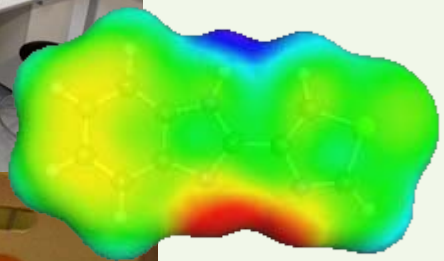
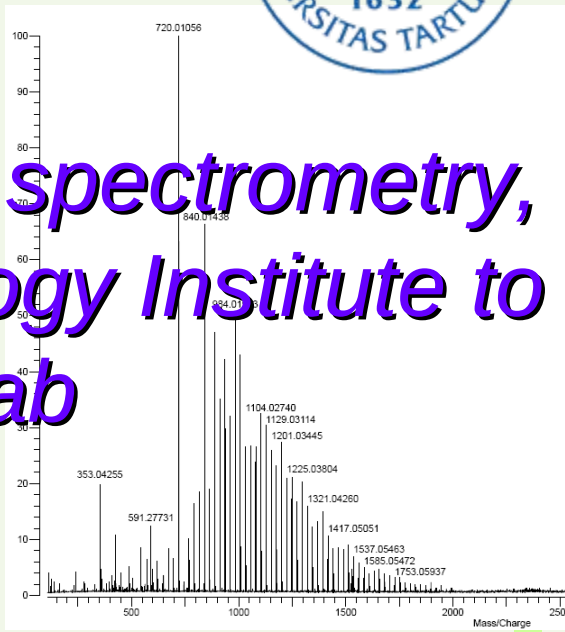
Metrology in Chemistry





from pipette to mass spectrometry, from National Metrology Institute to student lab

Ivo Leito

University of Tartu

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Metrology = Science of measurement

- Metrology is relevant for any measurement: from the simplest to the most sophisticated

Chemical measurements ≈ Chemical analysis

Are Measurements Important?

Measurements:
80 billions of EUR or 1% of the GDP in Europe

The Assessment of the Economic Role of Measurements and Testing in Modern Society.
Survey directed by Geoffrey Williams, Pembroke College, Oxford, **2002**

Legislation?

Ca 25% of the EU legislation specifies chemical measurements (chemical analysis)

Estimated by the EC JRC IRMM

Some EU legislation …

Water Framework Directive (2000/60/EC)
Priority Substances Decision (2455/2001/EC)
Bathing Water Directive (76/160/EEC)
Birds Directive (79/409/EEC ; 97/49/EC)
Drinking Water Directive (80/778/EEC ; 98/83/EEC)
Environment Impact Assessment Directive (85/337/EEC)
Habitats Directive (92/43/EEC)
Integrated Pollution Prevention Control Directive (96/61/EC)
Major Accidents Directive (96/82/EC)
Nitrates Directive (91/676/EEC)
Plant Protection Products Directive (91/1414/EEC ; 98/47/EEC)
Sewage Sludge Directive (86/278/EEC)

Urban Wastewater Treatment Directive (91/271/EEC)
Cadmium Discharge Directive (83/513/EEC)
Hexachlorocyclohexane Discharge Directive (84/491/EEC)
Mercury Directive (84/156/EEC)
Mercury Discharges Directive (82/176/EEC)
Directive (79/869/EEC) concerning the methods of measurement and frequencies of sampling and analysis of surface water intended for the abstraction of drinking water
Fish Life Directive (78/659/EEC)
Groundwater Directive (80/68/EEC)
Shellfish Waters Directive (79/923/EEC)
Surface Water Abstraction Directive (75/440/EEC)
Technical guidance document in support of Commission Directive (93/67/EEC)

Radioactivity Council Decision 87/600 Emergency Information Exchange
Dir 97/24/EC Vehicle emission
2001/81/EC national emission ceilings for certain atmospheric pollutants
2002/3/EC ozone in ambient air

The list continues ...

Estimated Water Framework Directive (2000/60/EC) monitoring cost

1) Direct cost: for EU estimated at **billions of €**

2) Cost of wrong decision: amplification

- incorrect trend analysis
- incorrect water protection measures
- incorrect environmental remediation measures

BSE testing in the EU

Since 2001 animals older than 30 months & fallen stock above 24 months must be tested for BSE before entering food chain

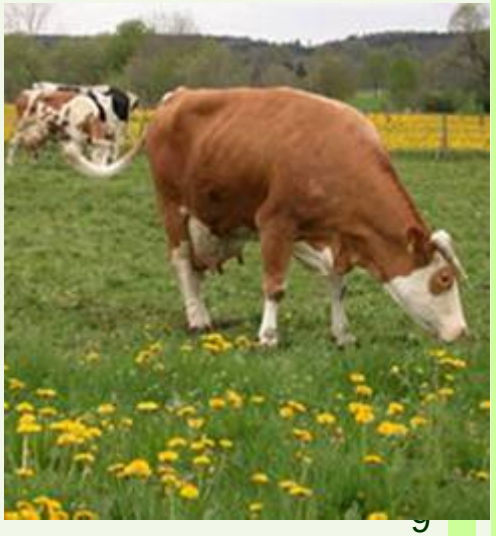
[Regulation (EC) No 999/2001]

➔ **10 - 11 million BSE tests/year in the EU**

~ € 500 million € /year

(on average ~ € 45 - 50 / test)
Estimation by DG SANCO 2005

Data from the EC JRC IRMM



So, chemical analysis ia a big business...

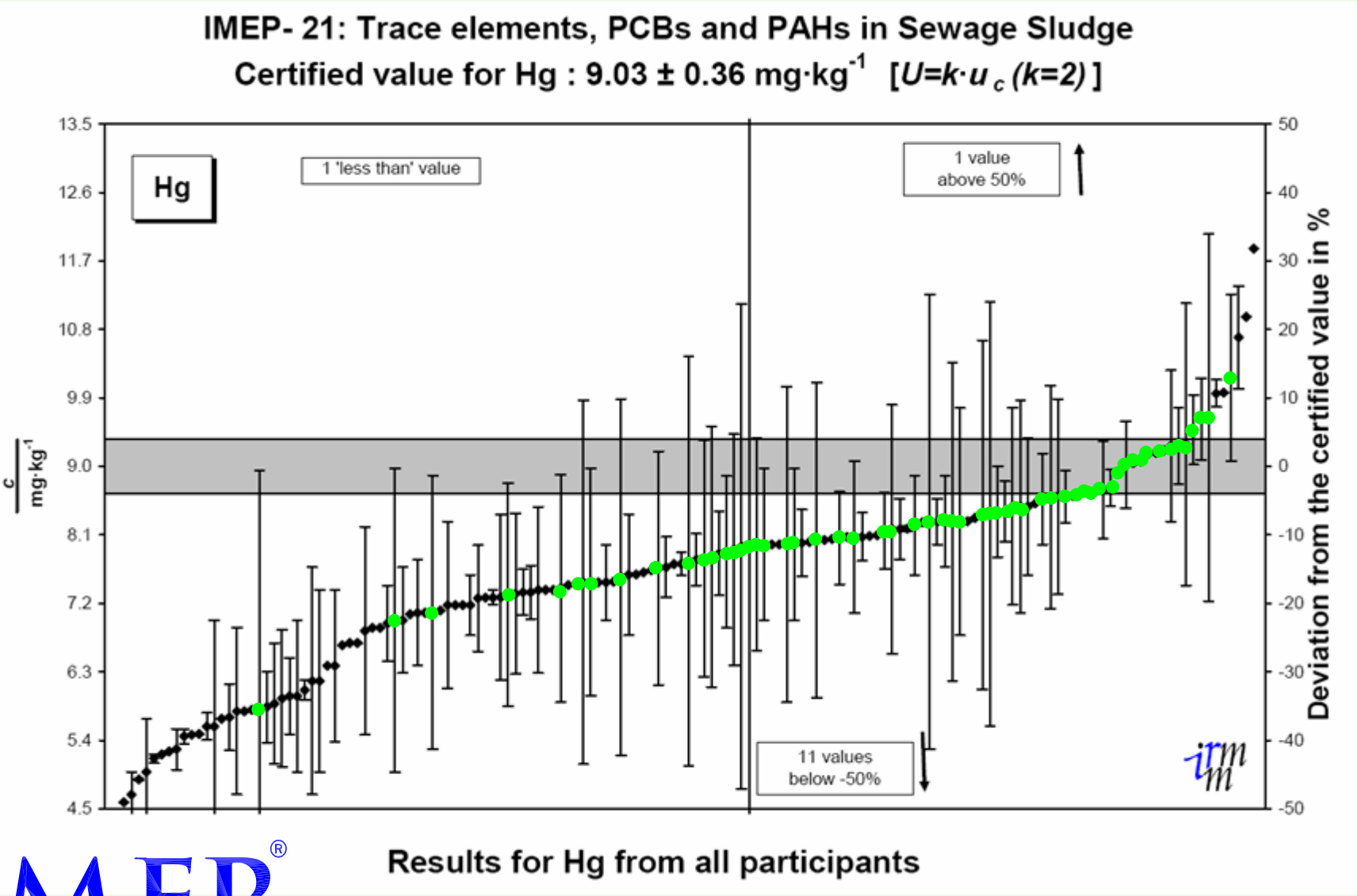
Are labs up to the task?

Between 5 and 30% of chemical analysis results are unsatisfactory!

Not quite ...

Metrology in Chemistry. Current Activities and Future Requirements in Europe. Prepared by B. King, EUR 19074 EN, Luxembourg, **1999**

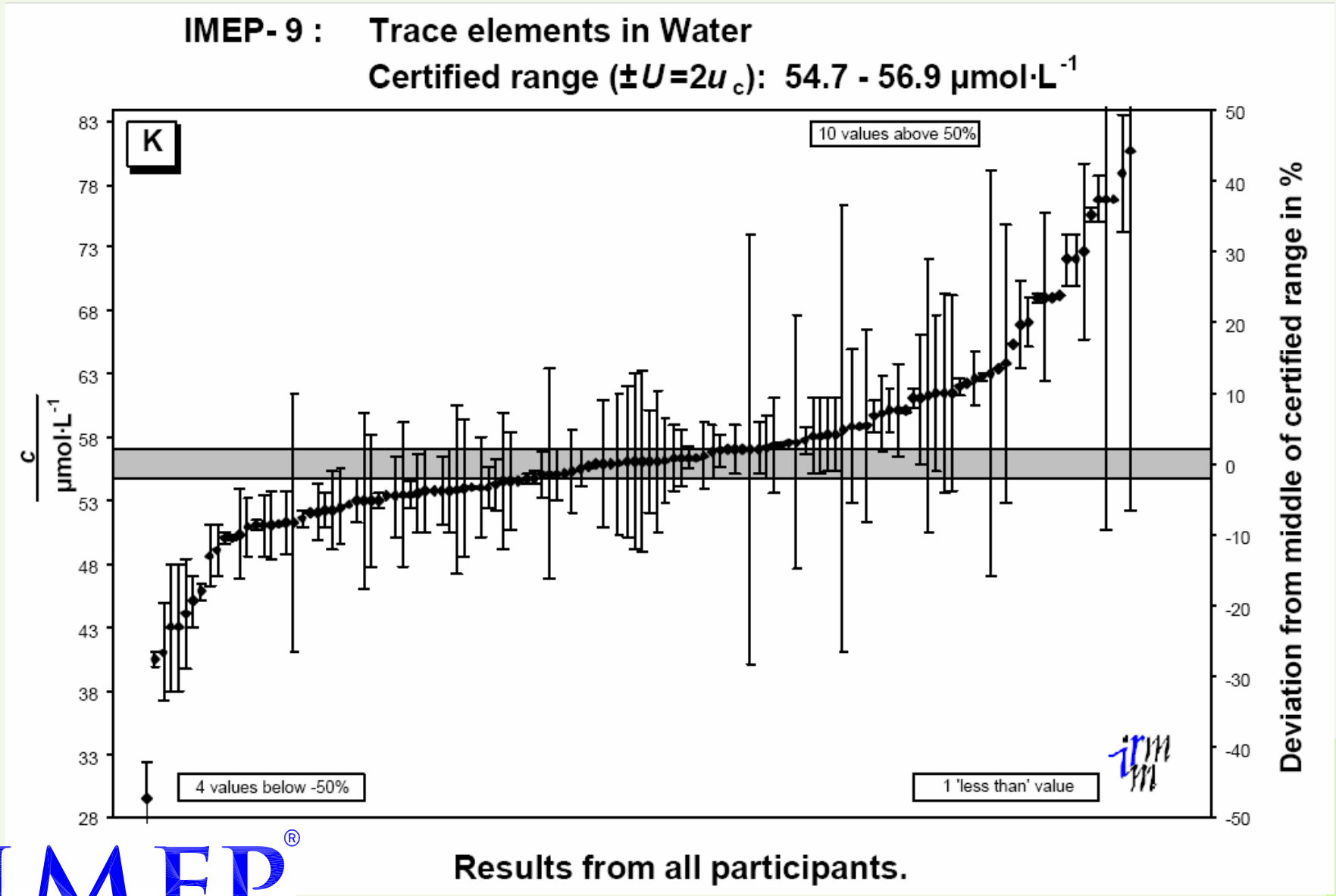
Example:



IMEP®

http://www.irmm.jrc.be/interlaboratory_comparisons/imep/

Another example:

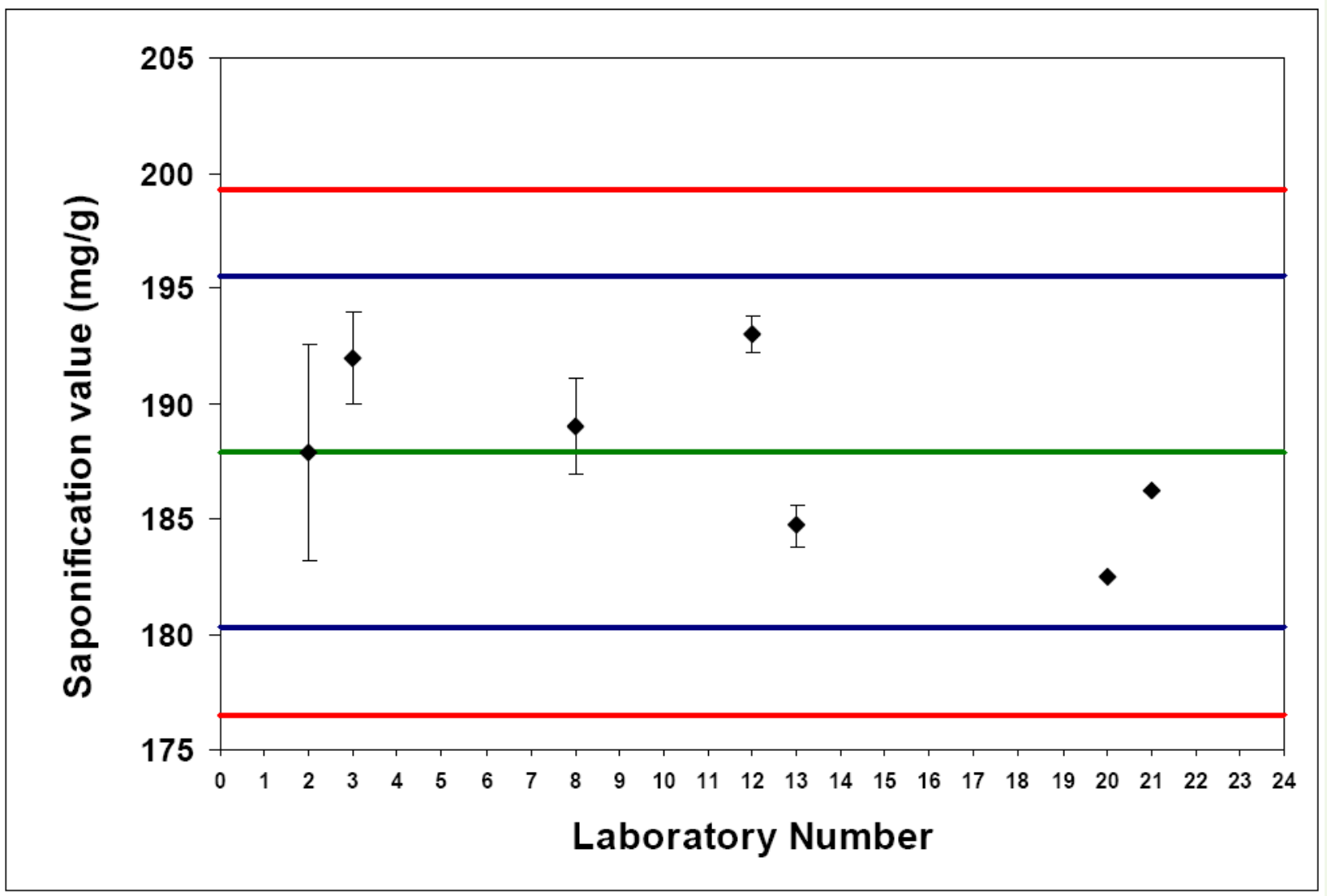


Results from all participants.

IMEP®

http://www.irmm.jrc.be/interlaboratory_comparisons/imep/

Yet another example:



EstOil-5

Two kinds of problems

- **Measurement poorly performed**
  - Measured value significally different from the reference value

- **Uncertainty poorly estimated**
  - The agreement between the measured value and the reference value is by itself not so bad, but the uncertainty of the lab's value is underestimated

How to achieve correct results?

- Be **educated**!
- Use **validated** measurement procedures
- Estimate and report measurement **uncertainty**
- Establish **traceability** of measurement results
- Participate in **inter-laboratory comparison** measurements
- Accredited **quality management system**

Not new ...

... but not included in most chemistry curricula!

Key to success: Education

There is a huge need for

educated workers and managers

in laboratories, industry, agencies, …

The Paradox

- There is a huge need for measurement-related knowledge
- Measurements are “big business”

- And yet, laboratories fail to find competent people ...
  - not enough students are educated in this field
  - important issues not included in curricula ...

Some widespread misconceptions

Analytical chemistry is just a tool, you just apply it

Everybody can measure, no special knowledge needed

You just buy the kit, no?

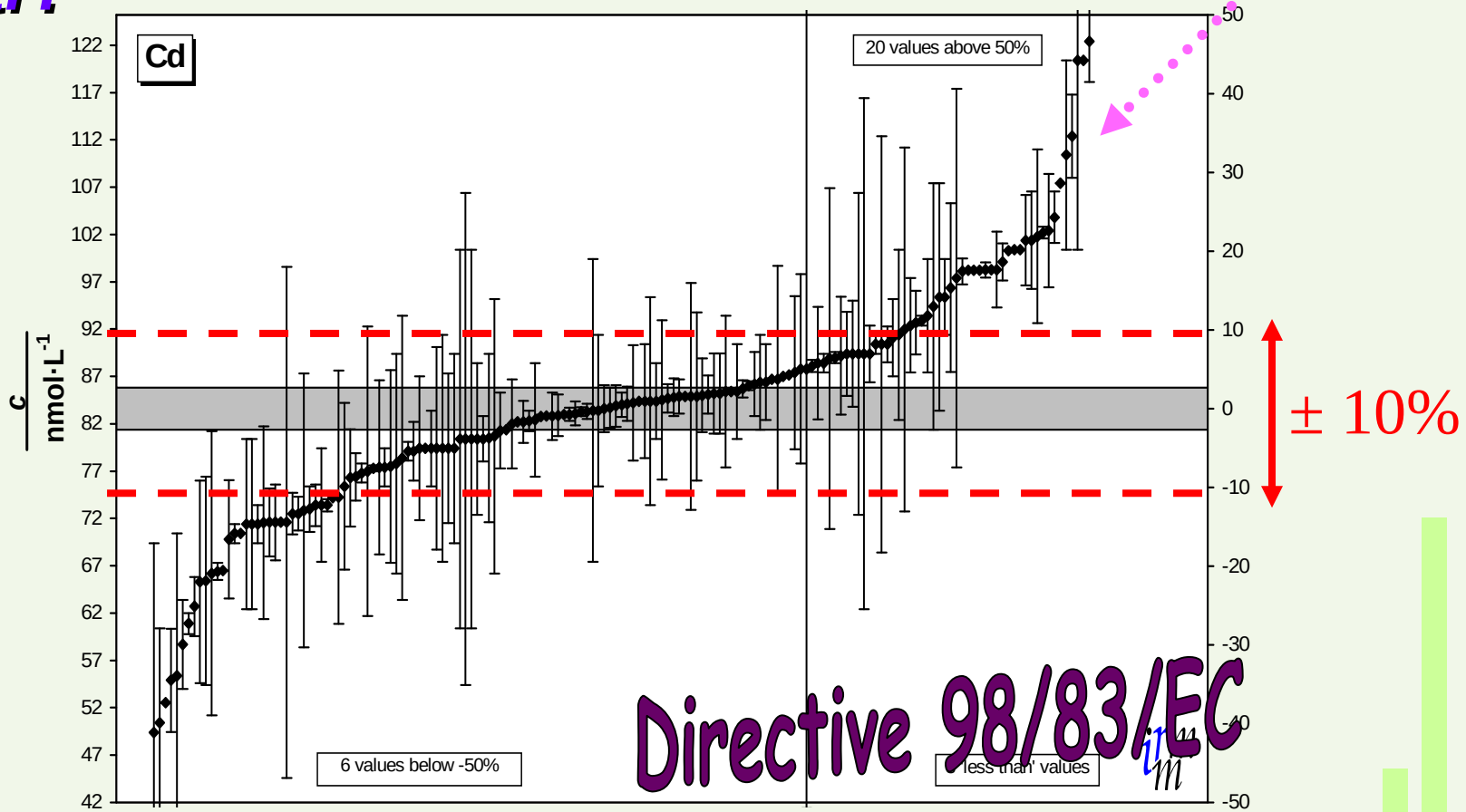
The plug and play idea !

analysis is trivial ?

Trivial?

IMEP- 9 : Trace elements in Water

Certified range ($\pm U = 2u_c$): 81.0 - 85.4 nmol·L^{-1} ??



Directive 98/83/EC

Results from all laboratories.

Water for human consumption

IMEP®

Does education help?

EstOil-3 Final Report 02.11.2007

The z-scores are calculated according to equation 1 and are presented in Table 6.

Table 6. Participant z-Scores.

Lab number[a]	z scores[b] Moisture content	FFA content	PV	P content	SAPV	B-SITO content
1		-1.0	-0.1			
2						0.1
3						0.2
4		-0.1	0.6		-1.2	
5		0.3	1.6			
6	-0.1	-0.7	-0.5	0.2	0.8	
7		-0.1	2.1	0.1	0.6	
8	-0.2					
9	-0.5					
10	0.8	1.8	-3.6	-1.5	1.7	
11	-1.9	-0.9	0.1	0.8		
12	14.2	-0.7	1.6			0.1
13	15.5	-2.2	0.1		0.2	
14		-0.8	4.9			-1.8
15		-0.6	-0.2			
16		-0.9	-1.8		0.1	0.5
17		17.6	-0.2			-6.2
18		0.7	-0.1	0.0	1.0	
19		7.4	0.0		6.4	
20		-0.1	0.1	6.8	0.1	
21		-0.5	0.0			-6.4
22	36.1	2.0	-2.0			
23			0.2			
24	0.9	5.7	-2.0	-1.7	-1.4	
25		-0.4	-0.3	1.4		
26		0.6			0.3	
27			0.4			
28	0.9	-0.3	-0.4	0.5	-1.2	
29		2.6	0.8	0.2		
30		4646.9				
31	7.7					-0.5
32	2.8	-0.3	0.0		-1.1	
33	0.8	-0.3	0.1			-0.4
34						1.7

[a] The participating laboratories are given in random order that is different from the order given in Table 3 but is identical to the order given in Table 4. [b] According to the ISO Guide 43-1: acceptable result is marked in green, doubtful result in yellow and unacceptable result in red.

Estoil 3

EstOil-4 Final Report 24.09.2008

The z-scores are calculated according to equation 1 and are presented in Table 6.

Table 6. Participant z-Scores.

Lab number[a]	Moisture content	FFA content	PV	P content	SAPV	B-SITO content	ERUC content
1		-1.3	2.2				
2						0.7	
3	0.2	0.9	-0.3	0.8	0.3	0.0	-1.0
4		-0.9	1.3	1.0	-0.4		
5		0.1	0.2				
6	-0.4	-0.6	-1.0	0.0	-0.2		
7	-3.6	-0.9			0.6	-1.8	
8	-0.8						
9	-1.3						
10	-1.8	2.0	0.5	-2.0			
11	0.3	0.8	-0.3	0.0			
12		-0.1	-0.4		0.3		-1.0
13		-0.9	0.8				
14		-0.9	-0.9	-0.4			1.8
15		-0.6	-1.0	-1.3	-0.4		
16		2.1	2.9		5.5		
17		-0.1	-0.6		0.1		0.4
18		-0.4	-0.4				
19	1.2	-0.9	7.4	-5.8	0.3		
20		1.2			-0.9		
21			-0.7				
22	-8.0	-0.4	-0.9	-0.6	-2.1		-4.6
23		-0.8					11.8
24	9.4					0.7	
25	-15.4	0.3	-0.2	-6.6	0.4		60.1
26	0.3	-0.1	1.3			0.7	-1.0
27		2.0	-1.1	-0.6			0.4
28	1.8	6.9			1.0		
29		1.6	-0.3				-8.2
30				-0.6			
31		-0.2	0.5	1.1	-0.2		
32	-0.7	-0.4	0.2	1.8	-1.3		
33		0.1	-0.4	-0.5			
34	-3.6	-0.4	-0.9	0.5	0.3	-0.4	0.4
35	-0.5	-1.5	0.5	0.8	2.4		
36			-0.7				
37	19.6	0.2	0.0		4.4		0.2

[a] The participating laboratories are given in random order that is different from the order given in Table 3 but is identical to the order given in Table 4. [b] According to the ISO Guide 43-1: acceptable result is marked in green, doubtful result in yellow and unacceptable result in red.

Estoil 4

EstOil-5 Final Report 09.11.2009

The z-scores are calculated according to equation 1 and are presented in Table 6.

Table 6. Participant z-Scores.

Lab number[a]	Moisture content	FFA content	PV	P content	SAPV	B-SITO content	ERUC content
1						-1.1	
2	0.0	-0.3	0.3	-1.4	0.0		
3					1.1	-16.6	
4	-0.2						
5	-0.6						
6	17.4	5.4	10.5				
7	-6.7	-1.2	1.6	1.1			
8		-0.6	-0.7		0.3		-1.2
9							1.0
10		1.0	0.8				
11		-0.6	0.5				
12	9.3	2.4	-1.1	35.7	1.3		
13		-0.1			-0.8		
14			-0.7				
15	0.9						1.0
16							0.1
17		-0.9	0.2			0.0	
18	1.3	0.2	1.3			-0.3	-1.2
19			0.1	0.9			-0.6
20	-1.5	0.5	-0.5	-1.0	-1.4		
21	-38.2	-0.5	-1.8	-0.1	-0.4	1.3	1.0
22				0.4			

[a] The participating laboratories are given in random order that is different from the order given in Table 3 but is identical to the order given in Table 4. [b] According to the ISO Guide 43-1: acceptable result is marked in green, doubtful result in yellow and unacceptable result in red.

Estoil 5

EstOil-6 Final Report 08.11.2010

The z-scores are calculated according to equation 1 and are presented in Table 6.

Table 6. Participant z-Scores.

Lab number[a]	Moisture content	FFA content	PV	P content	SAPV	B-SITO content	ERUC Content
1	1.7	1.5	-0.9	-0.1	1.3		
2		0.1			-2.3		
3	5.1	0.9	4.0				
4		4.4	1.4				
5		-0.9	1.5	0.6			
6		-0.6	-0.4	3.2			
7	0.8	0.1	-0.5	-0.4	0.2	-0.1	-0.1
8	-0.3	-0.4	-0.6	-1.4	0.5		
9	-0.4						
10							1.2
11	-0.5	-1.1	-0.2	0.04	-0.4		0.4
12	0.6	-0.1	-0.5			0.9	-0.6
13		-0.3	-0.5				
14		-0.7	-0.6		-0.4		-1.3
15	-0.9						
16			-0.6				
17			-0.2				
18					0.1	-0.9	
19			0.8	-0.2			0.4

[a] The participating laboratories are given in random order that is different from the order given in Table 3 but is identical to the order given in Table 4. [b] According to the ISO Guide 43-1: acceptable result is marked in green, doubtful result in yellow and unacceptable result in red.

Estoil 6

There is improvement!

24.03.2011

http://www.ut.ee/katsekoda/ILC/

Applied Measurement Science

International master's programme

http://www.ut.ee/ams

Programme outline

- Interdisciplinary 3+2 master's degree program
  - Physical measurements
  - Chemical analyses
  - Metrology
  - Quality systems
  - Economic and legal aspects of measurements
- 120 ECTS
- Cross-sectorial
- International: **Tuition in english**

This combination of topics is unique in Europe!

Knowledge and skills

- Measurement and analysis methods
  - Physical and chemical basis
  - Hands-on work
- Factors affecting the results
- Calculation methods
- Knowledge necessary for assessment of quality of results
- Economic and legal aspects, quality systems

Programme structure

Obligatory Module (45 ECTS)

Courses: Measuring and Instrumentation, Measurement Data Processing, Lab of Physical Measurements, Practical Chemical Analysis Methods, Lab of Chemical Analysis Methods, Fundamentals of Metrology, Metrology in Chemistry, Seminar in Measurement Science, Quality management

Elective Module (30 ECTS, courses can be chosen from the list)

Courses: Materials Characterization and Testing, Structural Analysis, Measurements in Biochemistry, Measurements and the Law, Economic Aspects of Measurements, Signal Processing, Chemometrics, Environment and Measurement, Electrochemical Measurement and Analysis Methods, Nanometrology, Quality Systems etc

Optional Subjects

(**6 ECTS**, any courses can be chosen university-wide)

Internship

(**9 ECTS**, internship placement in industry or analysis or calibration laboratories)

Master's thesis

(30 ECTS, reasearch project with a topic related to measurement science**)**

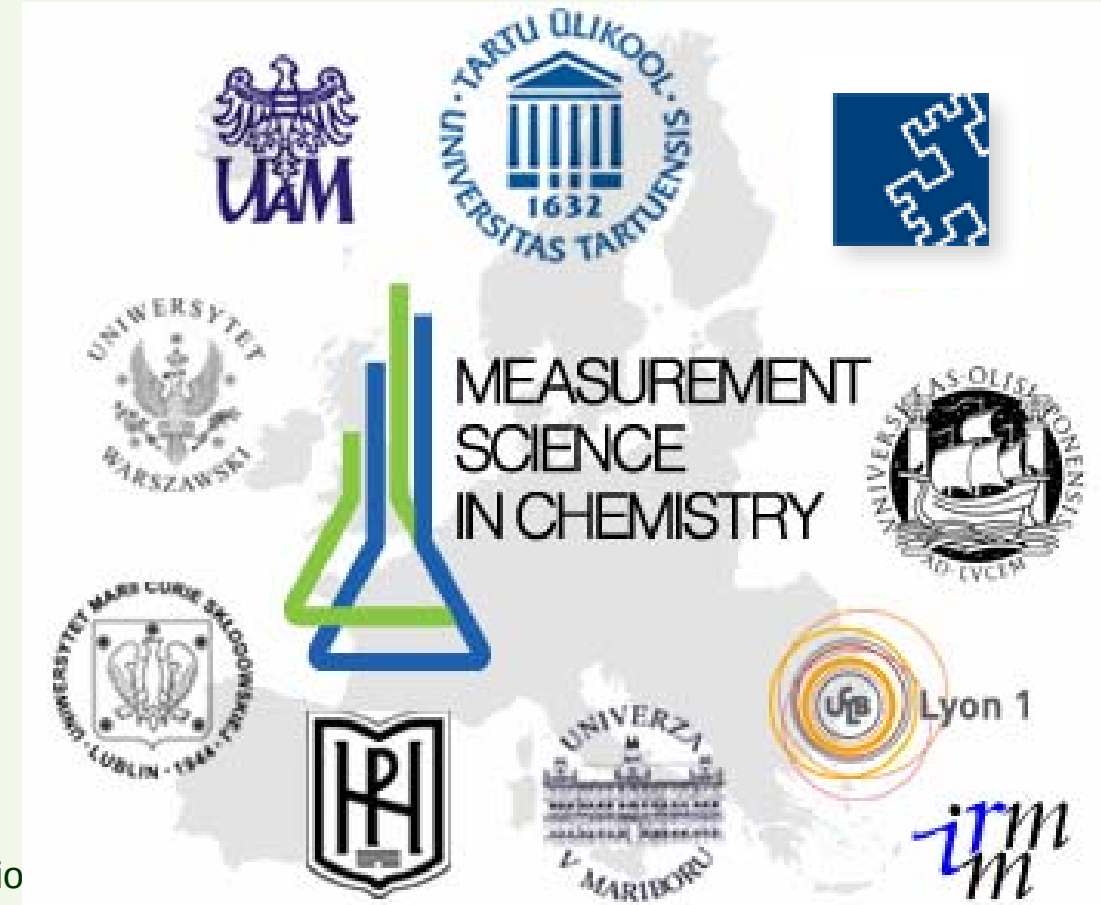
Measurement Science in Chemistry

www.msc-euromaster.eu

European Chemistry Thematic Network Association

EUROMASTER

- International
- 8 countries
  - Estonia, Slovenia, Bulgaria, France, Portugal, Poland, Romania, Finland
- 10 universities
  - Coordinator: University of Warsaw
  - Mentor-organization: EC-JRC IRMM



Measurement Science in Chemistry: Mission

- To contribute to **radical improvement of the education level of analytical chemistry** by being the best international provider of measurement science education in chemistry

"Jointly delivered" programme

- 3+2 **Master's** level programme
- Broad education in the basics of measurement science in chemistry (analytical chemistry)

- "Jointly delivered":
  - Study at home university, according to a local program
  - Added value by international activities
    - Summer school
    - Student and teacher exchange
    - Best practice in teaching

MSC consortium: short history

- 2001 Start of the IRMM TrainMiC (Training in Metrology in Chemistry) initiative
- 2005 AcadeMiC (Academics teaching Metrology in Chemistry) Summer School (Rogaška Slatina)
- 2007 AcadeMiC (Academics teaching Metrology in Chemistry) Summer School (Krakow/Wieliczka)
- 2007-… Applied measurement science international master's programme (University of Tartu)

MSC consortium: short history

- 2008 ECTNA awards to the consortium the **Chemistry Euromaster®** quality label

MSC consortium: short history
Summer schools

- Summer **2008 Celje** (Slovenia)
  - 35 participants
  - 8 countries

- Summer **2009 Blagoevgrad** (Bulgaria)
  - 43 participants
  - 9 countries

Summer schools

- Summer **2010 Lepanina** (Estonia)
  - 39 participants
  - 9 countries

- Summer **2011 Poznañ** (Poland)

Programme: generic structure

Year 1

- Fundamentals of Measurement Science (incl MS in Chemistry) (8-12 ECTS)
- Data evaluation and management (8-12 ECTS)
- Instrumental methods (8-12 ECTS)
- Sampling, sample preparation and separation methods (5-8 ECTS)
- Applications of analysis (16-24 ECTS)

Summer

- Summer School (30 ECTS)

Year 2

- Other subjects required by the home university
  (Subject to decision at university level)
- Master's thesis (30 ECTS)
  (reasearch project with a topic related to measurement science)

Programme Structure

- The structure on the previous slide is generic
  - At every university teaching follows a local program
  - Local differences exist between universities

- An important unifying link is the Summer school

Volume in ECTS

- The overall program volume is 120 ECTS
- 30 ECTS of the Summer school can either partially or fully replace some local courses at universities
  - This is to be decided at university level (the student, the local coordinator at the university and the university officials)

Enrollment

- Student first enrolls to one of the consortium universities as a master student
- Then separately applies for a place in the MSC program

Selection is based on academic excellence!

What comes in addition to local university programme?

- Summer school
- Homework during autumn semester of 2009
- Scrutiny of the master's thesis

MEASUREMENT SCIENCE IN CHEMISTRY

EUROMASTER CERTIFICATE OF COMPLETION

Annex to Diploma No

This is to certify that

ANABELA DE JESUS LOBO DA COSTA

(born in 17.01.1985) has successfully completed THE PAN-CONSORTIUM INTENSIVE COURSE (SUMMER SCHOOL 2008), which together with the master's programme in Mestrado em Química Analítica (Master of Science in Analytical Chemistry) constitute the jointly delivered master's programme – Euromaster in Measurement Science in Chemistry.

This certificate is issued by the University of Tartu on behalf of the international consortium „Measurement Science in Chemistry".

7-2/LO-581-H

1/5

This is the minimum requirement for the EDS!

Summer School

1. The Actual Summer School
   - Lectures, seminars
   - Group work
   - Student contest
   - Learning evaluation
2. Additional homework during autumn semester following the summer

- Final mark (a combination of 1 and 2) and ECTS points

Summer school content

- Validation of chemical analysis procedures
- Basic statistics, Statistical basis of calibration
- Traceability in chemical analysis
- Alternative Approaches for the Quantification of Measurement Uncertainty
- ISO 17025, Accreditation visit to real lab
- Sampling and sample preparation in food and environmental analysis
- Customer-analyst interactions
- Importance of reliable measurements to implement EU legislation

Master's Thesis

3. Scrutiny of the master's thesis
   - Extended English summary has to be presented
   - PPT presentation in English has to be prepared (to be put on the web)
   - The thesis will be scrutinized by the local coordinator at the home university (and if necessary by the MSC management committe)

Novel teaching approaches

"Learning by involvement"

- Contest of student teams
- Accreditation visits to ISO 17025 accredited laboratories


Metrology in chemistry on the move!
http://www.msc-euromaster.eu/