

QA/QC in the AQUAREF inter comparison exercise

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⁽⁶⁾ Ifremer, BE, ⁽⁷⁾ LNE, DMSI, ⁽⁸⁾ INERIS, ⁽⁹⁾ Ifremer, RBE-BE-LBCO

Summary



1. Schedule from data-base conception to participant reporting
2. Position QA/QC in trial from basic concepts to final implemented strategy
3. Data-base overview
4. Statistical treatment
5. Reference solutions from conception to assignation of the final value
6. Presentation of results (QC solution A, field blank) and discussion
7. Conclusion and perspectives

QA/QC in the AQUAREF inter comparison exercise

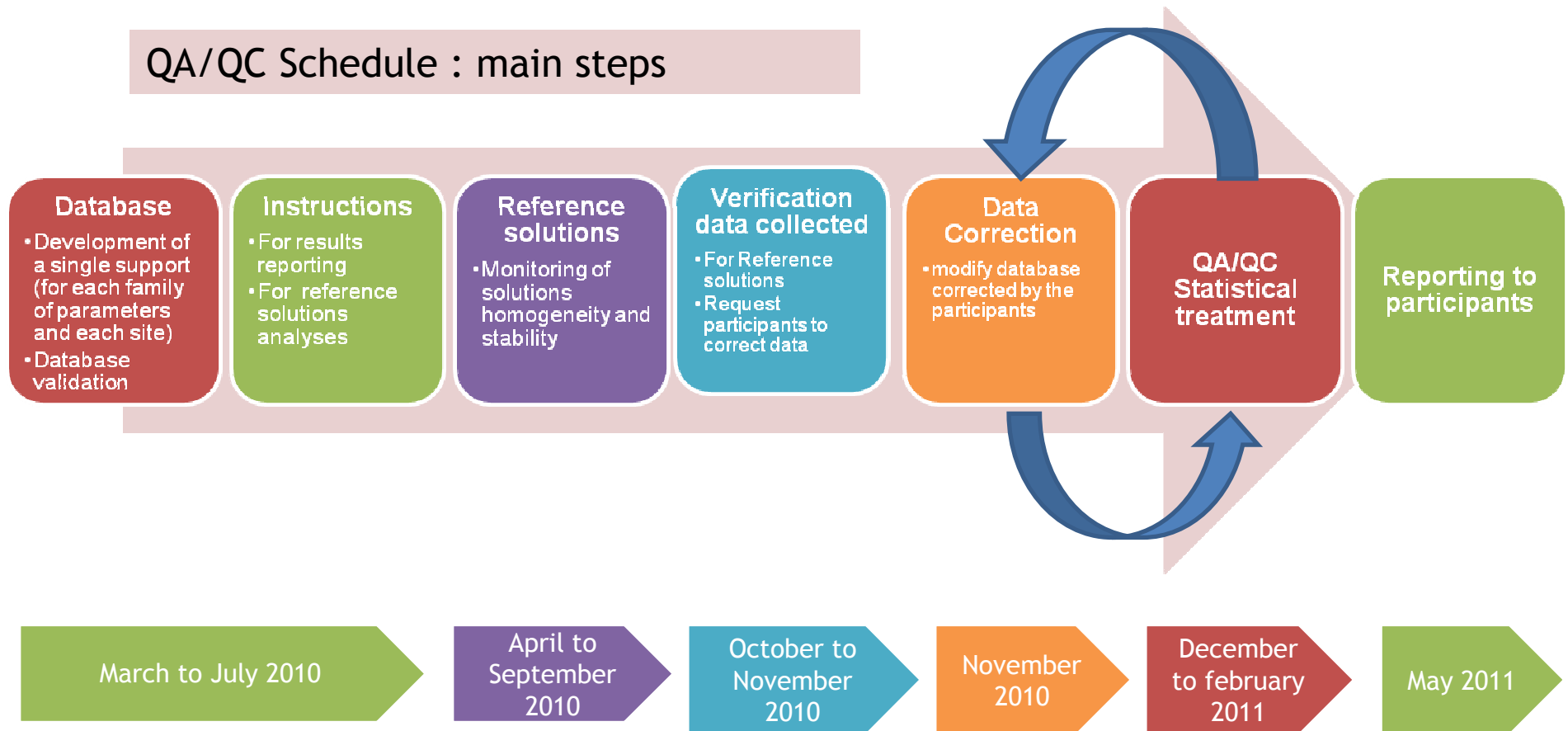
Schedule

**From database conception to
participant reporting**

General organization of the collaborative trial



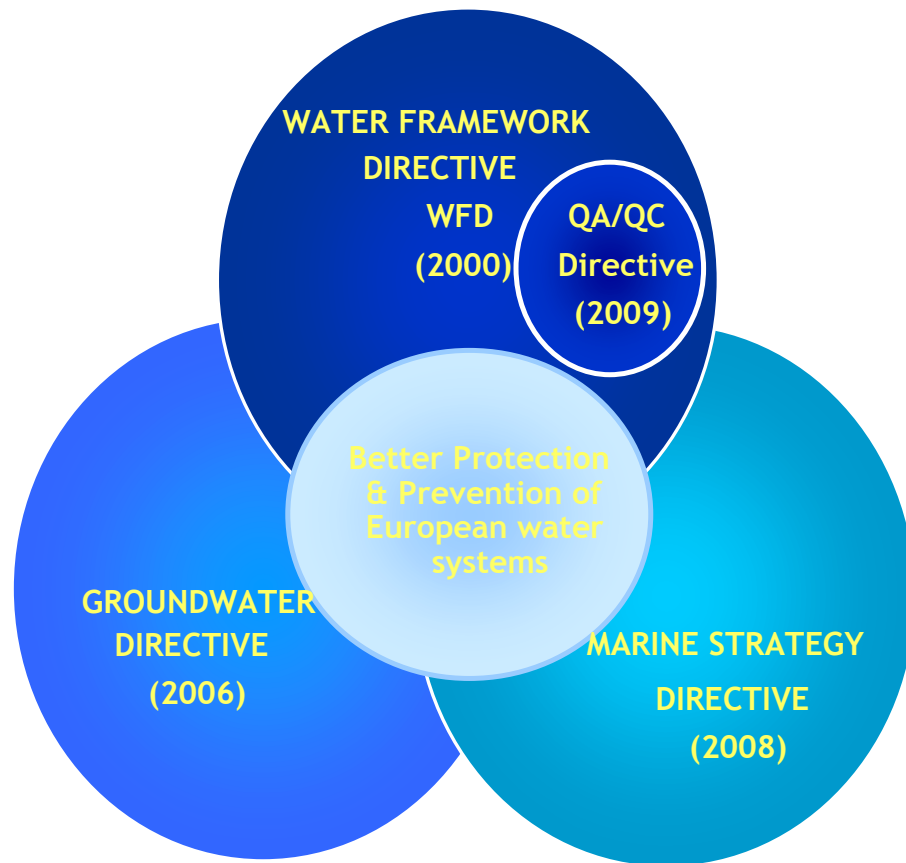
It is not a proficiency test



QA/QC in the AQUAREF inter comparison exercise

**From basic concepts to the final
implemented strategy**

From basic concepts... (1)



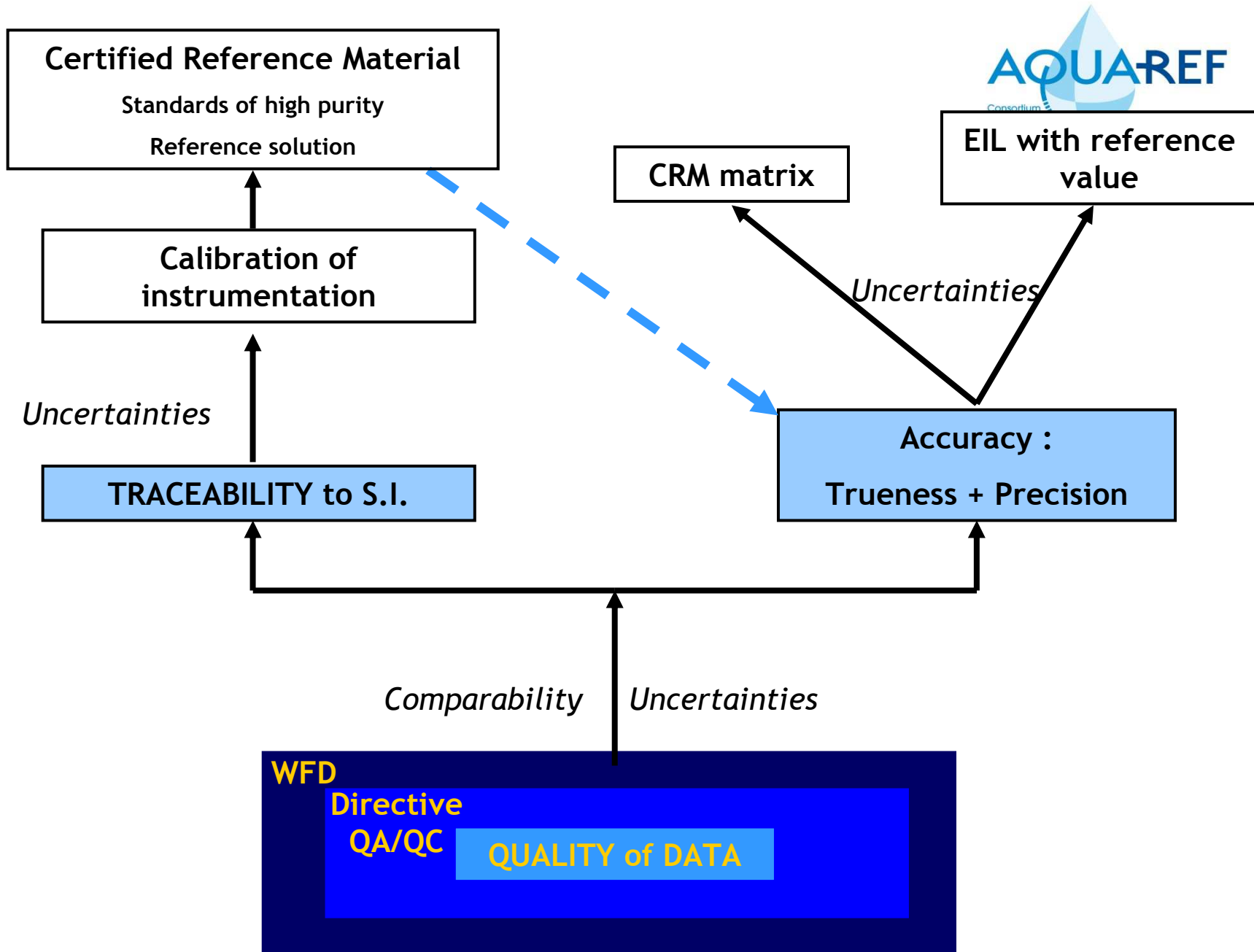
- ✓ Quality and comparability of data;
- ✓ Representativity of data;
- ✓ Rationalization of costs of monitoring;
- ✓ Evaluation of capabilities and competencies;

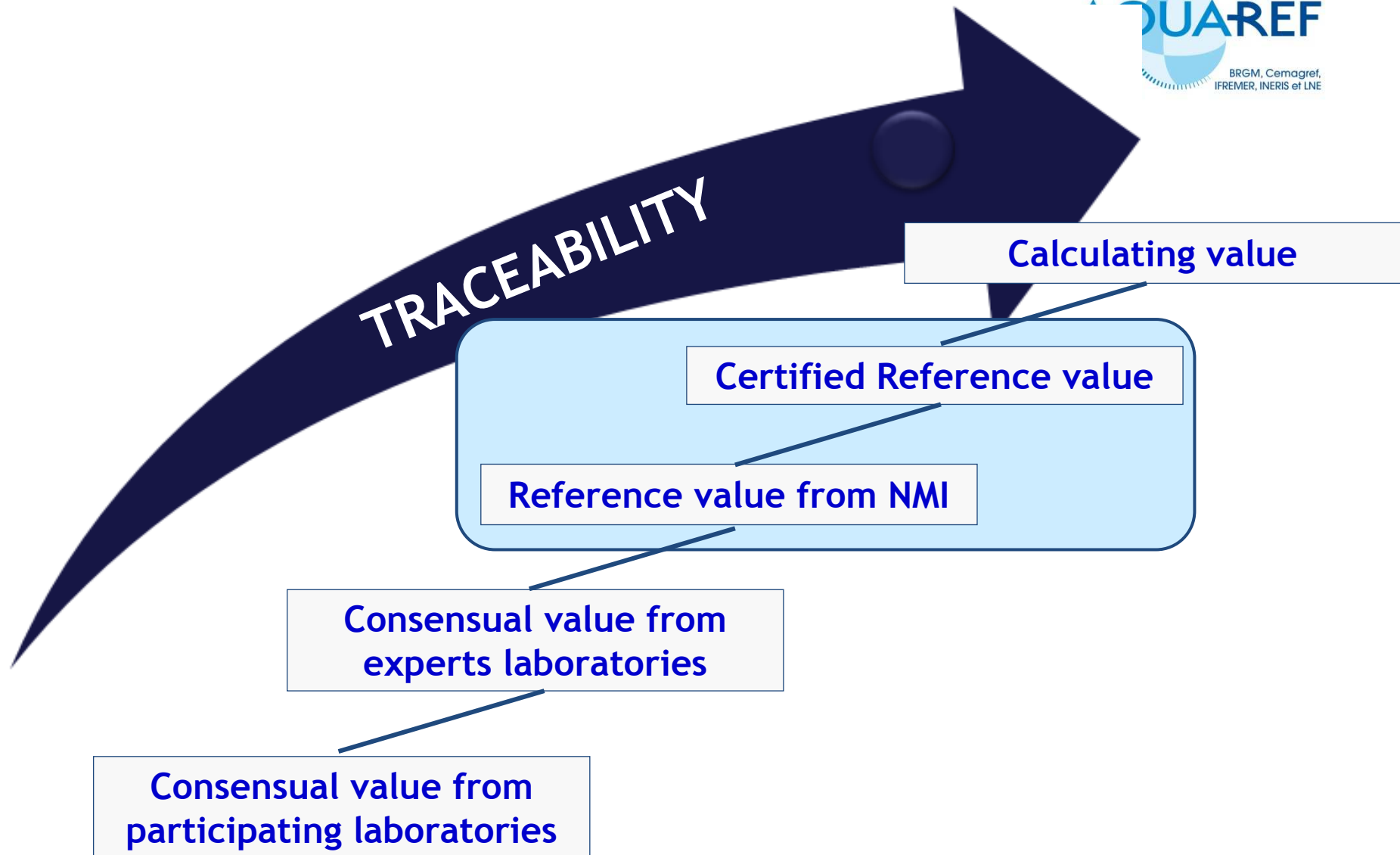
METROLOGIC INFRASTRUCTURE



QA/QC

From basic concepts... (2)





Measurement chains

Various types of pollutants

PAHS (16 EPA)

Pesticides (8)

Metals ()

Various types of devices



Various types of sites of deployment



- Relevance with the objectives of the inter comparison exercise;
- State of knowledge;
- Analytical capabilities;
- Acceptable costs

Thau site

Various types of analytical preparation

Solvent desorption Thermo desorption

Solvent desorption Thermo desorption

Acid Extraction

Various types of instrumental analysis



... To the final implemented QA/QC strategy (5)

Various types of pollutants

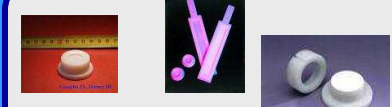
PAHS (16 EPA)

Pesticides (8)

Metals ()

REFERENCE PS → DEMONSTRATION of ACCURACY+UNCERTAINTIES

Various types of devices



Each PS pre-deployed → DEMONSTRATION of precision, minimum

Various types of

Analytical extract of PS → DEMONSTRATION of precision, minimum

Various types of

Relevant PS pre-deployed → DEMONSTRATION of precision, minimum

REFERENCE SOLUTIONS → ESTABLISHMENT of TRACEABILITY

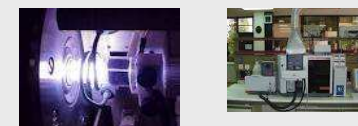
Various types of instrumental analysis



LC-Fluo/DAD/UV GC-MS

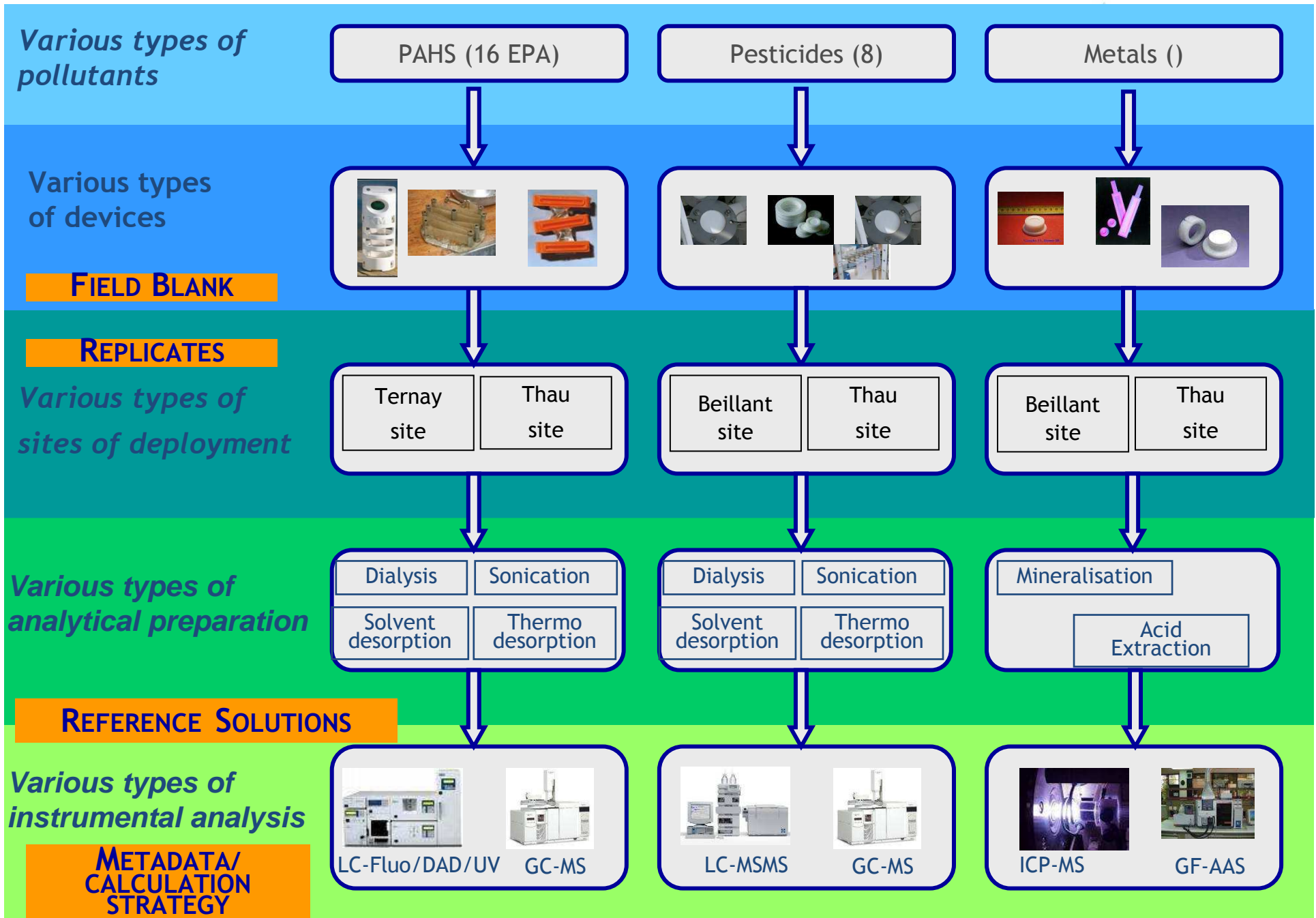


LC-MSMS GC-MS



ICP-MS GF-AAS

... To the final implemented QA/QC strategy (7)



QA/QC in the AQUAREF inter comparison exercise

Data base overview

Data base Overview (1)



Home Programmes Help

INTERLABORATORY TRIAL PASSIVE SAMPLERS 2010

April-July 2010: Passive samplers intercalibration exercise: 2010

This intercalibration is organized under the umbrella of AQUAREF, program 2010 (French laboratories involved in water monitoring <http://www.aquaref.fr>).

The objective is intercalibration exercise between expert laboratories involved in passive samplers development and deployment. The results of intercalibration exercises will be disseminated to French laboratories in charge of water monitoring. The main goal is to assess the potential role and efficiency of passive samplers for water pollutants measurements in coastal water. The targeted pollutants are the selected pesticides, the PAH and metals.

[Assistance Reporting Results](#)

RESULTS REPORTING

- [Access member area](#)
- [Application for Registration](#)
- TO ACCESS**
 - Login :
 - Password :
 - Choose your trial :
 - Make your choice
 - OK

You have 3 attempt(s) remaining
[Already registered](#) : [Forgotten your password?](#)

www.ineris.fr/eil/passivesamplers.php

Personal login and password



Data base Overview (2)



Transmission and validation Results



The screenshot shows the EIL INERIS website interface. At the top, there are logos for the French Republic and INERIS. The main header reads "EIL Essais Inter Laboratoires". Below this, there are navigation links for "Home", "Programmes", and "Help". The main content area is titled "INTERLABORATORY TRIAL PASSIVE SAMPLERS 2010".

On the left, there is a "Results Form : Parameters / site" table with columns for "Field blank", "Passive sampler 1", "Passive sampler 2", and "Passive sampler 3". The table contains numerical data for various parameters like "Date of analysis", "Volume of sample", "Temperature (°C)", "pH", "Conductivity", "Turbidity", "Total suspended matter", "Total dissolved matter", "Total phosphorus", "Total nitrogen", "Ammonium", "Nitrite", "Nitrate", "Calcium", "Magnesium", "Sulfate", "Chloride", "Fluoride", "Iron", "Copper", "Zinc", "Lead", "Cadmium", "Mercury", "Manganese", "Nickel", "Chromium", "Cobalt", "Molybdenum", "Selenium", "Vanadium", "Copper", "Zinc", "Lead", "Cadmium", "Mercury", "Manganese", "Nickel", "Chromium", "Cobalt", "Molybdenum", "Selenium", "Vanadium".

On the right, there is a "Passive samplers intercalibration" questionnaire form. The form includes fields for "Year" (set to 2010) and "Institute". Below these are several sections with yellow input fields: "Sampling site : BEILLANT", "Passive sampler (PS)", "Performance and Reference Compound (PRC)", "Storage", and "In situ deployment". A large "Metadata" watermark is overlaid on the questionnaire form.

At the bottom of the questionnaire form, there is a "Welcome the questionnaire" section with instructions: "Once the completed questionnaire, save it. Go to the website of EIL INERIS: www.ineris.fr/eil/ Choose the EIL '10_PS_ Questionnaire' Add your user name and password. On the page that appears next, click the button 'Browse ...' to select your questionnaire, then click the button 'Save' at which time your questionnaire is sent electronically to INERIS. On this same page, click 'OK ...' to stop sending the questionnaire."

Personal login and password



Data base Overview (3)



Extraction database
X File « DATA BRUT »



Site / QC or sampler / unit	Parameter	Personal Identification Participant	Value 1	Value 2	Value 3	Value 4	Metadata (40 colon for metadata)	
ETANG THAU - PAH - Solution A en µg/ml	Acenaphtene	10101	2.15	2.08	2.1	2		
ETANG THAU - PAH - Solution A en µg/ml	Site / QC or sampler / unit	Parameter	Personal Identification Participant	Value 1	Value 2	Value 3	Value 4	Metadata (40 colon for metadata)
ETANG THAU - PAH - Solution A en µg/ml	"	"	"	"	"	"	"	
"	"	"	"	"	"	"	"	
"	"	"	"	"	"	"	"	
TERNAY - PAH - Solution A en µg/ml	Acenaphtene	10101	2.15	2.08	2.1	2		
TERNAY - PAH - Solution A en µg/ml	Acenaphtene	10102	2.15	2.08	2.1	2		
TERNAY - pesticides - Solution A en µg/ml	Diuron	10101	2.15	2.08	2.1	2		
TERNAY - pesticides - Solution A en µg/ml	Diuron	10102	2.15	2.08	2.1	2		
TERNAY - pesticides - Solution A en µg/ml	Site / QC or sampler / unit	Parameter	Personal Identification Participant	Value 1	Value 2	Value 3	Value 4	Metadata (40 colon for metadata)
"	"	"	"	"	"	"	"	
"	"	"	"	"	"	"	"	
"	"	"	"	"	"	"	"	
BEILLANT - pesticides - Solution A en µg/ml	Simazine	10101	2.15	2.08	2.1	2		
BEILLANT - pesticides - Solution A en µg/ml	Simazine	10102	2.15	2.08	2.1	2		
BEILLANT - pesticides - Solution A en µg/ml	Simazine	XXXXX						
"	"	"						
"	"	"						
"	"	"						



Data base Overview (4)



Site / QC or sampler / unit	Parameter	Personal Identification Participant	Value 1	Value 2	Value 3	Value 4	Metadata (40 colon for metadata)
TERNAY - PAH - Solution A en µg/ml	Acenaphtene	10101	2.15		1	2	
TERNAY- PAH - Solution A en µg/ml	Acenaphtene	10102				2	
TERNAY - PAH- Solution A en µg/ml	Acenaphtene	XXXXX					
"	"	"					
"	"	"					
"	"	"					

Each File. XLS
« DATA BRUT »

ESSAIS D'APTITUDE des laboratoires par intercomparaison
* EIL ANALYTIQUE - INERIS *
TRAITEMENT STATISTIQUE sur les données remis par les participants

Validé

Après avoir choisi, dans le dossier 'DataBrut', un fichier xxxxx>DataBrut.XLS provenant de l'export de l'ensemble des résultats issus des saisies des participants, cette application sous EXCEL va créer un fichier xxxxxAnaStat.XLS disponible dans le dossier 'AnaStat'. Ces données xxxxxAnaStat.XLS sont destinées pour l'avis des experts (étape 2) et l'analyse statistique ultérieure (étape 3).

1/ Préparation Statistique ...
Pour Avis d'Expert en amont (Arrondissement décimales + Ajout de formules, BarreGraphes et Droite de HENRY)

2/ Avis d'Expert ...

3/ Avis d'Expert ...

3 0a du 28/03/11 sous Excel 2007 ©INERIS 2000-2011

QUITTER l'application
Recommandé (sans sauvegarder)

INFO...

Les Versions...

Accès au calculateur...

EIL Essais Inter Laboratoires

Statistical tool

Assigned value of solution A Robust Approach

Parameter en µg/mL	robust mean	robust standard deviation	Uncertainty assigned value	Standard deviation within laboratory	Standard deviation between laboratory	Repeatability en %	Reproducibility en %
Benzo_a_pyrène	2.364	0.938	0.303	0.113	0.936	4.0%	39.9%
Benzo_b_fluoranthène	2.270	0.722	0.250	0.128	0.719	5.3%	32.2%
Benzo_k_fluoranthène	2.254	0.613	0.205	0.107	0.610	6.2%	27.5%
Indéno_1,2,3c,d_pyrène	2.236	1.190	0.372	0.087	1.189	4.6%	53.3%
Acénaphthène	2.006	0.441	0.134	0.048	0.441	2.4%	22.1%
Anthracène	1.954	0.413	0.122	0.058	0.411	2.5%	21.3%
Benzo_g,h,i_pérylène	2.297	1.233	0.374	0.112	1.232	5.8%	53.8%
Fluoranthène	2.002	0.390	0.115	0.069	0.388	4.5%	19.7%
Fluorène	2.004	0.462	0.136	0.072	0.461	3.2%	23.3%
Naphtalène	2.113	0.520	0.196	0.061	0.519	2.8%	24.7%

QA/QC in the AQUAREF inter comparison exercise

Statistical treatment

Statistical treatment of QC A (1)



- According to the standards and guidelines
 - ISO 13528 (2005) « Statistical methods for use in proficiency testing by interlaboratory comparisons »
 - ISO 5725-5 (1998) « Accuracy (trueness and precision) of measurement methods and results – Part 5 : Alternative methods for the determination of the precision of the standard measurement method »
- Different approaches to determine the assigned value
 - Known values from formulation
 - Certified reference values
 - Reference values
 - Consensus values from expert laboratories
 - **Consensus values from participants**



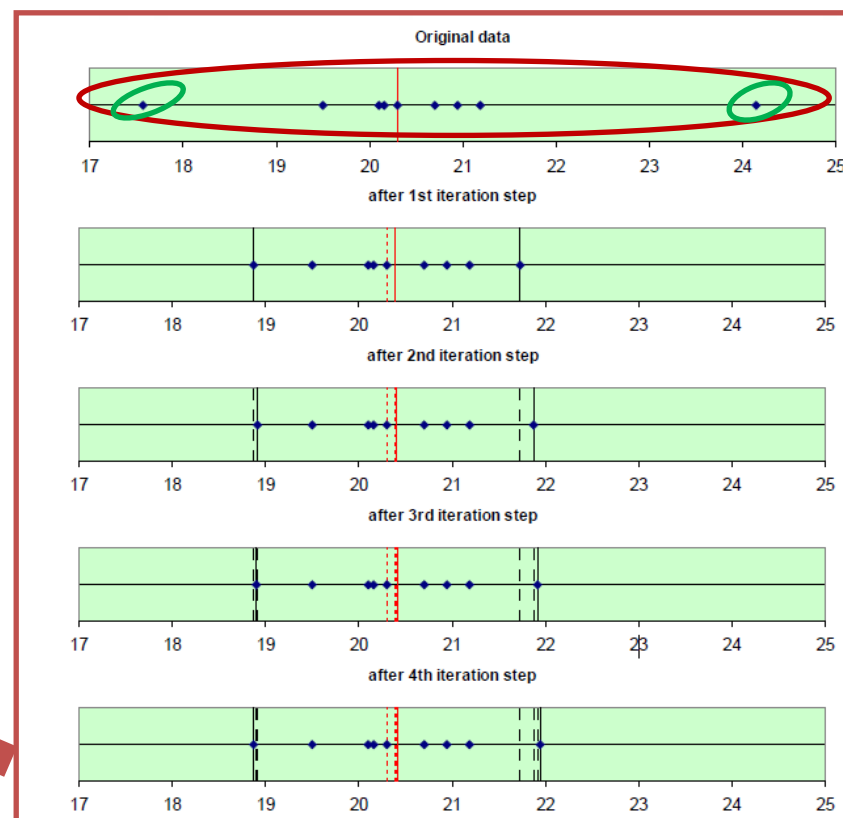
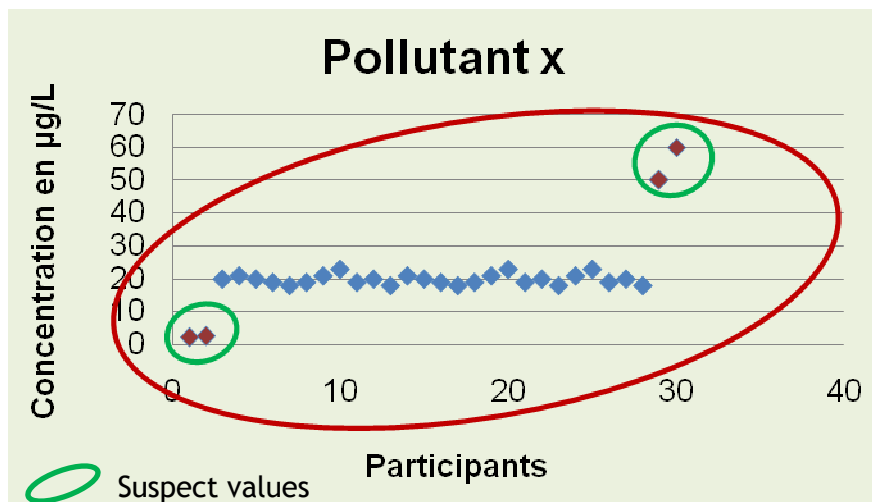
With this approach, the assigned value is the robust average of the results reported by all the participants: No exclusion of participants

Statistical treatment of QC A (2)

- Consensus values from participants
- Method implemented : Robust method



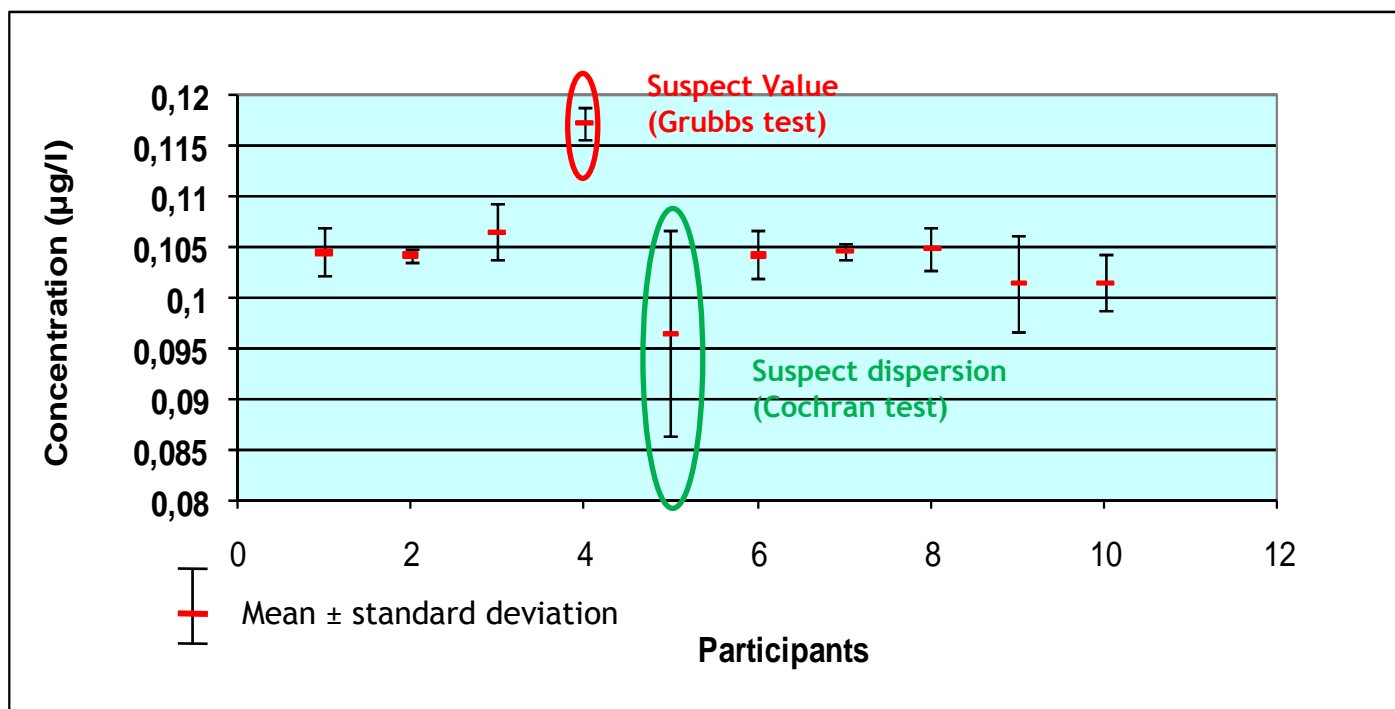
Calculate the assigned value and other statistical parameters from all data including those that might be deemed suspicious by an expert or a test for outliers. Data is processed to minimize the weight of suspect values, so that these do not significantly impact the result.



After x iteration
For each parameter :
✓ robust mean
✓ robust standard deviation

Statistical treatment of QC A (3)

- Research statistically different values
 - **Cochran test** : is a test of the **within-laboratory** variability
 - **Grubbs test** : is a test of **between-laboratory** variability



- Suspect values are studied to find correlation with
 - ✓ the implemented analytical strategies (metadata provided during the reporting)
 - ✓ the results of passive samplers measurements

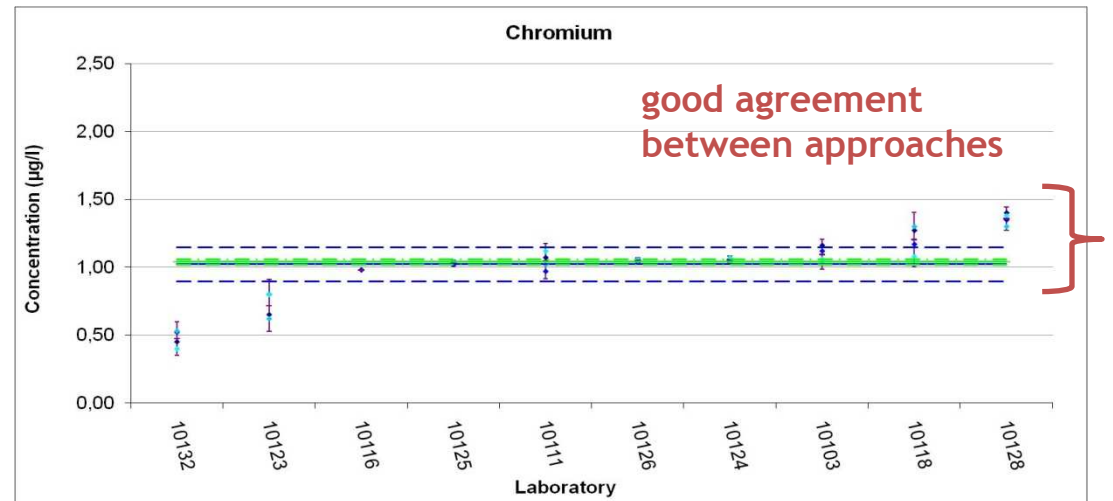
Statistical treatment of QC A (4)



- Comparison of the assigned value between :

Robust method	Based on consensus values from participants	Robust mean and Robust standard deviation
Reference value	Based on reference solution A	Reference value and uncertainty (k=2)

- In order to identify :
 - ✓ a good agreement whatever the approach adopted
- Otherwise identify the possible reasons for non agreement
 - ✓ a common bias in the results of the laboratories,
 - ✓ biased participant method(s) or several biased laboratories



Statistical treatment of QC A (5)



- This statistical treatment chosen for this trial was implemented

✓ Class of parameters and site

- ✓ PAH/Ternay
- ✓ PAH/EtangThau
- ✓ Pesticides/Beillant
- ✓ Pesticides/Ternay
- ✓ Metals/Ternay
- ✓ Metals/Etang Thau



Total for QC A
9 statistical treatments
were performed

✓ Class of parameters and all sites

- ✓ PAH/Ternay + Etang Thau
- ✓ Pesticides/Beillant + Ternay
- ✓ Metals/Ternay + Etang Thau



Last option was made possible because reference solution (sol A) was the same regardless of the site

QA/QC in the AQUAREF inter comparison exercise

**Reference solutions:
From conception to the assignation
of the final value**

Reference solutions: Summary









**TARGETS
EIL**

PAHS (16 EPA)

Pesticides (8)

Metals (8)

**TARGETS
REFERENCE
SOLUTION**

- ✓ Benzo (a) Pyrene 
- ✓ Benzo (b) Fluoranthene 
- ✓ Benzo (g, h, i) Perylene 
- ✓ Benzo (k) Fluoranthene 
- ✓ Indeno (1,2,3-cd) Pyrene 
- ✓ Naphtalene 
- ✓ Fluoranthene 
- ✓ Anthracene 
- ✓ Fluorene
- ✓ Acenaphtene

- ✓ Atrazine 
- ✓ Simazine 
- ✓ DEA
- ✓ DIA
- ✓ Diuron 
- ✓ Isoproturon 
- ✓ Alachlore 
- ✓ Acetochlore
- ✓ Metolachlore

- ✓ Cadmium 
- ✓ Nickel 
- ✓ Lead 
- ✓ Zinc 
- ✓ Copper 
- ✓ Manganese
- ✓ Cobalt
- ✓ Chromium 

SOLVENT

Acetone

Acetone

Nitric Acid (2 %)

**MASSIC
CONC.**

≈ 2 µg / ml ind.

≈ 2 µg / ml ind.

≈ 1 µg / l ind.

VOLUME

≈ 1 ml

≈ 1 ml

≈ 100 ml

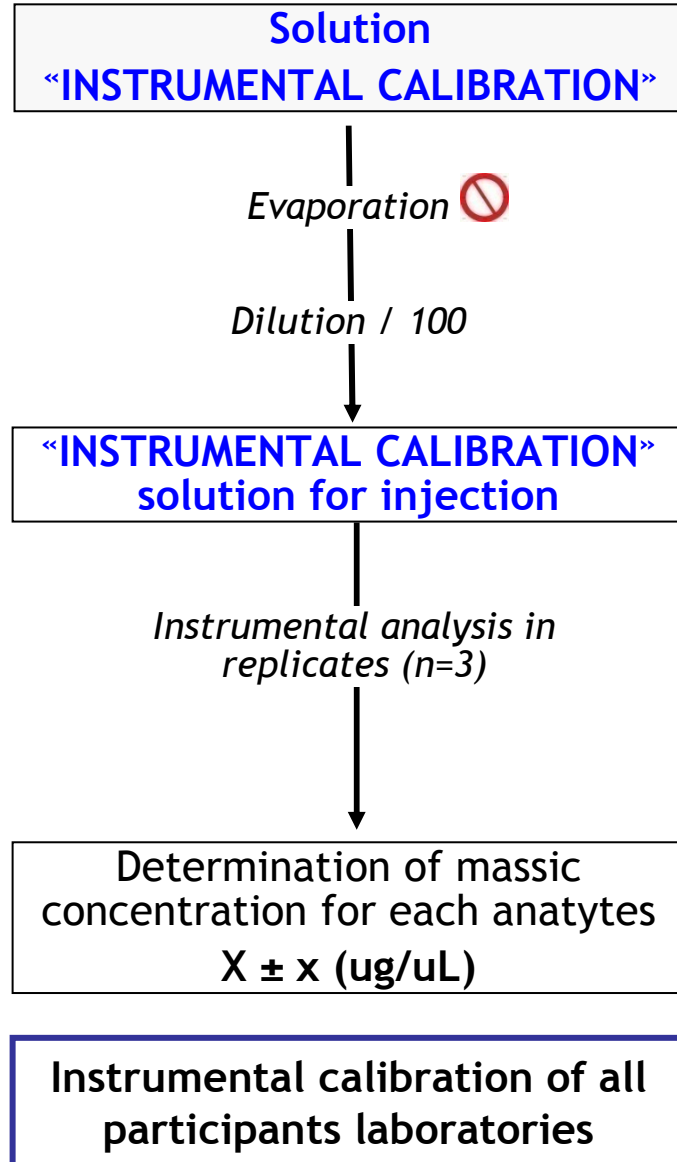


WFD (33 Priority substances)

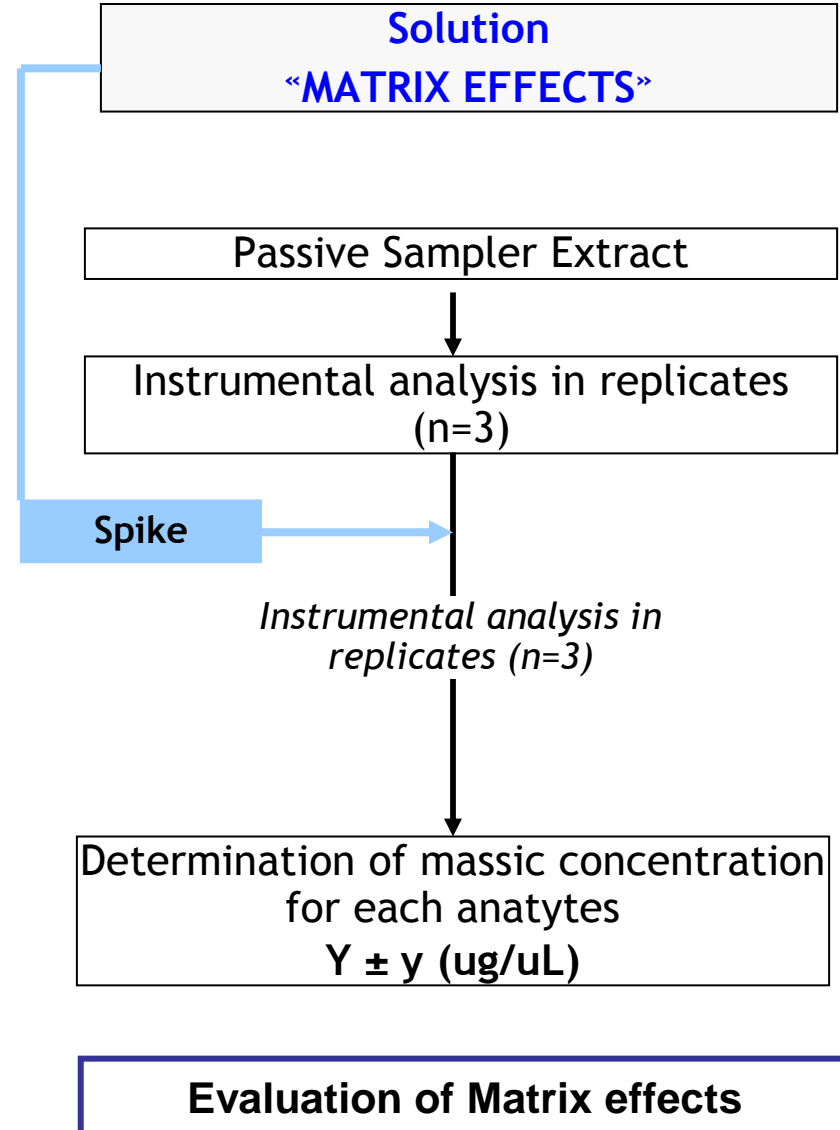


Substances of the ecological status

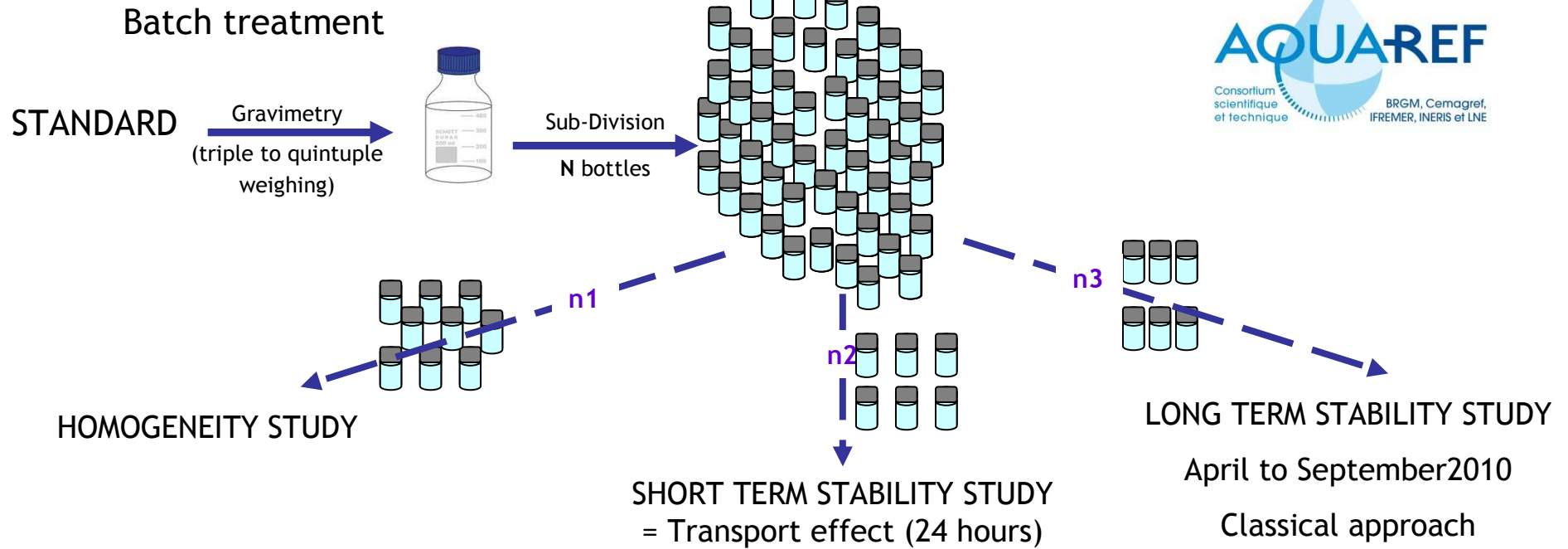
ORGANIC AND INORGANIC ANALYSIS OBLIGATORY



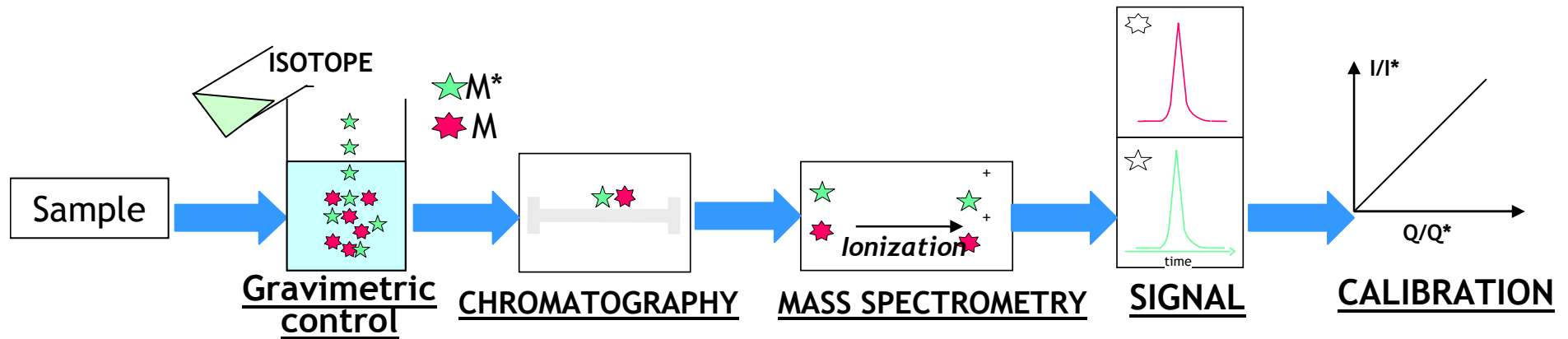
ORGANIC ANALYSIS, ONLY FACULTATIVE



PREPARATION of REFERENCE SOLUTIONS



PRIMARY METHOD ID-MS: Principles



METALS



Element	Reference Value Massic concentration \pm U (U expanded, k=2)
Cadmium	1.042 \pm 0.012 $\mu\text{g/l}$
Cobalt	1.005 \pm 0.080 $\mu\text{g/l}$
Chromium	1.040 \pm 0.020 $\mu\text{g/l}$
Copper	1.099 \pm 0.044 $\mu\text{g/l}$
Manganese	1.002 \pm 0.080 $\mu\text{g/l}$
Nickel	1.035 \pm 0.023 $\mu\text{g/l}$
Lead	1.049 \pm 0.015 $\mu\text{g/l}$
Zinc	1.025 \pm 0.071 $\mu\text{g/l}$

The study demonstrates :

- No inhomogeneity
- No instability

— — — ➔ Attribution of reference value with $U < 10\%$

PESTICIDES



	Reference Value Massic concentration \pm U (U expanded, $k=2$)
Alachlore	$2.05 \pm 0.09 \mu\text{g/ml}$
Acétochlore	$1.97 \pm 0.12 \mu\text{g/ml}$
DEA	$1.89 \pm 0.14 \mu\text{g/ml}$
DIA	$2.04 \pm 0.12 \mu\text{g/ml}$
Atrazine	$1.99 \pm 0.04 \mu\text{g/ml}$
Isoproturon	$2.02 \pm 0.08 \mu\text{g/ml}$
Diuron	$2.03 \pm 0.18 \mu\text{g/ml}$
Simazine	$2.23 \pm 0.1 \mu\text{g/ml}$
Métolachlore	$2.12 \pm 0.14 \mu\text{g/ml}$

The study demonstrates :

- No inhomogeneity
- No instability

— — — ➔ Attribution of reference value with $U < 10\%$

PAHs

	Reference Value Massic concentration \pm U (U expanded, k=2)
Naphtalene	$1.96 \pm 0.12 \mu\text{g/ml}$
Fluoranthene	$1.97 \pm 0.12 \mu\text{g/ml}$
Benzo (b) Fluoranthene	$1.99 \pm 0.12 \mu\text{g/ml}$
Benzo (k) Fluoranthene	$1.93 \pm 0.16 \mu\text{g/ml}$
Benzo (a) Pyrene	$1.87 \pm 0.12 \mu\text{g/ml}$
Indeno (1,2,3-cd) Pyrene	$1.73 \pm 0.36 \mu\text{g/ml}$
Benzo (g, h, i) Perylene	$1.88 \pm 0.32 \mu\text{g/ml}$
Acenaphtene	$1.94 \pm 0.12 \mu\text{g/ml}$
Anthracene	$1.93 \pm 0.48 \mu\text{g/ml}$
Fluorene	$1.93 \pm 0.08 \mu\text{g/ml}$



The study demonstrates :

- No inhomogeneity
- No instability



Attribution of reference value with $U < 10\%$, except for Anthracene, Benzo(ghi)perylene, Indeno(1,2,3-cd)pyrene

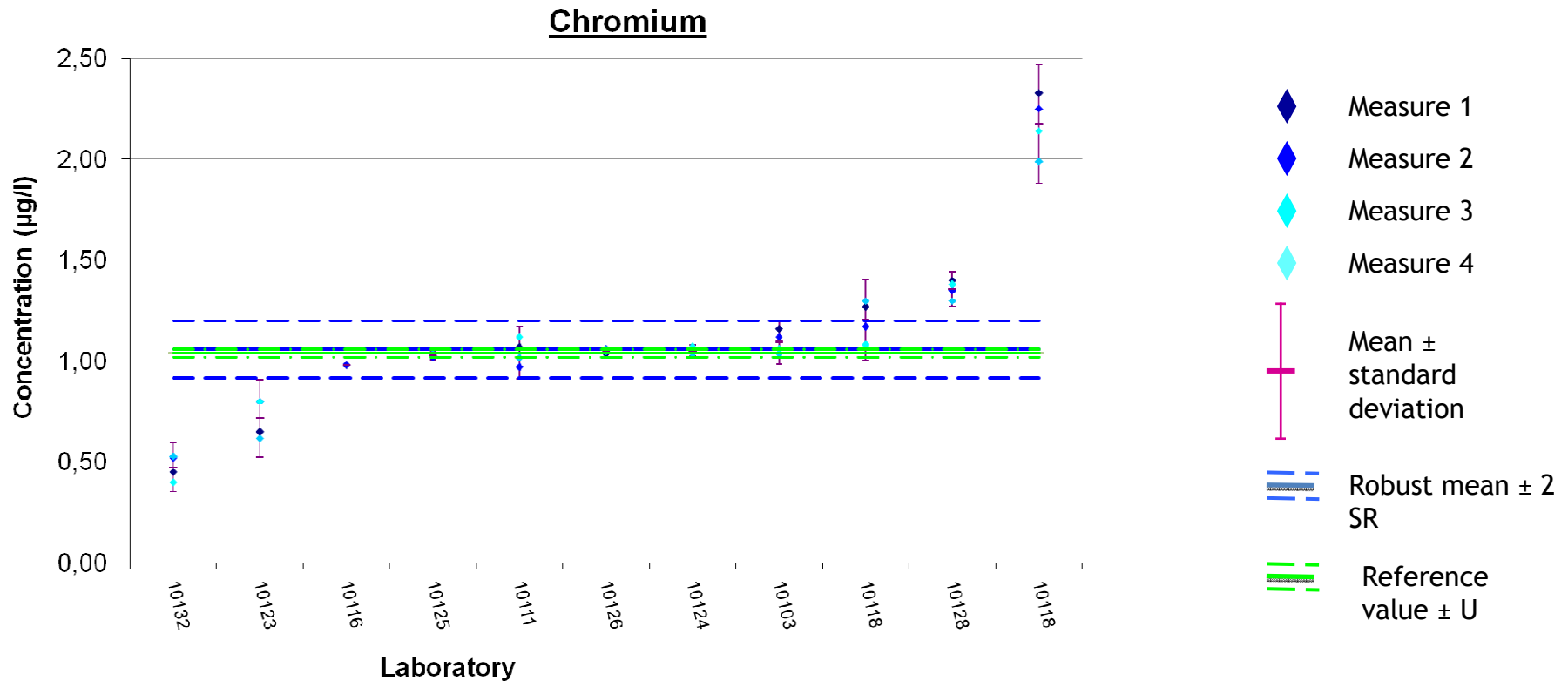
QA/QC in the AQUAREF inter comparison exercise

Presentation of results, discussions

LABORATORY QUALITY CONTROL : REFERENCE SOLUTIONS FOR VERIFICATION OF INSTRUMENT CALIBRATION (1)



METALS

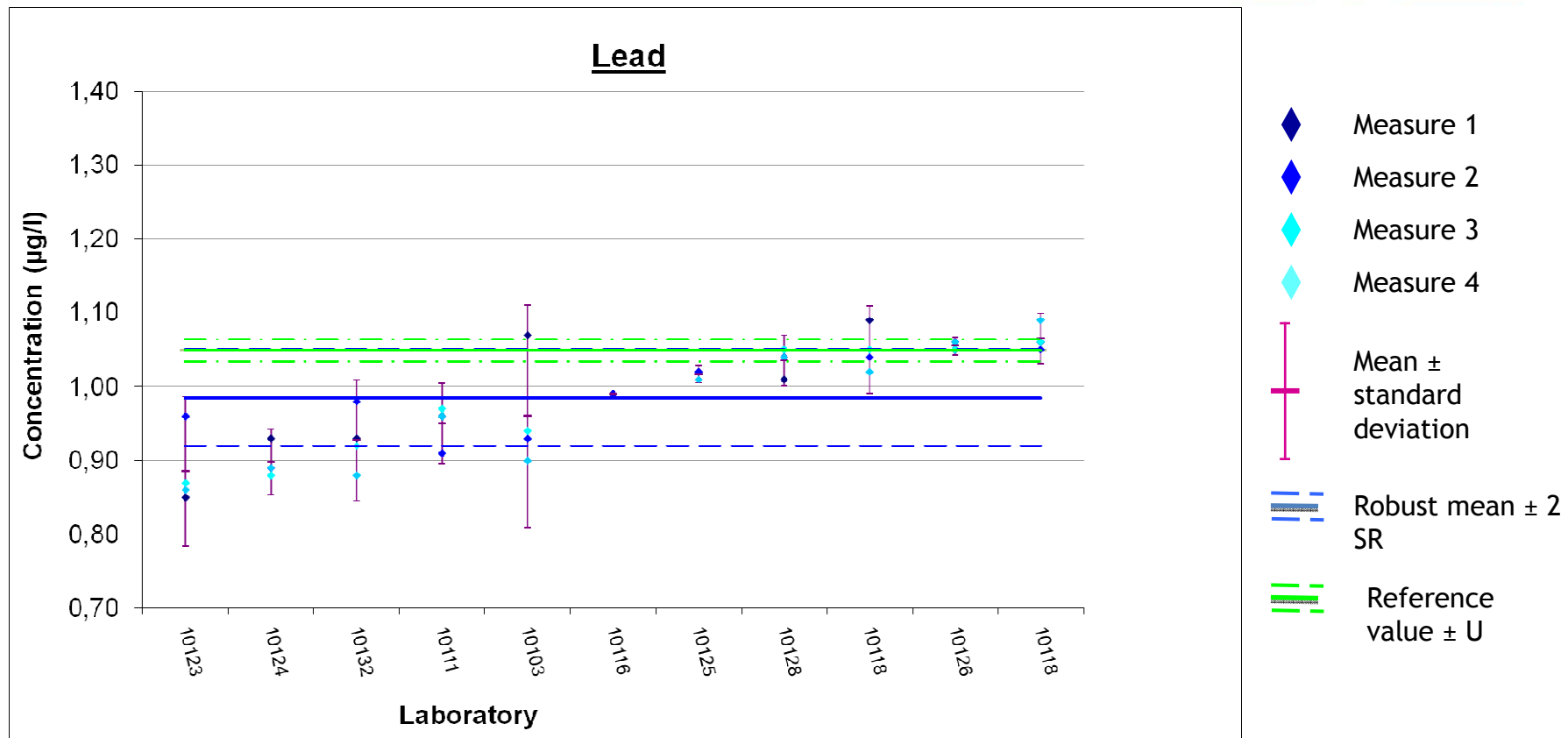


- Robust mean \approx reference value
- Accuracy : precision + trueness of measurements the general population

→ Mastery of participants

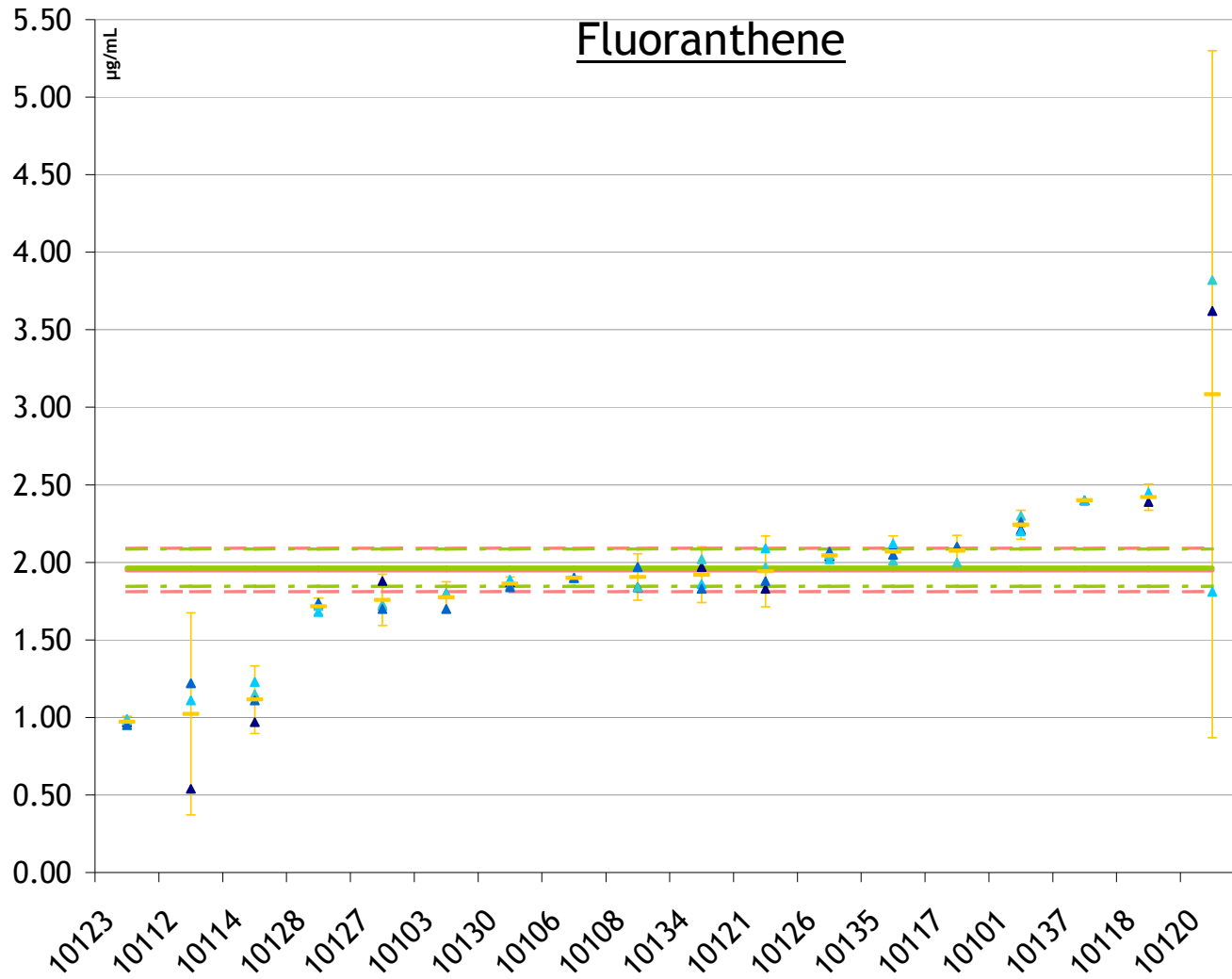
LABORATORY QUALITY CONTROL : REFERENCE SOLUTIONS FOR VERIFICATION OF INSTRUMENT CALIBRATION (2)

METALS



- No overlap between robust mean and reference value
- Lack of accuracy especially trueness : reference value needed in this case

PAHs



- ▲ Mesure 1
- ▲ Mesure 2
- ▲ Mesure 3
- ▲ Mesure 4
- Moyenne des mesures
- Moyenne robuste
- Moyenne robuste-2SR
- Moyenne robuste+2SR
- Valeur de référence

➤ Robust mean \approx reference value

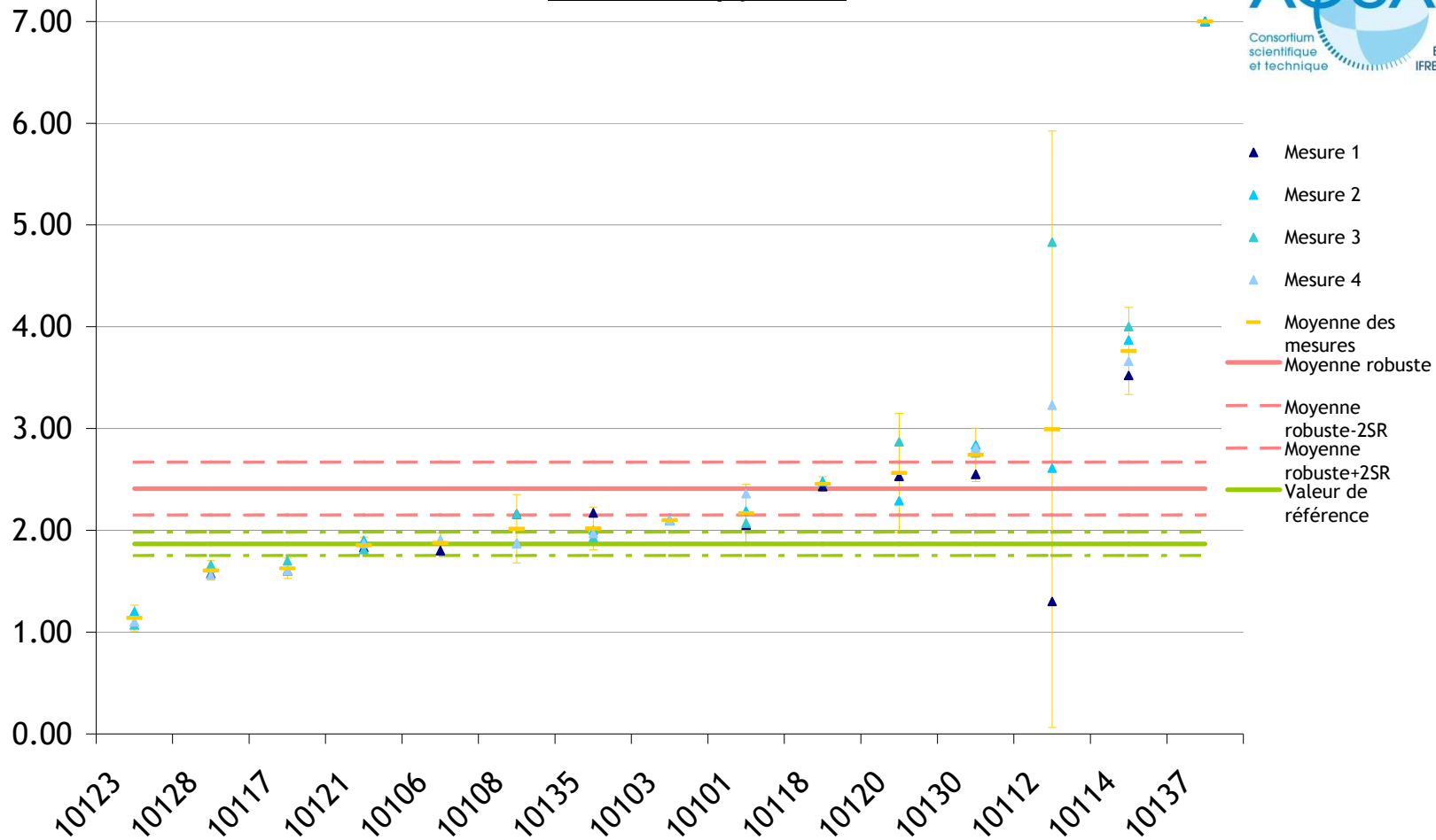
➤ Accuracy : precision + trueness of measurements the general population

—————➔ Mastery of participants

PAHs

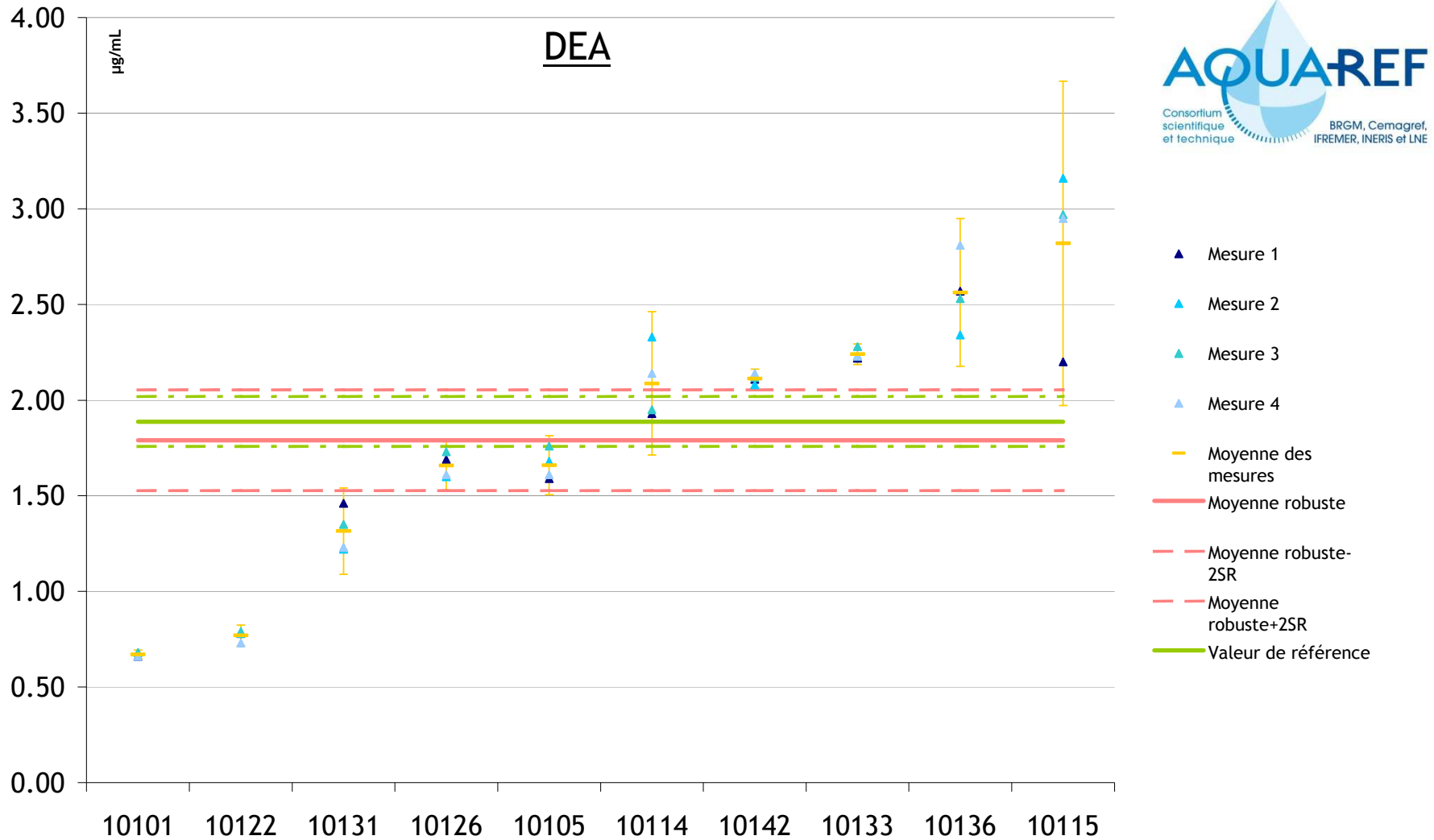
µg/mL

Benzo-a-pyrene



- No overlap between robust mean and reference value
- Lack of accuracy especially trueness : interest of reference value in such exercise

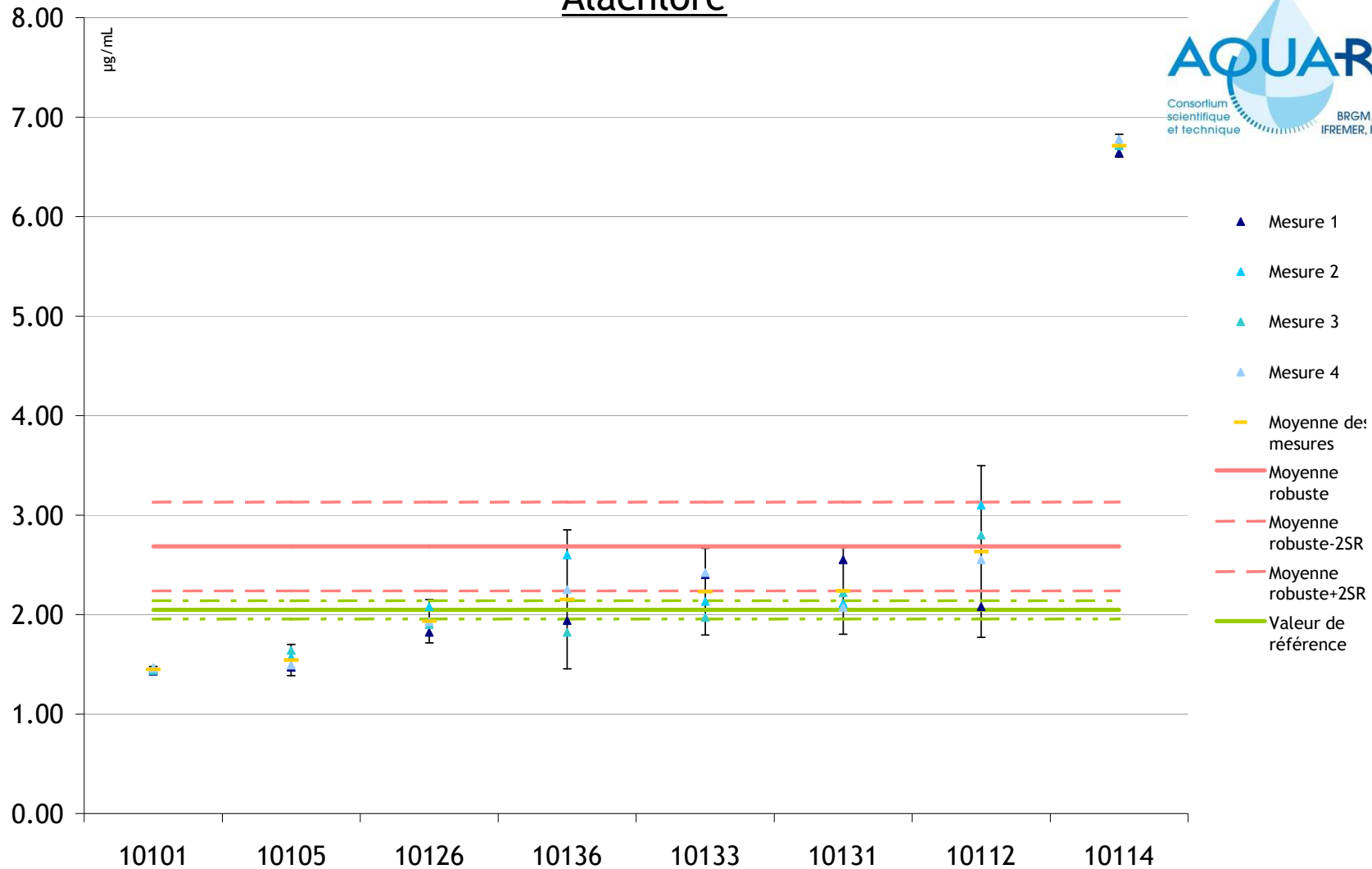
PESTICIDES



- Robust mean \approx reference value
- Accuracy : precision + trueness of measurements the general population

PESTICIDES

Alachlore

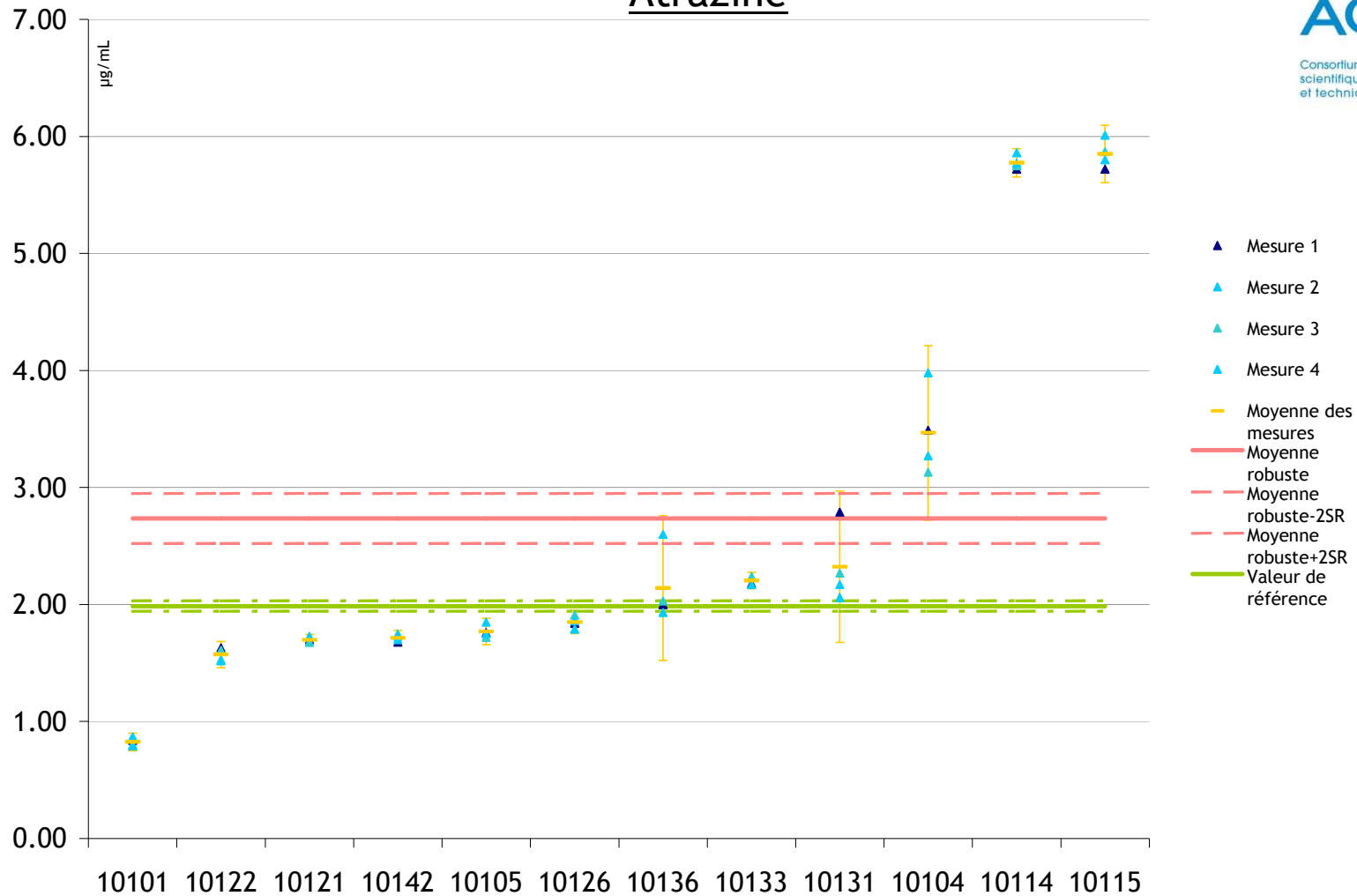


➤ No overlap between robust mean and reference value

➤ Lack of accuracy : interest of reference value in such exercise

PESTICIDES

Atrazine



➤ No overlap between robust mean and reference value

➤ Lack of accuracy especially trueness : interest of reference value in such exercise

FIELD QUALITY CONTROL : BLANK MEASUREMENTS (1/8)



METALS:

-Field blanks for metals display significant contamination depending on the element:

TERNAY participants		Field Blank	
Parameter ng/sampler	Mean	Min	Max
Cadmium	1.86	0.02	20.80
Chromium	3.62	0.80	9.10
Cobalt	0.92	0.02	6.34
Copper	6.31	1.06	26.00
Manganese	3.94	0.09	8.80
Nickel	9.42	0.51	65.00
Lead	3.63	0.05	33.97
Zinc	271.40	27.37	1300.00

THAU participants		Field Blank	
Parameter ng/sampler	Mean	Min	Max
Cadmium	0.76	0.03	3.35
Chromium	3.94	0.14	18.70
Cobalt	0.28	0.02	0.06
Copper	4.86	1.06	7.76
Manganese	2.67	0.95	4.56
Nickel	6.23	0.82	26.00
Lead	1.72	0.03	4.17
Zinc	788.49	18.00	3400.00




Discussed in the dedicated session p.m

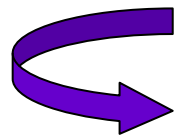
FIELD QUALITY CONTROL : BLANK MEASUREMENTS (2/8)



PESTICIDES:

-No field blanks positive except for one compound in one PS and in one site

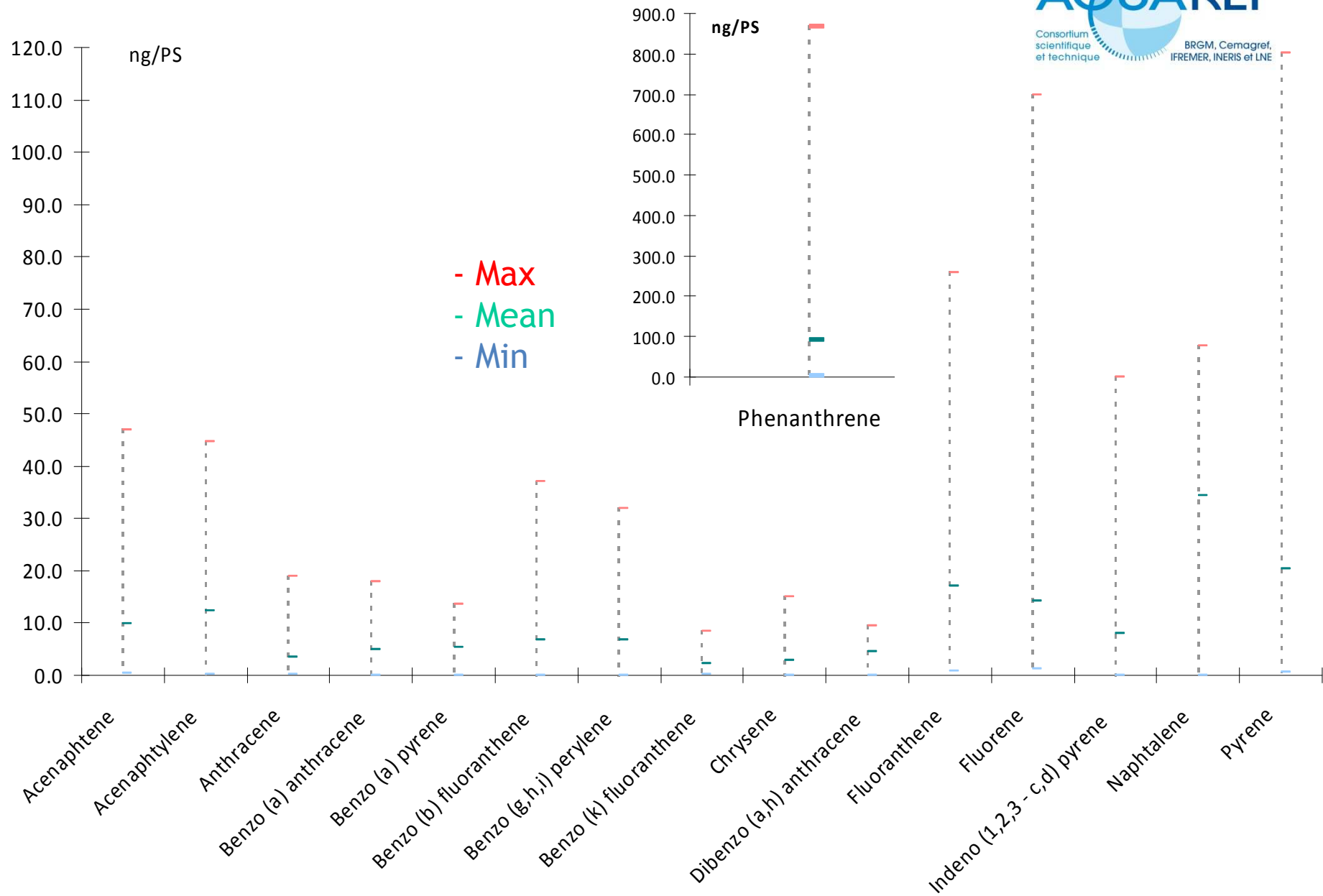
 Consistent with the physico-chemical properties of the selected molecules



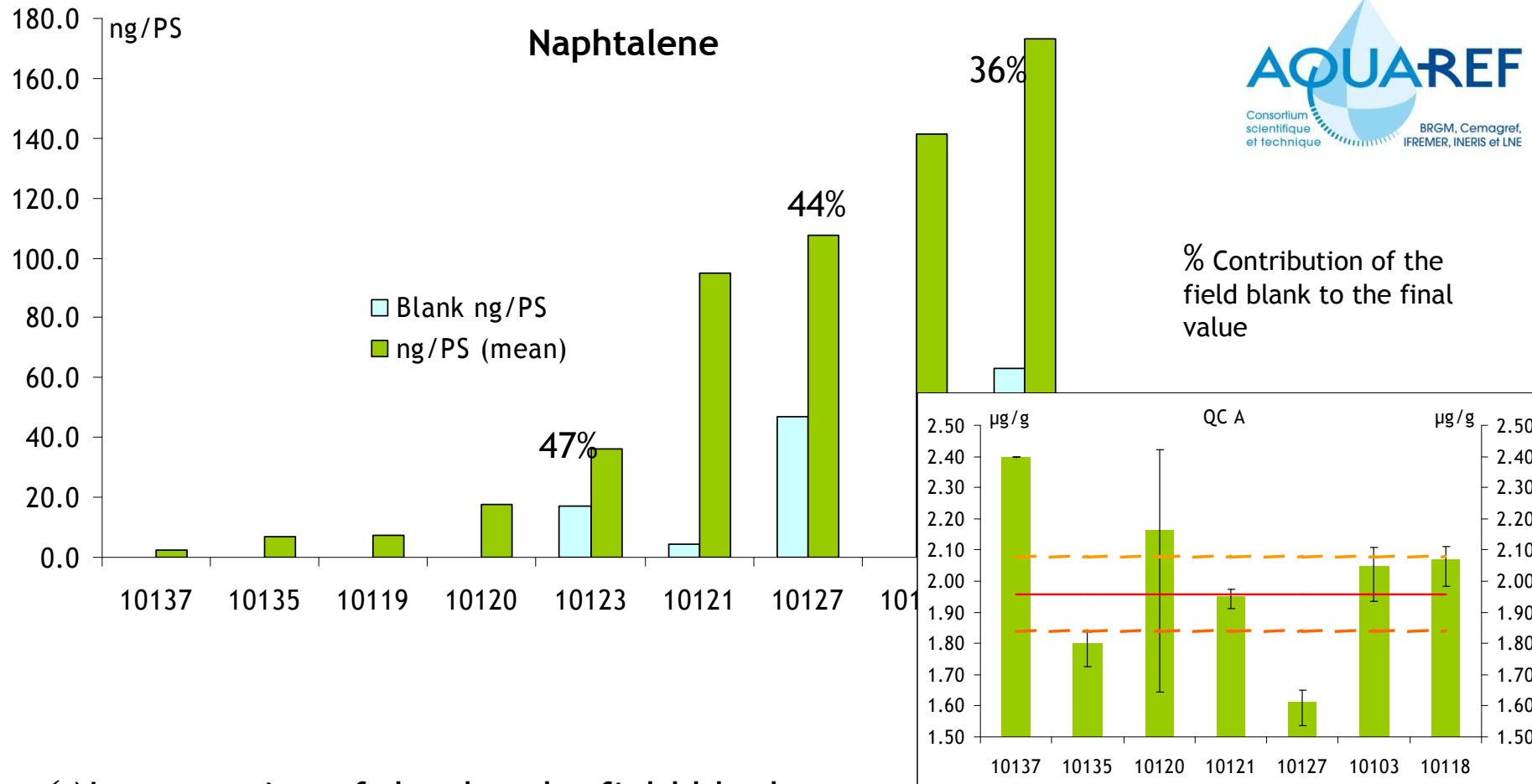
Focus on PAHs

FIELD QUALITY CONTROL : BLANK MEASUREMENTS (3/8)

Case study1: PAH Ternay

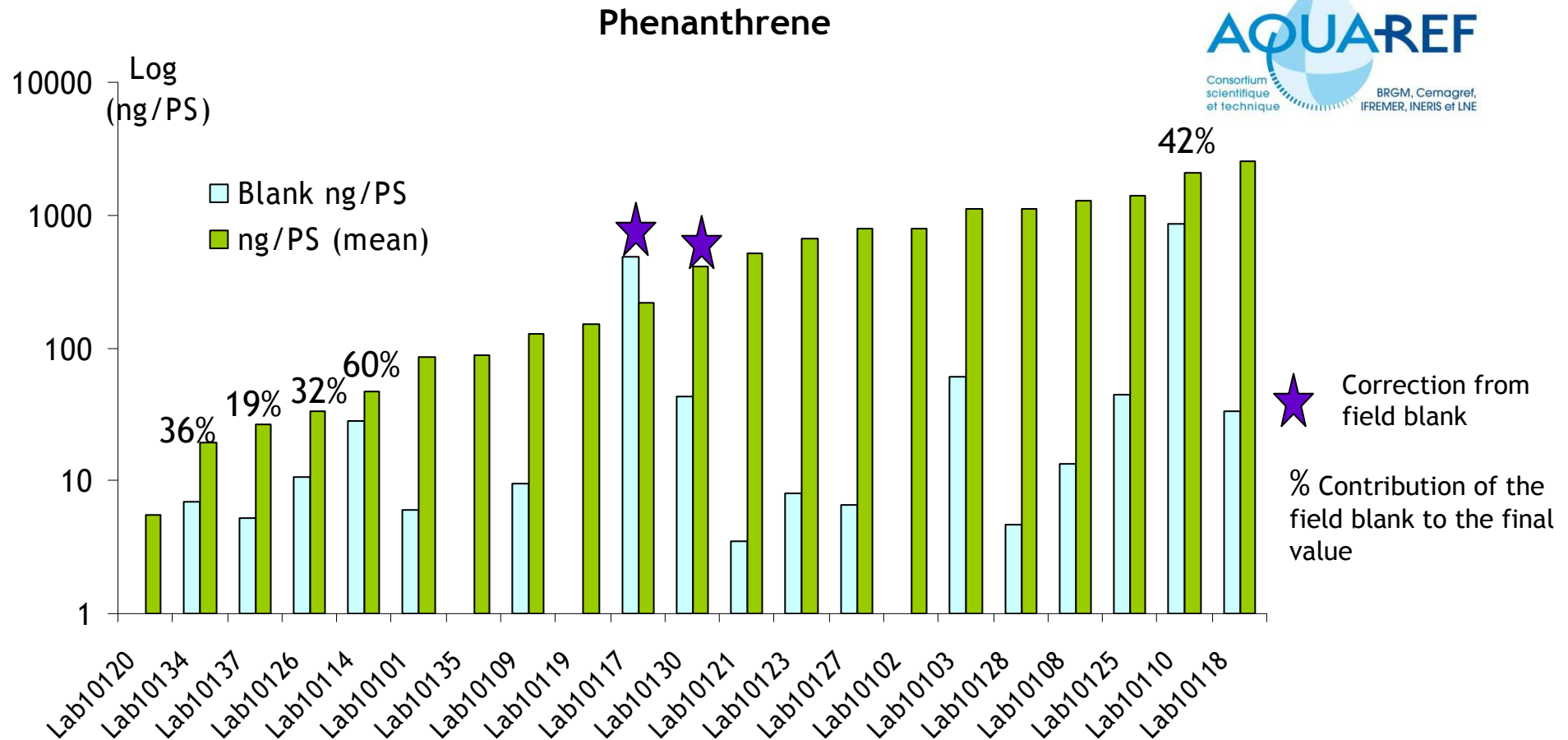


FIELD QUALITY CONTROL : BLANK MEASUREMENTS (4/8)



- ✓ No correction of the data by field blanks
- ✓ Field blanks close to 50% of deployed PS
- ✓ No correlation with the type of PS, the type of extraction technique nor type of instrumental analysis, the quantification approach
- ✓ No clear correlation with the QC A results

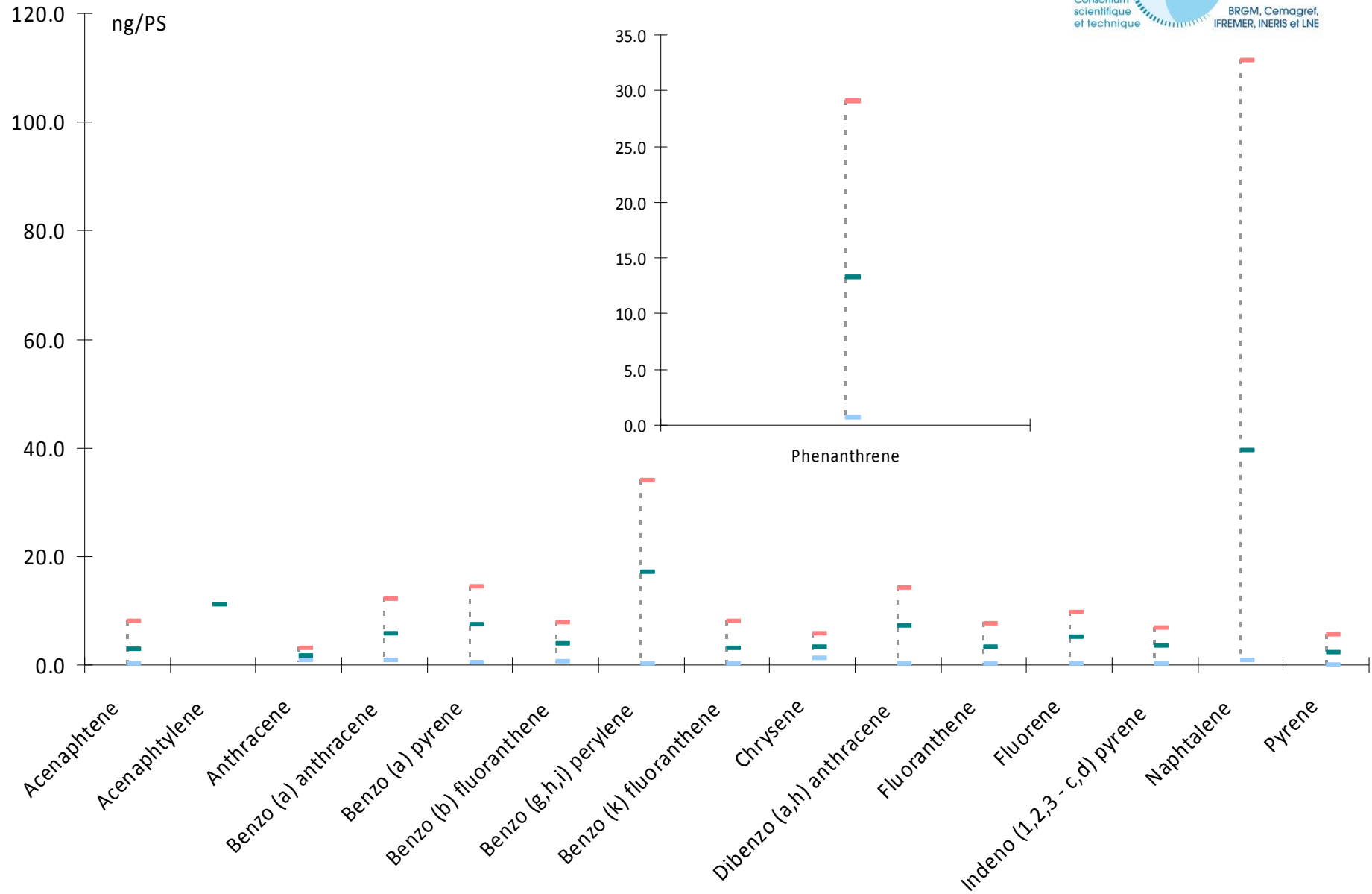
FIELD QUALITY CONTROL : BLANK MEASUREMENTS (5/8)



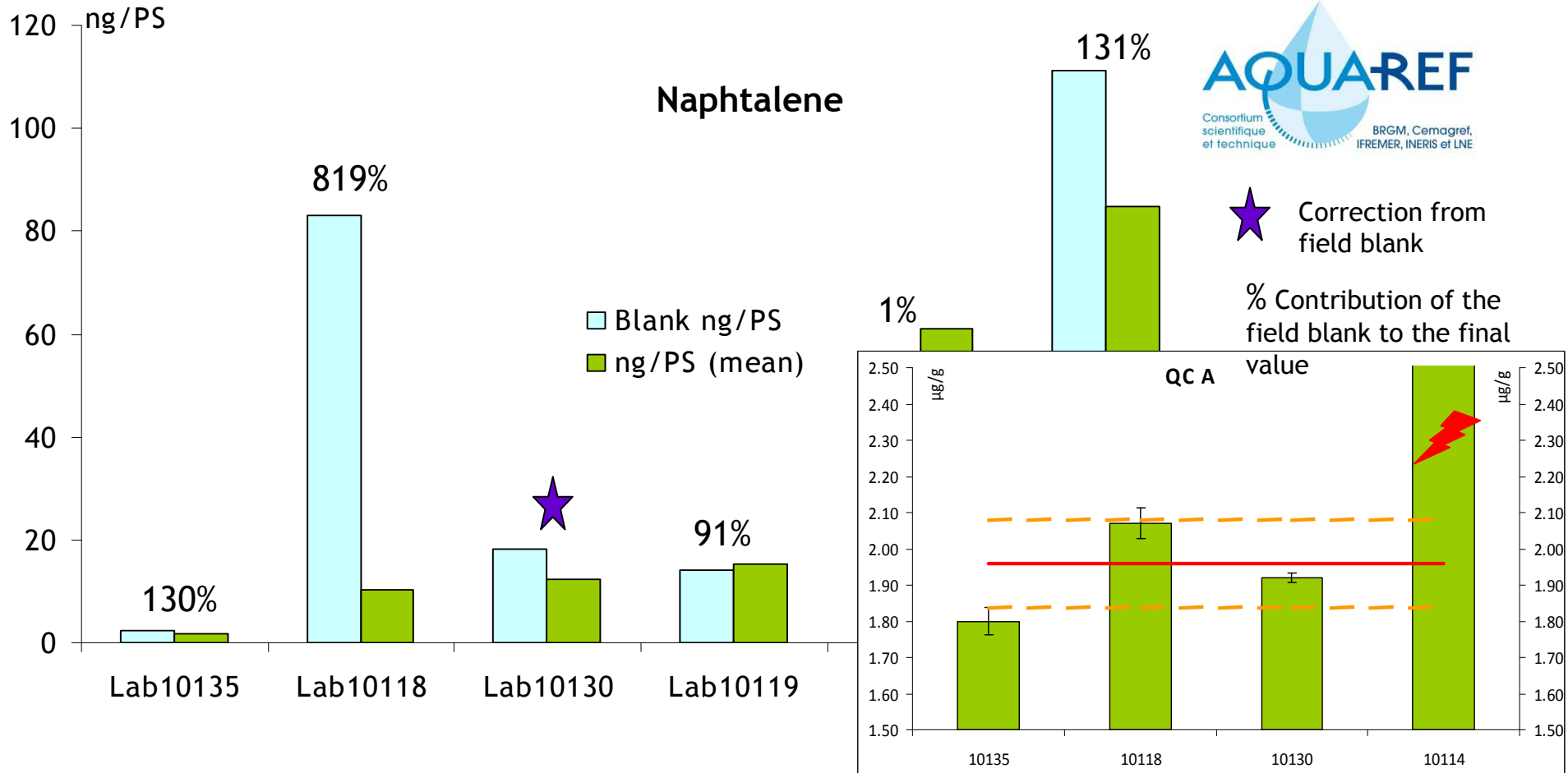
- ✓ No correction of the data by field blanks, except 2 labs.
- ✓ Field blanks close to 60% of deployed PS
- ✓ No correlation with the type of PS, the type of extraction technique nor type of instrumental analysis, the quantification approach

FIELD QUALITY CONTROL : BLANK MEASUREMENTS (6/8)

Case study 2: PAH Thau

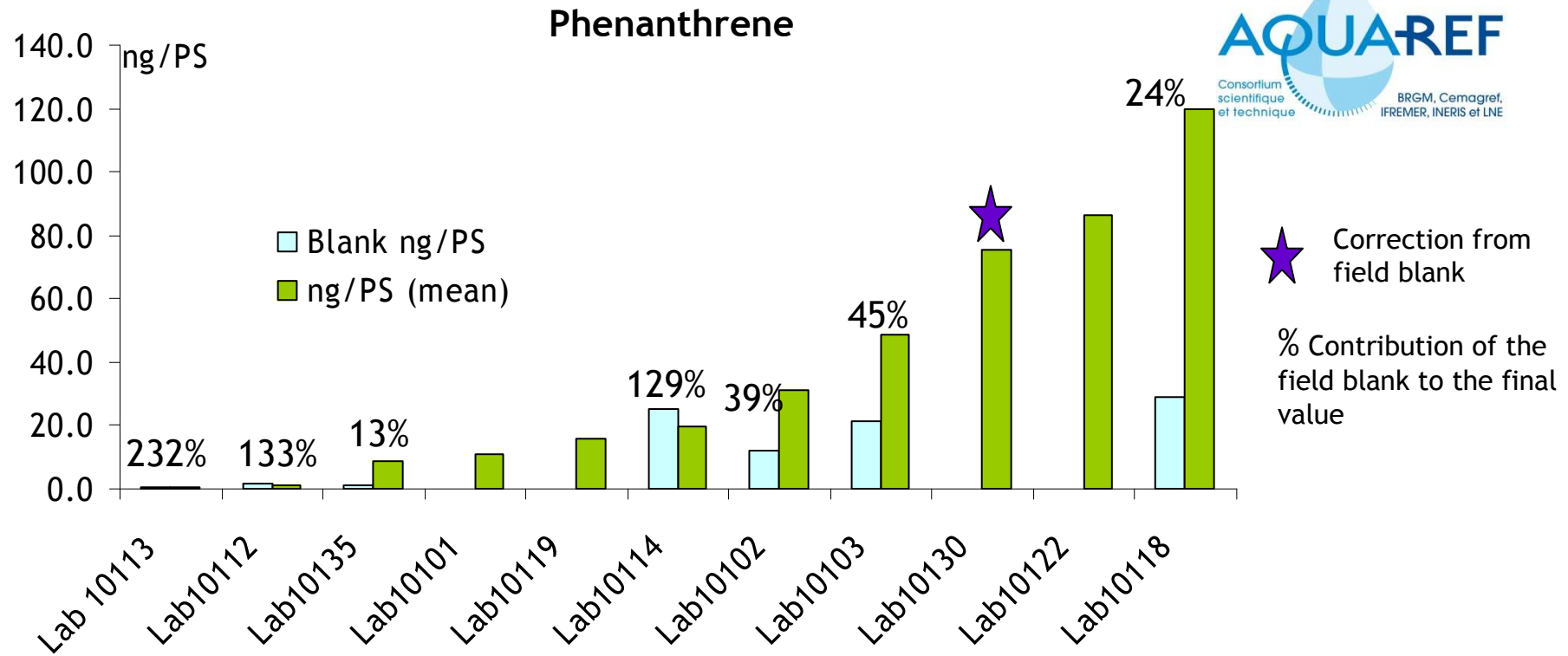


FIELD QUALITY CONTROL : BLANK MEASUREMENTS (7/8)



- ✓ No correction of the data by field blanks, except 1 lab.
- ✓ Field blanks close to 50% of deployed PS
- ✓ No correlation with the type of PS, the type of extraction technique nor type of instrumental analysis, the quantification approach
- ✓ No clear correlation with the QC A results

FIELD QUALITY CONTROL : BLANK MEASUREMENTS (8/8)



- ✓ No correction of the data by field blanks
- ✓ Field blanks > deployed PS
- ✓ No correlation with the type of PS, the type of extraction technique nor type of instrumental analysis or the quantification approach

QA/QC in the AQUAREF inter comparison exercise

Conclusion and Perspectives

Laboratory QC

- ✓ Results on reference solution (sol A) in accordance (in term of dispersion) with results of analytical intercomparison exercise in routine laboratories
- ✓ Systematic integration of control solution in analytical intercomparison exercise led to better evaluation of participants on results of these QC
- ✓ Interest of the reference value by comparison to the consensual value

Field QC

- ✓ Have to be taken into consideration
- ✓ Many issues not yet answered
- ✓ Importance of :
 - QA / QC (field and laboratory) with reference value
 - Replicat during deployment
 - Procedure (deployment and analysis)



- ✓ Importance of :
 - QA / QC (field and laboratory) with reference value
 - Replicat during deployment
 - Procedure (deployment and analysis)

- ✓ ISO 5667- *Part 23*: “*Guidance on passive sampling in surface waters*” published in February 2011
 - some aspects are to be completed light of these results
 - a procedure (deployment and analysis) for each type of passive sampler

Perspectives

Various types of pollutants

PROCEDURES/GUIDANCES

PAHS (16 EPA)

Pesticides (8)

Metals ()

Various types of devices



FIELD BLANK **BLANK ISSUE**

REPLICATES

Various types of sites of deployment

Ternay site Thau site

Beillant site Thau site

Beillant site Thau site

QC?

Various types of analytical preparation

Dialysis Sonication
Solvent desorption Thermo desorption

Dialysis Sonication
Solvent desorption Thermo desorption

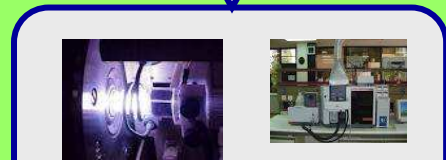
Mineralisation
Acid Extraction

REFERENCE SOLUTIONS

Various types of instrumental analysis


LC-Fluo/DAD/UV GC-MS


LC-MSMS GC-MS


ICP-MS GF-AAS

**METADATA/
CALCULATION
STRATEGY**