

# Joint Research Centre (JRC)

## Organisation of external quality assessment schemes



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

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

## Proficiency testing:

Determine the performance of individual laboratories for specific tests or measurements and to monitor laboratories' continuous performance



## Two internationally accepted guidance documents:

**ISO/IEC guide 43-1\***: Proficiency testing by interlaboratory comparisons (1997)

**IUPAC**: The international harmonised protocol for the proficiency testing of analytical chemistry laboratories\*

\* Thompson et al., Pure Appl. Chem, 78 (2006) 145-196

- 1. The JRC is not a commercial proficiency test (PT) provider**
- 2. The JRC does not make any profit with the organisation of PTs**

**?What is then the motivation of the JRC to organise PTs?**

## Lack of information on quality of data gained in different laboratories in EU

- **Comparability of data**
- **Systematic effects – biased methods**
- **Critical parameters**

### **Mostly linked to new scopes of analysis**

- Examples: acrylamide in food, furan in food, mineral oil in sunflower oil, melamine in milk powder



## Short program

- ✓ Contains all obligatory elements
- ✓ Attribution of performance indicators (z-scores)

## Free skate

➔ Adapted to particular problem

PT might include:

- › Different samples
- › Calibration check samples
- › Recovery check samples
- › Precision checks
- › Questionnaire on details of analysis method

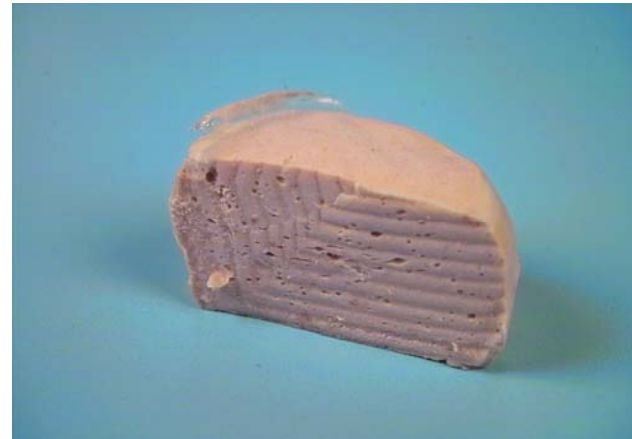
# *Short program*

## Test samples

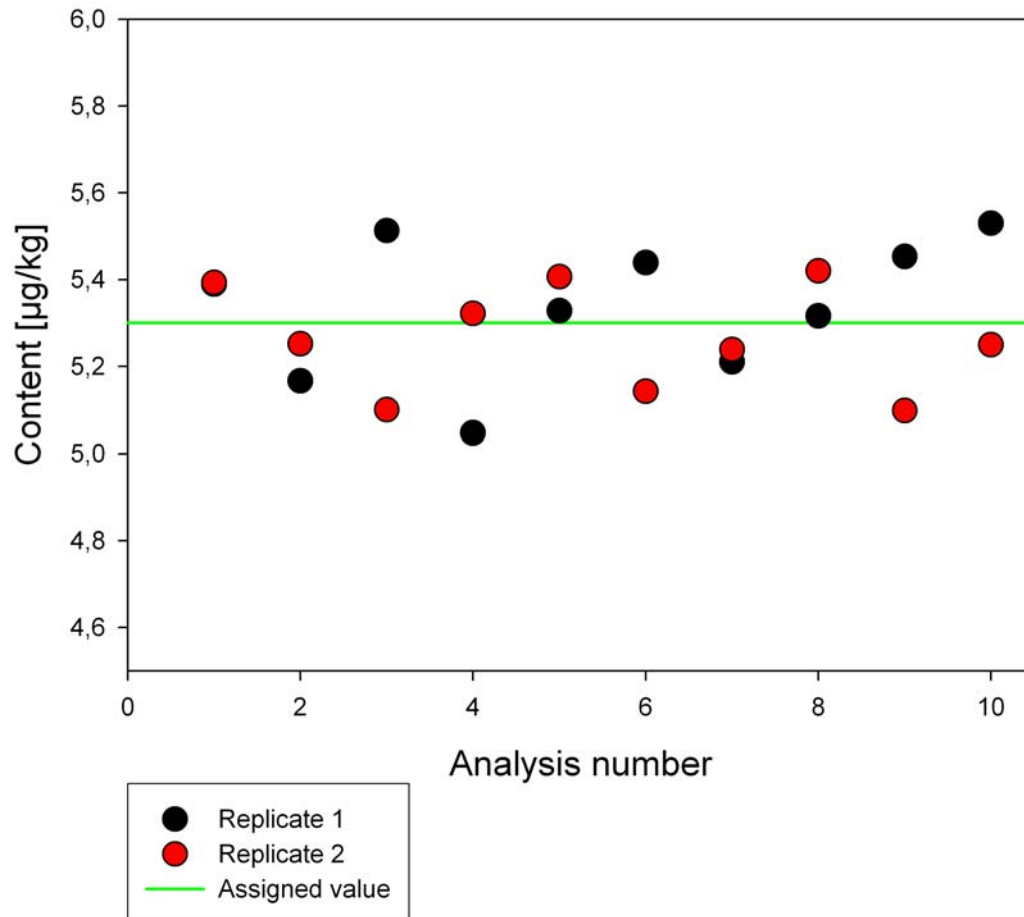
### Samples as close to reality as possible

#### Examples:

- Acrylamide in crispbread, potato crisps, butter cookies
- Furan in baby food
- PAHs in olive oil, sausage



## Homogeneity study BaP in sausage meat



## Statistical data treatment: ANOVA



ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.1845	9	0.131611	1.676575	0.216088	3.020383
Within Groups	0.785	10	0.0785			
Total	1.9695	19				

## ➤ Reference value

- From certificate
- From expert laboratories

## ➤ Application of robust statistics

- Different estimates of the assigned value
  - Often very close to each other

### CERTIFICATE OF ANALYSIS

**ERM®-BD273**


TOASTED BREAD		
	Mass Fraction	
	Certified value <sup>1)</sup> [ng/g]	Uncertainty <sup>2)</sup> [ng/g]
Acrylamide	425	29

1) Unweighted mean value of 11 accepted sets of data obtained in a different laboratory and/or with a different method of determination. The certified value is traceable to the SI.  
2) Expanded uncertainty with a coverage factor of  $k = 2$ , according to the Guide for the Expression of Uncertainty in Measurements, corresponding to a level of confidence of about 95 %.


This certificate is valid for one year after purchase.  
Sales date:  
The minimum amount of sample to be used is 1 g.

**NOTE**  
European Reference Material ERM®-BD273 was produced and certified under the responsibility of the IRMM according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available on the internet (<http://www.erm-orm.org>).

Accepted as an ERM®, Geel, December 2008

Signed: 

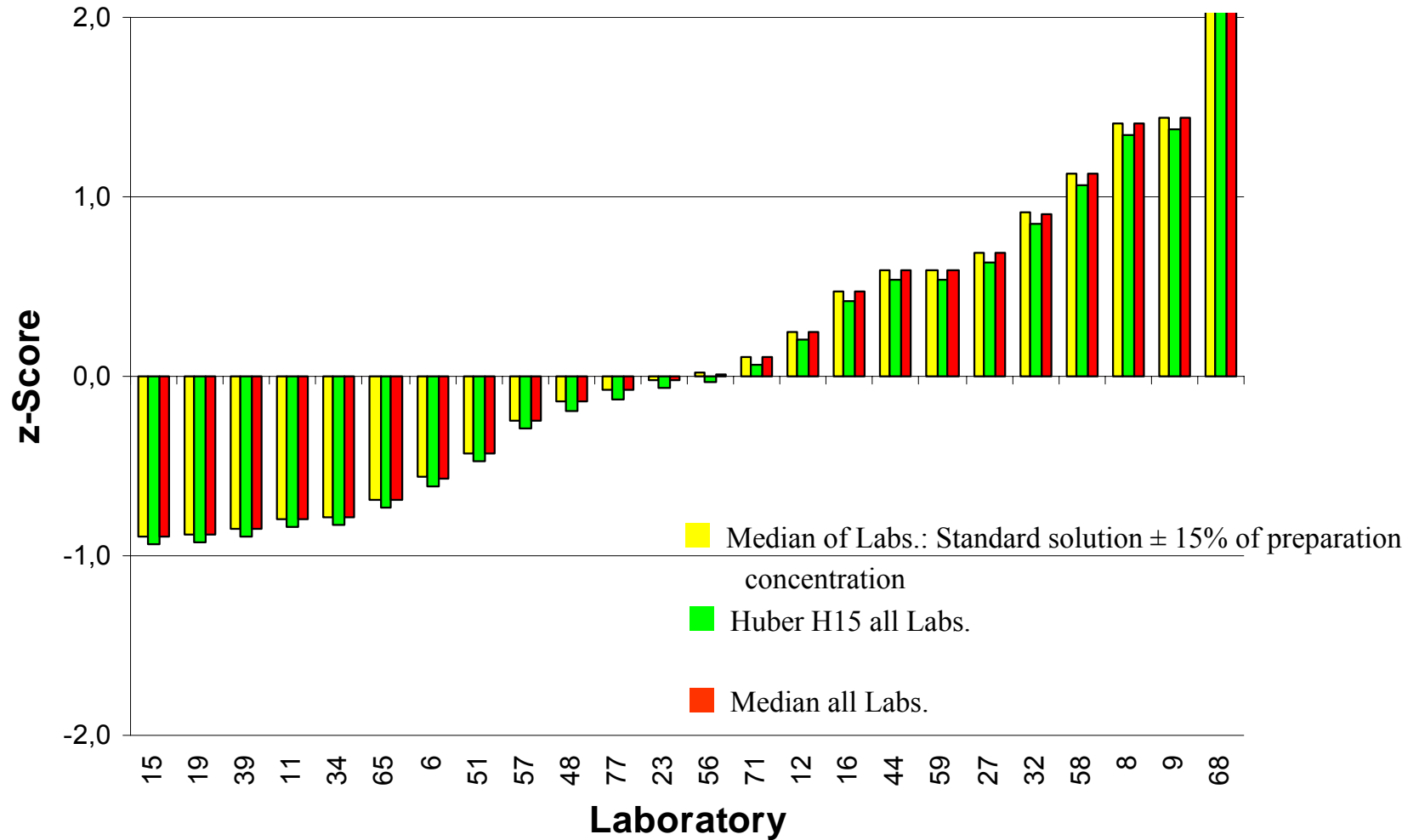
Prof. Dr. Hendrik Emons  
Unit for Reference Materials  
EC-JRC-IRMM  
Retieseweg 111  
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Registration No. 268-TEST  
ISO Guide 34 for the  
production of reference materials

All following pages are an integral part of the certificate.  
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## Application of robust statistics



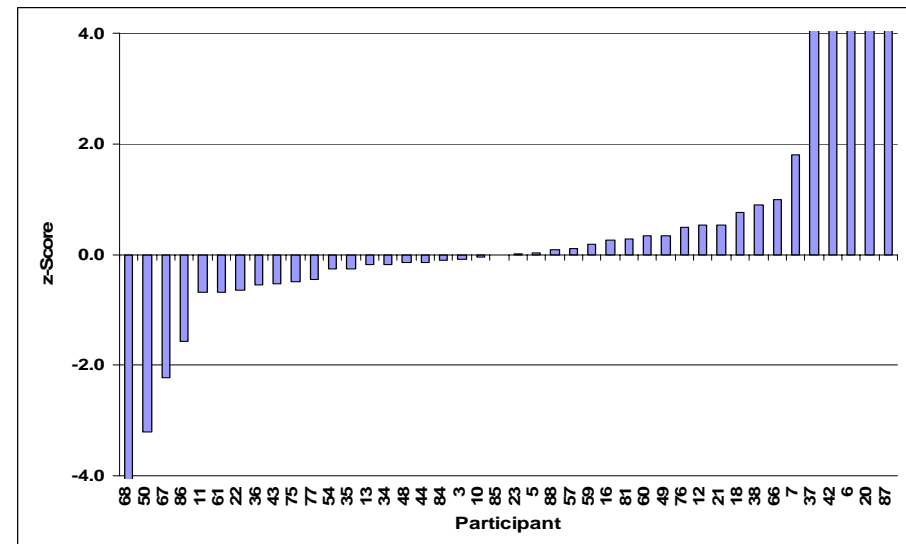
## Standard deviation for proficiency testing

- Provisions given in legislation
- Agreement among experts on fitness for purpose criterion
- Modified Horwitz equation
  - Advantage: independent of data set
    - Comparability of z-scores of different tests

## Performance indicators

- **Mostly z-scores**

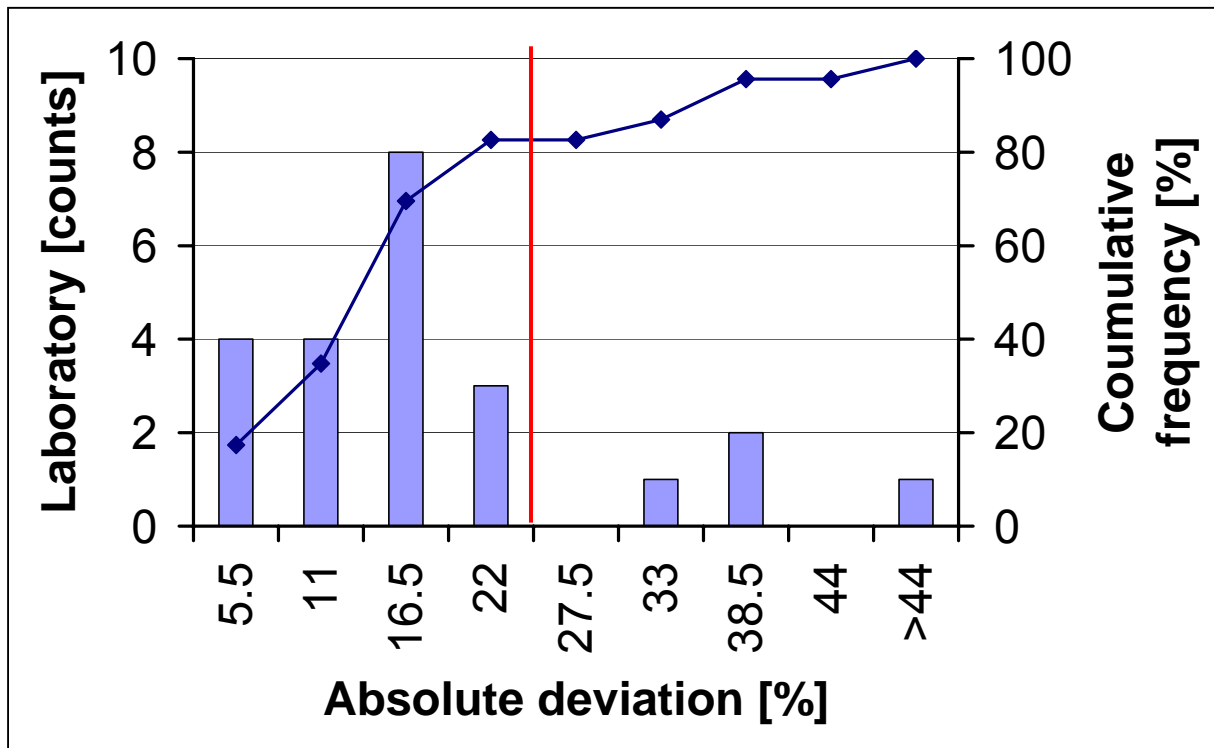
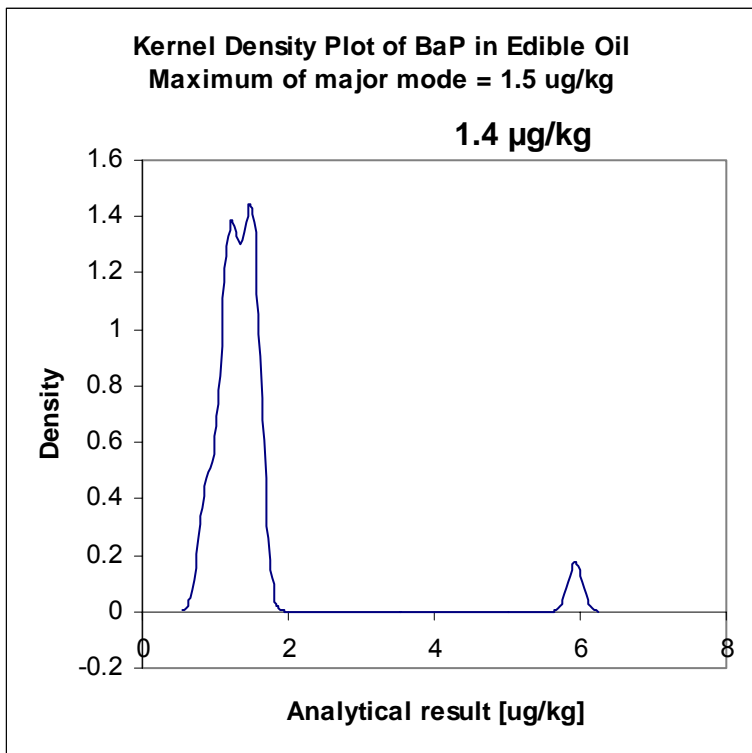
$$z_i = \frac{x_i - \hat{X}}{\sigma_P}$$



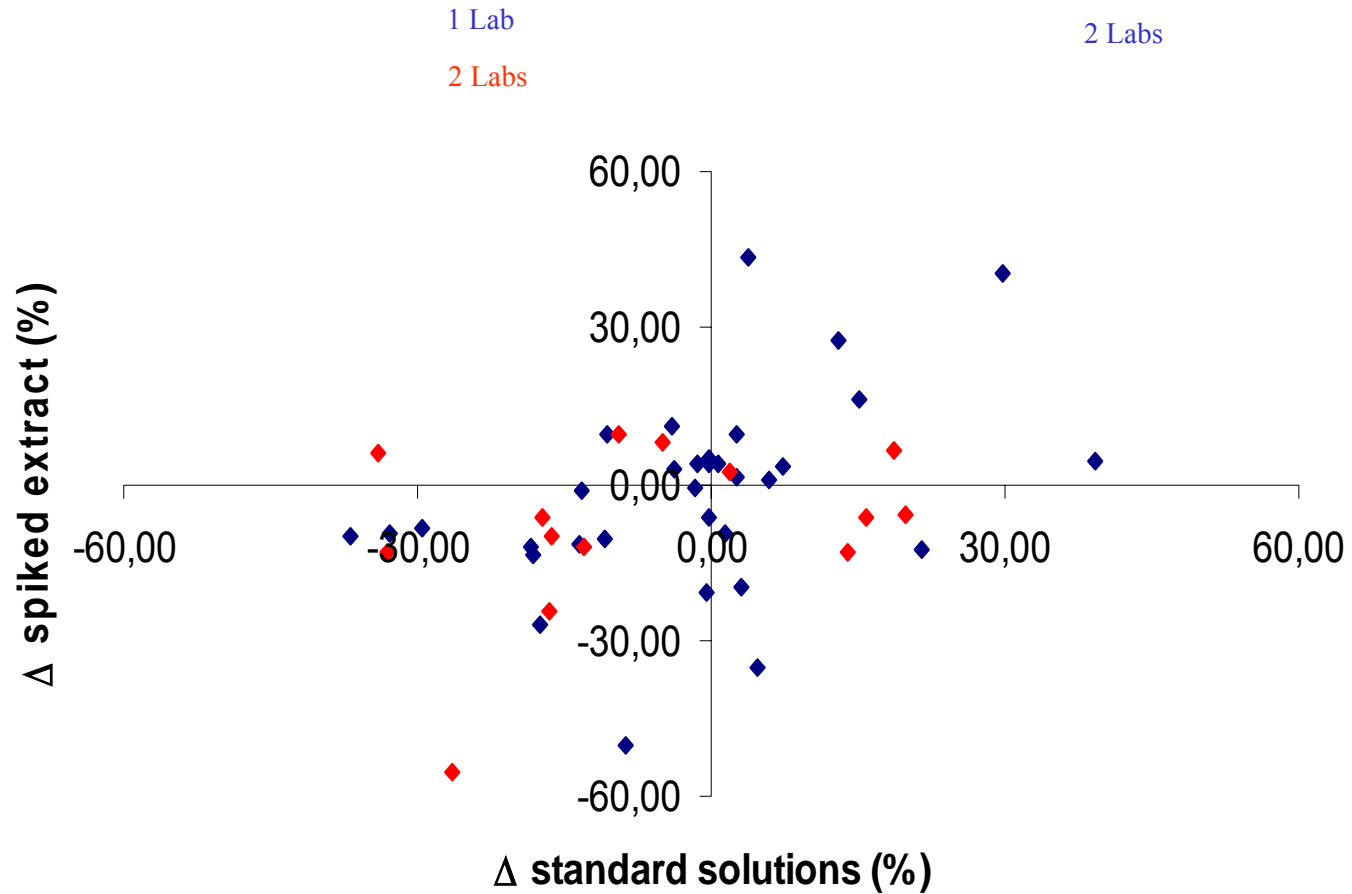
*Free Style*

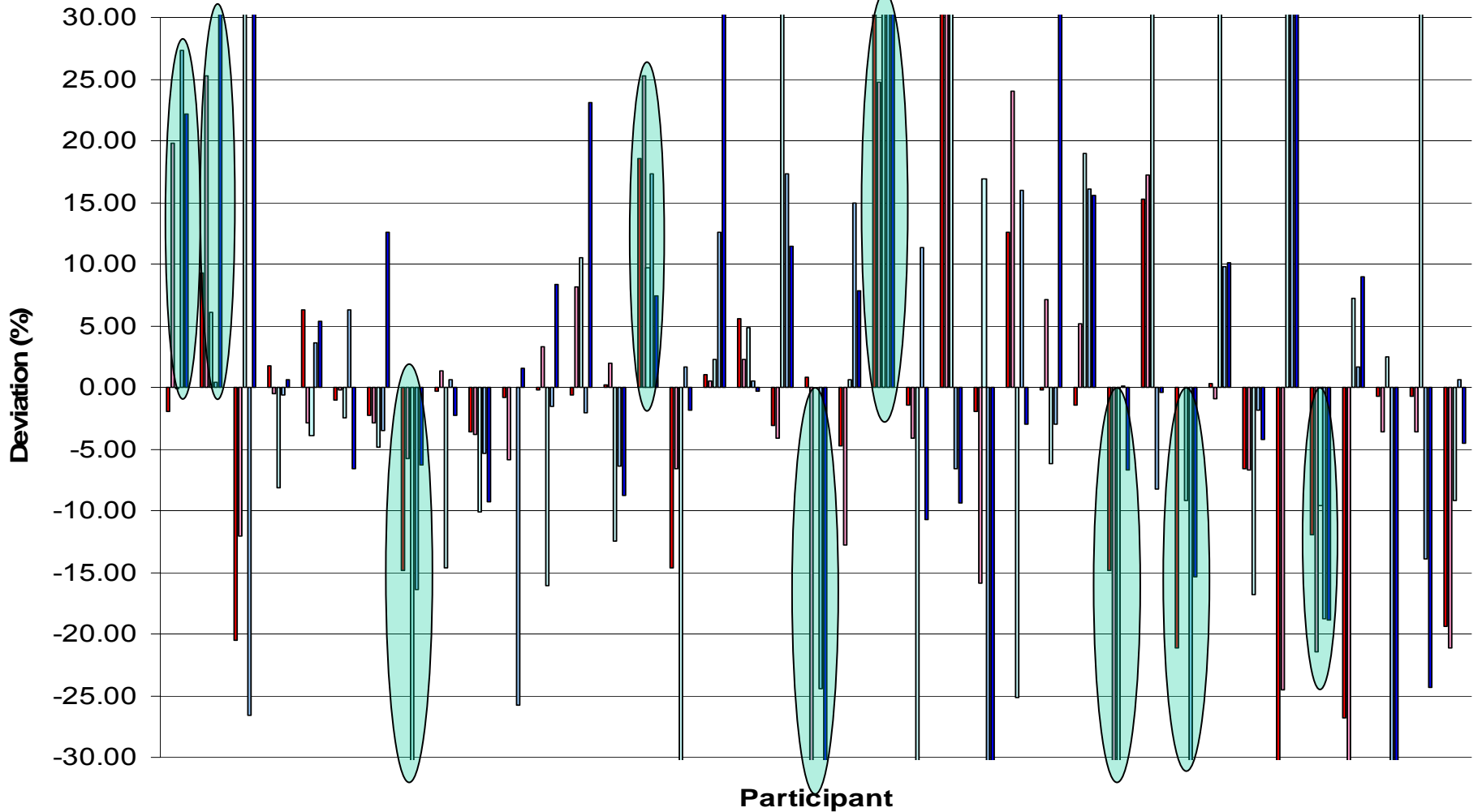
## BaP in olive oil

**82 % < 1s    96 % < 2s**

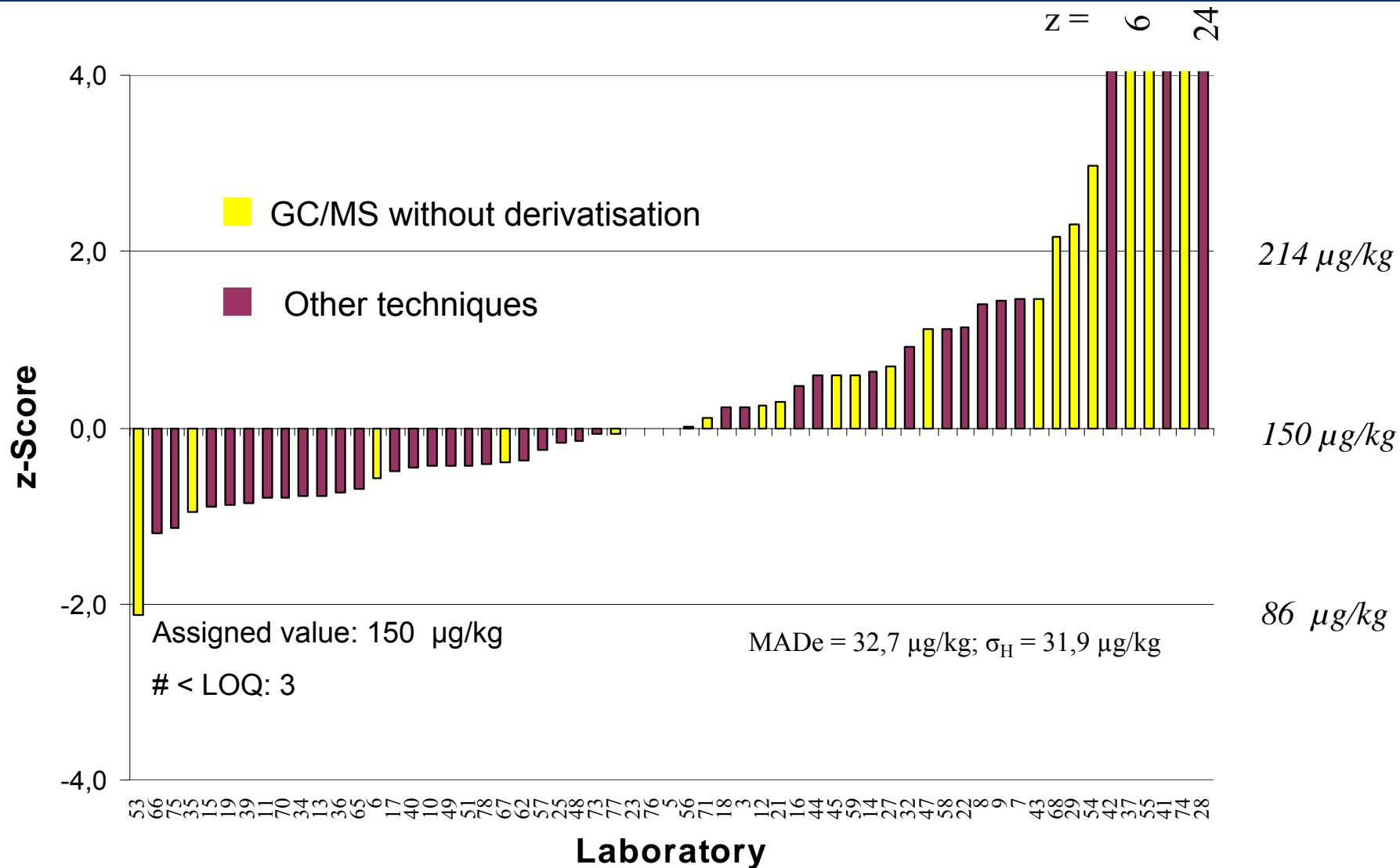


## Deviation of measured values from assigned values (%)





■ Standard A/C ■ Standard B/D ■ Crispbread 1 ■ Crispbread 2 ■ Crispbread 3



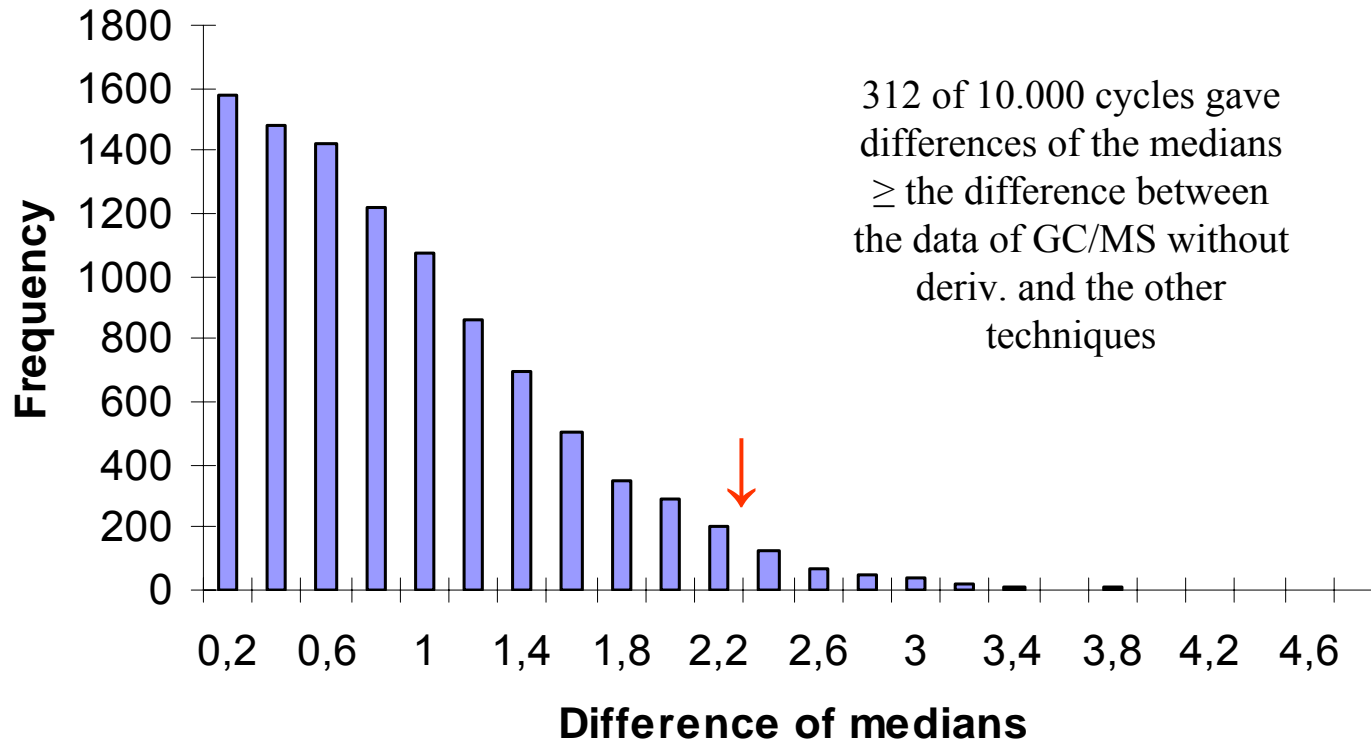
## How to detect influence of technique?

### Analysis of data set by resampling statistics (bootstrap)

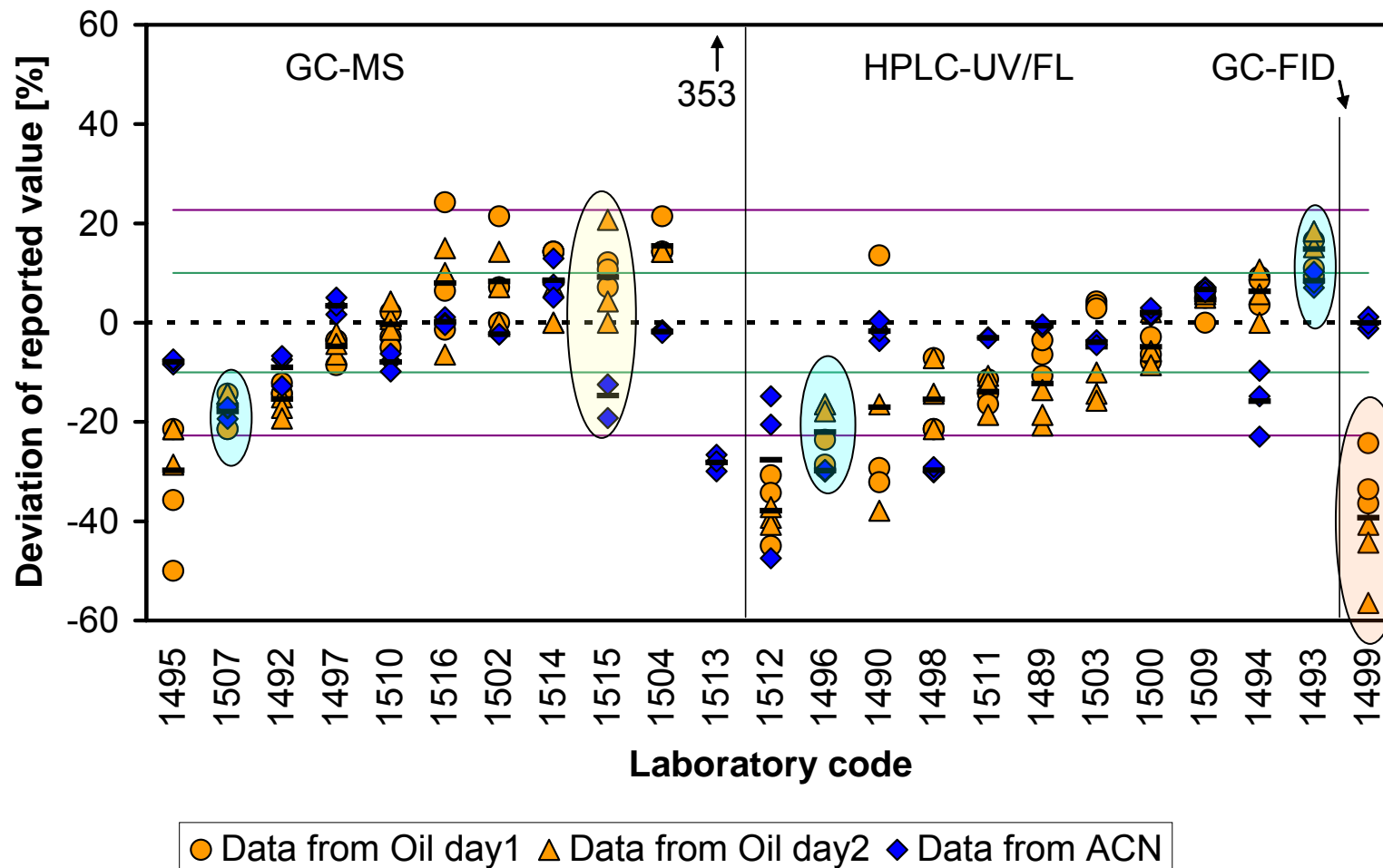
Principle: assumption that data are from one population  
(outliers eliminated)

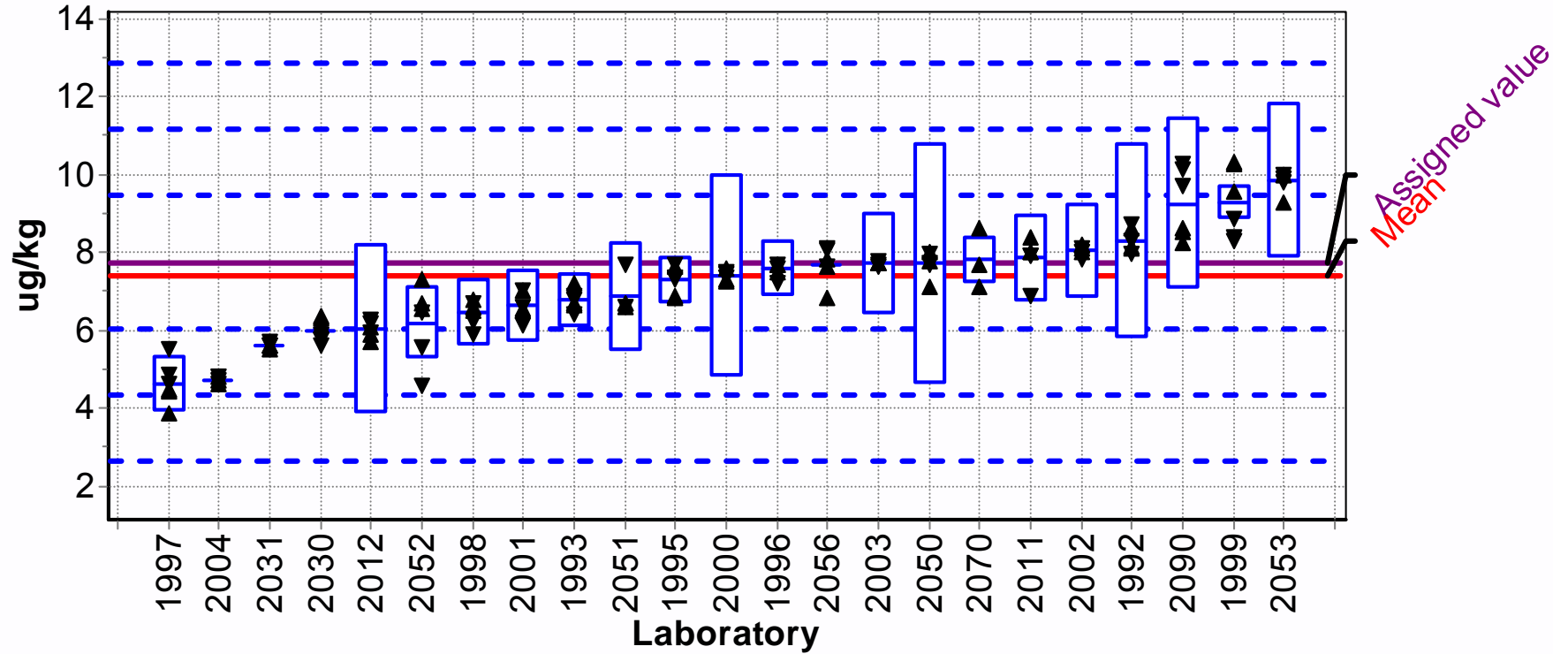
- 1) Determination of medians for GC/MS without deriv. and other techniques
- 2) Merge of all data
- 3) Take randomly selected sub-sample from data set and calculate median
- 4) Calculate absolute difference between new median and median of original data set
- 5) Perform 10.000 independent calculations
- 6) Estimation of probability for the difference between medians of original data sets for different techniques simply by the determination of its frequency

## Butter cookies by GC/MS without deriv compared with other techniques 10000 cycles

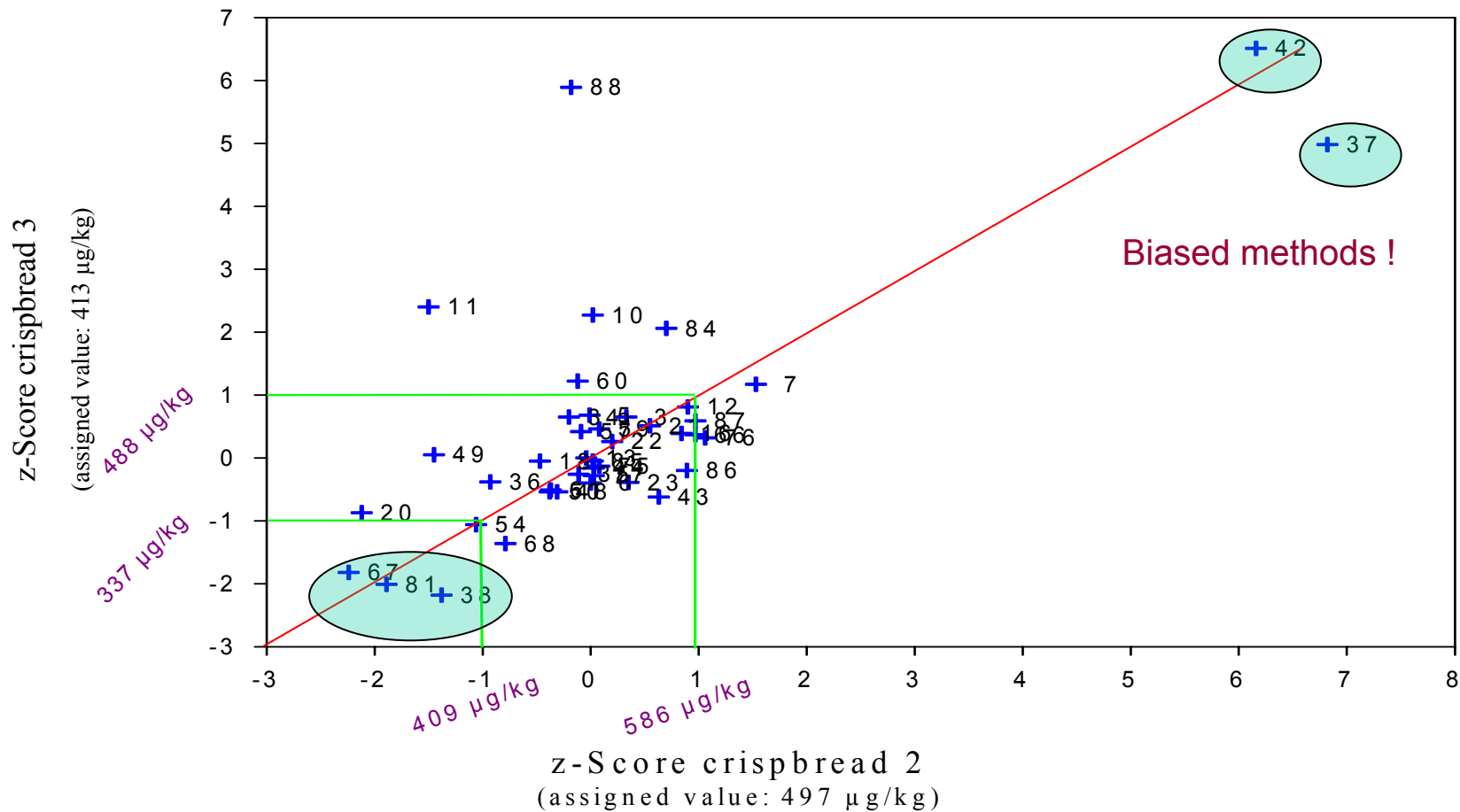


## BaP in olive oil

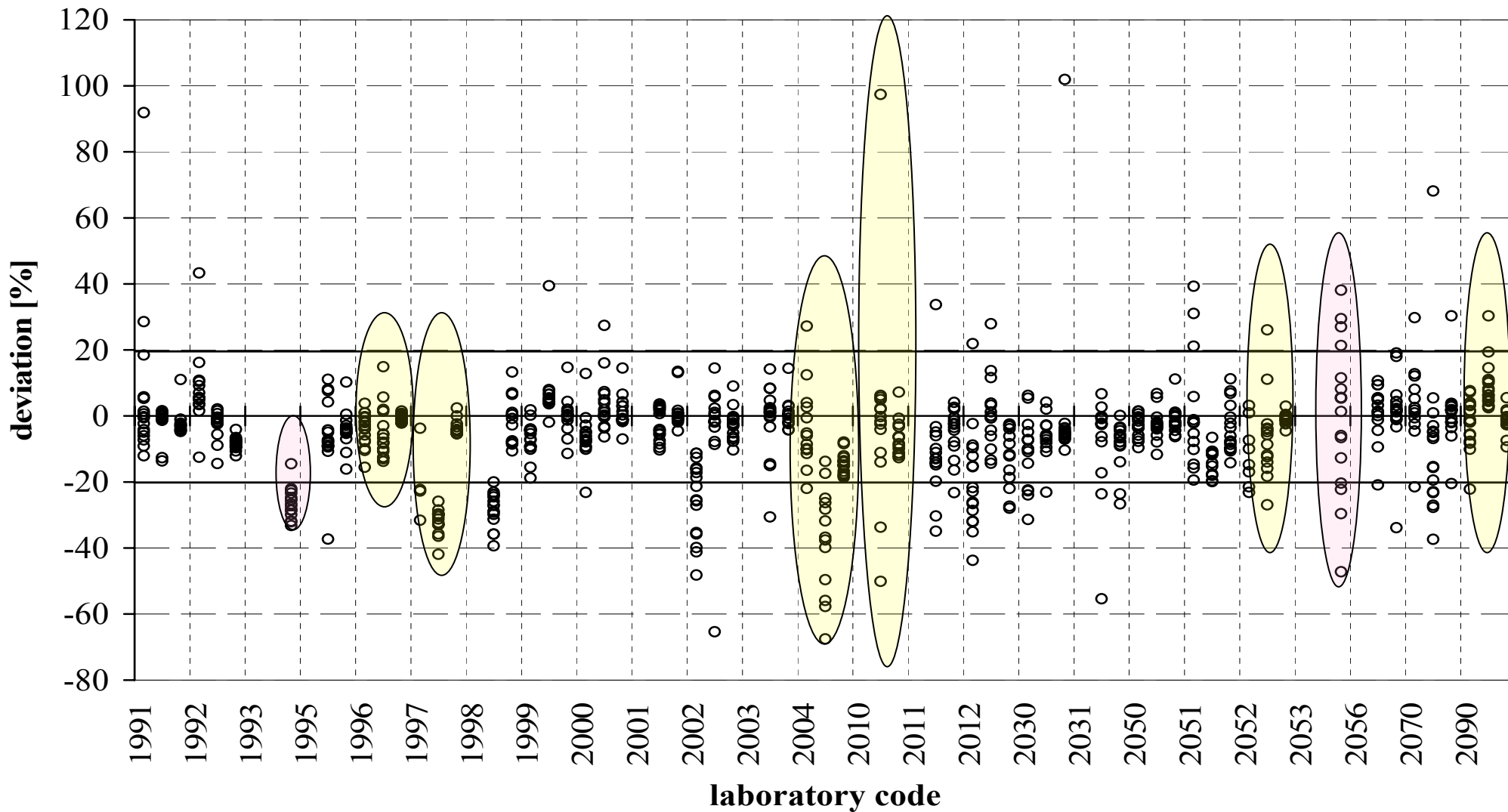




Comparison of performance indicators for CB2 and CB3



## Deviations for a set of analytes in solvent solution in three consecutive rounds



**PTs organised at the JRC for different purposes**

**Designed to answer to particular questions**

**Scale exceeds frequently that of commercial PTs**

**Extra efforts aim to provide Win-Win situation**