TC for Metrology in Chemistry

Highlights and challenges

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Metrology in Chemistry

Outline

- Meetings 2015
- TC-MC projects:
  - EURAMET project n. 1371/EURAMET.QM-K111“Key Comparison 1000 µmol/mol propane in nitrogen”
- EMRP projects:
  - EMRP HLT05 “Metallomics – Metrology for metalloproteins”

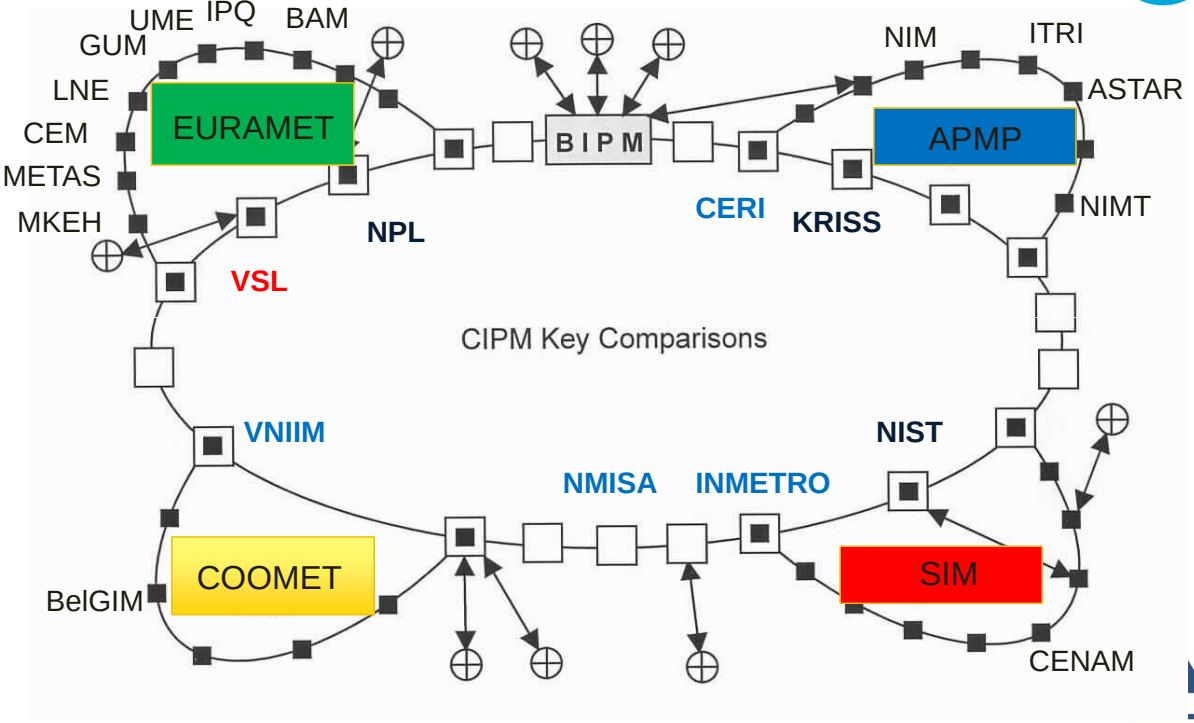
Metrology in Chemistry

Meetings 2015: Malta, 3-6 February

- From 10 to 30 participants in the various SC meetings
- 51 participants in the TC-MC from 31 European NMIs and DIs of 24 Countries + EU.
- Invited speakers from EURAMET Secretariat, BIPM, CIPM/CCQM.

Time	Tue, 03 Feb		Wed, 04 Feb				Thu, 05 Feb	Fri, 06 Feb
AM			SCGA	SCOA	SCEA	SCIA	TC-MC Plenary meeting	Plenary meeting
PM	Conve-nors meeting	ENV52	SCGA	SCOA	SCEA/SCIA joint meeting		TC-MC Plenary meeting – EMPIR worskhop	

CCQM-K111: Propane in Nitrogen Core comparison



EURAMET project 1371/ EURAMET.QM-K111

- Core Key Comparison in the gas field
- Key Comparison of 1000 µmol/mol propane in nitrogen
- VSL is coordinating both the CCQM and the EURAMET KCs
- VSL and NPL will act as a link to CCQM-K111
- The linking report will be made in cooperation among VSL, CERI and INMETRO
- Started in February 2015;
- Huge participation of European NMIs
- Participants: VSL, MKEH, GUM, METAS, CEM, UME, LNE, IPQ, BAM (9)

Timeline EURAMET.QM-K111

Event	Deadline
Agreement of protocol	August 2014 ✔
Registration of participants	August 2014 ✔
Preparation of mixtures	December 2014 ✔
Verification of mixture compositions	January 2015 ✔
Dispatch of mixtures	January 2015 ✔
Reports and cylinder arrived at VSL	April 2015
Re-verification of the mixtures	May 2015
Draft A report available	July 2015
Draft B report available	November 2015

HLT05 "Metallomics": Motivation

European and national directives require traceable results of clinical analysis

7.12.98 EN Official Journal of the European Communities L 331/1

of 27 October 1998

on *in vitro* diagnostic medical devices

"The traceability of values assigned to calibrators and/or control materials must be assured through available reference measurement procedures and/or available reference materials of a higher order."

Richtlinie der Bundesärztekammer zur Qualitätssicherung laboratoriumsmedizinischer Untersuchungen

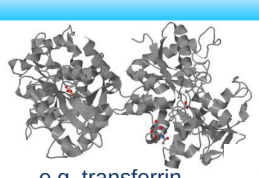
Gemäß Beschluss des Vorstandes der Bundesärztekammer vom 23. Novemb
veröffentlicht im Deutschen Ärzteblatt, Jg. 105, Heft 7, 15. Februar 2008, Seite A

SW: method specific reference value

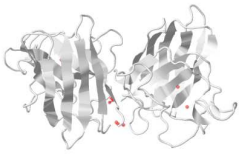
RMW: value obtained with reference method

1 lfd.	2 Analyt	3 Zulässige relative Abweichung des Einzelwertes bzw. des relativen quadratischen Mittelwertes	4 Gültigkeitsbereich der Spalten 3 und 5			5 Zulässige relative Abweichung beim Ringversuch	6 Zielwertart beim Ringversuch
			von	bis	Einheit		
23	Ferritin	13,5 %	10	600	µg/l	25,0 %	SW
27	Hämoglobin	4,0 %	2	20	g/dl	6,0 %	RMW
			1,2	12,4	mmol/l		
28	Hämoglobin A 1c (HbA1c) Haemoglobin beta chain (Blood)-N-(1-deoxyfructos-1-yl) haemoglobin beta chain	10,0 %	30	140	mmol/mol Hb	18,0 %	RMW
60	Transferrin	9,5 %	0,5	6	g/l	15,0 %	SW

Project Outline



e.g. transferrin



e.g. superoxide dismutase

WP 1 Non-covalently bound metalloproteins (LNE, LGC, PTB, UME)

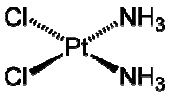
WP 2 Proteins with non-covalently bound metalloenzymes (PTB, BAM, REG2 (UNIABDN))

WP 3 Covalently bound metalloproteins, metallodrugs and mediating metabolites in cancer chemoprevention and treatment (LGC, BAM, LNE, UME, REG1 (DKFZ), REG2 (UNIAB

Glutathion peroxidase

WP 4 Development of new and complimentary methods (PTB, BAM, REG1 (DKFZ))

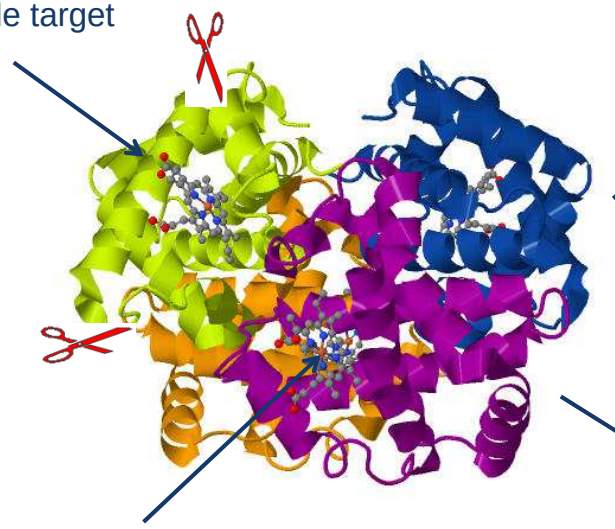
WP 5 Creating Impact (PTB, all partners)

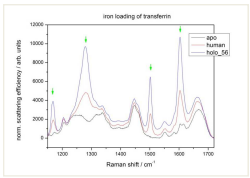


WP 6 JRP Management and Coordination (PTB, all partners)

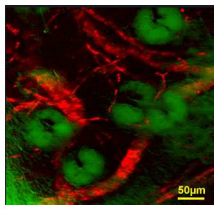
Quantification of Proteins

organic IDMS
peptide target





ID-RAMAN



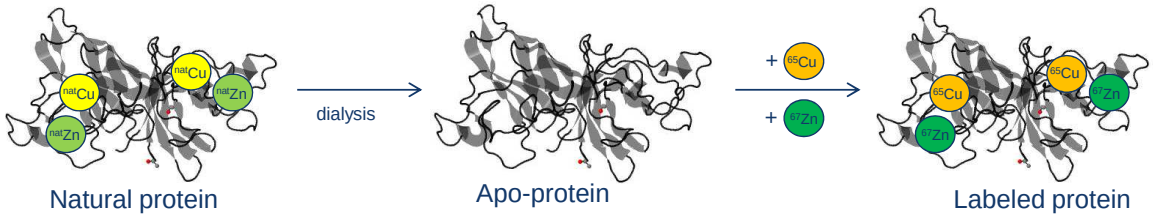
ICP-IDMS
determination of elements

optical methods
fluorescence or emission

Species specific Isotope Dilution Mass spectrometry

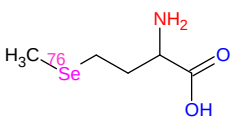
Challenge: Preparation of isotopically labeled spiked material

- Labeling via exchangeable ions



Natural protein — Apo-protein — Labeled protein

- Labeling of the protein backbone via isotopically labeled amino acids



Recombinant synthesis of proteins

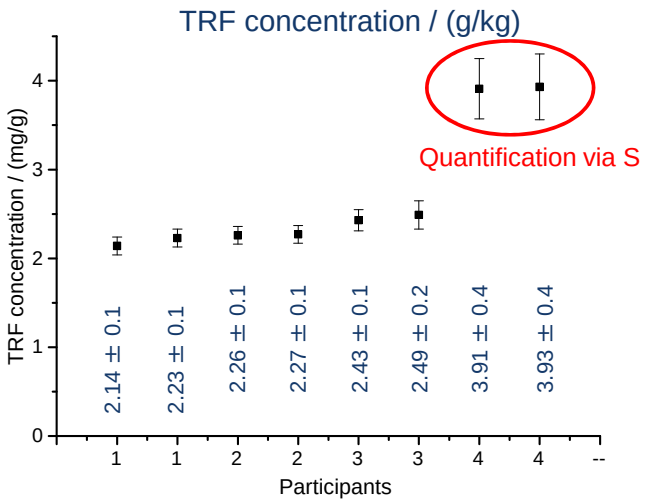
Replacing of Se-Cys with Cys and Met with ^{76}Se-Met → Labeled selenoproteins

→ ^{15}N-labeled proteins

EURAMET Intercomparison (Ref. 1351)

Determination of transferrin in human serum

Sample: BCR®-637 human Serum (untreated Serum)
Instrumentation: HPLC/ICP-MS
Monitored elements: Fe (participants 1-3), S (participant 4)
4 Participants
Shipped and stored at -80 ° C



Results:
Deviation between analysis via Fe and S
Problems with interferences in untreated serum