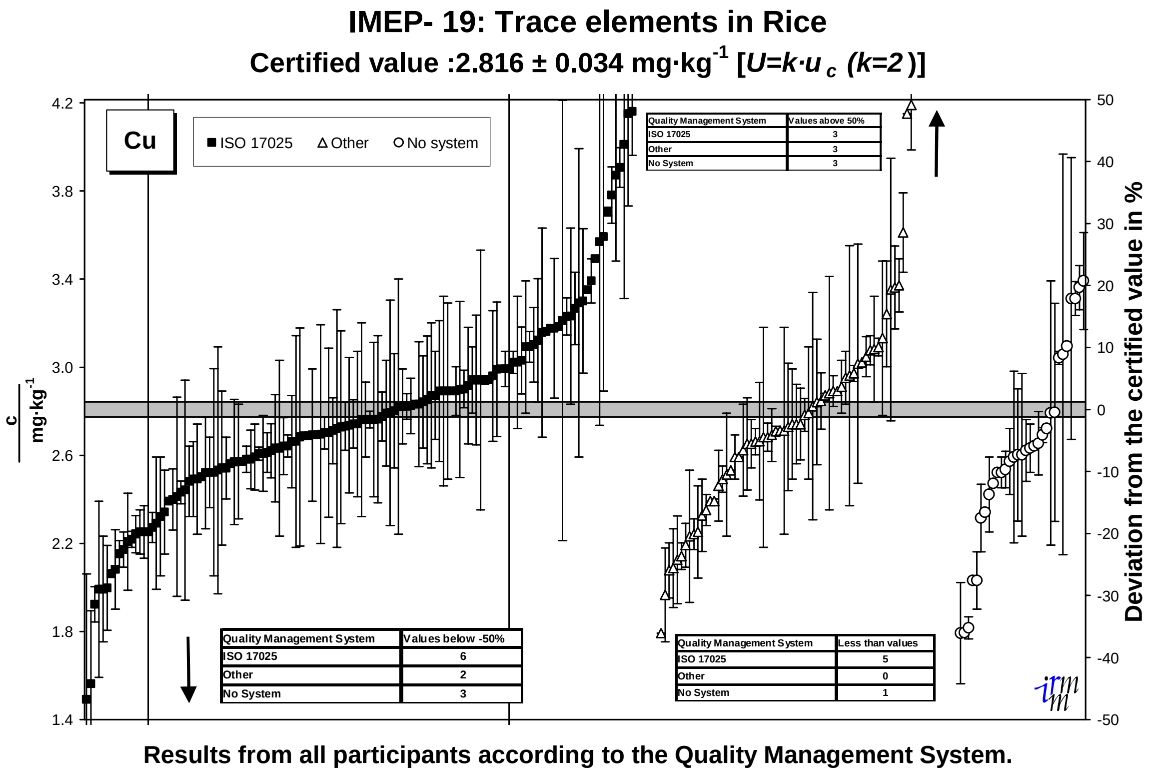

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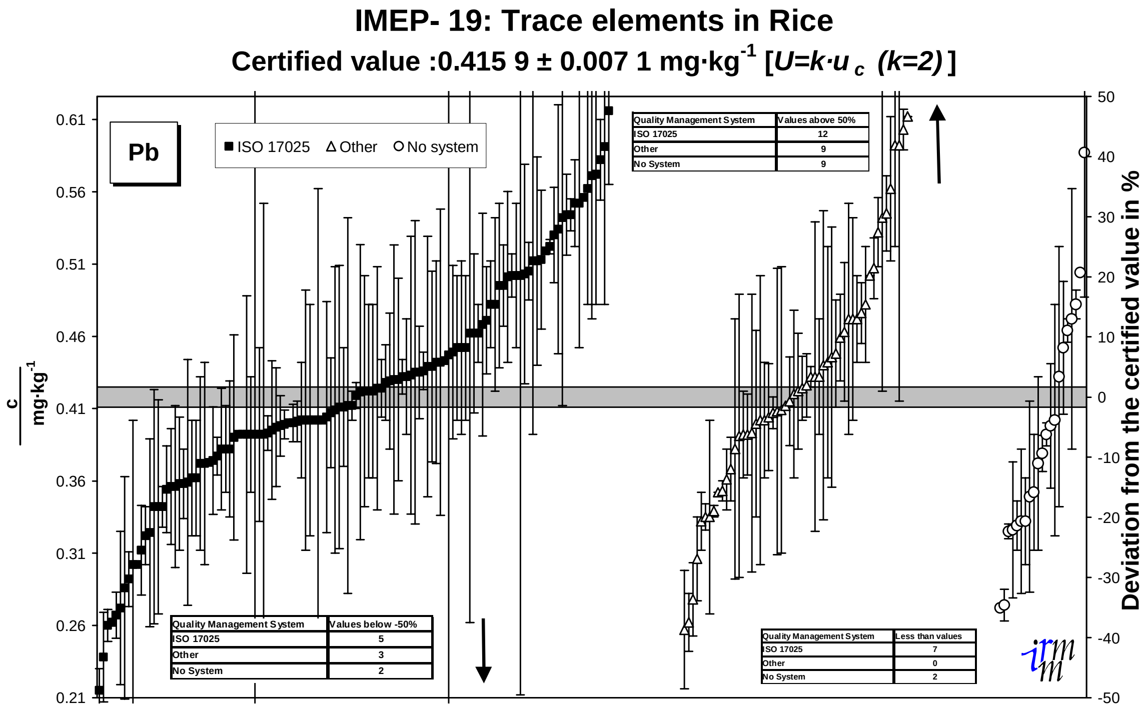
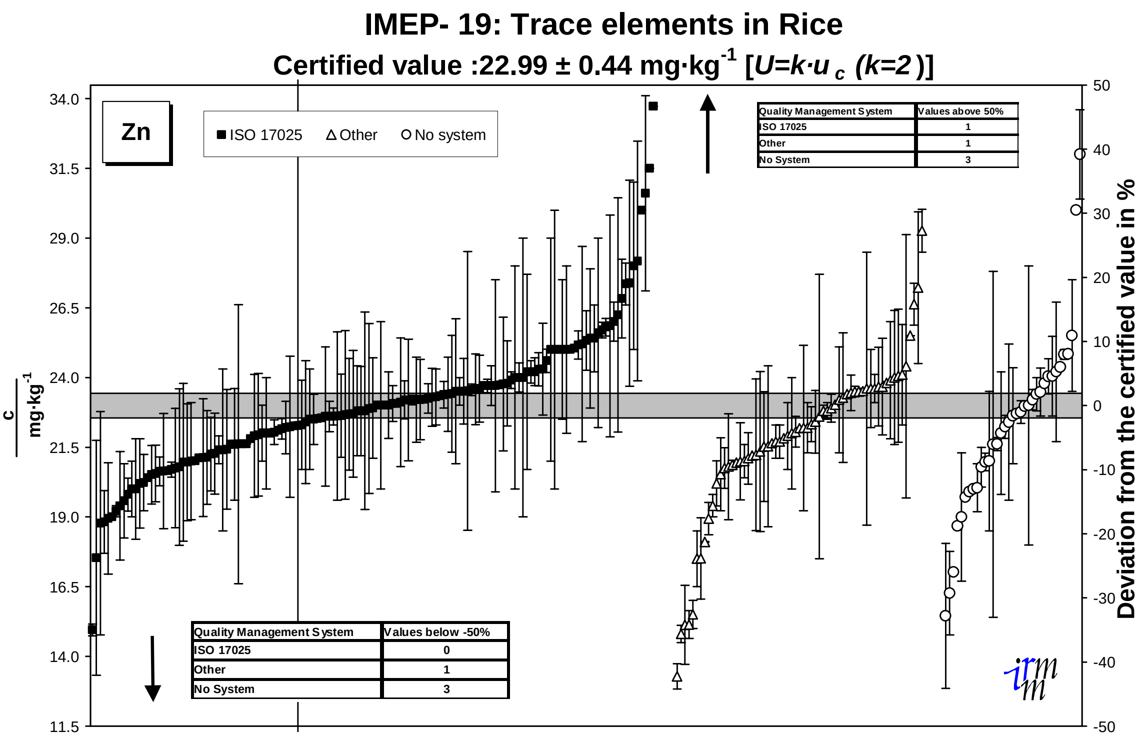


Results from all participants according to the Quality Management System.

Figure 24



Results from all participants according to the Quality Management System.

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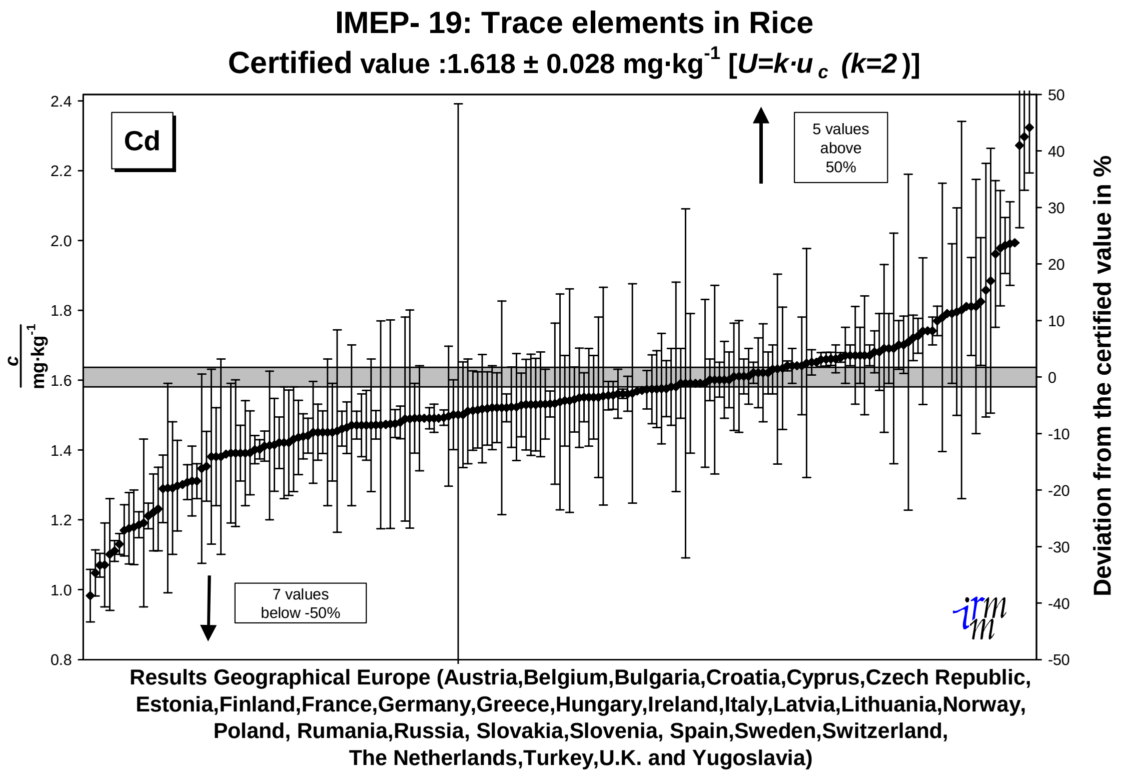
participants' results - All continents graphs by element

Figure 25



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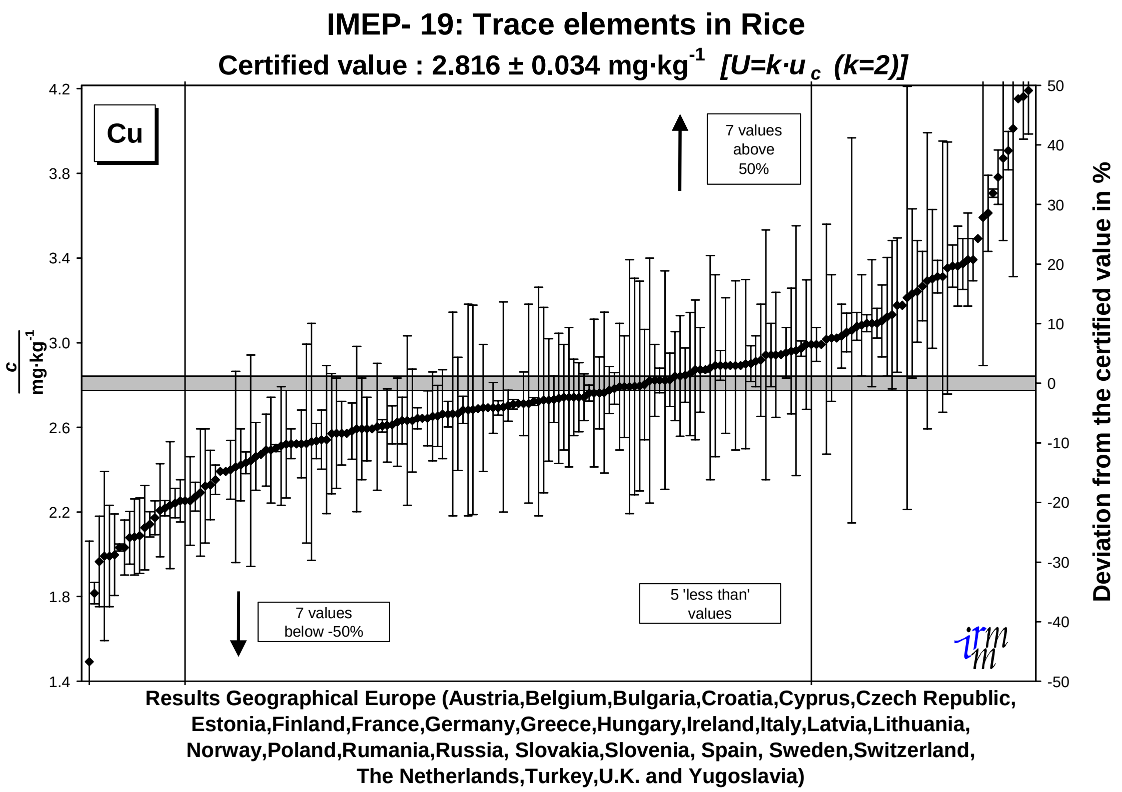

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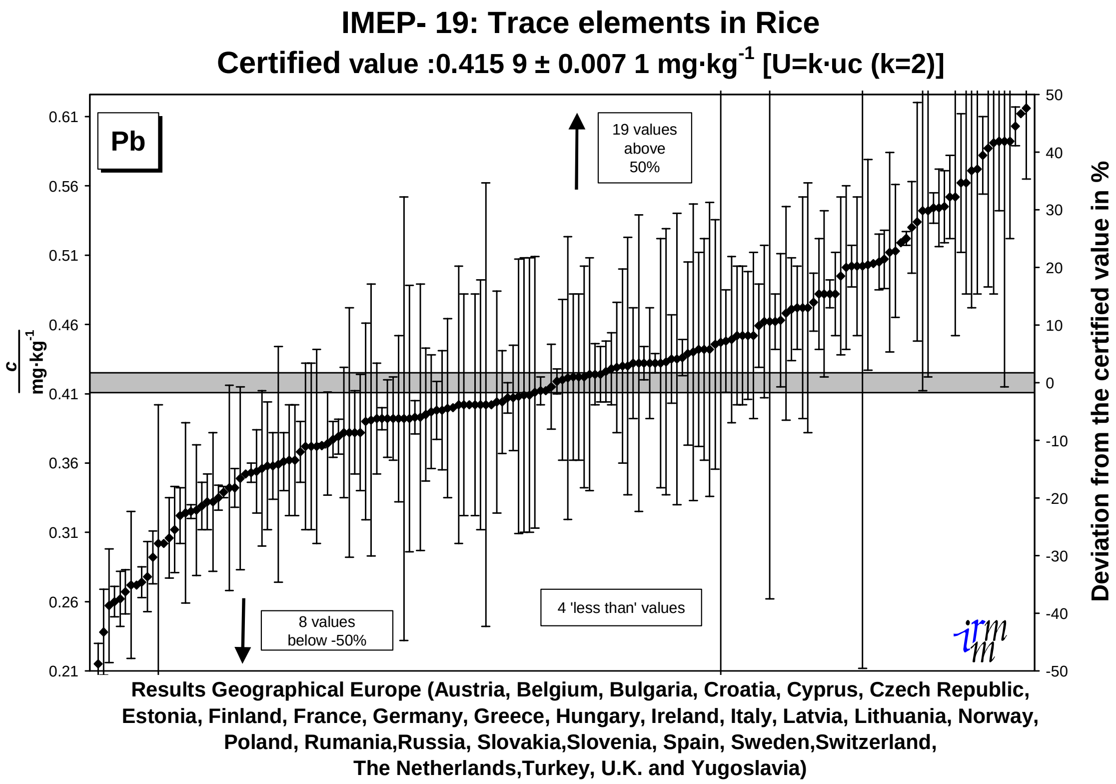

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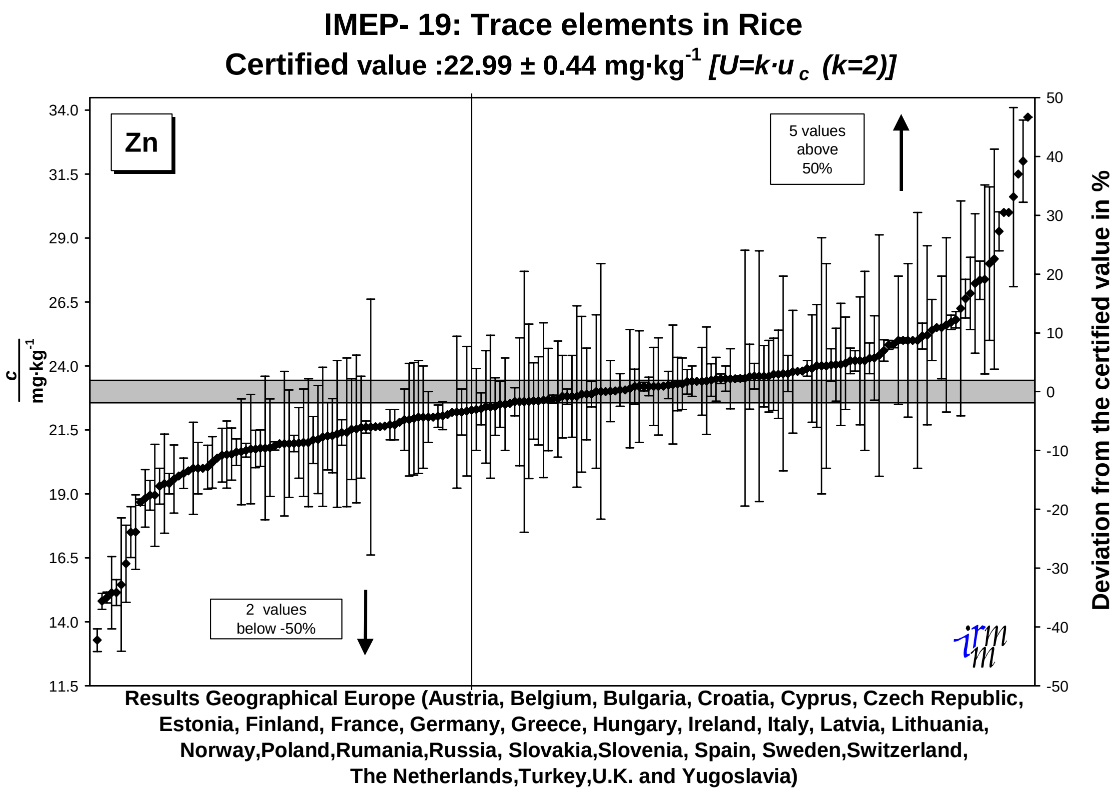

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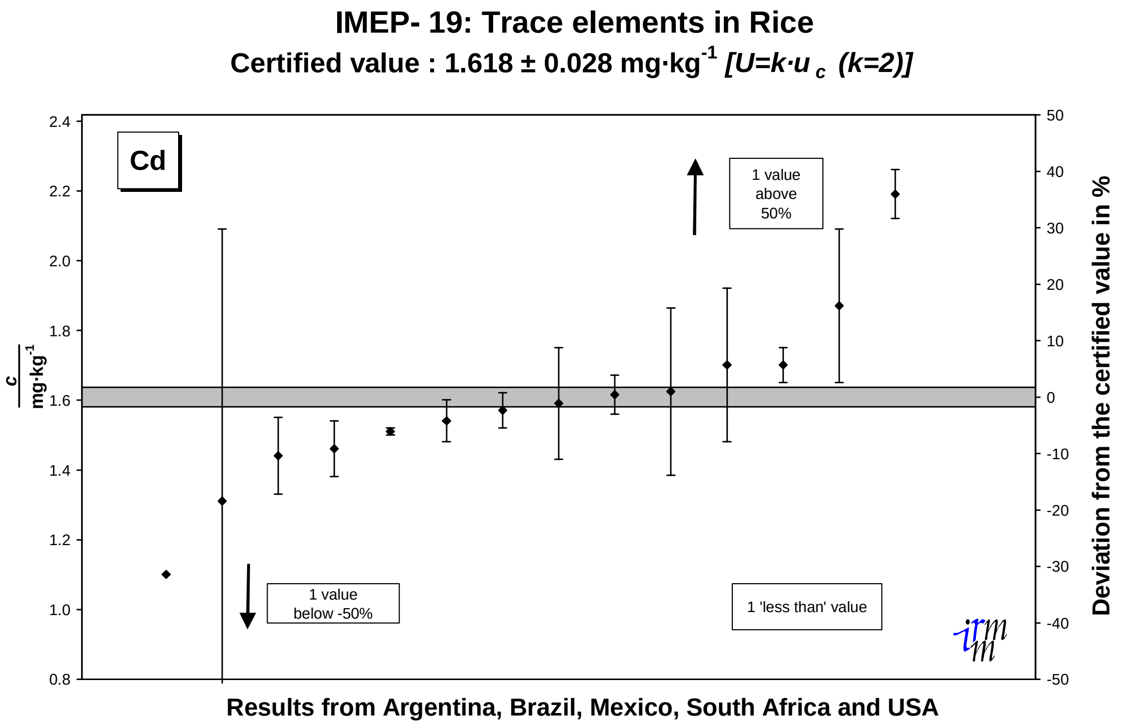


Figure 30

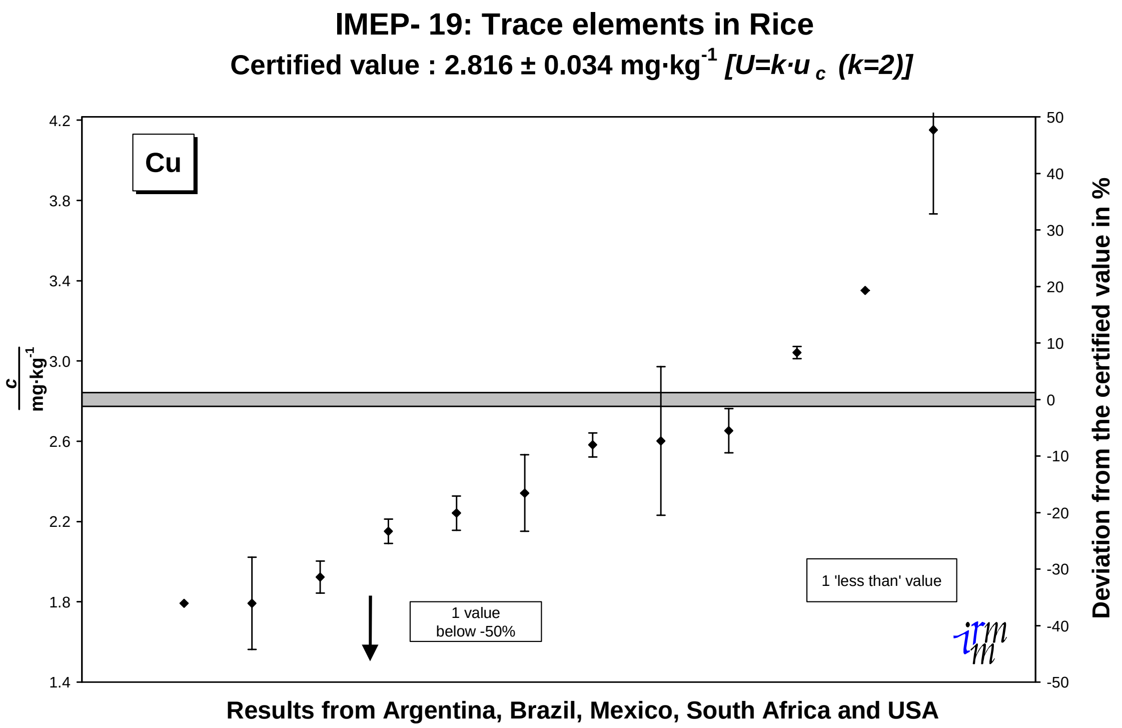


Figure 31

IMEP- 19: Trace elements in Rice
Certified value :0.415 9 ± 0.007 1 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*

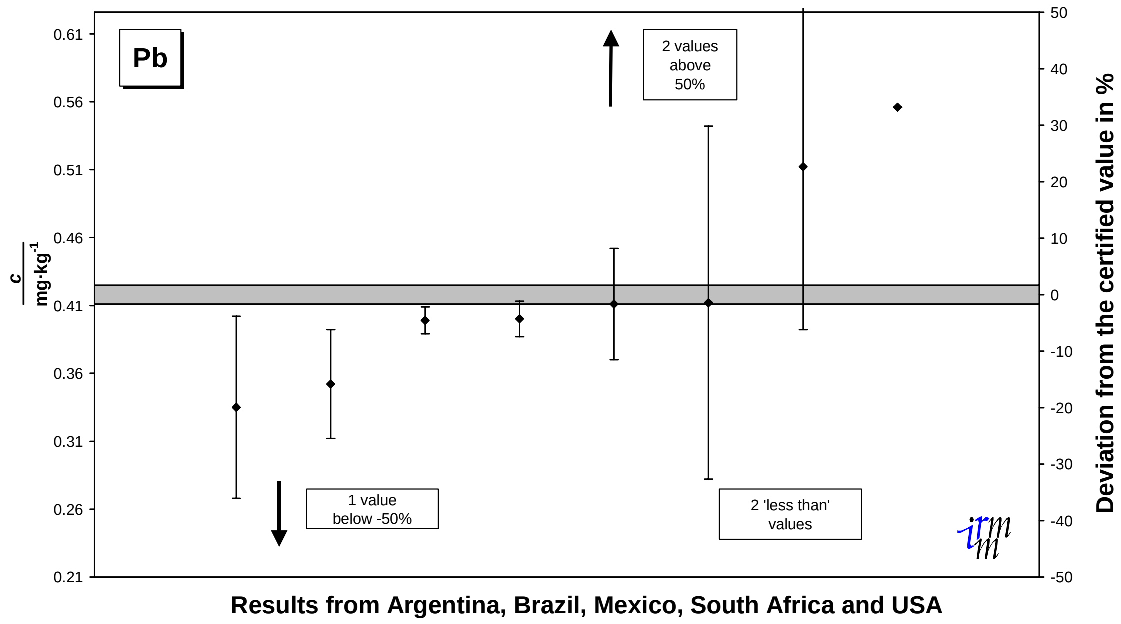


Figure 32

IMEP- 19: Trace elements in Rice
Certified value :22.99 ± 0.44 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*

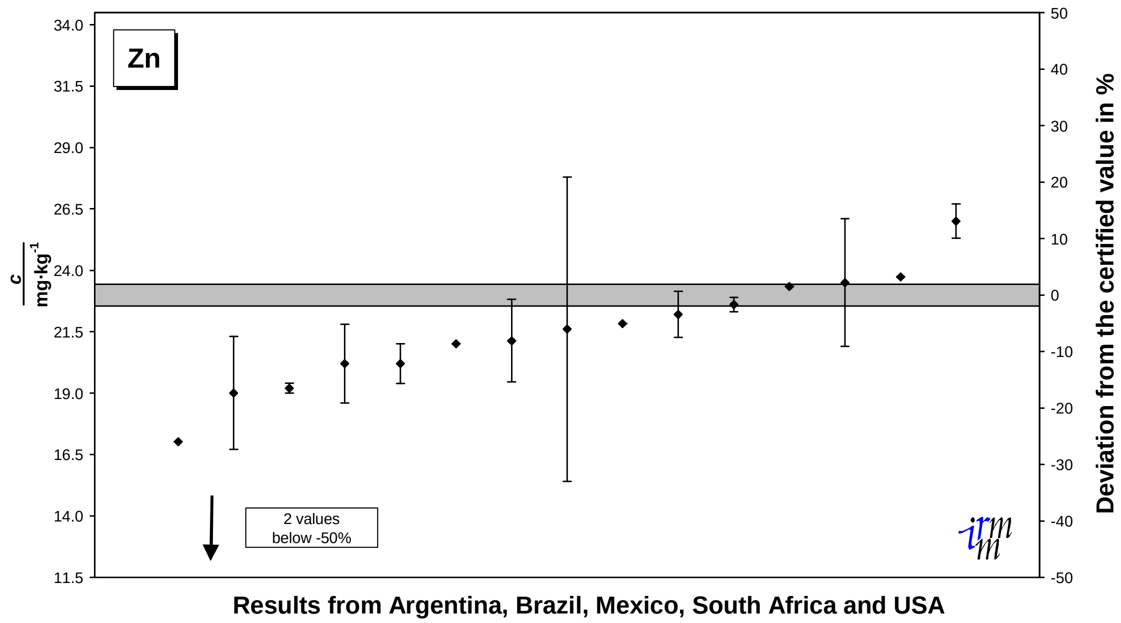
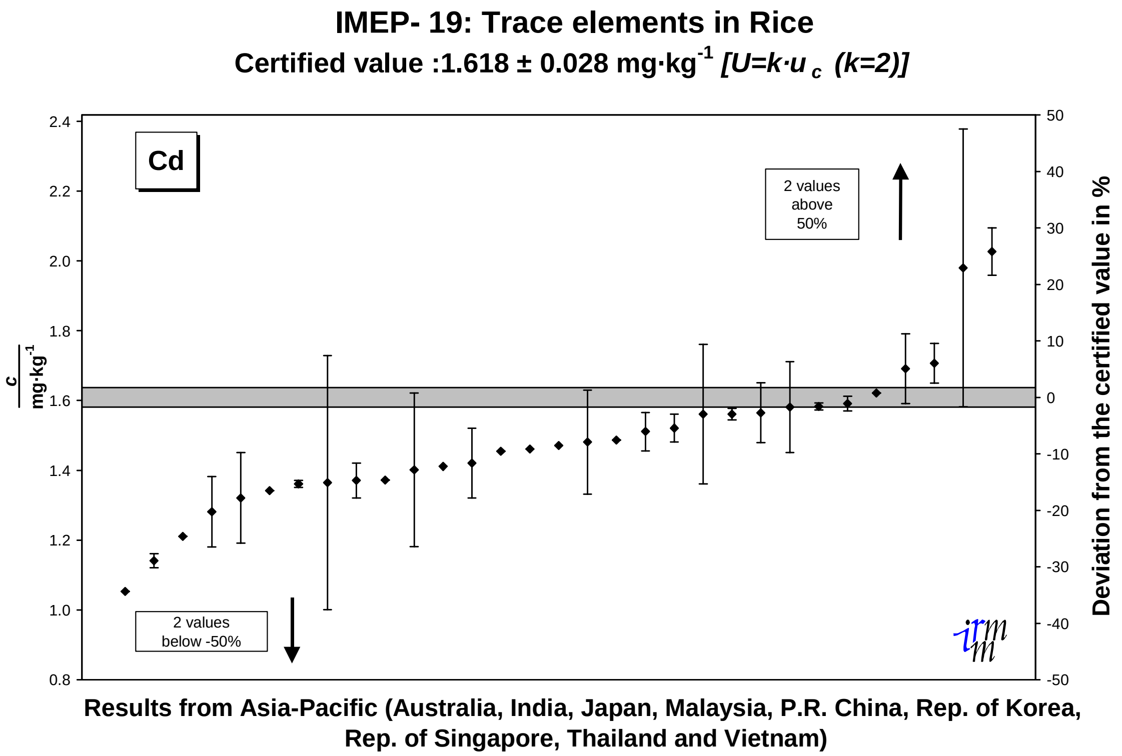


Figure 33



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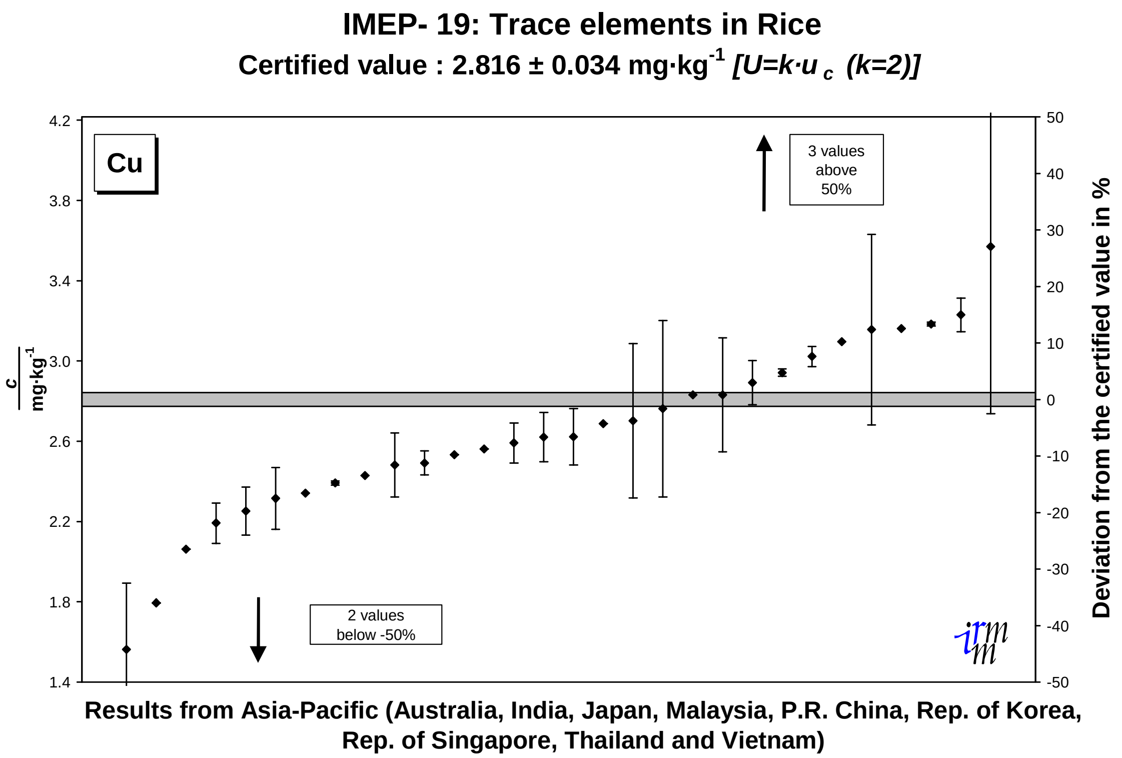

Figure 35

IMEP- 19: Trace elements in Rice
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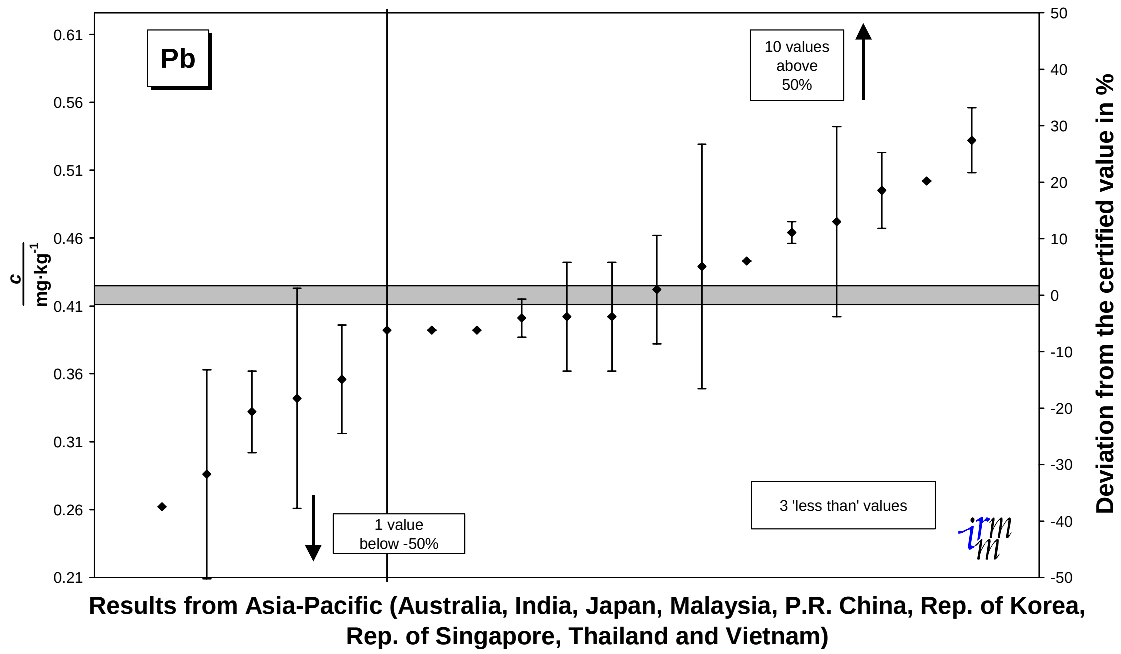
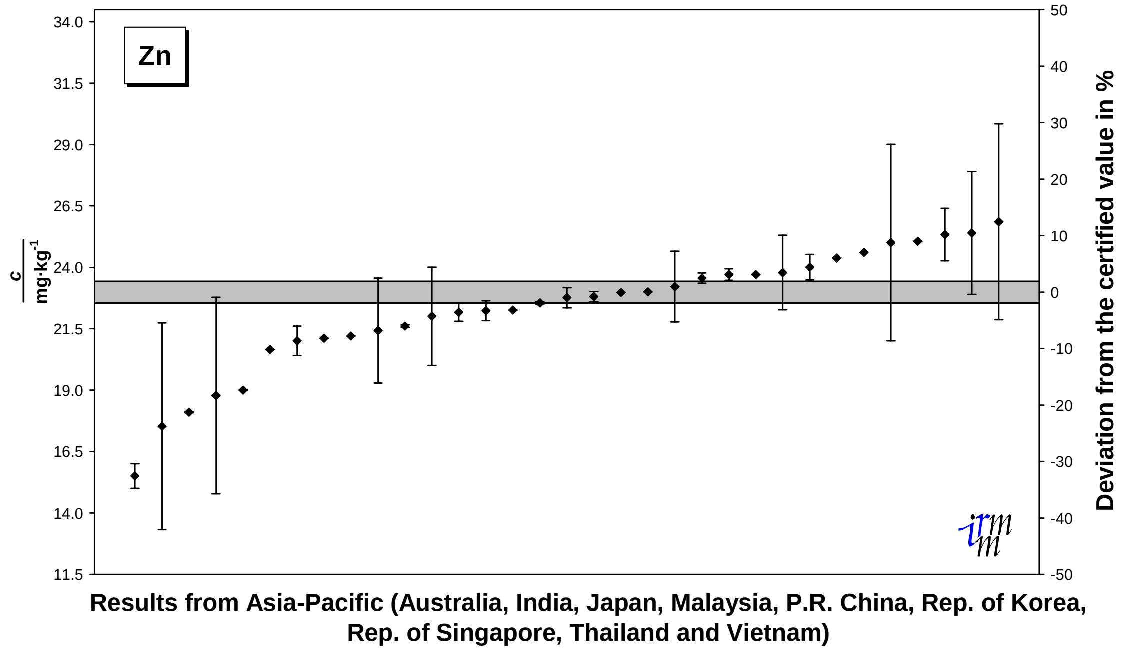


Figure 36

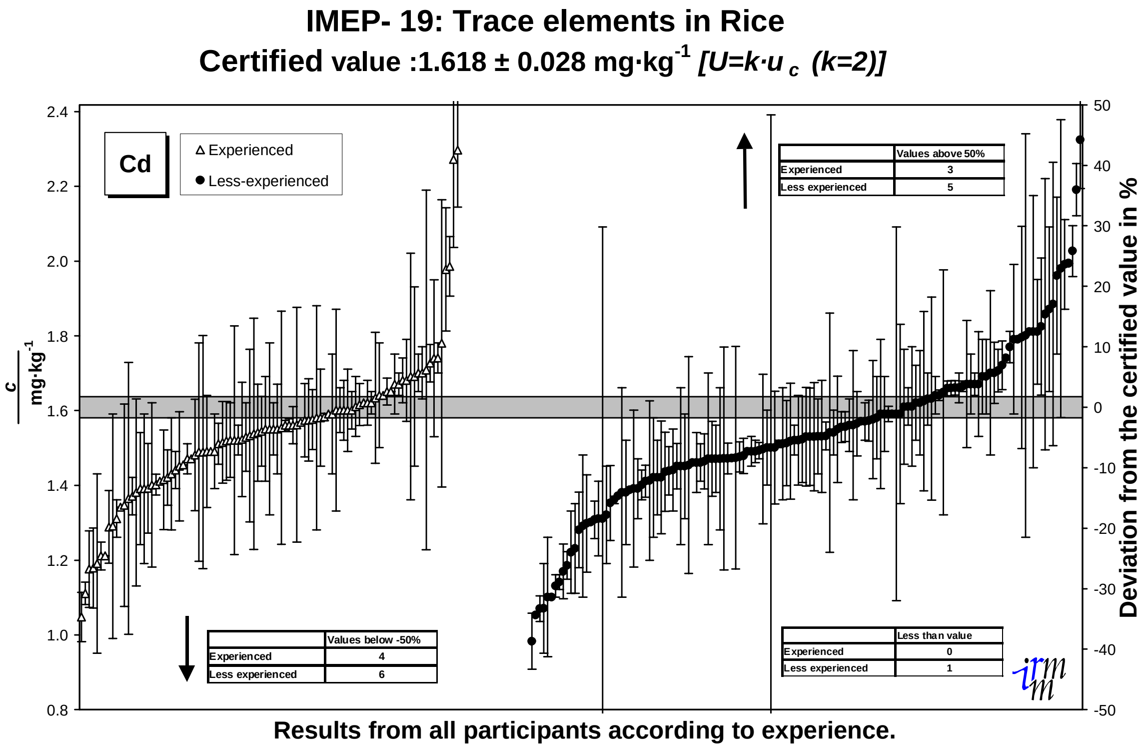
IMEP- 19: Trace elements in Rice
Certified value :22.99 ± 0.44 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



IMEP-19

participants' results - Questionnaire graphs by element

Figure 37



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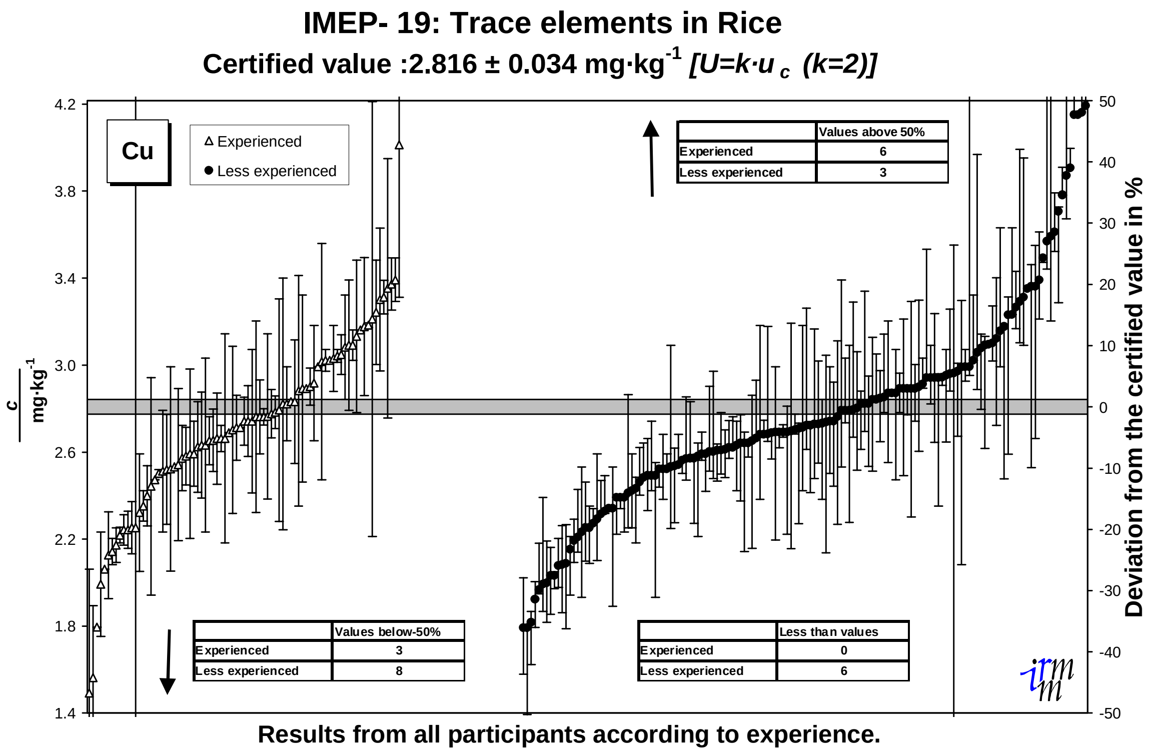
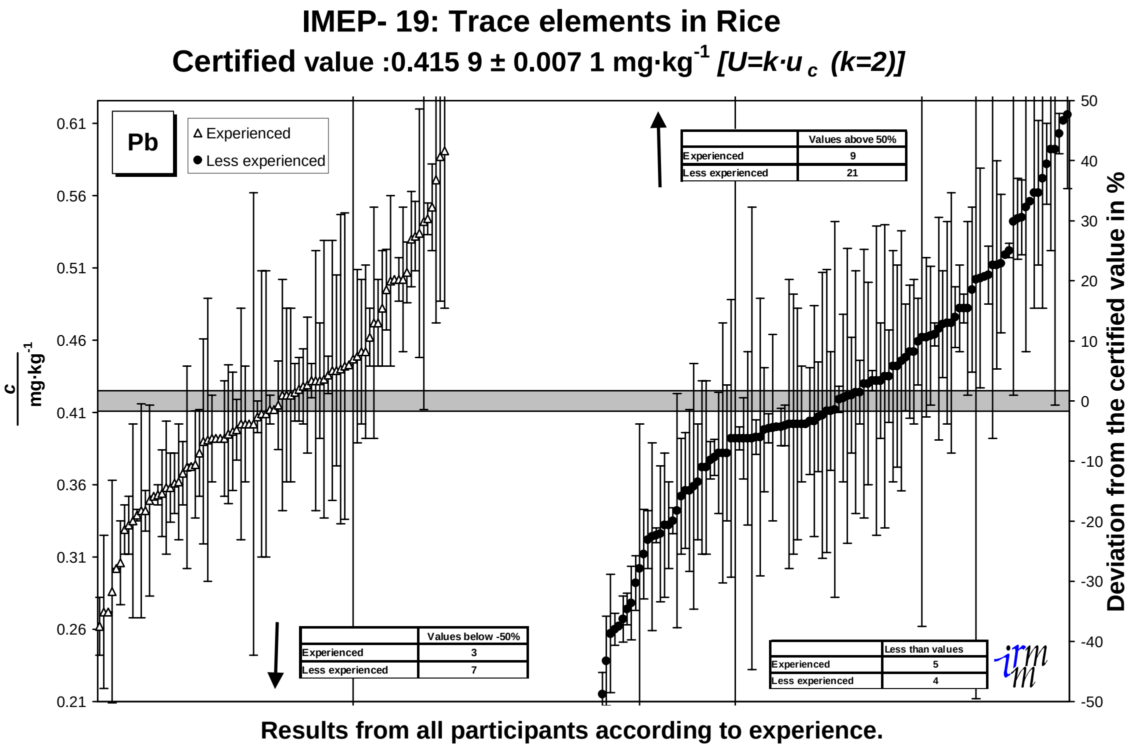
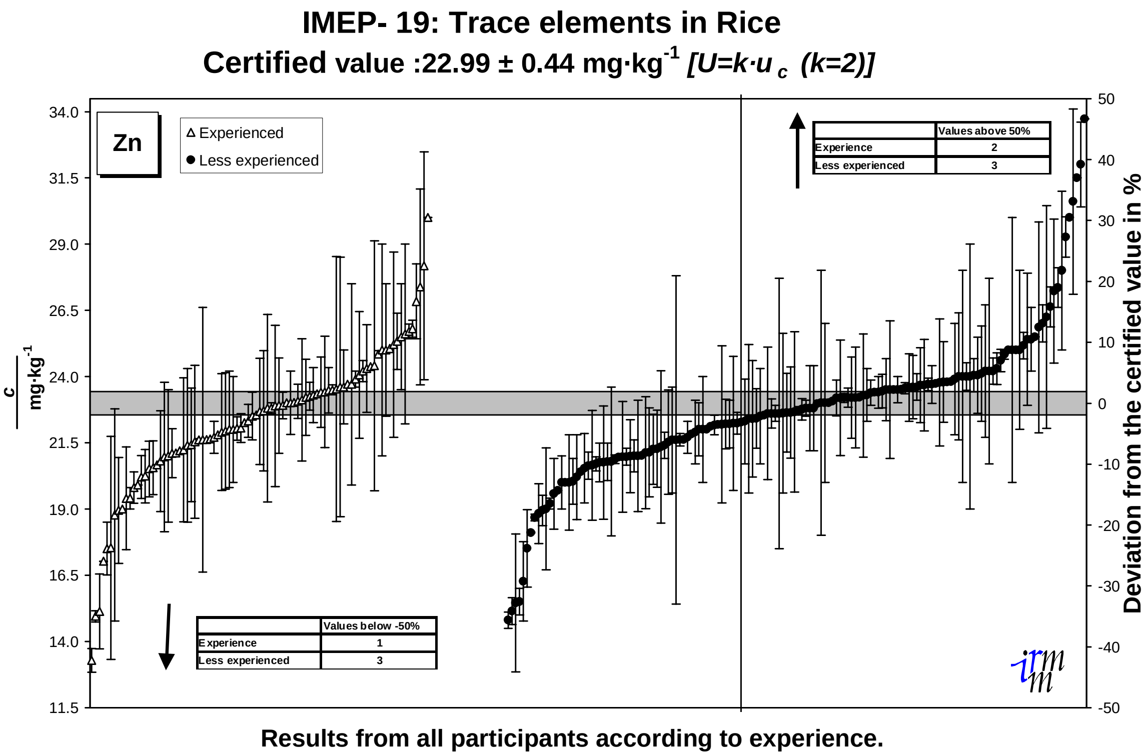


Figure 39



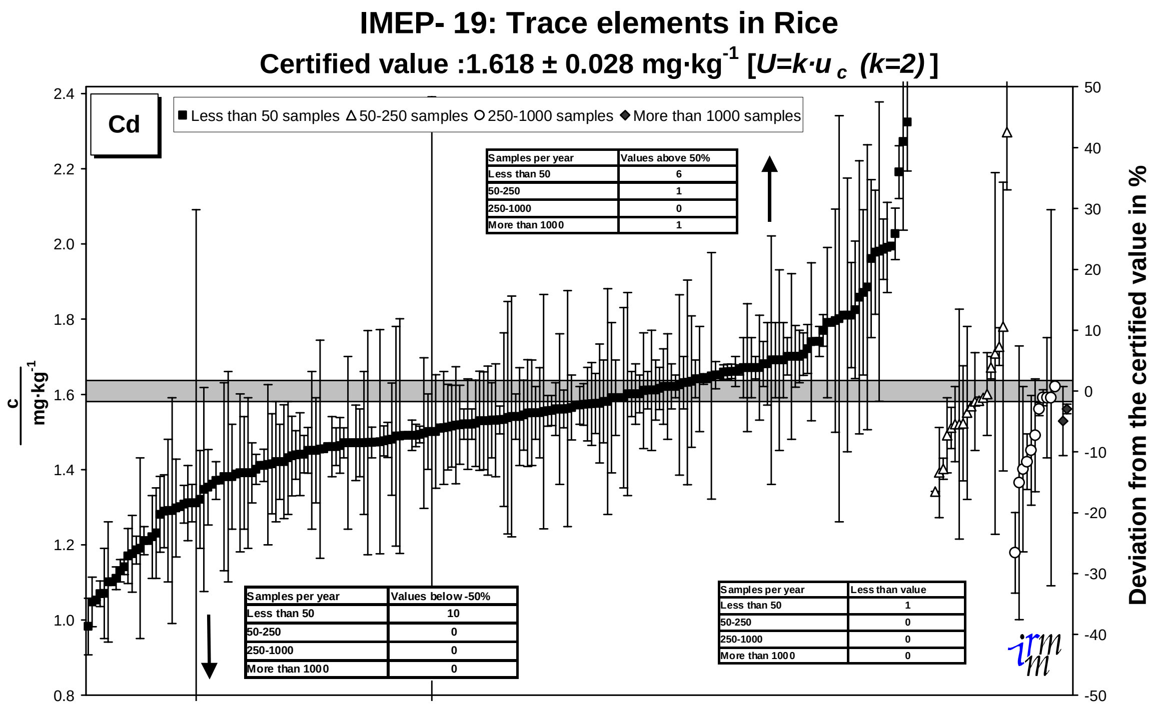
Results from all participants according to experience.

Figure 40



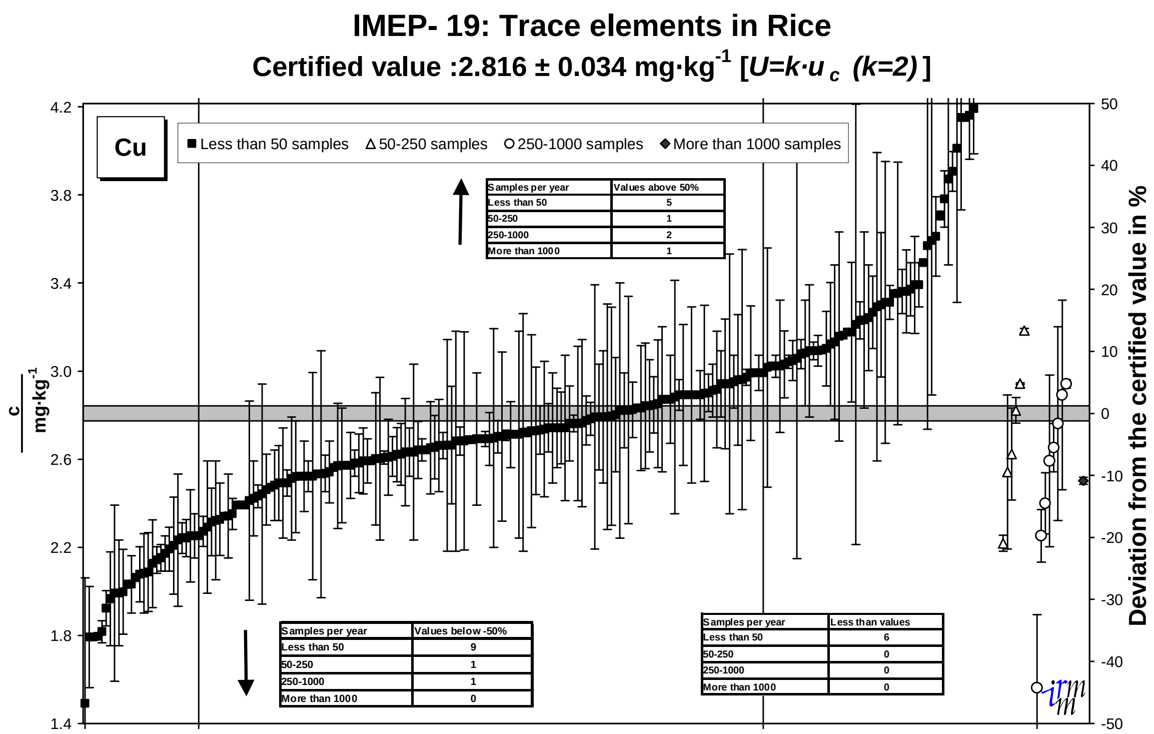
Results from all participants according to experience.

Figure 41



Results from all participants according to how many samples analysed per year.

Figure 42

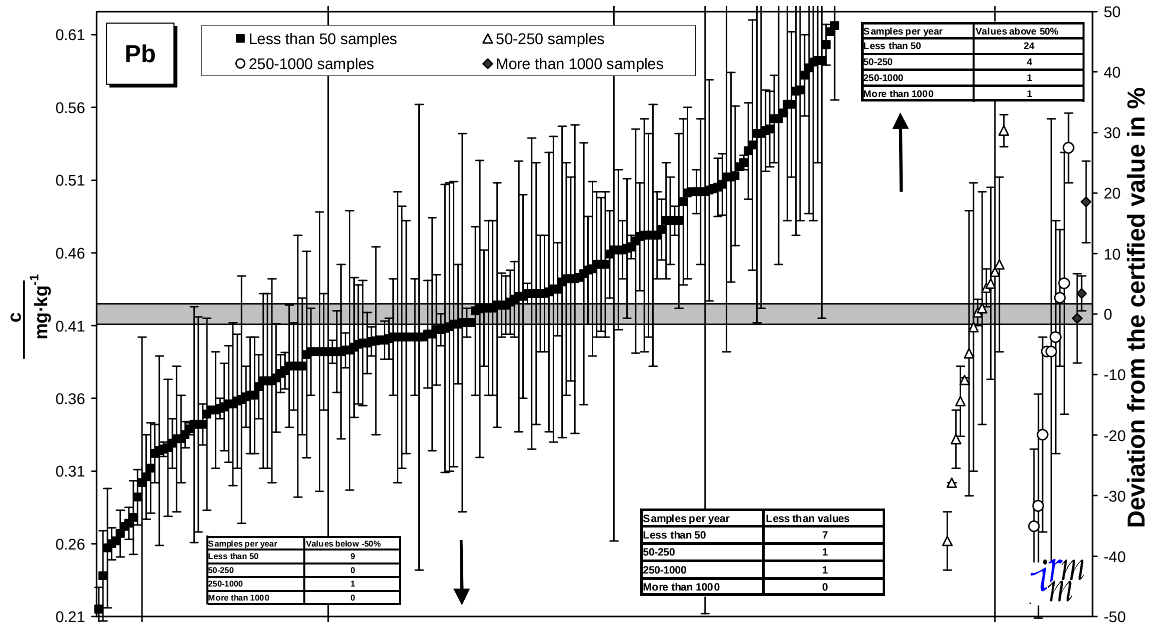


Results from all participants according to how many samples analysed per year.

Figure 43

IMEP- 19: Trace elements in Rice

Certified value :0.415 9 ± 0.007 1 mg·kg^{-1} [$U=k \cdot u_c$ ($k=2$)]

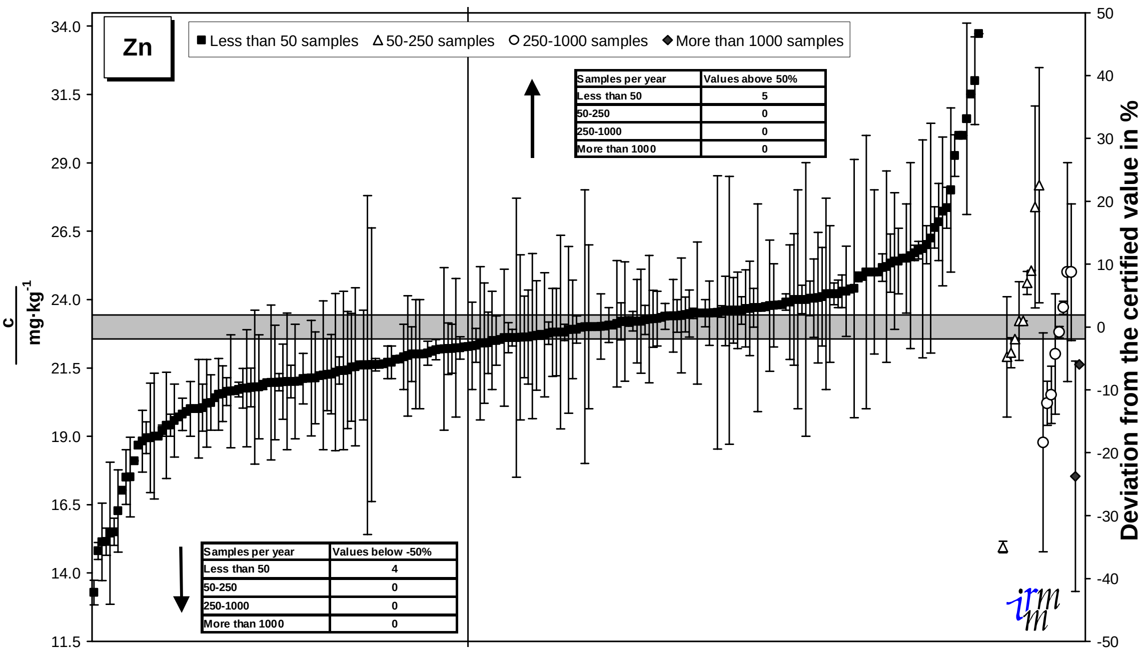


Results from all participants according to how many samples analysed per year.

Figure 44

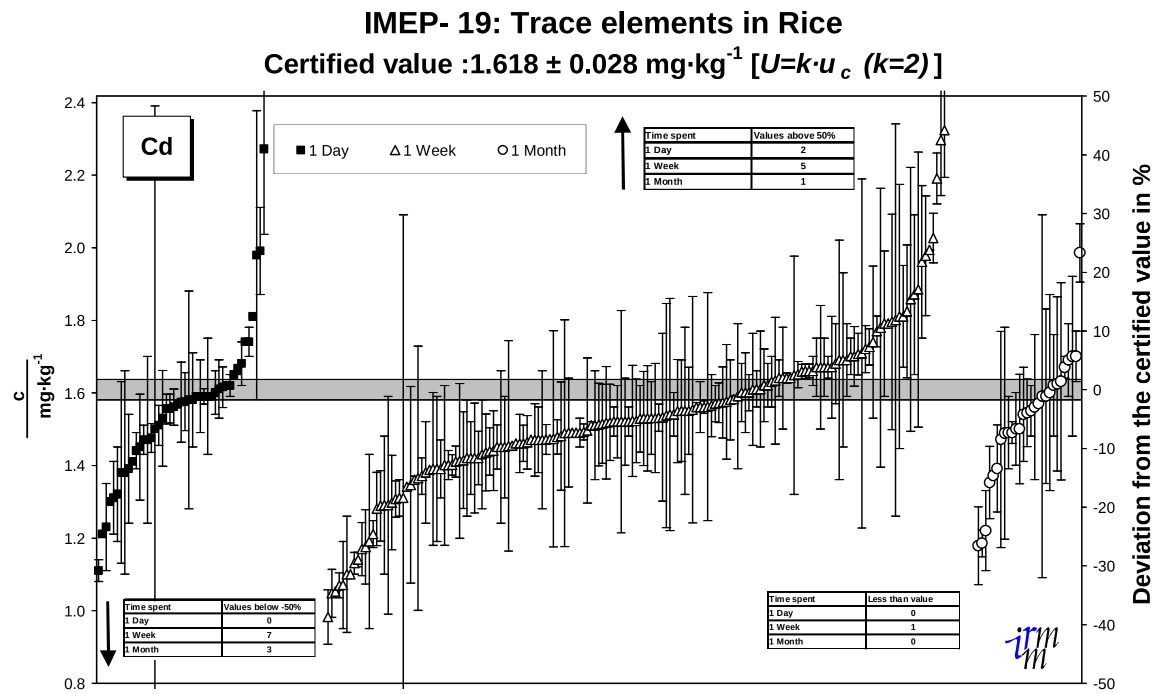
IMEP- 19: Trace elements in Rice

Certified value :22.99 ± 0.44 mg·kg^{-1} [$U=k \cdot u_c$ ($k=2$)]



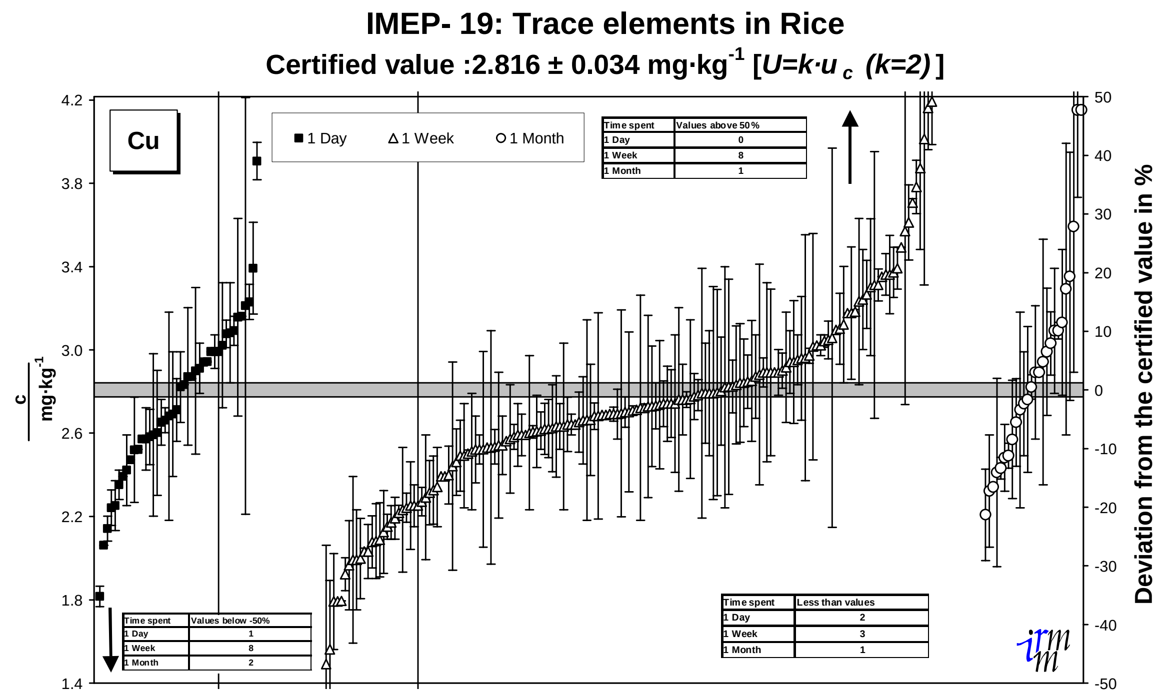
Results from all participants according how many samples analysed per year.

Figure 45



Results from all participants according to the time spent on the measurement.

Figure 46

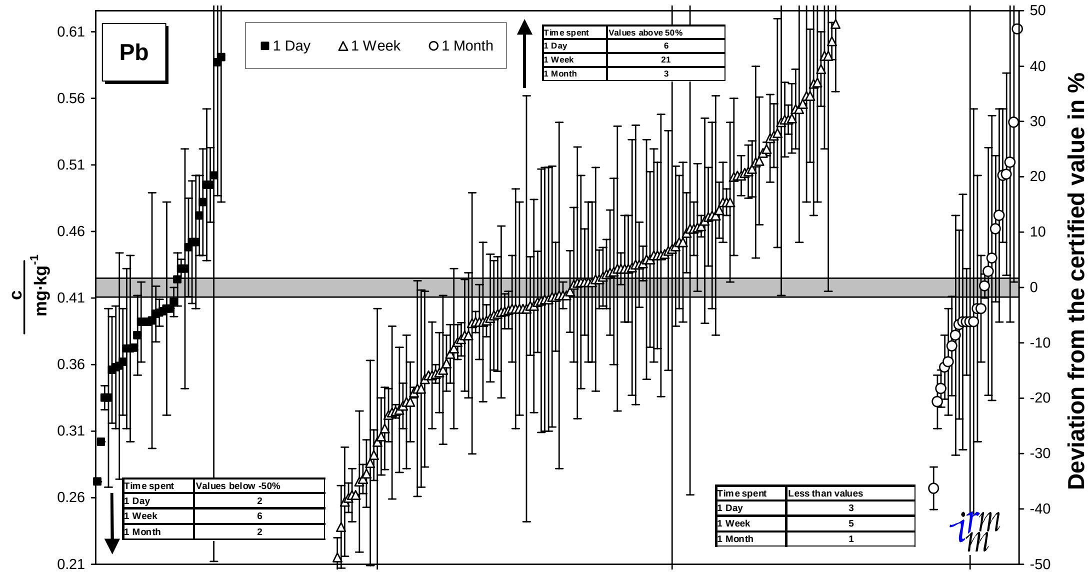


Results from all participants according to the time spent on the measurement.

Figure 47

IMEP- 19: Trace elements in Rice

Certified value :0.415 9 ± 0.007 1mg·kg^{-1} [$U=k \cdot u_c$ $(k=2)$]

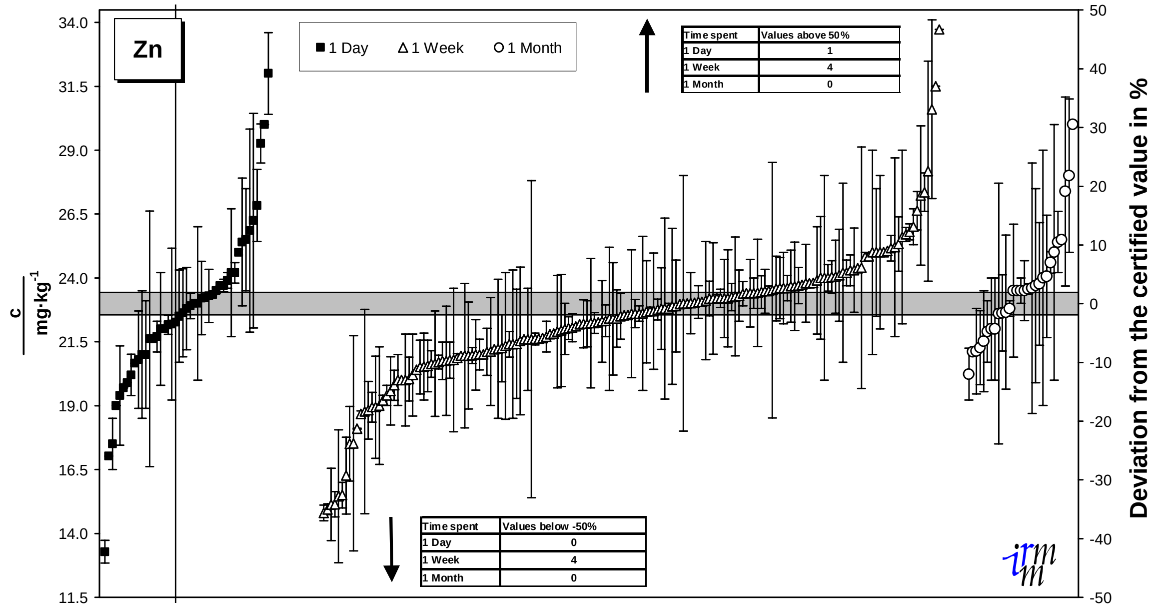


Results from all participants according to the time spent on the measurement.

Figure 48

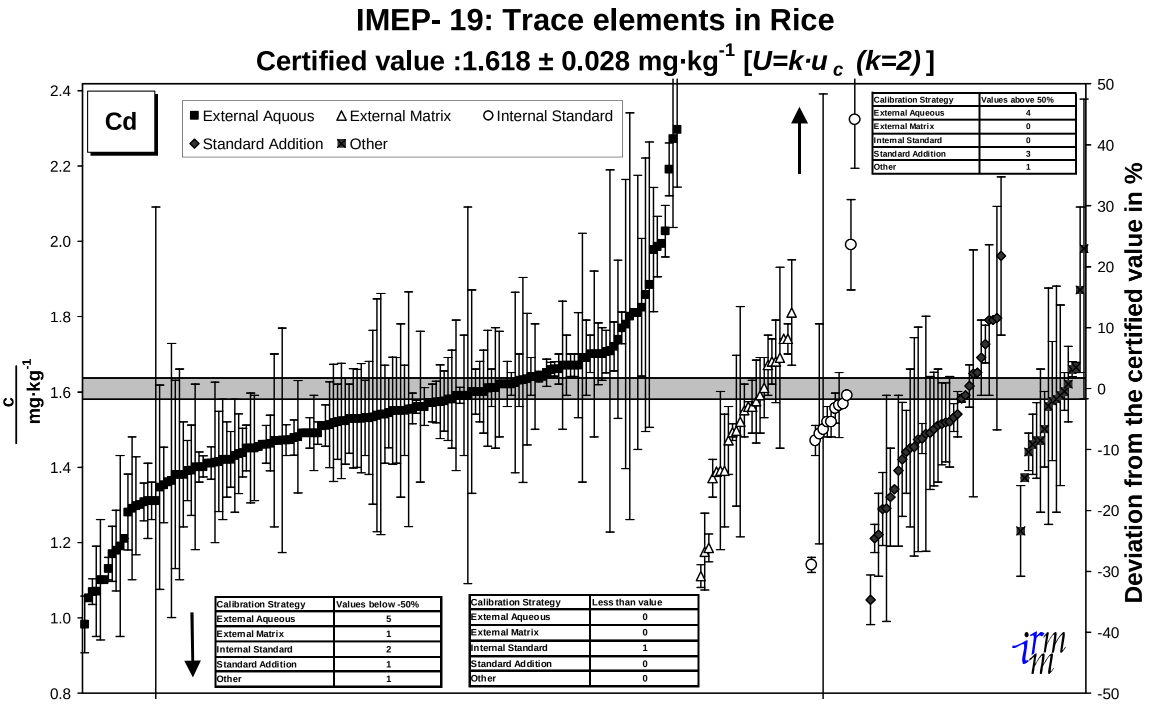
IMEP- 19: Trace elements in Rice

Certified value :22.99 ± 0.44 mg·kg^{-1} [$U=k \cdot u_c$ $(k=2)$]



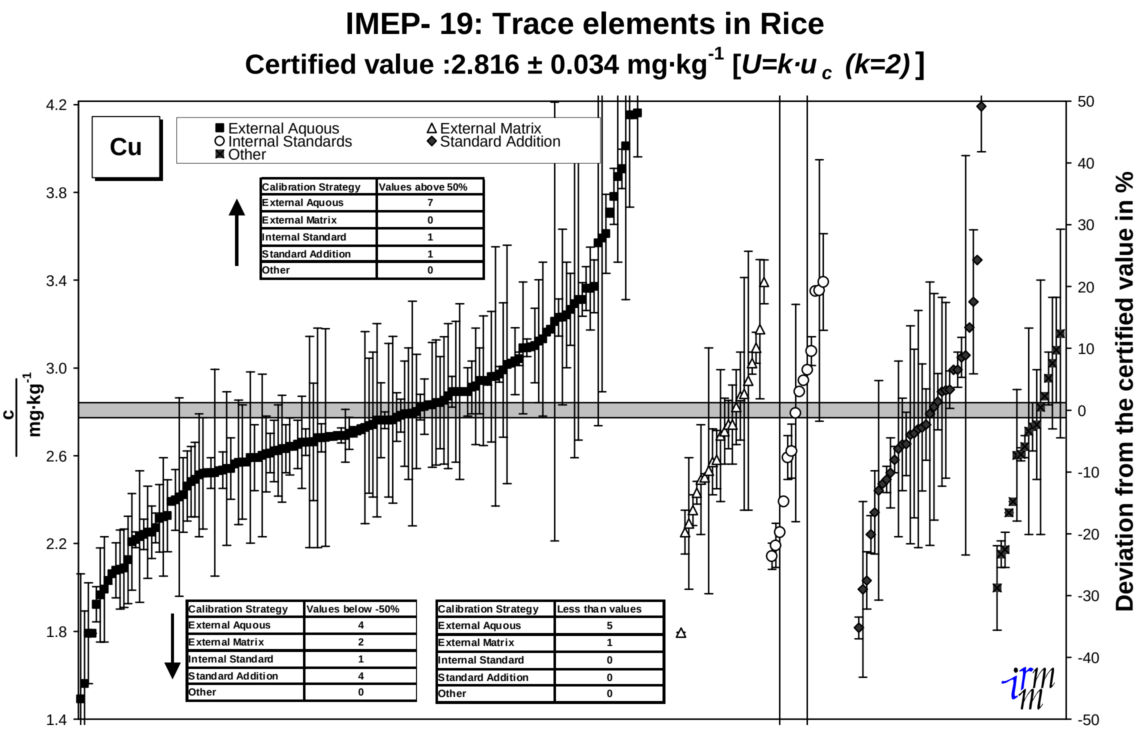
Results from all participants according to the time spent on the measurement.

Figure 49



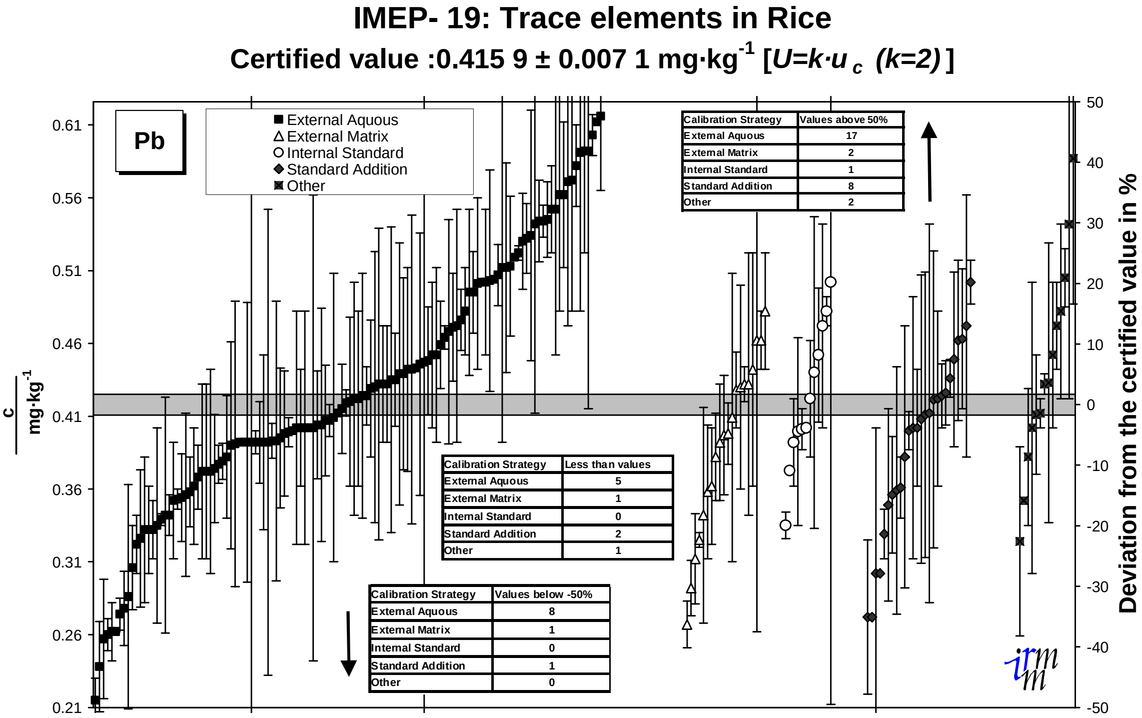
Results from all participants according to Calibration Strategy.

Figure 50



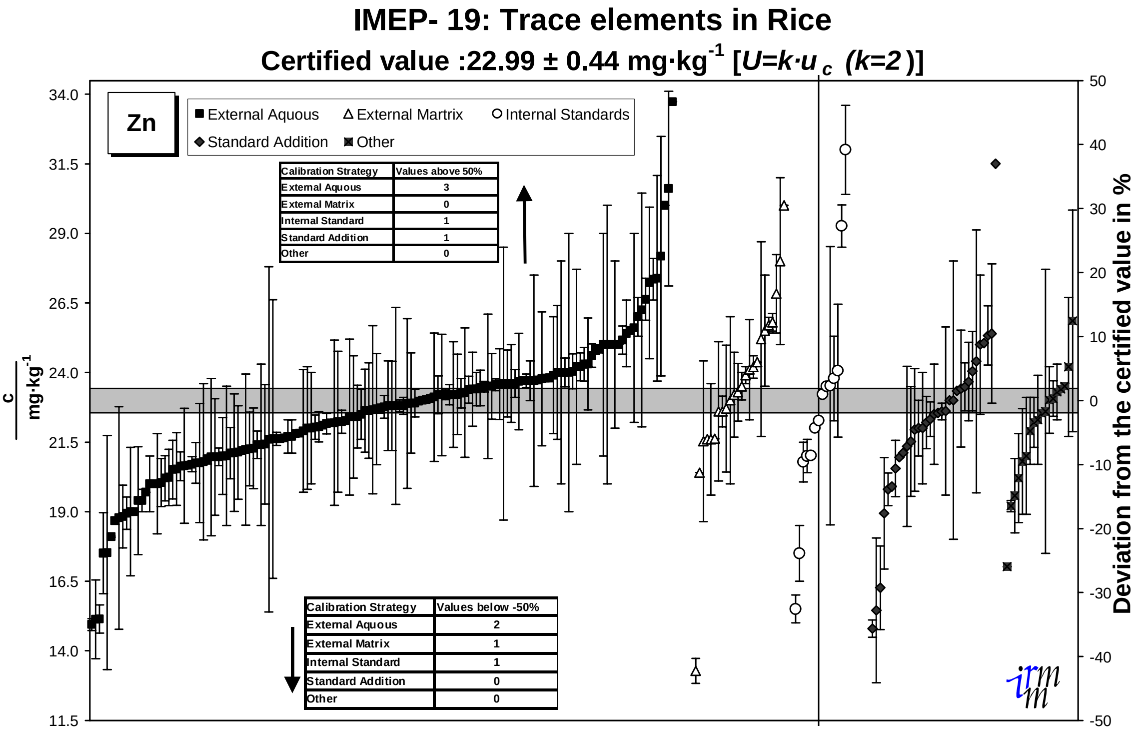
Results from all participants according to Calibartion Strategy.

Figure 51



Results from all participants according to Calibration Strategy.

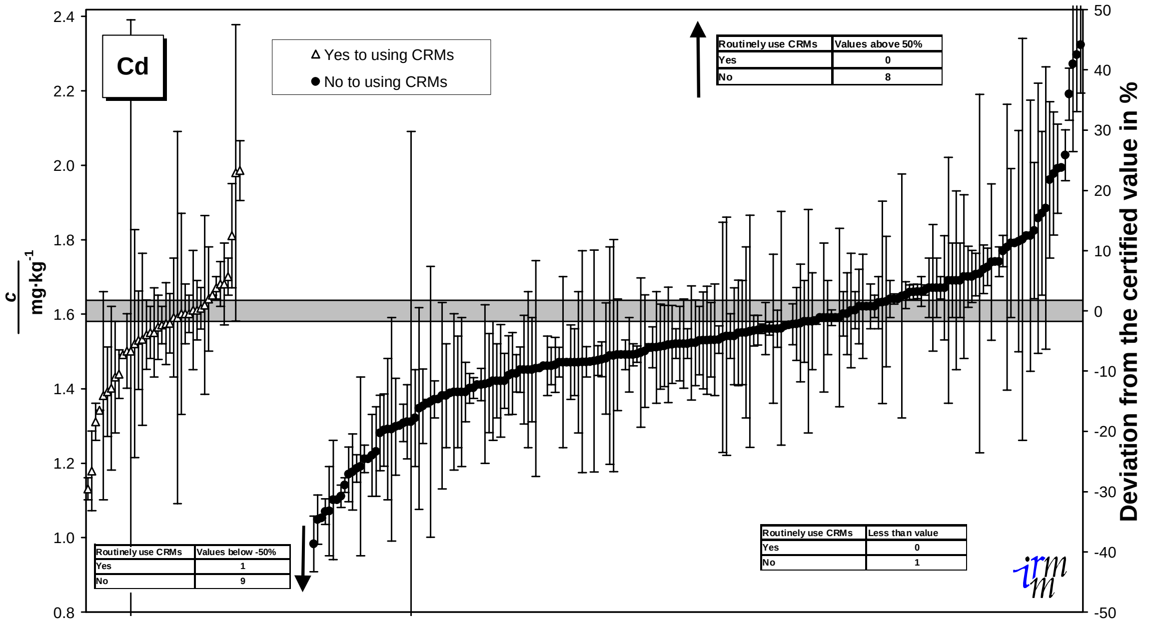
Figure 52



Results from all participants according to Calibration Strategy.

Figure 53

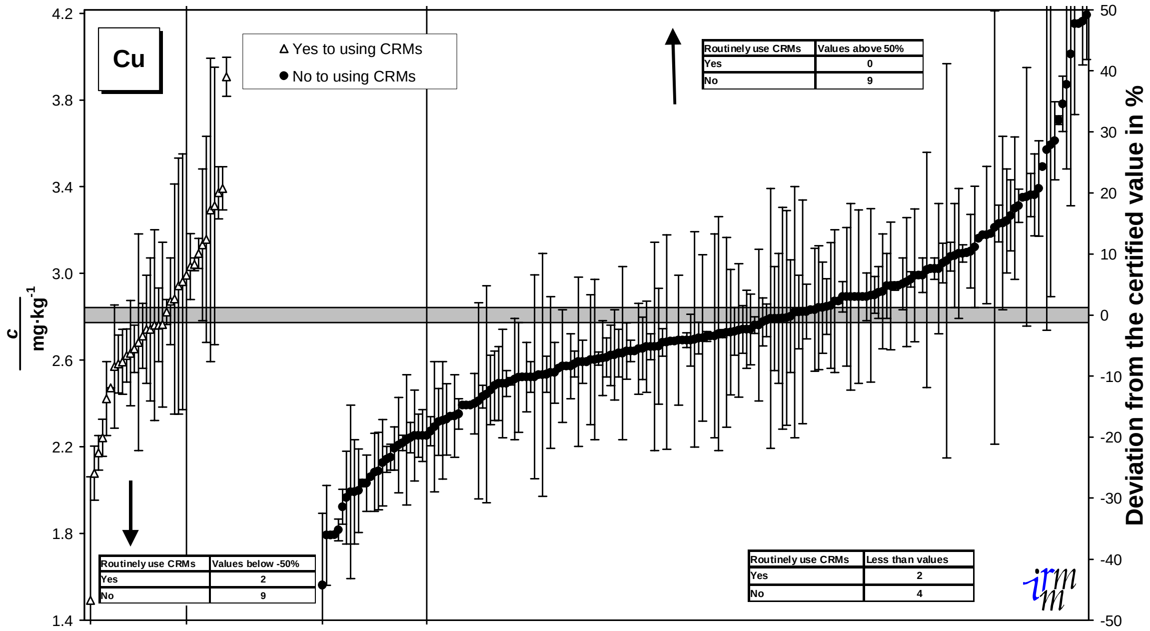
IMEP- 19: Trace elements in Rice
Certified value :1.618 ± 0.028 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants on the use of Certified Reference Materials. (CRMs)

Figure 54

IMEP- 19: Trace elements in Rice
Certified value :2.816 ± 0.034 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants on the use of Certified Reference Materials. (CRMs)

Figure 55

IMEP- 19: Trace elements in Rice
Certified value :0.415 9 ± 0.007 1 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*

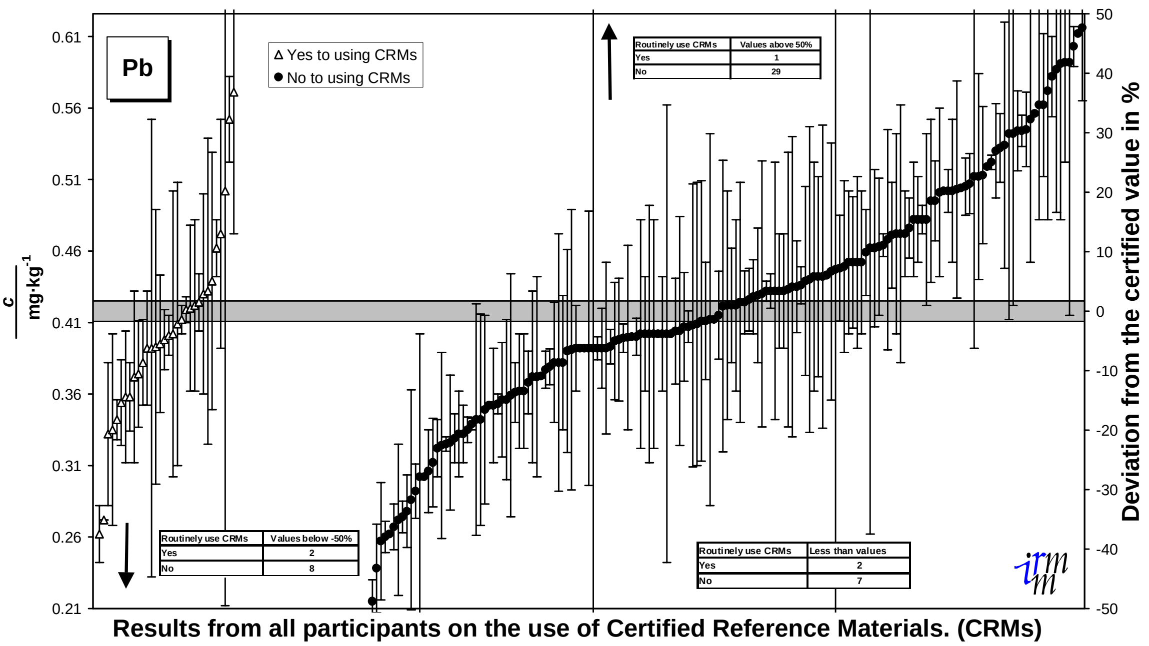


Figure 56

IMEP- 19: Trace elements in Rice
Certified value :22.99 ± 0.44 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*

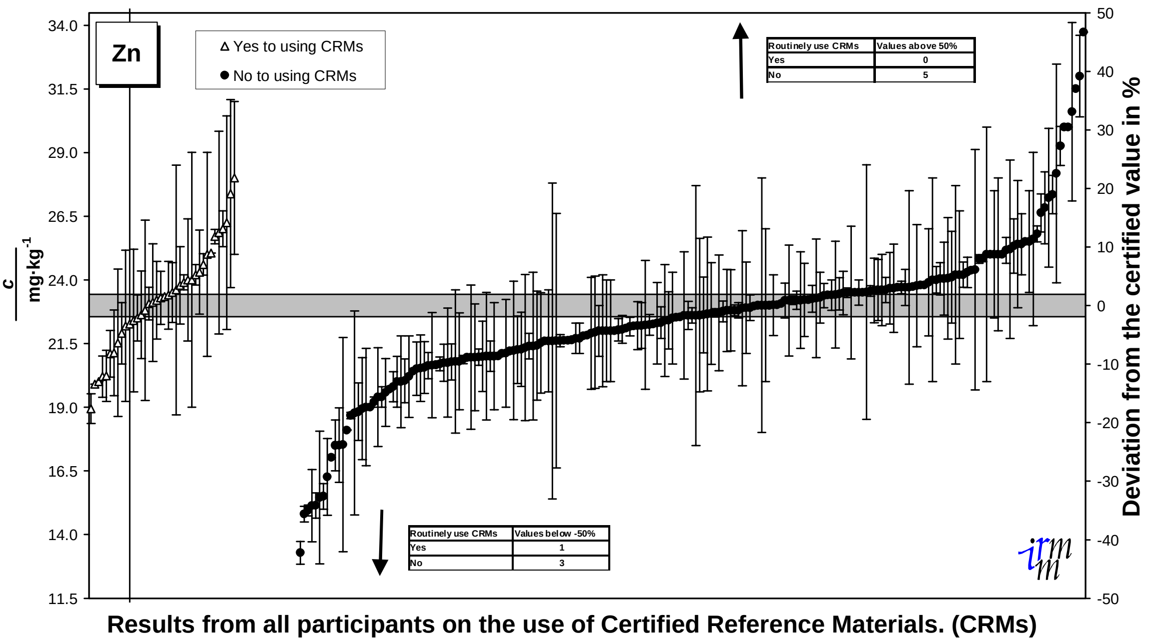
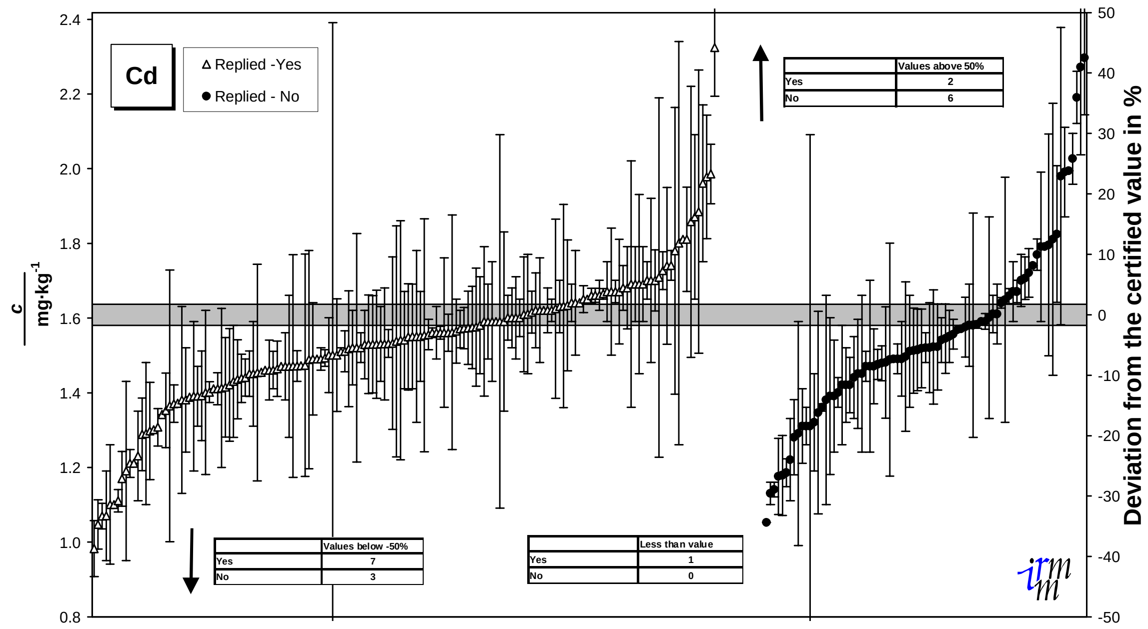


Figure 57

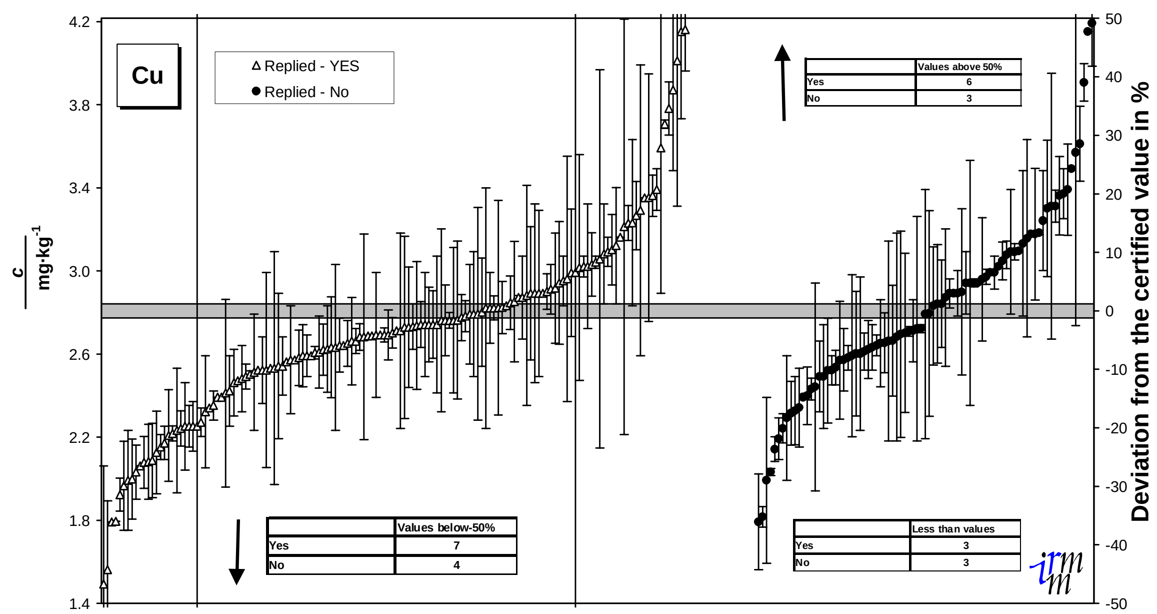
IMEP- 19: Trace elements in Rice
Certified value :1.618 ± 0.028 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants according to there participation in proficiency testing schemes.(PTs)

Figure 58

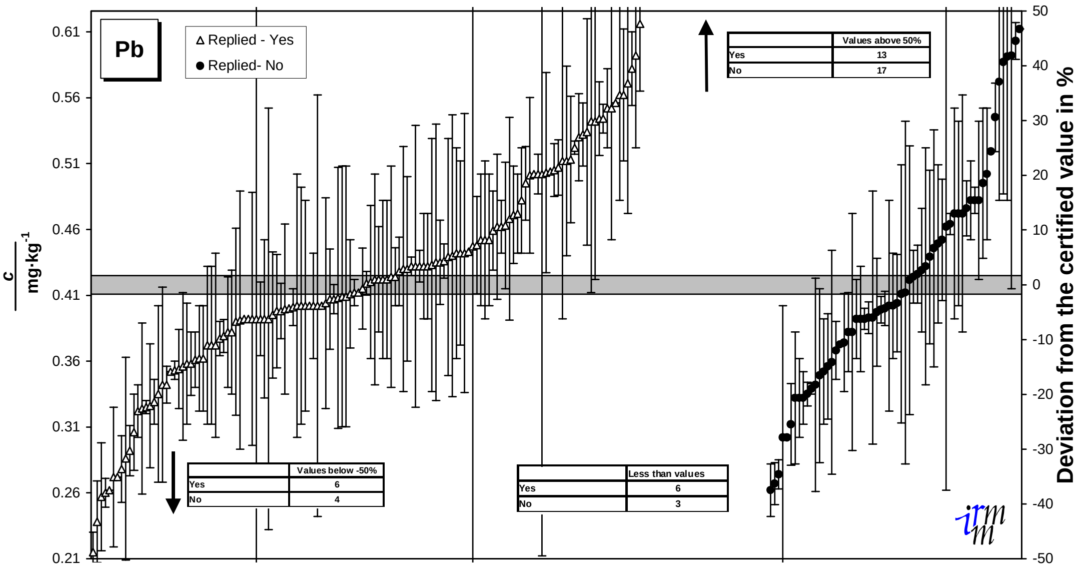
IMEP- 19: Trace elements in Rice
Certified value :2.816 ± 0.034 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants according to there participation in proficiency testing schemes. (PTs)

Figure 59

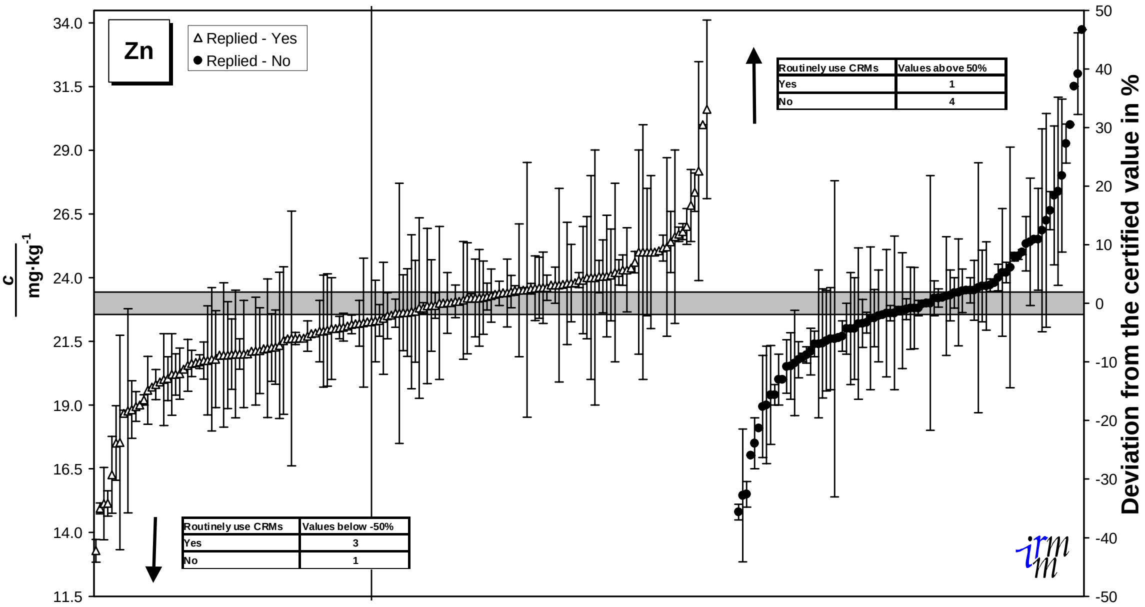
IMEP- 19: Trace elements in Rice
Certified value :0.415 9 ± 0.007 1 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants according to there participation in proficiency testing schemes. (PTs)

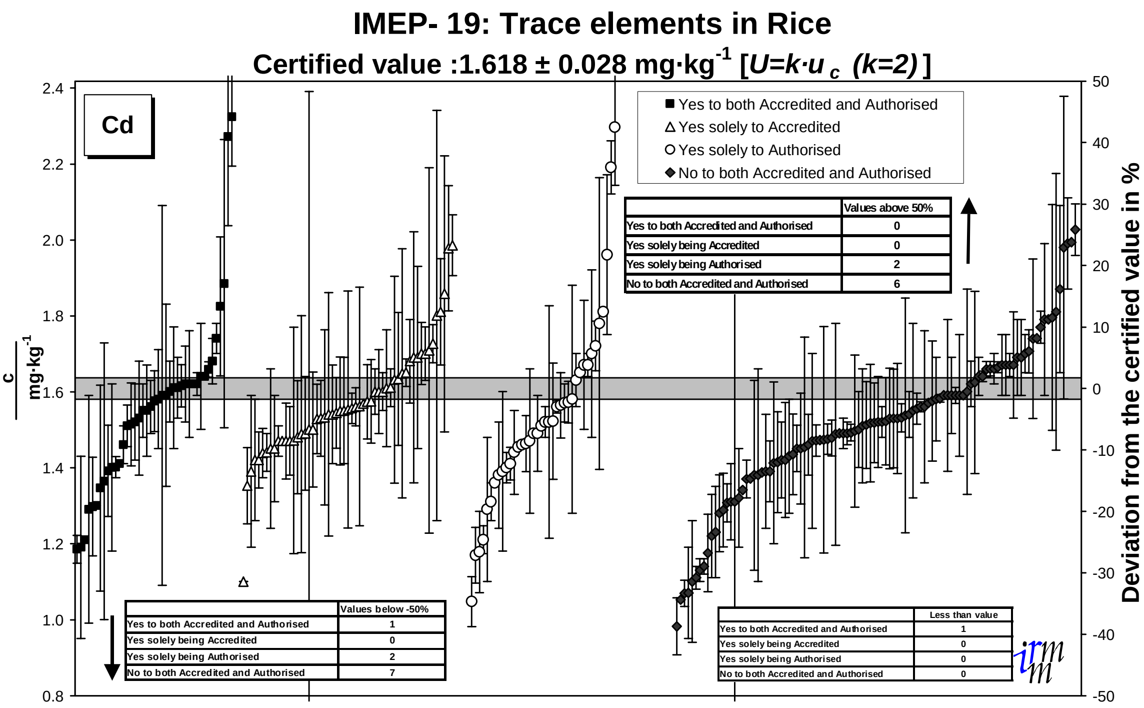
Figure 60

IMEP- 19: Trace elements in Rice
Certified value :22.99 ± 0.44 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



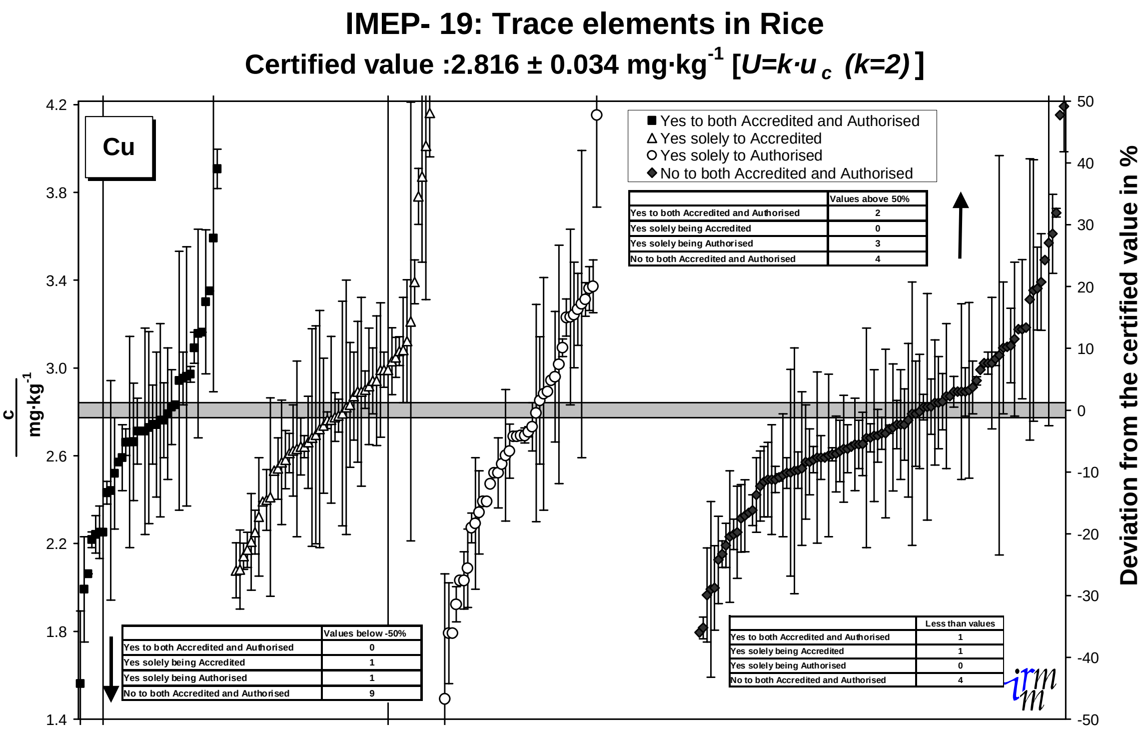
Results from all participants according to there participation in proficiency testing schemes. (PTs)

Figure 61



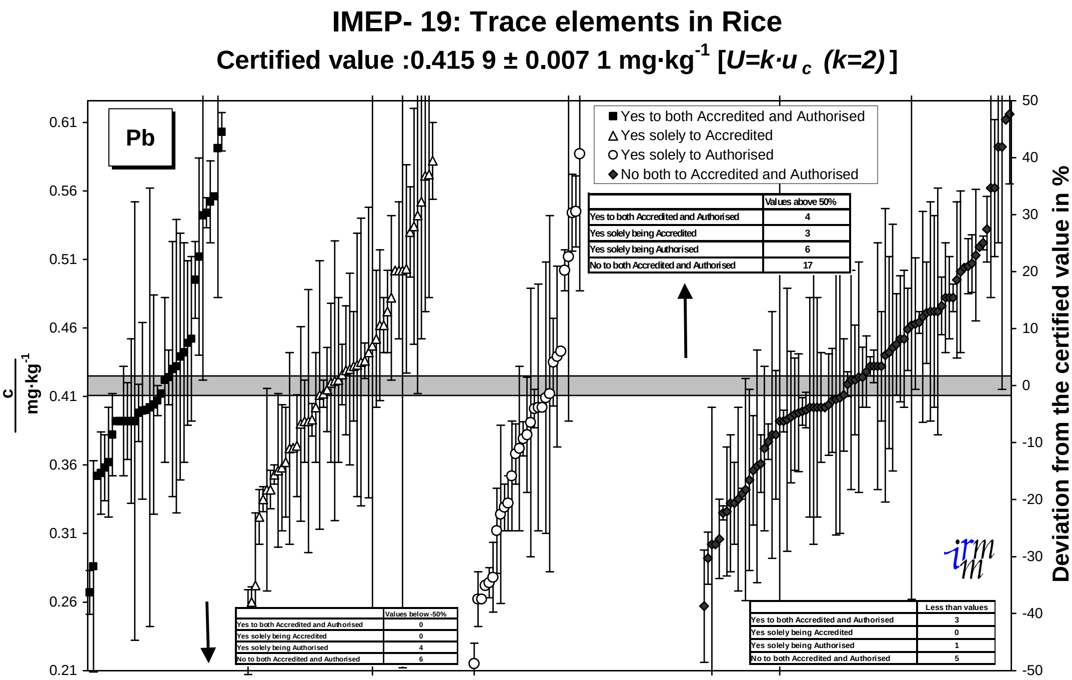
Results from all participants according to Accredited and/or Authorised.

Figure 62



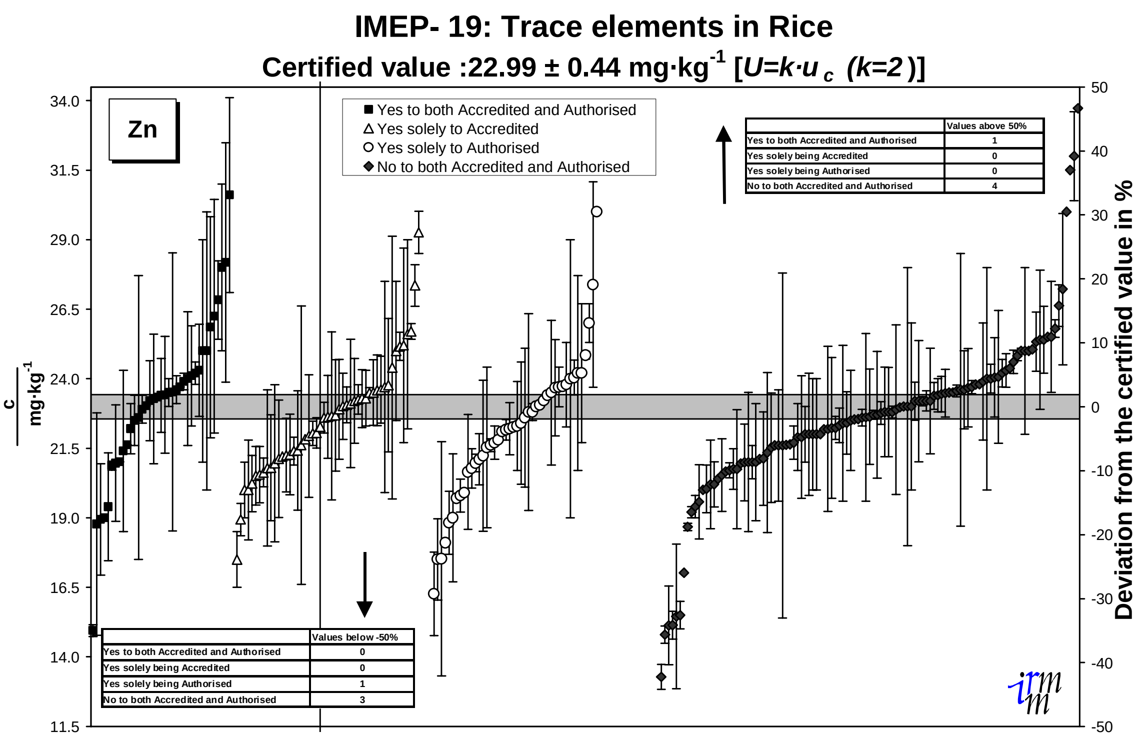
Results from all participants according to Accredited and/or Authorised.

Figure 63



Results from all participants according to Accredited and/or Authorised.

Figure 64



Results from all participants according to Accredited and/or Authorised.

Figure 65

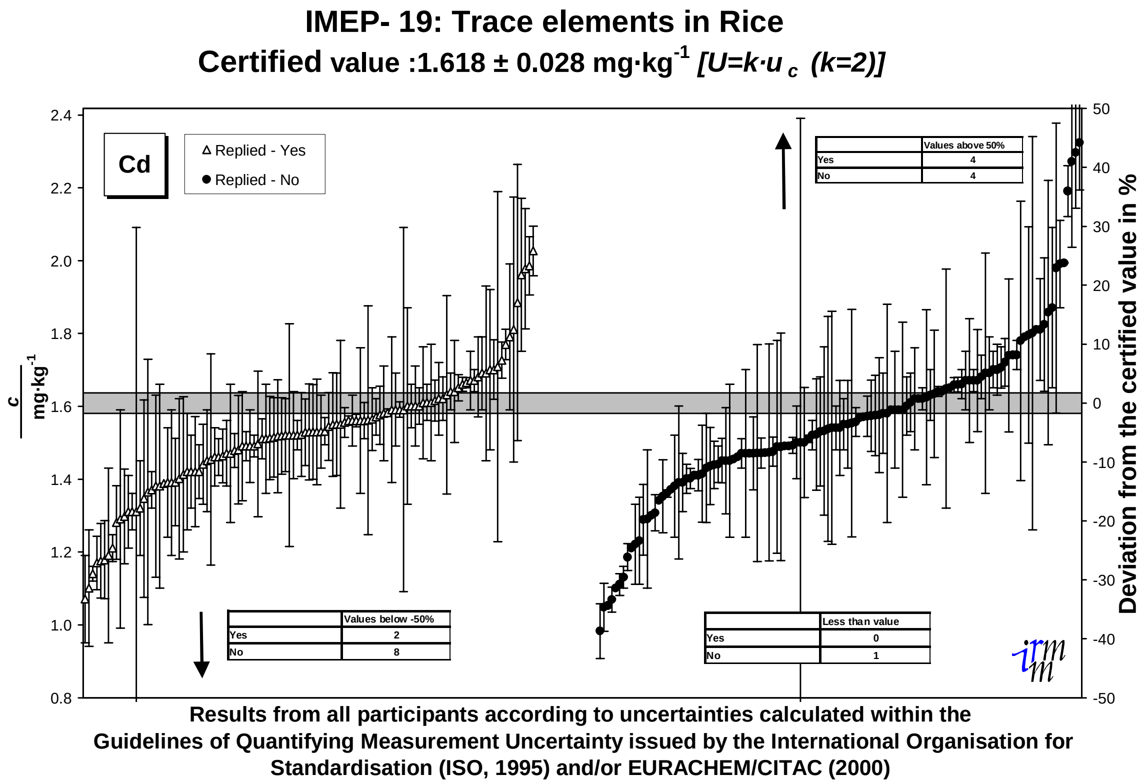


Figure 66

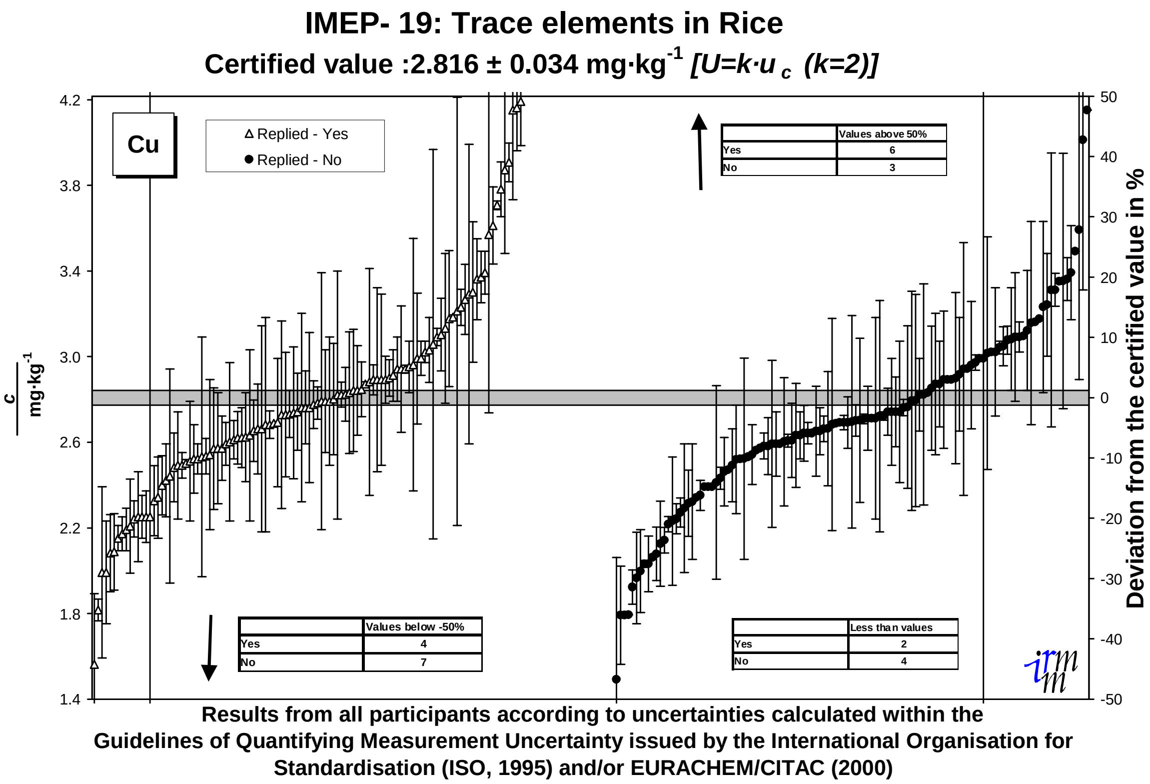
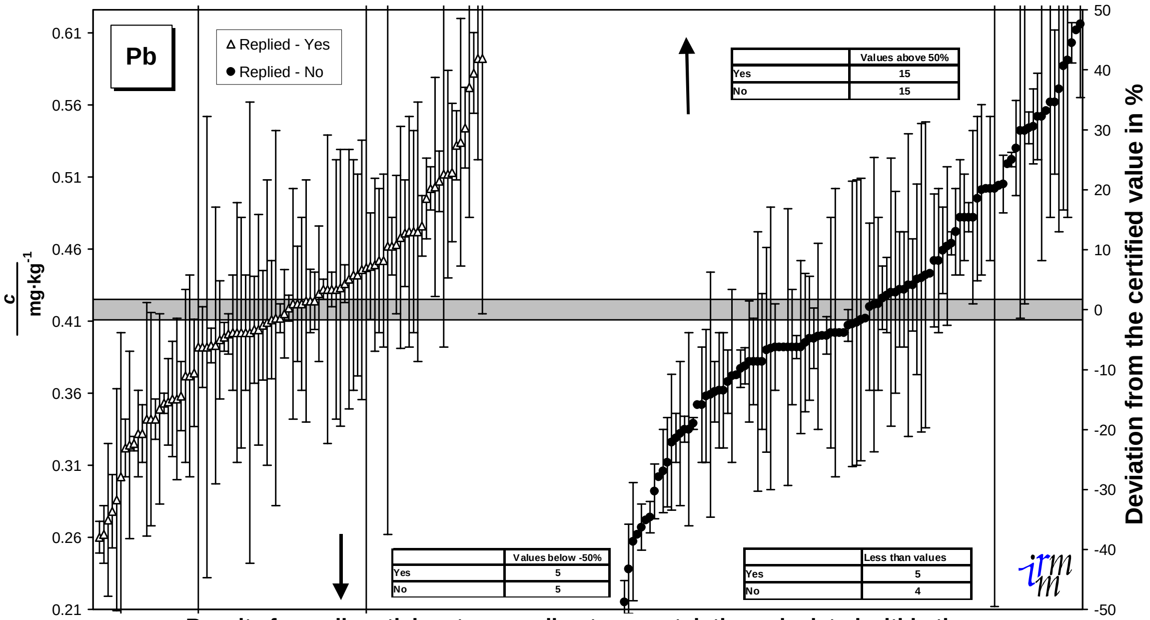


Figure 67

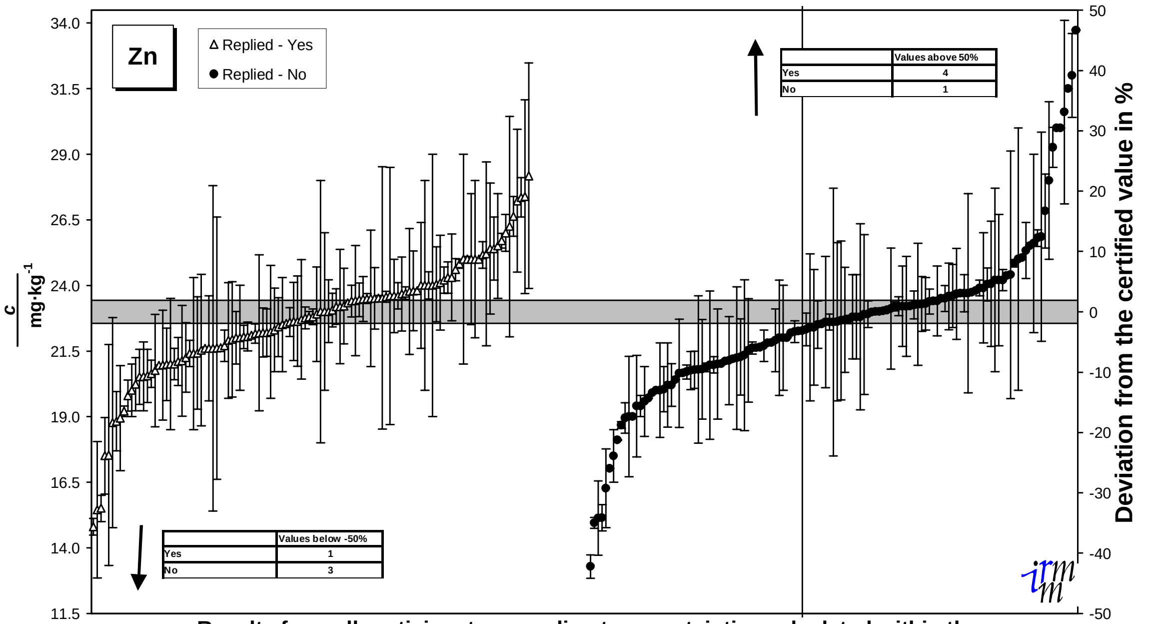
IMEP- 19: Trace elements in Rice
Certified value :0.415 9 ± 0.007 1 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants according to uncertainties calculated within the Guidelines of Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) and/or EURACHEM/CITAC (2000)

Figure 68

IMEP- 19: Trace elements in Rice
Certified value :22.99 ± 0.44 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants according to uncertainties calculated within the Guidelines of Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) and/or EURACHEM/CITAC (2000)

Figure 69

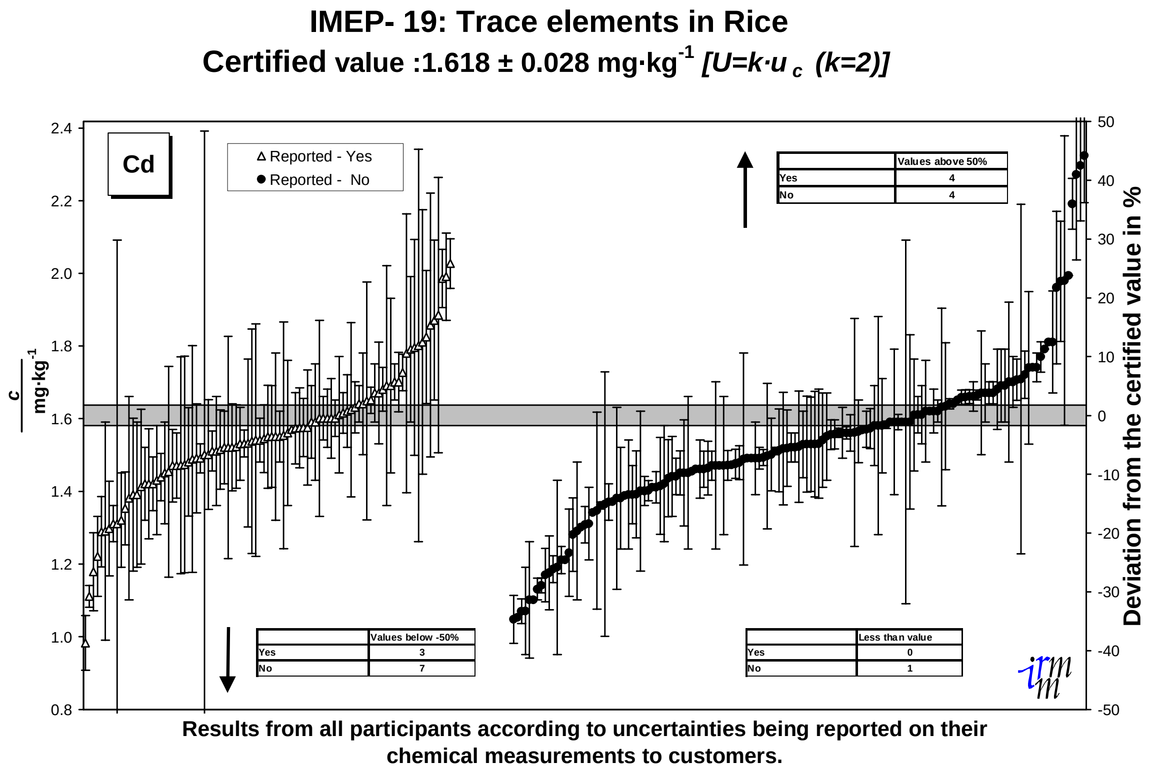


Figure 70

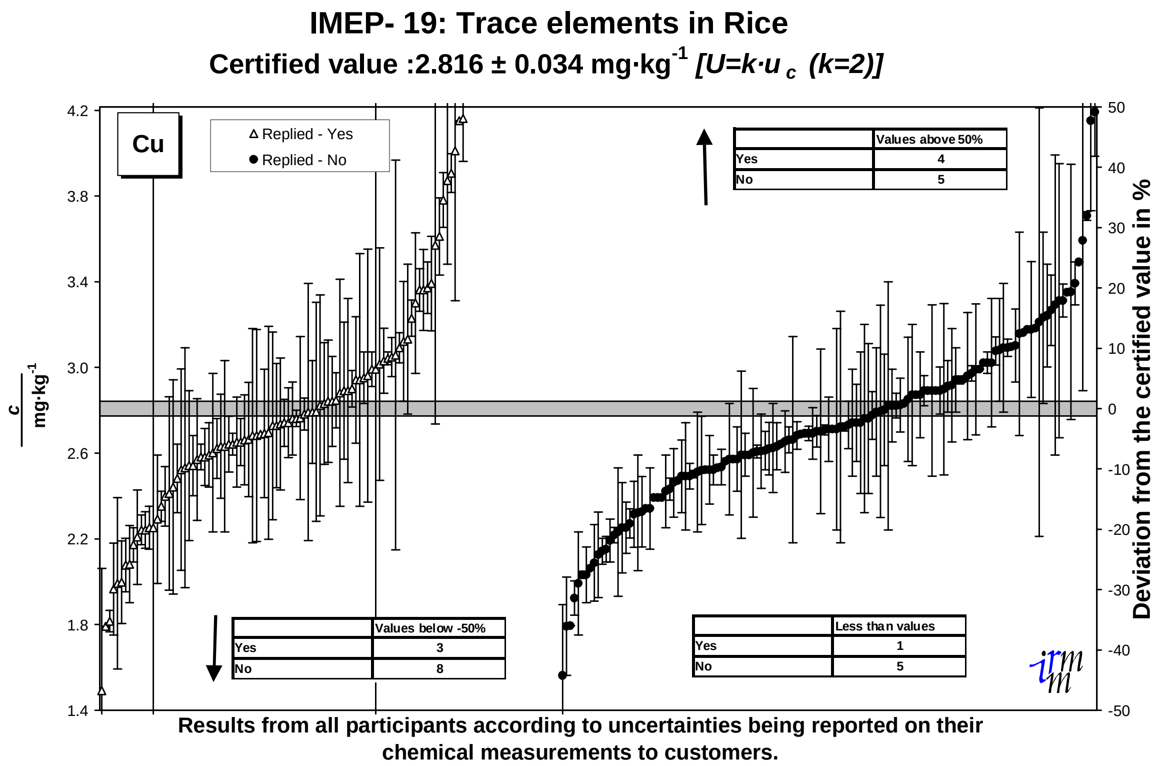


Figure 71

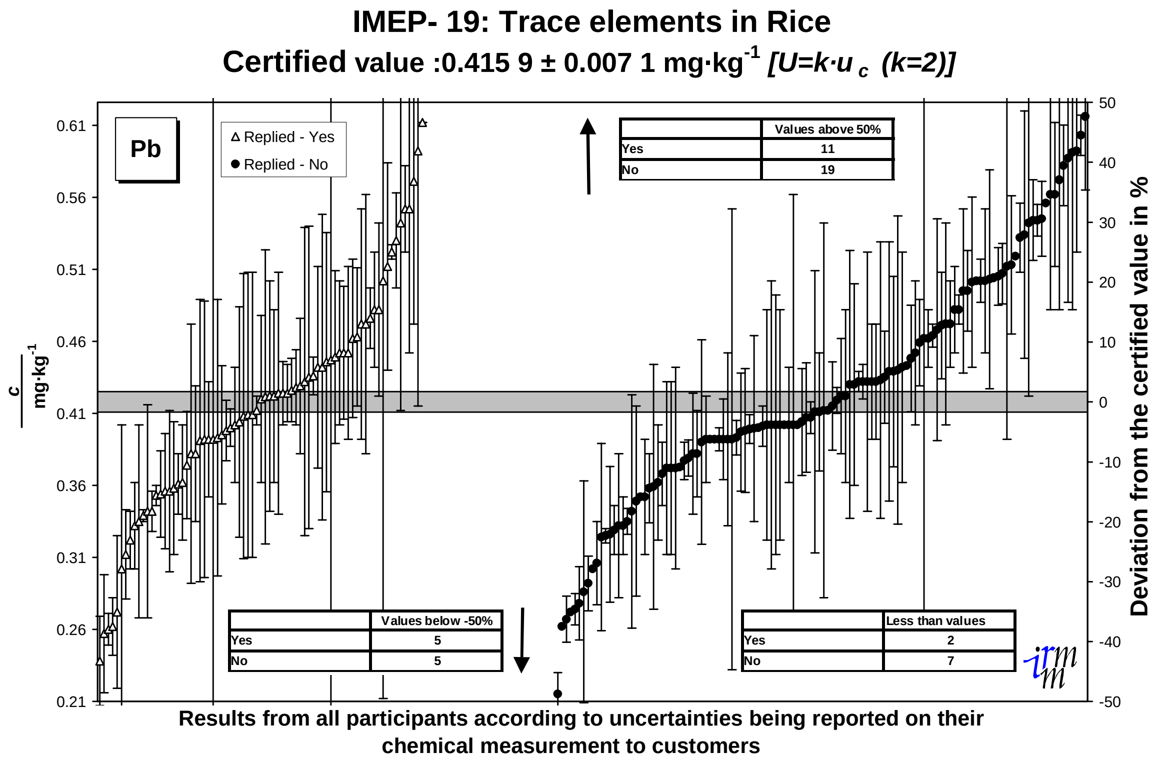
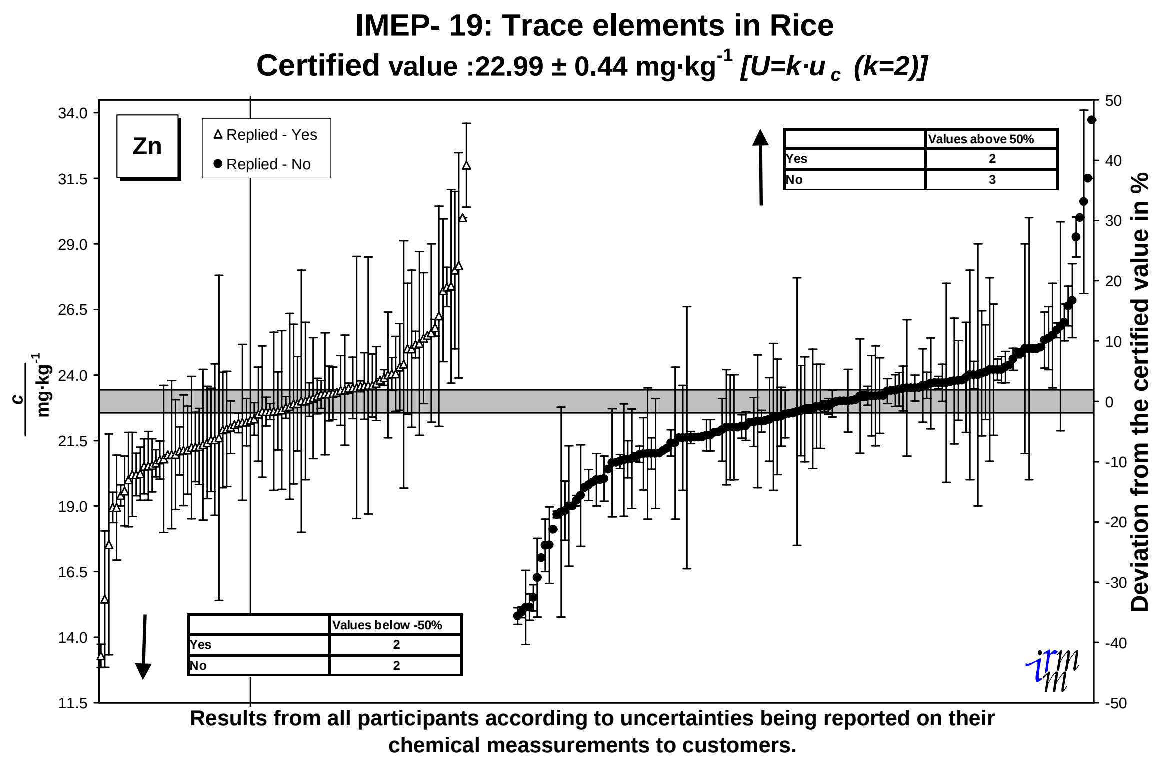


Figure 72



IMEP-19

participants' results - Measurement performance graphs by element

Figure 73

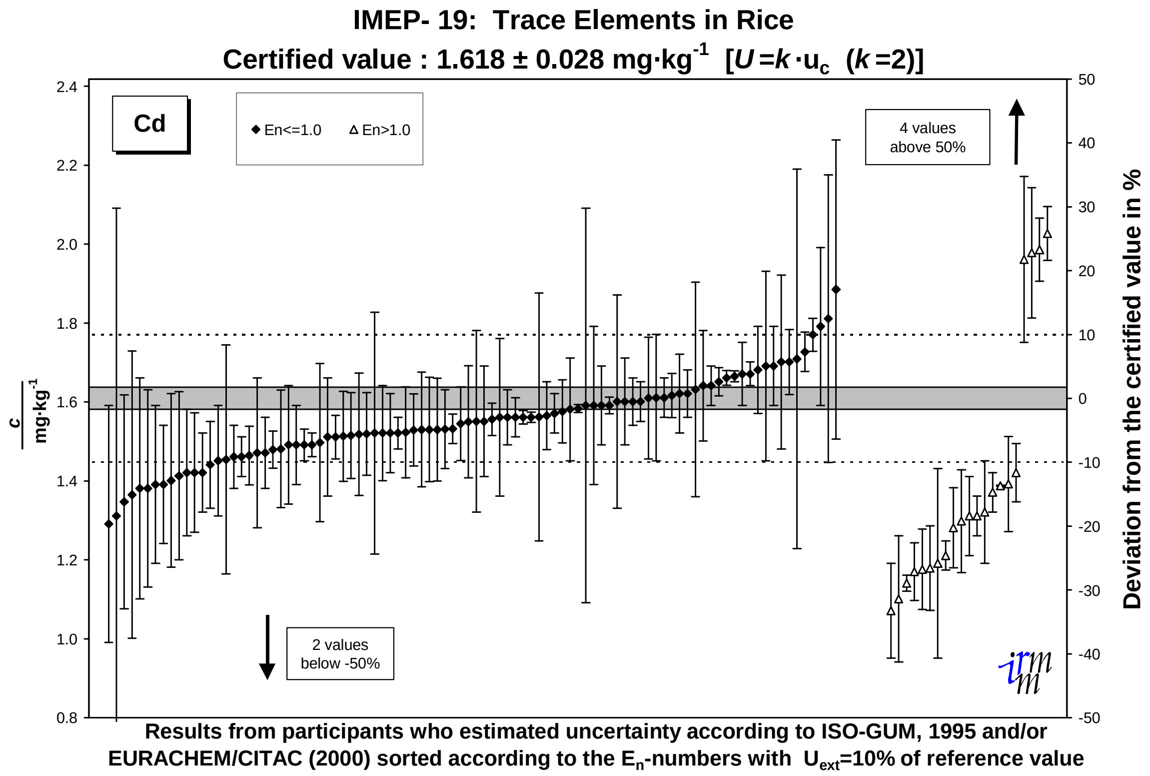


Figure 74

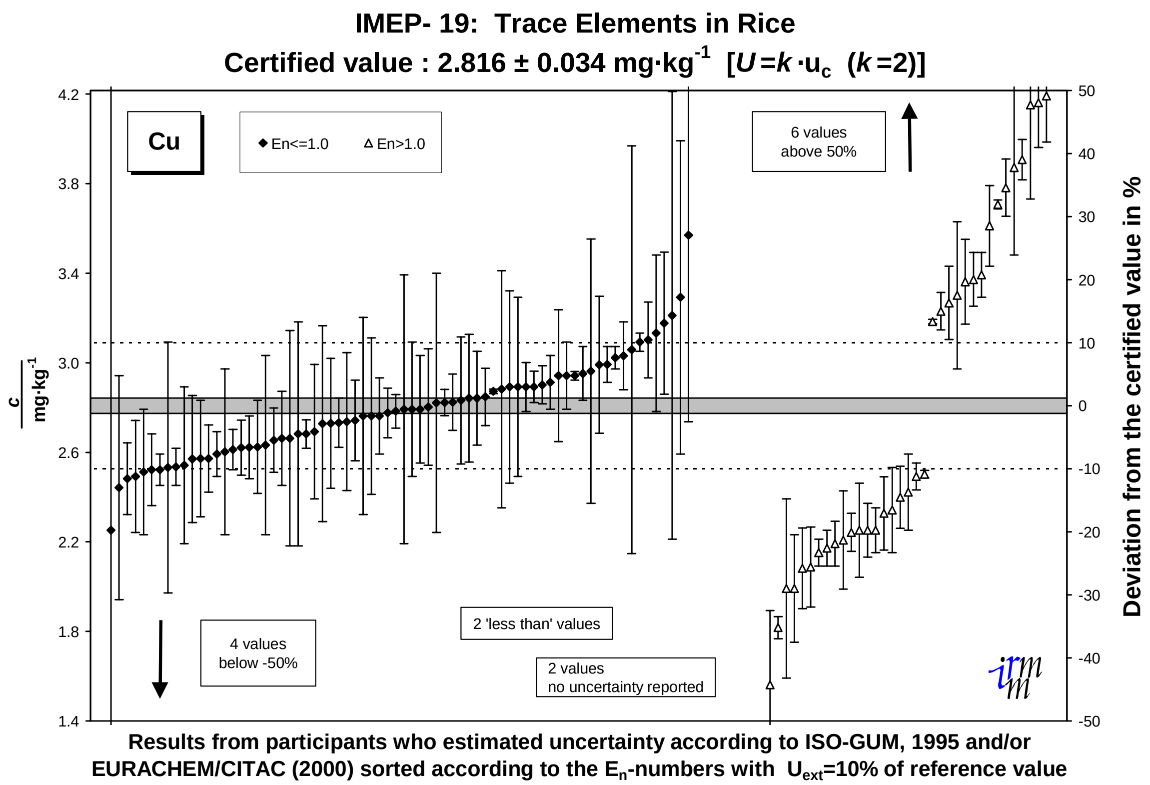


Figure 75

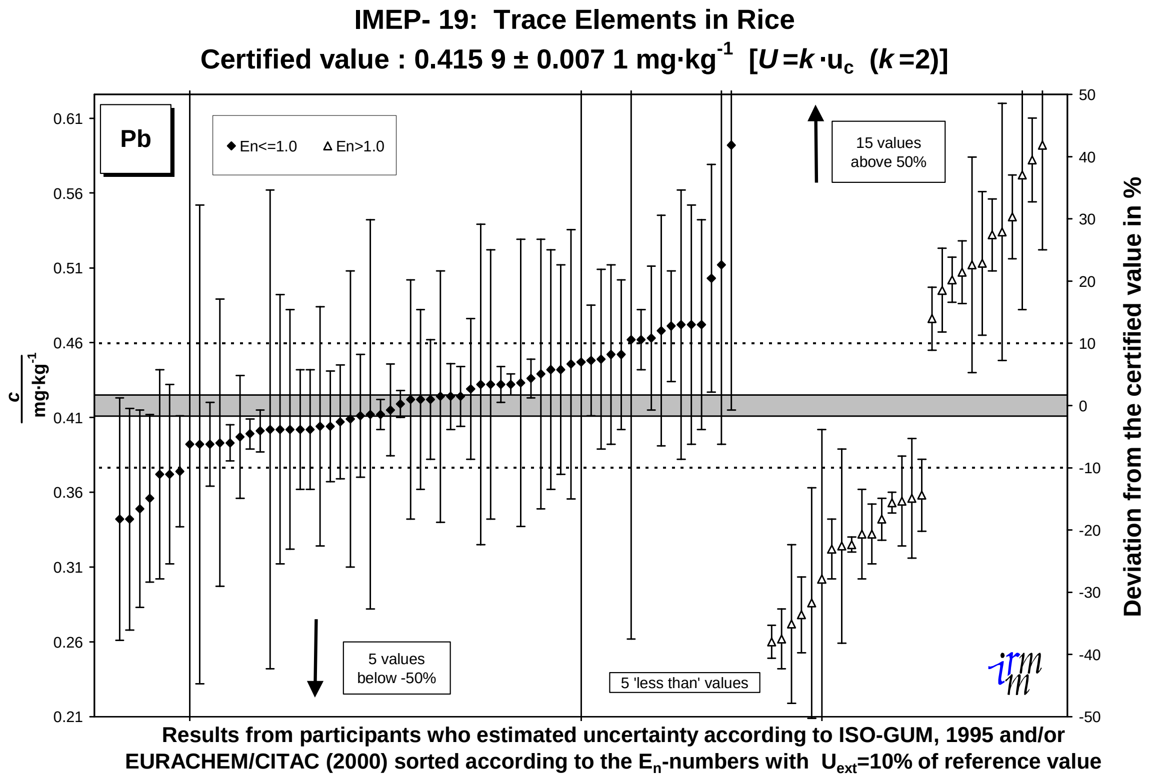
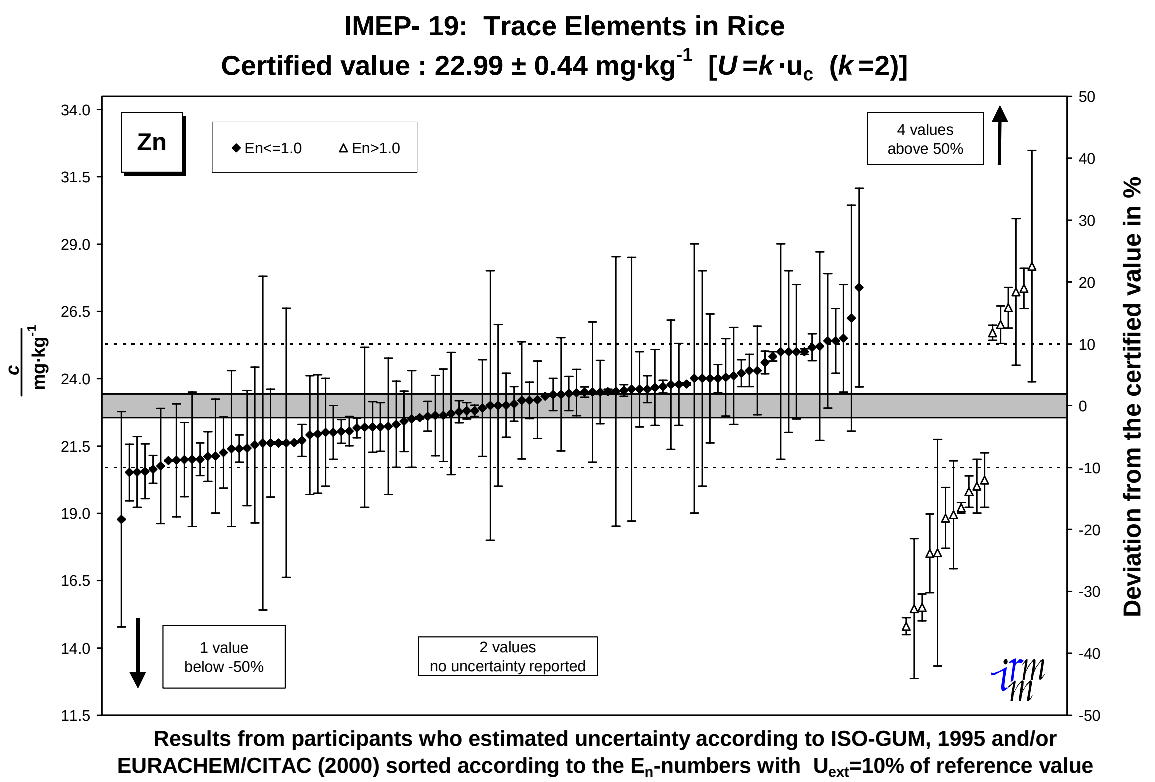
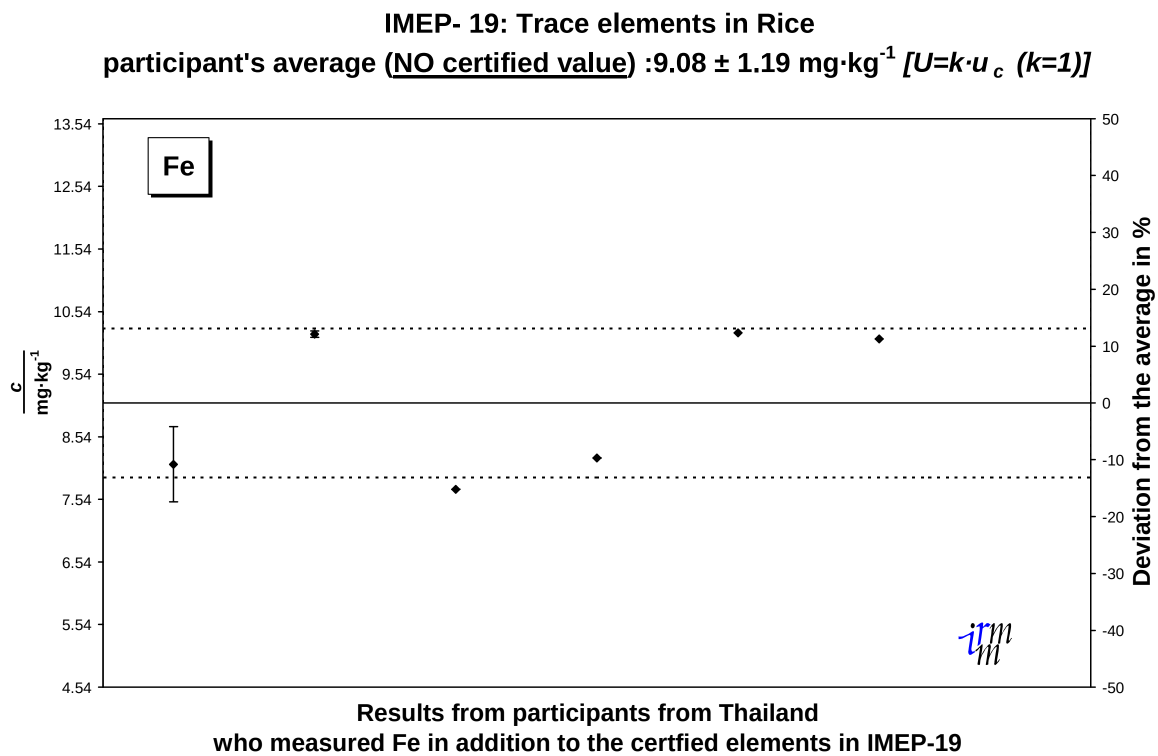


Figure 76



participants' results - IRON graph from Thailand

Figure 77



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DIRECTORATE GENERAL JRC
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IRMM
Institute for Reference Materials and Measurements

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IMEP-19: Trace Elements in Rice

Annex 2-Survey on the water content determination and correction for dry-mass

The determination of the water content in food matrices is a challenging task and to some extent "operationally defined" [1, 2]. Therefore IRMM has been approached several times in the past by laboratories from the food sector to organise an IMEP® interlaboratory comparison on the water content determination in a food matrix. Although the rice CTS used in IMEP-19 had not been certified for its water content, IRMM took the opportunity to provide a survey on the water content determination and correction for dry-mass using the information received from the IMEP-19 participants.

The results of the water content determination and correction for dry-mass are summarised in this Annex. In view of the importance of the water content determination in food analysis, IRMM asked the laboratories to provide information on the analytical method used. From the IMEP-19 questionnaire, participants were asked the average sample mass used for the water content determination, the determined water content in percentage, the applied dry-mass correction factor used in the measurement equation to calculate the total amount content of the trace elements present in the CTS, and whether the rice CTS was equilibrated with ambient humidity conditions prior to the water content determination.

Figure 1 shows by percentage the methods used for the water content determination:

82% of IMEP-19 participants applied the drying-oven method for the determination of the water content in the rice CTS. 24 participants out of those 82% used vacuum drying. 3 participants corrected for dry-mass using the Mettler LP16 infrared dryer and/or Moisture Analyser Mettler Toledo. Only 1 participant reported a value for the water content but did not report the method that was used for the water content determination. 17% of IMEP-19 participants did not determine the water content in the CTS at all. None of the IMEP-19 participants used Karl-Fischer titration to determine the water content in the CTS.

Figure 1 - Methods used for the water content determination

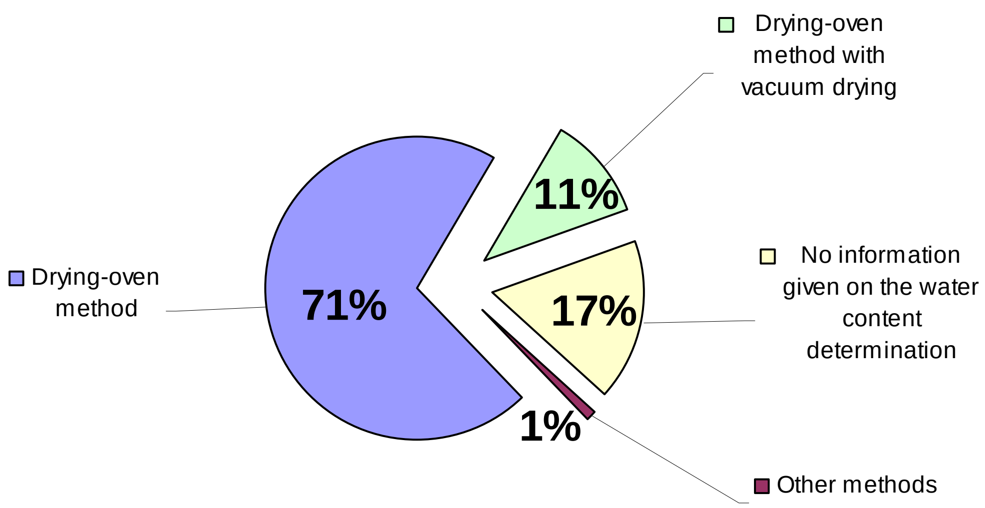
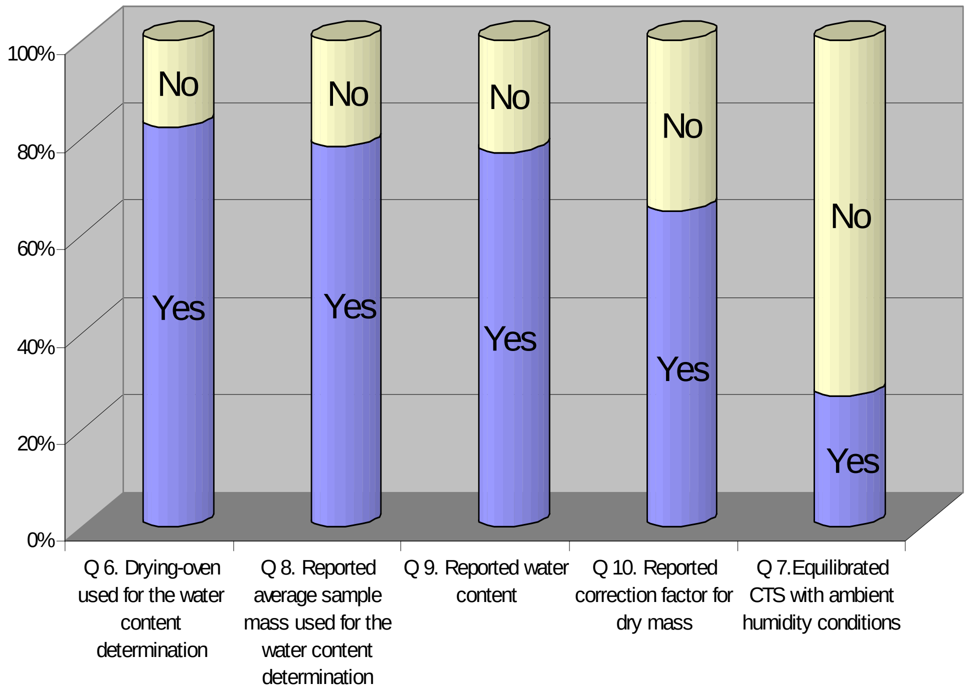


Figure 2 shows the IMEP-19 participants' response to the questions relating to the water content determination and correction for "dry-mass".

77% of IMEP-19 participants reported a value for the water content determination. 65% of IMEP-19 participants reported a value for the dry-mass correction. 78% of IMEP-19 participants reported the average sample mass in grams used for the water content determination and 27% of IMEP-19 participants equilibrated the rice CTS with ambient humidity conditions prior to water content determination

Figure 2 - IMEP-19 participants' responses to questions 6, 7, 8, 9 and 10 of the questionnaire, shown by %



To establish the certified reference values in IMEP-19 a thorough study was carried out at IRMM on the determination of the water content and the hygroscopic behaviour of the rice CTS. The correction factor for dry-mass was deduced from the water content measurements of the non-equilibrated rice CTS as well as from the water content measurements of the equilibrated rice CTS, taking also into account hygroscopicity. There were no significant differences observed from the measured amount contents of the elements to be certified in the rice CTS according to the two approaches.

Furthermore, the water content of the non-equilibrated rice CTS was measured with three independent methods, Karl-Fischer titration, drying-oven method and thermogravimetry. There were no significant differences to the results of the water content in the non-equilibrated rice CTS, for all three methods applied. Detailed information concerning the determination of the water content and the dry-mass correction in the rice CTS can be found in [3].

In Figure 3 to Figure 6 are the results for *Cd, Cu, Pb* and *Zn* which are sorted according to the measurements performed on the equilibrated or non-equilibrated rice CTS prior to the measurements.

Figure 3 - Results for Cd measurements from all participants according to sample equilibration prior to measurements

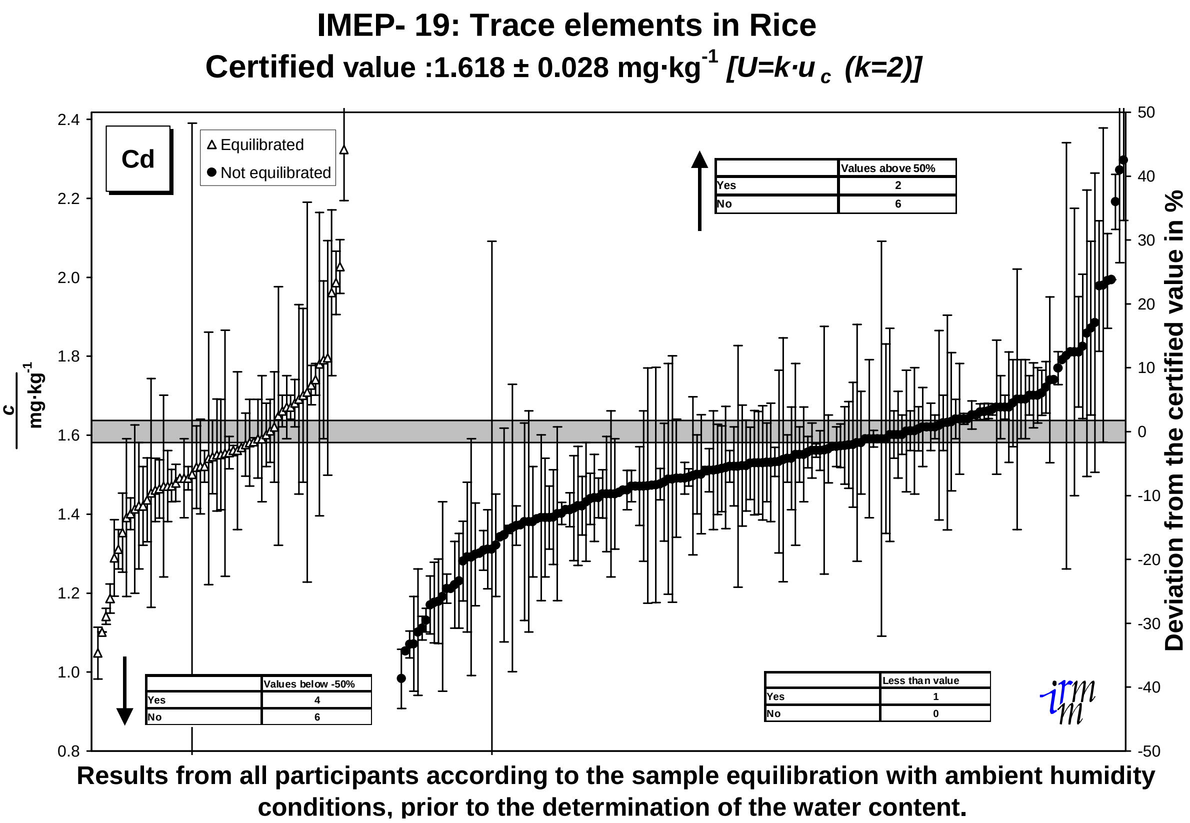
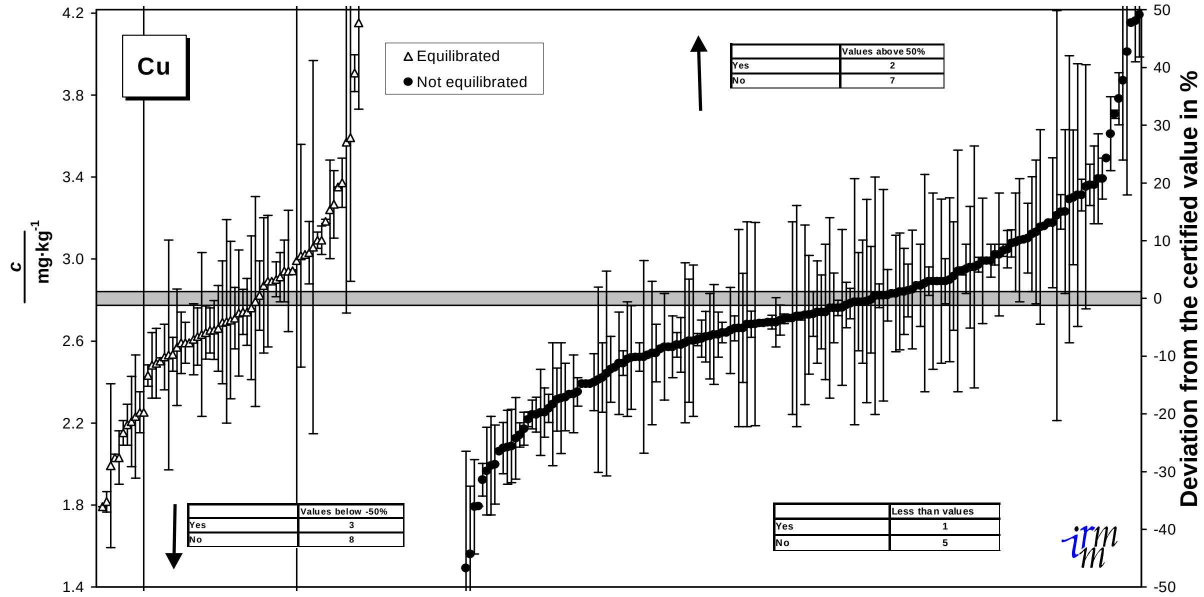


Figure 4 - Results for Cu measurements from all participants according to sample equilibration prior to measurements

IMEP- 19: Trace elements in Rice

Certified value :2.816 ± 0.034 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*

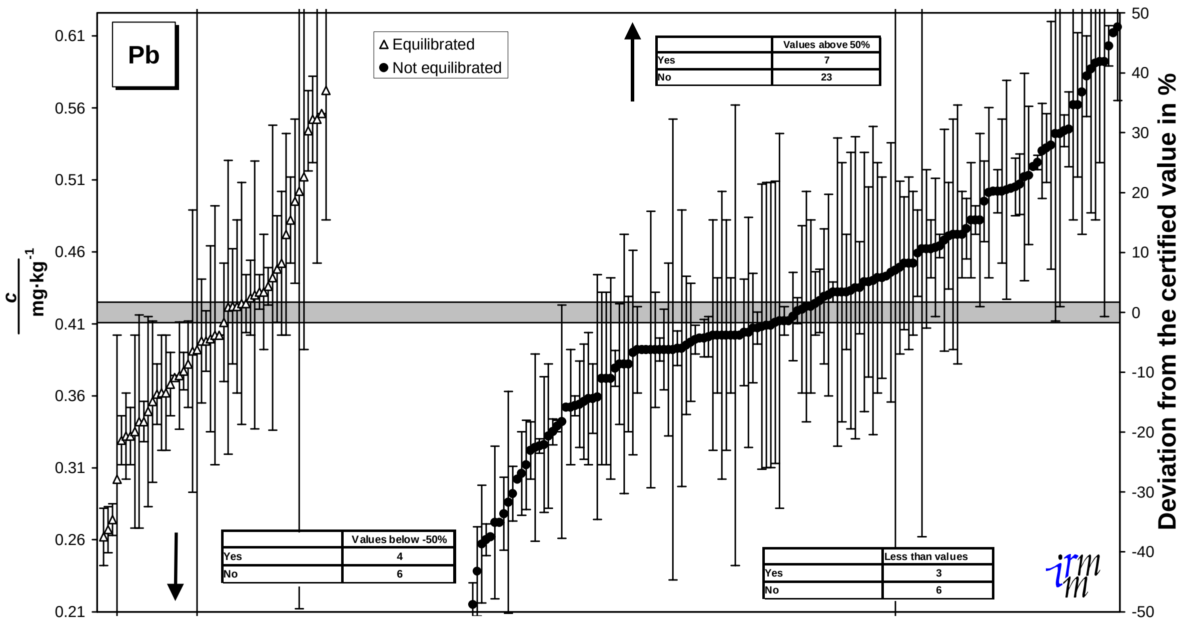


Results from all participants according to the sample equilibration with ambient humidity conditions, prior to the determination of the water content.

Figure 5 - Results for Pb measurements from all participants according to sample equilibration prior to measurements

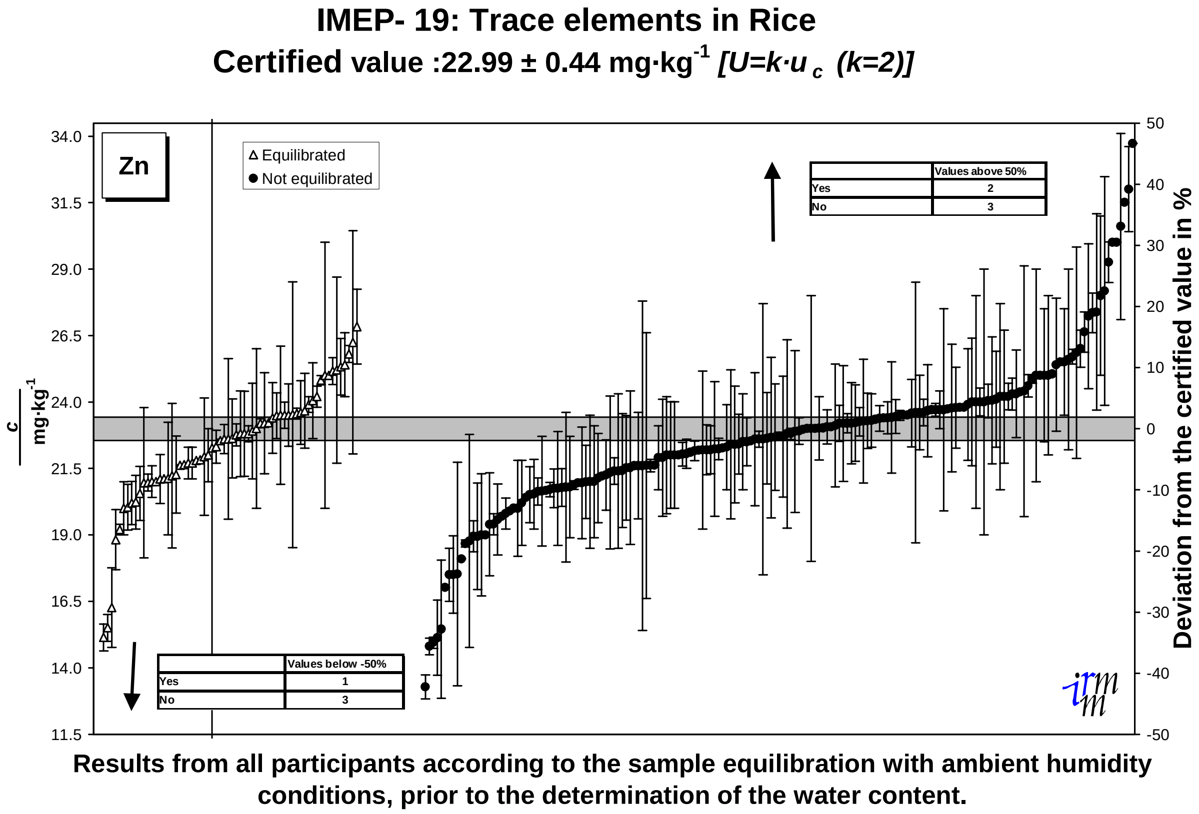
IMEP- 19: Trace elements in Rice

Certified value :0.415 9 ± 0.007 1 mg·kg^{-1} *[U=k·u$_c$ (k=2)]*



Results from all participants according to the sample equilibration with ambient humidity conditions, prior to the determination of the water content.

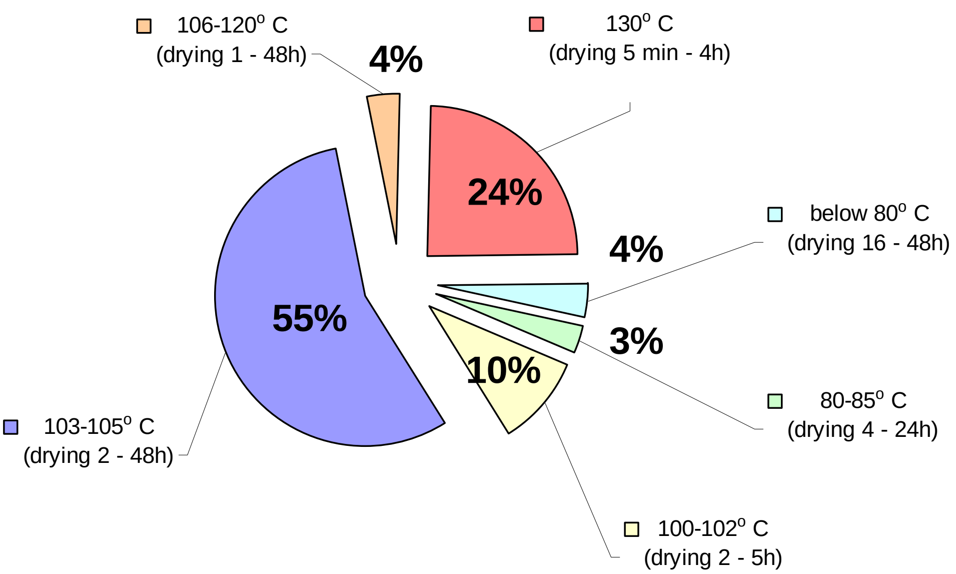
Figure 6 - Results for Zn measurements from all participants according to sample equilibration prior to measurements



43% of IMEP-19 participants used the drying-oven method for the water content determination on the non-equilibrated rice CTS applying different drying times and temperatures. Furthermore, they reported not only a value for the water content but also a correction factor for dry-mass. Half of these participants applied the drying-oven method at 105^{o} C, mostly using a drying time between 1 to 6 hours. 13 participants applied the drying-oven method at 105^{o} C, using a drying time between 12 hours and 1 day.

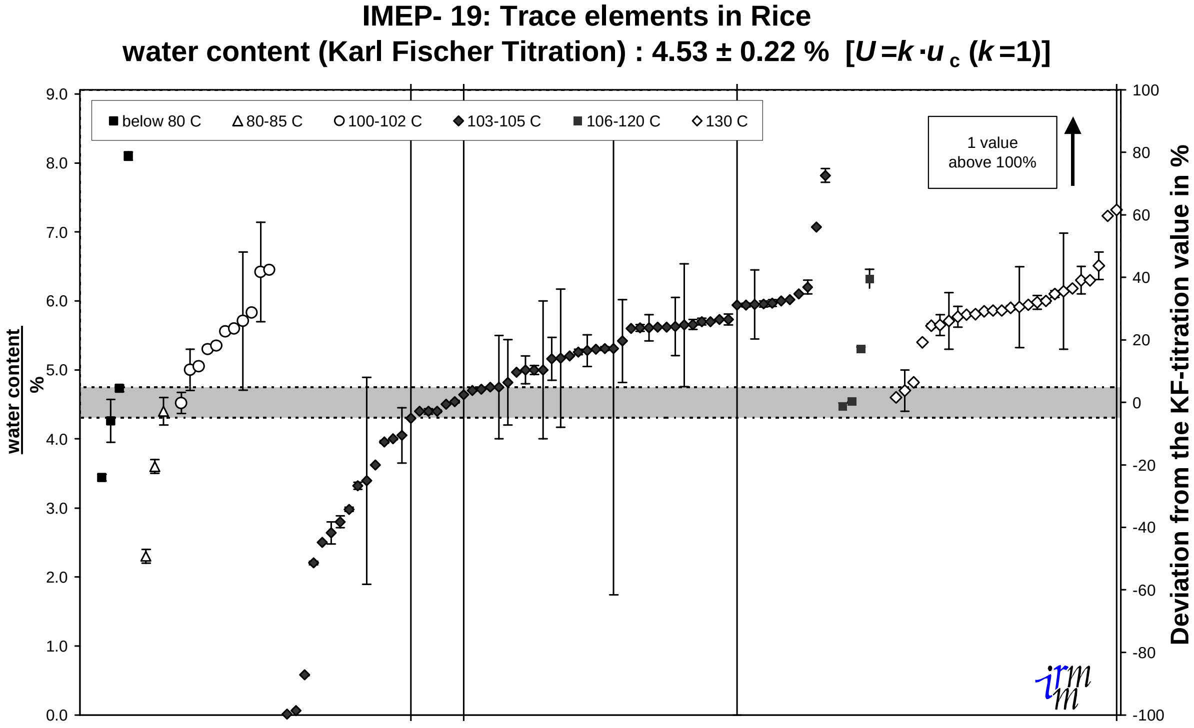
In Figure 7 the drying temperatures for oven-drying on the non-equilibrated CTS are shown. Drying times are given in parenthesis.

Figure 7 - Shows by percentage the drying temperatures used for the water content determination on the non-equilibrated CTS.



In Figure 8 the reported values of 43% of IMEP-19 participants are graphically displayed according to the applied drying temperatures. From Figure 8 a trend of increasing results for the water content with increasing drying-oven temperature can be observed. The water content was determined by IRMM using Karl-Fischer titration on 10 non-equilibrated rice CTS. This value within its uncertainty is given by the grey range between the broken lines in Figure 8. IRMM has to emphasise again that this is NOT a certified value for the water content in the rice CTS. This additional information is merely for the IMEP-19 participants.

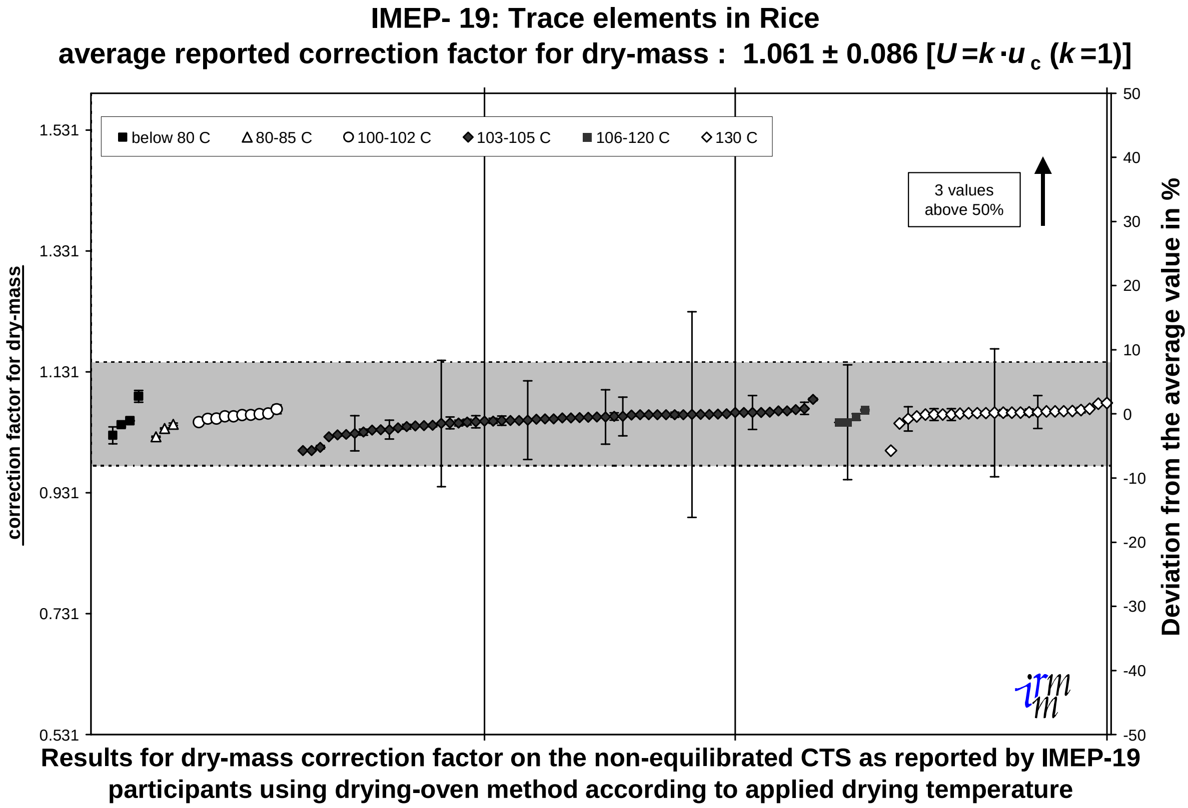
Figure 8 - Results for the water content measurements from 43% of IMEP-19 participants who applied drying-oven method and reported a value for the water content as well as for the correction for dry-mass



Results for water content on the non-equilibrated CTS as reported by IMEP-19 participants using drying-oven method according to applied drying temperature

In Figure 9 the reported values for the dry-mass correction factor are from the same participants as in Figure 8, according to the applied drying temperature. The average reported value (excluding the three values above 50% deviation) is given in the grey range between the broken lines. The reported dry-mass correction factors had been reported by the participants either as values below or above 1. To enable the calculation of an average value the reported dry-mass correction factors were all transferred to correction factors larger than 1. The displayed correction factors in Figure 9 correspond to the ratio of the sample mass before drying, to the sample mass after drying, also including a possible correction for moisture uptake while sample handling. The participants applied this "dry-mass correction" to the mass of the sample used for the measurements to correct for the mass loss due to drying in combination with a correction for possible hygroscopicity.

Figure 9 - Reported correction factor for dry-mass from 43% of IMEP-19 participants who applied drying-oven method and reported a value for the water content as well as for the correction for dry-mass



In IMEP-19 the importance of the water content determination was clearly identified, from the information given by the IMEP-19 participants. It was confirmed that the drying-oven method is the most common method used for water content determination in this kind of matrix. This is obviously due to the fact that for a lot of food matrices the drying-oven method is obligatory stated as reference method in the CODEX Alimentarius [4]. The positive response to this survey encourages IRMM to investigate the feasibility of an IMEP® comparison on water content determination in food matrices in the future.

1. S. Rückhold et al. *Fresenius J. Anal. Chem* (2000) 368: 522-527
2. S. Rückhold et al . *Fresenius J. Anal. Chem* (2000) 370: 189-193
3. E. Vassileva, C. R. Quétel, P. D. P. Taylor "Influence of the Correction for Moisture Content on the Quality of the Certification of Cd, Cu and Pb Mass Fractions in Rice" - - *in preparation*
4. CODEX Alimentarius, Standard and Standard Methods, volume 13, codexstan-234.1999

IMEP-19: Trace Elements in Rice
Annex 3-Additional Information contents table

Table 1. Laboratories who participate regularly in Proficiency Testing schemes (PTs*)*

Country	**Proficiency Testing schemes**	**No. of Labs**
ARGENTINA	IMEP-9, AQUACON EC ITALY. (Project 2c.4.1-Italian Research)	2
AUSTRALIA	FAPAS - Central Science Laboratory UK , NOAA.	2
AUSTRIA	IFA Tulln, ALFA PTs for agricultural, W&V, Insp. Health Protection NL, FAPAS-CSL UK, Several IMEPs, LVU Germany, CHEK, IAEA.	6
BELGIUM	OVAM, AQUQCHECK, FAPAS.	2
BRAZIL	CCQM-LGC, PT-IAEA, IAEA, AQUACHEC PTs.	3
BULGARIA	IMEP, FAPAS UK.	2
CROATIA	IFA Tulln Austria.	1
CYPRUS	IMEP, FAPAS UK.	2
CZECH REPUBLIC	FAPAS det. trace elements in foods, APLAC-trace, Nationale PT, CAFI, Ekocentrum Ostrava CZ, SZU Praha, Vitana Byšice, CZ.	8
ESTONIA	Food Analysis Performance Assessment Scheme FAPAS.	1
FINLAND	FAPAS, CHEK, Livsmedelverket Sweden, VTT Biotekn, Swedish National Food Administration.	3
FRANCE	BCR, BIPEA, IRMM, IAEA, CSL-FAPAS, CRL-ISS-Roma. (Community Ref Laboratory)	3
GERMANY	FAPAS, Central Science Lab, Nestec, CHEK, BGVV, LVU, EDQM, IMEP, DIN, r-concept, GSSR, Wageningen.	7
HUNGARY	Trace Elements FAPAS, MUVA (milk products), National proficiency ringtest in animal feeds, Other determinations of trace elements.	5
INDIA	Egg Powder proficiency testing organised by NABL.	1

Table 1. Laboratories who participate regularly in Proficiency Testing schemes (PTs)

Country	**Proficiency Testing schemes**	**No. of Labs**
IRELAND	FAPAS, Quasimeme.	2
ITALY	Metals in feed. (Università Cattolica del S.Cuore)	1
JAPAN	FAPAS, Central Science Lab UK, NATA, IMEP, ISO17025 & GUIDE 43, Japan-Environmental Measurement & Chemical.	5
LATVIA	Swedish National Food Administration, LIVSMEDELS VERKET Sweden.	2
LITHUANIA	National Food Administration Uppsala Sweden, LIVSMEDELS VERKET, MUVA KEMPTEN.	2
MALAYSIA	FODAS by Department of Chemistry Malaysia.	1
MEXICO	Intercomparation test of heavy metals in liver, Comision Nacional del Agua, Proficiency testing nutritional components in food, IMEP Water & Lead in wine, CENAM, Proficiency Test of Swedish Food Administration, GE.	5
NORWAY	FAPAS, Quasimeme, Livsmedelsverket Sweden, NRL-ISS, AACC (D- and VMP-series), NJF, Norsk Matanalyse.	3
P.R. CHINA	IMEP, NATA, CIQ.	1
POLAND	BADANIA MIEDZYLABORATORYJNE PZH, FAPAS, IPE WEPAL WAGENINGEN, ISS-CRL, METALS Pb Cd Cu Zn Hg NAT INSTITUTE OF HYGIENE, Pb Cd Cu Zn Hg PZH WARSZAWA, Cd Cu Pb Zn IN FOOD ORGANISER - NATIONAL INS, WATER PB CD ZN OZNACZANIE ZAWARTOSCI METALI W SRODKACH SPOZYWCZYC, ZAKLAD BADANIA ZYWNOSCI I PRZEDMIOTOW UZYTKU PANST, OZNACZANIE ZAWARTOSCI METALI SZKODLIWYCH DLA ZDROW, BADANIA BIEGLOSCI W ZAKRESIE OZNACZANIA ZAWARTOSCI, Politechnika Krakowska, IMEP, PROFICIENCY TESTING BY INTERLABORATORY COMPARISONS, Joint aquacon phare interlaboratory exercise joint, OZNACZANIE ZAWARTOSCI METALI W SRODKACH SPOZYWCZYC, Determination of heavy metal content in biologica.	32
RUMANIA	IMEP-12, IMEP-16.	1
RUSSIA	IMEP-6, 9, 12, 14, GeoPT UK, The Open University, International Atomic.	2

Table 1. Laboratories who participate regularly in Proficiency Testing schemes (PTs*)*

Country	**Proficiency Testing schemes**	**No. of Labs**
SINGAPORE	Nestec NRC, CSL-FAPAS. (Metallic Con)	3
SLOVAKIA	NRC OMT SZU LIPTOVSKY MIKULAS, PTs, VUVH SK, CZPI Brno CZ, FAPAS-CSL, NATIONAL REFERENCE CENTER FOR REFERENCE MEASUREMENTS, Trace Elements in Wholemeal Flour and Mixed Vegetables, National reference centre Public Health Institute, NESTLE Cd Hg in Cereal product, WEPAL NETHERLANDS, IAEA, Food Research Institute Bratislava, GEMS Food.	23
SLOVENIA	IMEP, FAPAS-CSL.	2
SOUTH AFRICA	IAEA multi-element studies in any matrix.	1
SPAIN	FAPAS, INTER2000, BIPEA, INTERNATIONAL ANALYTIQUE GROUP (IAG), National Food Administration Istit., GEMS MÂº Agricultura Spain.	6
SWEDEN	Trace elements in foods National food administration.	1
SWITZERLAND	FAPAS, IPE.	2
THAILAND	FAPAS, NATA Australia, IAEA Austria, Aseanfoods Thailand, PHLS, IMEP, Laboratory performance study Institute of Nutritio Pb Cd Hg in sunflower seed and fish.	7
THE NETHERLANDS	KDLL, FAPAS, QM,COKZ.	3
TURKEY	FAPAS-UME, Caeal (Canadian Environmental Accreditation Body), Determination of Trace Elements Cd Cr Cu Fe Zn.	7
UNITED KINGDOM	FAPAS, ISS Central Science Laboratory DEFRA UK.	4
USA	Participate with National Food Processors.	1
VIETNAM	IMEP, NATA, CTS.	1
YUGOSLAVIA	IMEP.	1

Table 2. Summary of the self-declared status, Accredited and/or Authorised for Cd and Pb

Country	Cd & Pb - Yes to Accredited/ Authorised	Cd & Pb - Yes only to Accredited	Cd & Pb - Yes only to Authorised	Cd & Pb - No to Accredited/ Authorised	No. of laboratories
ARGENTINA	0	0	2	1	3
AUSTRALIA	1	1	0	1	3
AUSTRIA	2	4	6	0	12
BELGIUM	0	0	2	2	4
BRAZIL	0	0	1	4	5
BULGARIA	1	0	1	1	3
CROATIA	1	1	2	0	4
CYPRUS	0	0	1	4	5
CZECH REPUBLIC	2	7	0	1	10
ESTONIA	0	1	0	1	2
FINLAND	0	3	0	0	3
FRANCE	1	1	1	3	6
GERMANY	4	3	0	2	9
GREECE	0	0	0	1	1
HUNGARY	4	4	1	5	14
INDIA	0	0	1	1	2
IRELAND	1	1	0	1	3
ITALY	0	1	0	1	2
JAPAN	0	0	2	6	8
LATVIA	1	0	1	1	3
LITHUANIA	0	1	0	3	4
MALAYSIA	1	0	0	1	2
MEXICO	2	1	2	1	6
NORWAY	2	0	1	0	3
P.R. CHINA	1	0	0	0	1
POLAND	1	6	4	25	36
REP. OF KOREA	0	0	1	2	3
RUMANIA	1	0	0	1	2
RUSSIA	0	1	0	1	2
SINGAPORE	3	1	0	0	4
SLOVAKIA	6	14	2	19	41
SLOVENIA	0	0	1	4	5
SOUTH AFRICA	0	0	1	1	2
SPAIN	1	2	2	1	6
SWEDEN	0	0	0	1	1
SWITZERLAND	2	1	2	0	5
THAILAND	0	0	2	7	9
THE NETHERLANDS	1	0	1	3	5
TURKEY	1	1	8	5	15
UNITED KINGDOM	2	1	0	2	5
USA	0	0	0	2	2
VIETNAM	1	0	0	1	2
YUGOSLAVIA	0	0	1	1	2
TOTALS	**43**	**56**	**49**	**117**	**265**

Figure 1 - Summary of the self-declared status, Accredited and/or Authorised for Cd & Pb

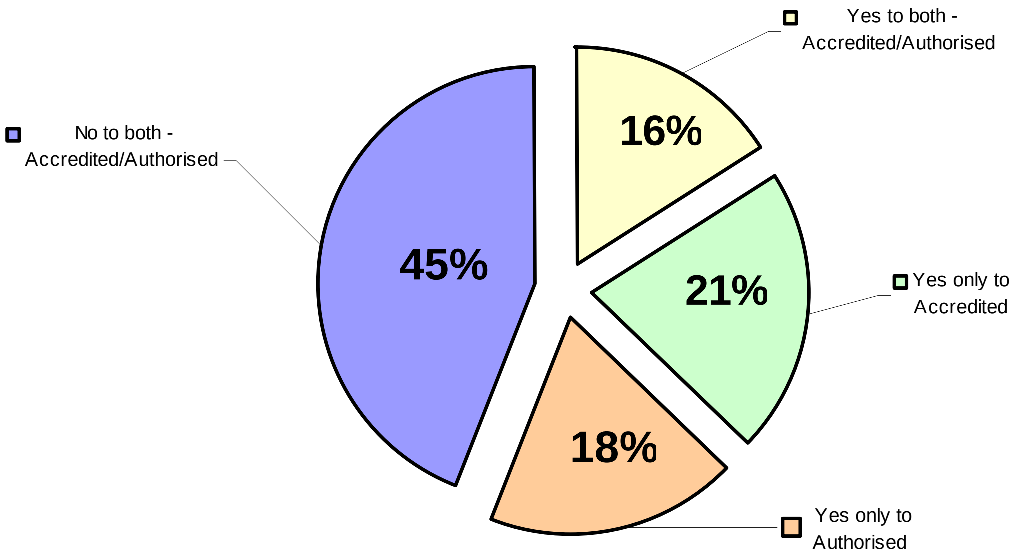


Table 3. Summary of the self-declared status, Accredited and/or Authorised for Cu

Country	Cu - Yes to Accredited/ Authorised	Cu - Yes only to Accredited	Cu - Yes only to Authorised	Cu - No to Accredited/ Authorised	No. of laboratories
ARGENTINA	0	0	2	1	3
AUSTRALIA	2	1	0	0	3
AUSTRIA	2	3	0	7	12
BELGIUM	0	0	2	2	4
BRAZIL	0	0	1	4	5
BULGARIA	1	0	1	1	3
CROATIA	1	0	3	0	4
CYPRUS	0	0	1	4	5
CZECH REPUBLIC	2	7	0	1	10
ESTONIA	0	1	0	1	2
FINLAND	0	0	2	1	3
FRANCE	1	0	1	4	6
GERMANY	3	3	1	2	9
GREECE	0	0	0	1	1
HUNGARY	4	4	1	5	14
INDIA	0	0	1	1	2
IRELAND	1	0	1	1	3
ITALY	0	1	0	1	2
JAPAN	0	0	1	7	8
LATVIA	1	1	1	0	3
LITHUANIA	0	1	0	3	4
MALAYSIA	1	0	0	1	2
MEXICO	3	0	2	1	6
NORWAY	2	0	1	0	3
P.R. CHINA	1	0	0	0	1
POLAND	1	8	4	23	36
REP. OF KOREA	0	0	1	2	3
RUMANIA	1	0	0	1	2
RUSSIA	0	1	0	1	2
SINGAPORE	3	1	0	0	4
SLOVAKIA	6	13	2	20	41
SLOVENIA	0	0	1	4	5
SOUTH AFRICA	0	0	1	1	2
SPAIN	1	1	2	2	6
SWEDEN	0	0	0	1	1
SWITZERLAND	2	1	2	0	5
THAILAND	0	0	2	7	9
THE NETHERLANDS	1	1	1	2	5
TURKEY	0	1	9	5	15
UNITED KINGDOM	2	1	0	2	5
USA	0	0	0	2	2
VIETNAM	0	0	1	1	2
YUGOSLAVIA	0	0	1	1	2
TOTALS	**42**	**50**	**49**	**124**	**265**

Table 4. Summary of the self-declared status, Accredited and/or Authorised for Zn

Country	Zn - Yes to Accredited/ Authorised	Zn - Yes only to Accredited	Zn - Yes only to Authorised	Zn - No to Accredited/ Authorised	No. of laboratories
ARGENTINA	0	0	2	1	3
AUSTRALIA	2	1	0	0	3
AUSTRIA	2	3	0	7	12
BELGIUM	0	0	2	2	4
BRAZIL	0	0	1	4	5
BULGARIA	1	0	1	1	3
CROATIA	1	0	3	0	4
CYPRUS	0	0	1	4	5
CZECH REPUBLIC	2	7	0	1	10
ESTONIA	0	1	0	1	2
FINLAND	0	0	3	0	3
FRANCE	1	0	1	4	6
GERMANY	3	3	1	2	9
GREECE	0	0	0	1	1
HUNGARY	4	3	1	6	14
INDIA	0	0	1	1	2
IRELAND	0	0	1	2	3
ITALY	0	1	0	1	2
JAPAN	0	0	1	7	8
LATVIA	1	1	1	0	3
LITHUANIA	0	1	0	3	4
MALAYSIA	1	0	0	1	2
MEXICO	2	1	2	1	6
NORWAY	2	0	1	0	3
P.R. CHINA	1	0	0	0	1
POLAND	1	8	4	23	36
REP. OF KOREA	0	0	1	2	3
RUMANIA	0	0	0	2	2
RUSSIA	0	1	0	1	2
SINGAPORE	2	1	0	1	4
SLOVAKIA	6	10	2	23	41
SLOVENIA	0	0	1	4	5
SOUTH AFRICA	0	0	1	1	2
SPAIN	1	1	2	2	6
SWEDEN	0	0	0	1	1
SWITZERLAND	2	1	2	0	5
THAILAND	0	0	2	7	9
THE NETHERLANDS	1	0	1	3	5
TURKEY	0	1	9	5	15
UNITED KINGDOM	2	1	0	2	5
USA	0	0	0	2	2
VIETNAM	0	0	1	1	2
YUGOSLAVIA	0	0	1	1	2
TOTALS	**38**	**46**	**50**	**131**	**265**

Figure 2 - Summary of the self-declared status, Accredited and/or Authorised for Cu

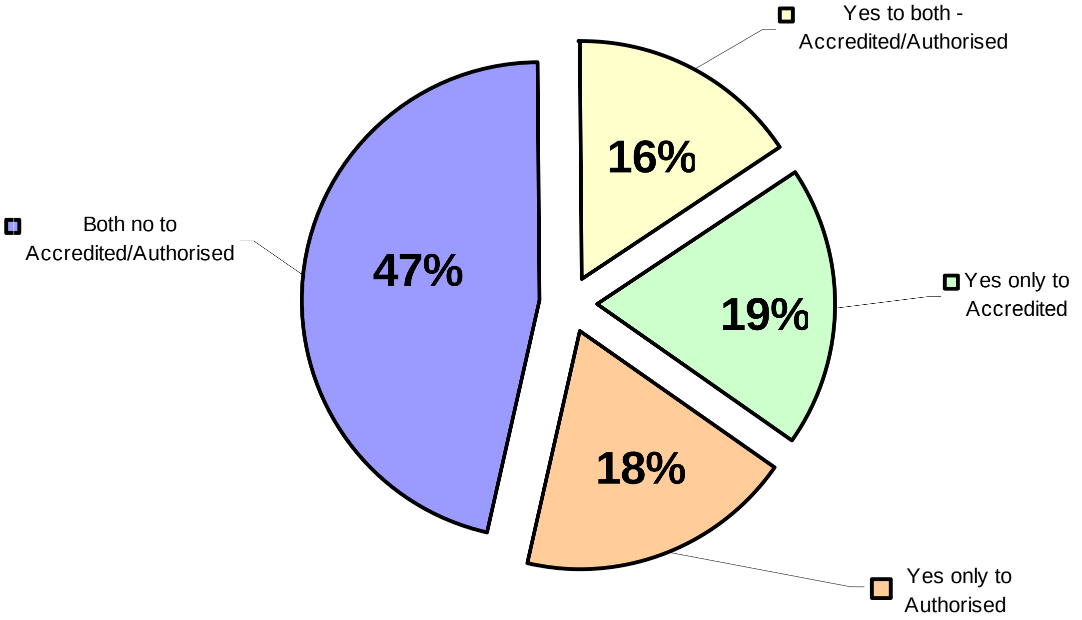


Figure 3 - Summary of the self-declared status, Accredited and/or Authorised for Zn

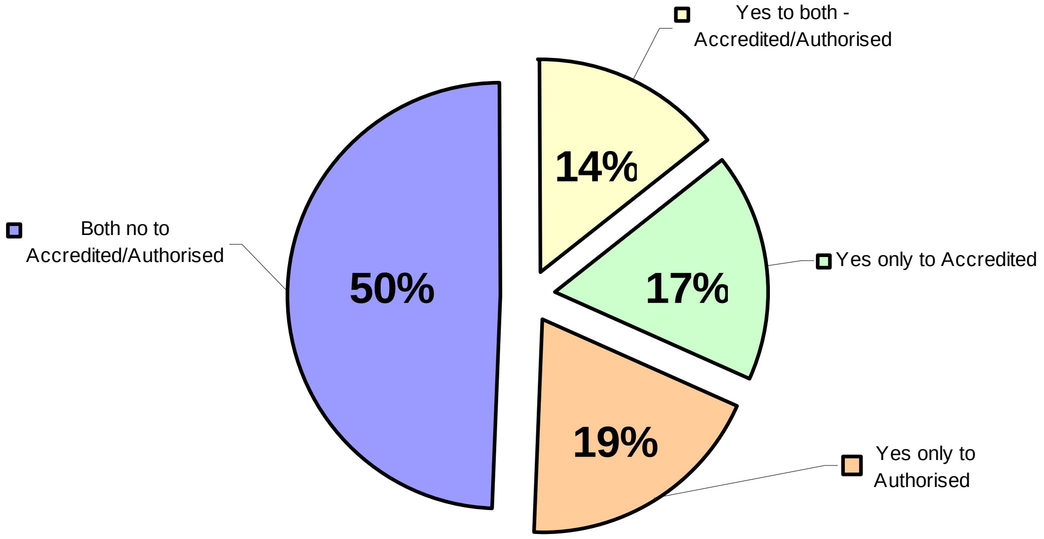


Table 5. Positive responses to Question 4, digestion step, for Cd and Pb

Country	Cd & Pb - Yes to separation & pre-concentration	Cd & Pb - Yes only to separation	Cd & Pb - Yes to only to pre-concentration	Cd & Pb - No to separation & pre-concentration	No. of laboratories
ARGENTINA	0	0	0	3	3
AUSTRALIA	0	0	0	3	3
AUSTRIA	0	0	0	12	12
BELGIUM	0	0	0	4	4
BRAZIL	0	0	0	5	5
BULGARIA	0	1	0	2	3
CROATIA	0	0	0	4	4
CYPRUS	0	0	0	5	5
CZECH REPUBLIC	0	0	0	10	10
ESTONIA	0	0	0	2	2
FINLAND	0	0	0	3	3
FRANCE	0	0	0	6	6
GERMANY	0	0	0	9	9
HUNGARY	0	0	0	14	14
INDIA	0	0	0	2	2
IRELAND	0	0	0	3	3
ITALY	0	0	0	2	2
JAPAN	1	0	1	5	7
LATVIA	0	0	0	3	3
LITHUANIA	0	0	0	4	4
MALAYSIA	0	0	0	2	2
MEXICO	2	1	0	3	6
NORWAY	0	0	0	3	3
POLAND	5	1	10	20	36
REP. OF KOREA	0	0	0	3	3
RUMANIA	0	0	0	2	2
RUSSIA	0	0	0	1	1
SINGAPORE	0	0	0	4	4
SLOVAKIA	0	1	1	39	41
SLOVENIA	0	0	1	4	5
SOUTH AFRICA	0	0	0	1	1
SPAIN	0	0	0	5	5
SWEDEN	0	0	0	1	1
SWITZERLAND	0	0	0	5	5
THAILAND	2	0	0	6	8
THE NETHERLANDS	0	0	0	5	5
TURKEY	1	0	1	13	15
UNITED KINGDOM	0	0	0	5	5
USA	0	0	0	2	2
VIETNAM	0	0	0	2	2
YUGOSLAVIA	0	0	1	1	2
TOTAL	**11**	**4**	**15**	**228**	**258**

Table 6. Negative responses to Question 4, digestion step, for Cd and Pb

Country	Cd & Pb - Yes to separation & pre-concentration	Cd & Pb - Yes only to separation	Cd & Pb - Yes to only to pre-concentration	Cd & Pb - No to separation & pre-concentration	No. of laboratories
GREECE	0	0	0	1	1
JAPAN	0	1	0	0	1
P.R. CHINA	0	0	0	1	1
RUSSIA	0	0	0	1	1
SOUTH AFRICA	0	0	0	1	1
SPAIN	0	0	0	1	1
THAILAND	0	0	0	1	1
TOTAL	**0**	**1**	**0**	**6**	**7**

Figure 4 - Positive responses to Question 4, digestion step, for Cd & Pb.

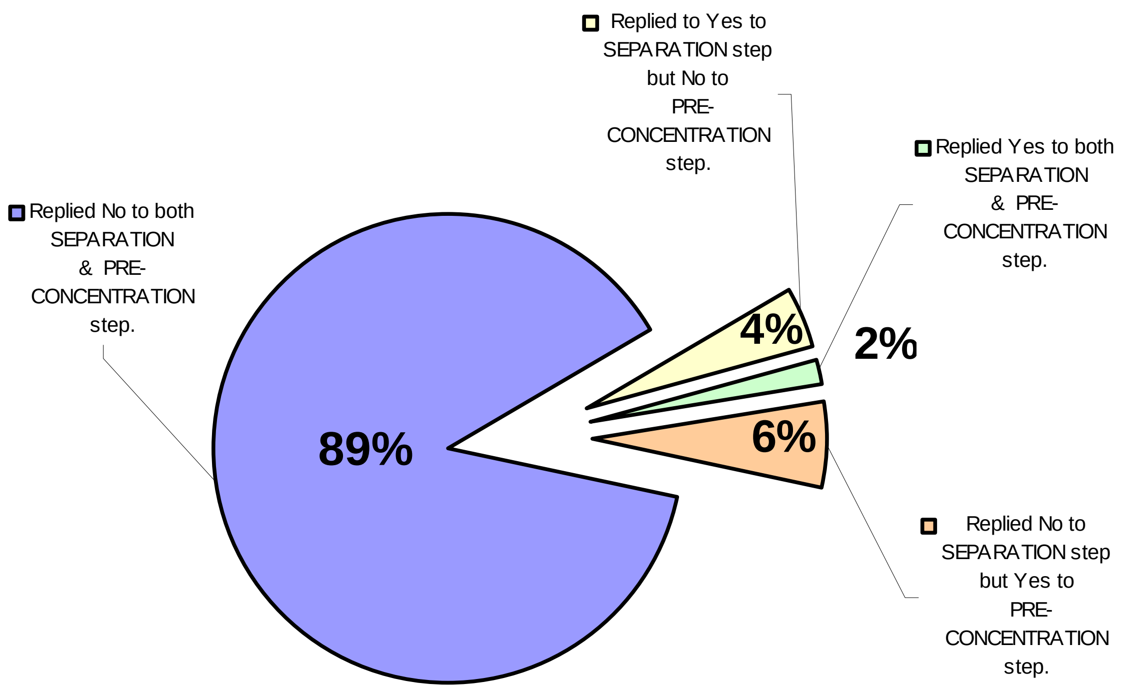


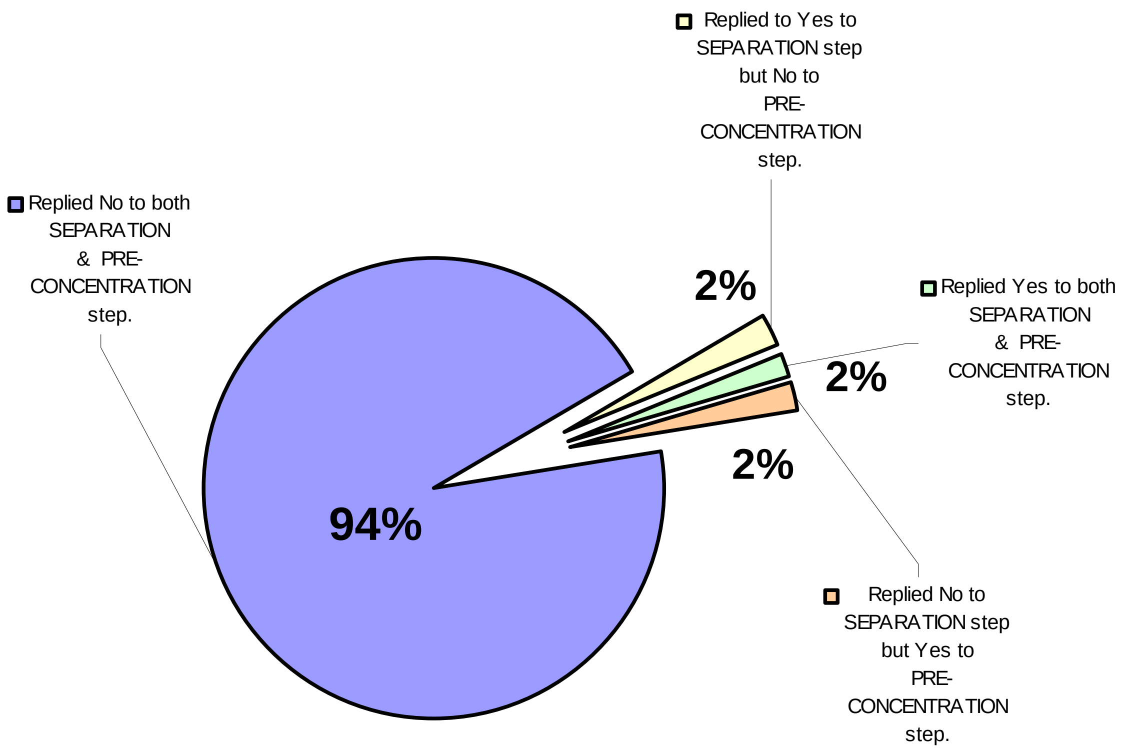
Table 7. Positive responses to Question 4, digestion step, for Cu and Zn

Country	Cu & Zn - Yes to separation & pre-concentration	Cu & Zn - Yes only to separation	Cu & Zn - Yes to only to pre-concentration	Cu & Zn - No to separation & pre-concentration	No. of laboratories
ARGENTINA	0	0	0	3	3
AUSTRALIA	0	0	0	3	3
AUSTRIA	0	0	0	12	12
BELGIUM	0	0	0	4	4
BRAZIL	0	0	0	5	5
BULGARIA	0	1	0	2	3
CROATIA	0	0	0	4	4
CYPRUS	0	0	1	4	5
CZECH REPUBLIC	0	0	0	10	10
ESTONIA	0	0	0	2	2
FINLAND	0	0	0	3	3
FRANCE	0	0	0	6	6
GERMANY	0	0	0	9	9
HUNGARY	0	0	0	14	14
INDIA	0	0	0	2	2
IRELAND	0	0	0	3	3
ITALY	0	0	0	2	2
JAPAN	1	0	1	5	7
LATVIA	0	0	0	3	3
LITHUANIA	0	0	0	4	4
MALAYSIA	0	0	0	2	2
MEXICO	2	1	0	3	6
NORWAY	0	0	0	3	3
POLAND	1	1	0	34	36
REP. OF KOREA	0	0	0	3	3
RUMANIA	0	0	0	2	2
RUSSIA	0	0	0	1	1
SINGAPORE	0	0	0	4	4
SLOVAKIA	0	1	1	39	41
SLOVENIA	0	0	0	5	5
SOUTH AFRICA	0	0	0	1	1
SPAIN	0	0	0	5	5
SWEDEN	0	0	0	1	1
SWITZERLAND	0	0	0	5	5
THAILAND	1	0	0	7	8
THE NETHERLANDS	0	0	0	5	5
TURKEY	1	0	1	13	15
UNITED KINGDOM	0	0	0	5	5
USA	0	0	0	2	2
VIETNAM	0	0	0	2	2
YUGOSLAVIA	0	0	1	1	2
TOTAL	**6**	**4**	**5**	**243**	**258**

Table 8. Negative responses to Question 4, digestion step, for Cu and Zn

Country	Cu & Zn - Yes to separation & pre-concentration	Cu & Zn - Yes only to separation	Cu & Zn - Yes to only to pre-concentration	Cu & Zn - No to separation & pre-concentration	No. of laboratories
GREECE	0	0	0	1	1
JAPAN	0	1	0	0	1
P.R. CHINA	0	0	0	1	1
RUSSIA	0	0	0	1	1
SOUTH AFRICA	0	0	0	1	1
SPAIN	0	0	0	1	1
THAILAND	0	0	0	1	1
TOTAL	**0**	**1**	**0**	**6**	**7**

Figure 5 - Positive responses to Question 4, digestion step, for Cu & Zn



EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC
JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP-19: Trace Elements in Rice

Annex 4-Documentation, Forms, Letters and Certificates contents table

Figure 1

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC

JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP®

Geel, 15 April 2002
IM/L/28/02

International Measurement Evaluation Programme

IMEP-19 Trace Elements in Rice

The International Measurement Evaluation Programme (IMEP®) was established and is operated by the Institute for Reference Materials and Measurements (IRMM) in order to picture objectively the degree of equivalence of chemical measurements by comparing them with external reference values (not derived from participant's results). Previous IMEP® interlaboratory comparisons have focused on different elements in various matrices such as water, sediment, serum, wine and others. Information about these activities can be found on the IMEP website http://www.imep.ws.

Participating laboratories receive a Certified Test Sample (CTS) (with undisclosed amount content values), which is to be measured using routine analytical procedures. The measurement results of participants will be evaluated against metrological reference values obtained using a primary method of measurement (Isotope Dilution Mass Specrometry). IMEP® is open to all laboratories and full confidentiality is guaranteed with respect to the link between measurement results and the participants' identity.

IRMM is now launching the IMEP-19 interlaboratory comparison that focuses on the analysis of total amount contents of Cd, Pb, Cu and Zn in rice. The CTS is available in glass bottles containing 15 g of rice. Each participant receives one bottle. A participation fee of 200 € per laboratory is requested. (In the frame of an EU supporting programme to EU candidate countries, participation for laboratories from these countries is free of charge. This applies for Bulgaria, Cyprus, Czech Republic, Estonia, Hungary, Latvia, Lithuania, Malta, Poland, Rumania, Slovakia and Slovenia.)

The samples will be available around June 2002. You can express your interest to participate until 31st May 2002 and the deadline for reporting results would be 30th September 2002. As a first feedback, participating laboratories will receive the reference values in November 2002. The full participants' report will be distributed in March 2003.

If you would be interested in joining this IMEP-19 interlaboratory comparison, please fill in the registration sheet and return it to your Regional Co-ordinator or IRMM. A list with the regional co-ordinators for this round will be available on the IMEP website http**://www.imep.ws.**

Yours sincerely

Dr.Y. Aregbe
IMEP-19 Co-ordinator
IM Unit - IRMM

Retieseweg, B-2440 Geel, Belgium
Tel.: +32-(0)14-571 673 •Fax: +32-(0)14-571 865 • imep@irmm.jrc.be • yetunde.aregbe@irmm.jrc.be •
www.imep.ws • http://www.irmm.jrc.be

Figure 2

IMEP-19 : TRACE ELEMENTS IN RICE

REGISTRATION SHEET

We would like to participate in the IMEP-19 interlaboratory comparison. The participation fee is 200 Euro (after receipt of the sample)

(In the frame of an EU supporting programme to EU candidate countries, participation for laboratories from these countries is free of charge. This applies for Bulgaria, Cyprus, Czech Republic, Estonia, Hungary, Latvia, Lithuania, Malta, Poland, Rumania, Slovakia and Slovenia.)

Please complete the form in capital letters

NAME	: ....
FIRM/INSTITUTE	:...
DEPARTMENT	: ....
ADDRESS	: ....
	....
	....
COUNTRY	: ....
TELE.	: ....
FAX.	: ....
E-MAIL	: ....

DATE :

SIGNATURE :

(*)Please return this sheet to the IMEP regional co-ordinator in your country (the list of IMEP regional co-ordinators can be found on http//www.imep.ws) or directly to
Dr. Yetunde Aregbe at IRMM, Retieseweg, B-2440 Geel, Belgium,
Fax: + 32 (0) 14 571 865

The IMEP® programme works in close co-operation with regional co-ordinators. Hence it is possible that you receive this registration sheet via different communication channels. You can register via IRMM or via your regional co-ordinator. All registrations are centralised at IRMM. In case more than one registration from your laboratory is received, the name of the person who registers will be important. Different names will count as multiple registrations.

Figure 3

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC

JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP®

Geel, 28th June 2002
IM/L/47/02

IMEP-19: Trace Elements in Rice

Dear «title» «surname»,

Please find enclosed the sample together with the following documentation: 1) Instruction letter on how to report your results and questionnaire information.
2) IMEP-19 sample confirmation form, which must be returned immediately to IRMM.

The IMEP-19 interlaboratory comparison involves the determination of total amount contents of Cd, Cu, Pb, and Zn in rice. Participants may select to analyse the elements relevant for their application. The Certified Test Sample (CTS) is in glass bottles containing 15 g of rice. The deadline for reporting the results and returning the completed questionnaire is 30th September 2002. A first feedback, concerning the IMEP-19 reference values, is foreseen for November 2002. The report for the participants containing the graphical display of all laboratory results as well as the reference values will be distributed by March 2003.

The results should be reported electronically via the Internet to IRMM. Instructions for reporting your results can be found at http://www.instruction19.imep.ws Therefore you have been allocated a laboratory identification number (Lab-ID) and a Key-code number, please use these numbers when reporting online.

The login page on the IMEP web site is located at: http://www.data19.imep.ws

Your LAB-ID No:- «Person_id»

Your KEY-CODE No:- «KeyCode»

When you have submitted your results and questionnaire information you will receive an e-mail as a confirmation of your reported results within the next days. On receipt of this e-mail please check your reported results carefully for any errors. In case you need to adjust any of your results, you will need to send an e-mail (imep@irmm.jrc.be) or fax (+32 14 571 865), with the amended details as soon as possible. If we don't receive a reply we assume that your reported results are correct and your results will then be transferred to our database. Once the results have been entered into our database there will be no further possibility for any changes.
If you have any questions or problems, please do not hesitate to contact us.
Yours sincerely,

Dr. Y. Aregbe
IMEP-19 Co-ordinator, IRMM – JRC – EC

Retieseweg, B-2440 Geel, Belgium
Tel.: +32-(0)14-571 673 •Fax: +32-(0)14-571 865 • imep@irmm.jrc.be • yetunde.aregbe@irmm.jrc.be •
www.imep.ws • http://www.irmm.jrc.be

Figure 4

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC

JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

«title» «firstname» «surname»
«companyinstitute»
«address»
«Town»
«zip» «country»

LAB-ID No. «person_id»

IMEP-19

Trace Elements in Rice
Confirmation of safe receipt - IMEP-19 Rice sample

Please return this form immediately to IRMM, this confirms that the sample package arrived.
(in case it is damaged, please contact us immediately).
Please complete or amend the address information in case needed.
(capital letters).
We have received the sample package in good order

Date of package arrival:……………………..

Signature:……………………………………

Please return the form to:

Dr. Y. Aregbe
IMEP-19 Co-ordinator
IRMM – JRC – EC
Retieseweg
B-2440 GEEL, Belgium

Tel : +32 (0) 14 571 673
Fax : +32 (0) 14 571 865
e-mail : imep@irmm.jrc.be

Retieseweg, B-2440 Geel, Belgium
Tel.: +32-(0)14-571 673 •Fax: +32-(0)14-571 865 • imep@irmm.jrc.be • yetunde.aregbe@irmm.jrc.be •
www.imep.ws • http://www.irmm.jrc.be

Figure 5

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC
JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP-19: Trace Elements in Rice

GUIDELINES to Participants on Reporting Results & completing the Questionnaire Online

The result reporting web page

Please use your allocated laboratory identification number (Lab-ID) and Key-code number when reporting online. The login page on the IMEP web site is located at: http://www.data19.imep.ws

IMEP is in a transition phase of changing to a new database system, but we still would like to provide the service of online reporting to our participants in IMEP-19. Therefore we have to ask you to follow strictly the guidelines for result reporting and filling in the questionnaire.

Retieseweg, B-2440 Geel, Belgium
Tel.: +32-(0)14-571 673 •Fax: +32-(0)14-571 865 • imep@irmm.jrc.be • yetunde.aregbe@irmm.jrc.be •
www.imep.ws • http://www.irmm.jrc.be

Figure 6

Completing the Results Report Form

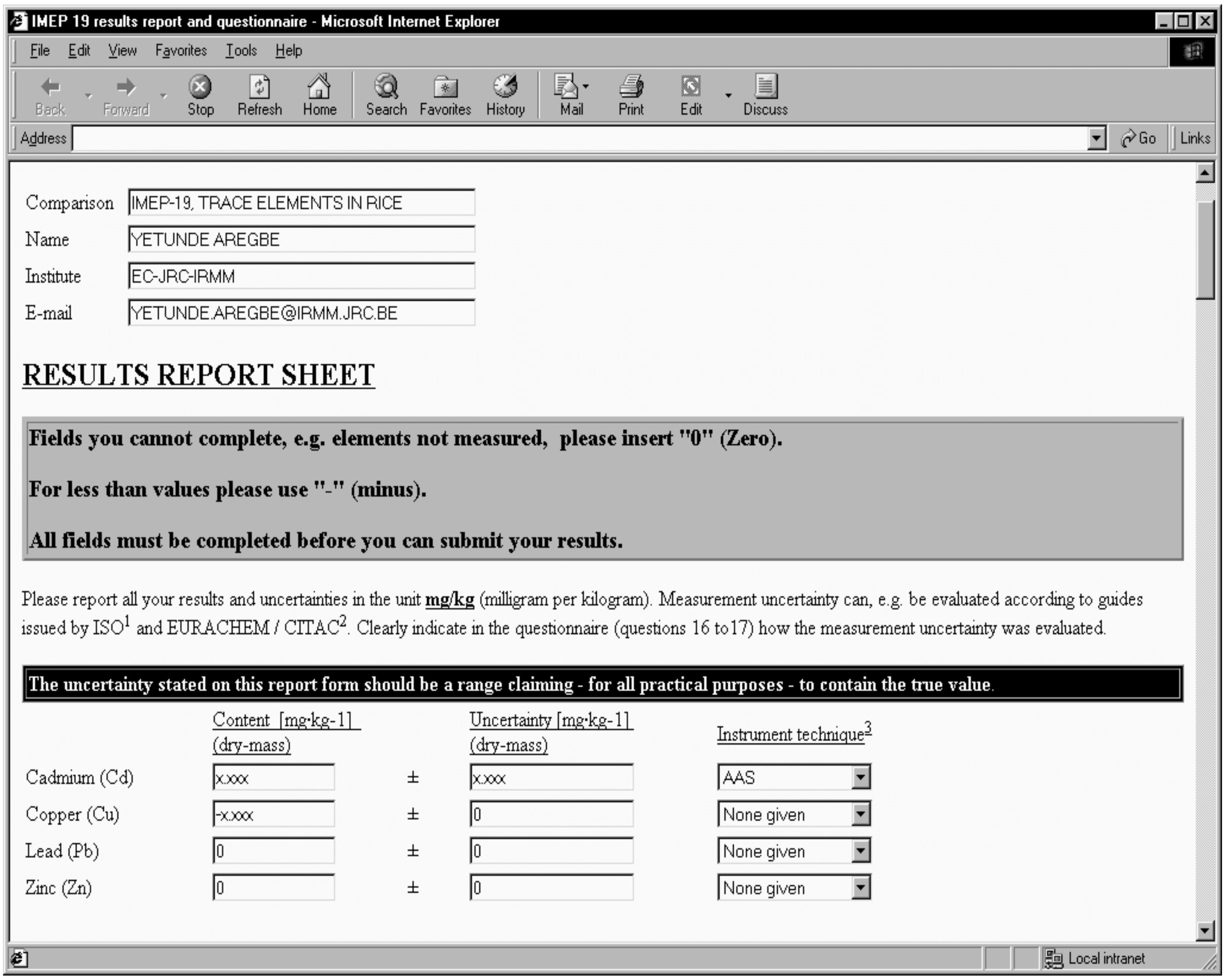
1. You have to report the element content and its uncertainty in **mg·kg^{-1}**

2. The fields in the Result Report Form are defined as **numerical decimal fields**. Data input using scientific format is not possible.

3. Due to the fact that the fields in the Result Report Form are defined as numerical fields you must enter “0” (zero) if you have not measured one element.

If you report an upper limit as a result you have to enter “–“ (minus sign) instead of the “<” less than sign.

(see example below, where “x.xxx” stands for a reported numerical result).



IMEP 19 results report and questionnaire - Microsoft Internet Explorer

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Comparison: IMEP-19, TRACE ELEMENTS IN RICE
Name: YETUNDE AREGBE
Institute: EC-JRC-IRMM
E-mail: YETUNDE.AREGBE@IRMM.JRC.BE

RESULTS REPORT SHEET

Fields you cannot complete, e.g. elements not measured, please insert "0" (Zero).

For less than values please use "-" (minus).

All fields must be completed before you can submit your results.

Please report all your results and uncertainties in the unit **mg/kg** (milligram per kilogram). Measurement uncertainty can, e.g. be evaluated according to guides issued by ISO[1] and EURACHEM / CITAC[2]. Clearly indicate in the questionnaire (questions 16 to17) how the measurement uncertainty was evaluated.

The uncertainty stated on this report form should be a range claiming - for all practical purposes - to contain the true value.

	Content [mg·kg-1] (dry-mass)		Uncertainty [mg·kg-1] (dry-mass)	Instrument technique[3]
Cadmium (Cd)	x.xxx	±	x.xxx	AAS
Copper (Cu)	-x.xxx	±	0	None given
Lead (Pb)	0	±	0	None given
Zinc (Zn)	0	±	0	None given

Local intranet

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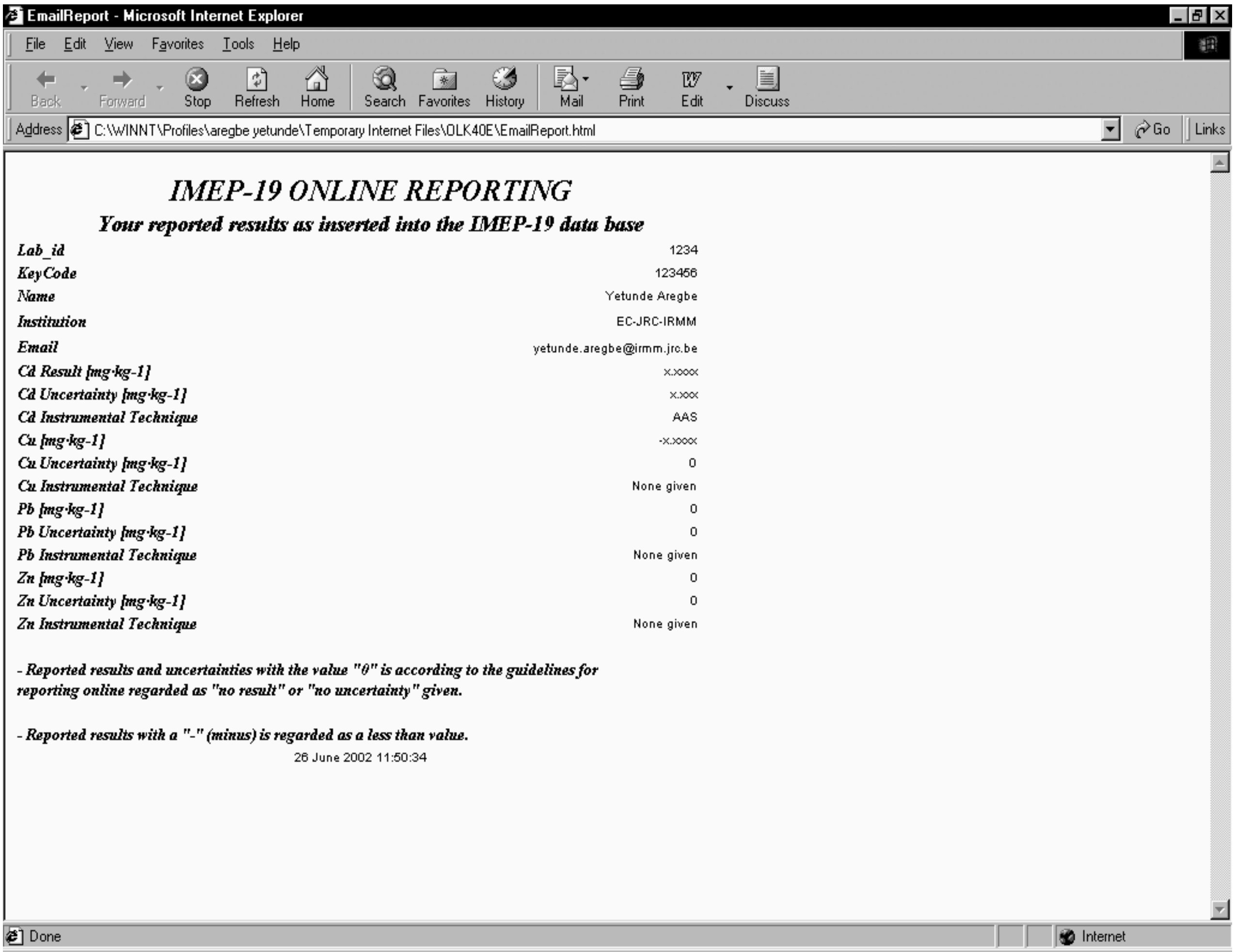
www.imep.ws • http://www.irmm.jrc.be

Figure 7

4. As a confirmation of your reported results you will receive by email an HTML file with your reported results in numerical format (see example below)



As soon as all the participants' results are entered in the IMEP-19 database, you will receive a certificate with the IMEP-19 reference values including your reported results in a regular text format (see table below as an example):

Element	Reported Measurement Result (mg·kg^{-1}) (dry-mass)	Reported Uncertainty (mg·kg^{-1}) (dry-mass)
Cd	x.xxx	x.xxx
Cu	<x.xxx	No unc. reported
Pb	No value reported	No unc. reported
Zn	No value reported	No unc. reported

Figure 8

Completing the Questionnaire Form

1. You must enter data in every field, otherwise your questionnaire information cannot be submitted. If a question cannot be answered you must enter N/A (not applicable)

2. Particularly for Questions 4) and 6) please make sure that you enter N/A in all the fields in the table that are not applicable.

3. Text fields are a maximum of 100 characters.

Instructions for Question 6

Due to requests from laboratories in the food sector, IRMM would like to provide a survey about applied water content determination and dry-mass correction in IMEP-19. A special appendix will be included in the IMEP-19 report, to emphasize the importance of this matter in food analysis.

Method A

Drying-oven-YES:
State temperature and drying time applied for oven drying. If vacuum (reduced pressure) was applied state pressure in mbar. In case vacuum-drying was NOT applied insert N/A in this field.

Method B

Karl-Fischer titration-YES:
In case elevated temperature was used during KF-titration state extraction temperature in °C, otherwise insert N/A in this field. If delay time “t (delay)” was used as stop criterion state delay time in seconds, if “stop drift” was used as stop criterion state which value (µL/min) was set, otherwise insert N/A. State the extraction time in minutes applied for the KF-titration

Instructions for Questions 8 to 10

Please report all values including uncertainty***!***

Instructions for Question 10

Factor for dry-mass correction:
Insert the applied dry-mass correction factor used in the measurement equation to calculate the total amount content of the trace elements present in the Rice sample. (This factor is deduced from the water content determination but also accounts for possible corrections due to the hygroscopicity of the rice material).

Figure 9

IMEP-19

Trace Elements in Rice
RESULTS REPORT FORM
(Deadline 30th September 2002)

(Please use capital letters)

Name :
Organisation :
Address :

Country :
Email address :
LAB-ID No. :

Please report all your results and uncertainties in the unit **mg·kg^{-1}** (milligram per kilogram). Measurement uncertainty can, e.g. be evaluated according to guides issued by ISO[1] and EURACHEM/CITAC[2]. Clearly indicate in the questionnaire (questions 16 to17) how the measurement uncertainty was evaluated.

The uncertainty indicated on this report form, should be a range claiming - for all practical purposes - to contain the true value

Element	Content (mg·kg^{-1}) (dry-mass)	Uncertainty (mg·kg^{-1}) (dry-mass)	Instrumental technique[3]
Cd			
Cu			
Pb			
Zn			

Date:

[1]International Organisation for Standardisation, "Guide to the Expression of Uncertainty in Measurement", © ISO, ISBN 92-67-10188-9, Geneva, Switzerland, 1995

[2] EURACHEM/CITAC, "Quantifying Uncertainty in Analytical Measurement", Second Edition, 2000, http://www.eurachem.bam.de/index.htm or http://www.measurementuncertainty.org/mu/guide

[3] Please use the acronyms given at the end of the questionnaire.

Figure 10

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IMEP-19 - Trace Elements in Rice
QUESTIONNAIRE FORM

The purpose of this questionnaire is to enable the organiser to compare measurement performance with additional factors such as analytical techniques, water content determination, quality management system in use, accreditation and present this to the participants in a graphical form.

ALL ANSWERS WILL BE TREATED CONFIDENTIALLY
(Non-disclosure of the identity of the laboratories)
PLEASE COMPLETE THIS FORM TOGETHER WITH THE RESULT FORM.

1. **a) How does your laboratory consider itself, experienced or less/non-experienced in the analysis of the following elements in Rice? b) Also please indicate how many samples does your laboratory routinely analyse per year for these elements.**

   (Please indicate by using a tick in the table below for each element)

Elements	Experienced	Less/non-experienced	Number of Rice samples analysed per year			
			< 50	50-250	250-1000	> 1000
Cd						
Cu						
Pb						
Zn						

2. **Was the IMEP-19 Certified Test Sample (CTS) treated according to the same analytical procedure as routinely used for this sample type?**

   YES NO

3. **Was the CTS treated in accordance to National or other standards?**

   YES NO
   If "YES", which one?…………………………………………………………………………….

   …………………………………………………………………………………………………

Figure 11

4. Did the analytical procedure involve: a) A digestion step?

YES (If YES **please complete the table below for each element)**

NO **(**if NO **please indicate in the table below which sample mass was used)**

(Enter N/A in fields that are not applicable)

Elements	Sample mass used (g)	Acids or reagents used	Type of destruction or equipment used (Microwave, high pressure ashing, dry ashing etc)
Cd			
Cu			
Pb			
Zn			

Did the analytical procedure involve: b) A Separation step?
c) A Pre-concentration step?

(Please indicate by using a tick in the table below for each element)

Elements	b) A Separation step?		c) A Pre-concentration step?	
	Yes	No	Yes	No
Cd				
Cu				
Pb				
Zn				

5. How long did you spend carrying out your measurement?

<1 Hour   <1 Day   <1 Week   1 Month

6. Indicate which method was used for the "water content determination"?

(**Please complete the tables below, enter N/A in fields that are not applicable)**

Method A

Drying-oven		Temperature (°C)	Drying time (hours)	Vacuum - drying pressure (mbar)
Yes	No			

Method B

Karl-Fischer titration		Temperature (°C)	Stop criteria		Extraction time (min)
Yes	No		drift (μL/min)	time (s)	

Solvent used for KF-titration **…………………………………………………..……….**

Figure 12

7. Was the Rice sample equilibrated with ambient humidity conditions, prior to the determination of the water content?

YES NO

8. What was the average sample mass (g) used for the "water content determination" with its uncertainty?

…………**g** ± ………**g**

9. What was the determined water content in the Rice sample with its uncertainty, state by percentage?

………… **%** ± …………**%**

10. What was the applied correction factor for dry-mass with its uncertainty?

………… ± …………

11. Which "calibration strategy" was used for the measurements?

External aqueous standards addition | Internal standard | Standard

External matrix matched standards | Isotope dilution | Other

If "Other", please specify:……………………………………………….…

12. Does your laboratory routinely use Rice Certified Reference Materials (CRMs) for quality assurance? YES NO

If "YES", state which CRM, the supplier and how the CRMs are used in your laboratory (e.g. validation of procedures, calibration of instruments, other):
....

13. Does your laboratory participate regularly in a proficiency-testing scheme in order to assess performance for this type of analysis? YES NO

If "YES", state which proficiency-testing scheme and organiser:

....…………

14. Is your laboratory working according to the guidelines of ISO 17025 or another quality management system? Please indicate the quality management system in use?

ISO 17025 | No quality management system in use.

Other (e.g. EN45000, ISO 25, ISO 9000 series, CEN, GLP, EPA, TQM, national standards)
Please indicate:
...…………………………………………………………………….…………………

Figure 13

15. Is your laboratory accredited or authorised (e.g. by national law or regulatory authority) for measurements of trace elements in Rice? Please indicate by using a tick in the table below for each element.

Elements	Accredited		Authorised	
	Yes	No	Yes	No
Cd				
Cu				
Pb				
Zn				

16. Do you routinely report uncertainties on chemical measurements to your customers?

YES NO

17. Are your reported uncertainties in IMEP-19 calculated according to the Guides for Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) and/or EURACHEM/CITAC (2000)?

YES NO

If "NO", how was the measurement uncertainty evaluated?

....

Acronyms for the Analytical Techniques in IMEP-19			
Anodic stripping voltammetry	ASV	Hydride-atomic absorption spectroscopy	H-AAS
Atomic absorption spectroscopy	AAS	Inductively coupled plasma	ICP
Cathodic-stripping voltammetry	CSV	Inductively coupled plasma-atomic emission spectrometry	ICP-AES
Cold vapour-atomic absorption spectroscopy	CV-AAS	Inductively coupled plasma-mass spectrometry	ICP-MS
Direct current plasma	DCP	Inductively coupled plasma-optical emission spectrometry	ICP-OES
Electrothermal atomic absorption spectroscopy	ETAAS	Infrared spectrometry	IR
Flame atomic absorption spectroscopy	FAAS	Ion chromatography	IC
Flame atomic emission spectroscopy	FAES	Metal hydride system	MHS
Flame atomic fluorescence spectroscopy	FAFS	No statement	None given
Flow injection analysis system-atomic absorption spectroscopy	FIAS-AAS	Other	OTHER
Graphite furnace-atomic absorption spectroscopy	GF-AAS	Potentiometric stripping analysis	PSA
High resolution-inductively coupled plasma-mass spectrometry	HR-ICP-MS	Spectrophotometry	Spectroph.
Hydride generation	HG	X-ray fluorescence	XRF
Hydride generation-atomic absorption spectroscopy	HG-AAS		

Figure 14

(Sample of e-mail sent)

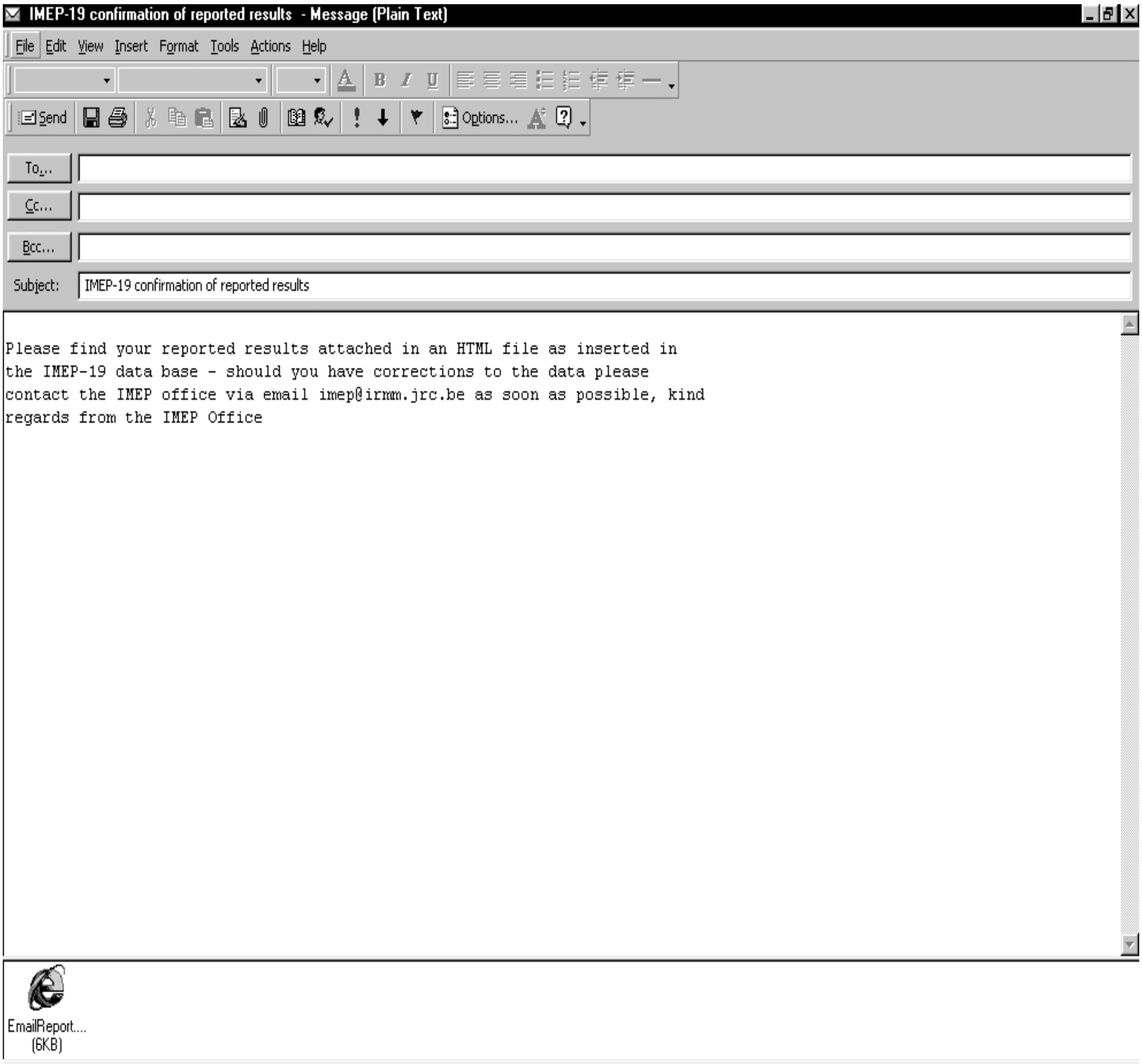

Figure 15

(Sample of HTML file – confirmation of reported results)

IMEP-19 ONLINE REPORTING

Your reported results as inserted into the IMEP-19 data base

Lab_id

KeyCode

Name

Institution

Email

Cd Result [mg·kg-1]

Cd Uncertainty [mg·kg-1]

Cd Instrumental Technique

Cu [mg·kg-1]

Cu Uncertainty [mg·kg-1]

Cu Instrumental Technique

Pb [mg·kg-1]

Pb Uncertainty [mg·kg-1]

Pb Instrumental Technique

Zn [mg·kg-1]

Zn Uncertainty [mg·kg-1]

Zn Instrumental Technique

- Reported results and uncertainties with the value "0" is according to the guidelines for reporting online regarded as "no result" or "no uncertainty" given.

- Reported results with a "-" (minus) is regarded as a less than value.

Figure 16

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC
JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP®

IM/L/80/02
October 2002

IMEP-19

Trace Elements in Rice

Certified Reference Values

Element	*Certified value mg·kg^{-1} (dry-mass)*	*Expanded uncertainty U, k=2 mg·kg^{-1} (dry-mass)*
Cadmium	***1.618***	***0.028***
Copper	***2.816***	***0.034***
Lead	***0.415 9***	***0.007 1***
Zinc	***22.99***	***0.44***

Dr. Y. Aregbe
IMEP-19 Co-ordinator
IRMM

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IMEP: an IRMM programme, with the aim to evaluate the performance of chemical measurements and establish their degree of international equivalence

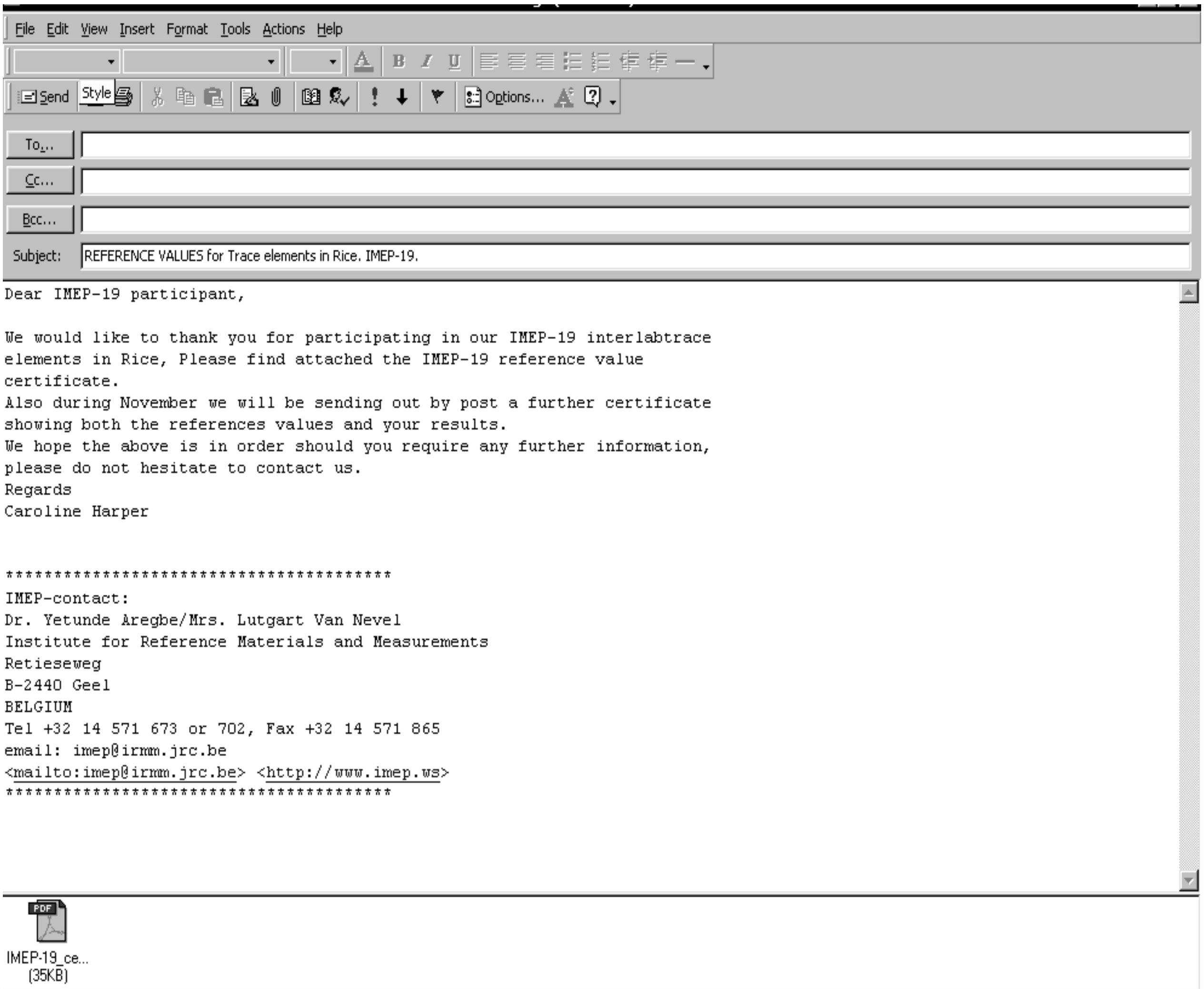
Institute for Reference Materials and Measurements
European Commission – Joint Research Centre
B-2440 GEEL (Belgium)

The certified reference values on this certificate were derived from measurements applying primary measurement procedures.
The following institutes collaborated with IRMM in the production or certification of the IMEP-19 rice samples

Bundesanstalt für Materialforschung und -prüfung
Dr J. Vogl and Dr. M. Ostermann
Unter den Eichen 87
D-12205 Berlin
Germany
http://www.bam.de

National Metrology Institute of Japan
Higashi 1-1-1, Tsukuba Central 5-2,
Tsukuba, 305-8565,
Japan
http://www.nmij.jp

Figure 17



Dear IMEP-19 participant,

We would like to thank you for participating in our IMEP-19 interlabtrace elements in Rice, Please find attached the IMEP-19 reference value certificate.
Also during November we will be sending out by post a further certificate showing both the references values and your results.
We hope the above is in order should you require any further information, please do not hesitate to contact us.
Regards
Caroline Harper

****
IMEP-contact:
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<mailto:imep@irmm.jrc.be> <http://www.imep.ws>
****

Figure 18

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC
JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP®

Geel, 27th November 2002
IM/L/94/02/«LAB_ID»

«Title» «firstname» «surname»
«companyinstitute»
«address»
«town»
«country» «zip»

Dear «Title» «surname»,

IMEP-19: trace elements in rice

Please find enclosed the IMEP-19 reference value certificate together with your individual IMEP-19 certificate. IRMM has issued individual certificates to each participant in IMEP-19. This certificate includes your reported measurement value for the IMEP-19 Certified Test Sample, the IMEP-19 Certified Reference Values and the deviation of your reported value from the certified value in percentage.

Furthermore E_n-scores [1] have been calculated for those participants in IMEP-19 who reported measurement results with uncertainties which were calculated according to the Guides for Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) and/or EURACHEM/CITAC (2000). Please note that for participants who did not state that they calculated the reported uncertainty according to the (ISO, 1995) and/or EURACHEM/CITAC (2000) guides no E_n-scores were issued.

Maximum levels of metals in foodstuff are set in the commission regulation (EC) 466/2001. In absence of performance characteristics for the uncertainty of the measured value for measurements of metals in rice in this regulation, IRMM selected as performance characteristic a range of ±10% from the reference value as extended uncertainty (U_{ext}) to calculate the E_n-scores. It can be assumed to represent an uncertainty range that is "fit for purpose" for measurements of trace metals in foodstuff.

The IMEP-19 participant's report is under preparation and on its completion will be dispatched to you during the first quarter of 2003.

We would like to thank you for taking part in this comparison and hope you have found your participation useful.

Yours sincerely,

Yetunde Aregbe

Dr. Y. Aregbe
IMEP-19 Co-ordinator

[1] The E_n-scoring is based on single performance statistics: ISO/IEC GUIDE 43-1:1997 (E)

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Figure 19

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JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

IMEP®

IM/L/94/02/«LAB_ID»
November 2002

Certificate

IMEP-19 Trace Elements in Rice

Issued to: «title» «firstname» «surname»
«companyinstitute»
«town» «zip», «country»

Element	*Reported value* $mg \cdot kg^{-1}$	*Reported uncertainty* $mg \cdot kg^{-1}$	*Certified value* $mg \cdot kg^{-1}$ *(dry-mass)*	*Expanded uncertainty U, k=2* $mg \cdot kg^{-1}$ *(dry-mass)*	*Deviation from certified value*	*E_n-score** *(E_n-numbers)*
Cadmium	«Cd»	«unc_Cd»	**1.618**	**0.028**	«Cd_Dev_Cert_Value»	«Cd_10_Enscore»
Copper	«Cu»	«unc_Cu»	**2.816**	**0.034**	«Cu_Dev_Cert_Value»	«Cu_10_Enscore»
Lead	«Pb»	«unc_Pb»	**0.415 9**	**0.007 1**	«Pb_Dev_Cert_Value»	«Pb_10_Enscore»
Zinc	«Zn»	«unc_Zn»	**22.99**	**0.44**	«Zn_Dev_Cert_Value»	«Zn_10_Enscore»

$$* E_n = \frac{x - X_{ref}}{\sqrt{U_x^2 + U_{ext}^2}}$$

$|E_n| \leq 1$ satisfactory
$|E_n| > 1$ not satisfactory

x: Reported value of IMEP-19 participant
X_{ref}: Certified IMEP-19 reference value
U_x: Uncertainty of reported participant's value
U_{ext}: Extended uncertainty: ±10% from the certified IMEP-19 reference value

Please note that E_n-scores were only issued to participants who reported measurement results with uncertainties. Which have been calculated according to the Guides for Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) and/or EURACHEM/CITAC (2000).

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Japan
http://www.nmij.jp

Figure 20

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IMEP®

IM/L/94/02/«LAB_ID»
November 2002

Certificate

IMEP-19 Trace Elements in Rice

Issued to: «title» «firstname» «surname»
«companyinstitute»
«town» «zip», «country»

Element	*Reported value mg·kg^{-1}*	*Reported uncertainty mg·kg^{-1}*	*Certified value mg·kg^{-1} (dry-mass)*	*Expanded uncertainty U, k=2 mg·kg^{-1} (dry-mass)*	*Deviation from certified value*	*E_n-score* (E_n-numbers)*
Cadmium	«Cd»	«unc_Cd»	**1.618**	**0.028**	«Cd_Dev_Cert_Value»	-
Copper	«Cu»	«unc_Cu»	**2.816**	**0.034**	«Cu_Dev_Cert_Value»	-
Lead	«Pb»	«unc_Pb»	**0.415 9**	**0.007 1**	«Pb_Dev_Cert_Value»	-
Zinc	«Zn»	«unc_Zn»	**22.99**	**0.44**	«Zn_Dev_Cert_Value»	-

$$* E_n = \frac{x - X_{ref}}{\sqrt{U_x^2 + U_{ext}^2}}$$

$|E_n| \le 1$ satisfactory
$|E_n| > 1$ not satisfactory

x: Reported value of IMEP-19 participant
X_{ref}: Certified IMEP-19 reference value
U_x: Uncertainty of reported participant's value
U_{ext}: Extended uncertainty: ±10% from the certified IMEP-19 reference value

Please note that E_n-scores were only issued to participants who reported measurement results with uncertainties. Which have been calculated according to the Guides for Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) and/or EURACHEM/CITAC (2000

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