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Institute for Reference Materials and Measurements

IMEP-21
Trace Elements, PCBs, PAHs in Sewage Sludge
Report to Participants

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IMEP®

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The mission of IRMM is to promote a common and reliable European measurement system in support of EU policies.

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Summary

The International Measurement Evaluation Programme (IMEP®) is an Interlaboratory Comparison (ILC) scheme in support of EU policies (e.g. Consumer Protection and Public Health, Single Market, Environment, Research and Technology, External Trade and Economic Policy). It is founded, owned and co-ordinated by the Institute for Reference Materials and Measurements (IRMM) of the European Commission's Directorate-General Joint Research Centre.

The aim of this interlaboratory comparison programme is to picture objectively the degree of equivalence and the quality of chemical measurements. Contrary to most other external quality assessment schemes, participating laboratories in IMEP® can compare their measurement results and uncertainty statements with external certified reference values, obtained completely independent from the participants' result. These reference values are required to demonstrate traceability and they should have a demonstrated and adequately small uncertainty, as evaluated according to international guidelines. Participants in IMEP® use their routine analytical procedures to measure the IMEP-certified test sample (CTS). Therefore they can assess the quality of their results on an international forum by comparing their values to the IMEP-reference values.

The European Union promotes the use of sewage sludge as fertilizer in agriculture. The Council Directive 86/278/EEC of 12 June 1986 sets rules for the protection of the environment, and in particular of the soil, when sewage sludge is used in agriculture. In this directive limit concentration ranges of metals in sewage sludge are laid down. In national legislation the concentration ranges for metals can be set below these limits. Furthermore limits for Polychlorinated Biphenyls (PCBs), and Polycyclic Aromatic Hydrocarbons (PAHs) congeners in sewage sludge are set in some of the national legislations. The directive 86/278/EEC is discussed to be revised and lower levels for metals and most probably also upper levels for PCBs and PAHs concentrations will be included. Participants in IMEP-21 were offered to measure the total amount content of 7 metals, 6 PCB congeners and 11 PAH congeners: Cd, Cr, Cu, Pb, Hg, Ni, Zn, PCB_28, PCB_52, PCB_101, PCB_118, PCB_153, PCB_180, sum_PCBs, anthracene, benz(a)anthracene, benz(a)pyrene, benz(b+k)fluoranthene, benzo(ghi)perylene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, pyrene, sum_PAHs.

IMEP-21 was organised in collaboration with The European Committee for Standardisation (CEN) upon approval of DG EuropAid, the Association of the European Geological Surveys (EuroGeoSurveys) Geoscientific Laboratory Network and the European Co operation for Accreditation (EA). 3228 measurement results on 26 analytes were reported by 204 participants from 44 countries in IMEP-21. Amongst those were participants nominated by CEN, EuroGeoSurvey and by national accreditation bodies (NABs) but also laboratories from Western Balkan countries (IRMM's CARDS support), new member states and other countries participated in IMEP-21. Individual IMEP-21 certificates were issued to all the IMEP-21 participants. This certificate includes the IMEP-21 certified reference values, the reported results and the performance scores of the participant.

The IMEP-21 report to participants presents organisatorial details about the project. Participants' results are presented in a graphical way together with the reference value and are sorted according to different criteria based on the replies from the questionnaire from which also numerical information is included.

IMEP®

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

invites participants to report results together with the best estimate of the expanded measurement uncertainty

enables result-oriented rather than procedure oriented evaluation of performance

demonstrates a degree of equivalence in measurement results on the international measurement infrastructure

IMEP®

Characteristics of IMEP®

Policy making and policy implementation aims at setting up a legal set of rules providing a maximum of consumer protection within healthy working and living environments and a prospering economy. In many cases implementation of international and national legislation is based on high quality chemical measurement results. Therefore laboratories need to be able to demonstrate that their measurement results are reliable, comparable and in compliance with legislation, international standards, and international recognition arrangements that support the free trade goal 'measured once, accepted everywhere'.

In support of this need, the Joint Research Centre - Institute for Reference Materials and Measurements (JRC – IRMM) operates for the European Commission the International Measurement Evaluation Programme (IMEP®), which focuses on the construction of an internationally structured measurement system. IMEP® is a metrological interlaboratory comparison tool publicly available to all laboratories, having different functions in the international measurement infrastructure. IMEP enables laboratories to assess their measurement performance and at the same time allows them to demonstrate their competence on a high quality level to accreditation, authorisation, and inspection bodies as well as to their regular customers.

In IMEP®, participating laboratories compare their results with certified reference values. They receive the characterised IMEP certified test sample with undisclosed certified reference values. To guarantee the high metrological quality, the reference measurements are performed by institutes with internationally demonstrated and mutually recognised measurement capabilities [1]. They are required to demonstrate traceability and they should have a demonstrated and adequately small uncertainty, as evaluated according to international guidelines. Those certified reference values are completely independent from the participants' result. The underlying philosophy is that the best possible values will serve as reference and these are obtained from well-understood measurement processes in a complete transparent way rather than via a consensus approach.

IMEP® is a publicly accessible metrological Interlaboratory Comparison scheme organised in support of EU policies. Therefore IMEP® is addressing different analytes in different matrices and not offering ILCs on a regular basis for a specific analyte and matrix. It intends to picture the state-of-the-practice in measurement capabilities of laboratories at a specific moment in time. IMEP® guarantees the confidentiality with respect to the identity of its participants and their reported result. Participants in IMEP® measure the analytes under investigation applying their routine measurement procedures and analytical techniques. In IMEP®, laboratories have always been invited to state uncertainty estimates for their reported results. Contrary to most regular proficiency testing schemes, the IMEP® measurement performance criteria are not only set relative to the reported value, but also to the reported measurement uncertainty.

These specific features of the IMEP® programme make it a very valuable tool for international and European organisations or reference networks to verify measurement claims and monitoring the efficiency of multilateral arrangements.

A large number of laboratories participating in IMEP® have to comply with the ISO/IEC 17025 standard [2]. They need to meet the requirement of providing reliable measurement results within uncertainties. As laboratories are accredited against this standard, many of them need training to enable them to demonstrate measurement traceability, estimate uncertainty and perform validation. IRMM has already offered training activities to participants who request additional support after the completion of the respective IMEP® comparison. An example is the IMEP-EDUCational follow-up of IMEP-12 and IMEP-20, where unsatisfactory performing laboratories agreed to assist in general case studies. Results of these activities can be found on the IMEP-EDUC website [3].Further information about IMEP activities and previous IMEP ILCs is available on the IMEP® website [4].

IMEP-21

Sewage sludge is the residue remaining from the treatment of municipal sewage. It is an organic-rich waste produced primarily by physical processes. Sewage contains aqueous domestic waste as well as surface drainage and, in many cases, a component of treated and untreated industrial effluent. Sewage sludge tends to concentrate a wide range of substances, like metals, poorly biodegradable trace organic compounds and potentially pathogenic organisms, by absorbing or binding them to the organic matrix of the sludge. If not managed properly, untreated discharged sewage effluents can pose a high risk to environmental resources and human health. The purification process enables reclaimed water to be discharged to freshwater courses or used in other applications under conditions that pose a greatly reduced risk to the receiving environment. Increased levels of waste water purification lead, inevitably, to greater quantities of sludge for which environmentally sound management strategies are required [5].
The organic matter and the nutrients present make sewage sludge very suitable for landspread as fertiliser or organic soil improver. The European Union promotes the use of sewage sludge as fertilizer in agriculture. The Council Directive 86/278/EEC of 12 June 1986 sets rules for the use of sewage sludge in agriculture as to prevent harmful effects on the environment, and in particular on the soil [6]. In this directive limit values for Cd, Cu, Hg, Ni Pb and Zn concentrations in sludge for use in agriculture are specified. In the EU sludge policy varies depending on the degree of support that the recycling of sludge in agriculture has from the political level (Agricultural and/or Environment Ministries) and farmers' perception. Therefore the implementation of the Directive in national laws resulted in some cases in much lower defined maximum concentrations for metals. Furthermore limits for PCBs and PAHs congeners or the sum of congeners in sewage sludge are also set in some of these national legislations although they are not included in the Directive 86/278/EEC [7].

PCBs are easily distributed to the environment. A high amount of these compounds was released into the environment in the 50s and 60s and contamination from, e.g. waste water dumps, incinerators and buildings is still ongoing. They are persistent and they accumulate in the food chain. They are classified as probable cancerogenic as well as possibly teratogenic and mutagenic. They can also affect thyroids, intestine, reproductive and immunologic system. Furthermore they can be related to neurological, psychological and/or psychosomatic symptoms like migraine, depression, dysfunction of memory, sleepiness, nervousness, tiredness and impotence.

PAHs are of critical environmental concern due to distribution, persistence and toxicity. Some of the PAH congeners are extremely cancerogenic, e.g. benzo(a)pyrene or benzo(b)fluoranthene. They are components of a number of products (e.g. coke, tar) generated by natural or human-related thermal processes (fire, explosions, incineration, gasification, pyrolysis) on the basis of carbon. PAHs are generally relatively resistant, both against chemical and biochemical attack [8].

The directive 86/278/EEC is discussed to be revised and lower levels for metals and most probably also upper levels for PCBs and PAHs concentrations might be included in the next revision. Participants in IMEP-21 were offered to measure the total amount content of 7 metals, 6 PCB congeners and 11 PAH congeners: Cd, Cr, Cu, Pb, Hg, Ni, Zn, PCB_28, PCB_52, PCB_101, PCB_118, PCB_153, PCB_180, sum_PCBs, anthracene, benz(a)anthracene, benz(a)pyrene, benz(b+k)fluoranthene, benzo(ghi)perylene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, pyrene, sum_PAHs.

Collaboration with European Accreditation (EA)

By going for accreditation, laboratories prove their commitment to deliver the best quality in measurements and services. Accreditation is a very useful tool for them to demonstrate technical competence to their customers. Furthermore, the accreditation infrastructure is an important component of the European Acquis Communautaire regarding technical infrastructure. In order to further improve the efficiency of accreditation in chemistry with respect to the evaluation and demonstration of the performance of laboratories, the EA and IRMM agreed to intensify their ongoing co-operation. A formal "letter of intent for co-operation" was signed by the Chairman of the EA and the director of IRMM in the beginning of 2001[9].The EA-IRMM co-operation focuses on the chemical measurements and aims at improving 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. In 2005 the existing EA-IRMM cooperation agreement was extended towards training activities for accredited laboratories and for technical assessors in the field of Metrology in Chemistry.

Accredited laboratories need to meet the requirements, according to the ISO/IEC 17025 standard, of providing reliable measurement results within uncertainties. Recently this became a very important aspect in the collaboration agreement between IRMM and EA, because in general PT providers do not ask participants to report a measurement result within uncertainty. Therefore IMEP® serves as an important tool for the National Accreditation Bodies (NABs) to ensure compliance of their accredited laboratories with ISO/IEC 17025. They may nominate laboratories to participate in IMEP®, in order to evaluate their performance against independent reliable reference values and request the laboratories to take appropriate corrective actions if needed.

Collaboration with CEN

The MEDA programme is the principal financial instrument of the European Union for the implementation of the Euro-Mediterranean Partnership. The programme offers technical and financial support measures to accompany the reform of economic and social structures in the Mediterranean partners and it is implemented by DG EuropeAid Co-operation Office (DG EuropAid) [10]. The European Committee for Standardisation (CEN) is managing a regional technical assistance programme for the quality of enterprises in the MEDA region, called Euromed Quality programme [11, 12]. The EC-Euromed Quality Programme is targeted to the countries of MEDA (Algeria, Cyprus, Egypt, Israel, Jordan, Lebanon, Malta, Morocco, Palestine, Tunisia, Turkey and Syria). The main objective of the Euromed Quality Programme is to establish a stable environment that will help MEDA enterprises to develop and place on the market quality products, able to compete in the export markets. The programme intends to improve awareness and train a number of MEDA representatives in the different aspects of:

- accreditation,
- conformity assessment,
- legislation,
- market surveillance,
- metrology,
- standardization

In the frame of the Euromed Quality Programme, the development of interlaboratory comparisons is foreseen. These interlaboratory comparisons aim to compare the results of tests developed in different countries in the MEDA region, with the results of similar tests developed in laboratories in the EU. The interlaboratory comparisons will help to build mutual trust among the laboratories. In this framework IRMM and CEN have agreed to collaborate in view of participation of MEDA laboratories in IMEP-21, upon approval of DG EuropAid.

Collaboration with the EuroGeoSurvey

The Association of the European Geological Surveys (EuroGeoSurveys) and the JRC-Institute for Environment and Sustainability (IES) established a draft contract on "Interoperability and harmonisation of geoscientific spatial data in the framework of the INSPIRE directive (Infrastructure for Spatial Information in Europe) and of the EU sustainable development policy" [13, 14, 15]. The objective of this envisaged collaboration is the demonstration of the use of EuroGeoSurveys Geochemistry Working Group 'European Geochemical Baseline Atlas and Database' by making it accessible through the European Spatial Data Infrastructure within the framework of the European Commission initiative INSPIRE. In article 1 of this draft collaboration agreement the testing of geochemical laboratory methods for the harmonised monitoring of heavy metals in sediments and water, involving the EuroGeoSurveys Geoscientific Laboratory Network and the JRC's Institute for Reference Materials and Measurements is identified as an area of common interest. Together these two institutions will carry out tests and interpretation of tests on trace elements contents and heavy metal pollution in rock, soils, sediments and water, benefiting from IRMM's expertise in providing tools to create confidence in the quality of measurement results. As first activity cooperation for participation of laboratories nominated by the EuroGeoSurveys Geoscientific Laboratory Network was established in IMEP-21.

IMEP-21 in support of the chemical measurement infrastructure

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® contributes to this by providing support to EU policies. By offering IMEP® to testing and calibration laboratories, IRMM supports the EU Member States by ensuring confidence in their national measurement system. IMEP® therefore enables to assess whether national measurement systems are in place to provide for an equivalent implementation of directives across an enlarged EU. To specific groups of laboratories this support can be organised in the frame of collaboration agreements or specific support programmes (IRMM's CARDS support).

A direct way to support the chemical measurement infrastructure, is to link, when possible, laboratories situated on the different levels of the international measurement infrastructure. This is realised by using samples from the same material batch in the various interlaboratory comparison programmes organised on the different levels. Over the past few years, the International Committee for Weights and Measures (CIPM), the guardian of the International Measurement System (the SI), has taken several initiatives to improve the equivalence of chemical measurements worldwide. In October 1999, IRMM and other National Metrology Institutes signed the Mutual Recognition Arrangement (MRA), [16]. The MRA enables National Metrology Institutes (NMIs) to demonstrate their measurement capability by participating in key comparisons and pilot studies. The same sewage sludge material as used in IMEP-21 was also used for a key comparison and a pilot study of the Consultative Committee of Amount of Substance of the CIPM, (CCQM-K44 and CCQM-P70). Eight signatories of the MRA and 4 expert laboratories participated in CCQM-K44 & CCQM-P70. Results of this key comparison and the pilot study will be accessible via the Bureau International des Poids et Mesures (BIPM) web-site [17]. Hence IMEP-21 participants can compare their results with the results of laboratories that represent their country at the international measurement structure level and vice versa.

The IMEP-21 Certified Test Sample

The sewage sludge

The sludge material originates from different sewage plants in Italy and France. The sample is a dried and milled sewage sludge blend bottled in amber glass bottles each one containing ~ 40 g of material. The total organic carbon content in this material was around 15%.

Within, between bottle homogeneity and stability tests were carried out applying several analytical methods. For Cd, Hg and Pb solid sample Zeemann Atomic Absorption Spectrometry (SS-ZAAS) was carried out on 10 sub-samples of 10 bottles, furthermore additional test were done on 3 to 5 sub-samples from 3 to 5 bottles applying Isotope Dilution Mass Spectrometry (IDMS) for Cd, Hg, Pb and Zn. For Cu and Cr, the homogeneity was assessed by analysing 3 sub-samples from 10 bottles applying k_0-Neutron Activation Analysis (k_0-NAA). For Ni 3 subsamples from 5 bottles applying Isotope Dilution Mass Spectrometry (IDMS) and Atomic emission spectrometry (AES) were measured. Homogeneity and stability for PCBs and PAHs congeners were assessed by means of Gas-chromatography-mass spectrometry (GC-MS). Results from all these measurements were combined applying the analysis of variance ANOVA [18, 19, 20]. As a result this material was found to be appropriate for the needs of this comparison [21].

IMEP-21 Certified Reference value

Reference measurements are performed by institutes with internationally demonstrated and/or mutually recognised measurement capabilities. Institutes admitted to perform reference measurements in IMEP have to comply with the stringent technical requirements as set in the call for expressions of interest published in the Official Journal no. S 53 045071 of the European Communities [22]. This also accounts for IRMM as a reference laboratory in IMEP-21. Therefore IRMM had to demonstrate its measurement capability for the specific measurands in the sewage sludge material. As signatory of the Mutual recognition agreement (MRA) IRMM has already successfully proven the measurement capabilities to measure trace elements in various matrices [1]. IRMM participated previous to the establishment of the certified IMEP-21 reference values to the CCQM-K44 key comparison & CCQM-P70 pilot study where the same CTS was used and thus its measurement capabilities, to measure the metals under investigation in sewage sludge have been successfully confirmed by comparison to other NMIs and expert laboratories worldwide. NMIs support routine laboratories in their country with expert advice and calibration services, and may have a stated responsibility to assure that measurements are traceable. The results of CCQM K44 will be published in the Metrologia Technical Supplement and will be accessible via the BIPM web-site [17].

The IMEP-21 certified reference values are given in Table 1. The institutes involved in establishing the IMEP-21 certified reference values are listed in Table 2. The analytical techniques applied to establish the IMEP-21 reference values were ID-ICP-MS, ID-TIMS, k_0-NAA and GC-MS. Further details on the methodology of the IMEP-21 certification process can be found in the IMEP-21 certification report, which is publicly available via the IMEP web-site [21, 4].

IMEP-21 proves once more that the IMEP metrological tool is a complete transparent process. It supports routine laboratories by providing independent reference values. Therefore they can assess the quality of their results on an international forum by comparing their measurement results to those reference values. This is of major importance, particularly in cases when the certified reference value would not coincide with a consensus value derived from the participants' results (see Annex 1, Figure 3).

Table 1: IMEP-21 Certified reference values

analyte	certified value in $mg \cdot kg^{-1}$	expanded uncertainty in $mg \cdot kg^{-1}$ *U*, *k*=2
Cadmium	19.23	0.24
Chromium	209	10
Copper	843	40
Lead	619.8	8.7
Mercury	9.03	0.36
Nickel	68.5	1.0
Zinc	3160	72

analyte	certified value in $ng \cdot g^{-1}$	expanded uncertainty in $ng \cdot g^{-1}$ *U*, *k*=2
PCB_28	48.2	9.0
PCB_52	92.3	4.7
PCB_101	121.0	6.9
PCB_118	87.4	4.1
PCB_153	203.0	9.1
PCB_180	157	12
Sum_PCBs	709	20

analyte	certified value in $ng \cdot g^{-1}$	expanded uncertainty in $ng \cdot g^{-1}$ *U*, *k*=2
Anthracene	104	18
Benz(a)anthracene	386	56
Benz(a)pyrene	383	91
Benz(b+k)fluoranthene	1110	160
Benzo(ghi)perylene	508	58
Fluoranthene	916	63
Fluorene	90	22
Indeno(1,2,3-cd)pyrene	370	54
Naphthalene	116	27
Phenanthrene	746	77
Pyrene	1280	120
Sum_PAHs	6010	260

Table 2: IMEP-21 Reference laboratories

Logo	Address	Contact
	European Commission – Joint Research Centre Institute for Reference Materials and Measurements Isotope Measurement Unit Retieseweg 111 B-2440 Geel Belgium	http://www.irmm.jrc.be/imep/
	Studiecentrum voor Kernenergie Centre d'étude de l'énergie nucléaire Boeretang 200 2400 MOL Belgium e	http://www.sck.be
	Federal Institute for Materials Research and Testing Unter den Eichen 87 D-12205 Berlin Germany	http://www.bam.de
	VITO Flemish Institute for technological research Boeretang 200 BE- 2400 MOL Belgium	http://www.vito.be/
	Universität für Bodenkultur Wien University of Natural Resources and Applied Life Sciences Vienna, Austria /	http://www.boku.ac.at

IMEP-21 regional co-ordinators

Over the years a network of Regional Co-ordinators (RCs) was established for IMEP. RCs are typically people from institutions which are 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. The general contact list of RCs can be found on the IMEP website [4]. The RCs active in IMEP-21 are given in Table 3.

In the frame of collaboration agreements with other organisations, a co-ordinator from those organisations is appointed who liases with IRMM throughout the organisation of a specific IMEP ILC. For EA, Dr. Christian Lehmann from the "Deutsches Akkreditierungssystem Prüfwesen (DAP)" co-ordinated the nomination of laboratories with the NABs. Mrs Elisa Setién from CEN co-ordinated the nomination of laboratories from the MEDA region in the frame of the Euromed quality program. Dr. Andras Bartha from the Geological Institute of Hungary co-ordinated the nomination of laboratories from the EuroGeoSurveys Geoscientific Laboratory Network.

Table 3. Regional Co-ordinators for IMEP-21.

Institution/Organisation	Country	Institution/Organisation	Country
Institute of Environment	ALBANIA	Latvian National Accreditation Bureau (LATAK)	LATVIA
Instituto Nacional de Tecnologia	ARGENTINA	Angeles Aguilar Mexican Accreditation Body (ema)	MEXICO
NATA	AUSTRALIA	Measurement Standards Laboratory of New Zealand	NEW ZEALAND
IFA, Interuniversitäres Forschungsinstitut für Agrarbiotechnology Tulln	AUSTRIA	National Veterinary Institute	NORWAY
University of Sarajevo	BOSNA AND HERZEGOVINA	University of Warsaw	POLAND
National Center of Metrology	BULGARIA	RELACRE - Associação dos Laboratórios Acreditados de Portugal	PORTUGAL
Canadian Association for Environmental Analytical Laboratories (CAEAL)	CANADA	National Institute of Metrology	RUMANIA
State Office for Standardization and Metrology	CROATIA	Bureau of Measures and Precious Metals - ZMDM	SERBIA AND MONTENEGRO
State General Laboratory	CYPRUS	PSB Corporation	SINGAPORE
Czech Metrology Institute	CZECH REPUBLIC	Slovak Institute of Metrology (SMU)	SLOVAKIA
Danish Fundamental Metrology	DENMARK	Metrology Institute of the Republic of Slovenia (MIRS)	SLOVENIA
Egyptian Society of Analytical Chemistry	EGYPT	CSIR National metrology Laboratory	SOUTH AFRICA
University of Tartu	ESTONIA	SP	SWEDEN
Bureau National de Métrologie - LNE	FRANCE	Environmental Research and Training Center Technopolis	THAILAND
Aristotle University of Thessaloniki	GREECE	NMi -Van Swinden Laboratorium	THE NETHERLANDS
National Office of Measures (OMH)	HUNGARY	Laboratory of the Government Chemist	UNITED KINGDOM

IMEP-21 Organisatorial details

The planning of the comparison started in January 2005. Due to unforeseen shortage of human resources the timing of IMEP-21 had to be postponed. All collaborating organisations, all RCs and interested participants were personally informed by IRMM about this fact. Furthermore the revised timing was announced on the IMEP web-site. After registration of participants had been finalised in July 2005, the IMEP-21 CTS was distributed with accompanying documents to the participants during September 2005. Each participant received a personal key-code in order to report their results and questionnaire information online. Guidance documents were prepared and accessible via the IMEP web-site (see Annex 3). The deadline for the participants to report their results was the 30th November 2005. In December 2005 the IMEP-21 reference value certificate was publicly accessible on the IMEP web-site. The individual participant's certificate was dispatched by post to each participant in February 2006 (see Annex 3).

Sewage Sludge CTS mailing

Individual boxes were prepared at IRMM, containing the Sewage Sludge CTS material and relevant information documents (see Annex 3):

- **An info letter**: giving information relevant to the comparison, pointing out timings and practicalities concerning the on-line reporting including the individual identification number (Password Key).
- **The online reporting guideline**: issued to show how to report results and complete the Questionnaire information electronically via the IMEP web-site
- **The sample receipt form** to acknowledge that the CTS arrived at its destination in good order

The CTS were sent using express mail. For those countries with an active RC, the individual boxes were sent to the RC as one batch for distribution to the participants in their country. All other laboratories received their packages on individual basis.

Data collection

All IMEP-21 participants reported their measurement results online through the IMEP web-site.

This was enabled by a newly Oracle-based database system, developed at IRMM. As a consequence, the management of large amounts of data is facilitated hence reducing the number of transcription errors. Nevertheless, as last step of the reporting procedure, laboratories were asked to print the report form and return it to IRMM signed. Only after receipt of the signed copy, the online result was validated. IMEP accepted and implemented any corrections of submitted results until the reporting deadline. An example of the IMEP-21 report form is given in Annex 3.

In addition to the result report form also a questionnaire was offered (see Annex 3). The purpose of this questionnaire was to enable the organiser to correlate measurement performance with other factors such as analytical technique used, self-assessment of experience, accreditation and to present this partly to the participants in this report. Additional information gained from this questionnaire will serve to identify the state-of-the-practice in sewage sludge analysis and will be used to plan and identify needs of future IMEP ILCs.

All reported information is treated in a confident way. This means that IMEP does not reveal the link between identity of the laboratory and the reported results or information received by means of the questionnaire.

Participation in IMEP-21

Samples were distributed to all 217 registered laboratories from 47 countries. Measurement results were reported by 204 participants (94% of the registered laboratories) from 45 countries (Table 4). Among those were 80 laboratories nominated by their NABs, 14 MEDA laboratories nominated via the Euromed quality programme and 9 laboratories nominated via the EuroGeoSurveys Geoscientific Laboratory Network. In the frame of IRMM's support to candidate and Western Balkan countries (CARDS), 8 participants in IMEP-21 came from these regions. The remaining participants were from EU member states and also some from outside Europe.

Table 4 IMEP-21 number of registered and reporting laboratories per country

Country	Registered laboratories	Results received	Country	Registered laboratories	Results received
ALBANIA	2	1	KOREA, REP. OF	1	1
ALGERIA	1	1	LATVIA	5	5
ARGENTINA	3	2	LEBANON	2	2
AUSTRALIA	4	4	MALTA	1	0
AUSTRIA	4	3	MEXICO	5	5
BELGIUM	6	6	MOROCCO	2	2
BOSNIA - HERZEGOVINA	1	0	NETHERLANDS	7	5
BULGARIA	3	2	NEW ZEALAND	4	4
CANADA	10	10	NORWAY	4	4
CHILE	1	1	POLAND	5	4
CROATIA	2	2	PORTUGAL	17	16
CYPRUS	4	4	ROMANIA	1	1
CZECH REPUBLIC	10	10	SERBIA - MONTENEGRO	2	2
DENMARK	6	6	SINGAPORE	1	1
EGYPT	4	4	SLOVAKIA	10	10
ESTONIA	6	6	SLOVENIA	1	1
FINLAND	6	5	SOUTH AFRICA	2	1
FRANCE	10	10	SPAIN	5	5
GERMANY	8	8	SWEDEN	5	5
GREECE	4	4	SWITZERLAND	2	2
HUNGARY	12	12	THAILAND	4	4
INDIA	1	1	TUNISIA	1	1
ITALY	16	16	UNITED KINGDOM	4	4
JORDAN	2	1			
			TOTAL	**217**	**204**

Evaluation of Performance

Laboratories using routine methodologies are not expected to achieve the same level of precision as National Metrology Institutes using, if possible, primary or other internationally recognised measurement procedures [23]. It was therefore necessary to define a 'fit-for-purpose' quality requirement for the performance assessment of participating laboratories in IMEP 21. The Council Directive 86/278/EEC only gives limit values for metals in sewage sludge, but does not comment on any performance or method characteristics besides requirements for limits of detection. Requirements on repeated analysis of contaminants as in IMEP-21 are given in some of the national legislation or in European Directives of contaminants in foodstuff. Therefore the fit-for-purpose criteria in IMEP-21 were set relative to the certified reference values after discussion with the EA co-ordinator (Table 5). They are based closely on legislation and international/national norms [6, 7, 24].

Table 5: IMEP-21 fit-for-purpose performance criteria

Analyte	Fit-for-purpose criterion, σ
Cadmium	$0.1\ X_{ref}$
Chromium	$0.1\ X_{ref}$
Copper	$0.05\ X_{ref}$
Lead	$0.05\ X_{ref}$
Mercury	$0.1\ X_{ref}$
Nickel	$0.1\ X_{ref}$
Zinc	$0.05\ X_{ref}$
PCB_28	$0.15\ X_{ref}$
PCB_52	$0.15\ X_{ref}$
PCB_101	$0.15\ X_{ref}$
PCB_118	$0.15\ X_{ref}$
PCB_153	$0.15\ X_{ref}$
PCB_180	$0.15\ X_{ref}$
Sum_PCBs	$0.15\ X_{ref}$
Anthracene	$0.15\ X_{ref}$
Benz(a)-anthracene	$0.15\ X_{ref}$
Benz(a)pyrene	$0.2\ X_{ref}$
Benz(b+k)-fluoranthene	$0.15\ X_{ref}$
Benzo(ghi)-perylene	$0.15\ X_{ref}$
Fluoranthene	$0.15\ X_{ref}$
Fluorene	$0.2\ X_{ref}$
Indeno(1,2,3-cd)-pyrene	$0.15\ X_{ref}$
Naphthalene	$0.2\ X_{ref}$
Phenanthrene	$0.15\ X_{ref}$
Pyrene	$0.15\ X_{ref}$
Sum_PAHs	$0.15\ X_{ref}$

Scoring is the method of converting a participants' reported result into a standard that adds judgmental information about performance [25]. The different IMEP-21 performance assessment scores and assessment criteria are listed in Table 6.

Where X_{ref} is the reference value; u_{ref} is the combined standard uncertainty of the reference value; σ is the fit-for-purpose criterion, *x* is the result reported by the participant; u_x is the associated combined standard uncertainty

Table 6: IMEP-21 Performance evaluation scores

Performance scoring		Performance assessment criteria	
Percentage difference	$D\% = \frac{(x - X_{ref})}{X_{ref}} \cdot 100$	$\lvert D\% \rvert \leq 2\sigma / X_{ref} \cdot 100$ $\lvert D\% \rvert > 2\sigma / X_{ref} \cdot 100$	Satisfactory Unsatisfactory
z-score	$z = \frac{(x - X_{ref})}{\sigma}$	$\lvert z \text{ or } zeta' \rvert \leq 2$ $2 < \lvert z \text{ or } zeta' \rvert \leq 3$ $\lvert z \text{ or } zeta' \rvert > 3$	Satisfactory Questionable Unsatisfactory
zeta'-score	$zeta' = \frac{x - X_{ref}}{\sqrt{u_x^2 + \sigma^2}}$		
Acceptable Uncertainty	only issued if $\lvert zeta' \rvert \leq 2$.	if $u_{ref} \leq u_x \leq \sigma$ Otherwise	'YES' 'NO'

The z-score is the score that proficiency testing participants are most familiar with seen the wide applicability and acceptance. In IMEP participants are always invited to report their measurement results with uncertainties. The evaluation of performance in IMEP ILC is thus not only done relative to the reported measurement values but also to the reported measurement uncertainties. The applied scores are thoroughly described in [25, 26, 27, 28]. In IMEP-21 the percent difference, the z-score and the zeta' score were issued to the participants. The criticism all scores that include reported measurement uncertainties have to encounter is that participants could achieve satisfactory scores by increasing the uncertainties of their measurement results. Therefore IRMM developed recently in discussions with the EA Working Group for ILCs in the field of testing additional criteria for minimum and maximum acceptable uncertainties to complete satisfactory scores that take reported measurement uncertainties into account [29, 30]. This additional performance assessment criterion was also issued to the participants in IMEP-21. Seen the fact that the equation to calculate zeta'-scores propagates combined standard uncertainties, the following approach was applied in order to convert the reported uncertainties into combined standard uncertainties. For laboratories that reported a coverage factor *k*, the standard uncertainty was calculated by dividing the reported uncertainty (assumed to be an expanded uncertainty) by the *k* factor reported. When no coverage factor was reported, the reported uncertainty was considered as the range of a rectangular distribution (± a); the combined standard uncertainty was then calculated dividing this range by √3, according to *Appendix E*-of the EURACHEM/CITAC Guide (2000) Quantifying uncertainty [31]. From the equations in Table 6 it can be easily seen that:

$$\lim_{u_x \to 0} zeta'(u_x) = z$$

IMEP-21 individual certificate

IRMM has issued individual certificates to each participant in IMEP-21. On this certificate, the reported results, the certified reference values, the percent difference and the z-score are listed. For all participants who reported a measurement uncertainty the zeta'-score and, in case $\lvert zeta' \rvert \leq 2$, the acceptable uncertainty assessment criteria were given. An individual certificate template is presented in Annex 3.

Reported results

Participants in IMEP-21 were free to measure the analytes that were of interest to their laboratories. The inorganic analytes were measured by more than 80% of the participants. The most popular elements were Cu and Zn measured by 94% of the participants, followed by Cd, Cr, Pb, Ni and finally Hg that was measured by 80% of the participants. The organic analytes were measured by about half of the IMEP-21 participants. The most popular organic analytes were as expected the cancerogenic PAH congeners benz(a)pyrene and benz(b+k)-fluoranthene measured by 54% of the participants. Only 25% of the IMEP-21 participants measured all analytes under investigation, the inorganic metals as well as all organic congeners.

The first column in Table 7 lists the number of reported results per analyte, including the values that were reported as range with an upper limit ('less than value') rather than as total amount content. The second column in Table 7 lists the percentage of participants who reported a result with uncertainty in IMEP-21. The last column gives the percentage of participants who reported a coverage factor, *k*.

Measurement uncertainties were reported by 52%-65%, a coverage factor *k* was reported by 44%-56% of the IMEP-21 participants for the various analytes.

Laboratory performance evaluation

From Table 8 it can be seen that the number of participants with satisfactory z-scores ranges from 30% for anthracene to 80% for Cd. 76% of the participants obtained a satisfactory z-score for the sum_PAHs, 72% for the sum_PCBs.

Zeta'-scores were only calculated for those participants who reported an uncertainty with their result (see Table 7), since the *zeta'*-score equals the *z*-score for u_x=0 [28, 29, 30].

Table 9 summarises the number of laboratories with satisfactory, questionable or unsatisfactory *zeta'*-scores. The 4th column in Table 9 lists the satisfactory *zeta'*-scores in percentage relative to the number of calculated zeta'-scores. The number of participants with satisfactory *zeta'*-scores ranges from 40% for anthracene to 90% for Cu. 79% of the participants obtained a satisfactory *zeta'*-score for the sum_PAHs and the sum_PCBs.

The 5th column in Table 9 includes the criteria for maximum and minimum acceptable uncertainty. This means reported results with an uncertainty statement that falls in the acceptable range of $u_{ref} \leq u_x \leq \sigma$. The number of laboratories with a satisfactory *zeta'*-score and an acceptable uncertainty decreases from 72% for Cd to 8% for Fluorene compared to the number of laboratories with a satisfactory *zeta'*-score. The small number of laboratories with a satisfactory *zeta'*-score and an acceptable uncertainty can be partly explained because of the fact that for a few congeners the combined standard uncertainty u_{ref} was around 10%. This was due to material intrinsic homogeneity and short term stability characteristics of the sludge [21]. Since each participant received only 1 bottle to carry out their measurements, some of the IMEP-21 participants reported for those congeners combined standard uncertainties u_x only slightly smaller than u_{ref} (e.g. benz(a)pyrene, Annex 1 Figure 33).

The largest difference between *zeta*'-scores and zeta'-scores with acceptable uncertainty (70%!!) is observed for Cu. This is due to the fact that on the one hand there were some laboratories that reported a measurement uncertainty for their Cu result smaller than u_{ref}. On the other hand many participants reported an uncertainty larger than σ (see also Table 5). The smallest difference between *zeta*'-scores and zeta'-scores with acceptable uncertainty (10%!!) is observed for Ni, because the majority of the participants reported an uncertainty $u_{ref} \leq u_x \leq \sigma$. For Sum_PCBs the difference between *zeta*'-scores and zeta'-scores with acceptable uncertainty is 19%, for Sum PAHs it is 30%.

Table 7: Number of reported results (including 'less than values'), reported results with uncertainties (%), reported results with reported k-factors (%)

Analyte	No. of reported results (including 'less than values')	No. of reported uncertainties (%)	No. of reported k-factors (%)
Cadmium	190	65.26%	55.79%
Chromium	187	65.24%	55.08%
Copper	191	65.45%	55.50%
Lead	186	66.13%	55.38%
Mercury	164	63.41%	52.44%
Nickel	184	65.22%	53.80%
Zinc	191	64.40%	53.93%
PCB_28	95	62.11%	49.47%
PCB_52	100	61.00%	49.00%
PCB_101	103	60.19%	49.51%
PCB_118	98	60.20%	48.98%
PCB_153	104	59.62%	49.04%
PCB_180	103	60.19%	49.51%
Sum_PCBs	89	53.93%	46.07%
Anthracene	99	52.53%	43.43%
Benz(a)-anthracene	101	57.43%	48.51%
Benz(a)pyrene	110	60.00%	50.91%
Benz(b+k)-fluoranthene	110	59.09%	50.00%
Benzo(ghi)-perylene	108	59.26%	50.93%
Fluoranthene	107	61.68%	52.34%
Fluorene	99	51.52%	44.44%
Indeno(1,2,3-cd)-pyrene	108	58.33%	49.07%
Naphthalene	98	53.06%	44.90%
Phenanthrene	100	59.00%	50.00%
Pyrene	102	58.82%	49.02%
Sum_PAHs	101	52.48%	43.56%

Table 8: Number of laboratories in relation to z-scores

Analyte	No. of laboratories with satisfactory z-score	No. of laboratories with Questionable z-score	No. of laboratories with Unsatisfactory z-score	Satisfactory z-scores in percentage
Cadmium	151	23	15	79.89%
Chromium	79	69	39	42.25%
Copper	147	21	23	76.96%
Lead	89	45	52	47.85%
Mercury	102	21	40	62.58%
Nickel	105	55	23	57.38%
Zinc	126	28	37	65.97%
PCB_28	43	10	39	46.74%
PCB_52	58	13	28	58.59%
PCB_101	69	12	21	67.65%
PCB_118	60	8	28	62.50%
PCB_153	71	15	17	68.93%
PCB_180	72	12	17	71.29%
Sum_PCBs	63	8	17	71.59%
Anthracene	27	16	47	30.00%
Benz(a)-anthracene	48	17	33	48.98%
Benz(a)pyrene	69	24	13	65.09%
Benz(b+k)-fluoranthene	52	22	33	48.60%
Benzo(ghi)-perylene	51	16	36	49.51%
Fluoranthene	59	22	25	55.66%
Fluorene	43	12	35	47.78%
Indeno(1,2,3-cd)-pyrene	46	17	39	45.10%
Naphthalene	34	20	31	40.00%
Phenanthrene	58	15	26	58.59%
Pyrene	66	22	12	66.00%
Sum_PAHs	76	11	13	76.00%

Table 9: Number of laboratories in relation to zeta'-scores (zeta'-scores were only calculated for laboratories that reported an uncertainty, see also Table 7)

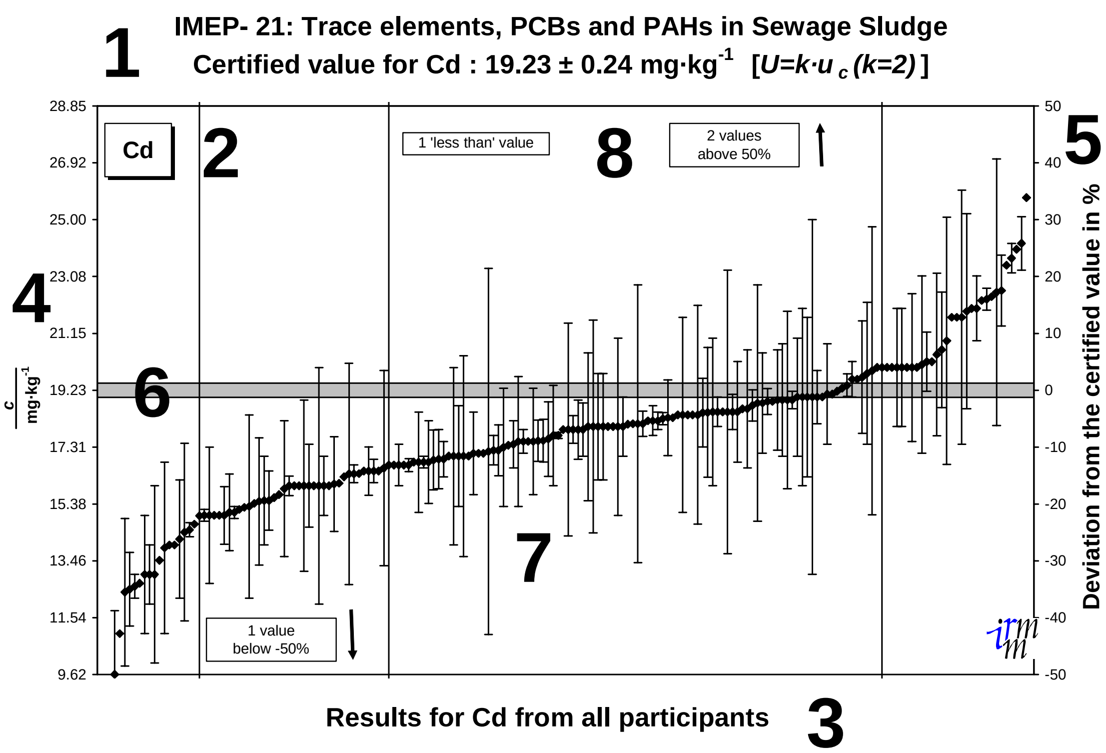
Analyte	No. of laboratories with Satisfactory zeta'-score	No. of laboratories with Questionable zeta'-score	No. of laboratories with Unsatisfactory zeta'-score	Satisfactory zeta'-score in percentage relative to the no. of calculated zeta'-scores	Satisfactory zeta'-score with $u_{ref} \leq u_x \leq \sigma$ in percentage relative to the no. of calculated zeta'-scores
Cadmium	107	11	6	86.29%	71.77%
Chromium	72	37	13	59.02%	34.43%
Copper	112	7	6	89.60%	20.00%
Lead	84	19	20	68.29%	29.27%
Mercury	77	7	20	74.04%	48.08%
Nickel	88	25	7	73.33%	62.50%
Zinc	93	17	13	75.61%	31.71%
PCB_28	28	10	21	47.46%	15.25%
PCB_52	40	8	13	65.57%	54.10%
PCB_101	48	5	9	77.42%	58.06%
PCB_118	38	6	15	64.41%	52.54%
PCB_153	50	7	5	80.65%	67.74%
PCB_180	49	8	5	79.03%	59.68%
Sum_PCBs	38	4	6	79.17%	60.42%
Anthracene	21	7	24	40.38%	11.54%
Benz(a)-anthracene	30	10	18	51.72%	18.97%
Benz(a)pyrene	50	8	8	75.76%	19.70%
Benz(b+k)-fluoranthene	38	10	17	58.46%	20.00%
Benzo(ghi)-perylene	36	12	16	56.25%	32.81%
Fluoranthene	41	14	11	62.12%	28.79%
Fluorene	26	10	15	50.98%	7.84%
Indeno(1,2,3-cd)-pyrene	37	12	14	58.73%	12.70%
Naphthalene	30	9	13	57.69%	17.31%
Phenanthrene	39	8	12	66.10%	25.42%
Pyrene	47	8	5	78.33%	40.00%
Sum_PAHs	42	7	4	79.25%	49.06%

IMEP graphical displays

Figure 1 shows how results are displayed in IMEP®. All participants' results of IMEP-21 are plotted in ascending order against the certified reference value (is middle of the reference range). All reported results are included in the graphs. The scale of the majority of the graphs is chosen for convenience displaying a ± 50% deviation from the reference value. In case a large spread of results was observed for an analyte the scale was enlarged to ± 100% deviation from the reference value. No results are excluded in IMEP®, but those that are off-scale are presented in textboxes on each graph.

A set of general graphs was prepared where the reported results were sorted according to different criteria based on information from the questionnaire, e.g. 'self-declared experience level', 'accreditation, authorisation status of the laboratory for this type of analysis', 'the analytical technique used' etc. All graphical displays are plotted in Annex 1 of this report.

Figure 1: Description of the content displayed in the result graph



1 Legend with project name and certified reference value for the displayed component.

2 Component name

3 Legend explaining details of the graph.

4 Scale with the value of the quantity expressed in absolute numbers.

5 Scale with the value of the quantity expressed in % relative deviation from the certified reference value.

6 Range (shaded) encompassing the certified reference value and its expanded uncertainty.

7 Participants' reported measurement result and self-declared reported uncertainty.

8 Box indicating results falling outside the scale of the graph.

Analytical techniques

IMEP® is result-oriented and hence does not focus on studying the different analytical techniques for this type of analysis in detail. To enable the graphical presentation of all results in relation to the analytical technique used, the various reported analytical techniques as presented in the first column of Table 10 are grouped according to the acronyms in column 2.

Graphs showing the reported results in relation to the analytical technique-group are given in Annex 1.

The techniques most frequently used to analyse the inorganic components in the sewage sludge were AAS and ICP-AES&ICP-OES. The majority of participants analysed the PCBs by either GC-MS or GC-ECD. GC-MS, HPLC-MS and HPLC-FLU were the most frequently used techniques to analyse the PAHs (Table 11 – Table 14).

All laboratories were informed on registration of the nominal metal, PCBs and PAHs content in the sewage sludge material. (see announcement letter in Annex 3).

Table 10: Reported analytical techniques and acronyms

Analytical technique	**Acronym**
Atomic absorption spectroscopy	AAS
Atomic emission spectroscopy	AES
Atomic fluorescence spectroscopy	AFS
Dual column Gas Liquid Chromatography - Electron Capture Detection	CG-ECD/ECD
Cold Vapour-atomic absorption spectroscopy	CV-AAS
Cold Vapour-atomic fluorescence spectroscopy	CV-AFS
Direct mercury analysis	DMA
Energy Dispersive X-ray Fluorescence - Conventional	EDXRF-CON
Electrothermal atomic absorption spectroscopy	ETAAS
Flame atomic absorption spectroscopy	FAAS
Flame atomic absorption spectrometry-Mercury/Hydride System	FAAS-MHS
Flame atomic emission spectroscopy	FAES
Flame atomic fluorescence spectroscopy	FAFS
Flow injection analysis system-atomic absorption spectroscopy	FIAS-AAS
Flow Injection Mercury System	FIMS
Gas chromatography-atomic fluorescence spectroscopy	GC-AFS
Gas Chromatography-Electron Capture Detector	GC-ECD
Gas chromatography-flame ionization detection	GC-FID
Gas chromatography-inductively coupled plasma-mass spectrometry	GC-ICP-MS
Gas chromatography-mass spectrometry	GC-MS

Analytical technique	**Acronym**
Graphite furnace-atomic absorption spectroscopy	GF-AAS
Gas Liquid Chromatography - Electron Capture Detection	GLC-ECD
Hydride generation-atomic absorption spectroscopy	HG-AAS
Hydride generation- inductively coupled plasma-mass spectrometry	HG-ICP-MS
High performance liquid chromatography photodiode array detection	HPLC-DAD
High performance liquid chromatography flame ionization detection	HPLC-FLD
high performance liquid chromatography- fluorescence detection	HPLC-FLU
High performance liquid chromatography- mass spectrometry	HPLC-MS
High performance liquid chromatography- ultra violet detection	HPLC-UV
High Resolution Gas chromatography- mass spectrometry	HR-GC-MS
Inductively coupled plasma- atomic emission spectrometry	ICP-AES
Inductively coupled plasma- Flow injection analysis system	ICP-FIAS
Inductively coupled plasma-isotope dilution- mass spectrometry	ICP-IDMS
Inductively coupled plasma-mass spectrometry	ICP-MS
ICP-MS with octopole collision cell	ICP-MS
Inductively coupled plasma-mass- Optical emission spectrometry	ICP-OES
Inductively coupled plasma-time of flight- mass spectrometry	ICP-TOF-MS
Proton (particle) induced X-ray emission spectrometry	PIXE
Potentiometric stripping analysis	PSA
Solvent extraction-Gas Chromatography - Electron Capture Detection	SE-GC/ECD
Wavelength Dispersive X-ray Fluorescence - without internal standard	WDXRF
Advanced Mercury Analyzer "Leco"	DMA
ASE Extraction/GC/MSD (Isotope Dilution)	Other

Table 11 Number of IMEP-21 participants reported results for metals per analytical technique

Analytical techniques	Cd	Cr	Cu	Ni	Pb	Zn
AAS & ETAAS & GF-AAS	44	36	32	37	38	32
F-AAS	31	27	37	31	35	41
ICP-AES & ICP-OES	84	95	92	88	85	87
ICP-MS	22	20	18	21	21	19
Other	9	9	12	7	7	12

Table 12 Number of IMEP-21 participants reported results for Hg per analytical technique

Analytical techniques	Hg
AAS & DMA & FAAS & FIAAS	56
AFS & CV-AVS & FAFS	12
CV-AAS	54
ICP-MS & FIMS	17
ICP-AES & ICP-OES	11
Other	14

Table 13 Number of IMEP-21 participants reported results for PCBs per analytical technique

Analytical techniques	PCB 28	PCB 52	PCB 101	PCB 118	PCB 153	PCB 180	PCB Sum
GC-ECD & GLC-ECD & SE-GC-ECD	49	48	52	49	53	52	40
GC-MS & HR-GC-MS	45	50	51	48	51	49	42
Other	1	2	0	1	0	2	7

Table 14 Number of IMEP-21 participants reported results for PAHs per analytical technique

Analytical techniques	Anthra-cene	Benz(a)-anthrazene	Benz(a)-pyrene	Benz(b+k)-fluorantene	Benzo(ghi)-perylene	Fluoran-thene
GC-MS & HR-GC-MS & HPLC-MS	61	62	63	62	61	62
HPLC-FLU	29	30	34	34	34	34
HPLC-UV & HPLC-DAD	5	5	8	8	8	6
Other	4	4	5	6	5	5

Analytical techniques	Fluorene	Indeno-(1,2,3-cd)-pyrene	Naph-talene	Phenan-trene	Pyrene	PAH Sum
GC-MS & HR-GC-MS & HPLC-MS	62	60	61	62	63	61
HPLC-FLU	29	32	27	29	28	26
HPLC-UV & HPLC-DAD	4	9	5	5	5	5
Other	4	7	5	4	6	9

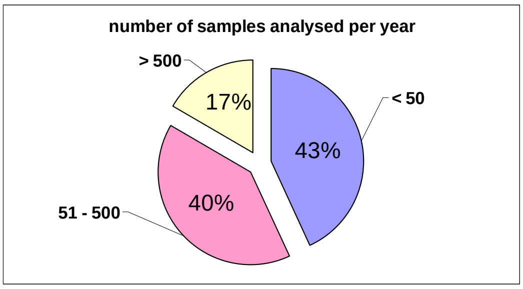
Evaluation of the questionnaire replies

204 laboratories completed the IMEP-21 questionnaire. Several conclusions can be drawn from this information provided via the questionnaires in a confidential way without quoting the identity of the laboratories. The evaluation of some of the replies is given in the following paragraphs. The graphical displays which present the participants' results sorted according to the criterion evaluated, can be found in Annex 1.

Number of samples analysed per year

Participants were asked to indicate the number of sewage sludge samples they analyse per year. Some 57% of the laboratories participating in IMEP-21 can be considered dealing on routine basis with the analysis of contaminants in sludge. They analyse yearly more than 50 samples. Only 17% analyse more than 500 samples per year. 43% of the participants analyse less than 50 samples per year.

Figure 2: Number of samples analysed per year



IMEP CTS analysed under routine conditions?

Participating laboratories in IMEP-21 were asked to analyse the Certified Test Sample (CTS) applying their routine procedures.

More than 90% treated the IMEP-21 sewage sludge sample for the analysis of the inorganic analytes according to their routine analytical procedure for this sample type. For the PCB analysis 88% replied that they applied their routinely used method. For PAH analysis all participants confirmed to apply their routinely used method. IMEP-21 results reflect therefore the actual measurement capability for the analytes under investigation for the given concentration range.

Digestion, separation or pre-concentration

A detailed summary of the sample treatment procedures applied per participant is given in Table 7 and Table 8 in Annex 2 of the report.

Official method

More than 60% of the laboratories used an official analytical method in their laboratories for the analysis of the metals in the sewage sludge, for the organic analytes only slightly more than 50% applied an official method. The most frequent standards and number of replies are listed in Table 1 – Table 3 in Annex 2 of this report. In addition Figure 65 – Figure 74 in Annex 1 display the measurement results according to an official method used for selected analytes.

Extraction clean up

The reply to the question whether an extraction clean-up was performed for the PCBs and PAHs analysis is displayed in Figure 3. 84% of the participants performed a clean-up prior to PCB analysis, but only 56% prior to PAH analysis.

Figure 3 Extraction clean-up for PCB analysis

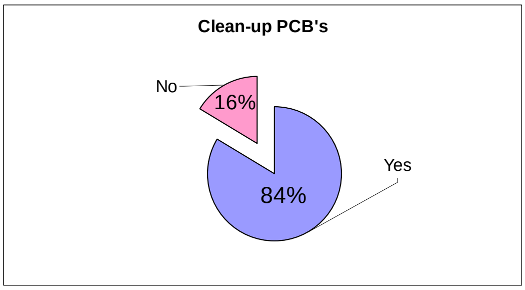
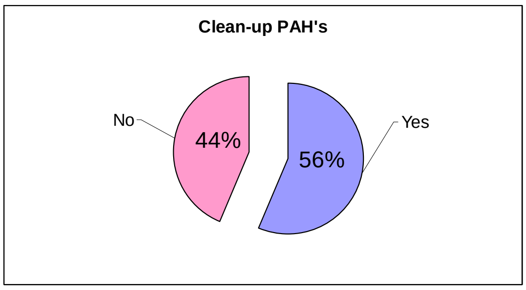


Figure 4 Extraction clean-up for PAH analysis



Certified Reference Material in use in laboratory

45% of the IMEP-21 participants have a sewage sludge certified reference material (CRM) at their disposal. From this population, 88 laboratories (96%) indicated the use of these CRMs for procedure validation purposes and 26 (28%) for instrument calibration. Multiple selections showed that 24 laboratories indicated the use of CRMs for both purposes.

An overview of all reported CRMs is given in Annex 2 in Table 4. The graphical presentation of the reported results in view of this criterion for selected analytes is given in Figure 62 – Figure 64 in Annex 1.

Participation in interlaboratory comparisons

169 Laboratories (82.8%) participated already in other interlaboratory comparisons.

71 of them were EA nominated, 8 were MEDA laboratories and 8 were laboratories nominated by the EuroGeoSurvey. The PT schemes and providers are listed in Table 5 in Annex 2.

Quality Management System

176 laboratories (86.3%) replied that they are working according to the recommendations of a quality management system. Which quality management system is followed is summarised in Table 15.

Table 15: The number of IMEP-21 participating laboratories in relation to the quality management system in use. Multiple answers were possible.

Quality management system in use			
EN 45000 series	**ISO 9000 series**	**ISO 17025**	**Other**
5	28	168	6

Accredited and/or Authorised

Results of laboratories that replied positive to the question if they were accredited or authorized (e.g. by law or regulatory authority) for this kind of analysis in a sewage sludge matrix are presented in Figure 53 – Figure 61 in Annex 1.

Reporting and calculating uncertainty

IMEP-21 participants were asked if they are familiar with the Guide To The Expression Of Uncertainty In Measurement (GUM) issued by the International Organisation for Standardisation (ISO, 1993) and/or sectoral guidance documents derived from this, such as the EURACHEM/CITAC Guide Quantifying Uncertainty in Analytical Measurement (2000) [31].

83% of the participants stated that they were familiar with the above mentioned guides. 66% also implemented these guidelines to calculate the uncertainty on the reported results. The different approaches to evaluate the uncertainty of those laboratories that did not use the above mentioned guidelines are given in Table 6 in Annex 2. 41% of the participating laboratories replied that they report uncertainties on chemical measurements to their customers.

Motivation for participation in IMEP-21

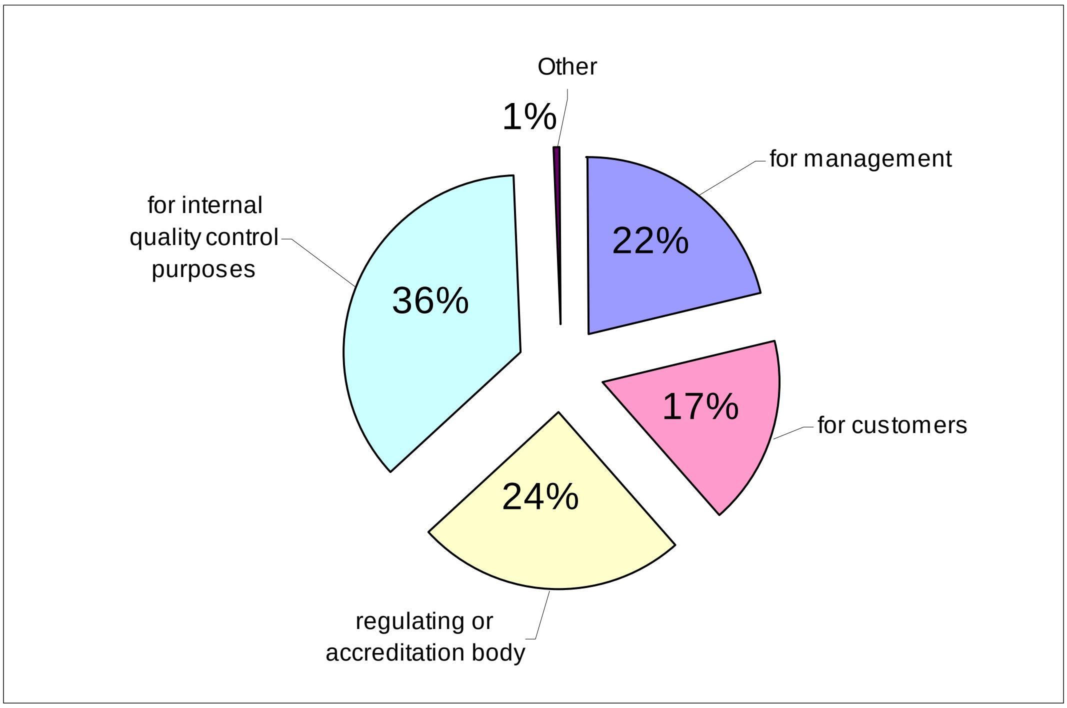
IMEP-21 participants were asked to indicate the most appropriate reply to the question “Was your participation to this IMEP comparison used to demonstrate your measurement capability to …”. The percentage of replies to the various choice possibilities are given in Figure 5.

Internal quality control purposes were the motivation for participation of 36% of the IMEP-21 participants. Demonstration of measurement capability to other parties such as their management was the motivation for 21%, such as their customers for 17% or such as their regulating or accreditation bodies for 24%.

Moisture content determination

The results for the measurements carried out by the reference laboratories on the sewage sludge are listed in the IMEP-21 certification report [21]. Via the IMEP-21 questionnaire the IMEP-21 participants provided also more detailed information concerning the determination of the moisture content of the sewage sludge. A graphical presentation of the moisture content measured by the IMEP-21 participants together with the moisture content as measured by IRMM is included in Figure 78 in Annex 1 of this report. IRMM emphasises that the IRMM result for the moisture content in Figure 78 does NOT represent a certified reference value. This additional information is merely out of interest provided to the IMEP-21 participants.

Figure 5: Motivation for participation in IMEP-21



Additional indicative values

After investigation of the chromatograms interferences in the determination of PCB_138, PCB_170 and chrysene were observed in this material. Therefore it was decided not to include these congeners in the certification campaign for IMEP-21. Nevertheless also these 3 analytes were measured by the reference laboratories in the scope of the certification of the material. Some of the IMEP-21 participants informed the IRMM ILC coordinator that they also measured these congeners in the sewage sludge material. Therefore also indicative values for the total amount content for PCB_138 , PCB_170, and chrysene are included in this report in Table 16. It has to be emphasised that these values are not IMEP-21 certified reference values, but given to interested participants as additional information.

Table 16: indicative values for PCB_170, PCB_138 and chrysene

analyte	indicative value in ng·g^{-1}	expanded uncertainty in ng·g^{-1} U, k=2
PCB_170	63.4	5.2
PCB_138	144.6	7.0
chrysene	371	46

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List of abbreviations

AES	Atomic emission specrometry
BAM	Bundesanstalt für Materialforschung und –prüfung (Berlin, Germany)
BOKU	Universität für Bodenkultur Wien
BIPM	Bureau International des Poids et Mesures (Paris, France)
CARDS	Community Assistance for Reconstruction, Development and Stabilisation
CCQM	Comité Consultatif pour la Quantité de Matière
CEN	European Committee for Standardisation
CIPM	International Committee for Weights and Measure
CITAC	Co-operation for International Traceability in Analytical Chemistry
CRMs	Certified Reference Materials
CTS	Certified Test Sample
EA	European Co-operation for Accreditation
EC	European Commission
EU	European Union
EURACHEM	A focus for Analytical Chemistry in Europe
EuroGeoSurveys	The Association of the European Geological Surveys
GC-MS	Gas Chromatography Mass Spectrometry
GUM	Guide for expression for Uncertainty in Measurement
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
IDMS	Isotope Dilution Mass Spectrometry
IES	Institute for Environment and Sustainability
IMEP®	International Measurement Evaluation Programme
ILC	Interlaboratory Comparison
IRMM	Institute for Reference Materials and Measurements
ISO	International Organisation for Standardisation
JRC	Joint Research Centre
MRA	Mutual Recognition Agreement
NAA	Neutron Activation Analysis
NAB	National Accreditation Body
PT	Proficiency Testing Scheme
RC	Regional Co-ordinator
SCK-CEN	Studiecentrum voor Kernenergie- Centre d’étude de l’énergie nucléaire
SS-ZAAS	Solid Sample Zeemann Atomic Absorption Spectrometry
TIMS	Thermal Ionisation Mass Spectrometry
VITO	Flemish Institute for technological research

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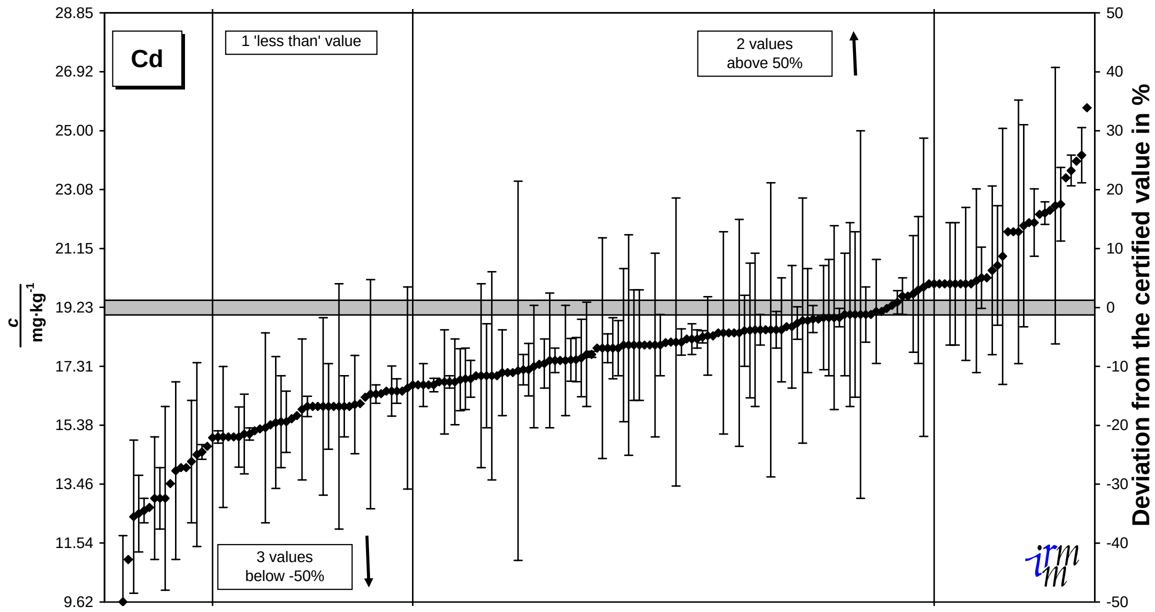
Annex 1 – Participants' results – General Graphs

Figure 1

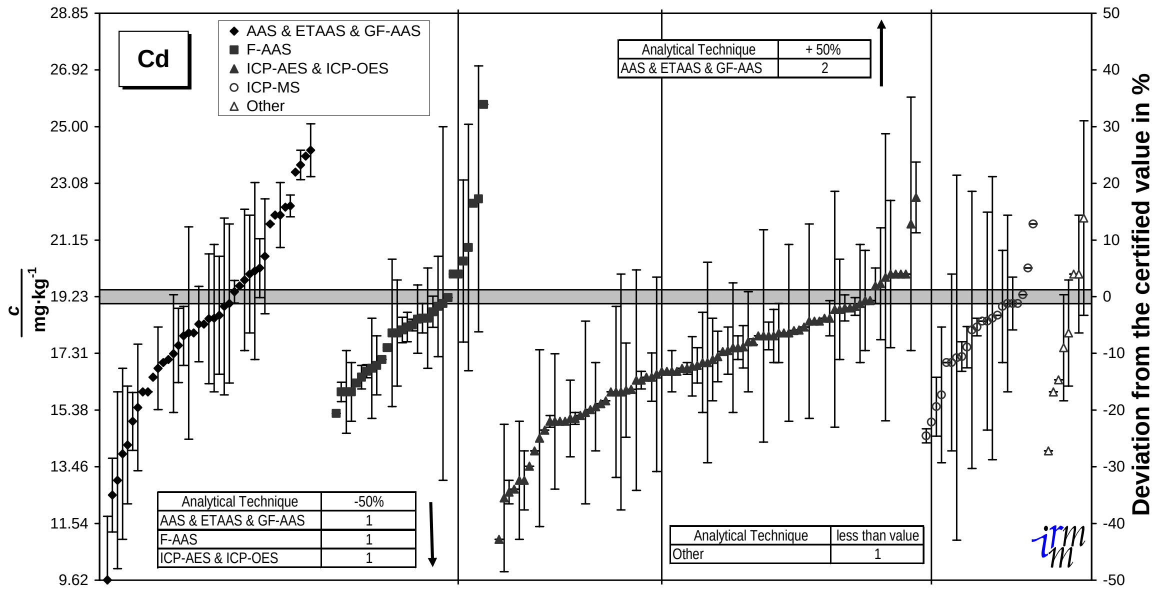
IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for Cd : 19.23 ± 0.24 mg·kg^{-1} [*U=k·u$_c$(k=2)*]



Results for Cd from all participants

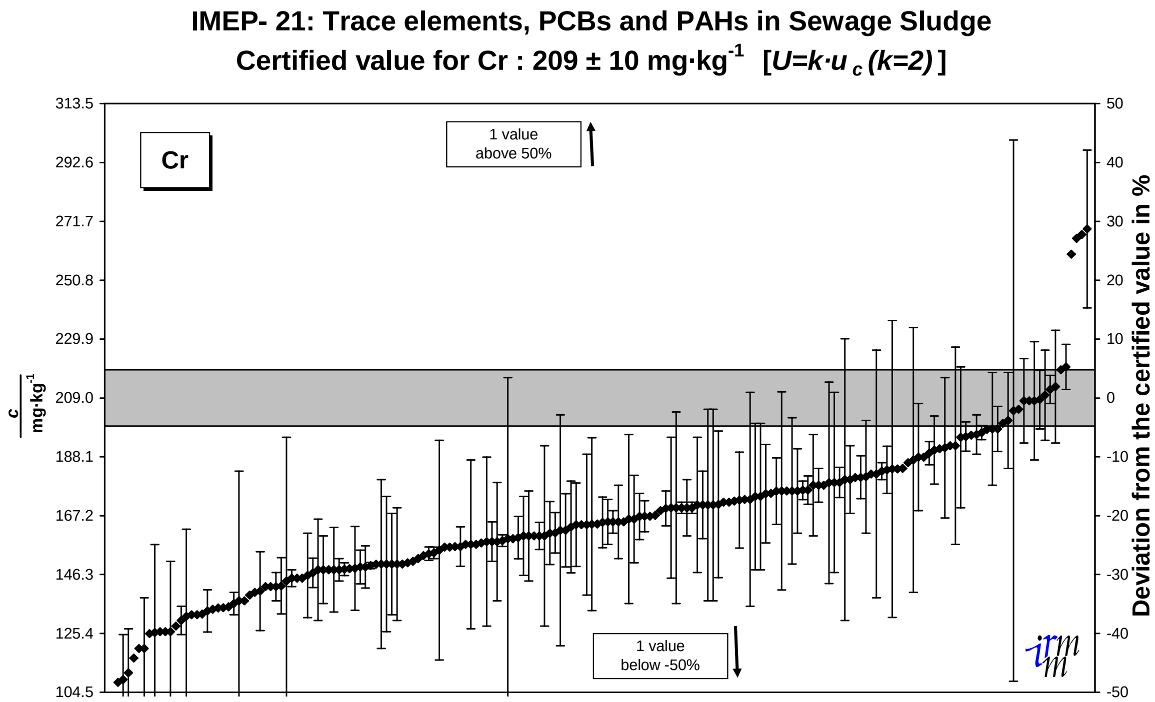
Figure 2

IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for Cd : 19.23 ± 0.24 mg·kg^{-1} [*U=k·u$_c$(k=2)*]



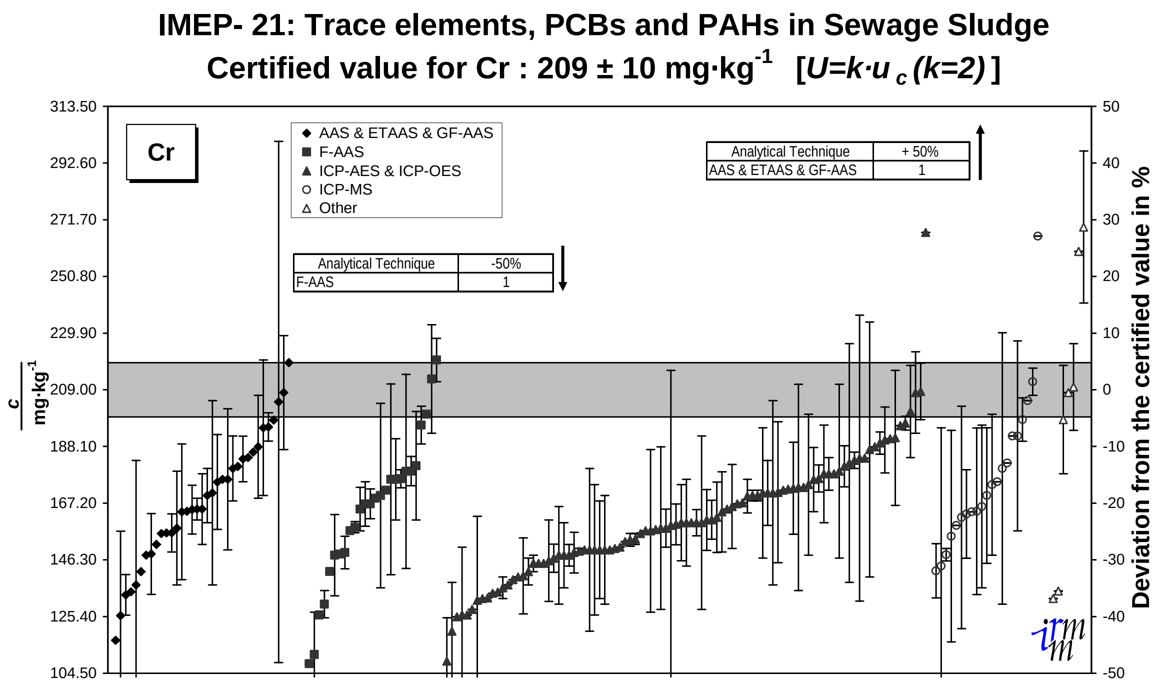
Results from all participants according to analytical techniques used

Figure 3



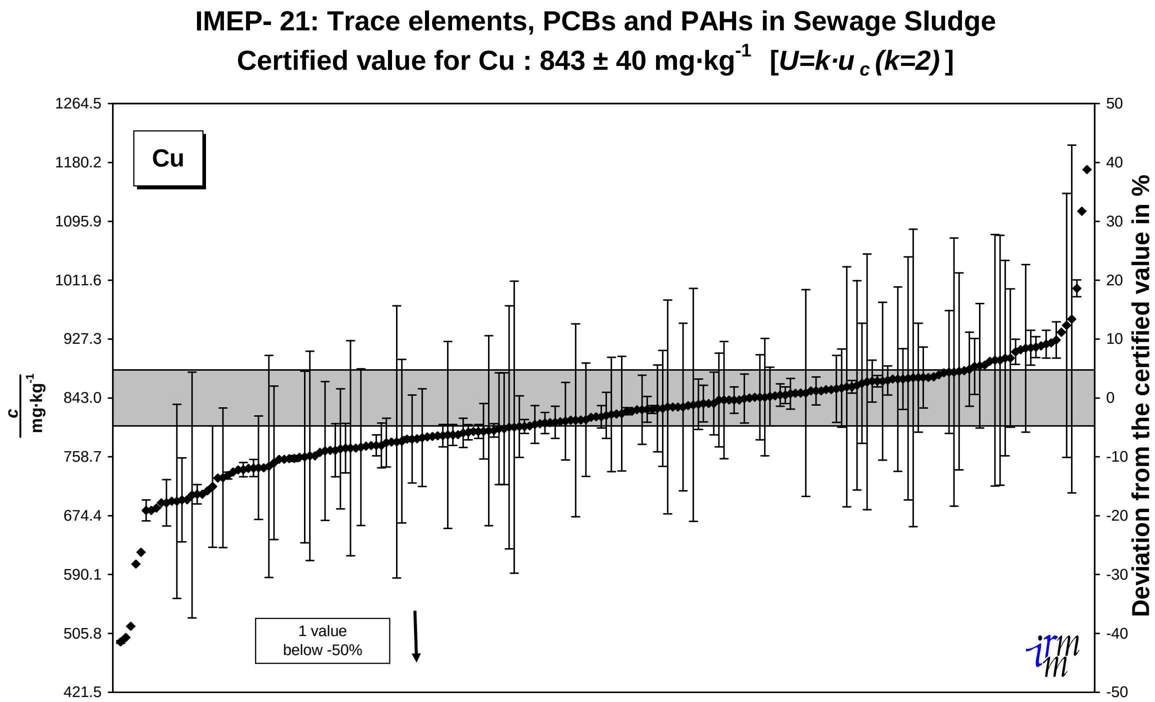
Results for Cr from all participants

Figure 4



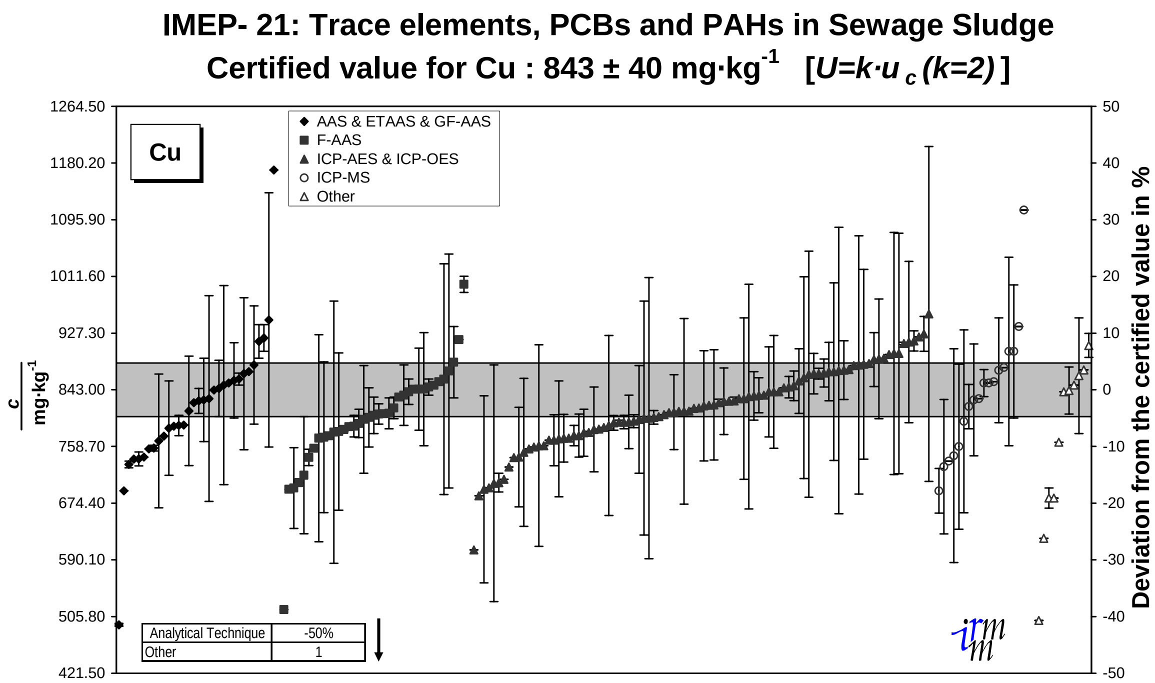
Results from all participants according to analytical techniques used

Figure 5



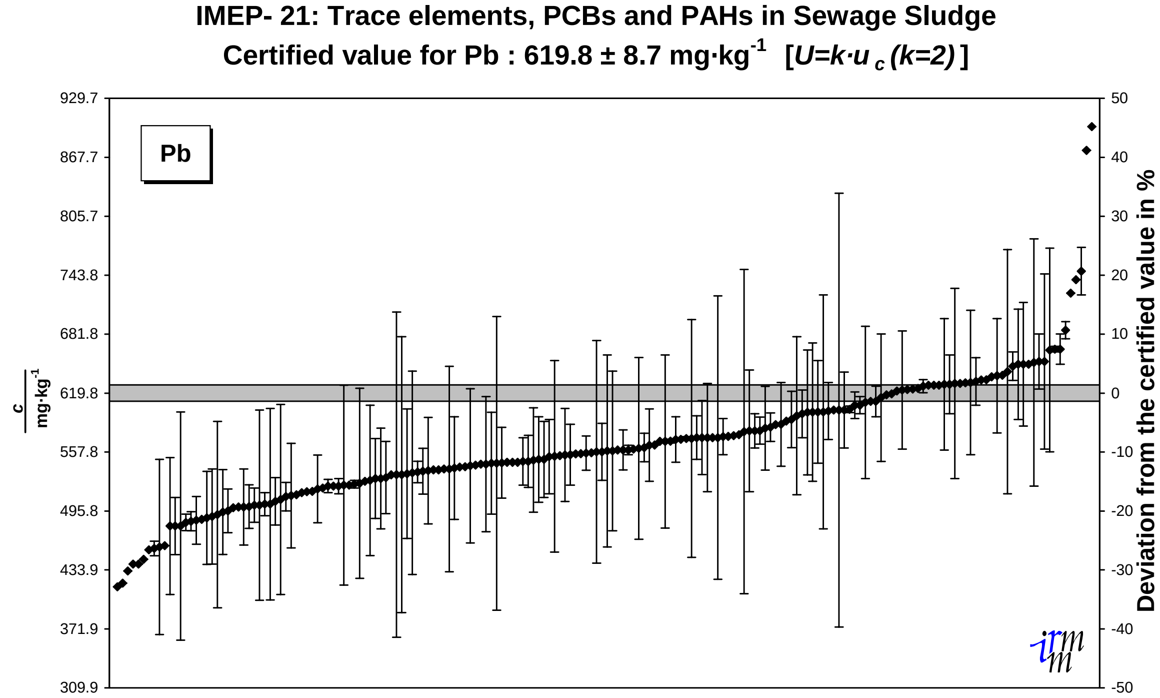
Results for Cu from all participants

Figure 6



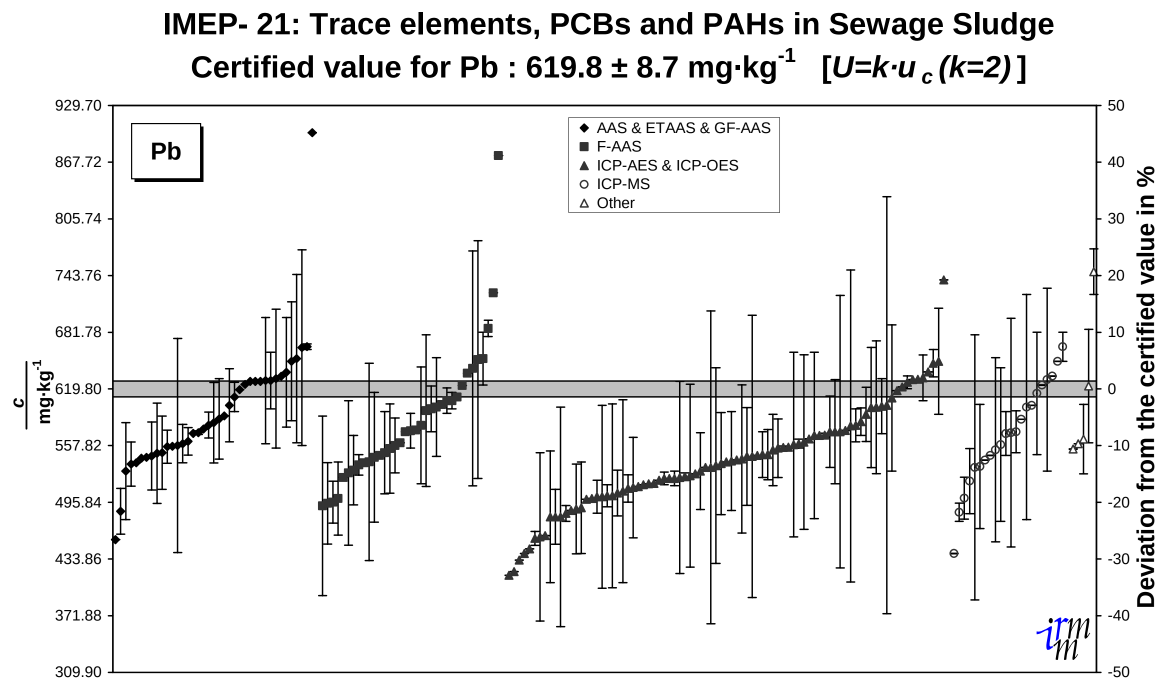
Results from all participants according to analytical techniques used

Figure 7



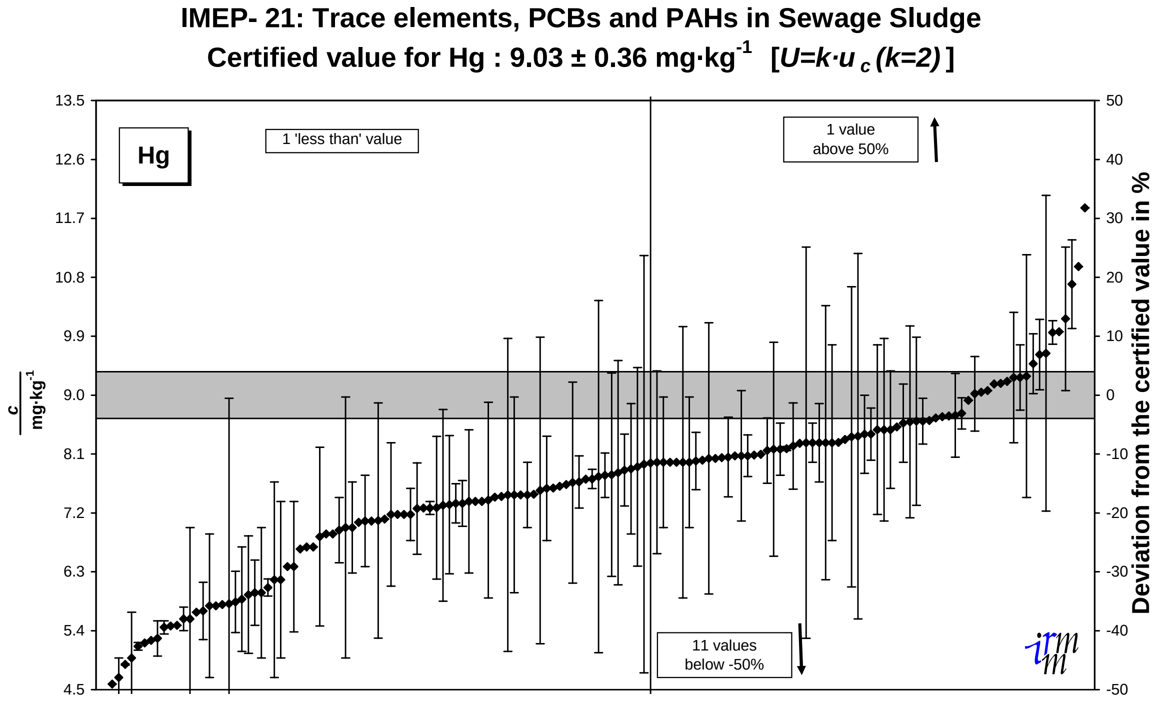
Results for Pb from all participants

Figure 8



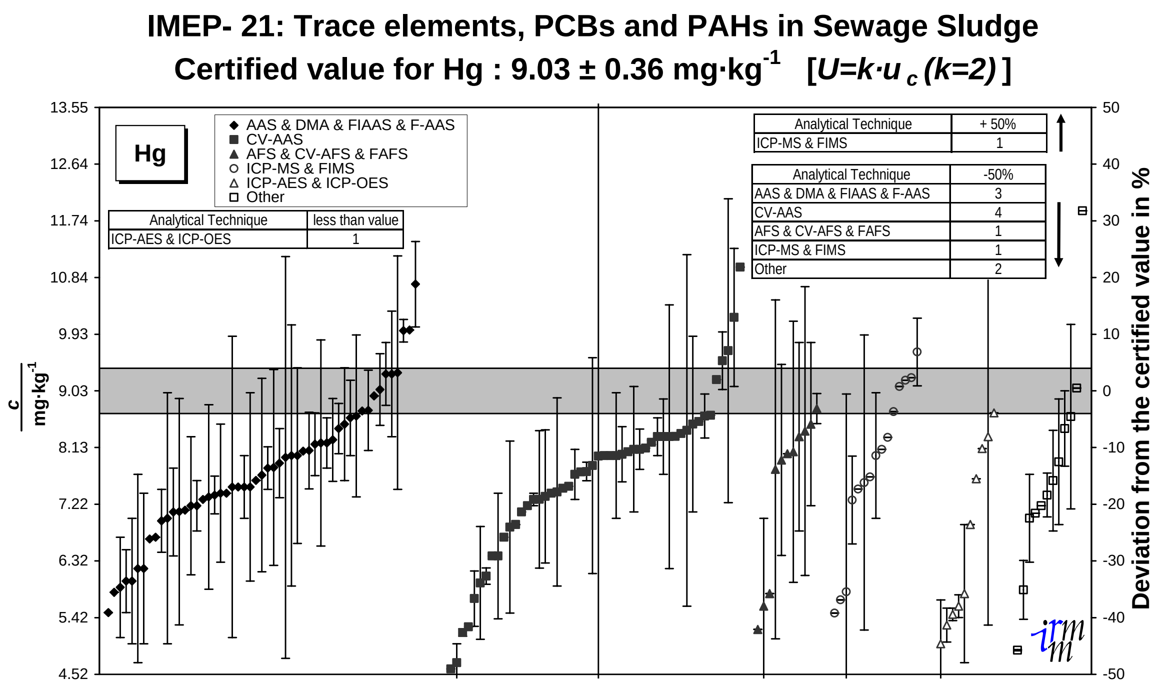
Results from all participants according to analytical techniques used

Figure 9



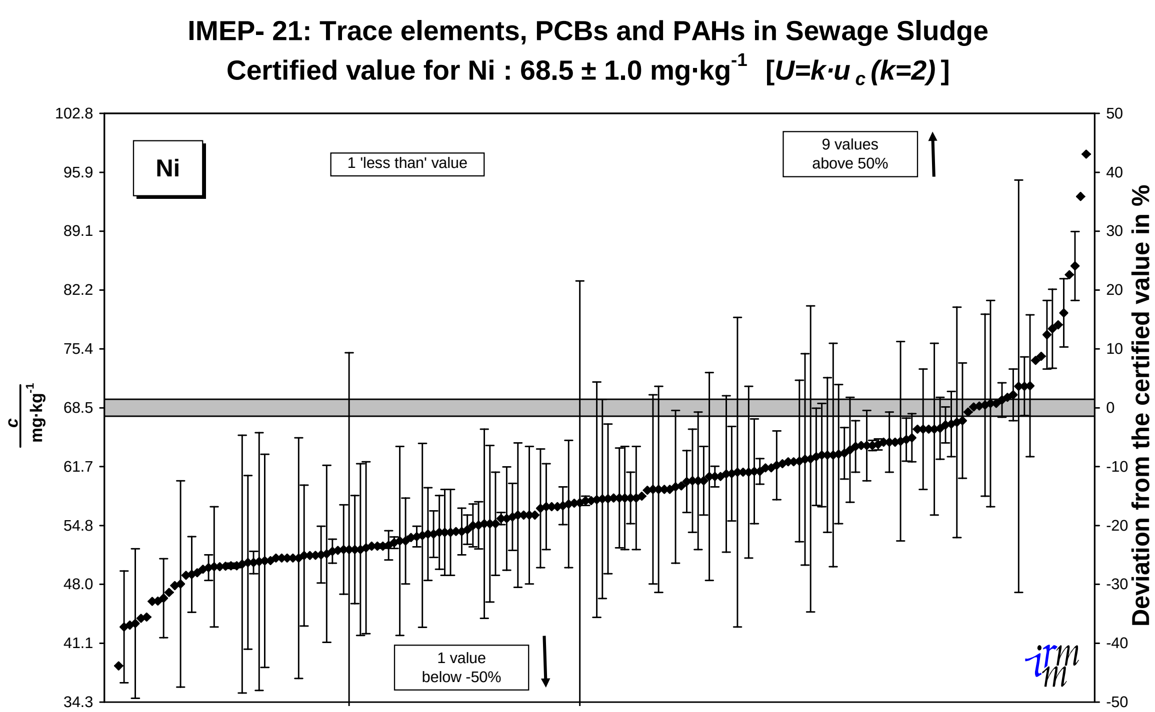
Results for Hg from all participants

Figure 10



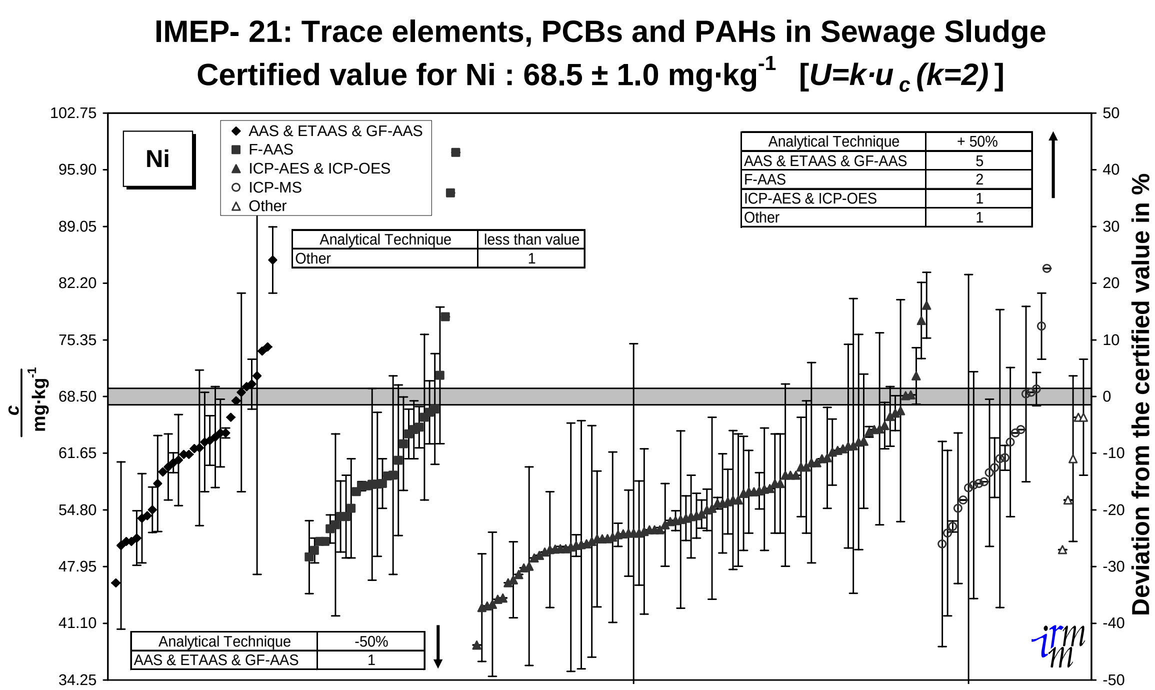
Results from all participants according to analytical techniques used

Figure 11



Results for Ni from all participants

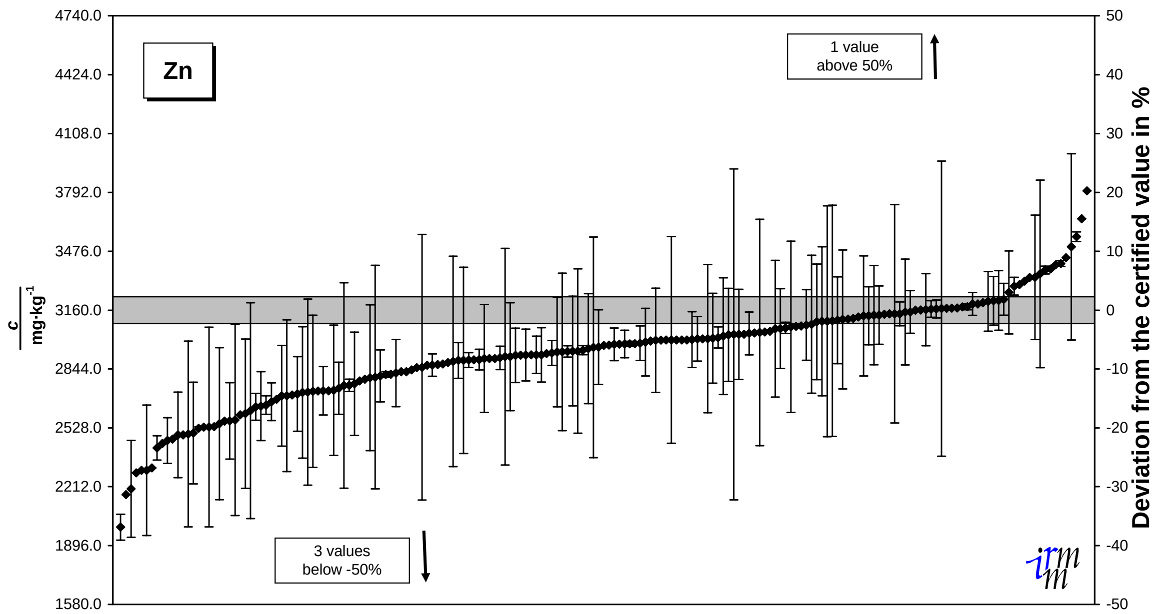
Figure 12



Results from all participants according to analytical techniques used

Figure 13

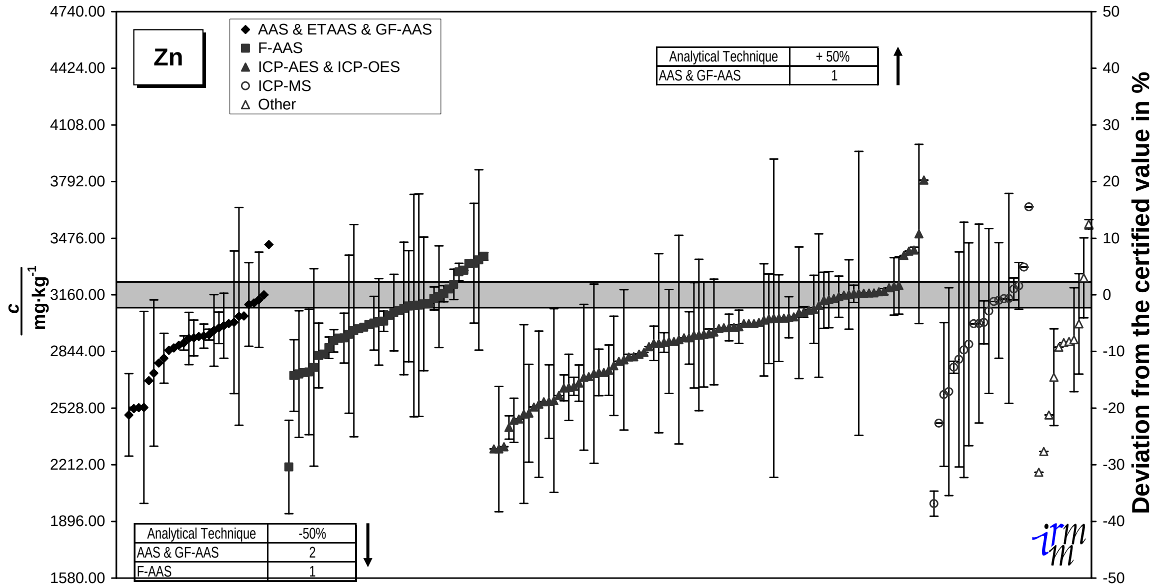
IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for Zn : 3160 ± 72 mg·kg^{-1} [$U=k \cdot u_c$ $(k=2)$]



Results for Zn from all participants

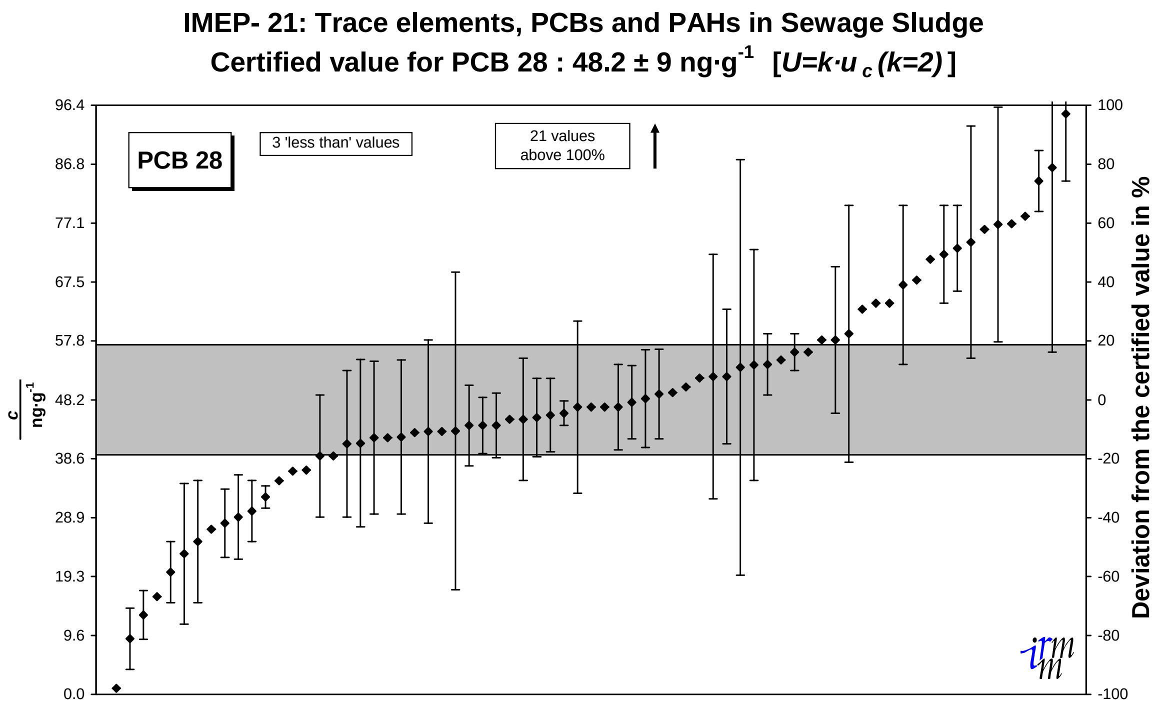
Figure 14

IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for Zn : 3160 ± 72 mg·kg^{-1} [$U=k \cdot u_c$ $(k=2)$]



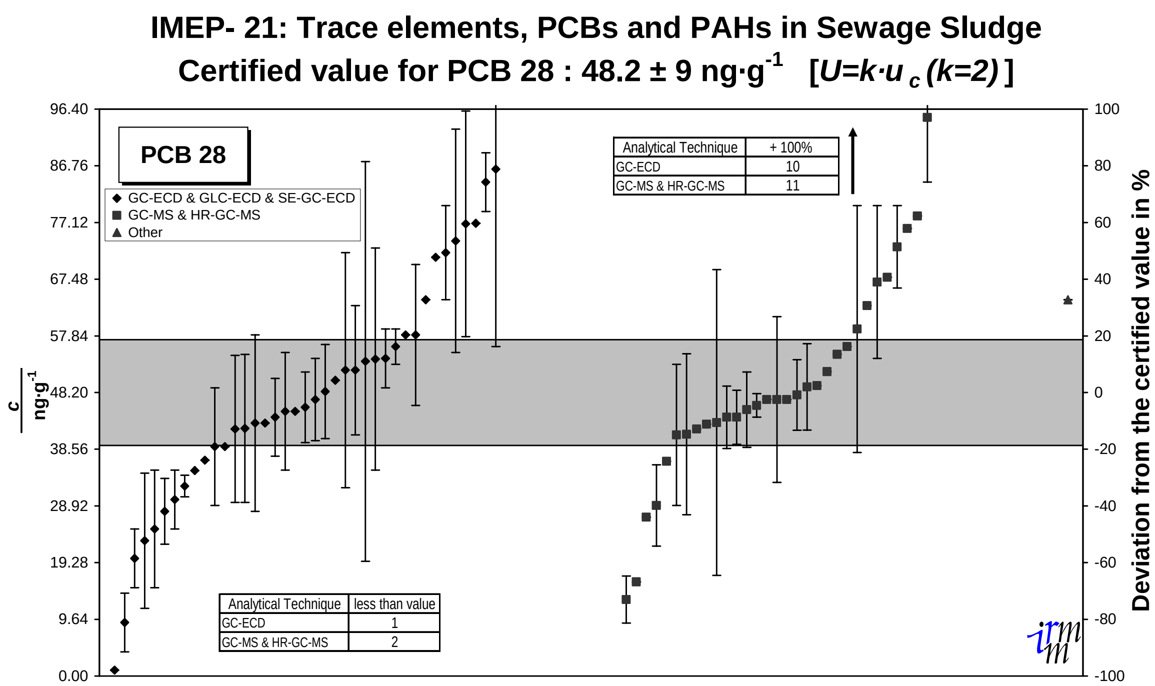
Results from all participants according to analytical techniques used

Figure 15



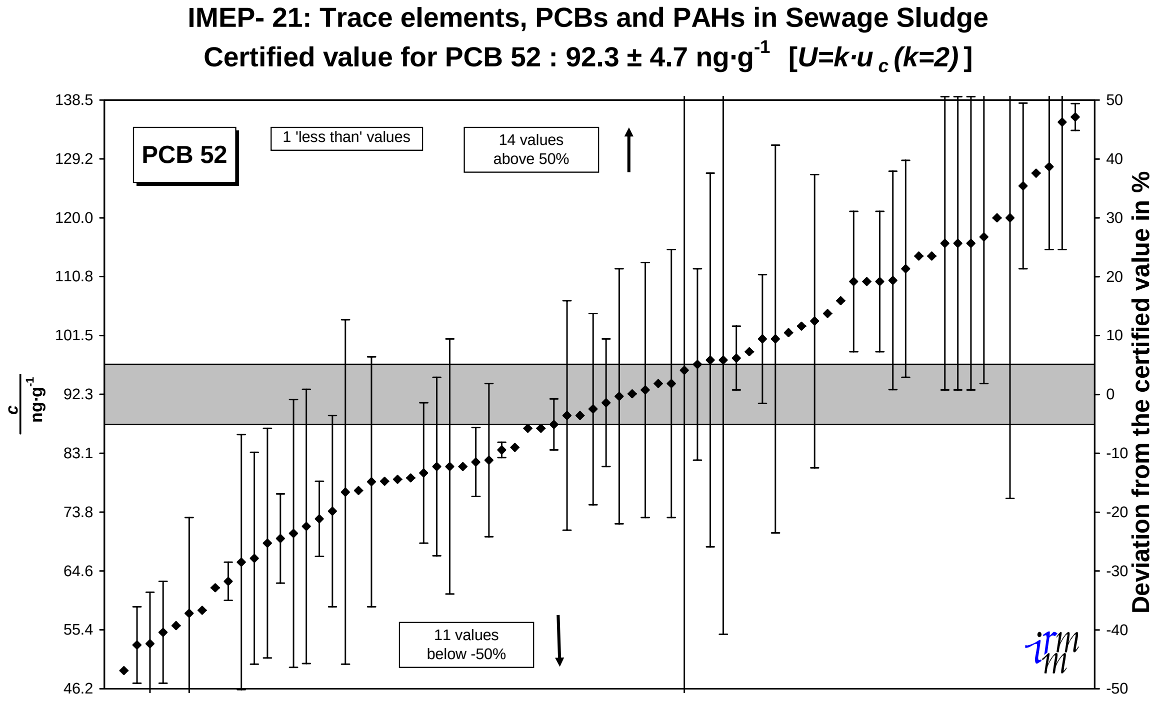
Results for PCB 28 from all participants

Figure 16



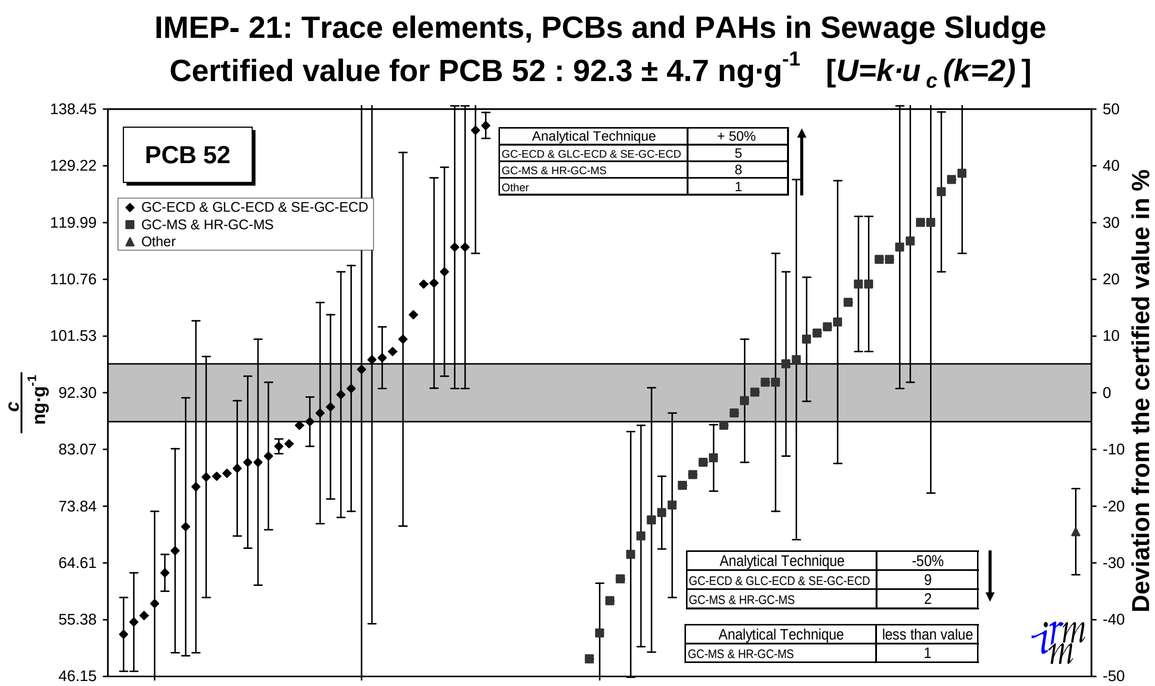
Results from all participants according to analytical techniques used

Figure 17



Results for PCB 52 from all participants

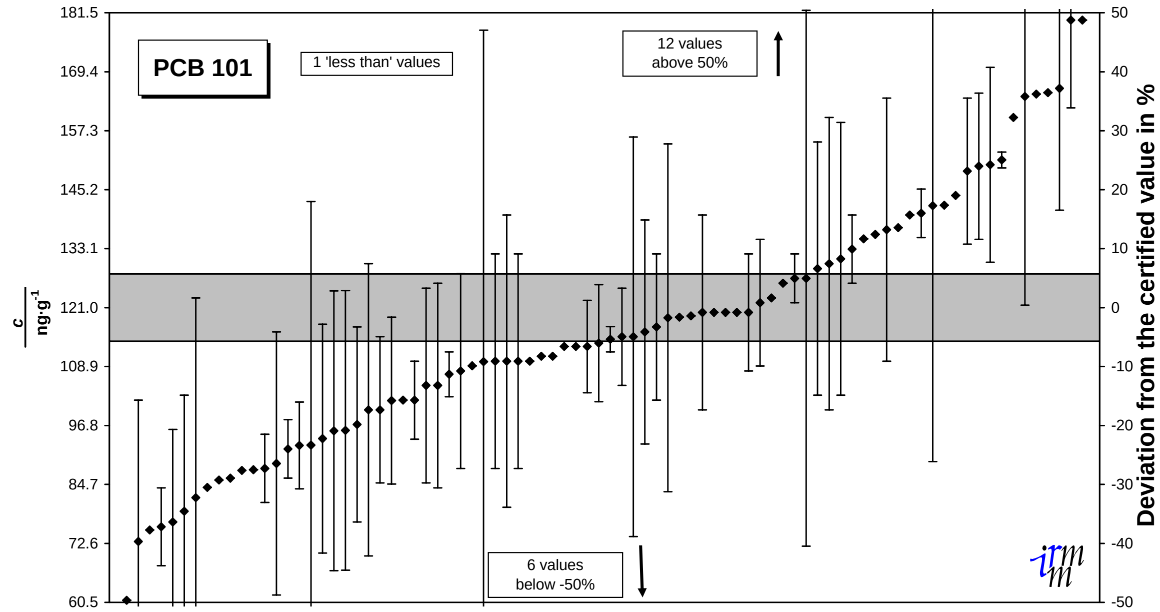
Figure 18



Results from all participants according to analytical techniques used

Figure 19

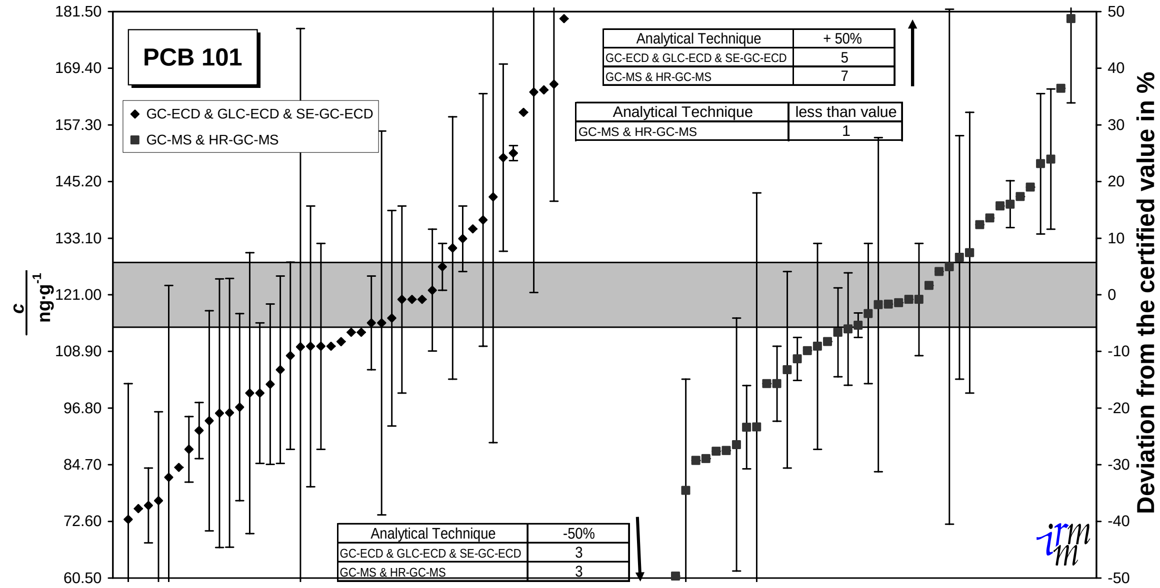
IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for PCB 101 : 121 ± 6.9 ng·g^{-1} [*U=k·u$_c$ (k=2)*]



Results for PCB 101 from all participants

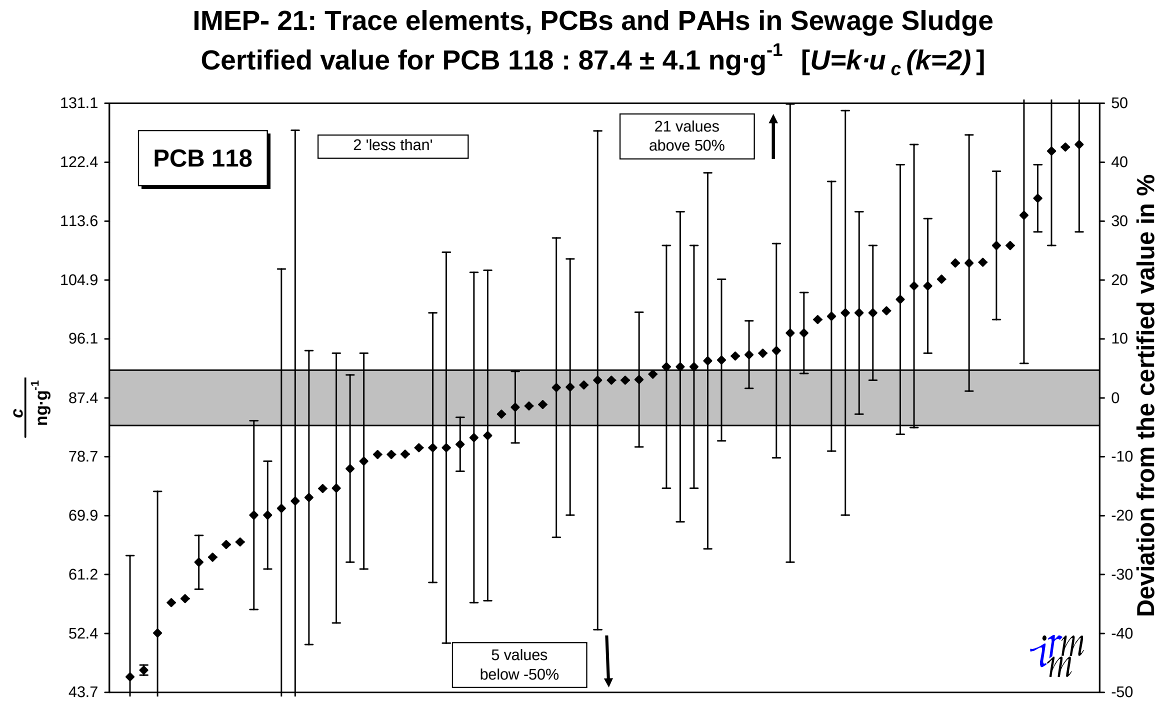
Figure 20

IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for PCB 101 : 121 ± 6.9 ng·g^{-1} [*U=k·u$_c$ (k=2)*]



Results from all participants according to analytical techniques used

Figure 21



Results for PCB 118 from all participants

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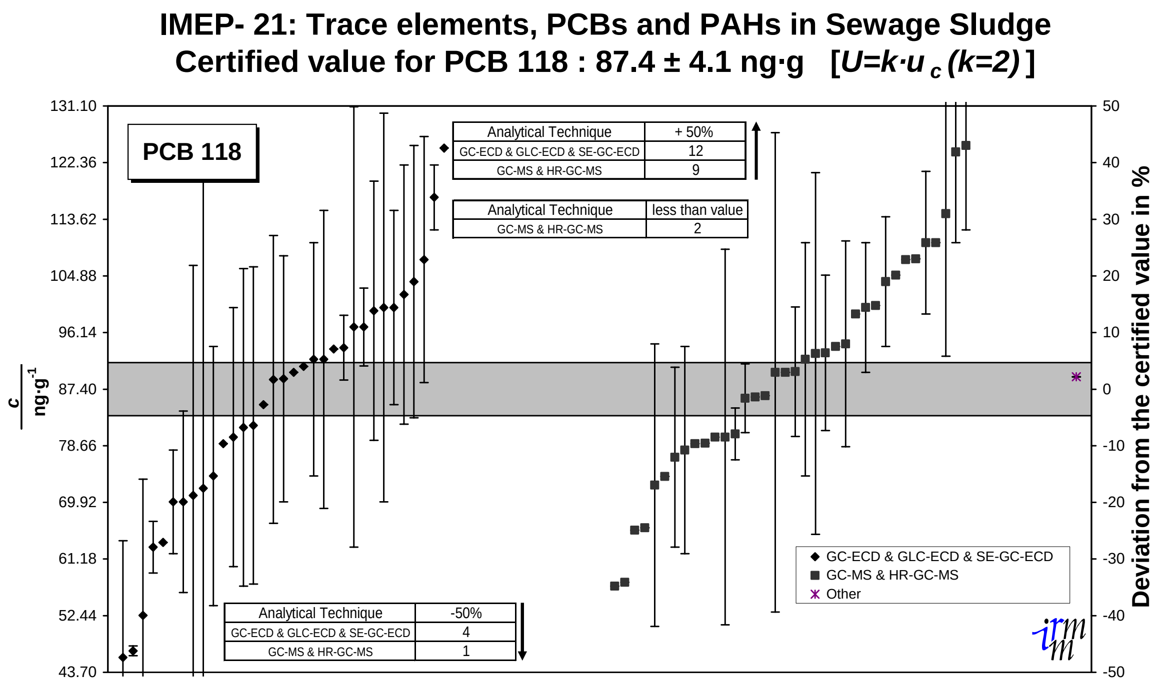
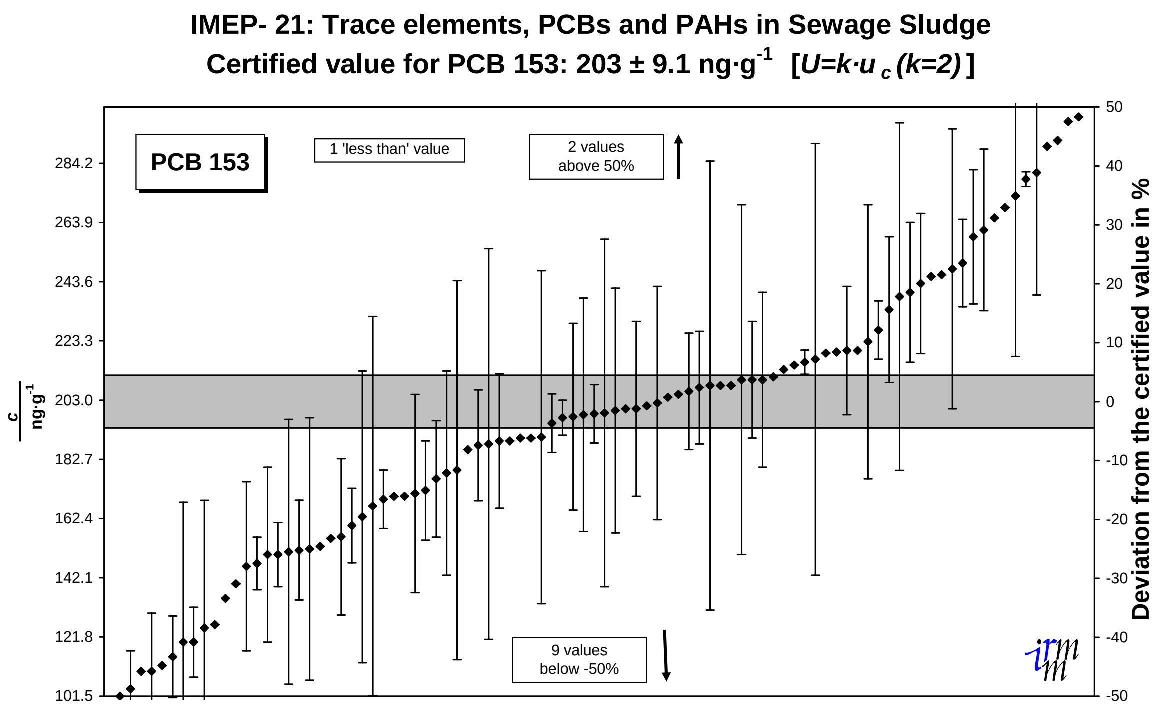


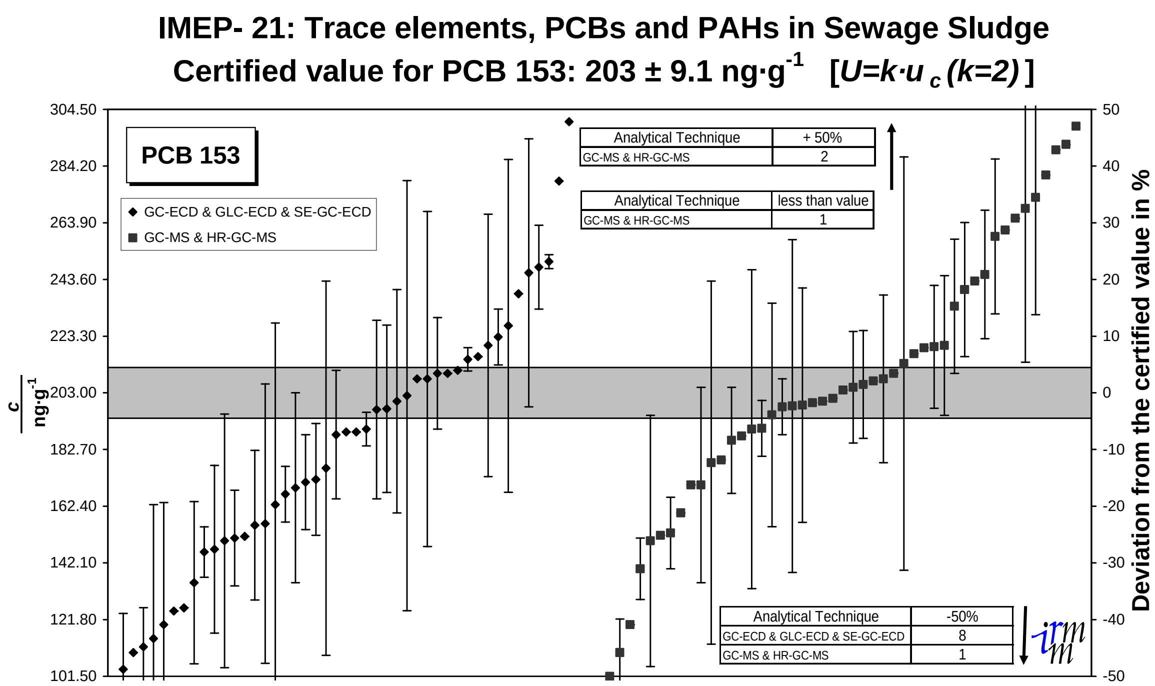
Results from all participants according to analytical techniques used

Figure 23



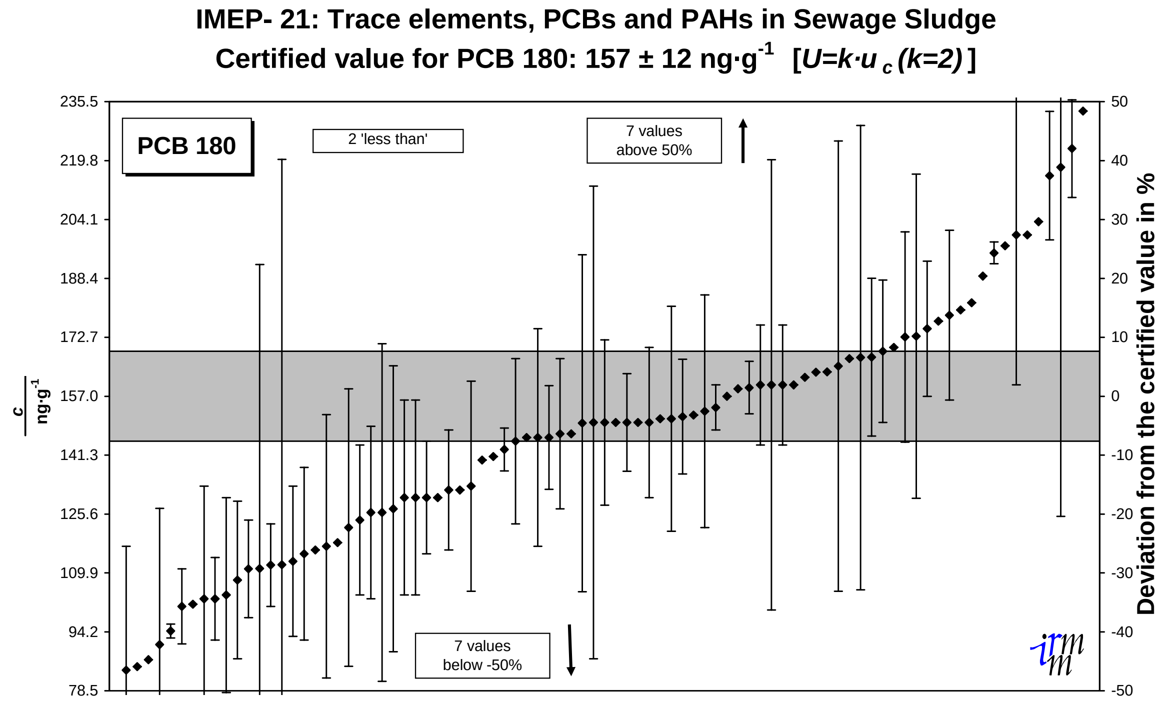
Results for PCB 153 from all participants

Figure 24



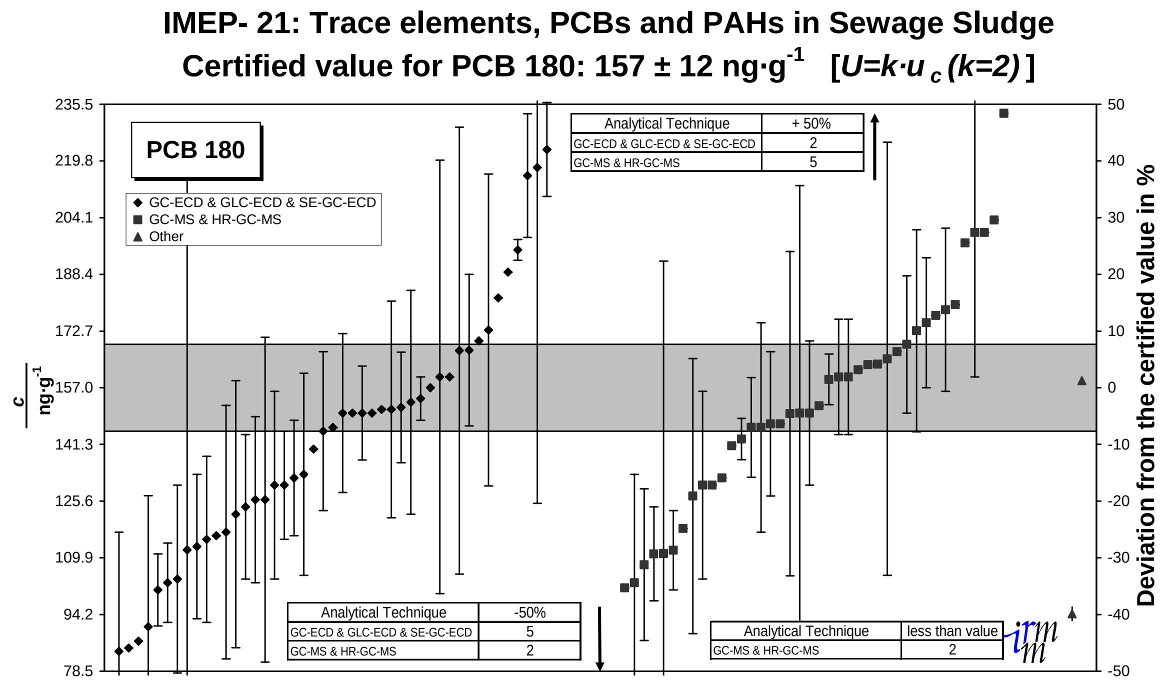
Results from all participants according to analytical techniques used

Figure 25



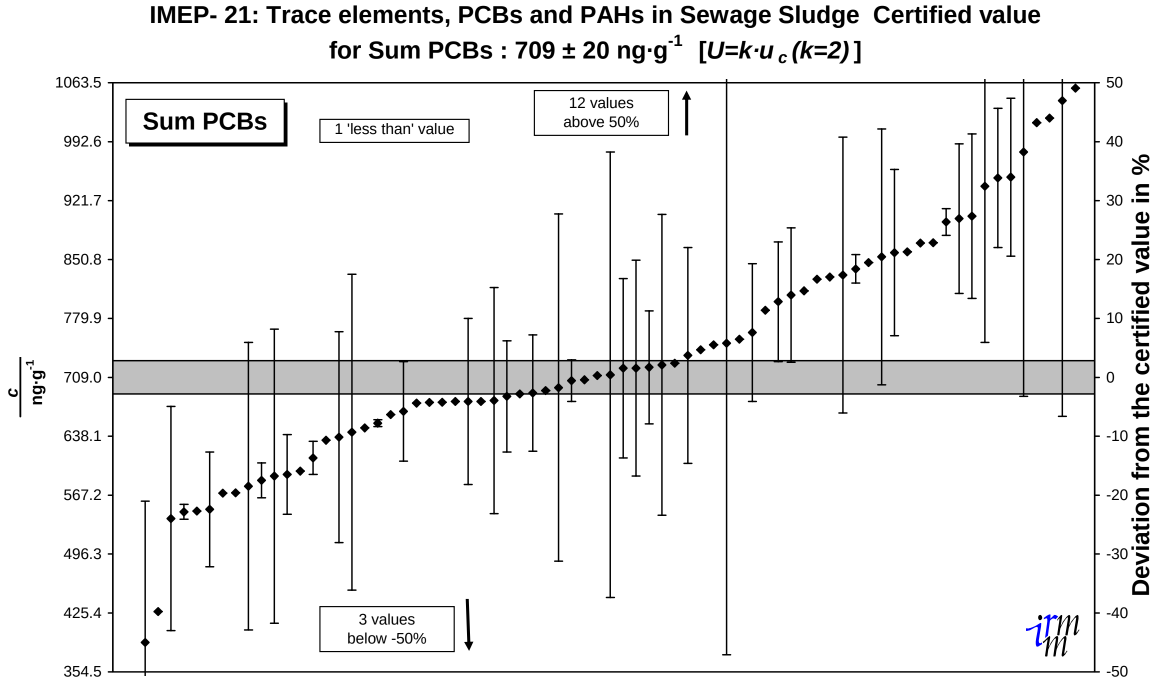
Results for PCB 180 from all participants

Figure 26



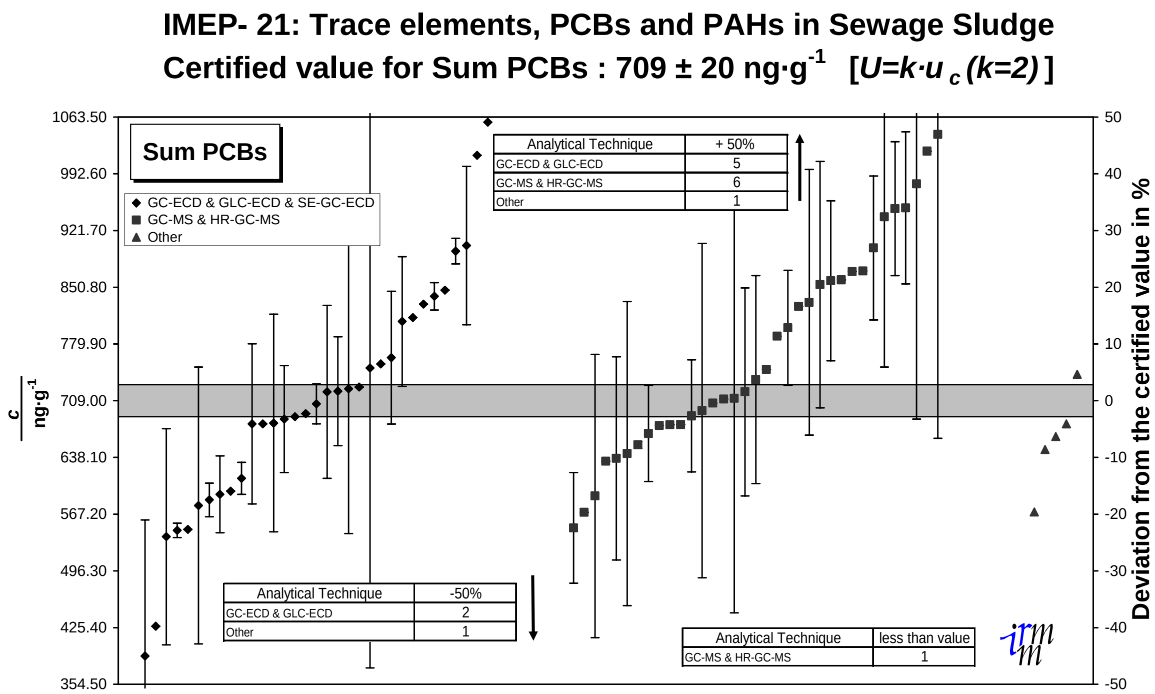
Results from all participants according to analytical techniques used

Figure 27



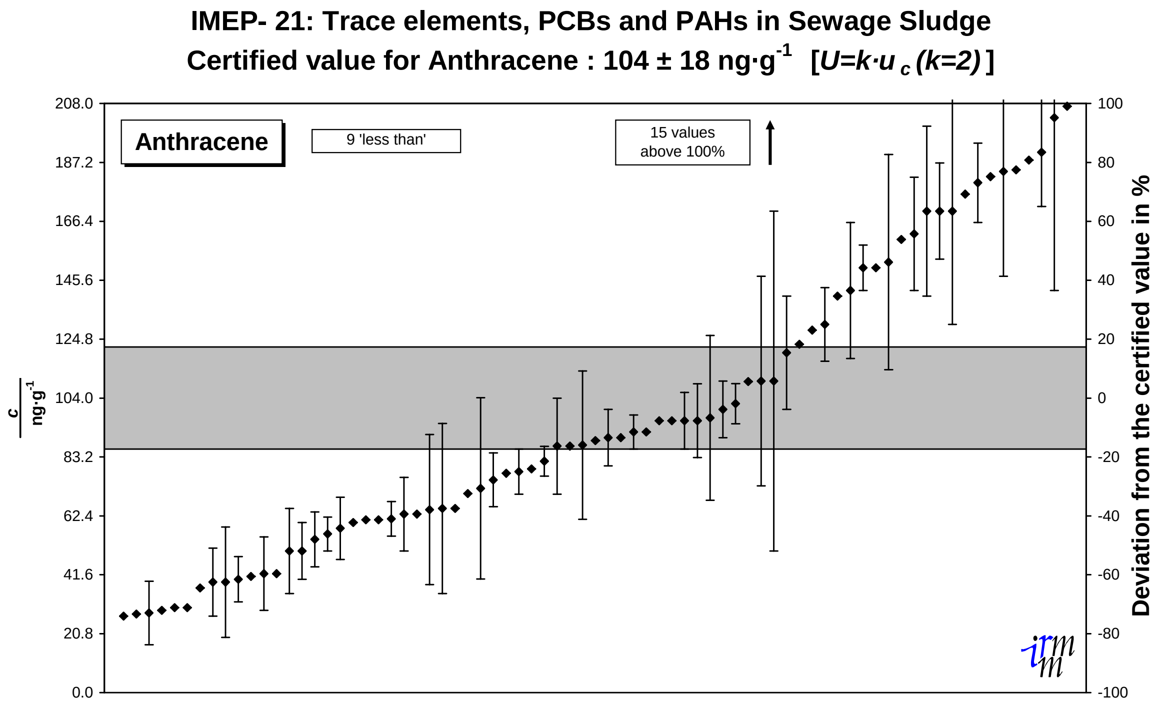
Results for Sum PCBs from all participants

Figure 28



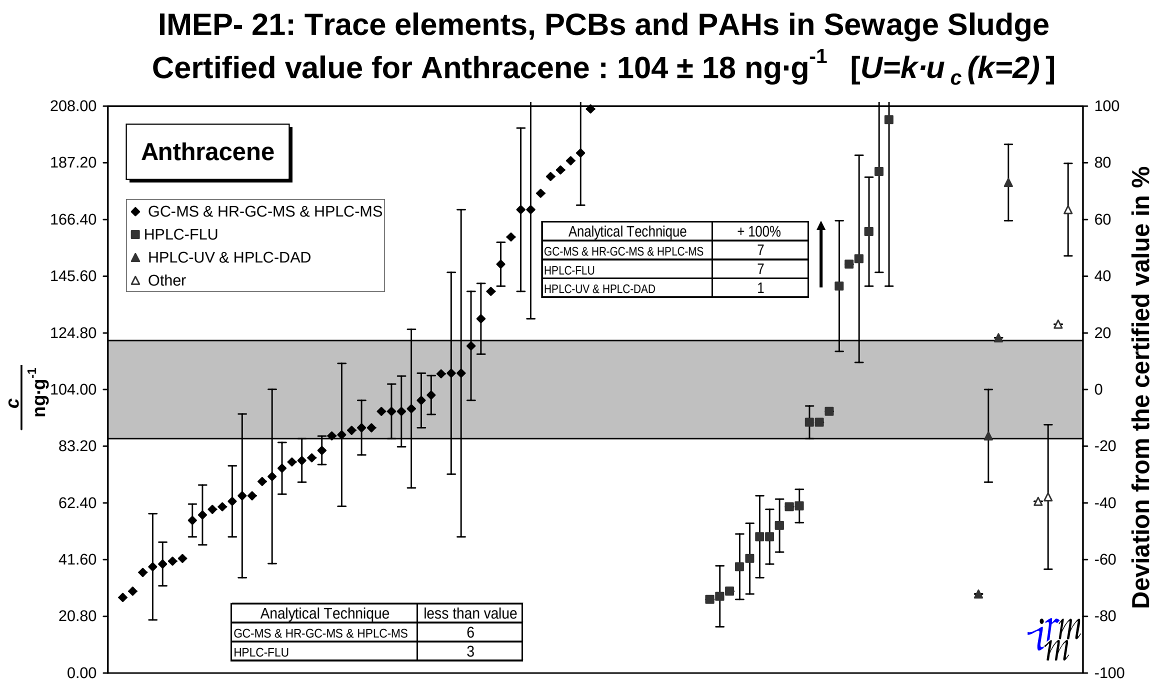
Results from all participants according to analytical techniques used

Figure 29



Results for Anthracene from all participants

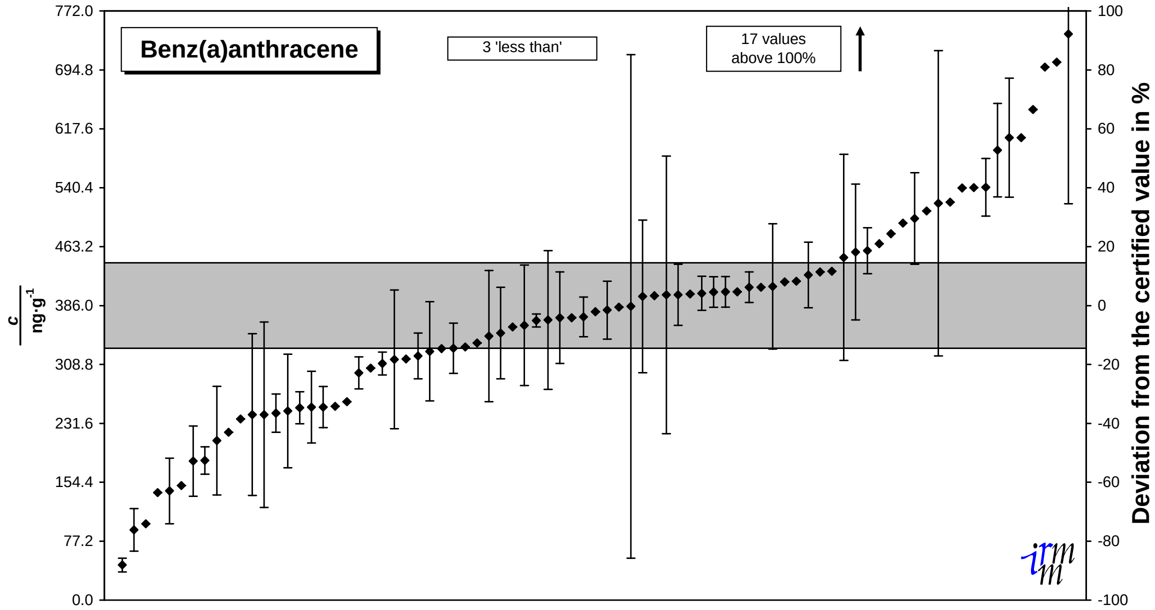
Figure 30



Results from all participants according to analytical techniques used

Figure 31

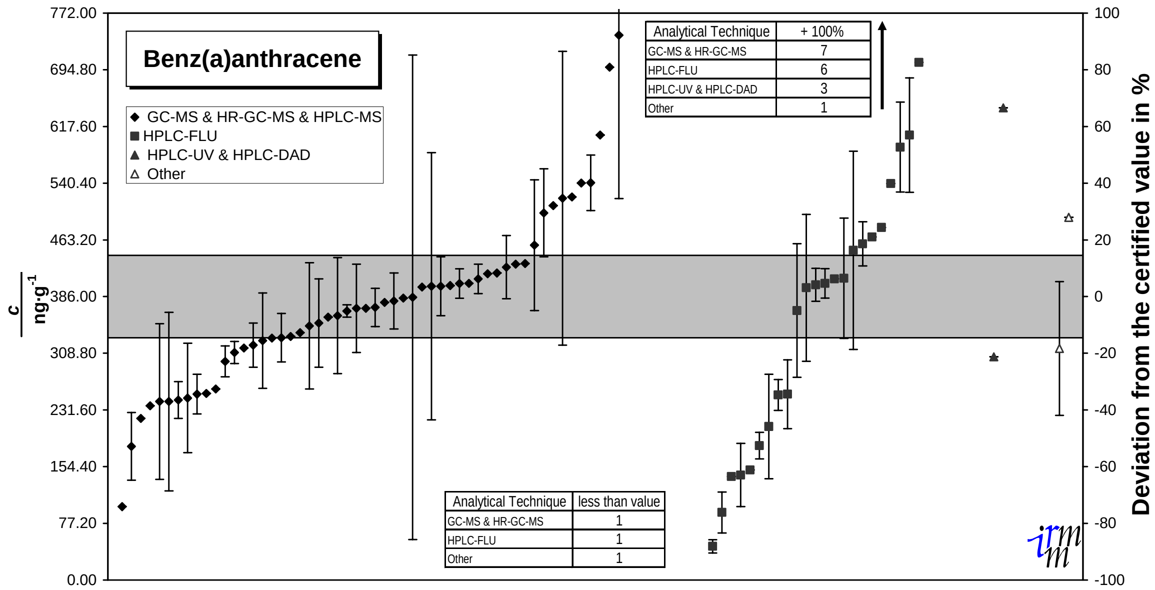
IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge Certified value for Benz(a)anthracene : 386 ± 56 ng·g^{-1} [$U=k \cdot u_c (k=2)$]



Results for Benz(a)anthracene from all participants

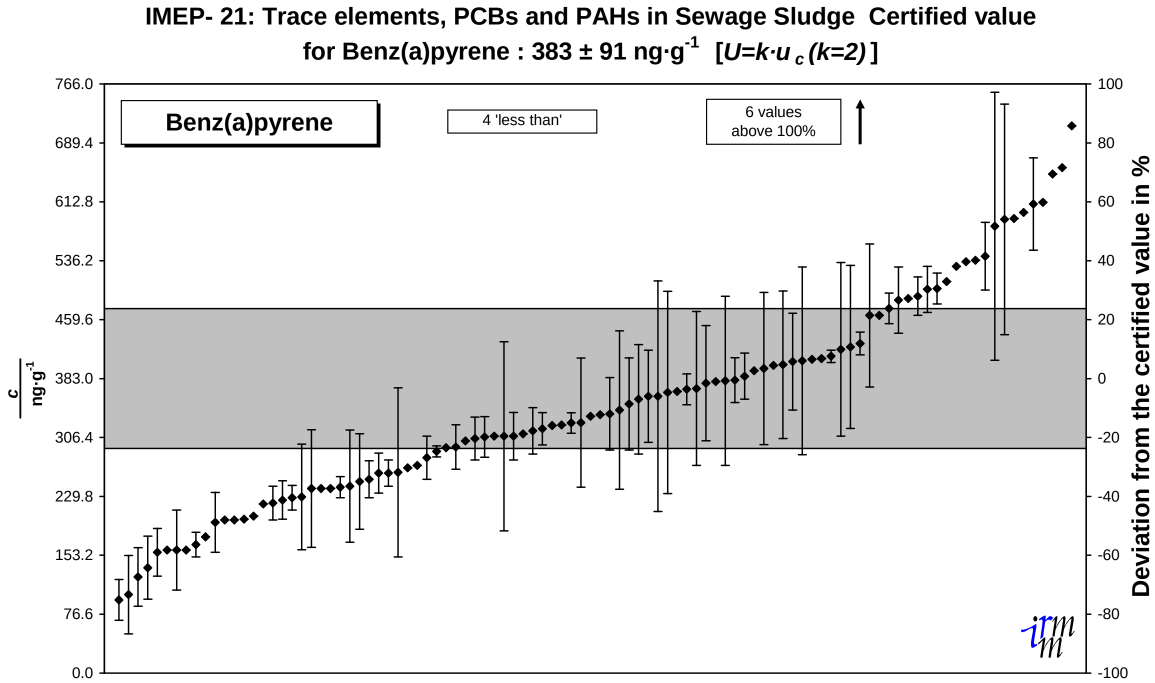
Figure 32

IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge Certified value for Benz(a)anthracene : 386 ± 56 ng·g^{-1} [$U=k \cdot u_c (k=2)$]



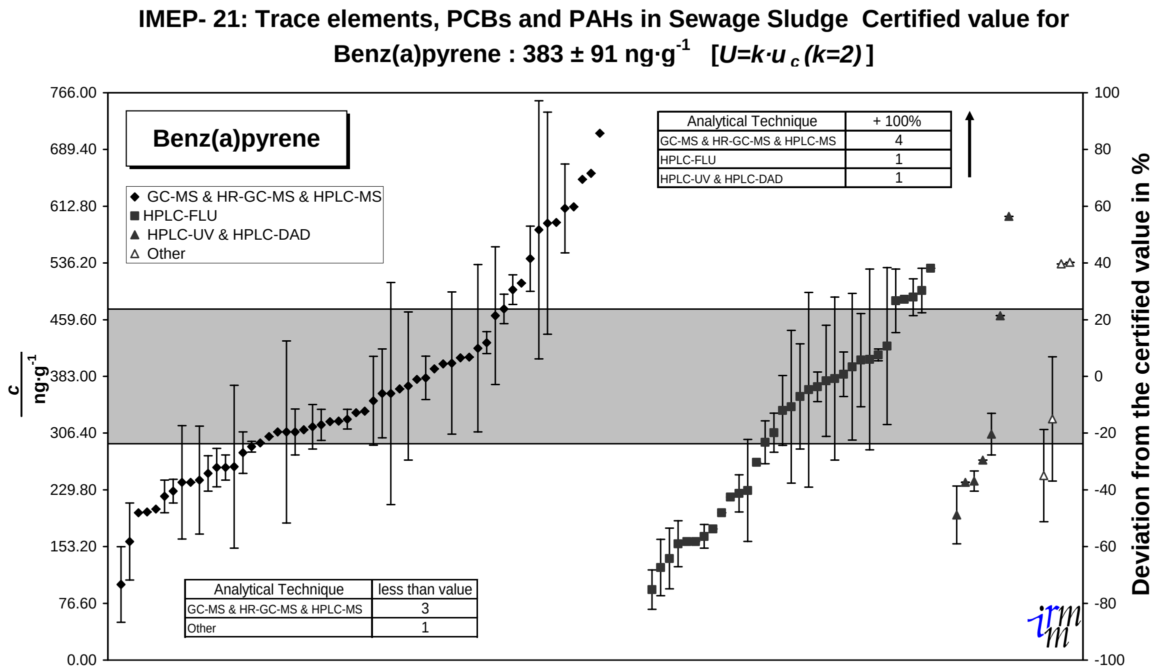
Results from all participants according to analytical techniques used

Figure 33



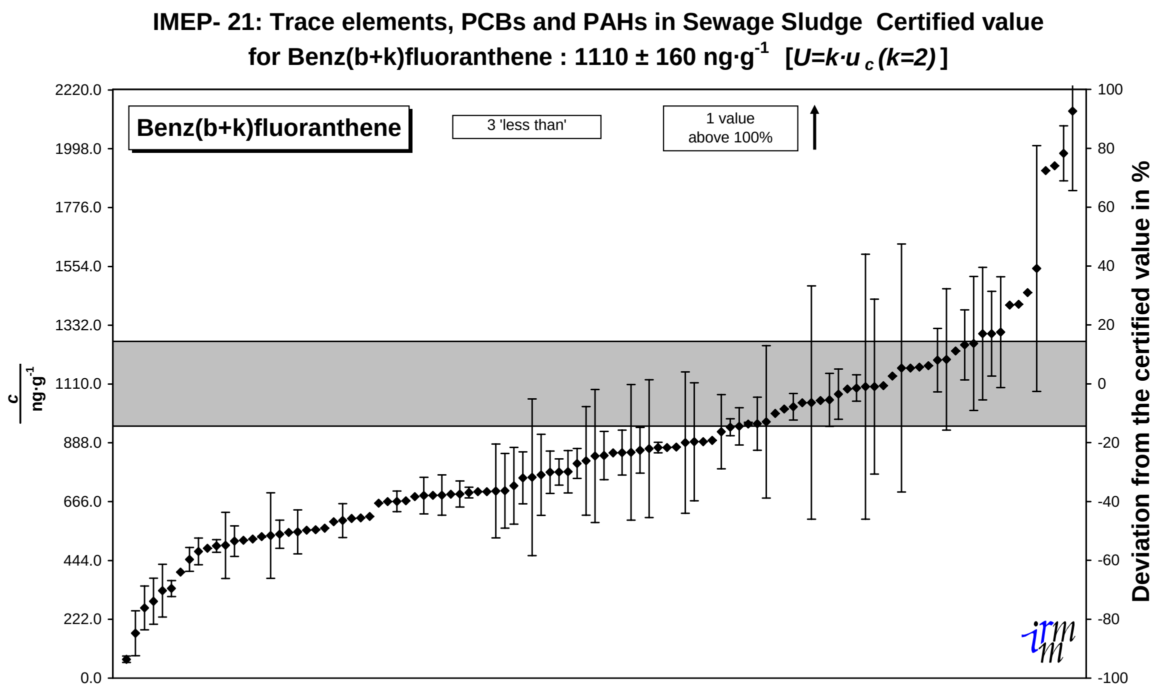
Results for Benz(a)pyrene from all participants

Figure 34



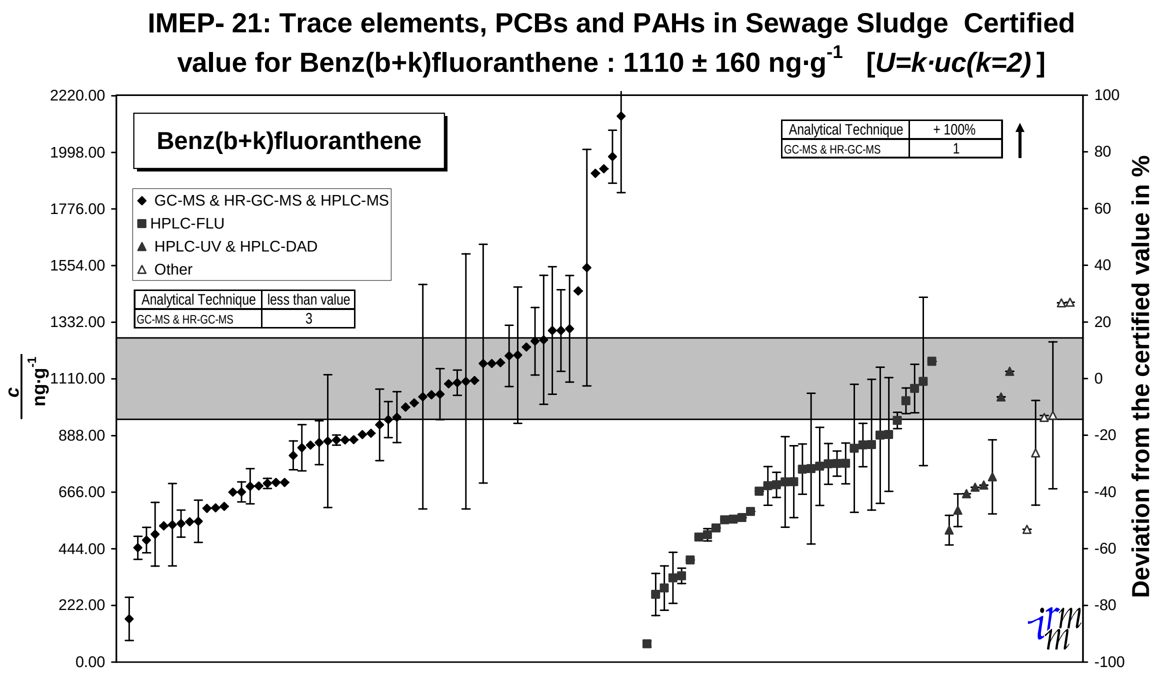
Results from all participants according to analytical techniques used

Figure 35



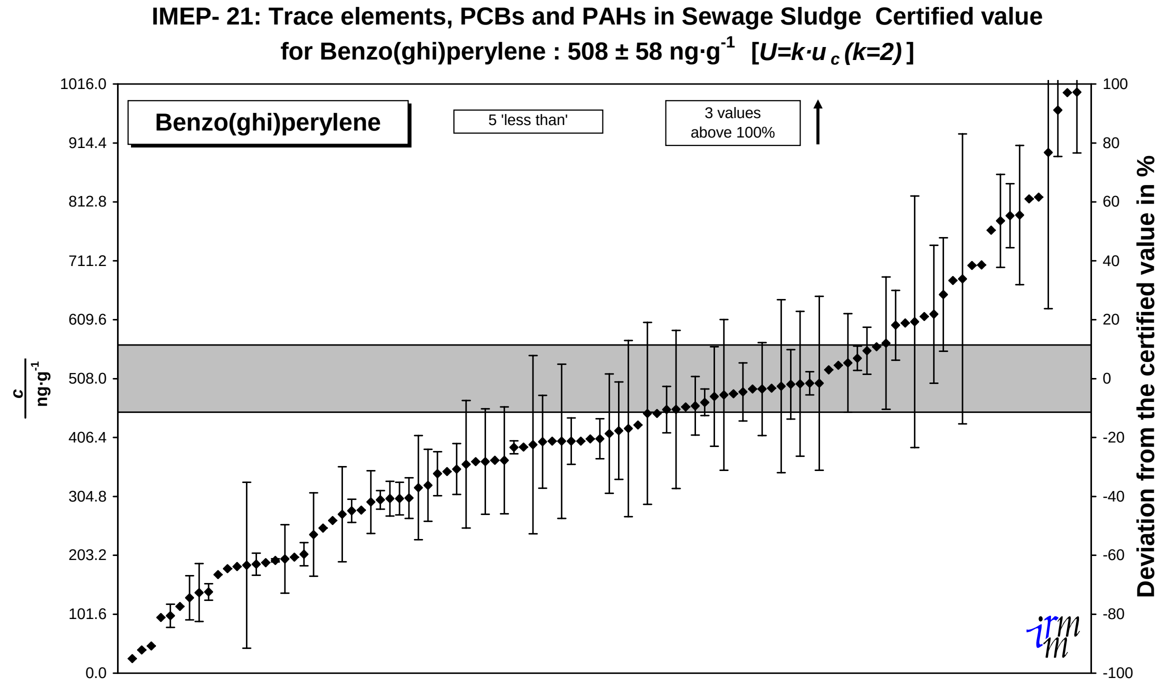
Results for Benz(b+k)fluoranthene from all participants

Figure 36



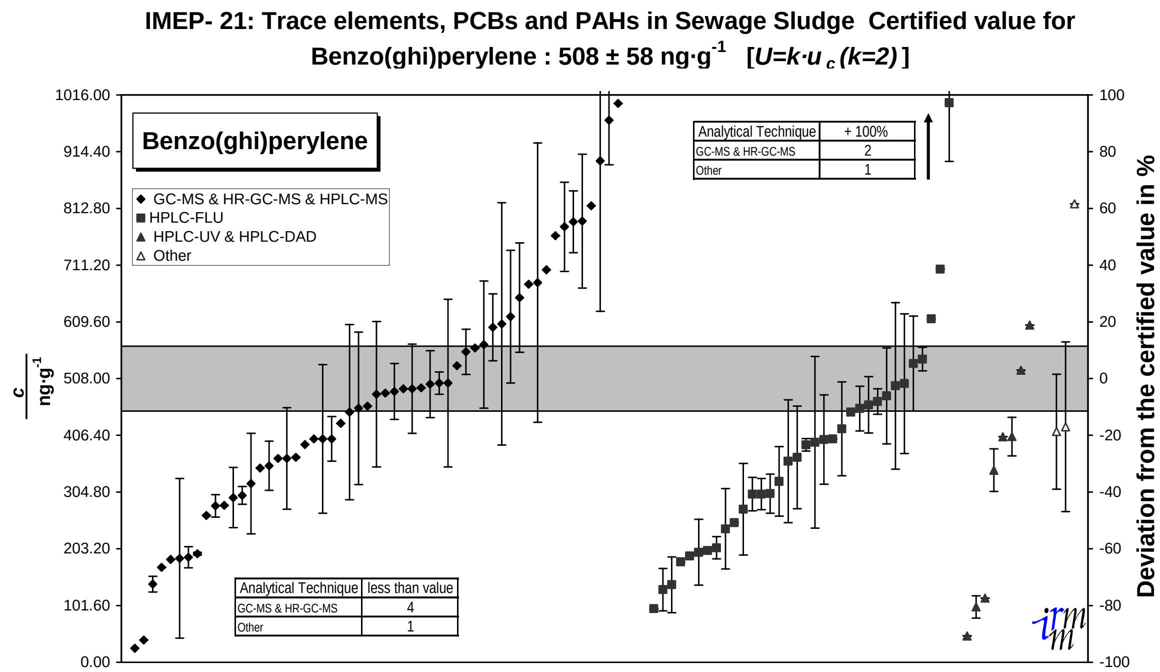
Results from all participants according to analytical techniques used

Figure 37



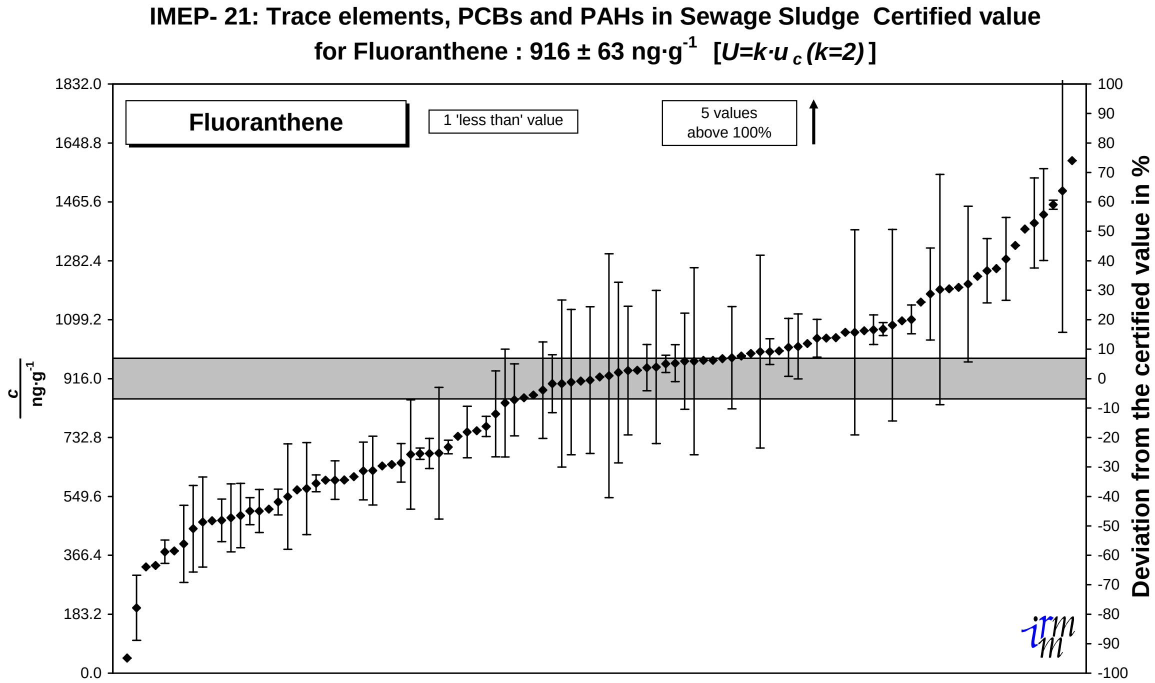
Results for Benzo(ghi)perylene from all participants

Figure 38



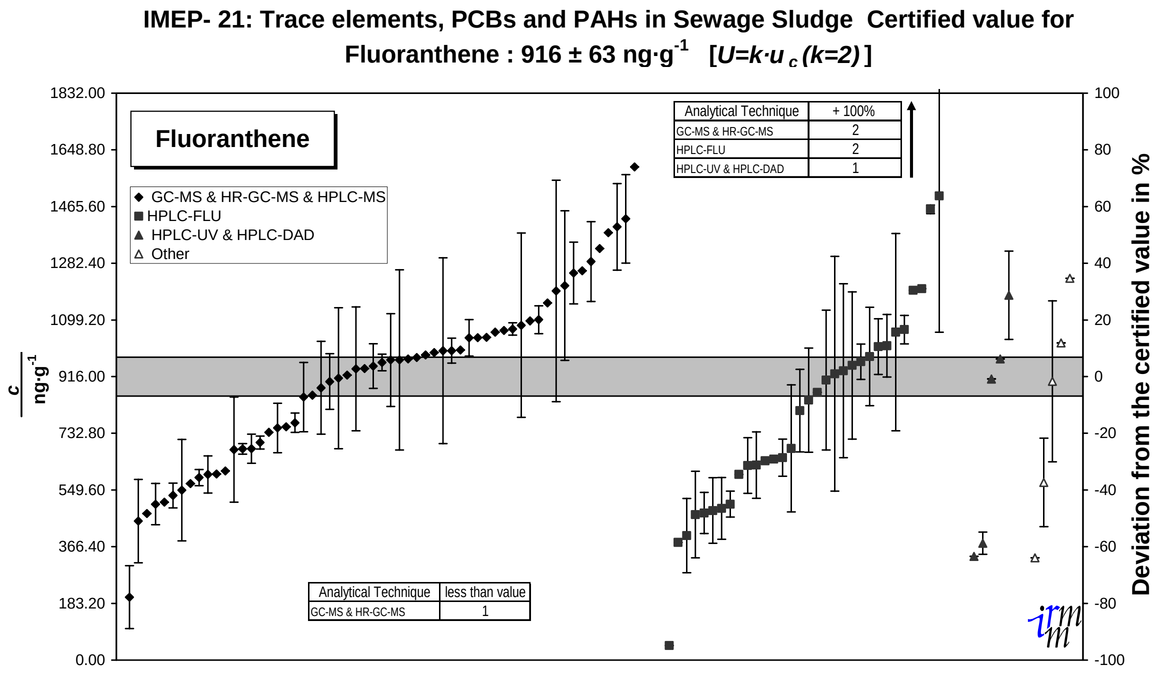
Results from all participants according to analytical techniques used

Figure 39



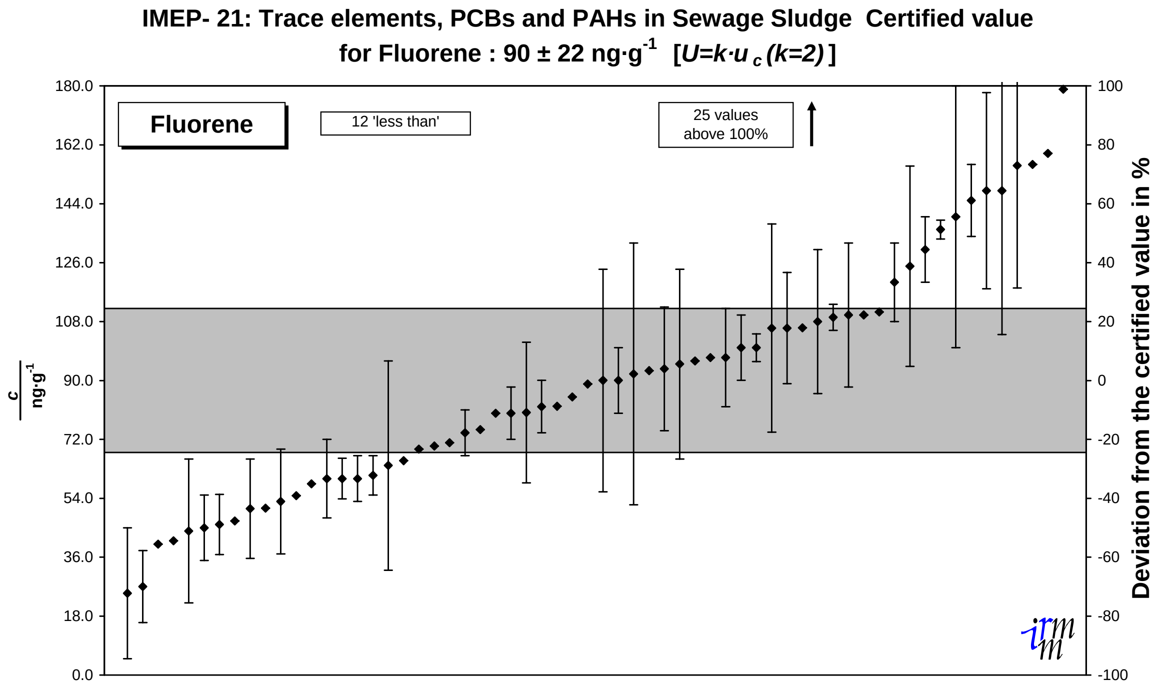
Results for Fluoranthene from all participants

Figure 40



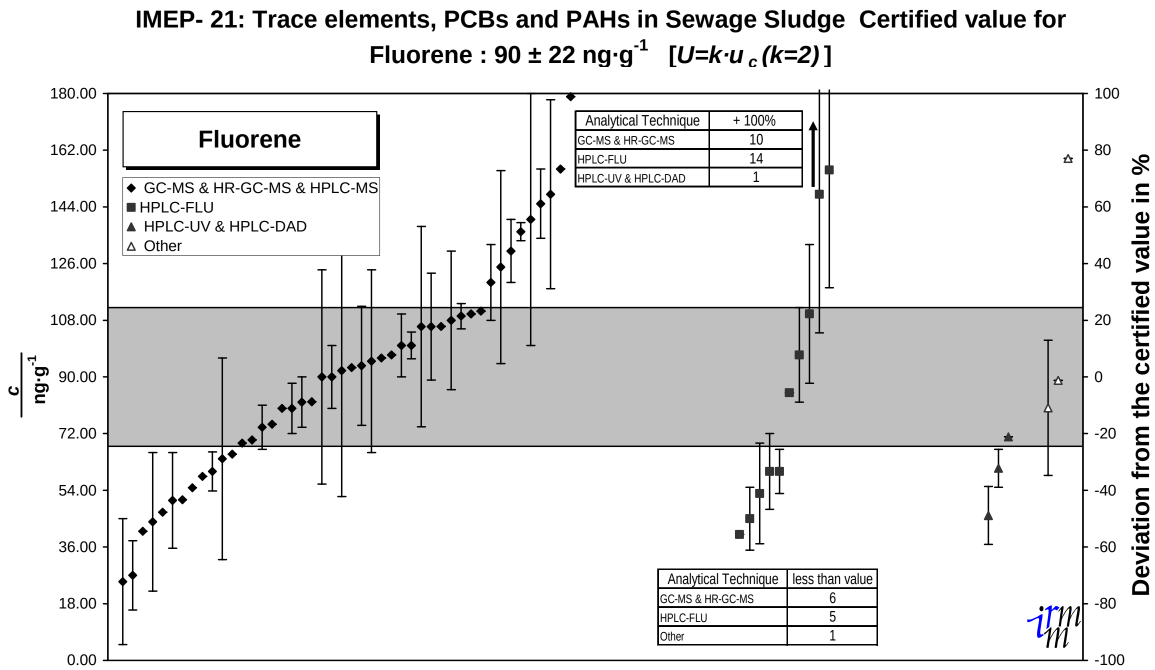
Results from all participants according to analytical techniques used

Figure 41



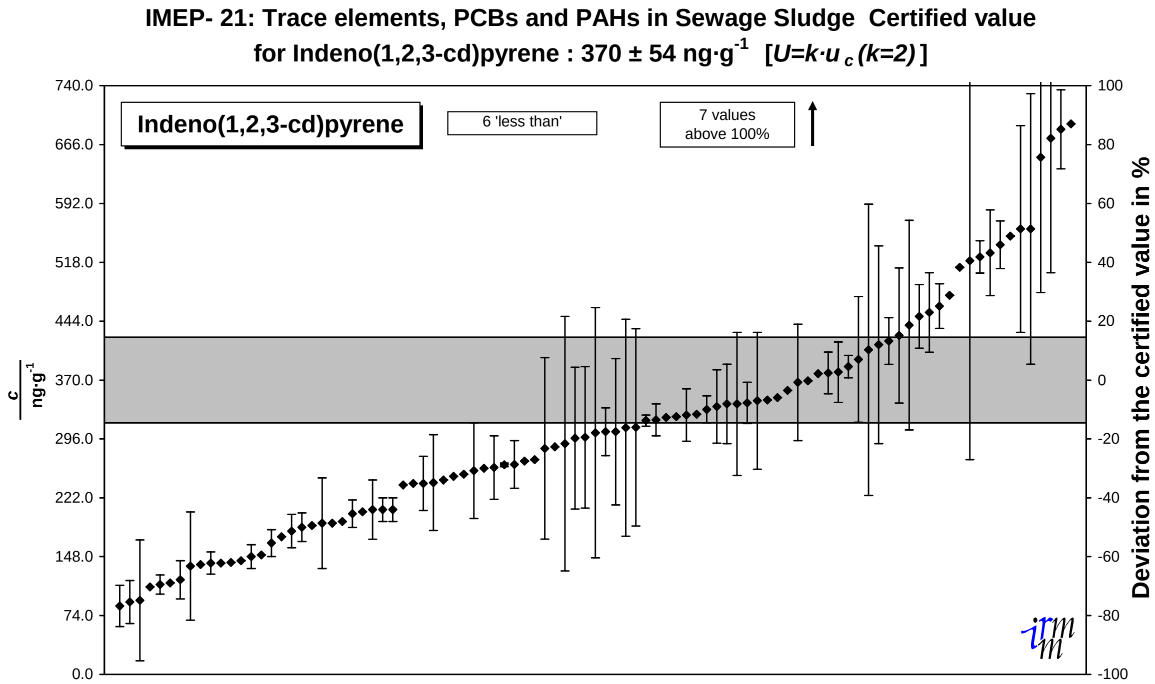
Results for Fluorene from all participants

Figure 42



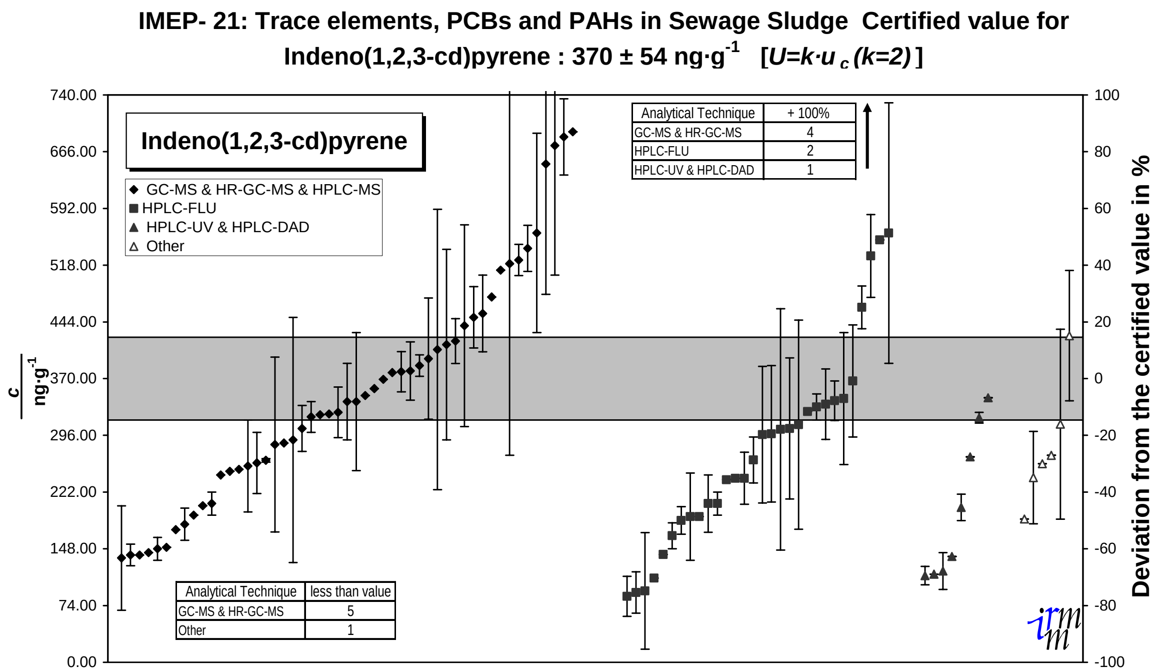
Results from all participants according to analytical techniques used

Figure 43



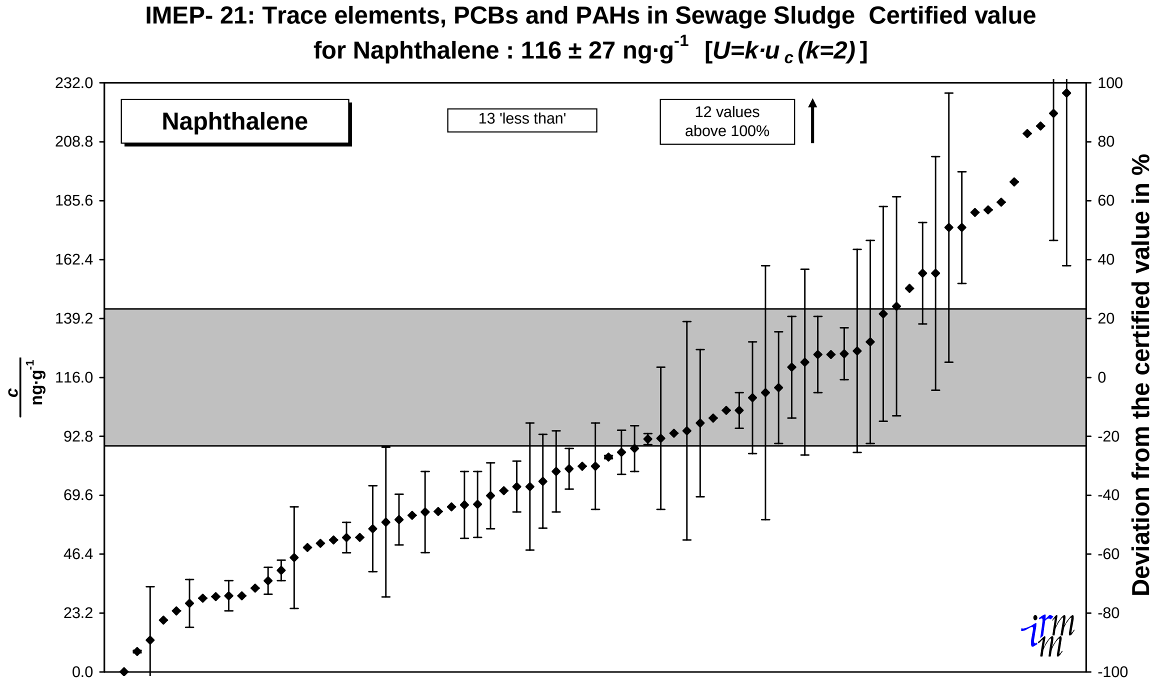
Results for Indeno(1,2,3-cd)pyrene from all participants

Figure 44



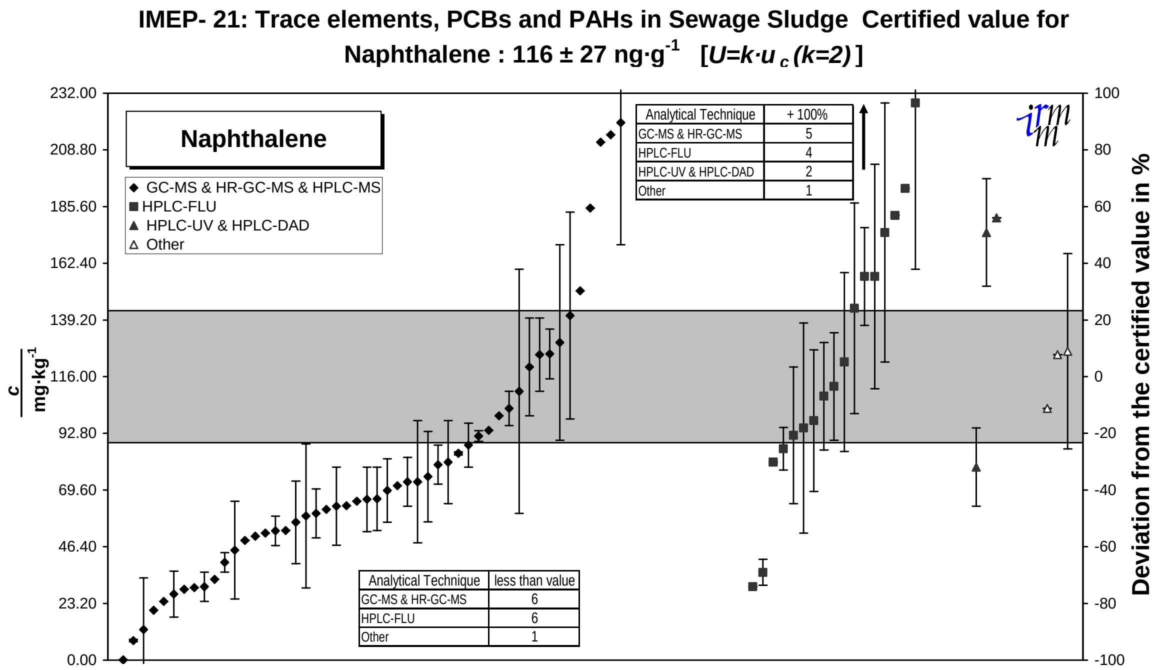
Results from all participants according to analytical techniques used

Figure 45



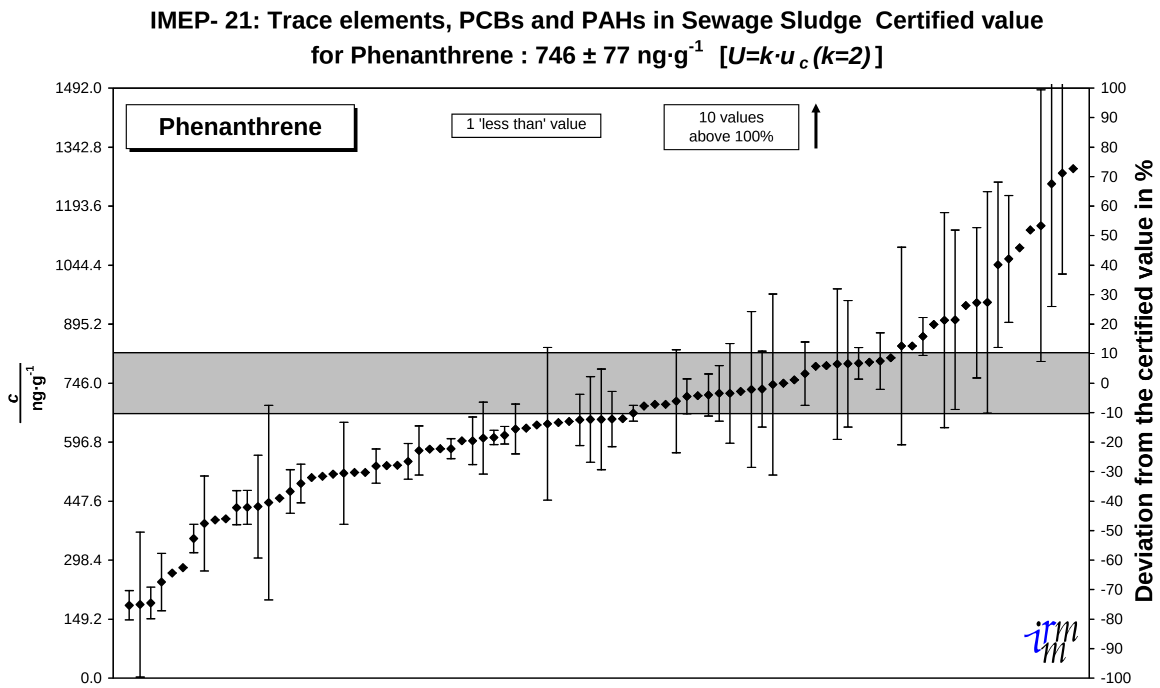
Results for Naphthalene from all participants

Figure 46



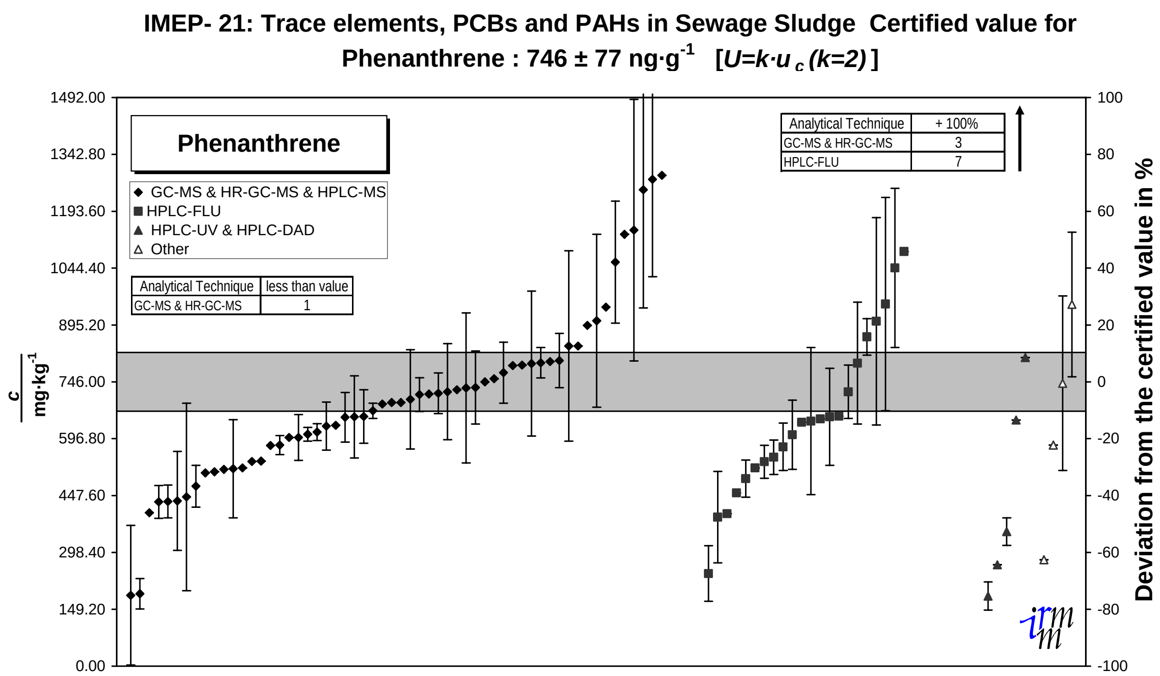
Results from all participants according to analytical techniques used

Figure 47



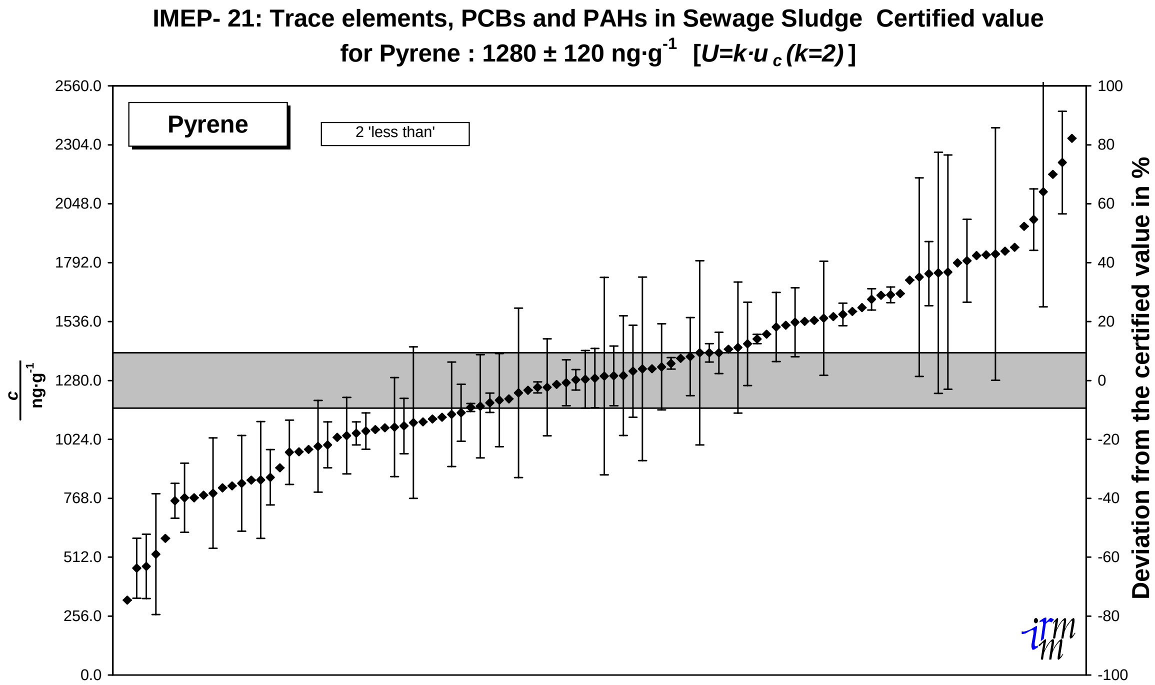
Results for Phenanthrene from all participants

Figure 48



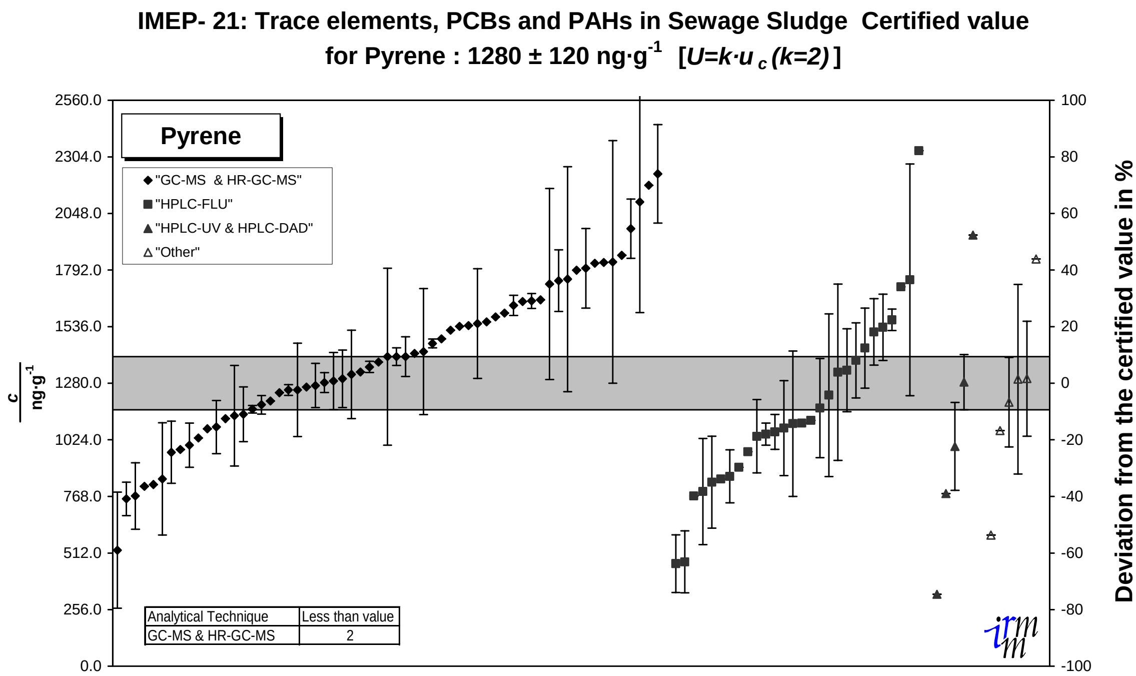
Results from all participants according to analytical techniques used

Figure 49



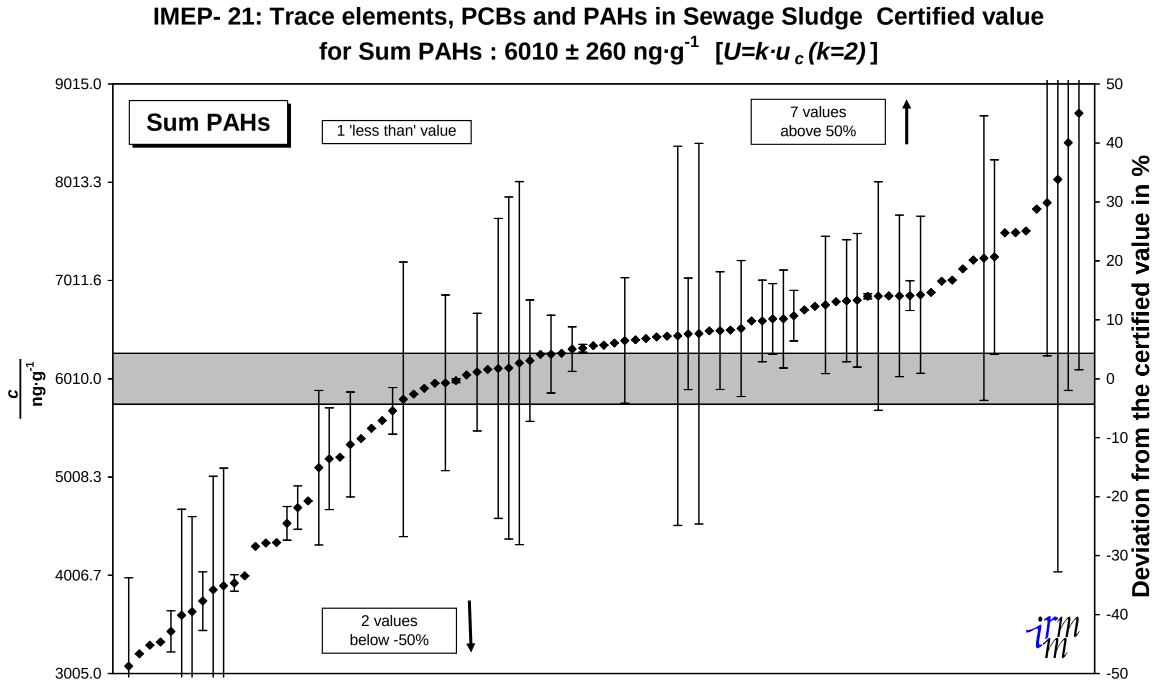
Results for Pyrene from all participants

Figure 50



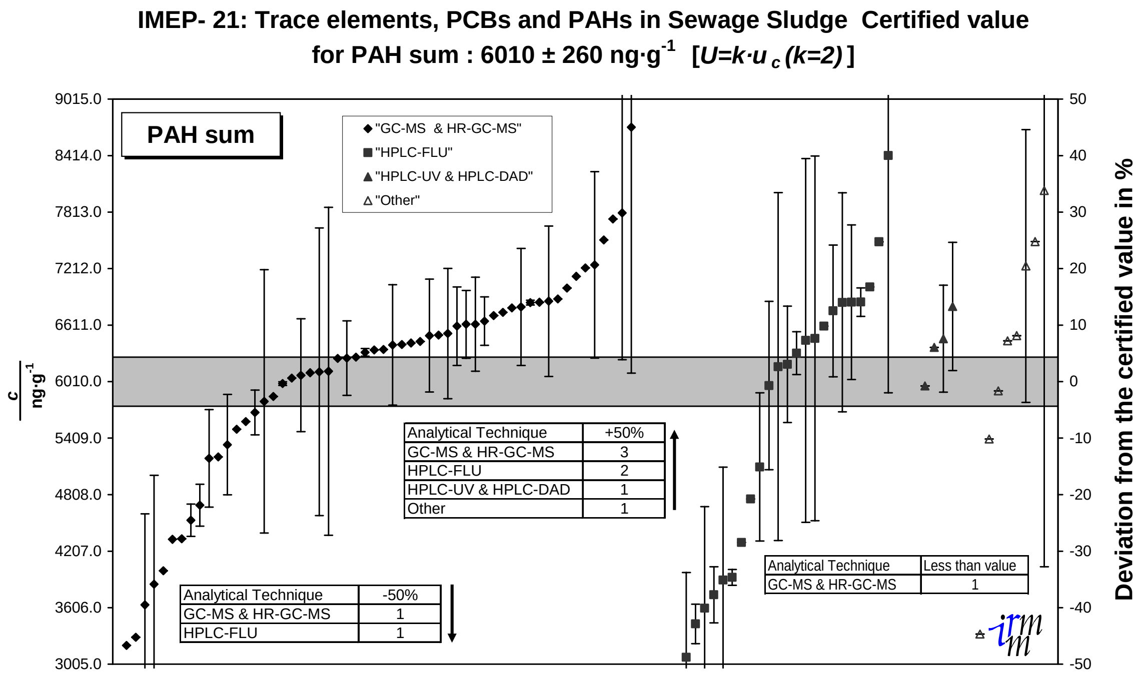
Results for Pyrene from all participants according to analytical techniques used

Figure 51



Results for Sum PAHs from all participants

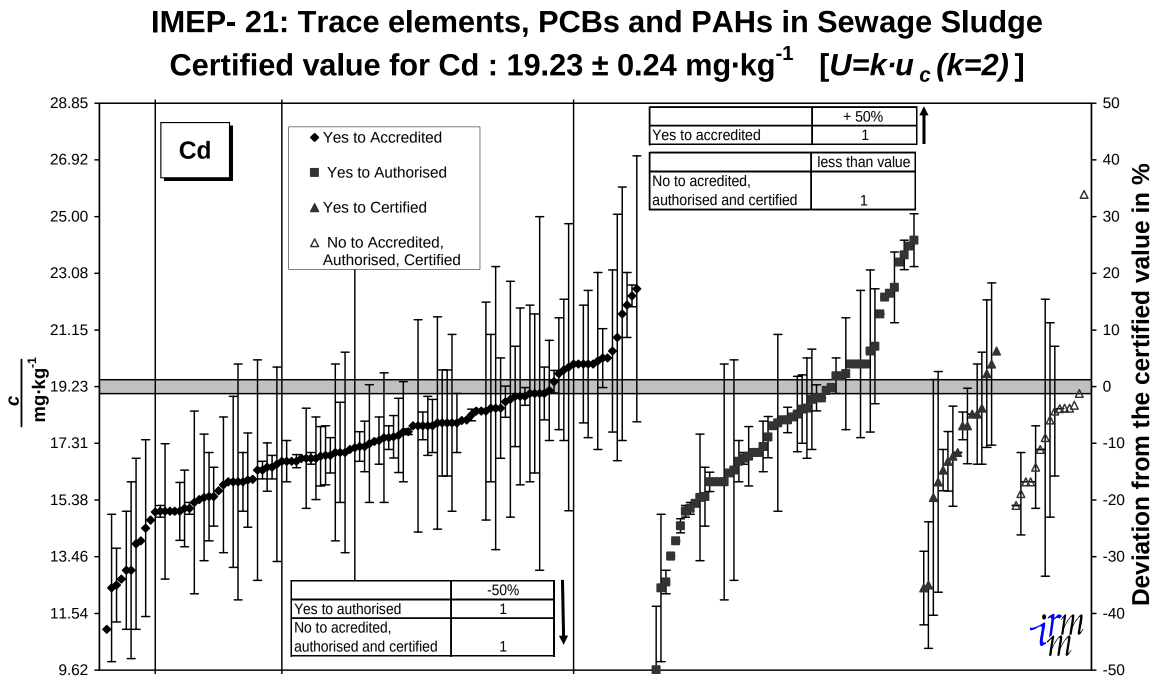
Figure 52



Results for PAH sum from all participants according to analytical techniques used

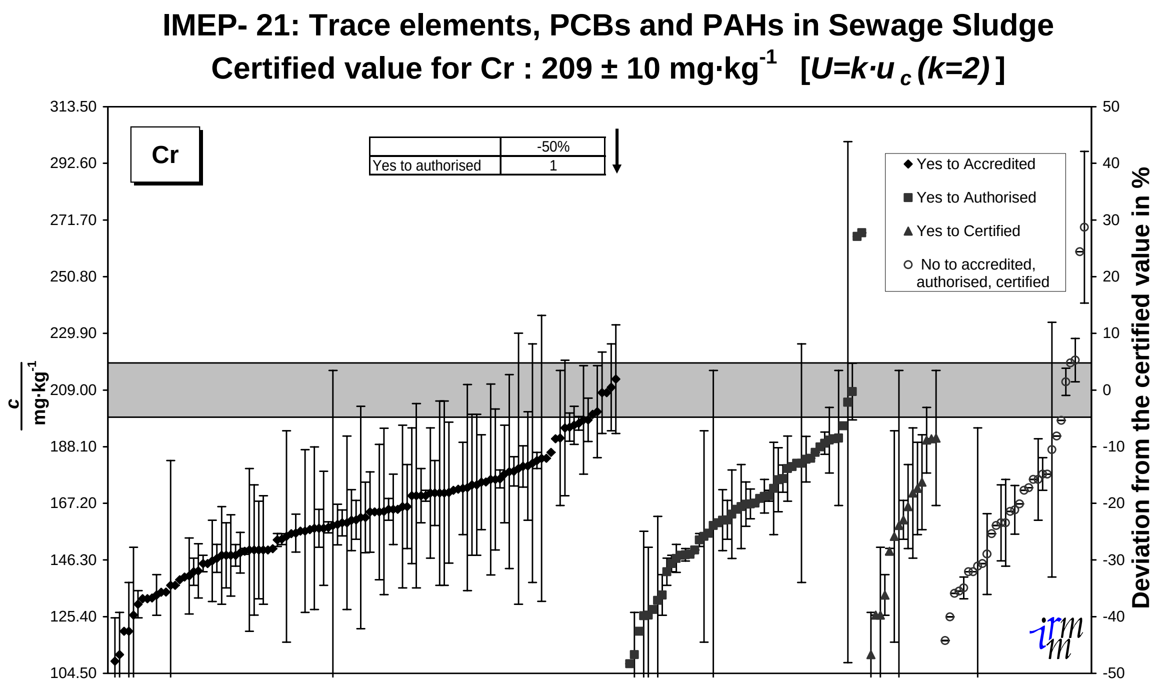
IMEP-21: Trace Elements, PCBs and PAHs in Sewage Sludge
Annex 1 – Participants' results – Questionnaire Graphs

Figure 53



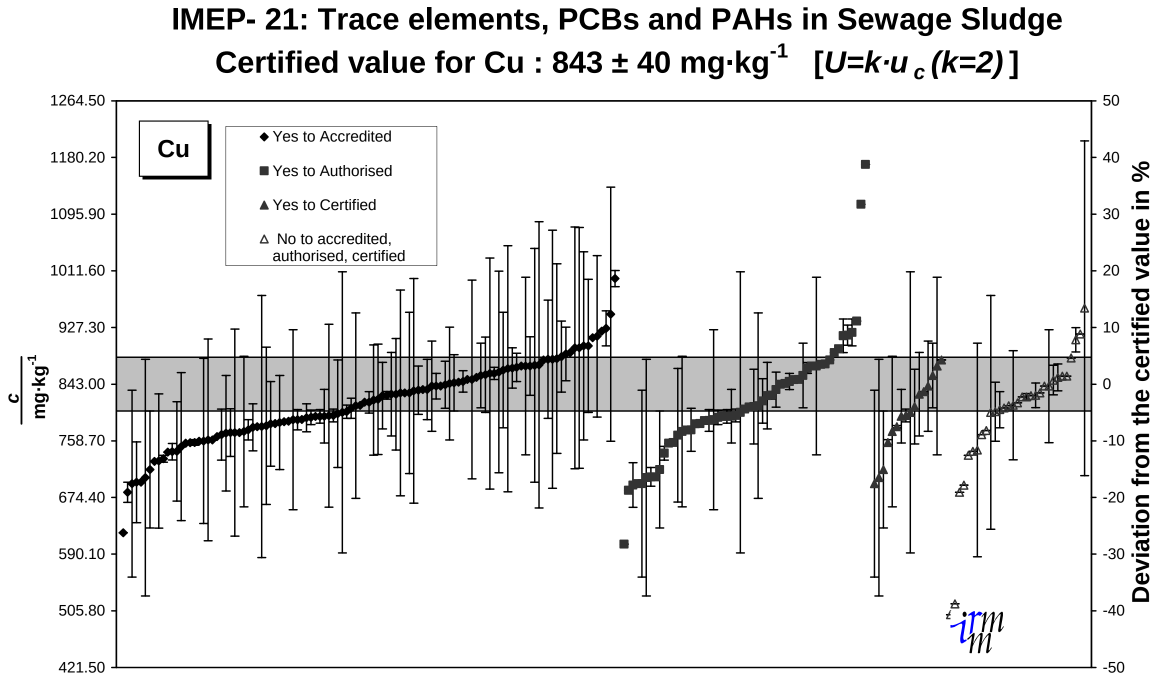
Results from all participants according to accredited, authorised, certified

Figure 54



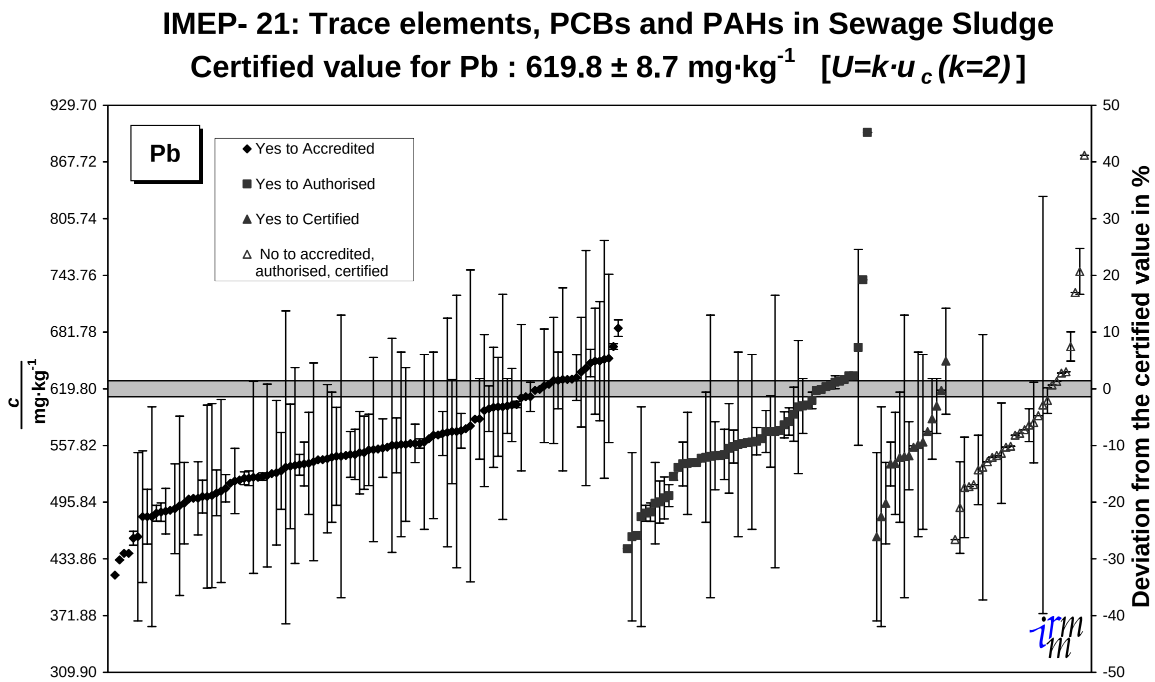
Results from all participants according to to accredited, authorised, certified

Figure 55



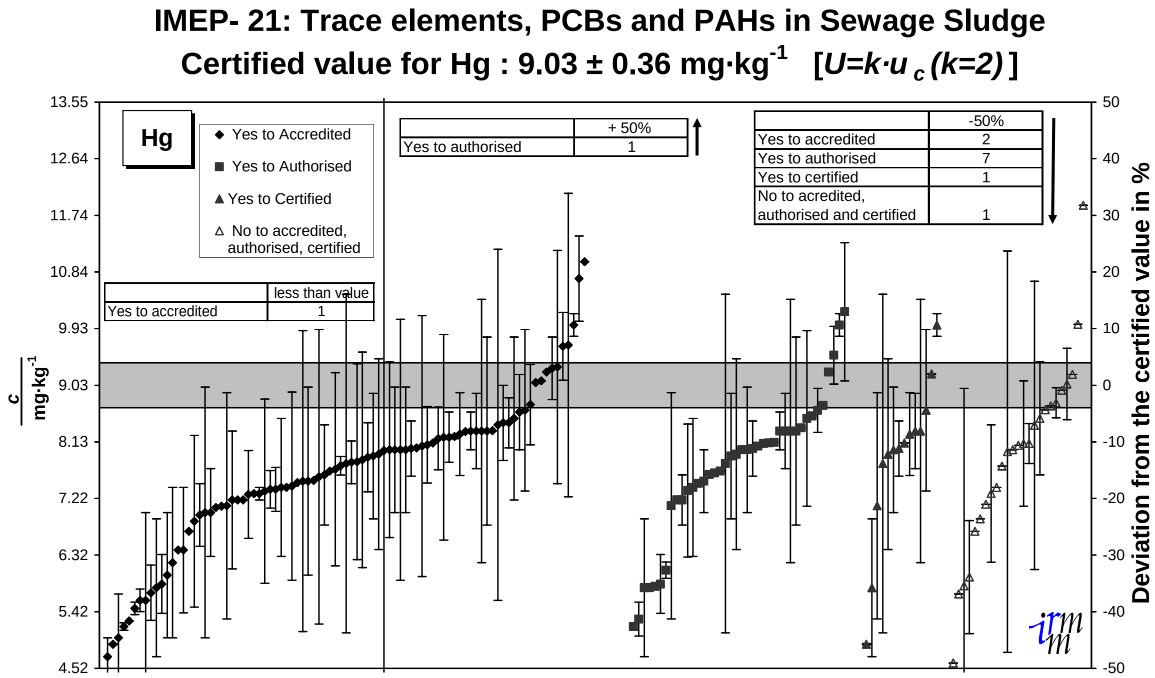
Results from all participants according to accredited, authorised, certified

Figure 56



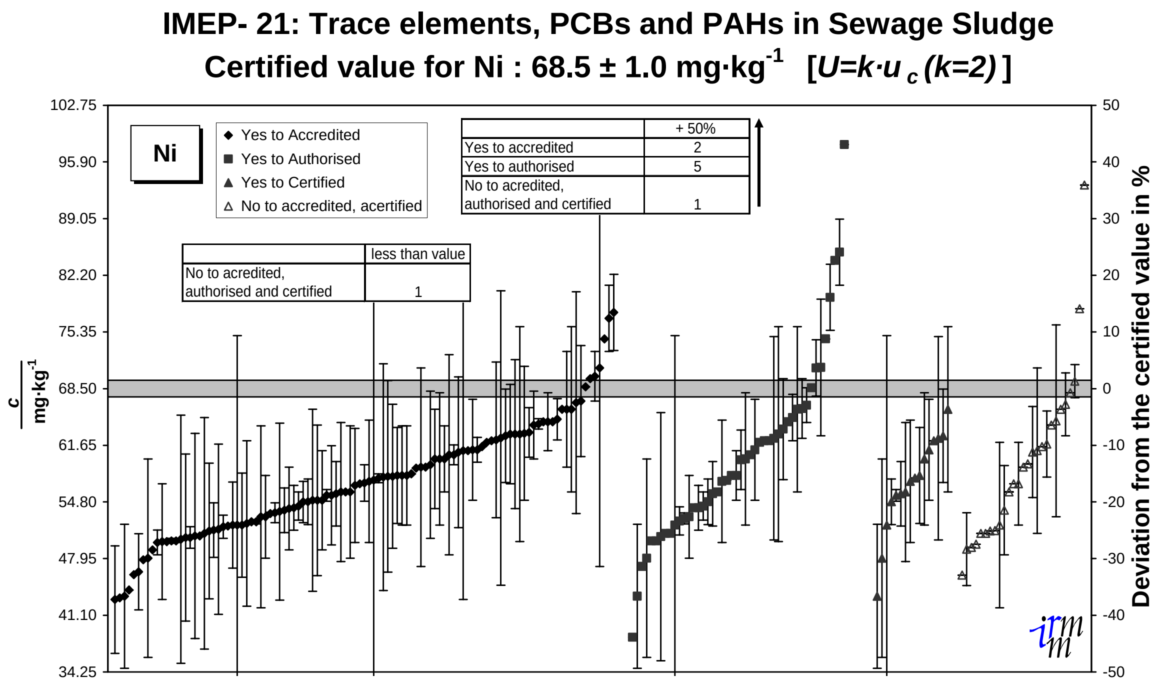
Results from all participants according to accredited, authorised, certified

Figure 57



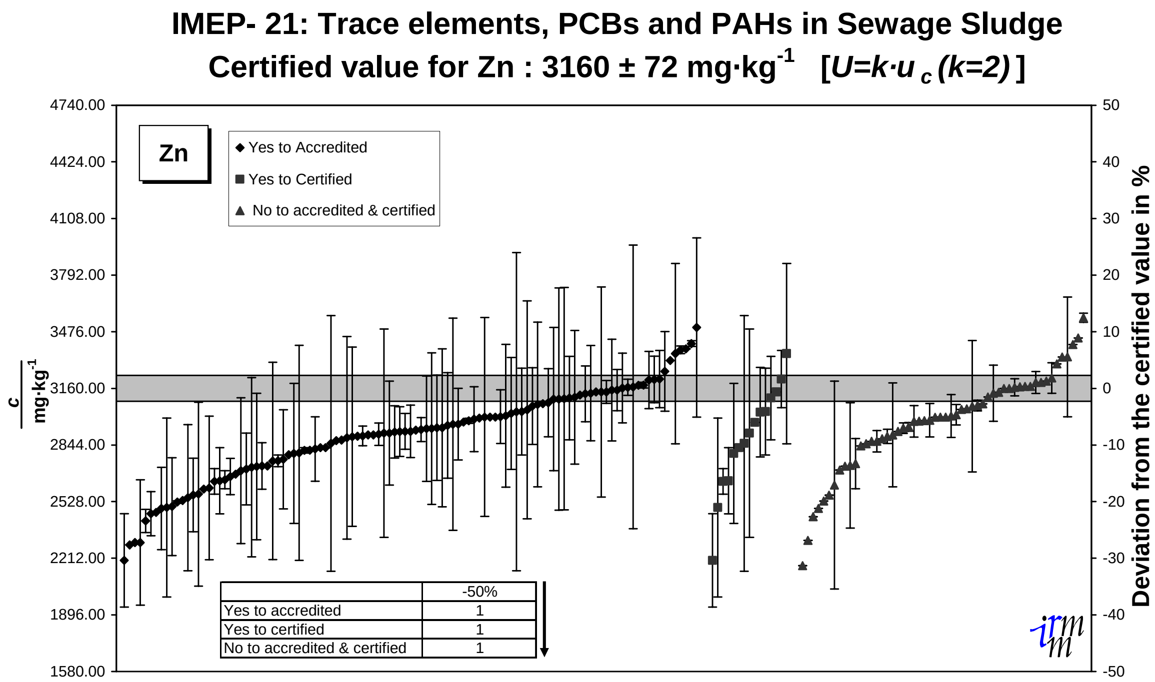
Results from all participants according to accredited, authorised, certified

Figure 58



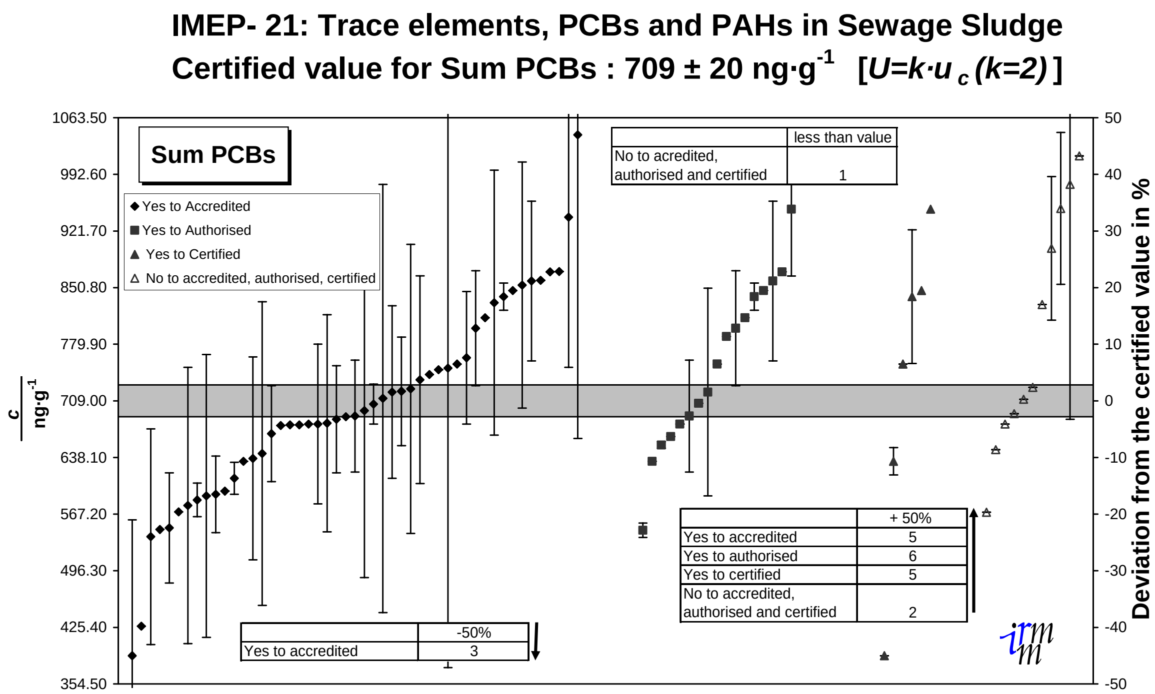
Results from all participants according to accredited, authorised, certified

Figure 59



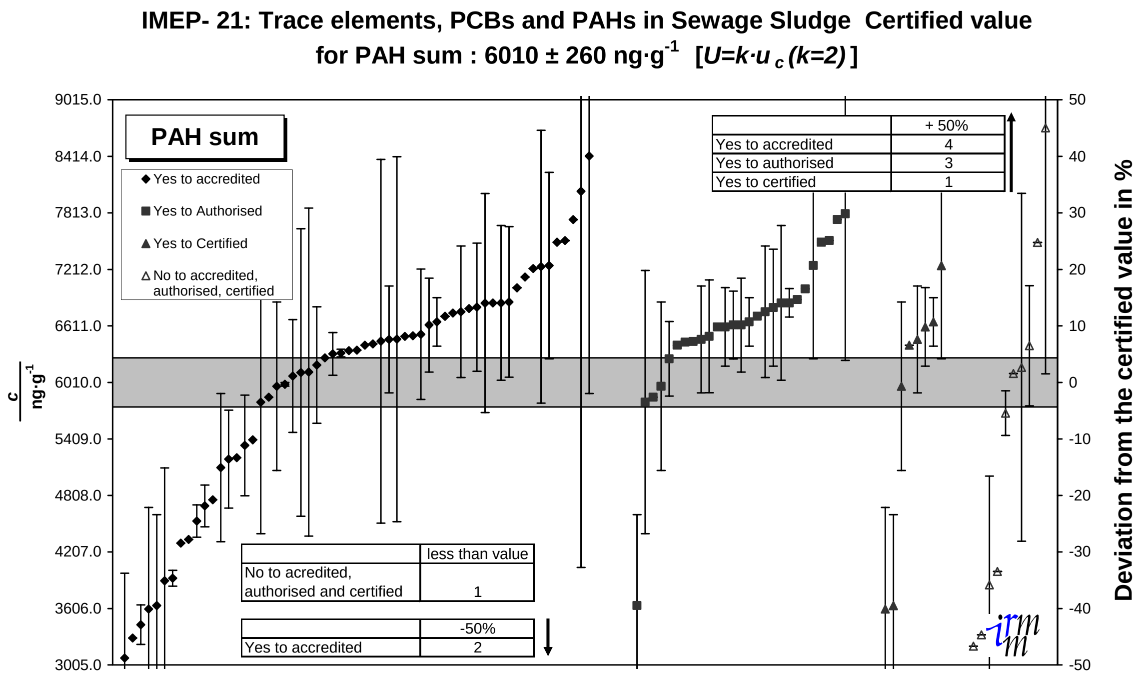
Results from all participants according to accredited, authorised, certified

Figure 60



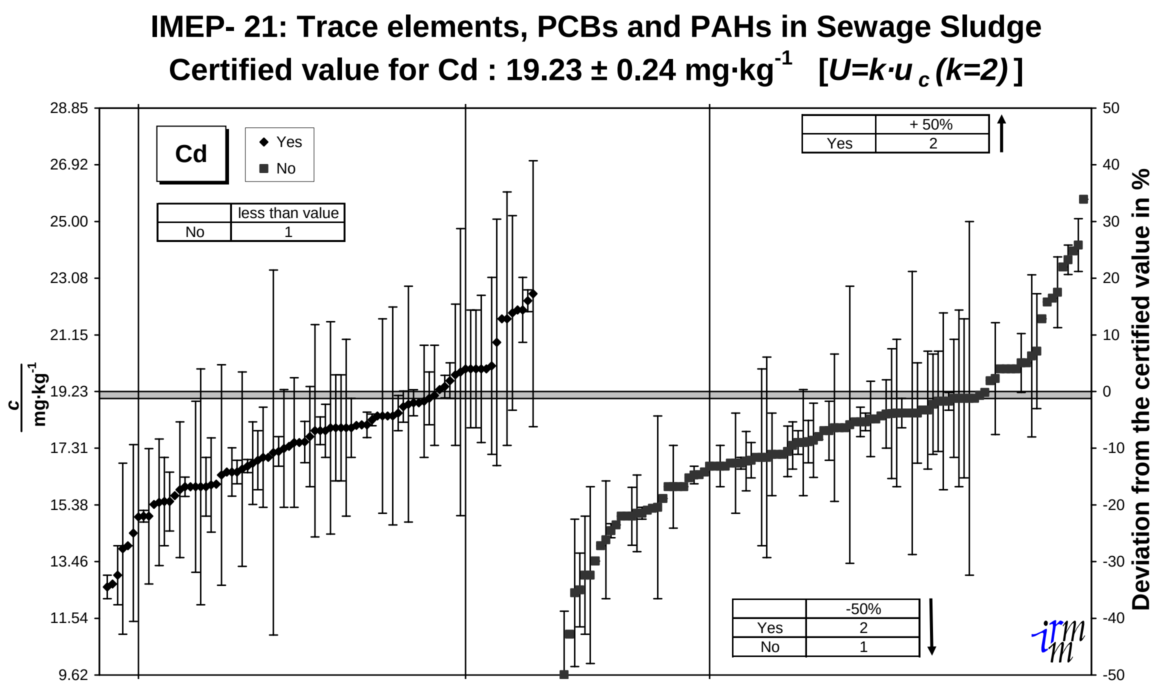
Results from all participants according to accredited, authorised, certified

Figure 61



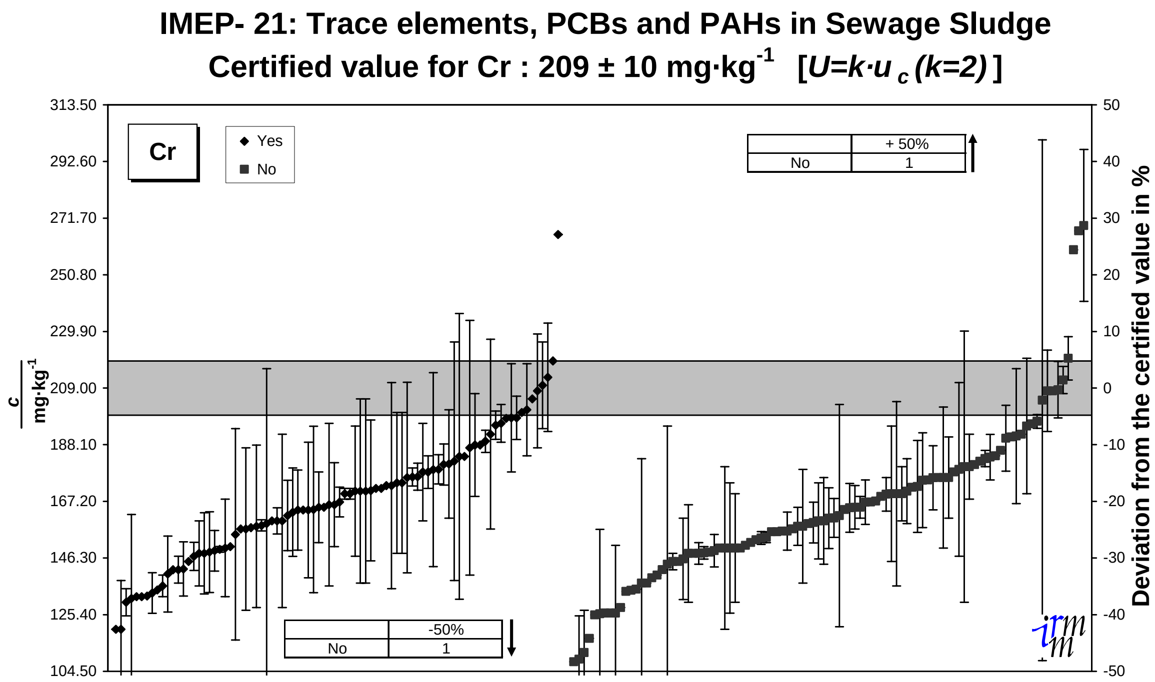
Results from all participants according to accredited, authorised, certified

Figure 62



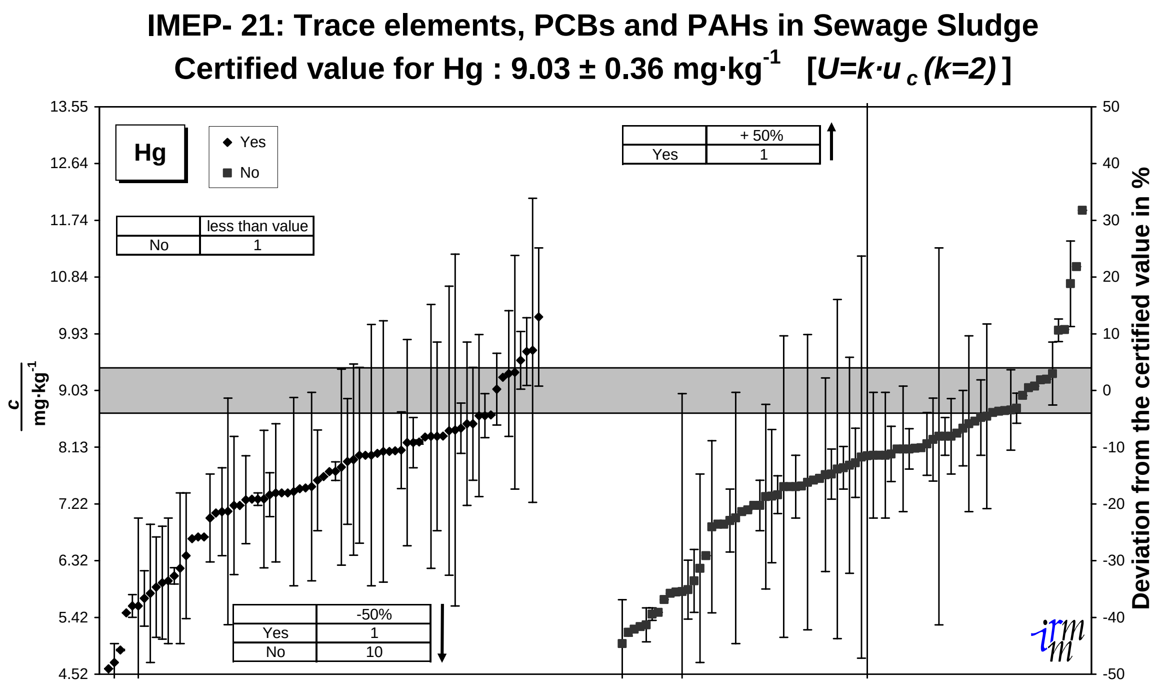
Results from all participants according to the use of Certified Reference Materials (CRMs)

Figure 63



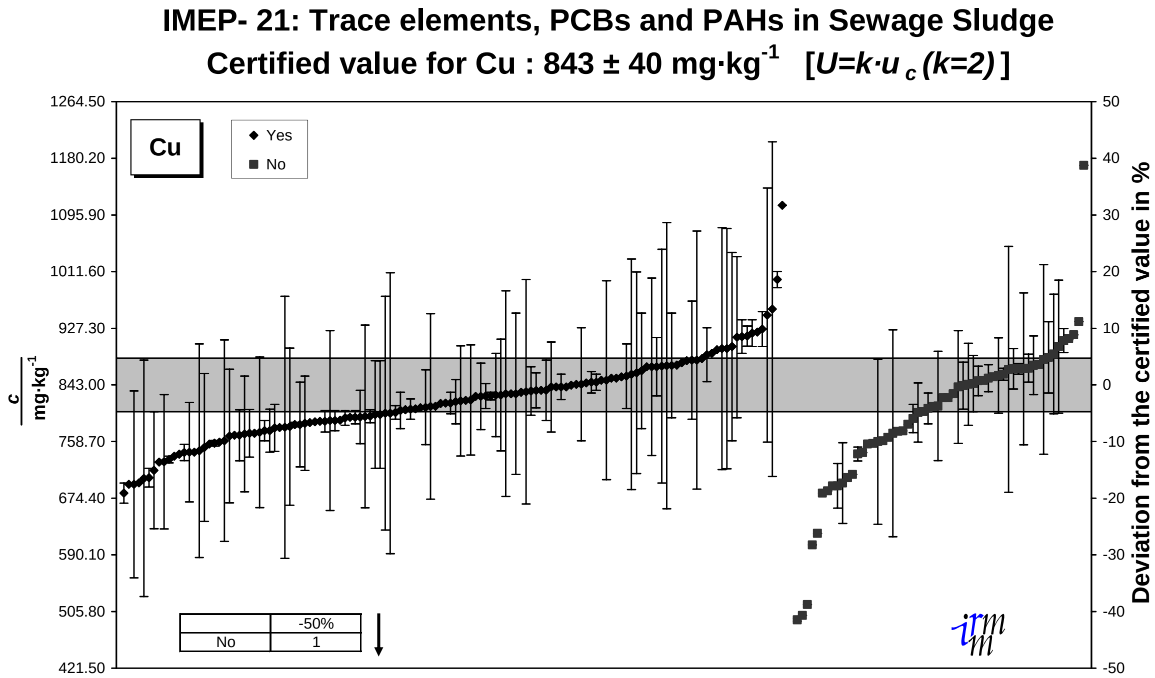
Results from all participants according to the use of Certified Reference Materials (CRMs)

Figure 64



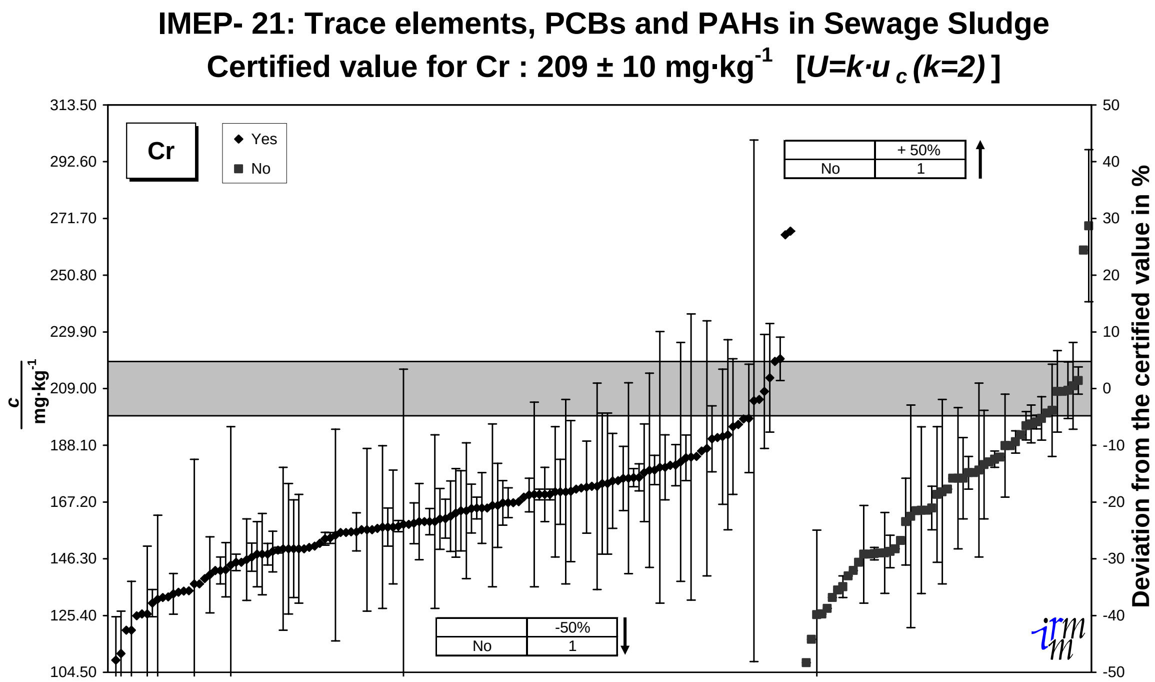
Results from all participants according to the use of Certified Reference Materials (CRMs)

Figure 65



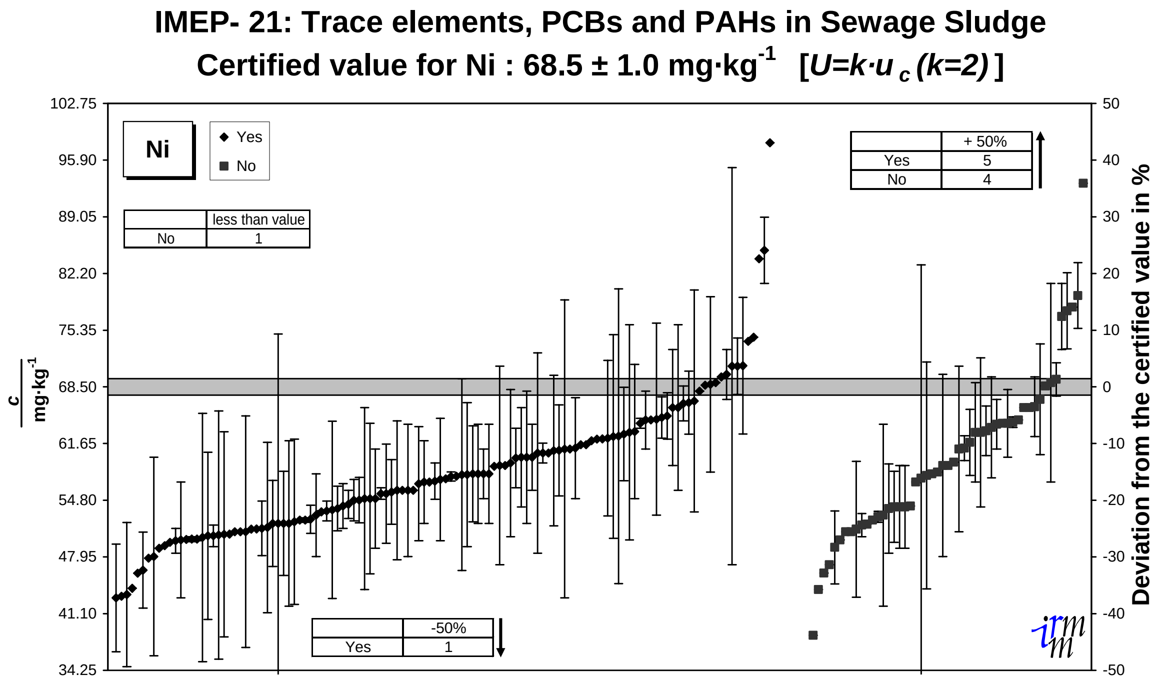
Results from all participants according to the use of any official method

Figure 66



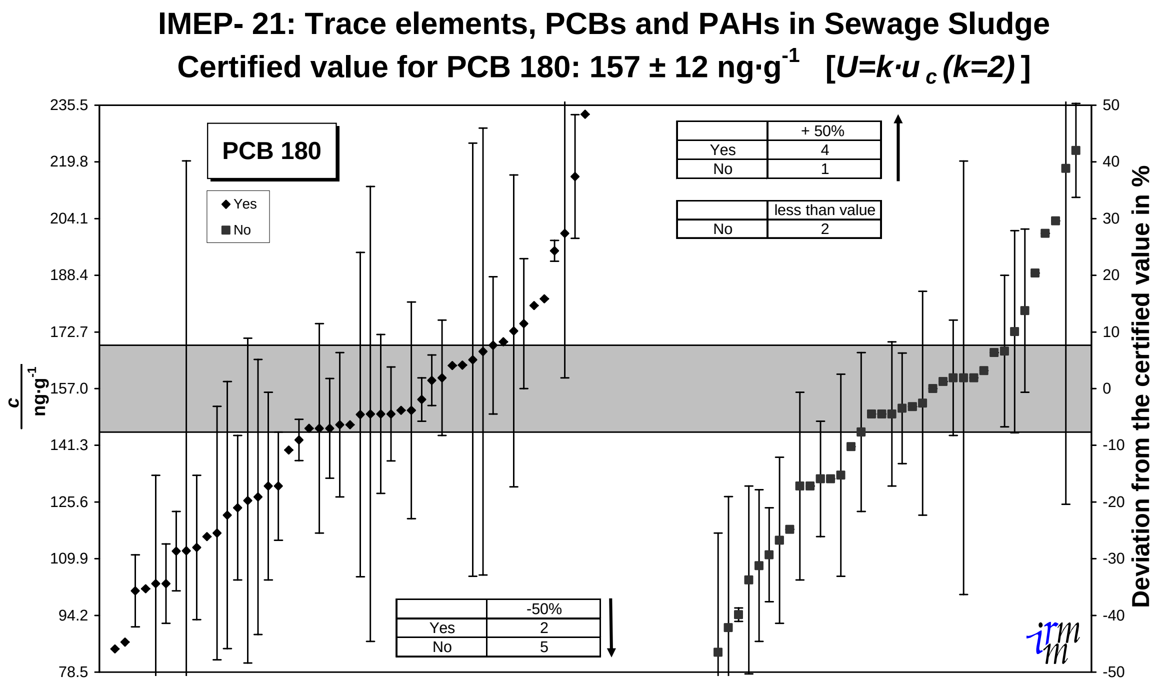
Results from all participants according to the use of any official method

Figure 67



Results from all participants according to the use of any official method

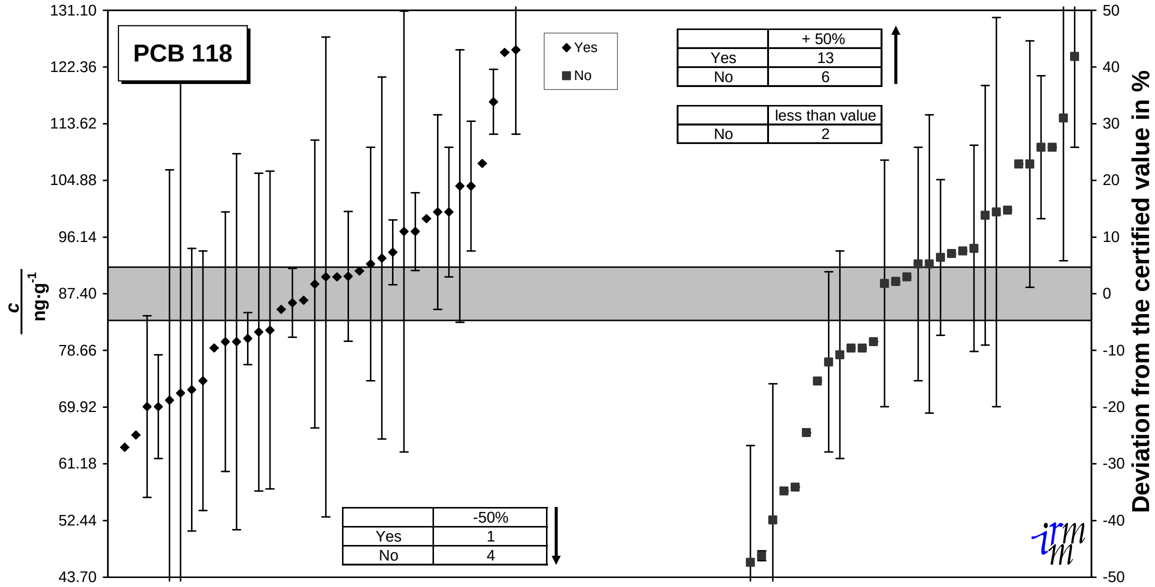
Figure 68



Results from all participants according to the use of any official method

Figure 69

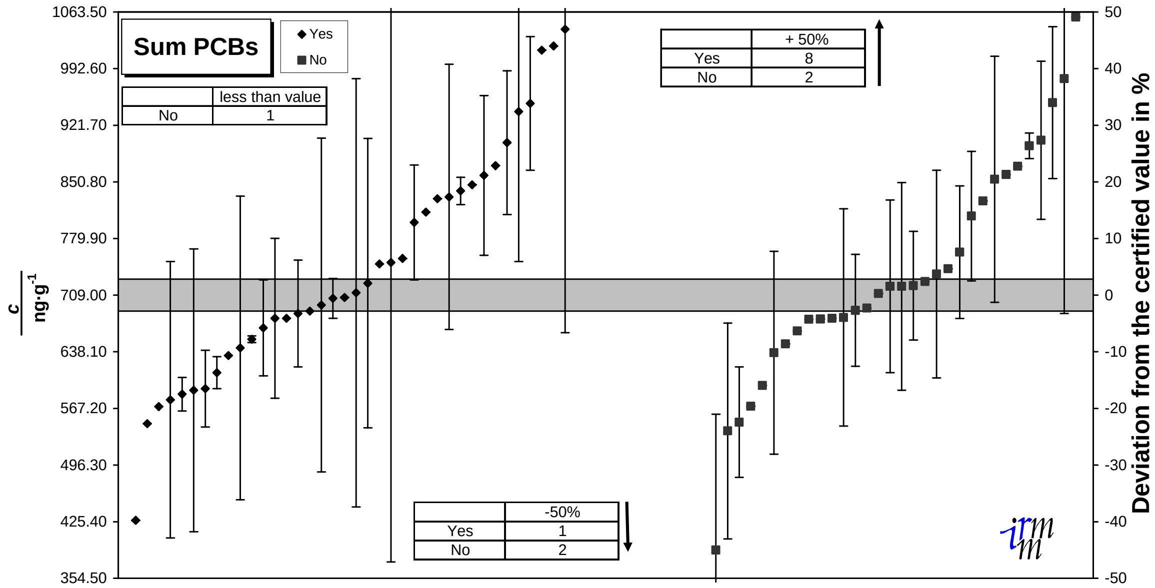
IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for PCB 118 : 87.4 ± 4.1 ng·g [*U=k·u c (k=2)*]



Results from all participants according to the use of any official method

Figure 70

IMEP- 21: Trace elements, PCBs and PAHs in Sewage Sludge
Certified value for Sum PCBs : 709 ± 20 ng·g^{-1} [*U=k·u c (k=2)*]



Results from all participants according to the use of any official method

Figure 71

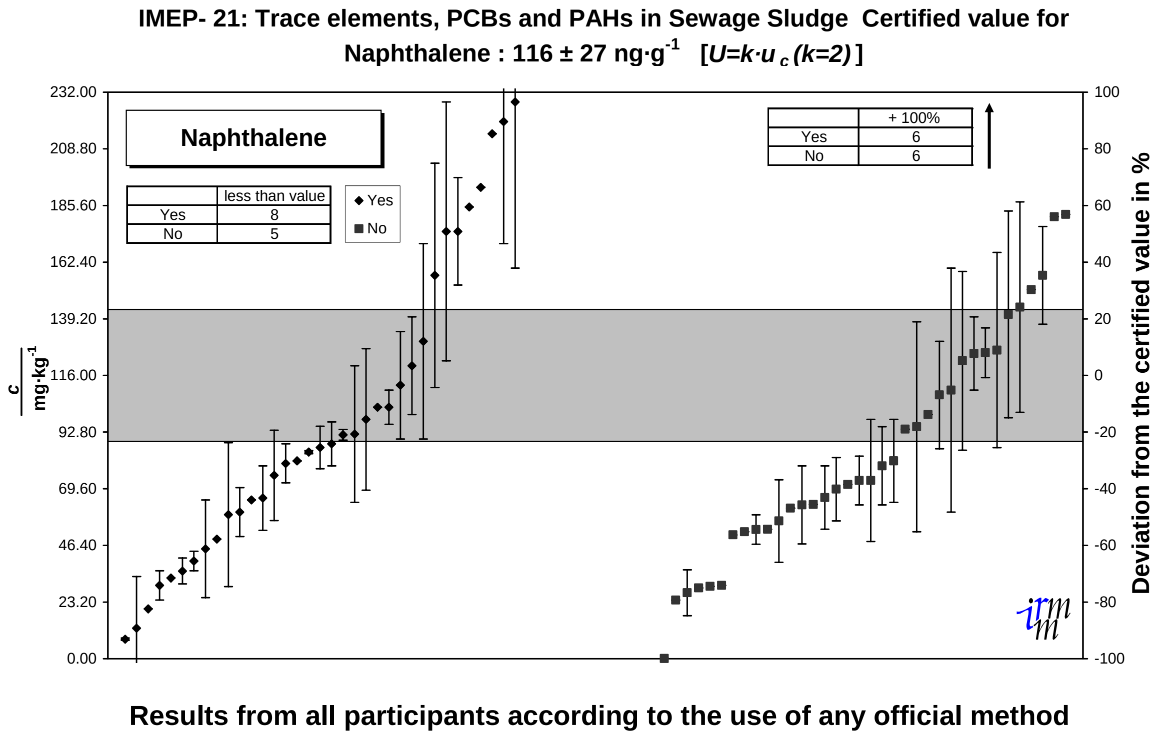


Figure 72

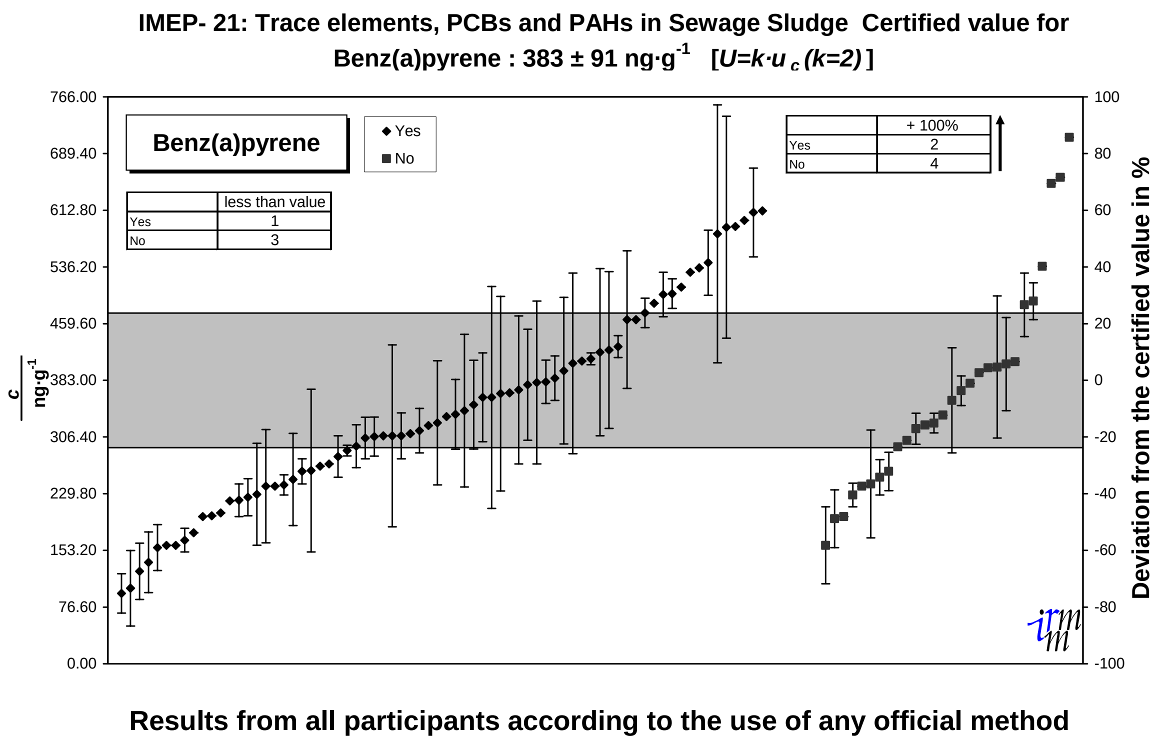
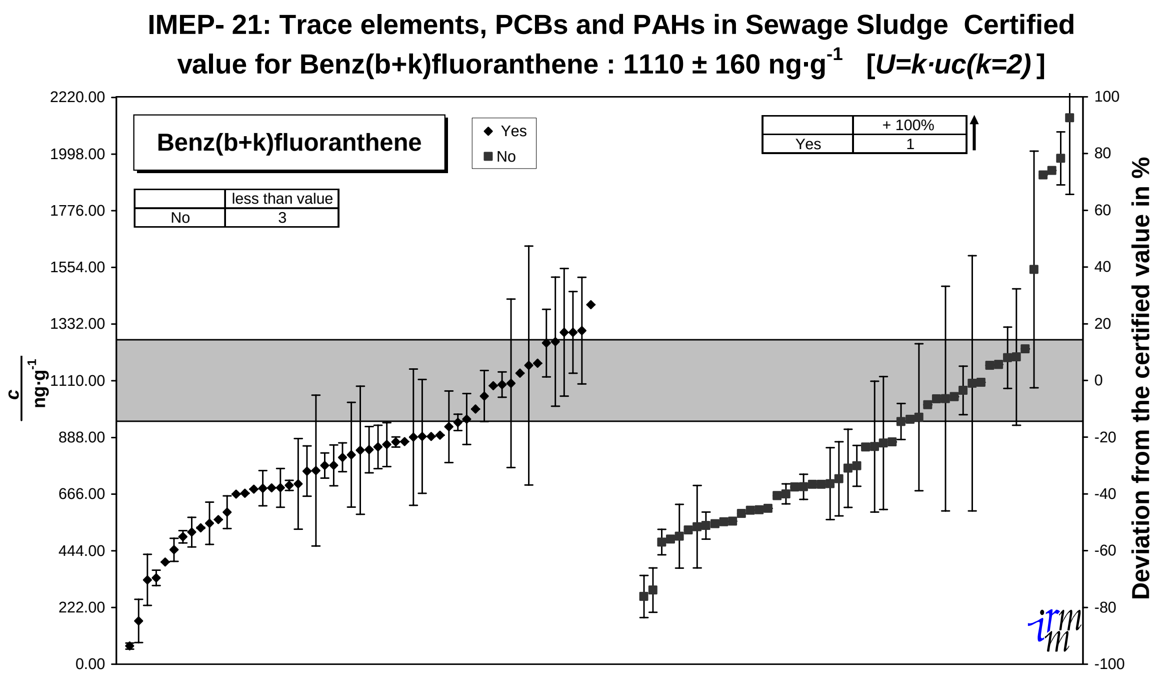
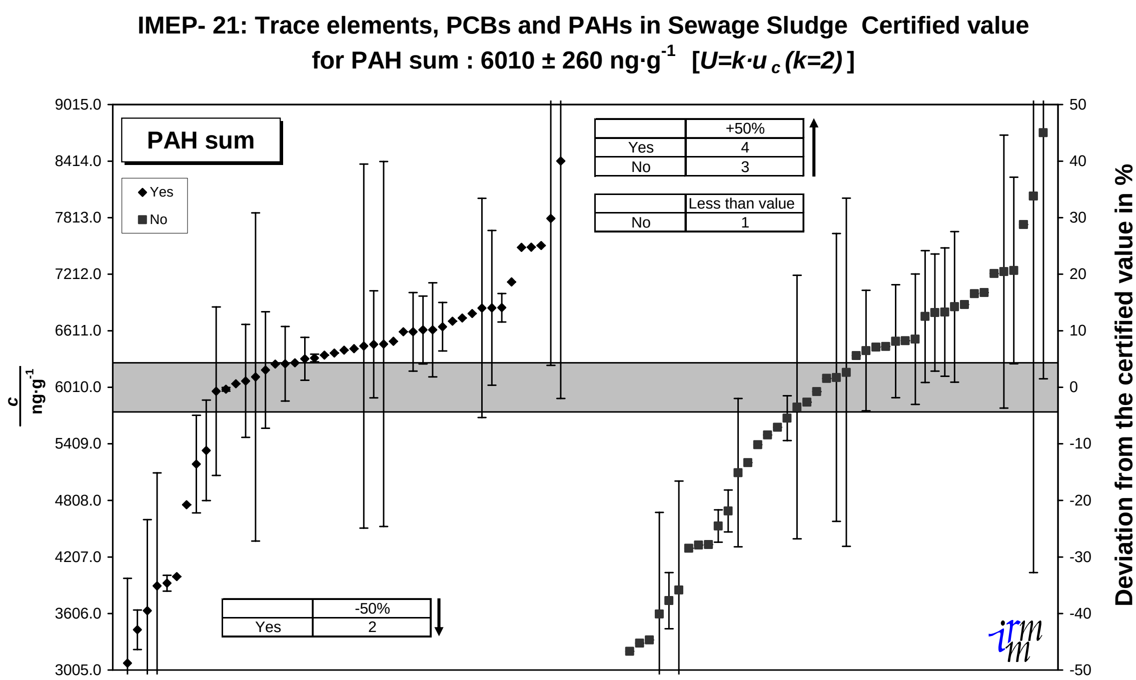


Figure 73



Results from all participants according to the use of any official method

Figure 74



Results for PAH sum from all participants according to the use of any official method

Figure 75

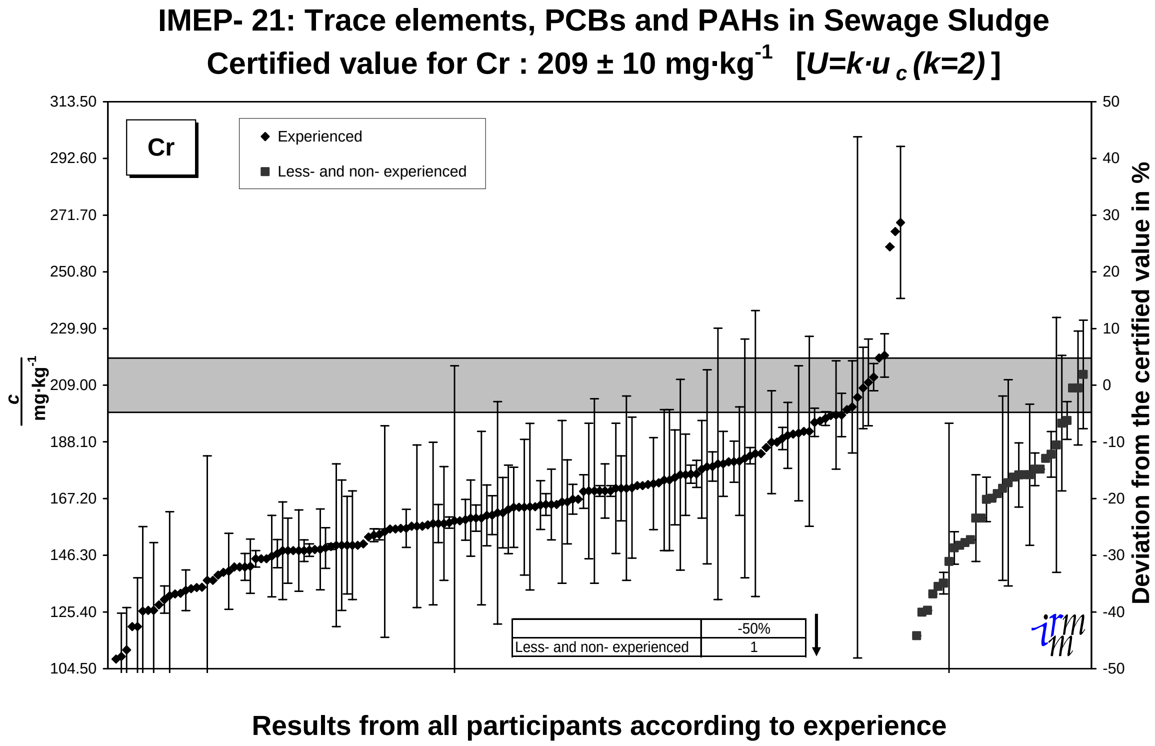


Figure 76

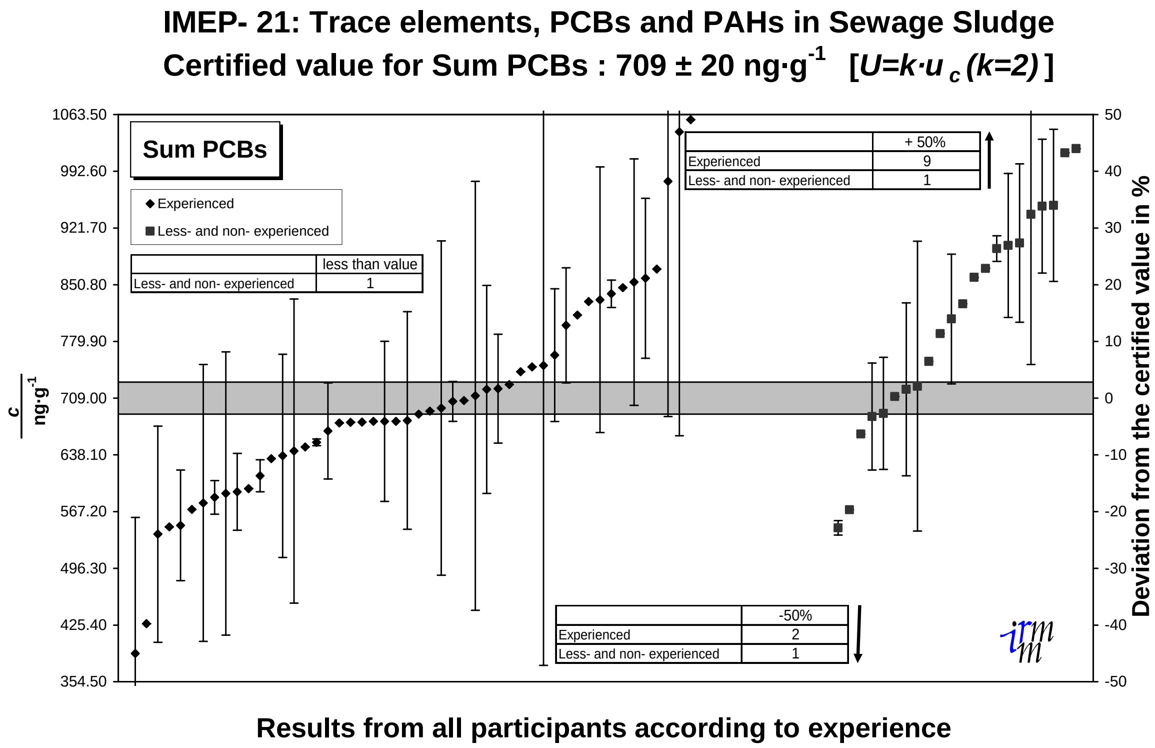
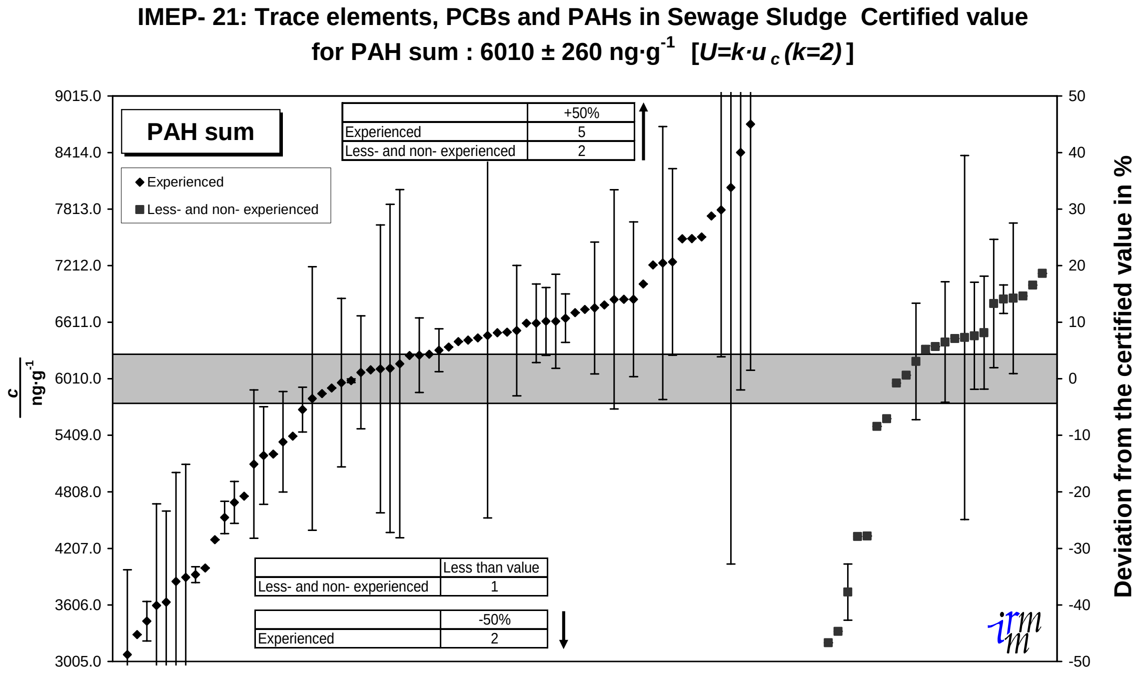


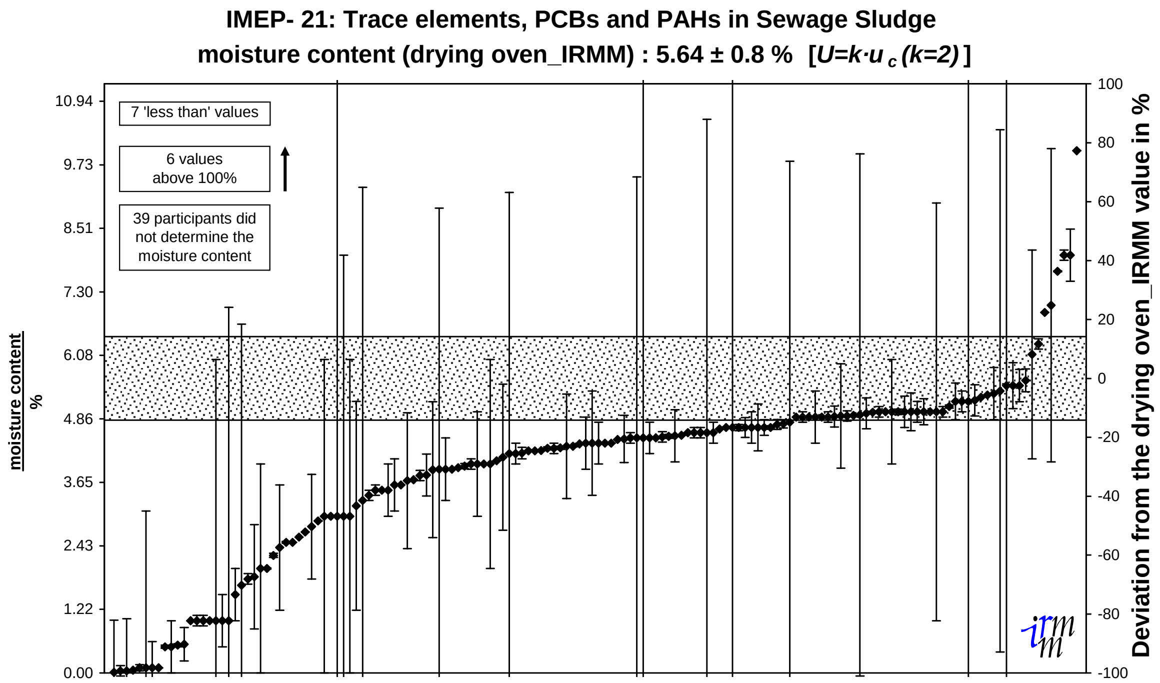
Figure 77



Results for PAH sum from all participants according to experience

Figure 78

IRMM emphasises that this is NOT a certified reference value for the moisture content in the CTS. This additional information is merely out of interest provided to the IMEP-21 participants.



Results for moisture content as reported by the IMEP-21 participants

Annex 2 – Additional information

Contents

IMEP-21: Trace Elements, PCBs and PAHs in Sewage Sludge

Annex 2 – Participants results – Tables

Table 1: Official method for inorganic analysis in IMEP-21

Official analytical method for inorganic analytes	Number of laboratories
APHA	1
Aqua regia digestion according to DIN 38414 S7 (specified in AbfKlärV)	1
ASTM,AOAC	1
Based on EPA 3050B	1
CMA	1
CMA/2/I/B.1 (cf. ISO 11885:1996) and CMA 2/II/A.3 (cf. EN 13656:2002)	1
CNR IRSA	1
Danish standard	1
Danish Standard (i.e. translated ISO/CEN)	1
Destruction according to NVN 5770 and analysis according to NEN 6426	1
DIN 38406 - E22, ¿SN EN ISO for individual elements	1
DIN EN 13346; DIN EN ISO 11885	1
DIN EN ISO 11885	2
DIN EN ISO 11885, Hg:DIN EN 1483	1
DS259, ICP	1
EN 13 346	2
EN 13804: 2002	2
EN 8288	1
EN ISO 11885	6
EN ISO 11885:1999	1
EN ISO 13346 and 11885	1
EN13656	1
EPA	1

Official analytical method

Official analytical method for inorganic analytes	**Number of laboratories**
EPA - 200.7, EPA 200.9, EPA 245.1	1
EPA 200.7 CLP-M	1
EPA 200.7, Standard Methods 3030E & 3120B and MOE3071	1
EPA 200.8 EPA 3051	1
EPA 3050A	1
EPA 3050B & EPA 200.8	1
EPA 3050B/200.7 for ICP-AES; and 7471A for Hg by CV-AAS	1
EPA 3050B-96+EPA 6010C-00	1
EPA 3051 + EPA 6010C	2
EPA 3051A	2
EPA 3052	1
EPA 6010	2
EPA 6010b, For Hg we used an internal procedure	1
EPA 7000 series	3
EPA 7131, 7190, 7210, 249.1, 7420, 7950	1
EPA and STANDARD METHODS FOR THE EXAMINATION OF WATER AND WASTEWATER	1
EPA-3052; ISO 11466; EPA-7473	1
ETHOS microwavw application N 0 E704, Nitric acid-Perchloric acid-Hydrofluoric acid digestion, 3030 I (Standard method for the examination water and w	1
For Cd, Cr, Cu, Ni, Pb and Zn following EPA method 3050. For Hg, based on EPA method 7471	1
Hg: NEN-ISO 16772; other NEN 6426	1
Hungarian Standard: MSZ 21470-50:1998.	1
In-House developed method based on EPA methods	1
IRSA CNR 3020	1
IRSA-CNR Quaderno 64, Metodo 10	1

Official analytical method for inorganic analytes	**Number of laboratories**
ISO	4
ISO 11047	2
ISO 11466	2
ISO 11885	6
ISO 8288	2
ISO 8288, EN 1233	1
ISO 8288; EVS-EN 1233; EVS-EN 1483	1
ISO AND ASTM	1
ISO-11885 and NS-4768 (Hg)	1
ISO14869-1	1
metals ISO 11885 (CMA2/I/B.1)	2
Italian Law (D.M. 13/9/99)	2
Italian Standard UNI 10780:1998, Appendix B (slightly modified)	1
M SZ	1
Marine Environmental Studies Labaoratory, Monaco	1
MEWAM Methods for the determination of metals in soils, sediments and sewage sludge.. ISBN 0 11 751098 1	1
MSZ 21470-50:1998	3
MSZ 21470-50:1998 (Hungarian Norm) and EPA 6010B	1
MSZ 318:1987, MSZ 21470-50:1998	1
National TNV 757440 (Hg), DIN 38406Teil 22 (metal)	1
NEN 6426:1995 nl	1
NEN 6966 and NEN-ISO 16772	1
NF EN 13346	1
NF EN 13346 - NF EN ISO 11885	1

Official analytical method for inorganic analytes	**Number of laboratories**
NF EN ISO 11885	1
NF EN ISO 11885 + NF EN 1483	1
NF EN ISO 11885, NF EN ISO 5961, NF EN 1483	1
PN-EN 13346, PN-EN ISO 11885, PN-EN 12338	1
PN-EN 13346: april 2002	1
PN-EN ISO 5961:2001; PN-EN 13346:2002; PN-ISO 8288:2002	1
SFS 5074:1990, in-house method based on SFS 5502:1990, Hg in-house method based on SFS-EN 1483:1997	1
SFS-EN ISO 15586,2004; SFS 3047, SFS 5074,	1
SM	1
SMEWW	1
SMEWW 3030	1
SS EN ISO 11885-1	1
SS028150-2	1
SS-EN ISO 11885-1 / SS-EN 13346	1
SS-EN ISO 15586:2003; SS-EN ISO 11885:1996	1
Standard Methods for Examination of Water and Wasterwater	4
STN/EN 13346	1
U.S. Environmental Protection Agency, Method 3051	1
US EPA	1
US EPA 200.2	1
US EPA 3050 modified, US EPA 6020 modified	1
US EPA SW-846 Method 6010B	1
US. EPA 3050B , 7471A	1
USEPA 200.2 Method	1

Official analytical method for inorganic analytes	Number of laboratories
USEPA 3050B -1996 and USEPA 3051:1994	1
USEPA 3051A, ISO 17294-1 and ISO 17294-2	1
USEPA3051, USEPA6020	1

Table 2: Official method for PCBs in IMEP-21

Official analytical method for PCBs	Number of laboratories
Abfall-Klärschlamm-Verordnung (AbfKlär-VO v. 15.4.1992)	1
CMA (VITO)	1
CMA 3/I	2
CMA3/I nEN5734	1
CNR-IRSA Q64/24B V3-88	1
Danish Srandard	1
DIN 38407-2	1
DIN 38414 S 20 and German sewage sludge ordinance	1
DIN 38414, Teil 20, DIN 37407, Teil 2,Din 51527, Teil 1, U.S. EPA Meth., 665 AA	1
DIN 38414-20	1
DIN 38414-20:1996-01	1
DIN 38414-S20	2
DIN 51527	1
ISO 10382:2002	5
EN ISO 6468	1
EPA 1668A	1
EPA 3540 B	1

Official analytical method

Official analytical method for PCBs	**Number of laboratories**
EPA 3545 + EPA 8270D	1
EPA 3550 / EPA 3665 / EPA 8082	1
EPA 8080	1
EPA 8082A	2
EPA 8270	1
EPA METHOD 3545	1
EPA-8082	1
European Standard Draft prEN 15308 Characterization of waste-CEN standard	1
Hungarien National Standard methods	1
In-House developed based on U.S. EPA methods	1
ISO	1
MSZ 21470-97:2002	1
MSZ 21470-98:2002	2
MSZ 21978-41:1999	1
NF EN ISO 6468, EPA 508, EPA 505, EPA 3546	1
NF XP X33-012	1
SNV3829	2
U.S. E.P.A. 3550A; 8080/8081	1
Unified analytical methods of Slovak environmental agency No. 150	1
US EPA 1668 B	1
USEPA 1668a	1
USEPA-8270	1
XP X 33-012	6

Table 3: Official method for PAHs in IMEP-21

Official analytical method for PAHs	**Number of laboratories**
CMA (VITO)	1
CMA 3/B	1
CNR-IRSA Q64/25 V3-90	1
Danish Srandard	1
DIN ISO 13877	1
EN ISO 17993, EPA 8310, EPA 5540	1
EPA	1
EPA 3540 B	1
EPA 3545 + EPA 8270D	1
EPA 3550	1
EPA 3550 / EPA 8270	1
EPA 550, EPA 3546	1
EPA 8270	4
EPA 8310	4
EPA METHOD 3545	1
EPA Method 610	1
EPA-8270	1
Hungarien National Standard methods	1
ISO	1
LUA-Merkblatt No.1 (Landesumweltamt Nordrhein-Westfalen)	3
LUFA	1
Method recommended from the danish environmental institute	1

Official analytical method

Official analytical method for PAHs	Number of laboratories
Method USEPA SW846 (3rd edition)	1
Methodenbuch VII des VDLUFA, VDLUFA-Verlag, Darmstadt	1
MSZ 21470-84:2002	2
MSZ 21978-40:1999	1
NEN 5771	1
NF XP X33-012	1
NS 9811	1
NVN 5731	1
ÖNORM L1200	1
PN-ISO 13877:2004	2
SNV 3829	2
SZME PAH BAC1M1	1
TNV 75 80 55 DIN 38 414 -23 I SO/FDIS 13877 US EPA Method 8310	1
U.S. E.P.A. 3550A;8270	1
Unified analytical methods of Slovak environmental agency No. 070	1
US EPA 3540 modified	1
USEPA SW846 8270	1
USEPA-8270	1
XP X 33-012	6

Table 4: CRMs in use by IMEP-21 participants

The CRMs uesd by the laboratories in IMEP-21	Number of laboratories
A.P.A.T (Enviromental Agency)	1
AROCHLOR 1260, 1248 (CHEM SERVICE), HIDROCARBUROS POLIAROMATICOS (CENAM), Cd, Cr, Cu, Hg, Pb, Zn (CENAM), Ni (Higt Purity)	1
BAM U 014 ; LGC 6113	1
BCR	1
BCR 088, LGC	2
BCR 143R, BCR145R	1
BCR 144 R	4
BCR 144R and NIST 2781 - 2782	1
BCR 144R- BCR 146R	1
BCR 145, Promochem	1
BCR 145R, BCR143R, BCR 146	1
BCR 145R CRM	2
BCR 146R IRMM	8
BCR 146R and 144R CRM	1
BCR -CRM 320 "S 3" - River sediment	1
BCR No146R, RM-7 (CZ)	1
BCR, Analytica CR	1
BCR, NBS	1
Be-1B - SCP Science	2
CRM 016-050 (sediment, no sludge)	1
CRM 029-050 from RTC (Resource Technology Corporation)	2
CRM 029-050, CRM 104-100 RTC	1

Certified Reference Materials

The CRMs uesd by the laboratories in IMEP-21	Number of laboratories
CRM 088 BCR (PAHs in Dried Sewage Sludge)	2
CRM 350 (River Sediment IRMM) - CRM-536 (Freshwater Harbour Sediment IRMM) - CRM104-100 (Contaminated Soil RTCorp.)	1
CRM 392, BCR	1
CRM 524,CRM535,CRM088, Riza1994A; Riza 1994B	1
CRM-ES Estuarian Sediment, CRM-RS-A River Sediment High Purity	1
CRMS-1 High Purity	1
Danish Water quality institut	1
EC1 supplier : National Water Research Institut, and LGC 6136 - supplier : BSI	1
EC-4 (National water research institute, Canada), IAEA-408 , CRM005-050	1
ERA 160B - Trace Metals, supplier Environmental Resource Associates	1
Estuarine sludge - Nist	1
Eurofins, QC Municipal Sludge	1
FAPAS	1
IRM-105 AND BDR-524	1
KS 96-10 wepal (marsep programme)	1
Laboratory of the Government Chemist, UK	2
LGC 6136	2
LGC 6137	1
LGC 6181	1
LGC 6182	1
LGC 6182, SRM 1944	1
Merck	1
National Institute of Standards & Technology - # 2782	1
NIST 2709	1

The CRMs uesd by the laboratories in IMEP-21	Number of laboratories
NWRI's EC-2, Analytika's No.7002	1
PCB Congeners in Soil - LGC, Gas works Contaminated Soil - LGC	1
QCT QUALITY CONTROL TECHNOLOGIES	1
RM 7004 Analytika	1
RTC	1
RTC 029-050	1
RTC CRM005-050 PROMOCHEM	1
RTC CRM031-040 Sewage Sludge	1
RTC CRM018-050 Metals on sewage sludge, BCR-CRM 459 PAH in coconut oil, BCR-536 Freshwater harbour sediment	1
Saint Joaquin Soil - LabserviceAnalytical	1
SETOC reference material from the Wageningen University	1
Sewage Sludge BE-1; SCP Science, 21800 Clark Graham, Baie d'Urfe', QC, Canada	1
Sewage Sludge CRM007-040, Dist by Promochem GmbH, Manufactured by Resource Technology Corp.	2
SRM ¿.12-3-13, SMU Slovakia	1
SRM 1944, NIST	1
VKI QC Municipal sludge A	2
VKI reference material	3
VKI-192-0595, RTC-CRM029-050,MBH Multielement Plasma Standard	1
VKI-27-1-0297; harbour sediment EC-4 (RM); RTC CRM018; PACS-1; HS-6 sediment (RM)	1
WT-L and WT-H, Slovak institute of metrology	3

Table 5: Proficiency testing schemes IMEP-21 laboratories participate in

Proficiency testing schemes	Number of laboratories
AARDE (VITO, Mol, BE), SETOC (WEPAL, Wageningen, NL), EU-SLUDGESUPPORT (Free University of Amsterdam, Amsterdam, NL), ...	1
Aarde, Vito; Fapas, KDLL, Vlarisub,	1
AGLAE	4
AGLAE - BIPEA	1
AGLAE, WEPAL	1
AIEA, GEMS, MEDPOL, CEEAE QUEBEC.	1
AMINAL/ OVAM/ VMM/ KIWA/ CHECK/ KDLL/ etc.	1
Análisis de Elementos Traza en solución acuosa - Instituto Nacional de Tecnología Industrial (INTI)	1
Analytical Product Group, Inc.; National Association of Testing Authorities, Australia; National Measurement Institute, Australia	1
APAT and UNICHIM	2
APAT IC001 (2004); UNICHIM POPs (5th, 6th, etc...) and COEPT Interlaboratory Ring Test; CIND (3th, 4th, etc)	1
AQS Baden-Württemberg, AQSL-Leitstelle Bayern	1
AQUACHECK	7
AQUACHECK, QUASIMEME, CALITAX, IAEA	1
Aquacheck, Relacre	5
ASLAB	5
ASLAB, CSLAB	2
CAEAL Proficiency Testing	5
CAEAL, NAPT, NY State (DOH), NWRI, Quasimeme, Umea, EPA, CFIA (note: not specific only to this matrix)	1
CALITAX	4
calitax - Labaqua	3

Proficiency testing schemes	Number of laboratories
CALITAX /Spain/ - waste water and sewage sludge, WEPAL /Netherlands/ - plant and soil	1
CALITAX-LABAQUA (from Spain), AQUACHECK (from United Kingdom)	1
CALITAX-LABAQUA (WATER, SOIL, SLUDGE, MICROBIOLOGY), AQUACHECK (WATER), IFA (WATER)	1
Canadian Association for Environmental Analytical Laboratories (CAEAL)	2
CCQM P39.1 IRMM	1
Centro Nacional de Metrología	2
Collaborative Trials, BIPEA	1
CONTEST; AQUACHECK	1
CSlab, Czech Republic	4
CZ: ASLAB - wastes, UKZUZ - sludges, soils, sediments	1
Dans le cadre de notre accréditation par le Centre d'expertise en analyse environnementale du Québec dans le domaine de l'eau	1
different proficiency testing organiser around germany	1
dioxins in food - Norwegian Inst Public Health; dioxins in soil/sediment - Orebro University, Sweden	1
DMU institute of the danish EPA	1
Eurofins	1
FAPAS	3
FAPAS and NARL	1
FAPAS, AACC, EU PT, SWEDISH NATIONAL FOOD ADMINISTRATION, CEN TC 327 ect.	1
FAPAS, LEAP	1
FEPAS, QM, AQUACHECK, HPA	1
Finnish Environmental Centre, once a year	1
IAEA Austria,APLAC,ASLAB Czech Republic,national scheme-VUVH Bratislava	1
IAEA intercomparison study for PCBs, petroleum hydrocarbons, metals	1

Proficiency testing schemes	Number of laboratories
IAEA436; IIS05A03; IFA Tulln	1
IAEA-MEL Monaco Trace elements in marine samples	1
ICHTJ Warsaw Poland-carrot;PK Crakow Poland-waste water;IMGW Wroclaw Poland-rain water;IMEP-19-rice;PIG Warsaw Poland-tea;PHARE-soil;	1
IFA Tulln, r-concept, IMEP, BAM	1
ILC-Waste water (National Institute of Chemistry, Slovenia); IFA-Tulln proficiency-testing (University of Natural Resources and Appl. Life Sciences)	1
IMEP, Aquacheck, DAP	1
IMEP12	1
IMEP-19; IMEP-20; FAPAS series 7 round 43; SWIFT-WDF (contract No SSPI-CT-2003-502492)round 1, round 2 ; QUA-NAS (GROWTH project G7RT-CT-2002-05110)	1
Intercal, CS-lab, QASIMEME, Labservice Italia	1
Intercalibrations organised by Finnish Environment Institute	1
International Proficiency test of Analytical Geochemistry Laboratories (GEOPT)	1
IRMM, IAEA, INRS, IRSID,	1
ISE, SETOC Programm, Wagheningen	1
ITM (SWEDISH), Eurofins	1
ITM, Sweden	4
JRC AQUACON-MedBas Subproject 10 (Metal determ. in Sewage Sludge & in Fly Ash) and NMI PT-scheme PAH on Diesel Particulates	1
Kiwa;Aquacheck;VLM;Vlarisub;VMM;KDLL;OVAM;VNA;IPH	1
Landesumweltamt	1
Latvian Environment, Geology and Meteorology agency	2
LGC Contest, Aquacheck, HSE Wasp, various ad hoc for dioxins/pcbs etc	1
Many National & International studies and Québec Proficiency testing for laboratory accréditation	1
MARSEP	1
Metals in drinking water, waste water, hazardous waste, soil, air, blood, urine, serum. PAH-s in edible oil.	1

Proficiency testing schemes	**Number of laboratories**
MPS - OH-01/03, Slovenská agentúra ¿ivotného prostredia (SR)	1
NATA	3
NATA (Australia) , APG (USA.)	1
NATA, NARL both from Australia	1
NEXT (DMU), NOVANA (DMU), Eurofins	1
NIVA, ITM, IMEP, QUA-NAS; SWIFT-WED, ...	1
NIVA, Norway	1
Organizer: Tartu Keskkonnauuringud (Tartu Environmental Researches)	1
Organochlorine pesticides organized by SZMD Belgrade	1
PAHs- UNICHIM - APAT	1
RIZA, Netherlands	3
Wageningen evaluating programmes for analytical laboratories, International soil-analytical exchange, Wageningen Agricultural University, Netherlands	2
PCB: FAPAS, QUALITYCONSULT; PAH: INCA, QUALITYCONSULT; METALS: QUALITYCONSULT, UNICHIM	1
Plant: WEPAL, Wageningen University, Netherland. Soil: FVM-NKTSZ, Hungary.	1
Proficiency test for the determination of trace elements (total) in soil and compost (RAS/2/11-2005), IAEA.	1
QualcoDanube	1
QualcoDanube interlaboratory comparison, WHO Collaborating Centre for Protection	1
QualcoDuna organised by VITUKI (Hun), Calitax (Esp), SWIFT-WFD (EU), QuaNAS (EU)	1
QUALITY CONTROL TECHNOLOGIES	1
QUANAS	5
QUANAS, VITUKI Intercalibration, Slovenian Waste water: MPP, SWIFT-WFD-PT	1
QUASIMEME	2
Quasimeme, IAEA, nordic intercomparisons	1

Proficiency testing schemes	Number of laboratories
r-concept	1
Ring tests	1
ring tests / VDLUFA; DAP; BAM; Bonner Enquete;	1
RING TESTS organized by UNICHIM (Italy) and APAT (Italy)	1
ring tests, e.g. BAM, LUA-NRW, AQS-BW etc	1
Ringversuch zur AbfKlärV, LfL Freising-Weihenstephan	1
RIZA, Netherlands	2
round robin test	1
SABS Inter Lab Comparisons on water samples	1
samples of ISE and SETOC by WEPAL	1
SETOC	1
Setoc (PAH, PCB), Quasimeme (PAH, PCB), NIVA (Zn, Cu, Cr, Ni, Pb, Cd, Hg)	1
SETOC (Wageningen University), Inter2000, Aquacheck, Leap Scheme	1
SETOC by WEPAL	1
SETOC, Wageningen University; NATA Australia.	1
We partecipate in APAT interlaboratory comparisons (Agenzia per la Protezione dell'Ambiente e per i Servizi Tecnici - Metrologia Ambientale)	2
Trace Elements in sewage sludge, Tartu Environmental Research Ltd. (Estonia);. Metals in sludge, ITM Stockholm university	1
UKZUZ Brno, Czech Republic	1
UMEA	1
UNICHIM	1
UNICHIM; COEPT; APAT	1
Vito (AARDE); Setoc (wageningen university)	1
VITUKI; FAPAS; AQACHECK AND IFA-TULLN	1

Proficiency testing schemes	Number of laboratories
Waganingen Univercity, Calitax	1
Wageningen evaluating programmes for analytical laboratories, International soil-analytical exchange, Wageningen Agricultural University, Netherlands	1
Water Anbalysis/Department of Science Service, Flour PT Programme/APLAC	1
Water, soil, sediment, sludge; Finnish Environment Institute (SYKE)	1
WEPAL (ISE, IPE)	1
WEPAL ISE and WEPAL SETOC	1
WEPAL, SETOC	1
WEPAL,UKZUZ	1
WEPAL-ISE/ Netherland; GEOPT/ Open University, UK	1

Table 6: Evaluation of measurement uncertainty not according to ISO-GUM or/and EURACHEM guidelines

Evaluation of measurement uncertainty in case the guides for quantifying uncertainty (ISO/EURACHEM) were not followed
American Water and Wastewater Examinations / EPA
Analysis of a quality control sample within each batch of samples
As standard deviation of measurement
Based on replicate analysis of CRM's and taking into account the bias
Based on results of daily quality control samples (own or CRMs if available) and instructions of Finnish Environment Centre and FINAS
berechnet aus Standardabweichungen
By combining relevant bias and within-lab reproducibility data, obtained during the method validation and/or QC programme
By CRM measurement
calculated according to the international standards used
Certified reference material, duplicate, standard spiking, procedural blank, surrogate standards
CMA

Evaluation of measurement uncertainty in case the guides for quantifying uncertainty (ISO/EURACHEM) were not followed
CMA (VITO)
CMA-method
Control cards for the last 20-100 controls or natural samples
El calculo de la incentidumbre se encuentra en proceso de acuerdo a las guias tecnicas de la Entidad Mexicana de Acreditacion
empiric
Expert jugdement...
From sample and quality assurance data
generally from performance data for replicate measurements of CRMs
internal procedure
like the Belgium CMA: uncertainty = 2*R + absolute bias
Mean value control chart
Ministry of Environment Protocol using Type A data
Nordquest
Nordtest Technical Report 537, 2004
Norme AFNOR XP T 90-220 Aout 2003
reproductibility and repetability test
standard deviation
Standard Methods for Examination of Water and Wastewater - 20 th
(stdev/average)*2
2xStd dev of 5 replicates
t*RSD/n-1/2 t=2.776 n=5
+- t*SD/sqr(n)

Evaluation of measurement uncertainty in case the guides for quantifying uncertainty (ISO/EURACHEM) were not followed
the measurment uncertainty was the statistical error and the fit error given by the GUPIX software
The relative standard deviation was calculated using three replicate analysis (Three independent measurement).
The uncertainties have been calculated with the french norm XP T 90-220 (august 2003)
There is no normalisation guide for the measerurement uncertainty in Holland (in develepment)
They are remainings from older days where a conservative general judgement was done
Uncertainties were calculated based mainly on repetabilities values generated from our regular quality control samples.
uncertainty in the opr values
uncertainty is based on known value samples
uncertainty is calcolated like two times reproducibility standard deviation of interlaboratory trial test (this laboratory partecipated positively.
Validation (parallel proves, 0-proves, controll-proves...)
Validation of the method
validation report
We evaluated according to the standard reference and according to our QC
Whith the results of external quality control
With the help of control charts.
With the soil control graphics board

Table 7: Replies per participant to sample digestion and acid mixture applied for metals analysis

Analyte	Digestion method	Acid mixture
all	acid mixture digestion	HNO3 : HCL (1:3)
all	aqua regia	HCl/HNO3 3:1
all	AQUA REGIA	NITRIC ACID / HYDROCHLORIC ACID
all	Aqua regia digestion according to DIN 38414 S7, open vessel procedure	21 ml HCl, 7 ml HNO3
all	Autoclave	HNO3/water; 1/1
all	autoclave	Nitric acid
all	Autoclave press	Nitric acid
all	Autoklav	nitric acid
all	Block heater	Aqua Regia
all	Closed microwave digestion system	aqua regia
all	Digested with repeted additions of nitric acid (HNO3) and hydrogen peroxide (H2O2) in hot plate	acid nitric concentrated
all	digestion in teflonvessel	HNO3+H2O2
all	Digestion on hotplate	Nitric/hydrochloric
all	DigiBloc 3000	50% Nitric & 30 % Hydrogen Peroxide
all	Heating and digestion during 12 hours	HCL: HNO3
all	high pressure ashing	cc.HNO3 and cc.H2O2
all	high pressure ashing	HCl + HNO3
all	high pressure ashing	HNO3/H2O2
all	High pressure ashing	Nitric acid
all	high pressure ashing	nitro hydro chloric acid

Analyte	Digestion method	Acid mixture
all	Hot Block Digestion	Nitric Acid and Hydrochloric Acid
all	Hot plate	Aqua regia
all	hot plate cllasical analitical method	Nitric acid-Perchloric acid-Hydrofluoric acid
all	hotblock at 105 C	Aqua Regia
all	Hotblock digestion	Nitric acid, hydrogen peroxide, and Hydrochloric acid
all	HOT-PLATE	HNO3-HCl
all	microonde	HCl-HNO3 (3:5)
all	microwave	5 ml H2O + 10 ml HNO3 65% + 3 ml HCl 32%
all	Microwave	5:2 H2O2:HNO3
all	microwave	6ml HF + 1ml HNO3
all	microwave	7 M HNO3
all	microwave	Aqua Regia
all	microwave	aqua regia
all	microwave	aqua regia
all	microwave	aqua regia
all	microwave	aqua regia
all	microwave	aqua regia (2.5 mL HNO3 + 7.5 mL HCL)
all	microwave	eau régale
all	microwave	HCl - HNO3 - HF - H3BO3 (6 - 2 - 2 - 3)
all	Microwave	HCl/HNO3
all	microwave	HCl/HNO3 3:1
all	microwave	HCL/HNO3/HF
all	microwave	HCl:HNO3 (3:1)

Analyte	Digestion method	Acid mixture
all	microwave	HCl:HNO3=3:1
all	microwave	HCl+ HNO3 (aqua regia)
all	microwave	HF and HNO3
all	MICROWAVE	HF/HNO3/HCL
all	microwave	HNO3
all	Microwave	HNO3
all	microwave	HNO3
all	microwave	HNO3 (5ml) and H2O2 (0.5ml)
all	microwave	HNO3 : HCl, 2 : 5
all	microwave	HNO3 + HCl
all	MICROWAVE	HNO3 + HCL
all	microwave	HNO3 + HCl
all	microwave	HNO3 + HCl + H2O2 (1:3:1)
all	microwave	HNO3 and HCl and HF
all	microwave	HNO3 and HF
all	microwave	HNO3, HCl, HF
all	MICROWAVE	HNO3, HCl, HF
all	microwave	HNO3/HCl
all	microwave	HNO3/HCl
all	microwave	HNO3:HCl (1:3)
all	microwave	HNO3+HCLO4
all	MICROWAVE	NITRIC + PEROXID
all	Microwave	nitric acid

Analyte	Digestion method	Acid mixture
all	microwave	nitric acid
all	microwave	nitric acid - hydrochloric acid
all	microwave	nitric acid + peroxide
all	microwave	nitric acid en hydrochloric acid
all	microwave	nitric acid, hydrogen peroxide
all	MICROWAVE	NITRIC ACID/H2O2/HCl 5/1/1
all	microwave	Nitric/sulphuric
all	microwave	yes
all	microwave (closed bomb)	HNO3/HCl/HF
all	Microwave Acid Digestion(temperature controlled)	5mL Nitric Acid(70%) and 1mL Perchloric Acid(60%)
all	microwave assisted (closed vessel)	HCl, HNO3; HF
all	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils	Nitric acid and Hydrochloric acid
all	microwave digestion	HNO3
all	microwave digestion	HNO3/HCL 67/33
all	Microwave, closed wessel	50% v/v HNO3
all	microwawe	conc. HNO3 (8mL) + 30% H2O2 (1 mL)
all	NF EN 13346 (Micowaves)	HNO3/ HCl
all	nitrohydrochloric acid	nitric acid, hydrochloric acid
all	open vessel hot block	nitric and peroxide
all	refluction	aqua regia
all	Reflux on digestion heating block	1:1:5 Nitric Acid:Hydrochloric Acid:Water
all	refluxing for 2 hours	21 ml HCl and 7 ml HNO3
all	Säureaufschluss 130°C; 2 Stunden	Königswasser

Analyte	Digestion method	Acid mixture
all	Tecator Heat mantel 120 degrees C	Aqua Regia
all	Thermal heating digestion in reflux system	Aqua regia (1HNO3 + 3HCl)
all	US EPA 3050 B	chloric acid and nitric acid
all	Wet digestion	HNO3
all	Wet digestion method	HNO3 , HCL , HCLO4
all	wet-ashing	conc. HNO3
Cd	microwave; filtration of the precipitate	HNO3+HF
Cd, Cr, Cu, Hg, Pb, Ni	minerisation plaque	HNO3, HCl
Cd, Cr, Cu, Ni, Pb, Zn	wet digestion - EPA SW 846 Method 3050	HNO3 + HCl
Cd, Cr, Cu, Ni, Zn	digestão ácida	água régia
Cd, Cr, Cu, Pb, Hg, Zn	MICROWAVE	HNO3/HF
Cd, Cr, Cu, Pb, Ni, Zn	acid digestion	HCl:HNO3 (3+1)
Cd, Cr, Cu, Pb, Ni, Zn	Acid digestion of Ash	HCl:HNO3
Cd, Cr, Cu, Pb, Ni, Zn	Acid Digestion on Hotplate	Nitric acid,Perchloric Acid
Cd, Cr, Cu, Pb, Ni, Zn	aqua regia	HCL:HNO3 (3:1)
Cd, Cr, Cu, Pb, Ni, Zn	ashing	nitric acid
Cd, Cr, Cu, Pb, Ni, Zn	autoclave digestion	HNO3
Cd, Cr, Cu, Pb, Ni, Zn	boiling acid	nitric acid and percloric acid
Cd, Cr, Cu, Pb, Ni, Zn	boiling under normal pressure	HNO3 + HCl
Cd, Cr, Cu, Pb, Ni, Zn	Chauffage à ébulition	HCl - HNO3
Cd, Cr, Cu, Pb, Ni, Zn	Closed teflon bomb digestion	HF,HNO3,HClO4
Cd, Cr, Cu, Pb, Ni, Zn	dry ashing at 450°C	HF, HClO4, HNO3
Cd, Cr, Cu, Pb, Ni, Zn	EN 13 346 part 8.2	aqua regia

Analyte	Digestion method	Acid mixture
Cd, Cr, Cu, Pb, Ni, Zn	extraction procedure under reflux conditions	agua regia
Cd, Cr, Cu, Pb, Ni, Zn	Furnace at 520 degrees C	Nitric acid
Cd, Cr, Cu, Pb, Ni, Zn	Graphite Heating Block wells with digital temperature control	HNO3/HCl (EPA 3050B)
Cd, Cr, Cu, Pb, Ni, Zn	Heat plate	HNO3 and HCl
Cd, Cr, Cu, Pb, Ni, Zn	high pressure	7M HNO3
Cd, Cr, Cu, Pb, Ni, Zn	high pressure	HNO3,HCl
Cd, Cr, Cu, Pb, Ni, Zn	High pressure digestion (autoclave)	HNO3
Cd, Cr, Cu, Pb, Ni, Zn	High pressure, 120'C (autoclave)	Nitric acid (1:1, about 33 %)
Cd, Cr, Cu, Pb, Ni, Zn	high temperature	HCl, HNO3
Cd, Cr, Cu, Pb, Ni, Zn	Hot Block Digestion @ 95 Celcius	HCL & Nitric Acids, 3:1 ratio
Cd, Cr, Cu, Pb, Ni, Zn	Hot digestion at atmosheric pressure	Nitric and perchloric acids
Cd, Cr, Cu, Pb, Ni, Zn	Hot plate	HNO3 / HCl 15:10
Cd, Cr, Cu, Pb, Ni, Zn	Hot Plate	Nitric and HF
Cd, Cr, Cu, Pb, Ni, Zn	Hot plate (NF X 31-147)	HClO4, HF
Cd, Cr, Cu, Pb, Ni, Zn	hot plate digestion	nitric
Cd, Cr, Cu, Pb, Ni, Zn	ISO 11466 digestion (open flask)	HCl+HNO3 (3:1)
Cd, Cr, Cu, Pb, Ni, Zn	micorwave or boiled with acid	HCL and HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	1st step:HNO3+ H2O2; 2nd step: HNO3+HCl
Cd, Cr, Cu, Pb, Ni, Zn	microwave	3ml HCl and 9ml HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	4mL HNO3 + 1mL HCl
Cd, Cr, Cu, Pb, Ni, Zn	microwave	5 ml HNO3 + 5 ml HCL.
Cd, Cr, Cu, Pb, Ni, Zn	microwave	6 ml of HCl + 2 ml HNO3 + 2 ml HF
Cd, Cr, Cu, Pb, Ni, Zn	microwave	aqua regia

Analyte	Digestion method	Acid mixture
Cd, Cr, Cu, Pb, Ni, Zn	microwave	Aquaregia
Cd, Cr, Cu, Pb, Ni, Zn	Microwave	Concentrated HNO3 5 ml
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HCl and HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HCl+HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HCl-HNO3-HF-Boric acid
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HF 2 mL + AQUA REGIA 8 mL + H2O2 1 mL 2°step 3mL sol sat H3BO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HF HNO3 H2O2
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HF, HNO3
Cd, Cr, Cu, Pb, Ni, Zn	Microwave	HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HNO3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HNO3 + H2O2
Cd, Cr, Cu, Pb, Ni, Zn	Microwave	HNO3 +H2O2
Cd, Cr, Cu, Pb, Ni, Zn	Microwave	HNO3/HCl (1/3)
Cd, Cr, Cu, Pb, Ni, Zn	Microwave	HNO3:HCl = 1:3
Cd, Cr, Cu, Pb, Ni, Zn	microwave	HNO3:HCl 3:1
Cd, Cr, Cu, Pb, Ni, Zn	microwave	nitric
Cd, Cr, Cu, Pb, Ni, Zn	microwave	nitric and hydrochloric acid
Cd, Cr, Cu, Pb, Ni, Zn	microwave (temterature program (250-600W) end temperatur 180 C	aqua regia (3.5 ml)
Cd, Cr, Cu, Pb, Ni, Zn	microwave digestion,Anton Paar Multiwave 3000	concHNO3 and H2O2
Cd, Cr, Cu, Pb, Ni, Zn	Microwave oven	aqua regia digestion
Cd, Cr, Cu, Pb, Ni, Zn	microwave, high pressure	HNO3, HCL

Analyte	Digestion method	Acid mixture
Cd, Cr, Cu, Pb, Ni, Zn	nitric acid and chlorid acid digestion	7 ml nitric acid, 21 ml chloric acid
Cd, Cr, Cu, Pb, Ni, Zn	no	HNO3 + HCl
Cd, Cr, Cu, Pb, Ni, Zn	open vessel	HF+HNO3+HClO4
Cd, Cr, Cu, Pb, Ni, Zn	pressure bomb	HNO3+H2O2
Cd, Cr, Cu, Pb, Ni, Zn	Reflux	Aqua regia
Cd, Cr, Cu, Pb, Ni, Zn	Reflux in covered beaker on thermostatically controlled hotplate	3:1 Hydrochloric acid:Nitric acid (Aqua Regia)
Cd, Cr, Cu, Pb, Ni, Zn	Wet acid digestion	HNO3+HF+HClO4
Cd, Cr, Cu, Pb, Ni, Zn	Wet acid open digestion	Nitric acid, Hydrochloric acid and Hydrogen Peroxide
Cd, Cr, Cu, Pb, Ni, Zn	Wet digestion	Nitric acid ,Hydrochloric acid ,Hydrogen peroxide
Cd, Cr, Cu, Pb, Ni, Zn	wet digestion	nitric acid and hydrochloric acid
Cd, Cr, Cu, Pb, Ni, Zn	wet digestion in closed system for 3 hours at 105 C	HNO3 and H2O2
Cd, Cr, Cu, Pb, Ni, Zn	wet digestion,open system	aqua regia
Cd, Cr, Cu, Pb, Zn	micrwave	hno3
Cd, Cr, Cu, Zn	dry ashing	HNO3
Cd, Cu, Pb, Ni, Zn	microwave	nitric acid
Cd, Cu, Pb, Ni, Zn	real total	HNO3+HF
Cd, Cu, Pb, Zn	ashing method	0.025NH2SO4 IN 0.5 In HCL 5DOUBLE ACIDE°
Cd, Cu, Pb, Zn	High pressure	nitric + hydrochloric
Cd, Cu, Zn	MICROWAVE DIGESTION - METHOD 3051 EPA	NITRIC ACID CONCENTRATED
Cr	ashing	Na2O2+HCl
Cr, Hg	microwave; filtration of the precipitate	aqua regia
Cu, Pb, Ni, Zn	acid digestion	HNO3+HCl
Cu, Pb, Ni, Zn	microwave; filtration of the precipitate	HNO3 + HF

Analyte	Digestion method	Acid mixture
ETGL1452233	bain marie	4 ml HNO3 + 2 ml H2SO4
Pb, Zn	microwave	HCl/HNO3
Hg	acid digestion	HNO3
Hg	Acid digestion of sample	H2SO4
Hg	atmospheric reflux	HNO3+HCl
Hg	autoclave digestion	HNO3+KMnO4
Hg	boiling	HNO3+H2SO4
Hg	Closed teflon bomb digestion	HNO3:HCl=1:1
Hg	Graphite Heating Block wells with digital temperature control	HCl/HNO3/KMNO4/NH2OH.HCl
Hg	HEATING IN A WATER BATH - METHOD 7471 EPA	WATER PURIFIED / AQUA REGIA / POTASSIUM PERMANGANATE / SODIUM CHLORIDE-HYDROXILAMINE SULFATE / STANNOUS SULFATE
Hg	Hot Block	H2SO4/HNO3 (4:1)
Hg	Microwave	HCl
Hg	microwave	HNO3 H2O2
Hg	microwave	HNO3/H2SO4
Hg	microwawe digestion	HNO3 and H2O2
Hg	Minéralisation à froid	Permenganate
Hg	NF EN 13346	HCl/HNO3
Hg	Niric acid digestion without ashing	Nitric acid
Hg	open digestion	H2SO4, HNO3
Hg	Open vessel	0.2mL HF / 2mL HCl / 1 mL H2SO4 / Final volume 50 mL
Hg	oven	hydrochloric acid : nitric acid 3:1 (v:v)
Hg	Over night at room temperature, then one hour at 50 degrees C	HNO3

Analyte	Digestion method	Acid mixture
Hg	Room temperature	HCL and HNO3
Hg	table top digestion	nitric/sulphuric
Hg	Test tubes in bored graphite heating block	3:1 Hydrochloric acid:Nitric acid (Aqua Regia)
Hg	Water bath	HNO3 / H2SO4 2.5: 1.25
Hg	Water-bath (90ºC)	2.5mL HNO3 + 5 mL H2SO4
Hg	Wet acid digestion	HNO3+H2SO4
Hg	Wet acid microwave digestion	Nitric acid
Hg	wet ashing	HCl+ HNO3
Hg	Wet digestion	H2SO4 + HNO3
Hg	wet digestion	nitric acid and sulfuric acid
Hg	AMA 254	non
Hg	Burned with oxygen (then AAS); Lego	nothing
Hg	Direct ashing, trapping	none
Hg	direct measurement	without acids
Hg	direct mercury analysis	no acid used
Hg	directly method - TMA	no
Hg	DMA analysis, no digestion	none
Hg	dry ashing	direct determination
Hg	Furnace and Catalyst in Mercury Analyzer	Not used
Hg	no	no
Hg	no	no
Hg	no digestion	no acid (direct measurement with mercury analyzer)
Hg	no digestion	no digestion

Analyte	Digestion method	Acid mixture
Hg	no digestion -	none
Hg	No digestion, direct mercury analysis	no acid
Hg	no digestion, from solid sample	without acids
Hg	without digestion	none
Hg	without digestion	none

Table 8: Percentage of participants who applied a separation and/or pre-concentration step

Analyte	separation	pre-concentration
Cd	12.58%	2.07%
Cr	12.08%	2.10%
Cu	12.50%	2.05%
Hg	19.23%	6.35%
Ni	12.33%	2.80%
Pb	12.16%	2.78%
Zn	12.58%	2.74%

Annex 3 – Documentation

Contents

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
Joint Research Centre

IMEP®

Geel, 24 January 2005
IM/L/09/05

International Measurement Evaluation Programme

IMEP-21 Trace elements, PCBs and PAHs in Sewage Sludge

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, fish 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. 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-21 interlaboratory comparison that focuses on the analysis of total amount contents of Cd, Cr, Cu, Hg, Ni, Pb, Zn, PCB's and PAH's in sewage sludge. The CTS is available in glass bottles containing 40g of the sewage sludge.

The analytes under investigation with nominal values in IMEP-21 are:

For metals	Hg	0.5-10 mg/kg	Cu, Pb	500-1000 mg/kg
	Cd	5-50 mg/kg	Zn	1000-5000 mg/kg
	Ni, Cr	100-500 mg/kg		
For PCB's	28,52,101,118,153,180	100-500 ng/g each		
For PAH's	sum PAH(11)	6000 ng/g		

In the framework of a collaboration between IRMM and CEN the participation in IMEP-21 of laboratories nominated by the Euromed Quality co-ordinator, Ms. Elisa Setién, is free of charge. (IRMM also covers for the dispatching costs).

Registration deadline will be **strictly** 21st February 2005. The samples will be dispatched in the beginning of April. Deadline for reporting results would be 20th June 2005. As a first feedback, the reference values for the analytes under investigation in the sludge material will be available on the IMEP website in July 2005. Individual certificates will be issued in September 2005. Participants' reports will be available in autumn of 2005.

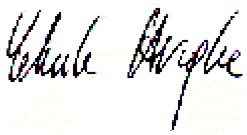
For participation in this IMEP-21 interlaboratory comparison, please register using the following on-line registration link for nominated participants from the MEDA region.

http://www.irmm.jrc.be/imepapp/registerForComparison.action?comparison=62

On registration, you will be asked to print the registration form and return it to IRMM.

With your participation in IMEP-21 you agree that your reported results will be forwarded to the Euromed Quality co-ordinator for the MEDA programme, Ms. Elisa Setién. For the rest your results will be strictly confidential, though.

Yours sincerely



IMEP-21 Co-ordinator
IM Unit – IRMM

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EUROPEAN COMMISSION
DIRECTORATE-GENERAL
Joint Research Centre

Institute for Reference Materials and Measurements

Geel, 19th September 2005
IM/L/23/05

IMEP-21: Trace elements, PCBs and PAHs in Sewage Sludge

Dear «title» «surname»,

Thank you very much for your participation in our interlaboratory comparison.

Together with this letter you will find the sample confirmation form. May we ask you to return this form immediately to IRMM, so that we know if you received the package in good order.

This IMEP-21 interlaboratory comparison involves the determination of Trace elements, PCBs and PAHs in Sewage Sludge. The Certified Test Sample is in an amber glass bottle containing about 40 gram sewage sludge powder.

Trace Element	mg/kg
Cd	5-50
Cu	500 - 1000
Cr	100 - 500
Hg	0.5 - 10
Ni	100 - 500
Pb	500 - 1000
Zn	1000 - 5000

PCB	ng/g
28	100 - 300
52	100 - 300
101	100 - 300
118	100 - 300
153	100 - 300
180	100 - 300
Sum PCBs	**600 - 1800**

PAH
anthracene
benz(a)anthracene
benz(a)pyrene
benz(b+k)fluorantene
benzo(ghi)perylene
fluoranthene
fluorene
indeno(1,2,3-cd)pyrene
Naphtalene
phenanthrene
pyrene
Sum PAHs: 6000 – 7000 ng/g

Deadline for reporting the results and returning the completed questionnaire is **30th November 2005**. A first feedback, concerning the IMEP-21 reference value, is foreseen for December 2005 on our website (www.imep.ws). Individual certificates will be issued early 2006 and the report will be made available in spring 2006.

Result reporting will be done electronically via the IMEP web-site. The result reporting-login is accessible via the url-link http://www.irmm.jrc.be/imepapp/jsp/loginResult.jsp . The url-link is also accessible from our website. Enclosed you will find some guidelines about how to report your results you will find these guidelines also via the website.

You have been allocated a personal code, the 'Password Key', for the on-line reporting of your results. Please fill in this number when requested when you are connected to the on-line reporting page.

Your Password Key = «participation_key»

When you have submitted your results and questionnaire information, you will be prompted to **print** the result report form. The paper version need to be returned **signed** to IRMM.
Please check your results carefully for any errors before submission. In case you need to adjust any of your results, please contact us on the following address: jrc-irmm-imep@cec.eu.int or by fax to the following number:+32 14 571 865. After result reporting deadline, no amendments of results are accepted anymore.

If you have any questions or problems, please do not hesitate to contact us.
Yours sincerely,

Dr. Y. Aregbe
IMEP-21 Co-ordinator, IRMM JRC

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«title» «firstname» «surname»
«companyinstitute_»
«department»
«address1»
«address2»
«address3» «address4»
«zip» «town»
«country»

IMEP-21

Trace elements, PCBs and PAHs in Sewage Sludge

Confirmation of receipt of the IMEP-21 Sewage Sludge samples

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).

REMARKS ?…………………………….

Date of package arrival:……………………..

Signature:……………………………………

Please return the form to:
Dr. Y. Aregbe
IMEP-21 Co-ordinator
DG-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium
Fax : +32 (0) 14 571 865
e-mail : yetunde.aregbe@cec.eu.int

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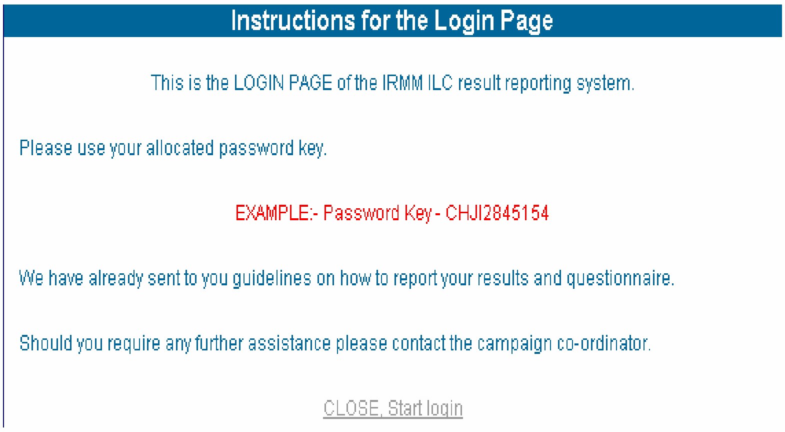
We are pleased to advise that the IMEP® online reporting system is now operational. These guidelines will explain how you can input your measurement result with uncertainty and how to enter the questionnaire information.

The result reporting is done on the Internet, the login page is located using the following URL

http://www.irmm.jrc.be/imepapp/jsp/loginResult.jsp

The following information page will appear. To obtain the login page, close down this screen.



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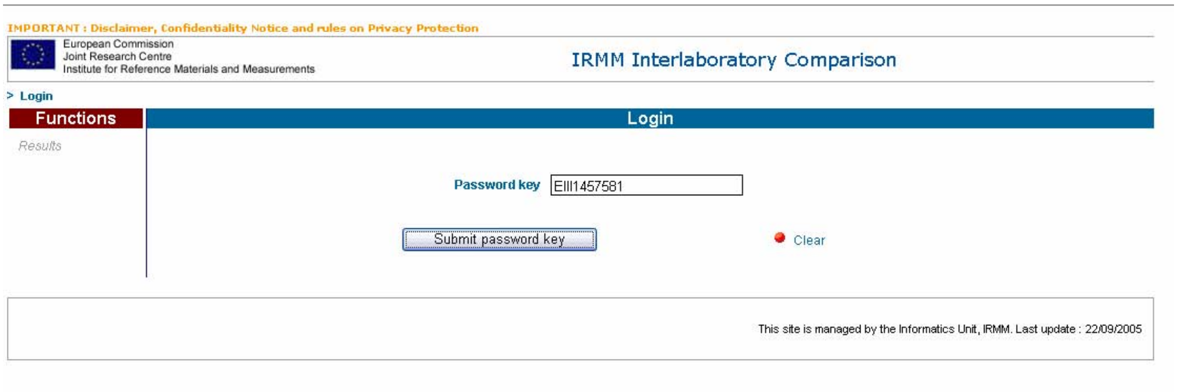
This is the login page.



Attention: The results must be reported as **dry mass** corrected values.

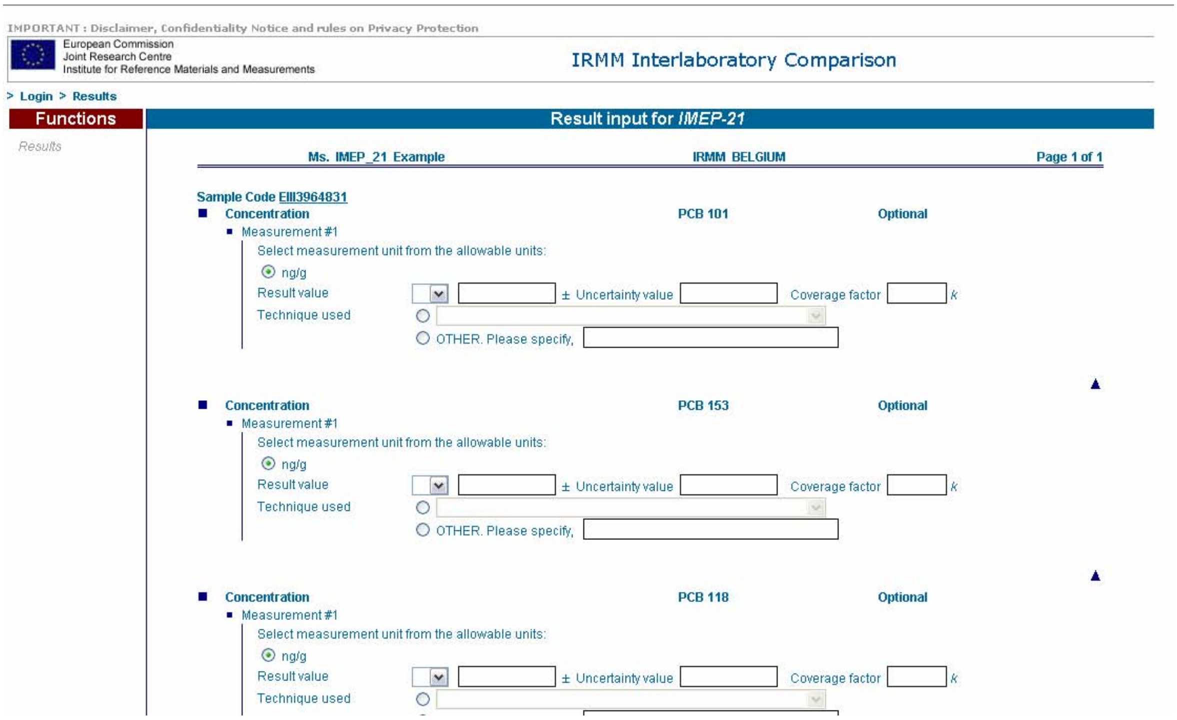
Please use your allocated password key, which was sent to you on the second page of the accompanying letter together with the sample.

EXAMPLE:- Password Key – ABCD1234567

Once you have entered your password key, press the *SUBMIT* button

(Please note that your password key is unique to the comparison you have registered to.)

The RESULT REPORT FORM



Completing the RESULT REPORTING Page.

1. The analytes are listed alphabetically, you will see first all the PCB measurements, then the PAH measurements and then the Trace Element measurements.

2. The unit of the results must be in ng/g dry mass for the PCB and PAH measurements and in mg/kg dry mass for the Trace Element measurements.

3. In the field marked "Result value" enter your measurement result using the 2nd box to your left. You only need to put in results for the analytes you have measured. (attention : the results must reported as dry mass corrected values)

If you need to report an upper limit as a result you will have to select the "<" from the drop down menu. (1st box from your left) Please be aware that the uncertainty field will now be disabled, so no input can be entered.

3. In the field marked "Uncertainty value" enter your measurement uncertainty. If you have not estimated an uncertainty for your result you will have to leave this field blank.

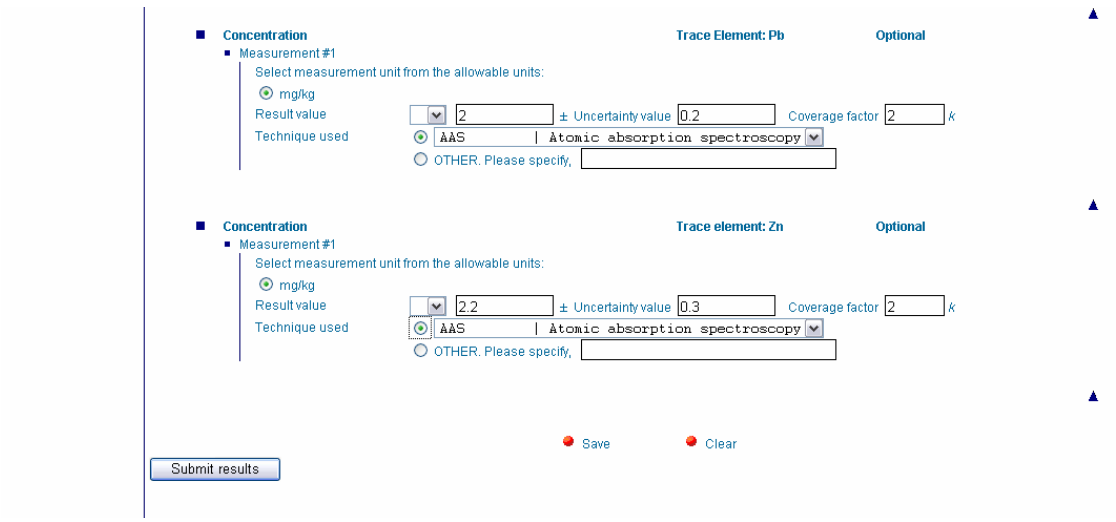
4. Input the coverage factor.

5. Select the field marked "Technique used" this will activate the drop down menu. Select the technique used. If the technique used is not listed, select the "OTHER" field and then specify.

When the "Technique used" field has been selected the "OTHER" field is disabled and no input can be entered.

Likewise should you select the "OTHER" field then the "Technique used" field is disabled.

Below is an example of a completed result screen.

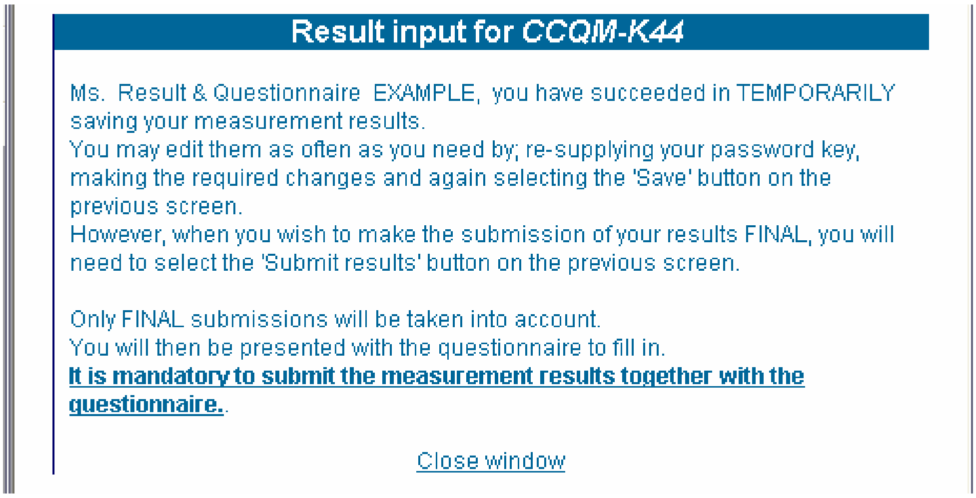


At this stage you can choose to **SAVE** your results or **SUBMIT** them.
SAVE -To SAVE your results press on the *SAVE* button, this will SAVE the data entered with the possibly to edit them as often as you need.

To reconnect to our system use the same URL link http://www.irmm.jrc.be/imepapp/jsp/loginResult.jsp and re-supply your password key. The result form will appear with the data that has already been entered. Make the required changes and select either the *SAVE* button or the*SUBMIT* button.

Remember to submit your results before the deadline date, as ONLY submitted results will be accepted.

This pop up page will appear advising you that your results have been saved.



How to SUMBIT your results

Once the *SUBMIT* button has been pressed, the questionnaire will appear ready for your input

The QUESTIONNAIRE FORM

Completing the QUESTIONNAIRE Page.

1. You must enter or select data to every question, otherwise your questionnaire information will not be submitted. Should you not complete a question or complete a question incorrectly a message will appear directing you to that relevant question. If you do not answer to a question you cannot submit the questionnaire.

2. Text fields are a maximum of 100 characters.

3. The questionnaire is structured as follows: Questions 1-9 are general questions; questions 10-54 are specific to trace elements analysis; questions 55-61 are specific to PCB analysis, questions 62-68 are specific to PAH analysis.

4. From question 10 on you have to click the 'non applicable measurement button' for questions related to an analyte that you did not measure.

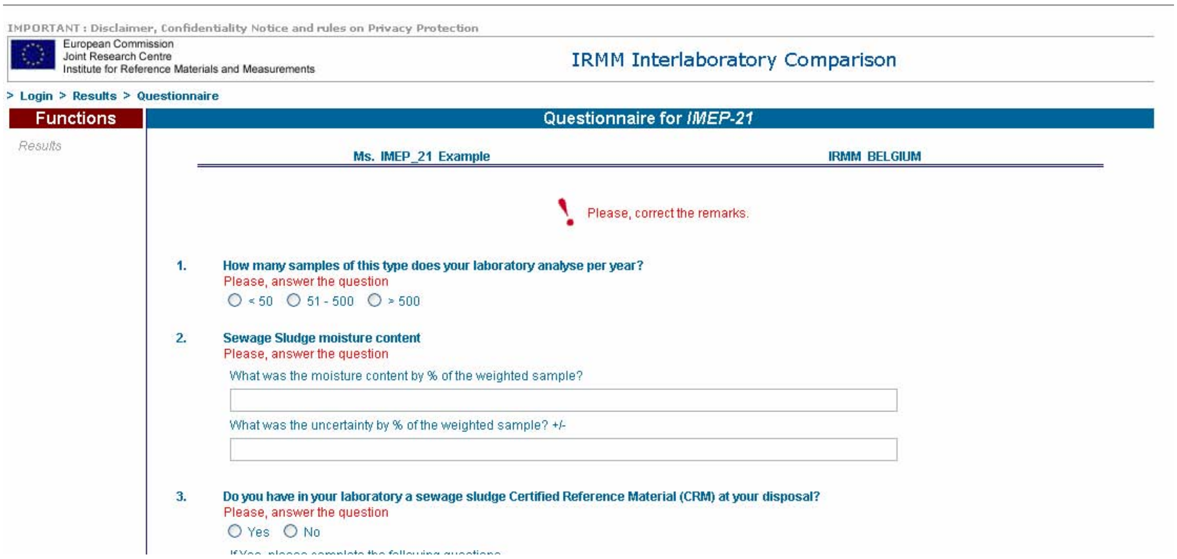
Please do not add comments to questions where it is not asked. For any comments entered where you have selected 'not applicable measurement', our system will automatically delete them when you submit your data.

FOR EXAMPLE- If you answer 'Cd non applicable measurement' PLEASE do not add any text there, **as our system will automatically delete your comments when you submit.**

Once you have completed the questionnaire, press the *SUBMIT QUESTIONNAIRE* button.

If you receive an error message, the system will direct you to that relevant question by adding a message in red text.

Below is an example of an error message screen. (Located at the top of the screen)



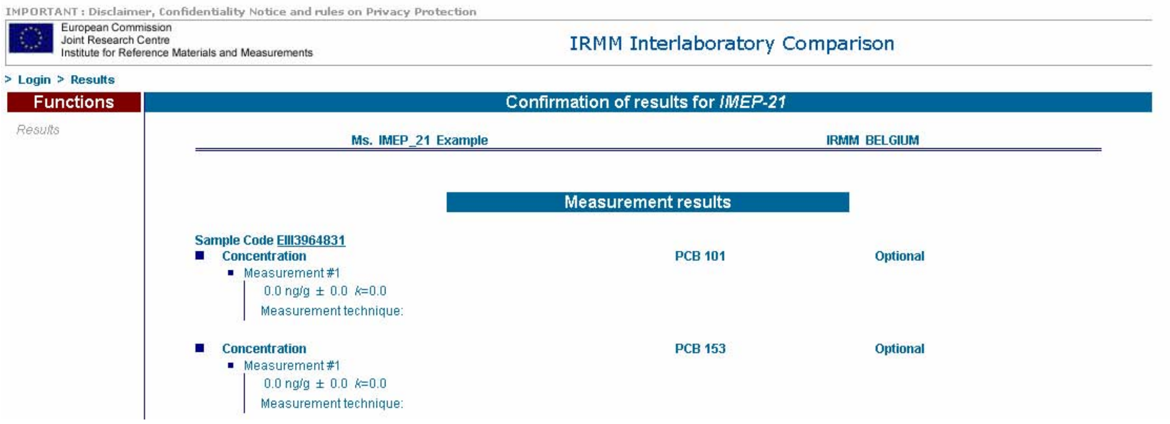
Before re-submitting your data please make sure that the following has been applied:-

a) Ensure all questions have been completed.

b) Ensure that the comment field has only been completed when asked.

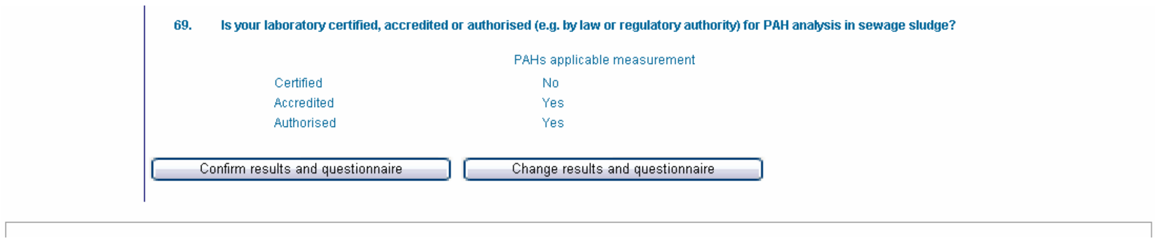
When you have made the necessary changes, press the *SUMBIT QUESTIONNAIRE* button.

A confirmation screen will appear showing the data entered.



Should any amendments need to be made press the *CHANGE RESULTS AND QUESTIONNAIRE* button, this will return you to the previous screen. Make the required changes and submit your data again.

Once more the confirmation screen will appear, check your data again. When all data is correct, press the *CONFIRM RESULTS AND QUESTIONNAIRE* button. (Located at the bottom of the screen)



It is IMPORTANT that you print off your result report form ONLY ONCE, from the available print option.
Please sign and fax this document to IRMM on Fax No. +32 (0)14 571 865

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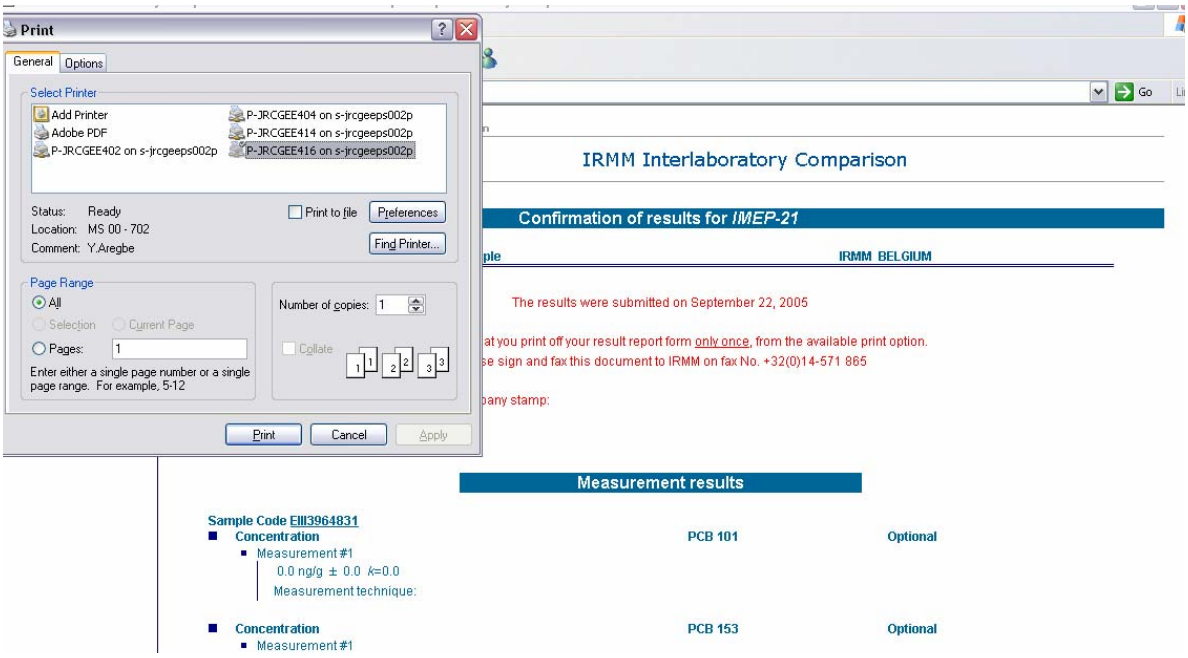
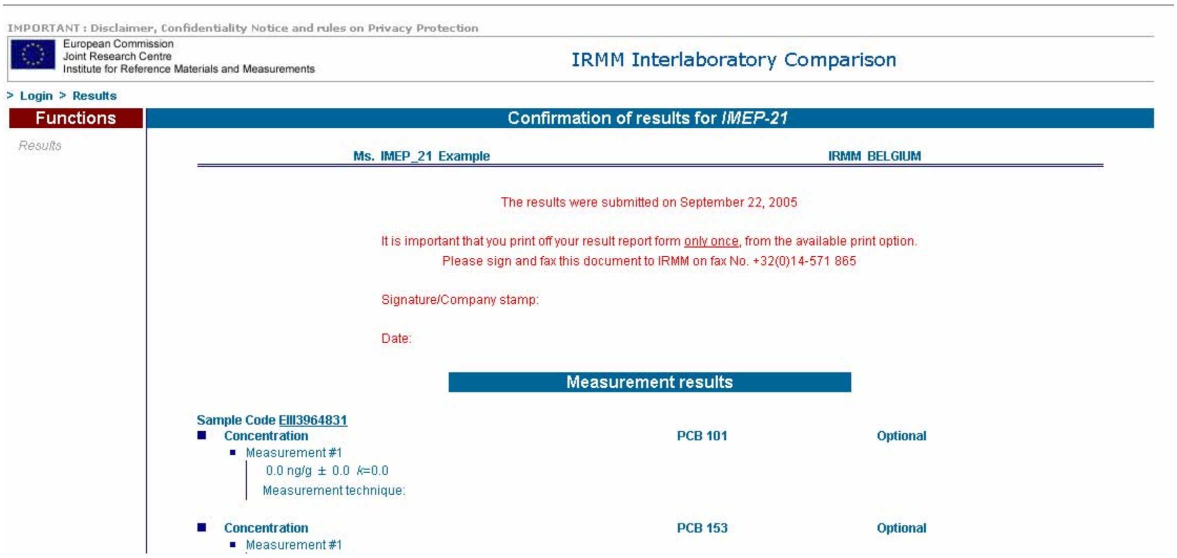


The final screen will conclude that your data has been accepted by IRMM, this message will appear at the top of the screen.



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DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements

Geel, 16 December 2005
IM/L/118/05

IMEP-21
Trace Elements, PCBs and PAHs in Sewage Sludge

Certified Reference Values for Trace Elements

analyte	certified value mg·kg^{-1} (dry mass)	expanded uncertainty *U*, *k*=2 mg·kg^{-1} (dry mass)
Cadmium	19.23	0.24
Chromium	209	10
Copper	843	40
Lead	619.8	8.7
Mercury	9.03	0.36
Nickel	68.5	1.0
Zinc	3160	72

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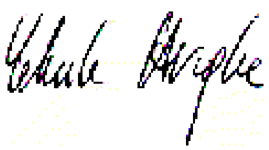
jrc-irmm-imep@cec.eu.int • http://www.irmm.jrc.be/html/interlaboratory_comparisons/imep

Certified Reference Values for Polychlorinated Biphenyls

analyte	certified value $ng \cdot g^{-1}$ (dry mass)	expanded uncertainty *U*, *k*=2 $ng \cdot g^{-1}$ (dry mass)
PCB_28	48.2	9.0
PCB_52	92.3	4.7
PCB_101	121.0	6.9
PCB_118	87.4	4.1
PCB_153	203.0	9.1
PCB_180	157	12
Sum_PCBs	709	20

Certified Reference Values for Polycyclic Aromatic Hydrocarbons

analyte	certified value ng·g^{-1} (dry mass)	expanded uncertainty *U*, *k*=2 ng·g^{-1} (dry mass)
Anthracene	104	18
Benz(a)anthracene	386	56
Benz(a)pyrene	383	91
Benz(b+k)fluoranthene	1110	160
Benzo(ghi)perylene	508	58
Fluoranthene	916	63
Fluorene	90	22
Indeno(1,2,3-cd)pyrene	370	54
Naphtalene	116	27
Phenanthrene	746	77
Pyrene	1280	120
Sum_PAHs	6010	260



Dr. Yetunde Aregbe
IMEP-21 Co-ordinator
IRMM

Dr. Philip Taylor
IM unit-head
IRMM

IMEP_21 Letter accompanying the individual certificate

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

Geel, 15 February 2006

IM/L/17/06

D04-IM(2006)D/2873

«title» «firstname» «surname»
«Organisation»
«ADDRESS» «ADDRESS2»
«town» «zip»
«country»

IMEP-21 Trace Elements, PCBs and PAHs in Sewage Sludge

Dear «title» «surname»,

Please find enclosed your individual certificate for IMEP-21. This certificate includes your reported results and the performance scores for your laboratory together with the IMEP-21 certified reference values. Furthermore the fit-for-purpose performance evaluation criteria, the performance scoring and the performance assessment criteria are listed. Enclosed you will also find a hard copy of the IMEP-21 material certificate which is also on-line available on our website.

In order to follow-up the receipt of this information, may we ask you to return the document 'Acknowledgement of receipt' at your earliest convenience !

The IMEP-21 report to participants is under preparation and on its completion will be made available on our website in Spring 2006. After booklet printing, you will also receive a personal hard copy of this report.

We sincerely hope you have found your participation in IMEP-21 useful. We would like to apologize for any inconvenience that might have occurred due to the fact that we were obliged to revise the time schedule for this interlaboratory comparison.

We would like to thank you for your participation in IMEP-21 and we hope to welcome you again as participant in one of our future interlaboratory comparisons.

Yours sincerely,

IMEP-21 Co-ordinator

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Institute for Reference Materials and Measurements

Geel, 15 February 2006

IM/L/14/06/«C_IMEP21»

Attachment to D04-IM(2006)D/2873

Individual Certificate

IMEP-21 Trace Elements, PCBs and PAHs in Sewage Sludge

Issued to: «title» «firstname» «surname»
«Organisation»
«ADDRESS» «ADDRESS2»
«town» «zip» «country»

	Certified in mg•kg^{-1}dry mass		Reported in mg•kg^{-1}				Performance scores			
Analyte	Reference value	*U*, *k*=2	Value	Uncertainty	Coverage Factor, *k*	Analytical technique	D%	*zeta'*	*z*	*Accept Unc.*
Cadmium	19.23	0.24	«Cd»	«Cd_unc»	«Cd_Kfactor»	«CD_TECHNIQUE»	«Cd_Dev»	«Cd_zeta»	«Cd_z»	«Cd_Min_Max_zeta»
Chromium	209	10	«Cr»	«Cr_unc»	«Cr_Kfactor»	«Cr_TECHNIQUE»	«Cr_Dev»	«Cr_zeta»	«Cr_z»	«Cr_Min_Max_zeta»
Copper	843	40	«Cu»	«Cu_unc»	«Cu_Kfactor»	«Cu_TECHNIQUE»	«Cu_Dev»	«Cu_zeta»	«Cu_z»	«Cu_Min_Max_zeta»
Lead	619.8	8.7	«Pb»	«Pb_unc»	«Pb_Kfactor»	«Pb_TECHNIQUE»	«Pb_Dev»	«Pb_zeta»	«Pb_z»	«Pb_Min_Max_zeta»
Mercury	9.03	0.36	«Hg»	«Hg_unc»	«Hg_Kfactor»	«Hg_TECHNIQUE»	«Hg_Dev»	«Hg_zeta»	«Hg_z»	«Hg_Min_Max_zeta»
Nickel	68.5	1.0	«Ni»	«Ni_unc»	«Ni_Kfactor»	«Ni_TECHNIQUE»	«Ni_Dev»	«Ni_zeta»	«Ni_z»	«Ni_Min_Max_zeta»
Zinc	3160	72	«Zn»	«Zn_unc»	«Zn_Kfactor»	«Zn_TECHNIQUE»	«Zn_Dev»	«Zn_zeta»	«Zn_z»	«Zn_Min_Max_zeta»

	Certified in ng•g^{-1}dry mass		Reported in mg•kg^{-1}				Performance scores			
Analyte	Reference value	*U, k*=2	Value	Uncertainty	Coverage Factor, *k*	Analytical technique	D%	*zeta'*	*z*	*Accept Unc*
PCB_28	48.2	9.0	«PCB_28»	«PCB_28_unc»	«PCB_28_Kfactor»	«PCB_28_TECHNIQUE»	«PCB_28_Dev»	«PCB_28_zeta»	«PCB_28_z»	«PCB_28_Min_Max_zeta»
PCB_52	92.3	4.7	«PCB_52»	«PCB_52_unc»	«PCB_52_Kfactor»	«PCB_52_TECHNIQUE»	«PCB_52_Dev»	«PCB_52_zeta»	«PCB_52_z»	«PCB_52_Min_Max_zeta»
PCB_101	121.0	6.9	«PCB_101»	«PCB_101_unc»	«PCB_101_Kfactor»	«PCB_101_TECHNIQUE»	«PCB_101_Dev»	«PCB_101_zeta»	«PCB_101_z»	«PCB_101_Min_Max_zeta»
PCB_118	87.4	4.1	«PCB_118»	«PCB_118_unc»	«PCB_118_Kfactor»	«PCB_118_TECHNIQUE»	«PCB_118_Dev»	«PCB_118_zeta»	«PCB_118_z»	«PCB_118_Min_Max_zeta»
PCB_153	203.0	9.1	«PCB_153»	«PCB_153_unc»	«PCB_153_Kfactor»	«PCB_153_TECHNIQUE»	«PCB_153_Dev»	«PCB_153_zeta»	«PCB_153_z»	«PCB_153_Min_Max_zeta»
PCB_180	157	12	«PCB_180»	«PCB_180_unc»	«PCB_180_Kfactor»	«PCB_180_TECHNIQUE»	«PCB_180_Dev»	«PCB_180_zeta»	«PCB_180_z»	«PCB_180_Min_Max_zeta»
Sum_PCBs	709	20	«sum_PCB»	«sum_PCB_unc»	«sum_PCB_Kfactor»	«sum_PCB_TECHNIQUE»	«Sum_PCBs_Dev»	«Sum_PCBs_zeta»	«Sum_PCBs_z»	«Sum_PCB_Min_Max_zeta»

	Certified in ng•g^{-1}dry mass		Reported in mg•kg^{-1}				Performance scores			
Analyte	Reference value	*U, k*=2	Value	Uncertainty	Coverage Factor, *k*	Analytical technique	D%	*zeta'*	*z*	*Accept Unc.*
Anthracene	104	18	«Anthracene»	«Anthracene_unc»	«Anthracene_Kfactor»	«Anthracene_TECHNIQUE»	«Anthracene_Dev»	«Anthracene_zeta»	«Anthracene_z»	«Anthracene_Min_Max_zeta»
Benz(a)-anthracene	386	56	«Benzaanthrazene»	«Benzaanthrazene_unc»	«Benzaanthrazene_Kfactor»	«Benzaanthrazene_TECHNIQU	«Benzaanthrace	«Benzaanthrace	«Benzaanthrace	«Benzaanthracene_Mi

						E»	ne_Dev»	ne_zeta»	ne_z»	n_Max_zeta»
Benz(a)pyrene	383	91	«Benzapyrene»	«Benzapyrene_unc»	«Benzapyrene_Kfactor»	«Benzapyrene_TECHNIQUE»	«Benzapyrene_Dev»	«Benzapyrene_zeta»	«Benzapyrene_z»	«Benzapyrene_Min_Max_zeta»
Benz(b+k)-fluoranthene	1110	160	«Benzbkfluorantene»	«Benzbkfluorantene_unc»	«Benzbkfluorantene_Kfactor»	«Benzbkfluorantene_TECHNIQUE»	«Benzbkfluoranthene_Dev»	«Benzbkfluoranthene_zeta»	«Benzbkfluoranthene_z»	«Benzbkfluoranthene_Min_Max_zeta»
Benzo(ghi)-perylene	508	58	«Benzoghiperylene»	«Benzoghiperylene_unc»	«Benzoghiperylene_Kfactor»	«Benzoghiperylene_TECHNIQUE»	«Benzoghiperylene_Dev»	«Benzoghiperylene_zeta»	«Benzoghiperylene_z»	«Benzoghiperylene_Min_Max_zeta»
Fluoranthene	916	63	«Fluoranthene»	«Fluoranthene_unc»	«Fluoranthene_Kfactor»	«Fluoranthene_TECHNIQUE»	«Fluoranthene_Dev»	«Fluoranthene_zeta»	«Fluoranthene_z»	«Fluoranthene_Min_Max_zeta»
Fluorene	90	22	«Fluorene»	«Fluorene_unc»	«Fluorene_Kfactor»	«Fluorene_TECHNIQUE»	«Fluorene_Dev»	«Fluorene_zeta»	«Fluorene_z»	«Fluorene_Min_Max_zeta»
Indeno(1,2,3-cd)-pyrene	370	54	«Indeno123cdpyrene»	«Indeno123cdpyrene_unc»	«Indeno123cdpyrene_Kfactor»	«Indeno123cdpyrene_TECHNIQUE»	«Indeno123cdpyrene_Dev»	«Indeno123cdpyrene_zeta»	«Indeno123cdpyrene_z»	«Indeno123cdpyrene_Min_Max_zeta»
Naphtalene	116	27	«Naphtalene»	«Naphtalene_unc»	«Naphtalene_Kfactor»	«Naphtalene_TECHNIQUE»	«Naphtalene_Dev»	«Naphtalene_zeta»	«Naphtalene_z»	«Naphtalene_Min_Max_zeta»
Phenanthrene	746	77	«Phenantrene»	«Phenantrene_unc»	«Phenantrene_Kfactor»	«Phenantrene_TECHNIQUE»	«Phenanthrene_Dev»	«Phenanthrene_zeta»	«Phenanthrene_z»	«Phenanthrene_Min_Max_zeta»
Pyrene	1280	120	«Pyrene»	«Pyrene_unc»	«Pyrene_Kfactor»	«Pyrene_TECHNIQUE»	«Pyrene_Dev»	«Pyrene_zeta»	«Pyrene_z»	«Pyrene_Min_Max_zeta»
Sum_PAHs	6010	260	«sum_PAHs»	«sum_PAHs_unc»	«sum_PAHs_Kfactor»	«sum_PAHs_TECHNIQUE»	«Sum_PAHs_Dev»	«Sum_PAHs_zeta»	«Sum_PAHs_z»	«Sum_PAHs_Min_Max_zeta»

The fit-for-purpose criteria for the certified test material were set based on legislation and international/national norms as follows:

Analyte	Fit-for-purpose criterion, σ
Cadmium	$0.1\ X_{ref}$
Chromium	$0.1\ X_{ref}$
Copper	$0.05\ X_{ref}$
Lead	$0.05\ X_{ref}$
Mercury	$0.1\ X_{ref}$
Nickel	$0.1\ X_{ref}$
Zinc	$0.05\ X_{ref}$
PCB_28	$0.15\ X_{ref}$
PCB_52	$0.15\ X_{ref}$
PCB_101	$0.15\ X_{ref}$
PCB_118	$0.15\ X_{ref}$
PCB_153	$0.15\ X_{ref}$
PCB_180	$0.15\ X_{ref}$
Sum_PCBs	$0.15\ X_{ref}$
Anthracene	$0.15\ X_{ref}$
Benz(a)-anthracene	$0.15\ X_{ref}$
Benz(a)pyrene	$0.2\ X_{ref}$
Benz(b+k)-fluoranthene	$0.15\ X_{ref}$
Benzo(ghi)-perylene	$0.15\ X_{ref}$
Fluoranthene	$0.15\ X_{ref}$
Fluorene	$0.2\ X_{ref}$
Indeno(1,2,3-cd)-pyrene	$0.15\ X_{ref}$
Naphtalene	$0.2\ X_{ref}$
Phenanthrene	$0.15\ X_{ref}$
Pyrene	$0.15\ X_{ref}$
Sum_PAHs	$0.15\ X_{ref}$

Performance scoring		**Performance assessment criteria**	
Percentage difference	$$D\% = \frac{(x - X_{ref})}{X_{ref}} \cdot 100$$	$\lvert D\% \rvert \leq 2\sigma / X_{ref} \cdot 100$ $\lvert D\% \rvert > 2\sigma / X_{ref} \cdot 100$	Satisfactory Unsatisfactory
z-score	$$z = \frac{(x - X_{ref})}{\sigma}$$	$\lvert z$ *or zeta'* $\rvert \leq 2$ $2 < \lvert z$ or *zeta'* $\rvert \leq 3$ $\lvert z$ *or zeta'* $\rvert > 3$	Satisfactory Questionable Unsatisfactory
zeta'-score	$$zeta' = \frac{x - X_{ref}}{\sqrt{u_x^2 + \sigma^2}}$$		
Acceptable Uncertainty	only issued if $\lvert zeta' \rvert \leq 2$.	if $u_{ref} \leq u_x \leq \sigma$ Otherwise	'YES' 'NO'

Where X_{ref} is the reference value; u_{ref} is the combined uncertainty of the reference value; σ is the fit-for-purpose criterion, x is the result you reported; u_x is the associated combined uncertainty derived as follows: When you reported a coverage factor (k), the combined uncertainty was calculated dividing the reported uncertainty by k. When no coverage factor was reported, the reported uncertainty was considered as the range of a rectangular distribution (± a); the combined uncertainty was then calculated dividing this range by $\sqrt{3}$, according to *Appendix E*-of the EURACHEM/CITAC Guide (2000) Quantifying uncertainty.

Dr. Yetunde Aregbe
IMEP-21 Co-ordinator
IRMM

Dr. Philip Taylor
unit-head
IRMM

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

Geel, 15 February 2006

«title» «firstname» «surname»
«Organisation»
«ADDRESS» «ADDRESS2»
«town» «zip»
«country»

IMEP-21 Trace Elements, PCBs and PAHs in Sewage Sludge
Acknowledgement of receipt 'individual certificate'

Dear «title» «surname»,

Since we need to follow up all dispatches,
we kindly ask you to
return this form on receipt
to one of the
following recipients

IMEP-FAX: +32 (0)14 571 865

IMEP–contact: jrc-irmm-imep@cec.eu.int

IMEP-office: Retieseweg 111, B-2440 Geel, Belgium

Date of receipt...
Signature……………………………………………….
Any comments ??
………………………………………………………………………………………………………

Thank you very much for your collaboration.

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571-211.
Tel.: +32-(0)14-571 673 • Fax: +32-(0)14-571 865
jrc-irmm-imep@cec.eu.int• http://www.irmm.jrc.be/html/interlaboratory_comparisons/imep

Sample Code BFSA2267860

- **Concentration** **PCB 101** **Optional**
  - Measurement #1

Select measurement unit from the allowable units:

◉ ng/g

Result value ± Uncertainty value Coverage factor *k*

Technique used

○ OTHER. Please specify,

- **Concentration** **PCB 153** **Optional**
  - Measurement #1

Select measurement unit from the allowable units:

◉ ng/g

Result value ± Uncertainty value Coverage factor *k*

Technique used

○ OTHER. Please specify,

- **Concentration** **PCB 118** **Optional**
  - Measurement #1

Select measurement unit from the allowable units:

◉ ng/g

Result value ± Uncertainty value Coverage factor *k*

Technique used

○ OTHER. Please specify,

- **Concentration** **PCB 180** **Optional**
  - Measurement #1

Select measurement unit from the allowable units:

◉ ng/g

Result value ± Uncertainty value Coverage factor *k*

Technique used

○ OTHER. Please specify,

- **Concentration** **PCB 28** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ◉ ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used ○

    ○ OTHER. Please specify,

- **Concentration** **PCB 52** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ◉ ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used ○

    ○ OTHER. Please specify,

- **Concentration** **PAH: Anthracene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ◉ ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used ○

    ○ OTHER. Please specify,

- **Concentration** **PAH: Benz(a)anthrazene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ◉ ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used ○

    ○ OTHER. Please specify,

- **Concentration** **PAH: Benz(a)pyrene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ◉ ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used ○

    ○ OTHER. Please specify,

- **Concentration** **PAH: Benz(b+k)fluorantene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAH: Benzo(ghi)perylene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAH: Fluoranthene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAH: Fluorene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    mol/kg ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAH: Phenantrene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAH: Indeno(1,2,3-cd)pyrene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAH: Naphtalene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAH: Pyrene** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PAHs (sum)** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **PCBs (sum)** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    ng/g

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **Trace element: Cd** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    mg/kg

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **Trace element: Cr** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    mg/kg

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **Trace Element: Cu** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    mg/kg

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **Trace Element: Hg** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    mg/kg

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **Trace Element: Ni** **Optional**
  - Measurement #1

    Select measurement unit from the allowable units:

    mg/kg

    Result value ± Uncertainty value Coverage factor *k*

    Technique used

    OTHER. Please specify,

- **Concentration** **Trace Element: Pb** **Optional**
  - Measurement #1

Select measurement unit from the allowable units:

mg/kg

Result value ± Uncertainty value Coverage factor *k*

Technique used

OTHER. Please specify,

- **Concentration** **Trace element: Zn** **Optional**
  - Measurement #1

Select measurement unit from the allowable units:

mg/kg

Result value ± Uncertainty value Coverage factor *k*

Technique used

OTHER. Please specify,

Save Clear

Submit results

1. How many samples of this type does your laboratory analyse per year?

○ < 50 ○ 51 - 500 ○ > 500

2. Sewage Sludge moisture content

What was the moisture content by % of the weighted sample?

What was the uncertainty by % of the weighted sample? +/-

3. Do you have in your laboratory a sewage sludge Certified Reference Material (CRM) at your disposal?

○ Yes ○ No

If Yes, please complete the following questions.

	Yes	No
Is the CRM used in your laboratory for validation of procedures?	○	○
Is the CRM used in your laboratory for calibration of instruments?	○	○

Please state which CRM and supplier.

4. Did your laboratory participate in other interlaboratory comparisons (round robin test/ring tests/collaborative trials)?

Yes	No
○	○

If Yes, please state which interlaboratory comparison scheme and which proficiency testing organiser.

5. Is your laboratory working according to a quality management system?

○ Yes ○ No

If Yes, please state which system. (you can make more than one choice)

- EN 45000 series ☐
- ISO 9000 series ☐
- ISO 17025 ☐
- Other (e.g. CEN, GLP, EPA, TQM, national standards) ☐

If Other, please supply additional information.

6. Do you report uncertainties on chemical measurements to your usual customers?

○ Yes ○ No

7. Are you familiar with the Guides for Quantifying Measurements Uncertainty issued by the International Organisation for Standardisation (ISO,1993) and/or EURACHEM (1995)?

○ Yes ○ No

8. Where the reported uncertainties calculated according to the in above mentioned guides?

○ Yes ○ No

If No, how was the measurement uncertainty evaluated?

9. Was your participation to this IMEP comparison used to demonstrate your measurement capability to: (you can make more than one choice)

- your management ☐
- your customers ☐
- regulating or accreditation body ☐
- participation was intended for internal quality control purposes ☐
- Other ☐

If Other, please supply additional information.

10. Status of experience for Trace Element analysis.

Cd applicable measurement ○ Cd non applicable measurement ○

	Experienced	Less- and non-Experienced
Does your laboratory consider itself, in matters of Cd analysis in sewage sludge at the given concentration level, as:	○	○

11.

Cr applicable measurement ○ Cr non applicable measurement ○

	Experienced	Less- and non-Experienced
Does your laboratory consider itself, in matters of Cr analysis in sewage sludge at the given concentration level, as:	○	○

12.

Cu applicable measurement ○ Cu non applicable measurement ○

	Experienced	Less- and non-Experienced
Does your laboratory consider itself, in matters of Cu analysis in sewage sludge at the given concentration level, as:	○	○

13.

Hg applicable measurement ○ Hg non applicable measurement ○

	Experienced	Less- and non-Experienced
Does your laboratory consider itself, in matters of Hg analysis in sewage sludge at the given concentration level, as:	○	○

14.

Ni applicable measurement ○ Ni non applicable measurement ○

	Experienced	Less- and non-Experienced
Does your laboratory consider itself, in matters of Ni analysis in sewage sludge at the given concentration level, as:	○	○

15.

Pb applicable measurement ○ | Pb non applicable measurement ○

Does your laboratory consider itself, in matters of Pb analysis in sewage sludge at the given concentration level, as:

○ Experienced ○ Less- and non-Experienced

16.

Zn applicable measurement ○ | Zn non applicable measurement ○

Does your laboratory consider itself, in matters of Zn analysis in sewage sludge at the given concentration level, as:

○ Experienced ○ Less- and non-Experienced

17. Was the applied analytical method for trace element measurements...

○ your routinely used method? ○ Not your routinely used method?

18. Was the trace element analysis performed following any official analytical method? (e.g. ISO/CEN)

○ Yes ○ No

If Yes, please specify wich official analytical method

---

19. What was the sample mass used for the trace elements analysis [g]?

20. Digestion method and acid mixture

Cd applicable measurement ○ | Cd non applicable measurement ○

Which digestion method (micrwave, high pressure ashing, etc...) was used?

Which acid mixture was used?

21.

Cr applicable measurement ○ | Cr non applicable measurement ○

Which digestion method (micrwave, high pressure ashing, etc...) was used?

Which acid mixture was used?

22.

Cu applicable measurement ○ Cu non applicable measurement ○

Which digestion method (micrwave, high pressure ashing, etc...) was used?

Which acid mixture was used?

23.

Hg applicable measurement ○ Hg non applicable measurement ○

Which digestion method (micrwave, high pressure ashing, etc...) was used?

Which acid mixture was used?

24.

Ni applicable measurement ○ Ni non applicable measurement ○

Which digestion method (micrwave, high pressure ashing, etc...) was used?

Which acid mixture was used?

25.

Pb applicable measurement ○ Pb non applicable measurement ○

Which digestion method (micrwave, high pressure ashing, etc...) was used?

Which acid mixture was used?

26.

Zn applicable measurement ○ Zn non applicable measurement ○

Which digestion method (micrwave, high pressure ashing, etc...) was used?

Which acid mixture was used?

27. Separation.

Cd applicable measurement ○ Cd non applicable measurement ○

	Yes	No
Did the analytical procedure for Cd involve a separation step?	○	○

28.

Cr applicable measurement	Cr non applicable measurement
◯	◯

	Yes	No
Did the analytical procedure for Cr involve a separation step?	◯	◯

29.

Cu applicable measurement	Cu non applicable measurement
◯	◯

	Yes	No
Did the analytical procedure for Cu involve a separation step?	◯	◯

30.

Hg applicable measurement	Hg non applicable measurement
◯	◯

	Yes	No
Did the analytical procedure for Hg involve a separation step?	◯	◯

31.

Ni applicable measurement	Ni non applicable measurement
◯	◯

	Yes	No
Did the analytical procedure for Ni involve a separation step?	◯	◯

32.

Pb applicable measurement	Pb non applicable measurement
◯	◯

	Yes	No
Did the analytical procedure for Pb involve a separation step?	◯	◯

33.

Zn applicable measurement	Zn non applicable measurement
◯	◯

	Yes	No
Did the analytical procedure for Zn involve a separation step?	◯	◯

34. Preconcentration.

Cd applicable measurement	Cd non applicable measurement
◯	◯

	Yes	No
Did the analytical procedure for Cd involve a preconcentration step?	◯	◯

35.

Cr applicable measurement ◯ Cr non applicable measurement ◯

Did the analytical procedure for Cr involve a preconcentration step? Yes ◯ No ◯

36.

Cu applicable measurement ◯ Cu non applicable measurement ◯

Did the analytical procedure for Cu involve a preconcentration step? Yes ◯ No ◯

37.

Hg applicable measurement ◯ Hg non applicable measurement ◯

Did the analytical procedure for Hg involve a preconcentration step? Yes ◯ No ◯

38.

Ni applicable measurement ◯ Ni non applicable measurement ◯

Did the analytical procedure for Ni involve a preconcentration step? Yes ◯ No ◯

39.

Pb applicable measurement ◯ Pb non applicable measurement ◯

Did the analytical procedure for Pb involve a preconcentration step? Yes ◯ No ◯

40.

Zn applicable measurement ◯ Zn non applicable measurement ◯

Did the analytical procedure for Zn involve a preconcentration step? Yes ◯ No ◯

41. Calibration strategy.

○ Cd applicable measurement ○ Cd non applicable measurement

Which calibration strategy was used for the Cd measurements?

- external standard ☐
- standard addition ☐
- internal standard ☐

If internal standard was used, please specify.

42.

○ Cr applicable measurement ○ Cr non applicable measurement

Which calibration strategy was used for the Cr measurements?

- external standard ☐
- standard addition ☐
- internal standard ☐

If internal standard was used, please specify.

43.

○ Cu applicable measurement ○ Cu non applicable measurement

Which calibration strategy was used for the Cu measurements?

- external standard ☐
- standard addition ☐
- internal standard ☐

If internal standard was used, please specify.

44.

○ Hg applicable measurement ○ Hg non applicable measurement

Which calibration strategy was used for the Hg measurements?

- external standard ☐
- standard addition ☐
- internal standard ☐

If internal standard was used, please specify.

45.

○ Ni applicable measurement ○ Ni non applicable measurement

Which calibration strategy was used for the Ni measurements?

external standard	☐
standard addition	☐
internal standard	☐

If internal standard was used, please specify.

46.

○ Pb applicable measurement ○ Pb non applicable measurement

Which calibration strategy was used for the Pb measurements?

external standard	☐
standard addition	☐
internal standard	☐

If internal standard was used, please specify.

47.

○ Zn applicable measurement ○ Zn non applicable measurement

Which calibration strategy was used for the Zn measurements?

external standard	☐
standard addition	☐
internal standard	☐

If internal standard was used, please specify.

48. **Is your laboratory certified, accredited or authorised (e.g. by law or regulatory authority) for one of the following analysis in sewage sludge?**

Cd applicable measurement	Cd non applicable measurement
○	○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

49.

Cr applicable measurement	Cr non applicable measurement
○	○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

50.

Cu applicable measurement	Cu non applicable measurement
○	○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

51.

Hg applicable measurement	Hg non applicable measurement
○	○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

52.

Ni applicable measurement ○ Ni non applicable measurement ○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

53.

Pb applicable measurement ○ Pb non applicable measurement ○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

54.

Zn applicable measurement ○ Zn non applicable measurement ○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

55. Status of experience for PCB analysis.

PCBs applicable measurement ○ PCBs non applicable measurement ○

	Experienced	Less- and non-Experienced
Does your laboratory consider itself, in matters of PCB analysis in sewage sludge at the given concentration level, as:	○	○

56. Applied analytical method for PCB measurements.

PCBs applicable measurement ○ PCBs non applicable measurement ○

	your routinely used method?	not a routinely used method?
Was the applied analytical method for PCB measurements...	○	○

57. Official analytical method for PCB measurements..

○ PCBs applicable measurement ○ PCBs non applicable measurement

Was the PCB analysis performed following any official analytical metod? (e.g.ISO/CEN)?

Yes ○ No ○

If Yes, please specify which official analytical method.

58. Sample treatment.

PCBs applicable measurement ○ PCBs non applicable measurement ○

Please briefly describe your measurement process by listing the types of reagents and apparatus applied.

Sample mass [g]?

Extraction procedure:

Temperature (°C)

Extraction time

Solvent

59. Clean-up.

○ PCBs applicable measurement ○ PCBs non applicable measurement

Was an extraction clean-up performed?

Yes ○ No ○

If Yes, please specify.

60. Instrumentation.

PCBs applicable measurement ○ PCBs non applicable measurement ○

Which separation apparatus (e.g. GC, LC,...) was used?

Which column?

61. Is your laboratory certified, accredited or authorised (e.g. by law or regulatory authority) for PCB analysis in sewage sludge?

PCBs applicable measurement ○ PCBs non applicable measurement ○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

62. Status of experience for PAH analysis.

PAHs applicable measurement ○ PAHs non applicable measurement ○

	Experienced	Less- and non-Experienced
Does your laboratory consider itself, in matters of PAH analysis in sewage sludge at the given concentration level, as:	○	○

63. Applied analytical method for PAH measurements.

PAHs applicable measurement	PAHs non applicable measurement
○	○

	your routinely used method?	not a routinely used method?
Was the applied analytical method for PAH measurements...	○	○

64. Official analytical method for PAH measurements..

○ PAHs applicable measurement ○ PAHs non applicable measurement

Was the PAH analysis performed following any official analytical metod? (e.g.ISO/CEN)?

Yes	No
○	○

If Yes, please specify which official analytical method.

65. Sample treatment.

PAHs applicable measurement	PAHs non applicable measurement
○	○

Please briefly describe your measurement process by listing the types of reagents and apparatus applied.

Sample mass [g]?

Extraction procedure:

Temperature (°C)

Extraction time

Solvent

66. Clean-up.

○ PAHs applicable measurement ○ PAHs non applicable measurement

Was an extraction clean-up performed?

Yes	No
○	○

If Yes, please specify.

67. Clean-up.

○ PAHs applicable measurement ○ PAHs non applicable measurement

Was an extraction clean-up performed?

	Yes	No
	○	○

If Yes, please specify.

68. Instrumentation.

PAHs applicable measurement	PAHs non applicable measurement
○	○

Which separation apparatus (e.g. GC, LC,...) was used?

Which column?

69. Is your laboratory certified, accredited or authorised (e.g. by law or regulatory authority) for PAH analysis in sewage sludge?

PAHs applicable measurement	PAHs non applicable measurement
○	○

	Yes	No
Certified	☐	☐
Accredited	☐	☐
Authorised	☐	☐

Clear

Submit questionnaire

European Commission

EUR 22242 EN – DG Joint Research Centre, Institute for Reference Materials and Measurements –
IMEP-21 Trace Elements, PCBs and PAHs in Sewage Sludge Report to Participants
Authors: Y.Aregbe, S. Bynens, L. Van Nevel, I. Verbist, P. Robouch, J. van de Kreeke and P.D.P. Taylor
Luxembourg: Office for Official Publications of the European Communities
2006 –151pp. – 21 x 29.7 cm
Scientific and Technical Research series

Abstract

The International Measurement Evaluation Programme (IMEP®) is an Interlaboratory Comparison (ILC) scheme in support of EU policies (e.g. Consumer Protection and Public Health, Single Market, Environment, Research and Technology, External Trade and Economic Policy). It is founded, owned and co-ordinated by the Institute for Reference Materials and Measurements (IRMM) of the European Commission's Directorate-General Joint Research Centre.

The aim of this interlaboratory comparison programme is to picture objectively the degree of equivalence and the quality of chemical measurements. Contrary to most other external quality assessment schemes, participating laboratories in IMEP® can compare their measurement results and uncertainty statements with external certified reference values, obtained completely independent from the participants' result. These reference values are required to demonstrate traceability and they should have a demonstrated and adequately small uncertainty, as evaluated according to international guidelines. Participants in IMEP® use their routine analytical procedures to measure the IMEP-certified test sample (CTS). Therefore they can assess the quality of their results on an international forum by comparing their values to the IMEP-reference values.

The European Union promotes the use of sewage sludge as fertilizer in agriculture. The Council Directive 86/278/EEC of 12 June 1986 sets rules for the protection of the environment, and in particular of the soil, when sewage sludge is used in agriculture. In this directive limit concentration ranges of metals in sewage sludge are laid down. In national legislation the concentration ranges for metals can be set below these limits. Furthermore limits for Polychlorinated Biphenyls (PCBs), and Polycyclic Aromatic Hydrocarbons (PAHs) congeners in sewage sludge are set in some of the national legislations. The directive 86/278/EEC is discussed to be revised and lower levels for metals and most probably also upper levels for PCBs and PAHs concentrations will be included. Participants in IMEP-21 were offered to measure the total amount content of 7 metals, 6 PCB congeners and 11 PAH congeners: Cd, Cr, Cu, Pb, Hg, Ni, Zn, PCB_28, PCB_52, PCB_101, PCB_118, PCB_153, PCB_180, sum_PCBs, anthracene, benz(a)anthracene, benz(a)pyrene, benz(b+k)fluoranthene, benzo(ghi)perylene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, pyrene, sum_PAHs.

IMEP-21 was organised in collaboration with The European Committee for Standardisation (CEN) upon approval of DG EuropAid, the Association of the European Geological Surveys (EuroGeoSurveys) Geoscientific Laboratory Network and the European Co operation for Accreditation (EA). 3228 measurement results on 26 analytes were reported by 204 participants from 44 countries in IMEP 21. Amongst those were participants nominated by CEN, EuroGeoSurvey and by national accreditation bodies (NABs) but also laboratories from Western Balkan countries (IRMM's CARDS support), new member states and other countries participated in MEP-21. Individual IMEP-21 certificates were issued to all the IMEP-21 participants. This certificate includes the IMEP-21 certified reference values, the reported results and the performance scores of the participant.

The IMEP-21 report to participants presents organisatorial details about the project. Participants' results are presented in a graphical way together with the reference value and are sorted according to different criteria based on the replies from the questionnaire from which also numerical information is included.

The mission of the Joint Research Centre is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of European Union policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Community. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

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