



**PTB**

# PT-Providers: Key Partners of European Metrology for Dissemination of Traceability in Analytical Chemistry



Detlef Schiel <sup>(1)</sup>, Olaf Rienitz <sup>(1)</sup>, Reinhard Jährling <sup>(1)</sup>, Bernd Güttler <sup>(1)</sup>, Holger Scharf <sup>(2)</sup>, Ralf Matschat <sup>(2)</sup>, Birkhahn <sup>(2)</sup>, Paola Fiscaro <sup>(3)</sup>, Guillaume Labarraque <sup>(3)</sup>, Ulrich Borchers <sup>(4)</sup>, David Schwesig <sup>(4)</sup>

(1) Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Germany, (2) Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany, (3) Laboratoire National de Métrologie et d'Essais (LNE), Paris, France, (4) IWW Water Centre, Mülheim, Germany

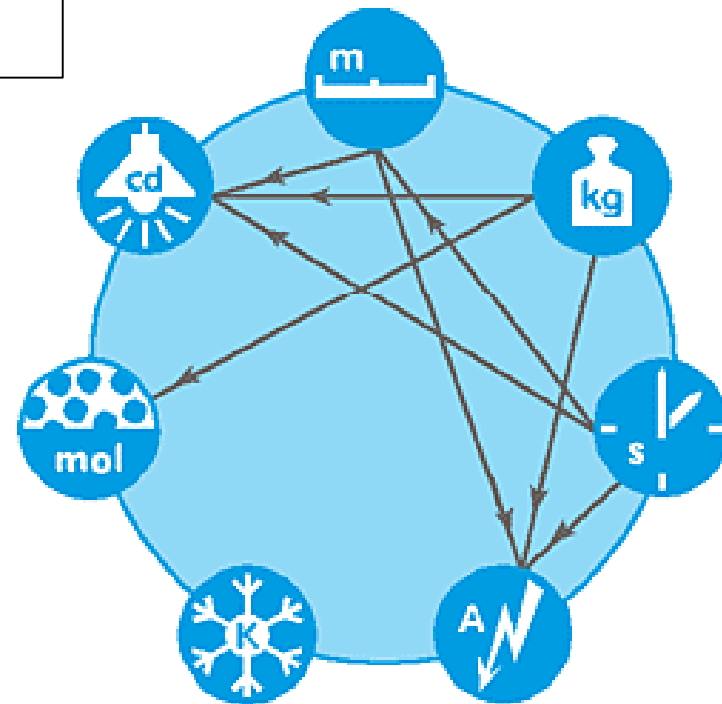


**Fundamental  
Constants**



**Federal Ministry  
of Economics  
and Technology**

- **Realization and dissemination of the SI units**
- **Provision of the basis for reliable (traceable, comparable) measurement results**



# Why comparability and traceability?

Increasingly required quality features of measurement results

## Comparability

## Traceability

EU Water Framework Directive 2000/60/EC (WFD)

Groundwater Directive 2006/118/EC

Drinking Water Directive 98/83/EC

Marine Strategy Framework Directive 2008/56/EC

In vitro Diagnostica 98/79/EG

ISO/IEC 17025

ISO/IEC 17043

# How can comparability be achieved ?

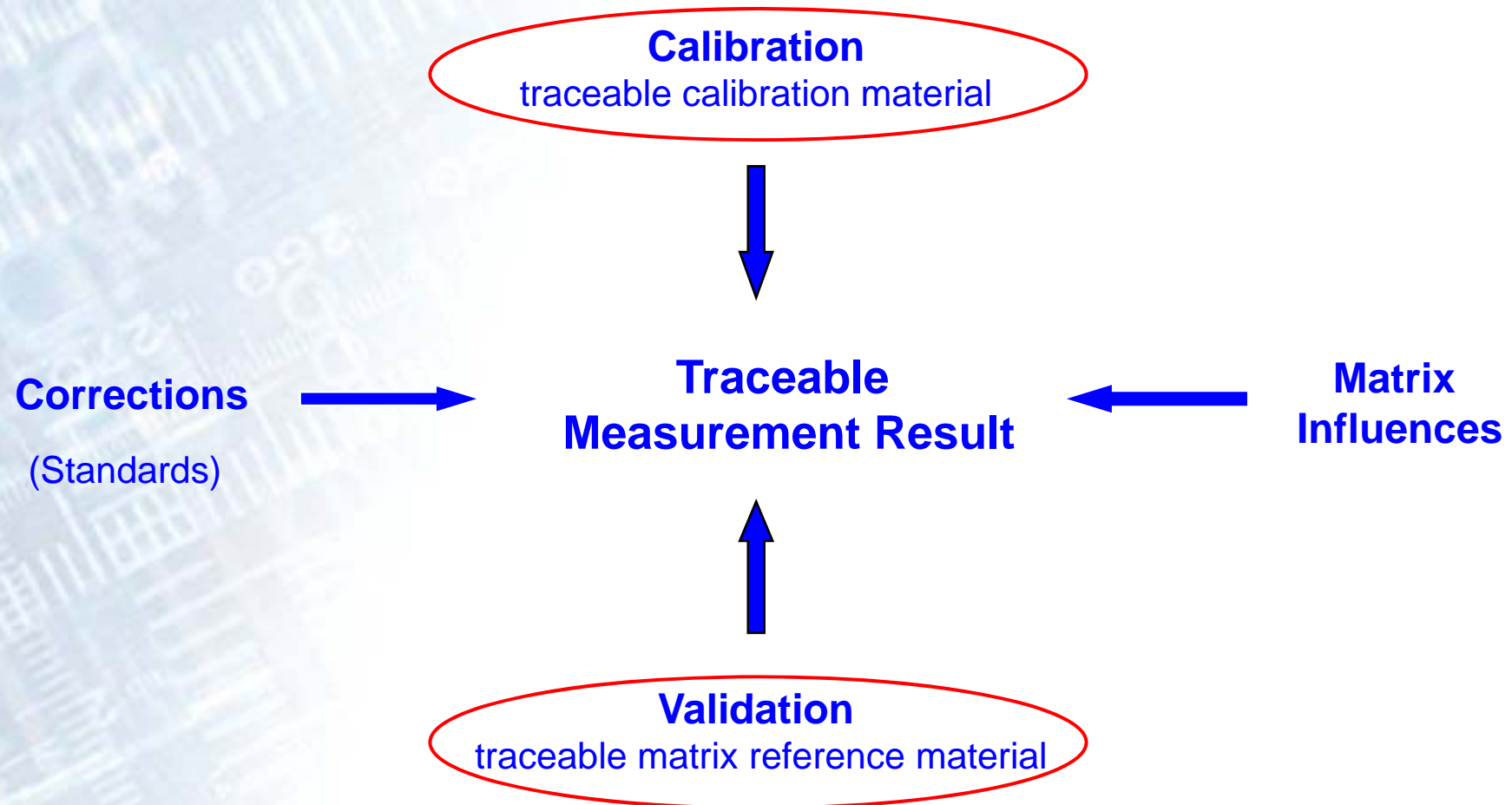
## Comparison measurements using consensus values

- Snapshot in time and
- circle of participants
- uncertain reference

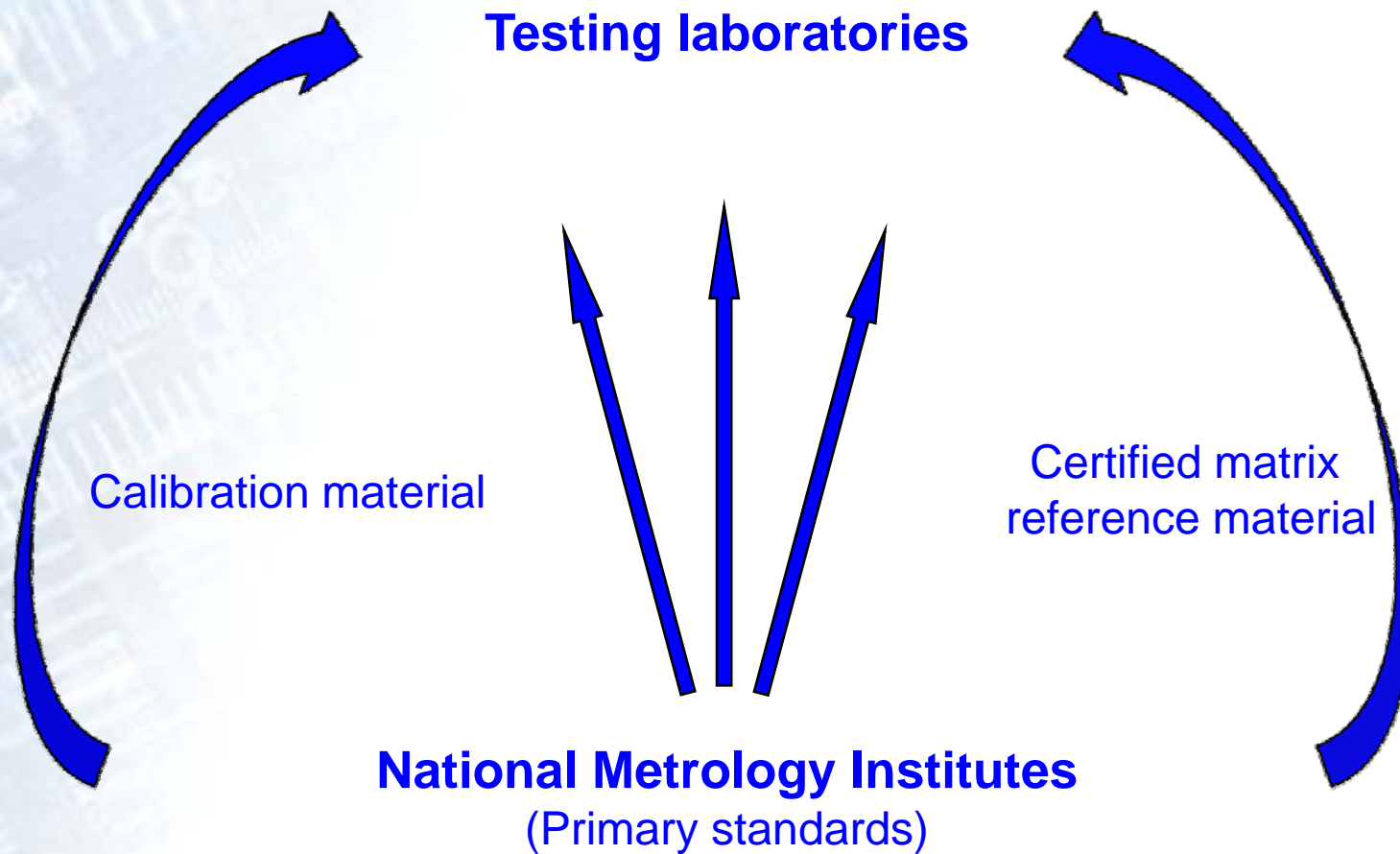
## Traceability to the SI

- Internationally accepted reference
- Sustainable
- Independent on participants

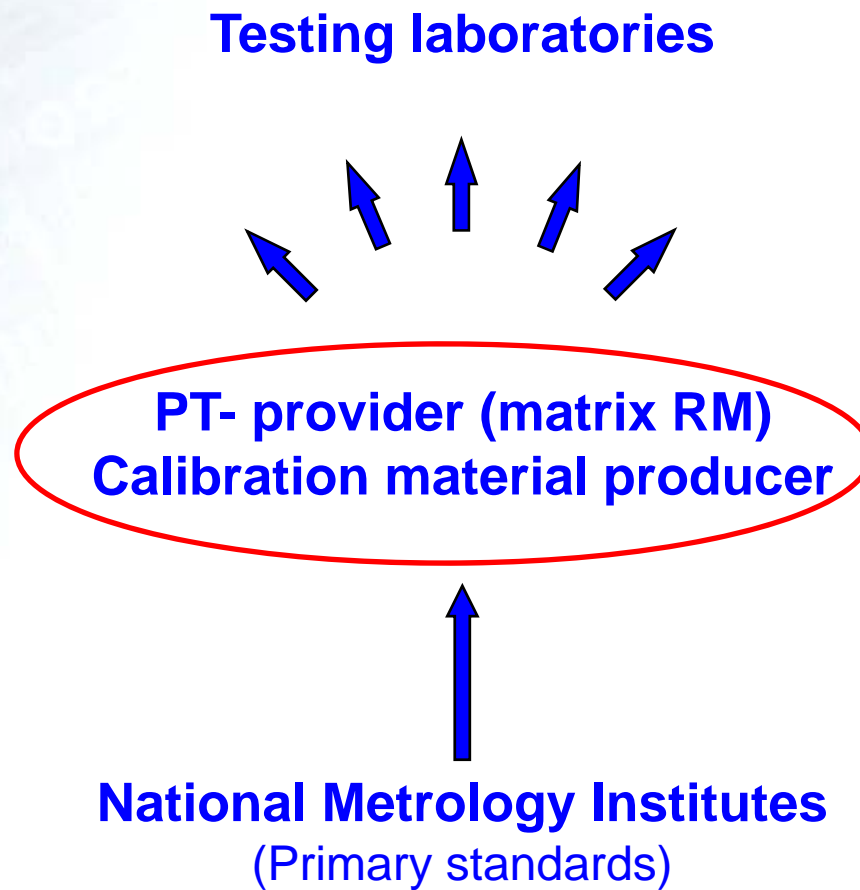
# How can traceability be achieved?



# Dissemination as usual



**Multiplication effect**  
**rational**  
**EU-wide**  
**Sustainable**



# Primary elemental calibration materials



$$n = \frac{m \cdot w_{\text{pur}}}{M}$$



BAM-A-primary-Cu-1  
LOT B27F17

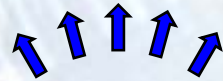
	matrix in %	impurity in mg/kg	sum 'aboveum/2 'below' in mg/kg	in mg/kg	not relevant (estimate)	under investigation
mass fraction	99,9968	32,33	22,38	9,95		
abs. uncertainty	0,0005	5,27	3,84	3,61		

H < 2,1																He < 0,001	
Li < 0,31	Be < 1,1											B < 3,2	C 0,04	N 0,2	O 1	F < 2	Ne < 0,001
Na 0,002	Mg < 0,05											Al < 0,07	Si < 0,002	P < 2	S 5,4	Cl < 0,6	Ar < 0,001
K < 0,002	Ca 0,1	Sc < 0,06	Ti < 0,32	V < 0,04	Cr 0,07	Mn 0,01	Fe < 5	Co < 0,11	Ni 1,64	Cu matrix	Zn 0,057	Ga < 0,11	Ge < 0,12	As 0,5	Se 0,22	Br < 0,014	Kr < 0,001
Rb < 0,05	Sr < 0,014	Y < 0,03	Zr < 0,015	Nb < 0,02	Mo < 0,06	Tc < 0,001	Ru < 0,03	Rh < 1,6	Pd < 0,014	Ag 11,3	Cd < 0,015	In < 0,05	Sn 0,14	Sb 1	Te < 0,22	I < 0,09	Xe < 0,001
Cs < 0,0057	Ba < 0,017	La < 0,002	Hf < 0,003	Ta < 0,003	W < 0,12	Re < 0,009	Os < 0,004	Ir < 0,007	Pt < 0,007	Au < 0,008	Hg < 0,03	Tl < 0,005	Pb 0,47	Bi 0,23	Po < 0,001	At < 0,001	Rn < 0,001
Fr < 0,001	Ra < 0,001	Ac < 0,001															
			Ce < 0,0057	Pr < 0,002	Nd < 0,21	Pm < 0,001	Sm < 0,007	Eu < 0,003	Gd < 0,001	Tb < 0,001	Dy < 0,001	Ho < 0,001	Er < 0,001	Tm < 0,001	Yb < 0,001	Lu < 0,002	
			Th < 0,02	Pa < 0,001	U < 0,001												



# Dissemination of elemental calibration materials

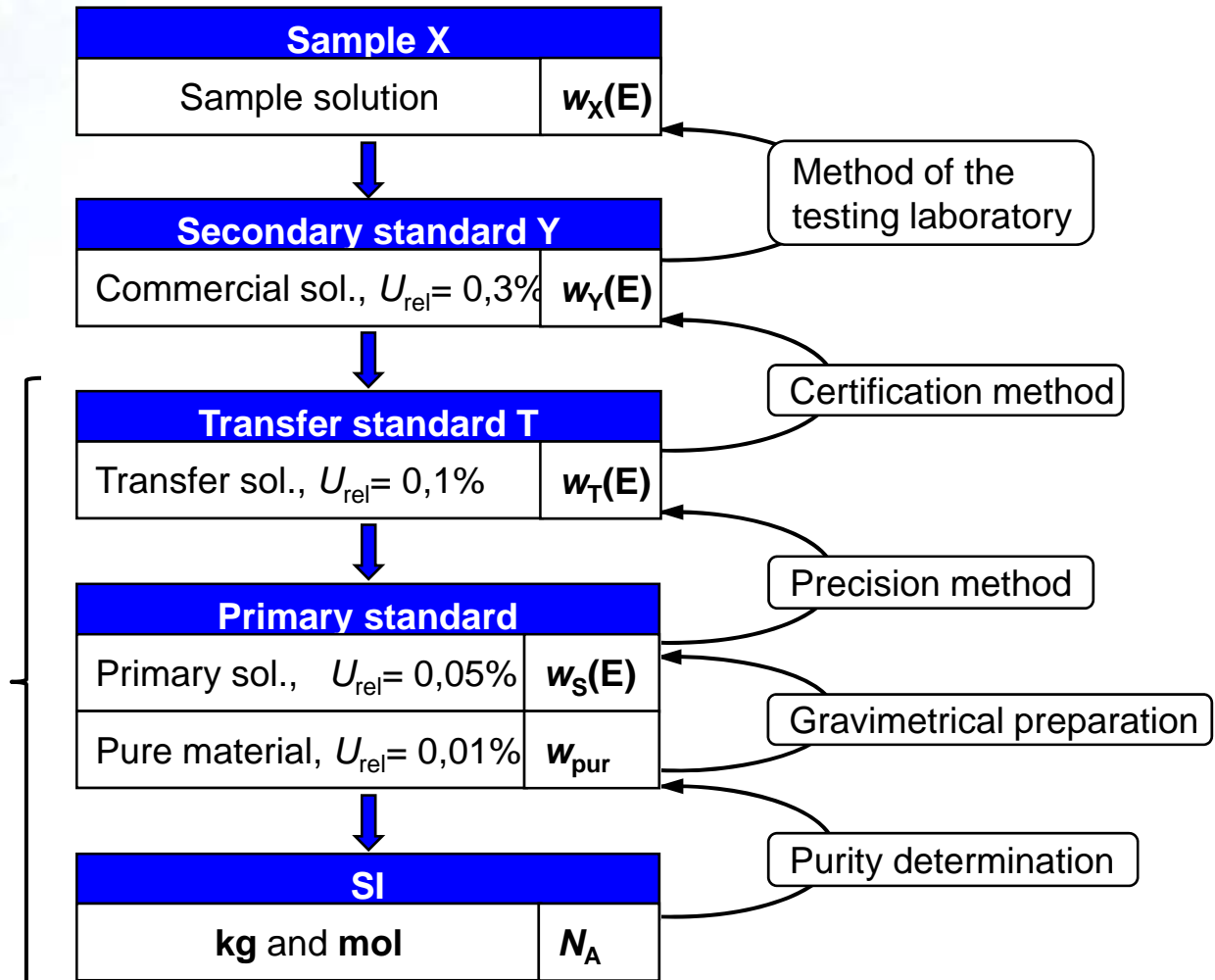
Testing laboratories



Calibration material producer

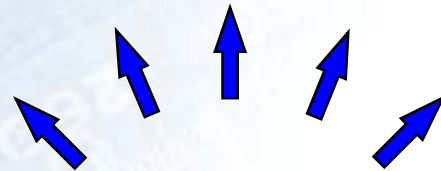


Primary basis (NMI)

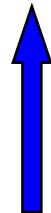


# Dissemination of matrix reference materials

Testing Laboratories (TL)



**PT-Providers (PTP)**  
experienced in chemical analysis



National Metrology Institutes (NMI)

**PT-providers need to be enabled to assign traceable reference values to ring trail samples**



Comparison measurement  
(regular)

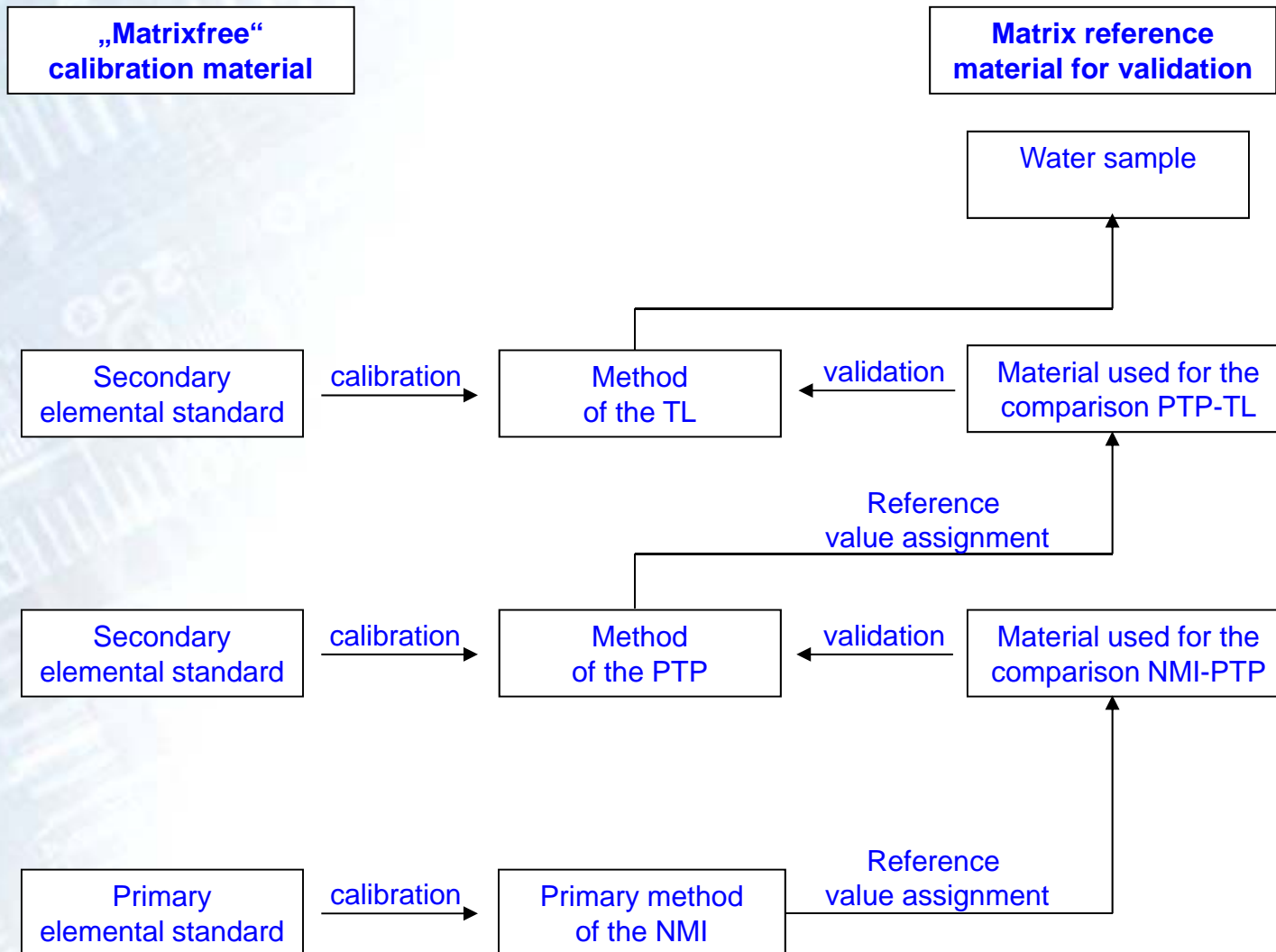


Advice in metrological  
uncertainties calculation



Comparison measurement

# Dissemination scheme (Euramet project 924)



NMI: national metrology institute  
PTP: calibration laboratory  
TL: testing laboratory

# European dissemination network (Euramet 924)



Demonstration of the applicability of this concept in the Euramet project 924 for priority substances Ni, Cd, Pb and Hg of the WFD as an example

## Participants

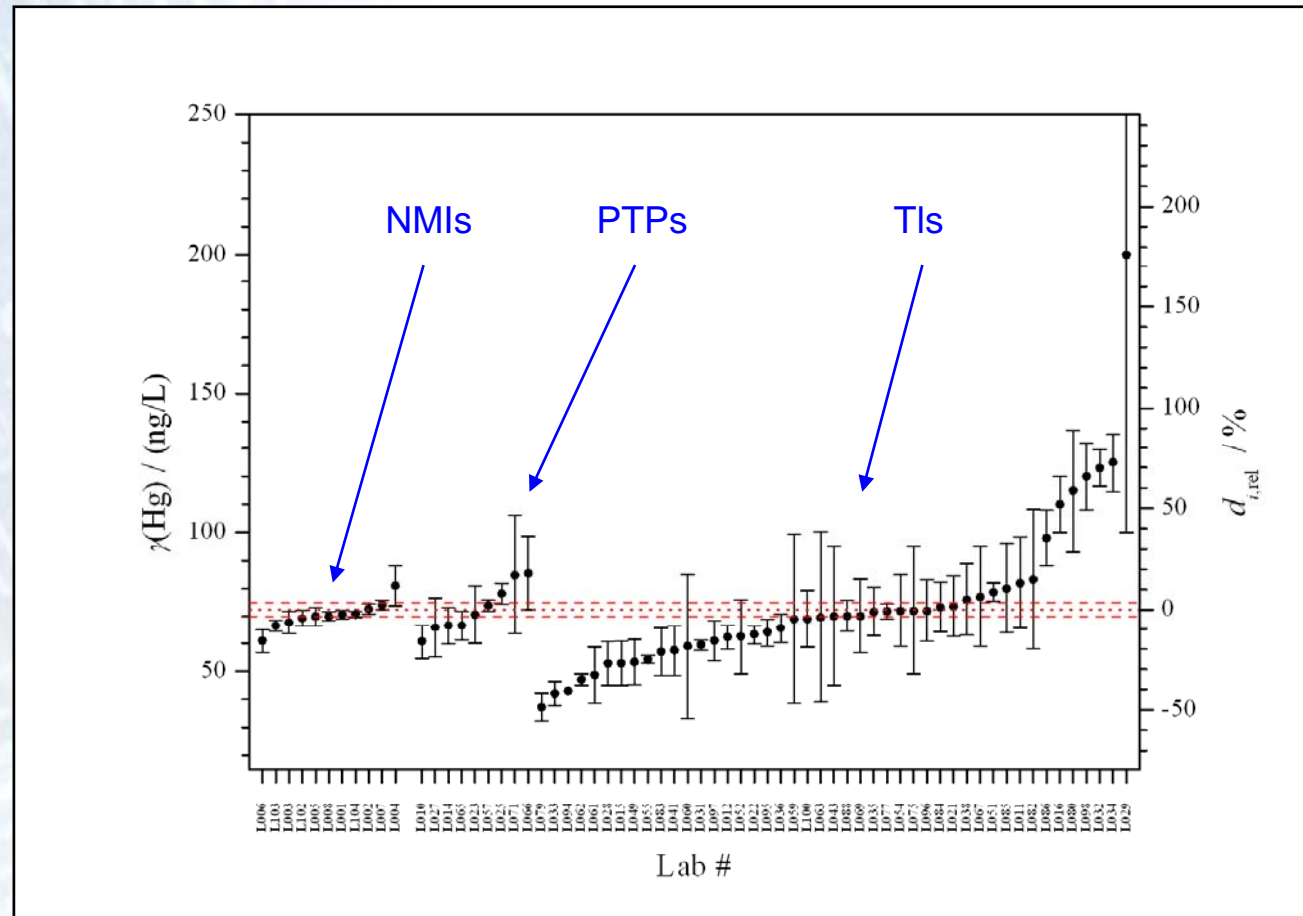
114 Laboratories from  
17 European countries

Among them:

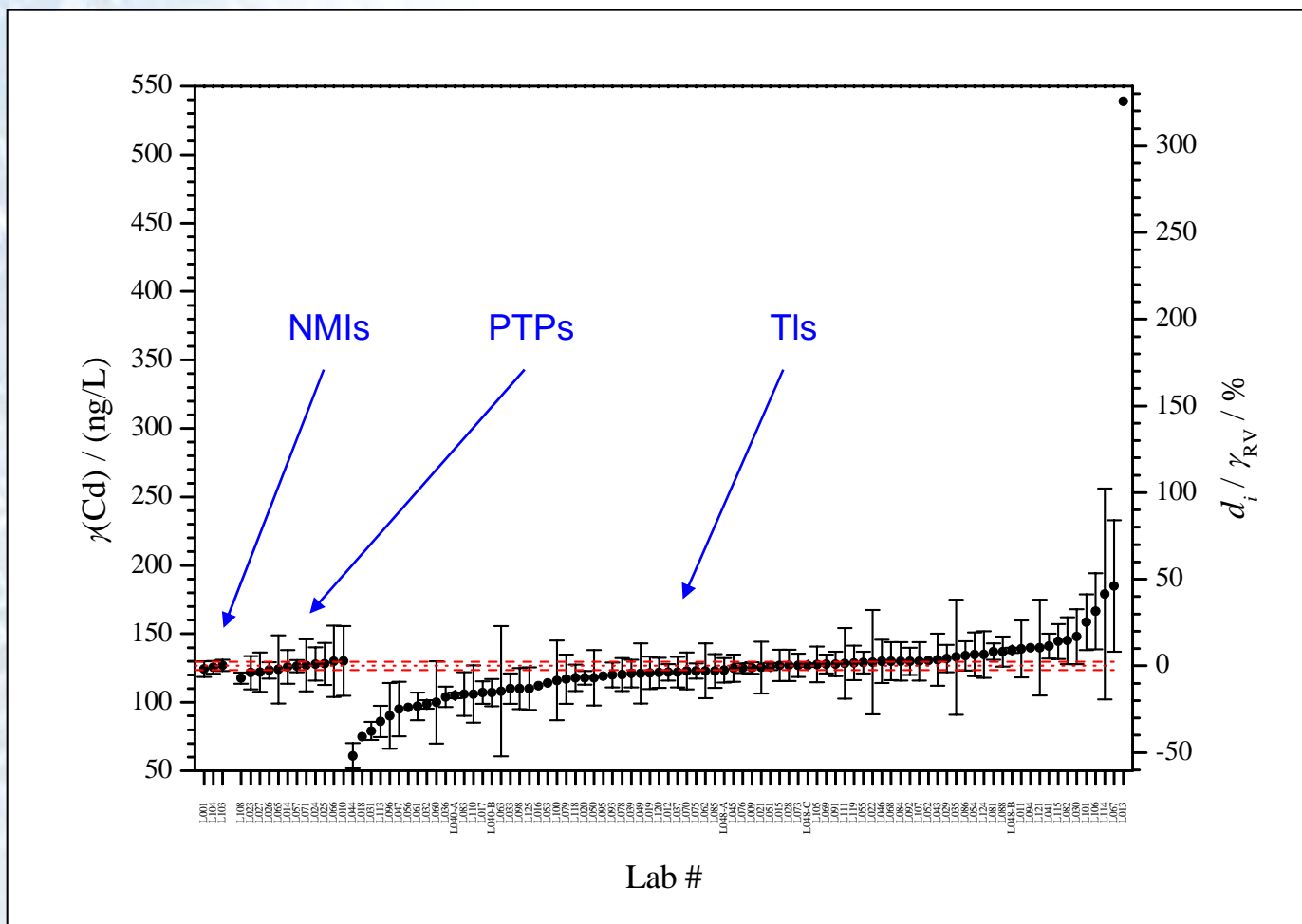
- 4 NMIs
- 22 PT-providers (expert lab.)

Schiel, D.; et. al, Accred Qual Assur (2011)16:489-498

# Comparison measurement results of Hg



# Comparison measurement results of Cd



- Metrological network can be set up
  - using an hierarchial dissemination structure
  - providing the basis for comparability
  - aiming to improve the quality of measurement results and
  - helping to implement directives and standards
- Expert laboratories among the PTPs could take over a central role in metrological dissemination
- They demonstrated for environmental analysis their good measurement capability which is appropriate for collaborating in metrological dissemination
- Enabling of expert laboratories does not need large efforts
- PTB is ready for a close cooperation with the expert laboratories

NMIs are going to develop primary methods and materials in several projects for :

- organic priority substances of the WFD e.g. TBT, PBDE, PAHs
- priority substances of laboratory medicine e.g. proteins and
- elemental calibration materials



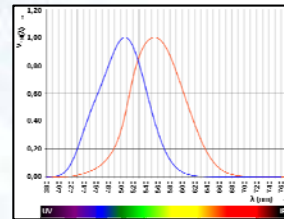
**Thank you  
For your attention**

- European metrological network for environmental analysis providing the basis for comparability using an hierarchial dissemination structure has successfully been tested within Euramet project 924
- Expert laboratories of PTPs demonstrated their good measurement capability appropriate for collaborating in metrological dissemination
- Such a dissemination structure has already been established for laboratory medicine in Germany for more than 15 years
- Outlook: NMIs are going to develop primary methods and materials for
  - organic priority substances of the WFD e.g. TBT, PBDE, PAHs,
  - priority substances of laboratory medicine e.g. proteins and
  - elemental calibration materials

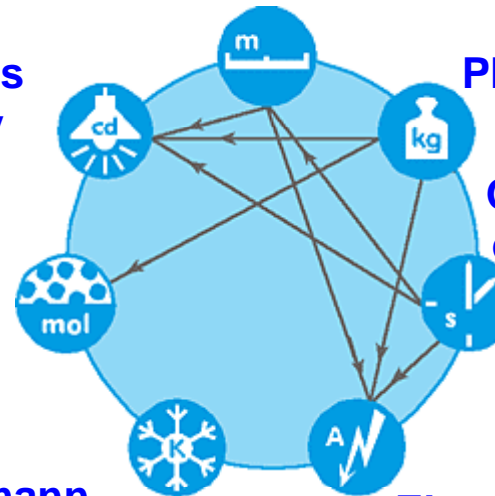
- Realization and dissemination of the SI units
- Provision of the basis for reliable measurement results



$c = \text{constant}$

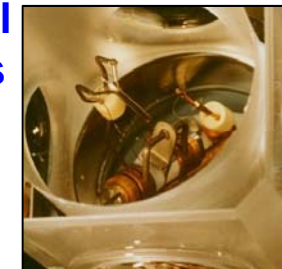


Luminous Efficacy

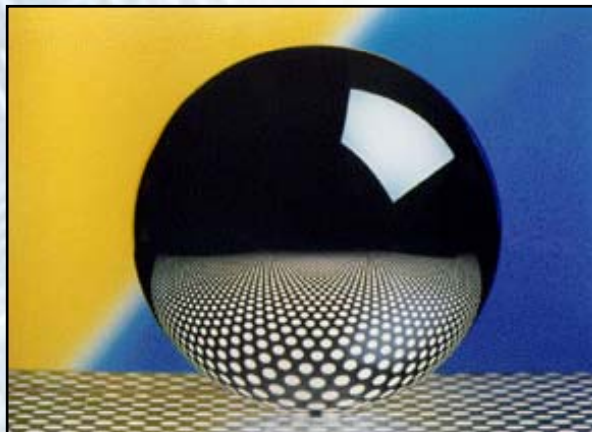


Planck-constant

Optical clocks



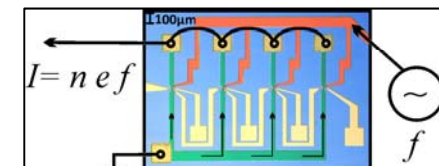
Avogadro-Constant



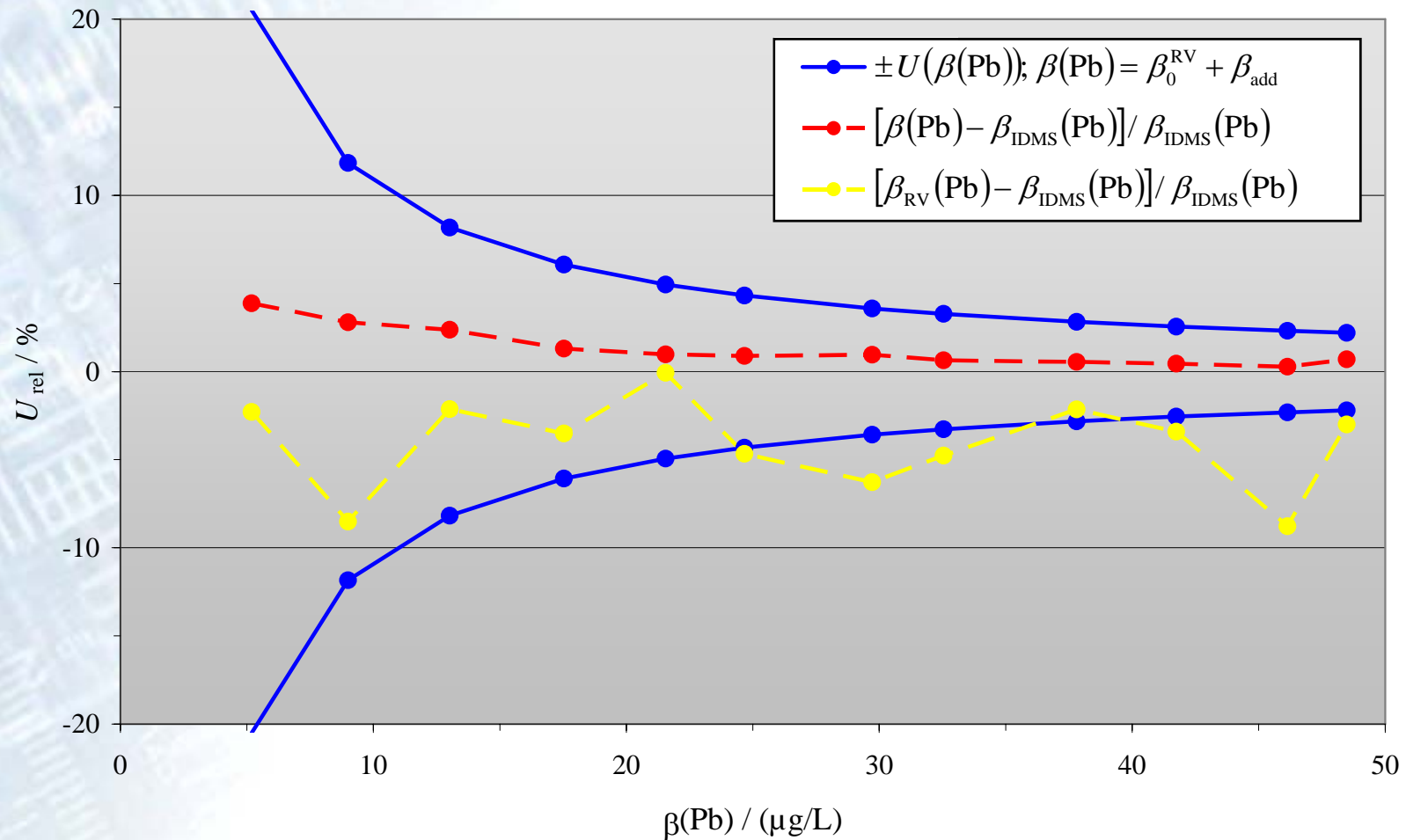
Boltzmann-Constant



Elementary Charge



# Traceable reference values for a drinking water ring trial from gravimetry and participants' results

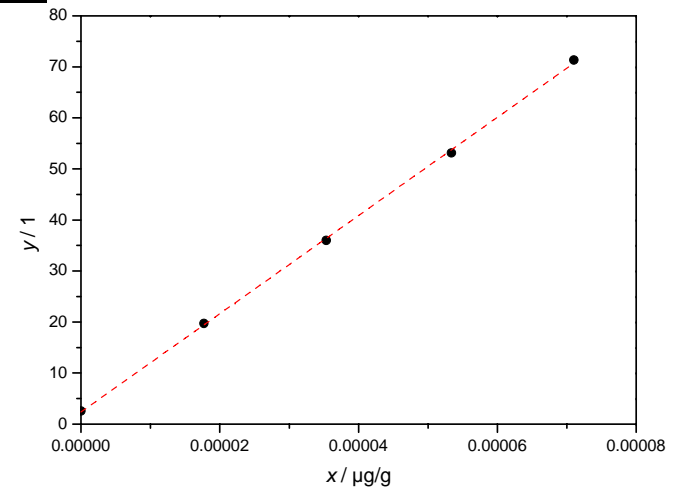


O Rienitz, D Schiel, M. Koch, U Borchers, Accred. Qual. Assur. **12** (2007), 615-622

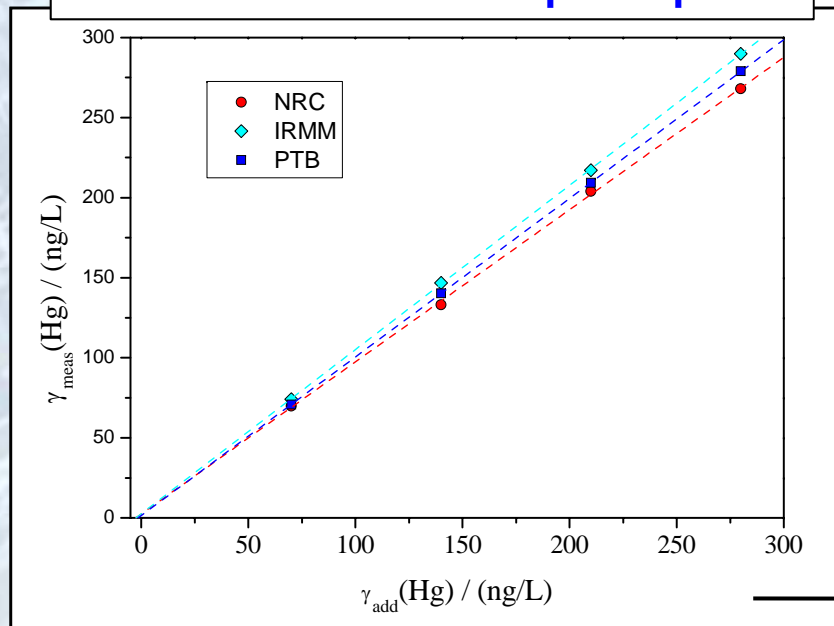
# Hg content of the fresh water used for gravimetical preparation of samples

Directly measured by standard addition IDMS

$$\gamma_{0, \text{IDMS}} = (2.6 \pm 0.5) \text{ ng/L}$$



From the results of the participants



$$\gamma_{\text{RV}} = \gamma_{\text{add}} + \gamma_0$$

$$\gamma_{\text{RV}} = (72.4 \pm 2.5) \text{ ng/L } (k=2)$$

$$\gamma_{0,p} = (2.1 \pm 1.9) \text{ ng/L}$$

# Further applications

- Hierarchical dissemination structure has been established in laboratory medicine in Germany for more than 15 year
- Regular ring trials of JCTLM are used for linking reference laboratories with PTB

Database of higher-order reference materials, measurement methods/procedures and services

BIPM Bureau International des Poids et Mesures

JCTLM Database Laboratory medicine and *in vitro* diagnostics

You are here: JCTLM-DB > Reference measurement methods/procedures > List

**Result of the search: list of reference measurement methods/procedures**

Your search criteria: Reference measurement methods/procedures; Analyte: calcium; Analyte category: Electrolytes; Matrix category: Blood serum

Save as PDF file | Modify your selection

**Results of the search**

**Ion chromatography method for calcium in blood serum**

University of Ghent ion chromatography reference method for serum calcium

Applicable matrice(s)	lyophilized, fresh, or frozen human serum
Full description of technique(s)	Ion chromatography
Quantity	Amount-of-substance concentration
Applicable range	1.25 mmol/l to 3 mmol/l
Expected uncertainty	1 % to 2 %
Reference(s)	Anal. Chem., 1994, 66, 2404-2408 Clin. Biochem., 1996, 29, 501-508 J. Chromatogr. A, 1997, 789, 557-568 Eur. J. Clin. Chem. Clin. Biochem., 1997, 35, 297-300 Scand. J. Clin. Lab. Invest., 1998, 58, 229-240
Comment(s)	The expanded uncertainty is relative
JCTLM DB identification number	NRMeth 16

**Call For Nominations**

- WG1 Cycle 8 (2011) CALL FOR NOMINATIONS: Reference materials & measurement procedures or methods
- WG2 Cycle 6 (2011) CALL FOR NOMINATIONS: Reference measurement services

IFCC International Federation of Clinical Chemistry and Laboratory Medicine

RELA - Homepage External quality control for Reference Laboratories

RfB Referenzinstitut für Bioanalytik

REL A Home

Welcome

login | Registration/Account

REL A in progress: order REL A 2011 | enter REL A 2011 results

former REL A results | Choose year...

REL A 2010

Calcium | show result plot

select lab analytes | full address

with limits of equivalence

For highlighting a specific result please click on the corresponding result line.

Labcode	A	e.u. A	B	e.u. B	Method
3	2.093	0.05	3.062	0.05	FAAS
8	2.082	0.025	3.006	0.014	ion chromatography
27	2.089	0.021	3.02	0.03	FAAS
63	2.071	0.025	2.99	0.036	FAAS
77	2.15	0.021	2.942	0.64	FAAS

Calcium

grey lines indicate a one-percent grid | e.u. - expanded uncertainty