

# Development of new Certified Reference Materials for supporting EU Environmental Legislation



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<http://irmm.jrc.ec.europa.eu>

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

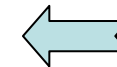
**IRMM - CONFIDENCE IN MEASUREMENTS®**



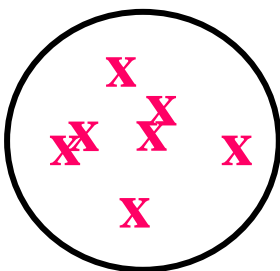
**Policy  
Implementation**



**Compliance with  
Legislation ?**

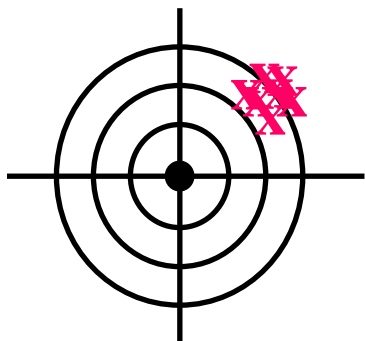
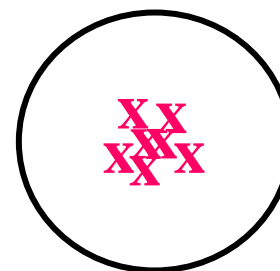


**Policy  
Monitoring**

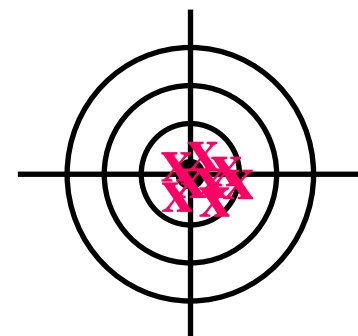


**Comparability**

- ◆ in time
- ◆ between labs

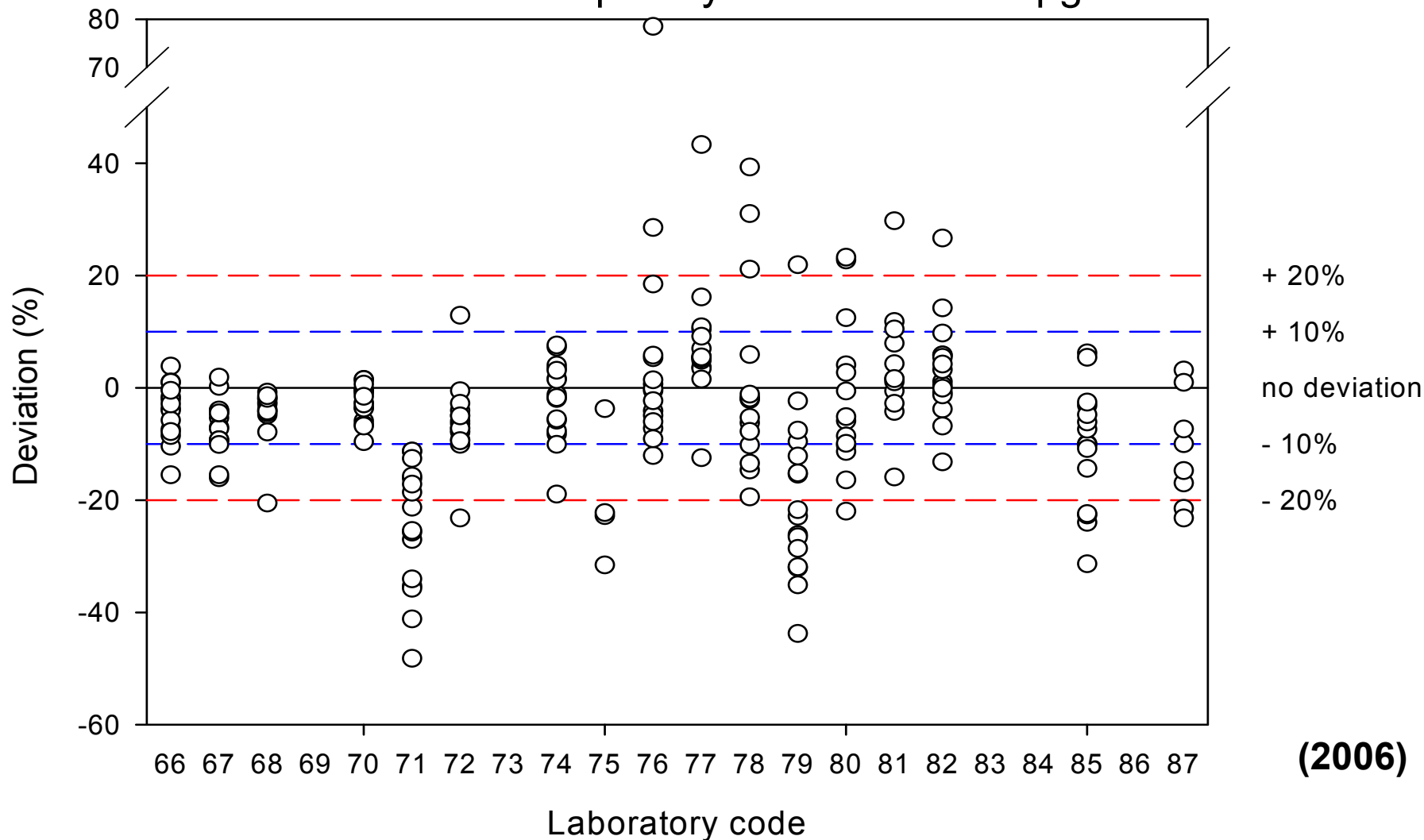


**Reliability**  
(‘accuracy’)



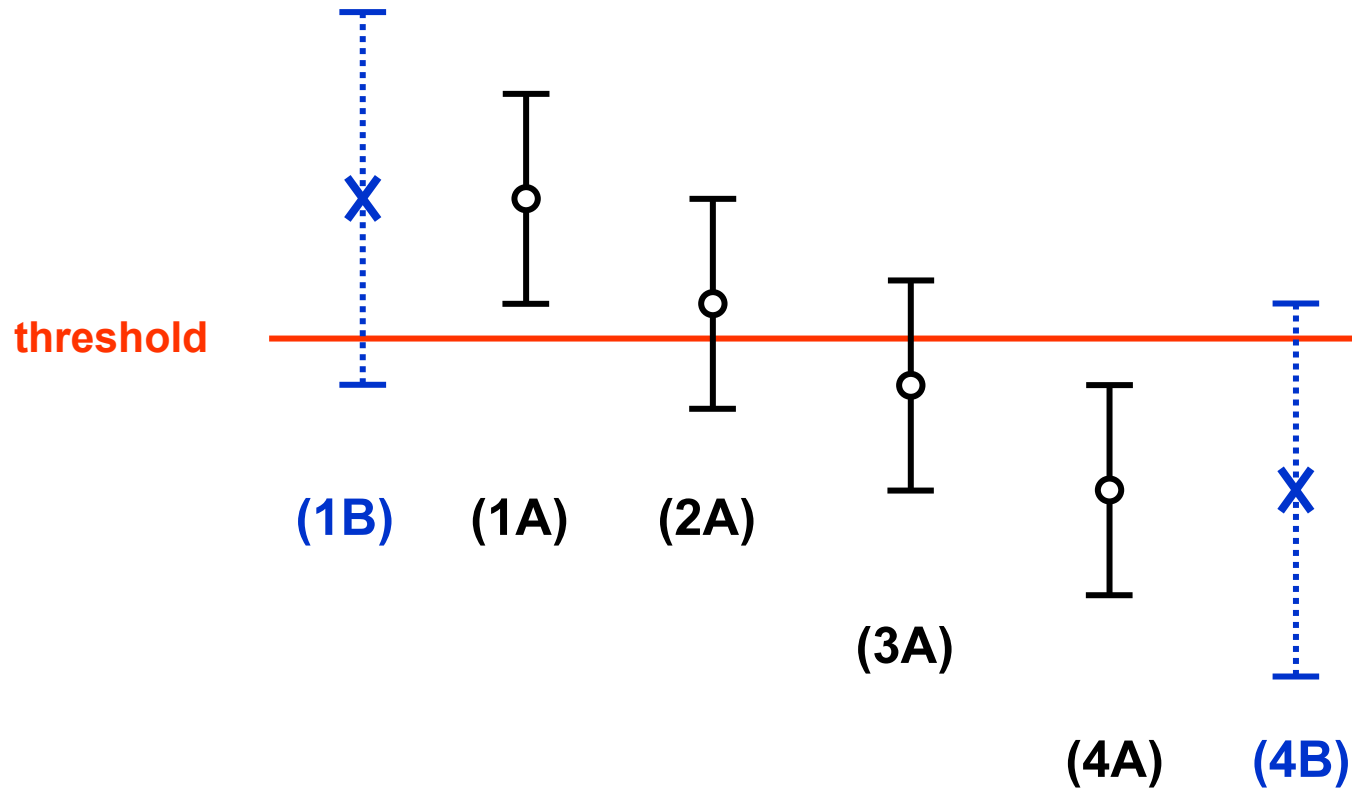
## Calibrant measurements

15+1 “EU priority PAHs” at 40-90 µg/L each in acetonitrile



(2006)

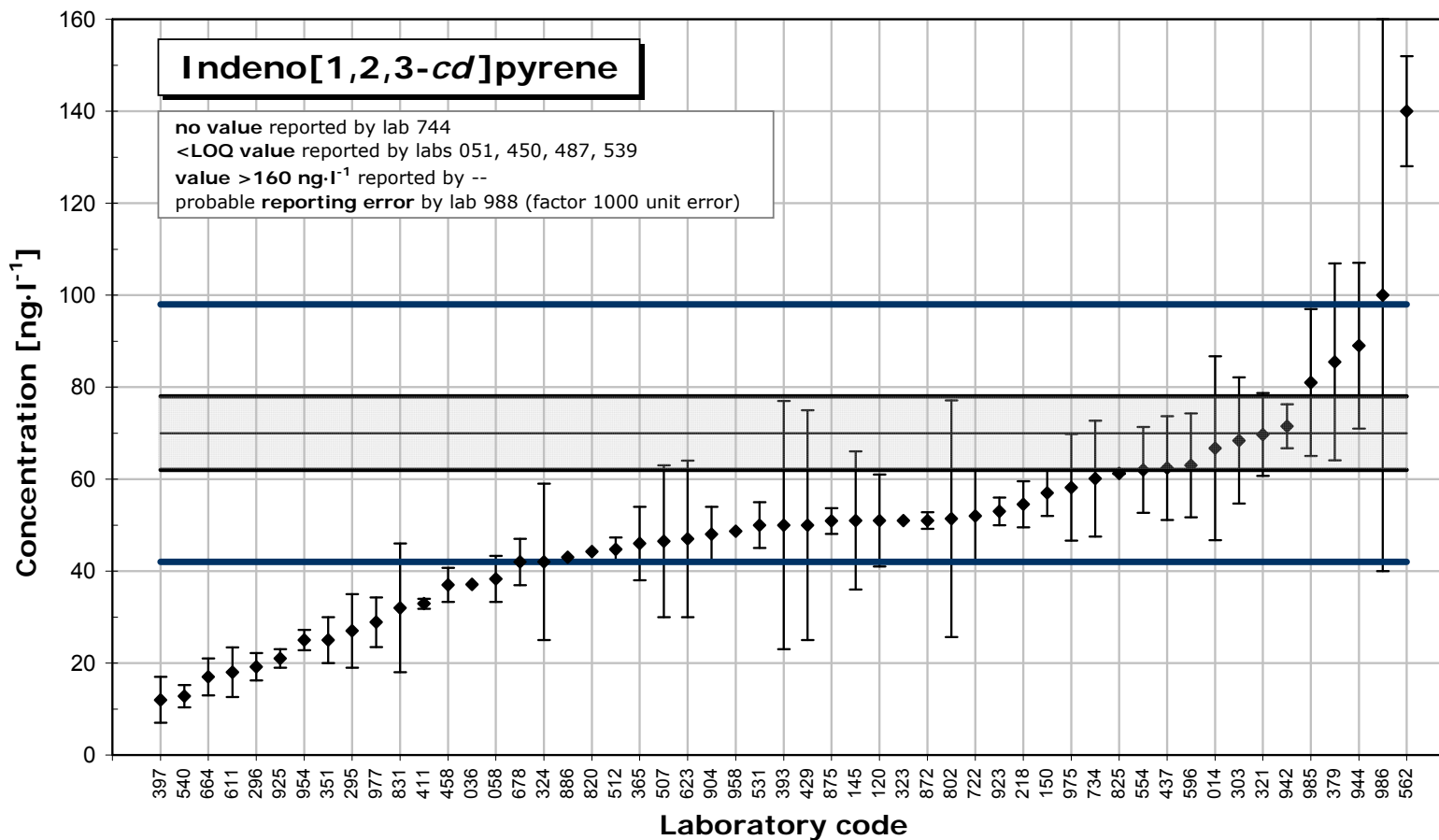
Analytical result:  $\bar{x} \pm u$



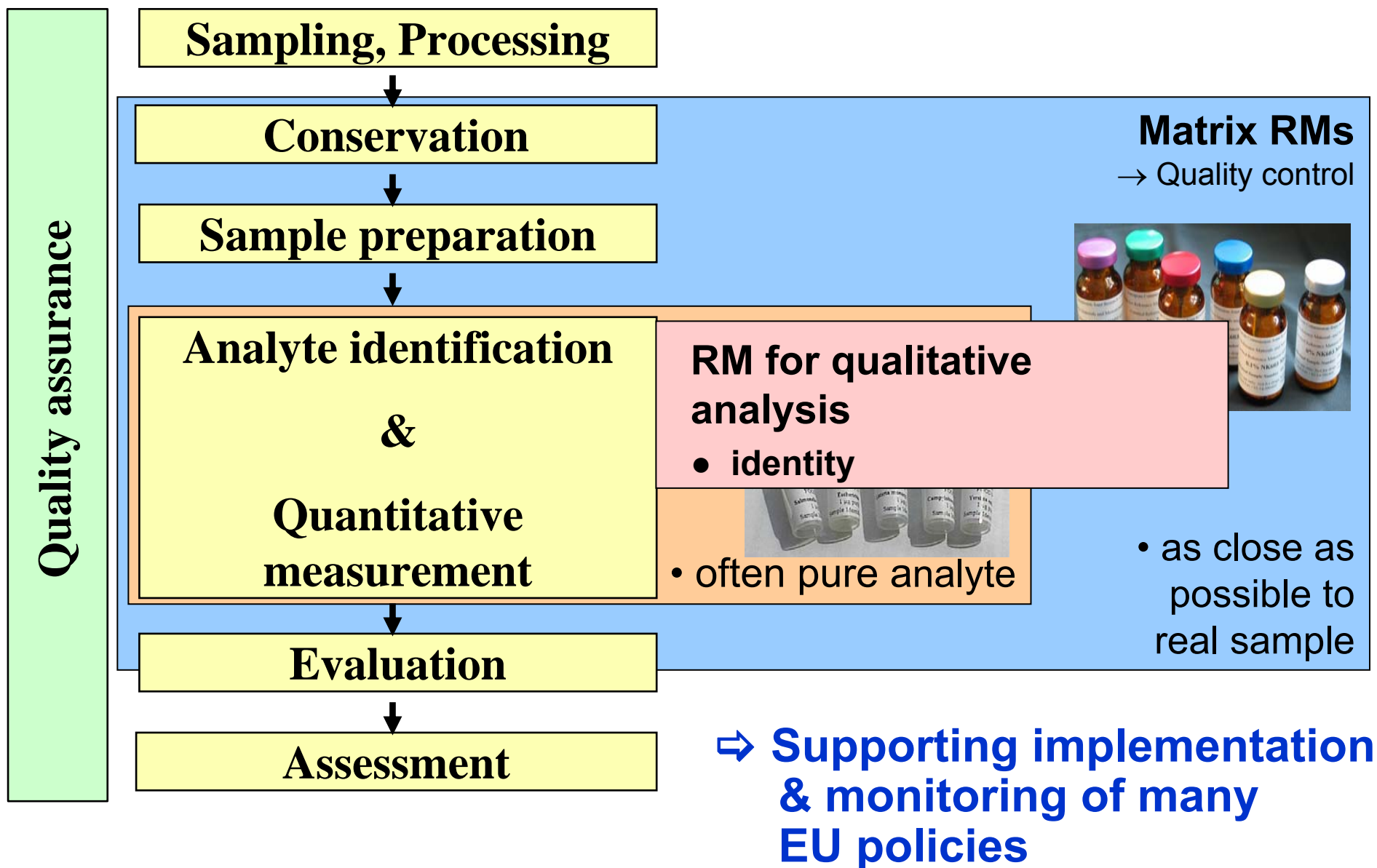
**Fit-for-decision:** by adequate uncertainty

## IMEP-23 (PAHs in water): results for indeno[1,2,3-*cd*]pyrene

Certified value:  $X_{ref} = (70 \pm 8) \text{ ng}\cdot\text{l}^{-1}$  ( $k=2$ ) **~ EQS**



**Water + Humic acid + PAH**





**Water Framework Directive  
2000/60/EC**

**Marine Strategy Framework  
Directive 2008/56/EC**



*etc.*

**Air Quality Directive  
EC 107/2004**

*etc.*

**Communication COM  
(2002) 179 final:  
Towards a Thematic  
Strategy for Soil  
Protection**



**Sewage Sludge  
Directive  
86/278/EEC**



**Are the relevant parameters to be analysed identified ?**

# Analysis

**Are analytical procedures available for the relevant parameters in adequate matrices & concentration ranges?**



**Can analytical procedures be executed with adequate performance characteristics ?**

**Are the 'instruments' for internal & external quality assurance available ?**

***Are competent laboratories available ?***

## non-certified RMs

- homogeneous subsamples
- appropriate stability

- statements on homogeneity & stability

- performance controls (precision, consistency) of methods or labs (internal & external)
- method developments



material characteristics

additional investigations

accompanying information

main applications

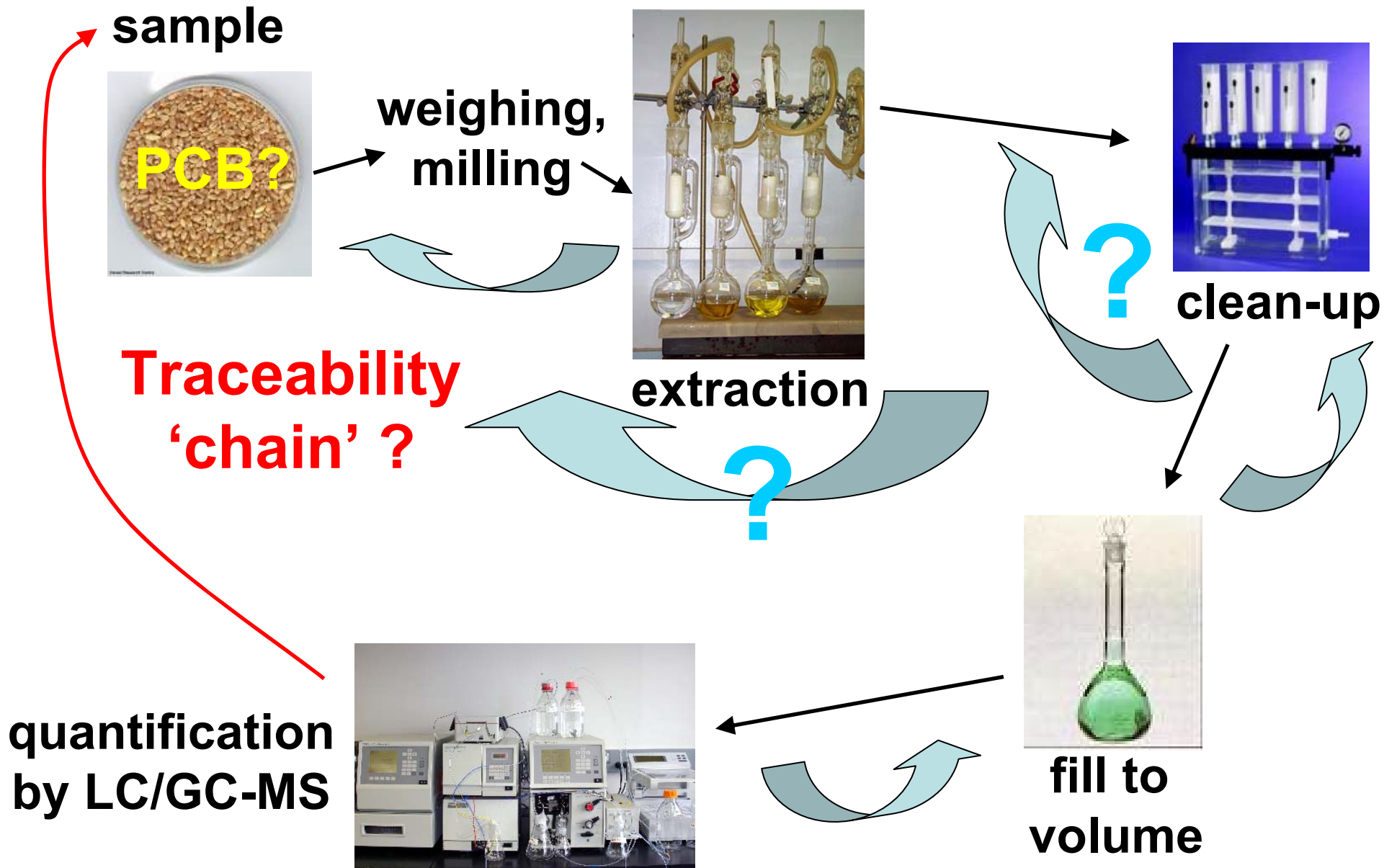
## Certified RMs

- homogeneous subsamples
- appropriate stability

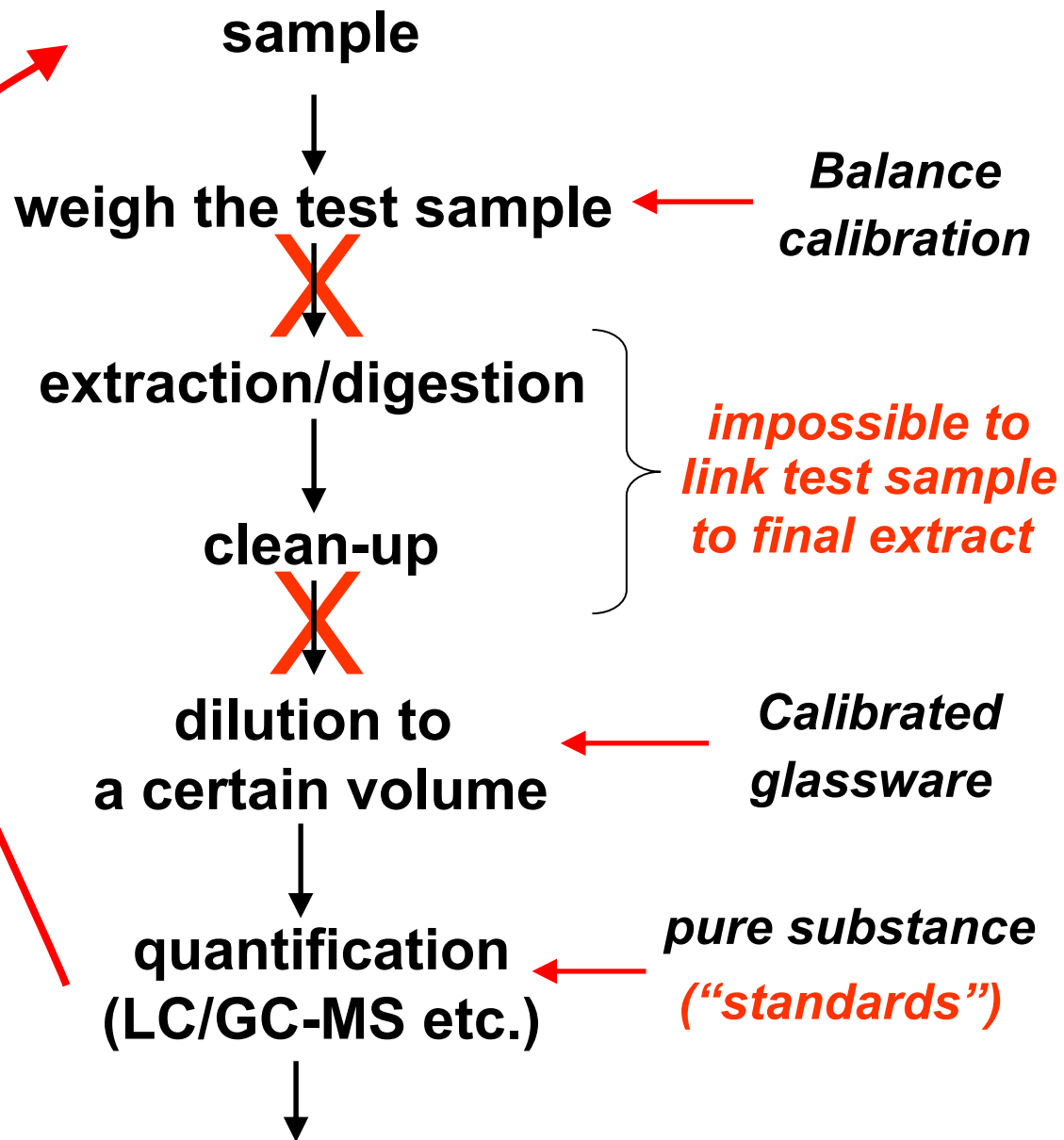
metrologically valid establishment of property value(s)

- property value(s) traceable to adequate reference system
- stated meas. uncertainty
- stated homogeneity & stability
- intended use

- calibration
- trueness control
- full method validation
- all QA/QC measures



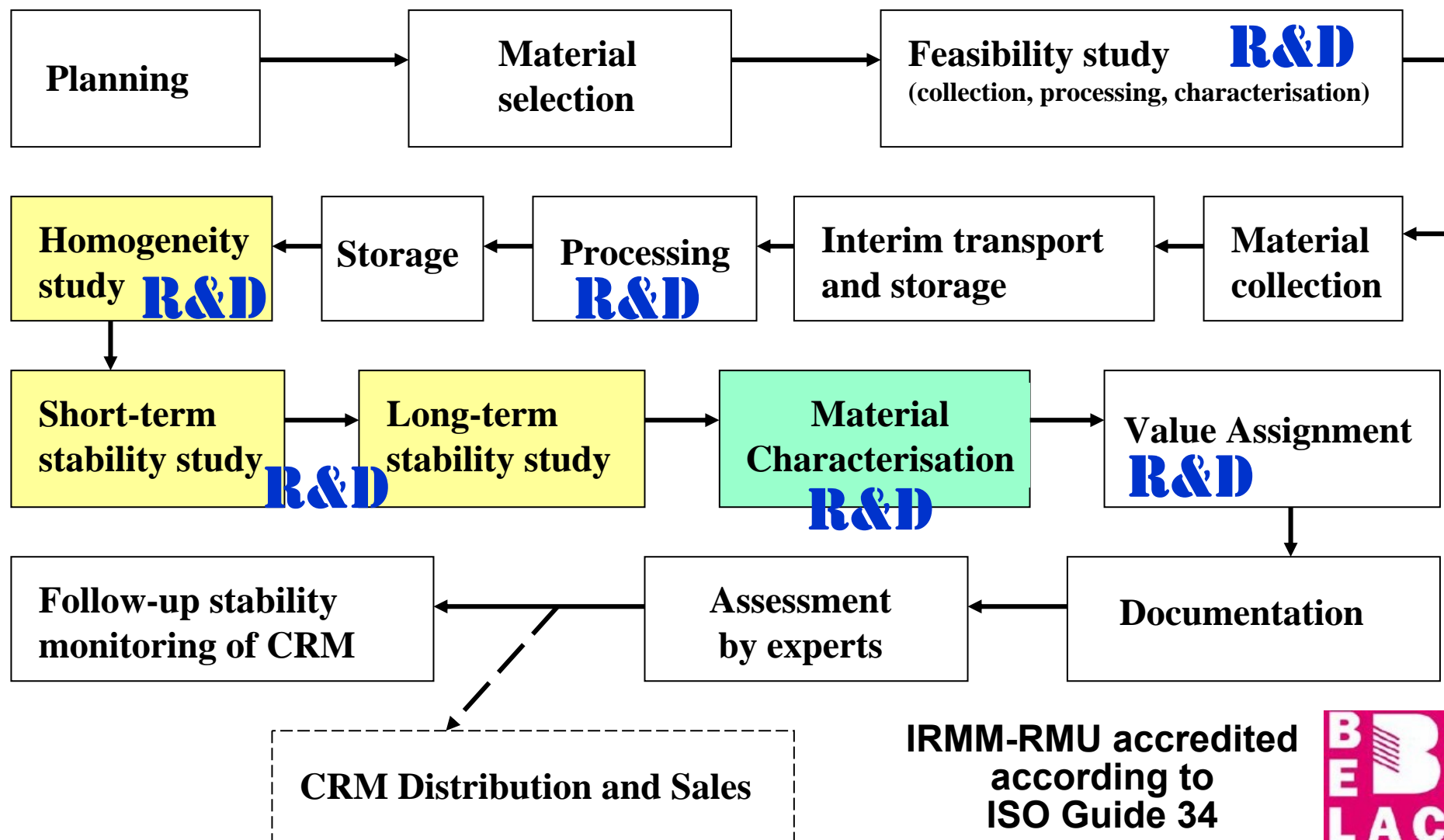
Certified reference material



## Uncertainty budget



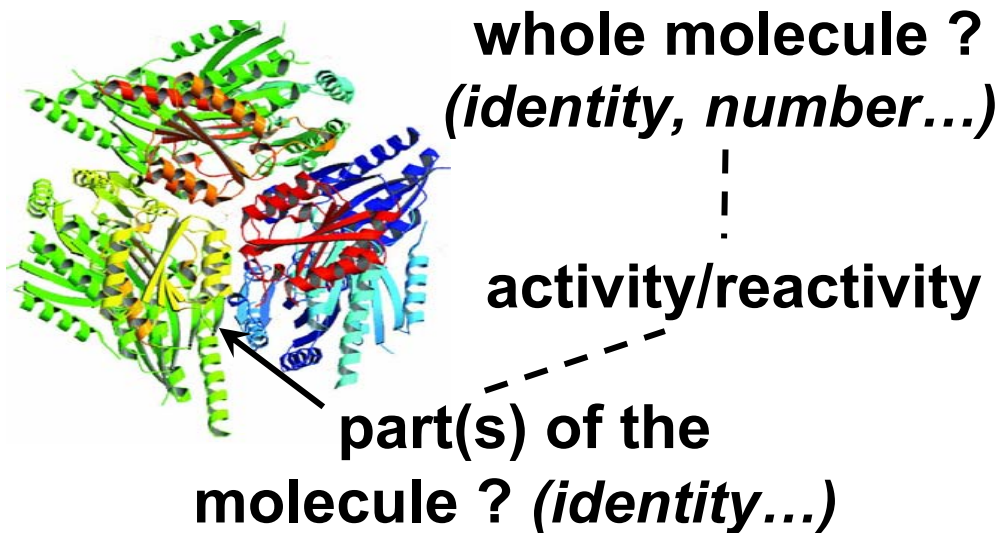
## PROBLEM DEFINITION



IRMM-RMU accredited  
according to  
ISO Guide 34



## Complex molecules / Functional properties



“trivial” for:  
simple ions (atoms)  
in homogeneous  
water (e.g.  $\text{Pb}^{2+}$ )

**but:** - multi-phase samples  
- ill-defined analytes  
(BOD, PFOS...)  
- sum parameters

## Problem: Definition of measurand & unit

- “temporary” solution:  
*measurands defined by the measurement procedure*  
(e.g., TOC, lipid content, pH, ...)

## EU Water Framework Directive 2000/60/EC & Daughter Directives

	$C_{EQS}$ [ng/L]	LOQ [ng/L]
Hg & Hg compounds*	50	$\leq 15$
Tributyltin compounds	0.2	$\leq 0.06$
Pentabromodiphenylether	0.2	$\leq 0.06$
C10-13 Chlorinated paraffins	400	$\leq 120$



no analytical procedure !

*Environmental Quality Standard in "total water"*

*(\* after 0.45  $\mu\text{m}$  filtration)*



Ref: QA Directive  
*(in press)*

Residues of sulfur in fuels are a significant source of atmospheric pollution and maximum permissible limits are systematically decreased (Directive 1999/32/EEC, Directive 2003/17/EC)

**MS laboratories controlling the fuels on the market need Certified Reference Materials to ensure the accuracy of measurement results. IRMM produces CRMs for low sulfur content in fuels.**



**Reference materials for different levels of sulfur in gas oil (diesel)**



## Directive 2002/95/EC 'Restriction of the use of certain hazardous substances in electrical and electronic equipment

“Member States shall ensure that, from 1 July 2006, new electrical and electronic equipment put on the market does not contain lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls (PBB) or polybrominated diphenyl ethers (PBDE).”



### Polymer CRMs:

- PE (*polyethylene*) and PP (*polypropylene*) certified for tetra-, penta-, hexa-, hepta-, octa-, deca-BDEs, deca-BBs & Br
- 2 polyethylene CRMs certified for As, Br, Cd, Cl, Cr, Hg, P, S, Sb



BDE ... brominated diphenylether

BB ... brominated biphenyl

## Environmental programmes:

**Ultra-clean laboratory, of 120 m<sup>2</sup>, with 16 work places and corrosion resistant construction for preparation of special spikes in the area of trace analysis such as U and Pu mixtures for quality control in particle measurement.**

**Analysis of swipe samples taken by European Safeguards (ES) and IAEA (International Atomic Energy Agency) inspectors.**



**Training of visitors from all over the world in the use of isotopic reference materials and uncertainty of measurements.**



- **Dioxin calibrants**
- **PAH calibrants**
- **Waters (waste, rain,...)**
- **Sediments**
- **Sewage sludges**
- **Soils**
- **Dusts / fly ashes**
- **Aquatic & terrestrial bioindicators**



**trace elements  
ions**



**selected  
organic  
pollutants**

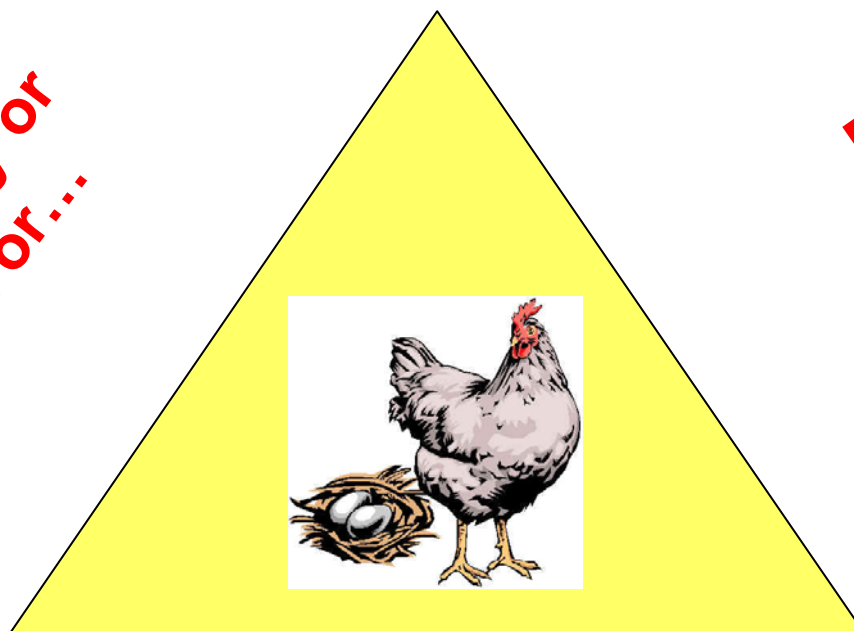
~~Driver:  
Curiosity ?~~

Analytical problem  
with decision relevance

Driver:  
Toxicology or  
Fraud or....

Driver:  
Legislation

Validated  
analytical  
methods



Reference  
Materials

Driver:  
Accreditation

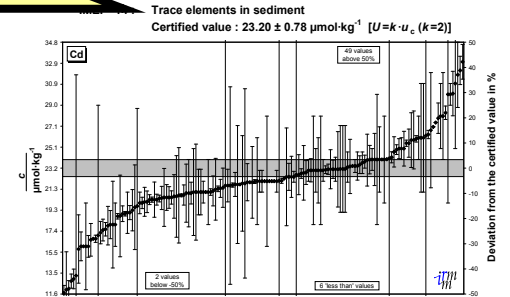
## AQA



**Validated Methods**

**Reference Materials**

**Proficiency Testing**



*Thank you !*



**RM Unit (Januar 2008)**