



# Report on activities carried out by the European Commission's Joint Research Centre

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# Workshops organised

- **European Workshop on Analytical Methods for the Determination of Acrylamide in Food Products, Oud-Turnhout, Belgium, 28-29 April 2003 (EUR 20766 EN)**
- **1. Meeting of Task Force Group on Acrylamide Analysis, Brussels, Belgium, 14 October 2003**



# Method and Data Collection

- **Review on analytical methods: Food Add. Contam. 20, 885 (2003)**
- **Hosting of European Monitoring Database: spreadsheet at [www.irmm.jrc.be](http://www.irmm.jrc.be)  
contact address: [acrylamide@irmm.jrc.be](mailto:acrylamide@irmm.jrc.be)**
- **Test material (crisp bread and cocoa powder) available upon request**



**Results of the collaborative trial  
on the analysis of acrylamide  
in two food matrices  
*carried out in 2003***





# Overview on participants

- **78 Laboratories applied for the participation and were supplied with test samples**
- **62 Laboratories reported results**
  - from 12 EU Member States and 3 non EU countries
- **Experience**
  - more than 25% less than 10 samples per month
  - 40% more than 25 samples per month



# Sample description

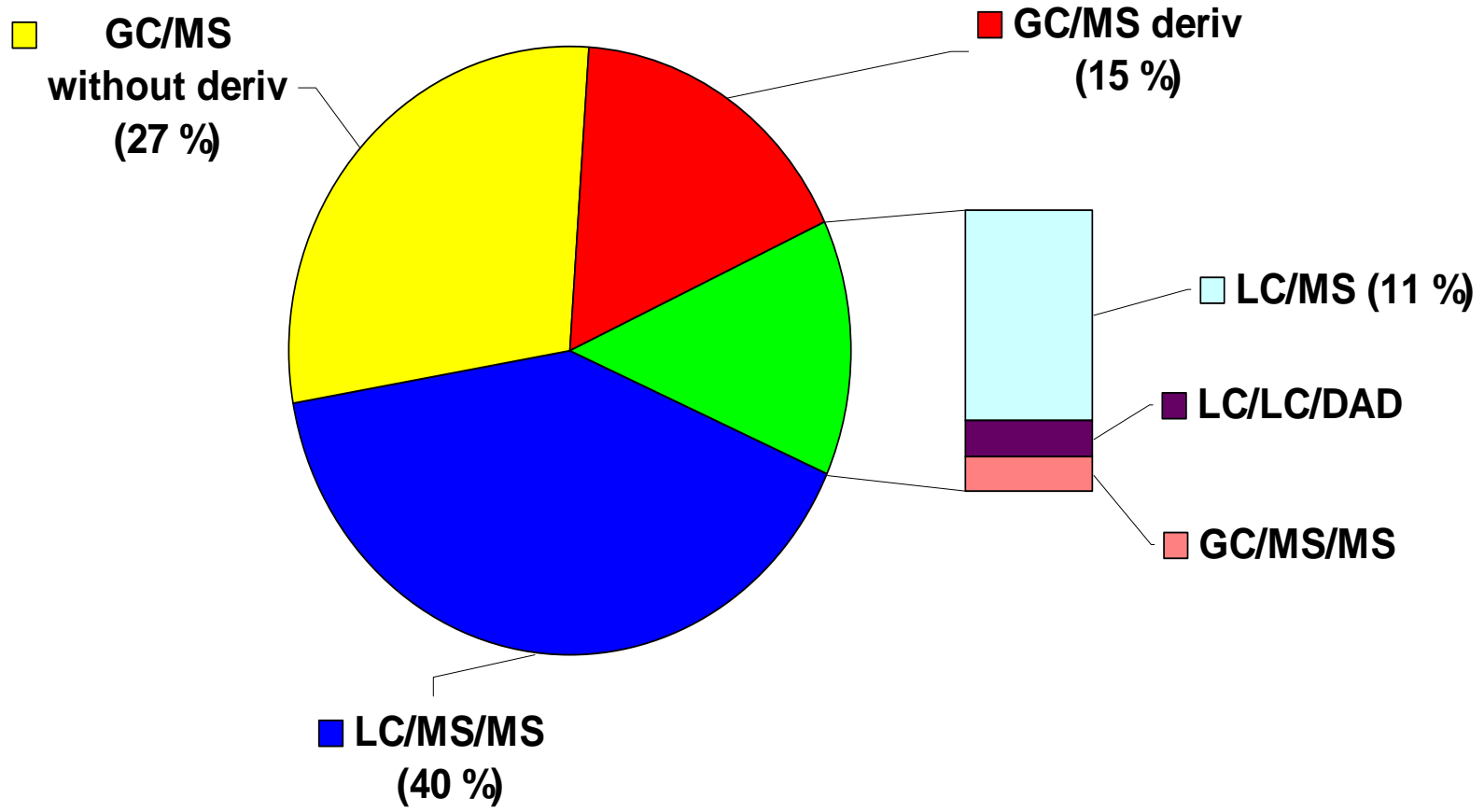
- 1) Crisp bread sample: real sample, acrylamide content: low (57  $\mu\text{g}/\text{kg}$ )**
- 2) Butter cookies sample: real sample, acrylamide content: medium (150  $\mu\text{g}/\text{kg}$ )**
- 3) White bread crumb extract: acrylamide content below LOQ**
- 4) Spiked white bread crumb extract: spiking level: 116  $\mu\text{g}/\text{kg}$**
- 5) Acrylamide standard solution**



# Analysis technique

86,6 % of participants applied isotopic labelled standards

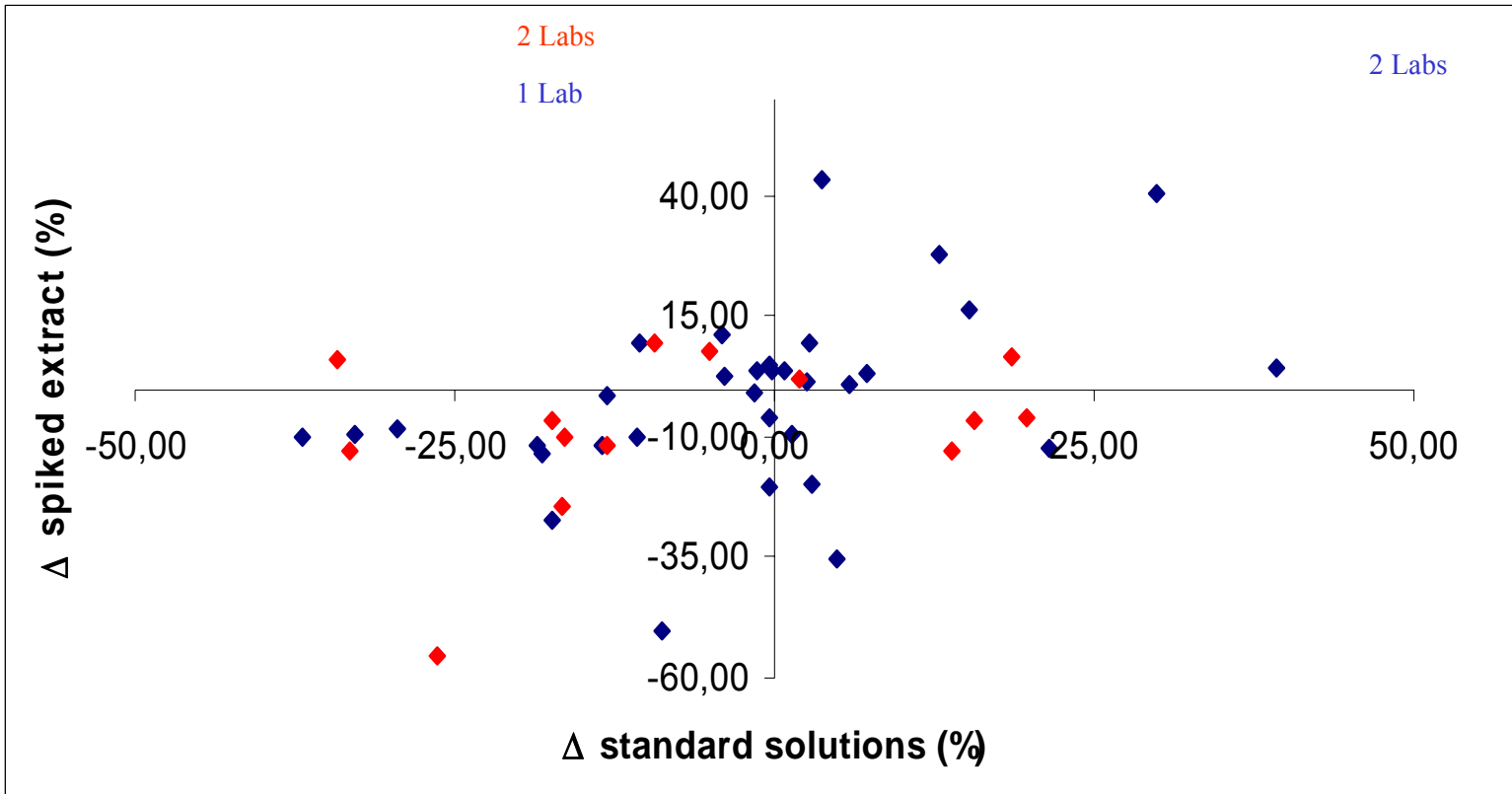
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# Calibration check

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



Standard in EtAc

Aqueous standard

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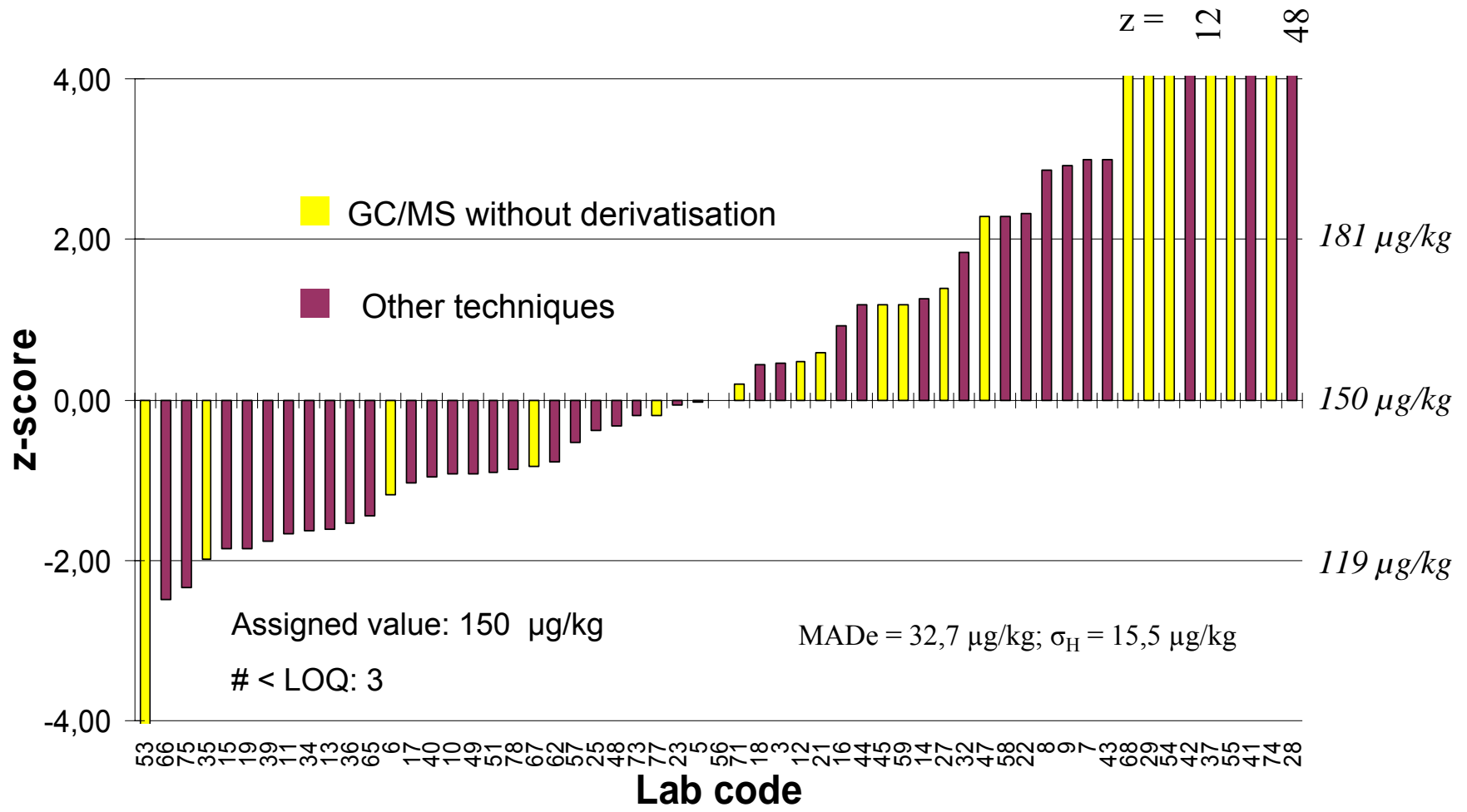
# Calculation of z-scores

- **Check of distribution of data**
  - Deviation from normal distribution
- **Application of robust statistics**
  - Different estimates of the assigned value
    - Median and Huber H15 were nearly equal
- **Estimation of standard deviation**
  - Modified Horwitz equation
    - Advantage: independent of data set
  - But point of discontinuity at 120  $\mu\text{g}/\text{kg}$



# z-Scores for butter cookies

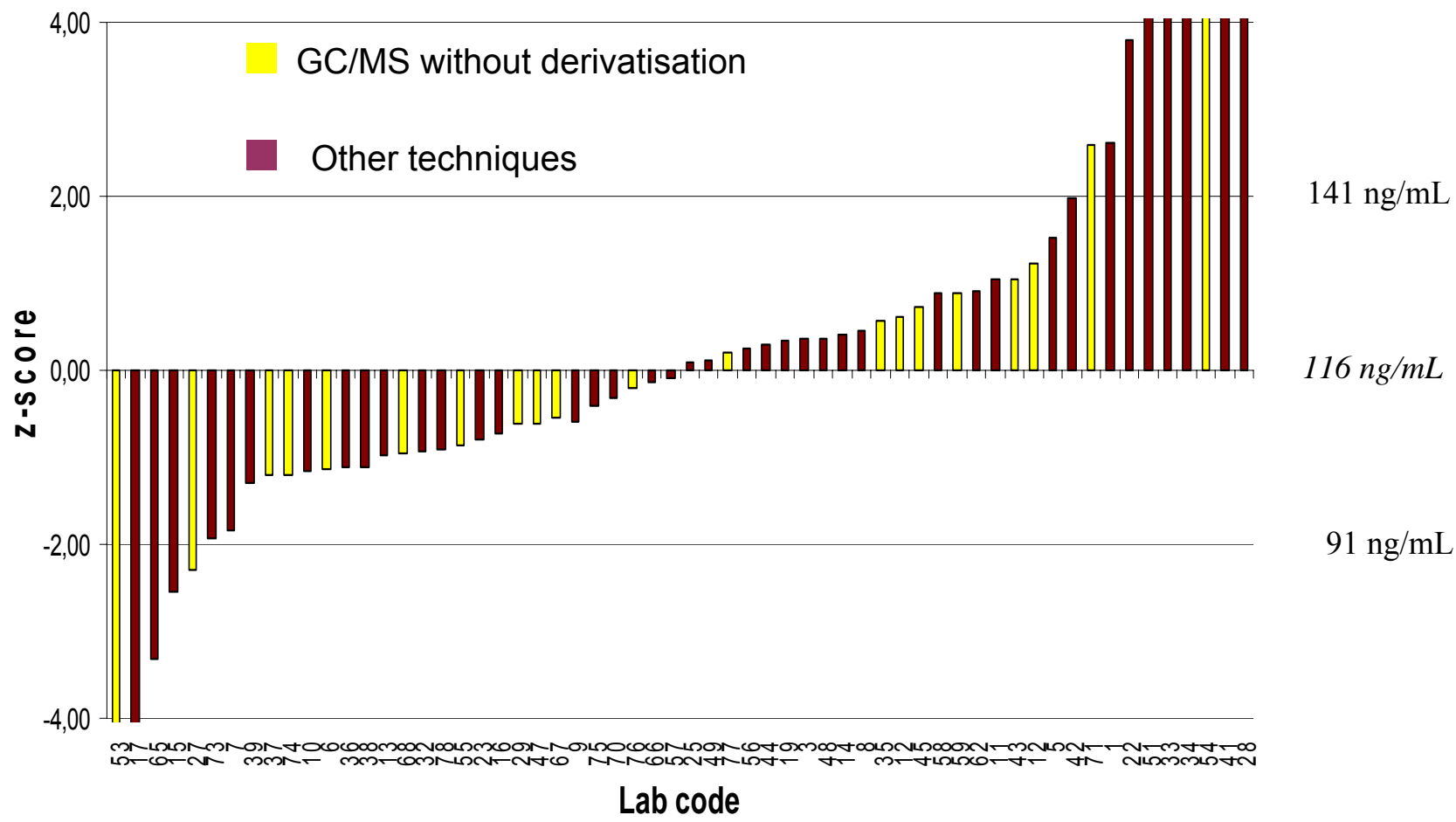
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# z-Scores for spiked extract

z-scores calculated with lower  $\sigma_H$



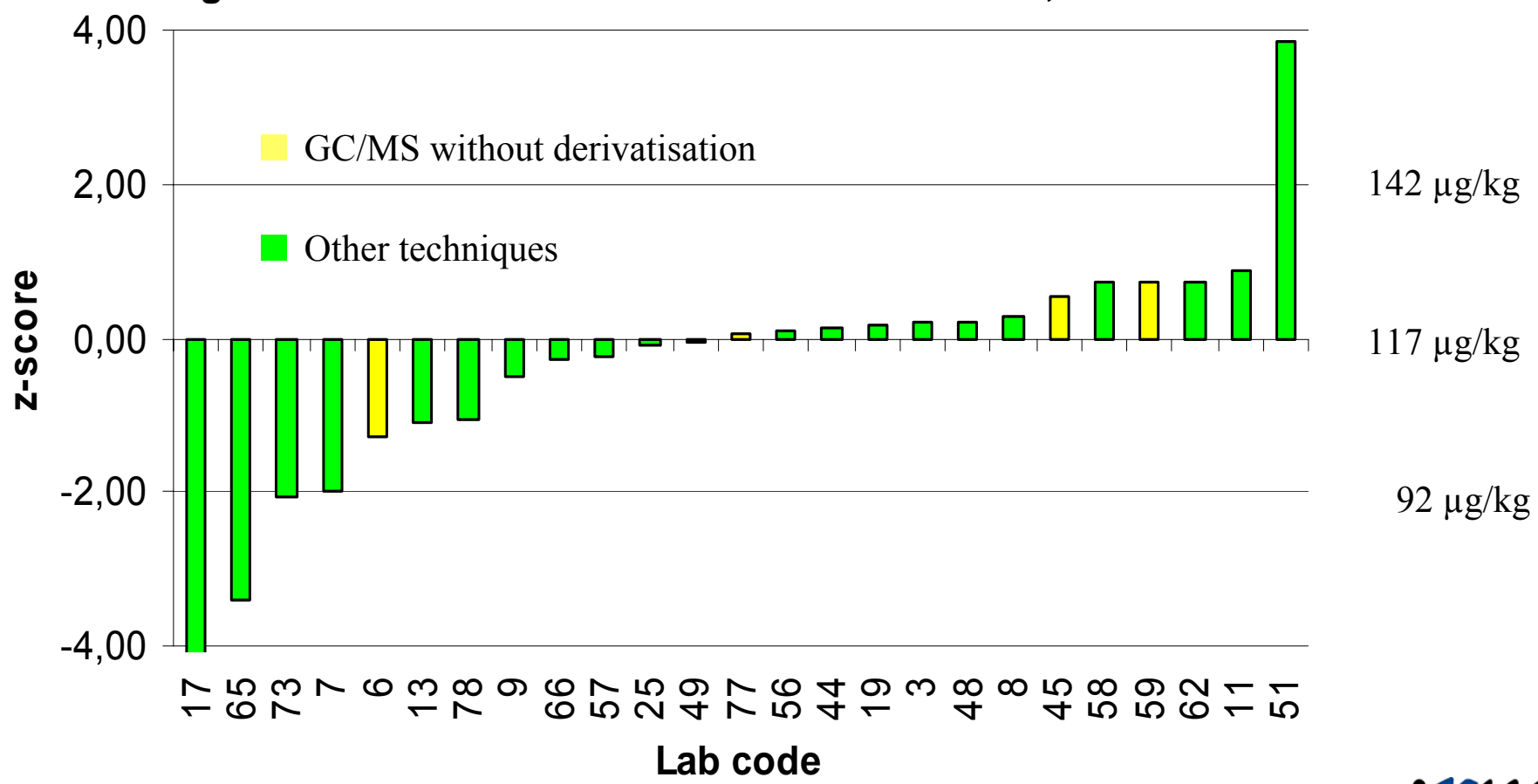
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# z-Scores for spiked extract - corrected

All participants, which reported deviations from the assigned value greater than  $\pm 15\%$  for the AA standard solution, were excluded



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# Summary

- Obviously laboratories have calibration problems
- Overestimation of AA content concerning analysis by GC/MS without derivatisation
- Additional training for many laboratories required



# Next steps

- **New round currently running with similar/same sample matrices (1 crisp bread sample, 1 extract of one real sample, 2 standards for calibration check)**
- **Method validation**
- **Proficiency test with coffee and cocoa powder**
- **Certification of reference material (crisp bread)**

