

RMCC Meeting 2014, Amman

# Proficiency test system of the IAEA Terrestrial Environment Laboratory

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## Topics

- Concept of the PTs and materials
- PT evaluation scheme
- Sample preparation
- Results and their interpretation



## Concept of the Proficiency Test

- **Traditions:**

- Keeping the Mutual agreement at the second ALMERA meeting 2005
- Regular sample set: water, biota, soil type material
- Predefined evaluation system
- Both natural and anthropogenic radionuclides



## Concept of the Proficiency Test

- **New elements:**

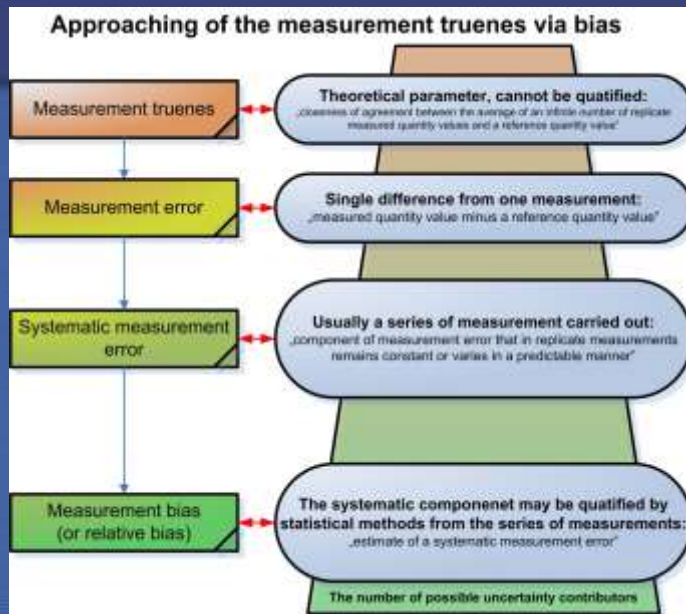
- The analytes were not defined in advance (the participant should select them from a list)
- New PT evaluation scheme (MARB, %)
- Bias trend analysis
- QC sample with known activity values

- PT history

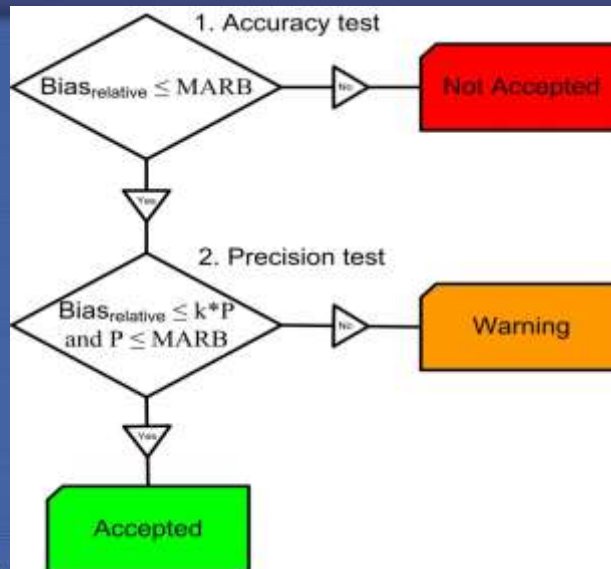


## Proficiency Test Evaluation Method

- **Key parameter:**
  - **M**aximum **A**cceptable **R**elative **B**ias specified in % (MARB)
  - The MARB value derived from method parameters considering general laboratory circumstances:
    - 30% relative efficiency detector
    - 100 cm<sup>3</sup> sample volume in cylindrical geometry
    - reasonable counting time (10000-80000 sec)
    - Typical uncertainty budget (calibration source, efficiency curve fitting, counting statistics, long term stability, variation of the background)



## Evaluation scheme



## Proficiency Test Evaluation Method

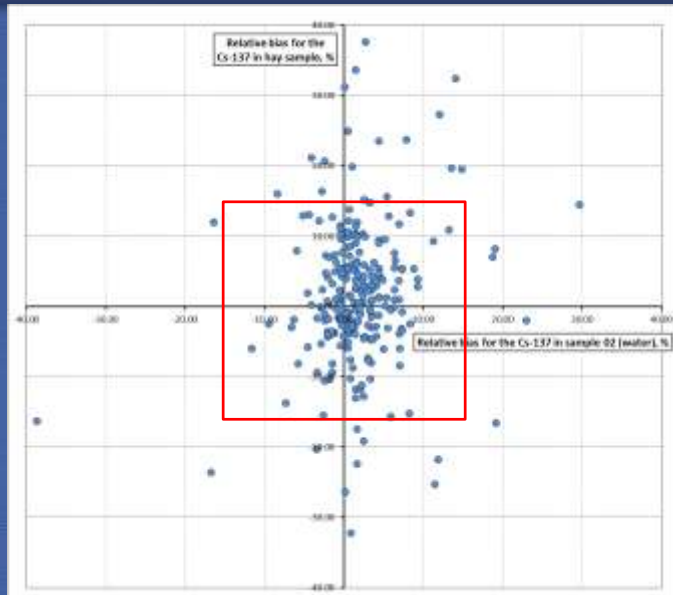
- **Bias trend analysis**
  - Master sample, master analytes (Cs-134, Cs-137)
    - Master sample: water
    - Slave samples: biota, soil
- Rules
  - Same radio-analytical method
  - Similar activity level
  - The activity should be five times higher than the MDA value (supposing standard laboratory circumstances, ISO 11929)
  - Data presentation on the X-Y chart: slave sample versus master sample



## Bias trend analysis (Cs-137 water-hay)

World-wide  
2012

245 participants



## Evaluation of Z-scores

The Z-scores were derived by the following equation:

$$Z = \frac{Value_{Reported} - Value_{Target}}{s^*}$$

Where  $s^*$  the robust standard deviation without refinement, calculated by the following formula:

$$s^* = 1.483 \cdot \text{median of } |Value_{Reported} - Value_{Target}|$$

It should be emphasized the Z-score is a relative parameter, because the value of the robust standard deviation used for the calculation is derived from the reported results influenced by the performance of the participants.



## The PT samples and analytes in

- Water samples
  - Sample 01 spiked tap water
  - Sample 02 spiked tap water
  - Sample 03 (QC) spiked deionised water

(The assigned values of the radionuclides was specified in the cover letter)
- Biota or Food sample
  - Sample 04 (for anthropogenic isotopes)
    - hay
    - flour
    - seaweed
    - rice
- Mineral matrices
  - Soil, sediment, ores



## Sample preparation

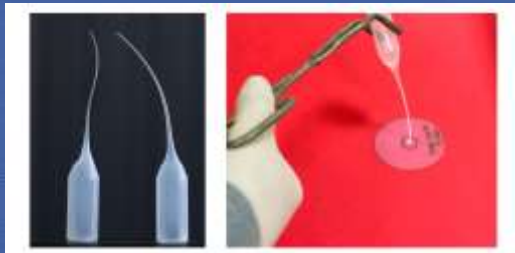
- Water samples
  - From SRM solutions (with low uncertainty) using gravimetric dilution steps
  - All dilution process included into the material balance and the contingent discrepancy included into the preparation uncertainty budget
  - Control measurement of the sample sets
- Spiked biota sample
  - From SRM solutions (with low uncertainty) using gravimetric dilution steps
  - Bulk homogeneity check during the preparation and final homogeneity check on the bottled samples
  - Control measurement
  - Dry weight tracking during the preparation steps



## Sample preparation

### General principles for spiked samples

- Certified high precision isotope solutions are used
- Material balance (weight tracking) during the entire preparation steps
- Establishing the traceability chain
- **Water**
  - Gravimetric dilution
  - Validation by point source preparation
  - Checking the final dilution by control measurement



## Sample preparation

### General principles for characterised samples

- Results of independent expert laboratories
- Most appropriate and robust mathematical method for deriving target values and uncertainties
- Establishing the traceability chain
- Homogeneity check using ANOVA pattern

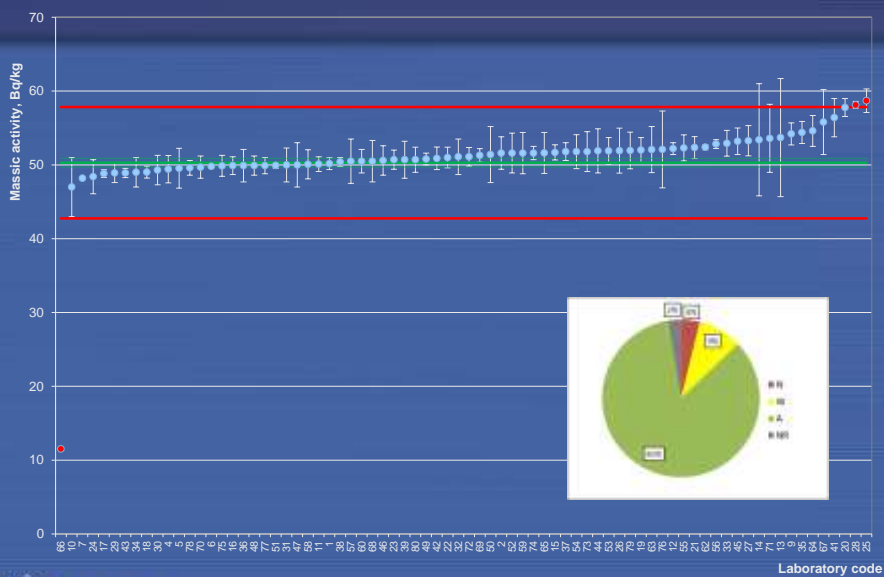


# Results

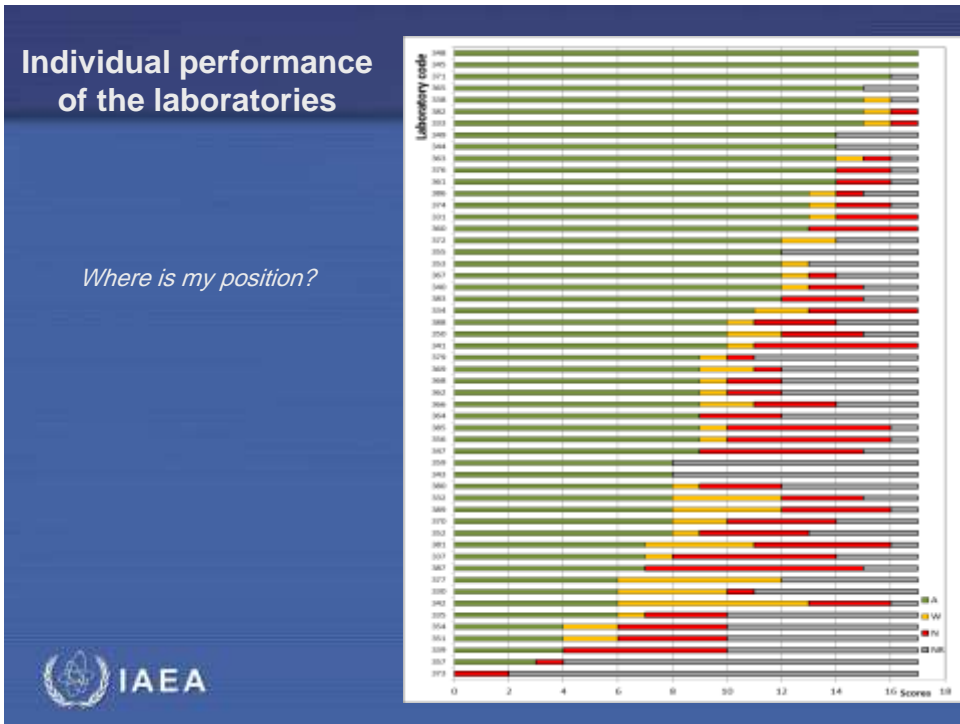
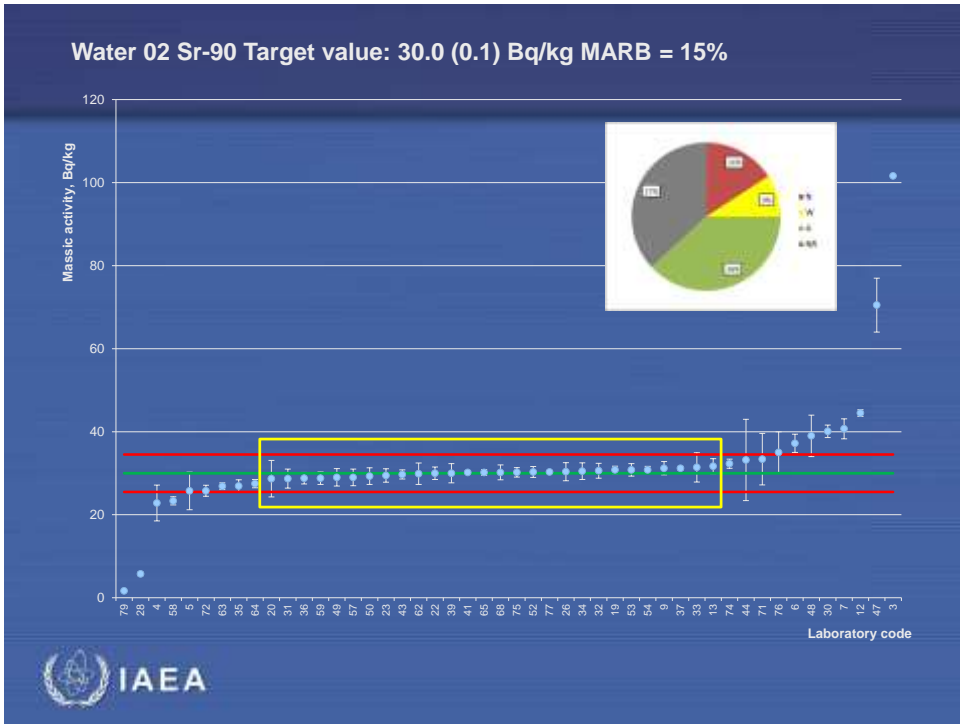
(as the participants want to see)



Water 01 Cs-137 Target value: 50.3 (0.3) Bq/kg MARB = 15% in 2013





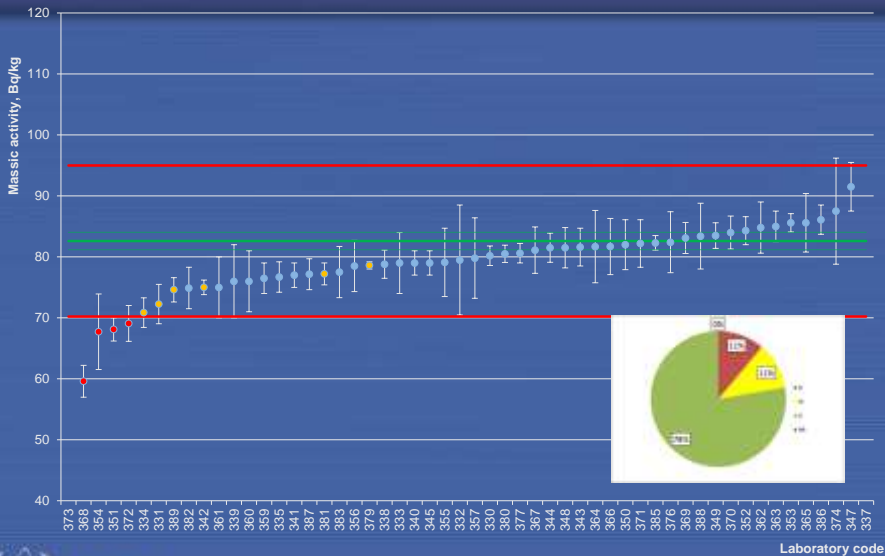


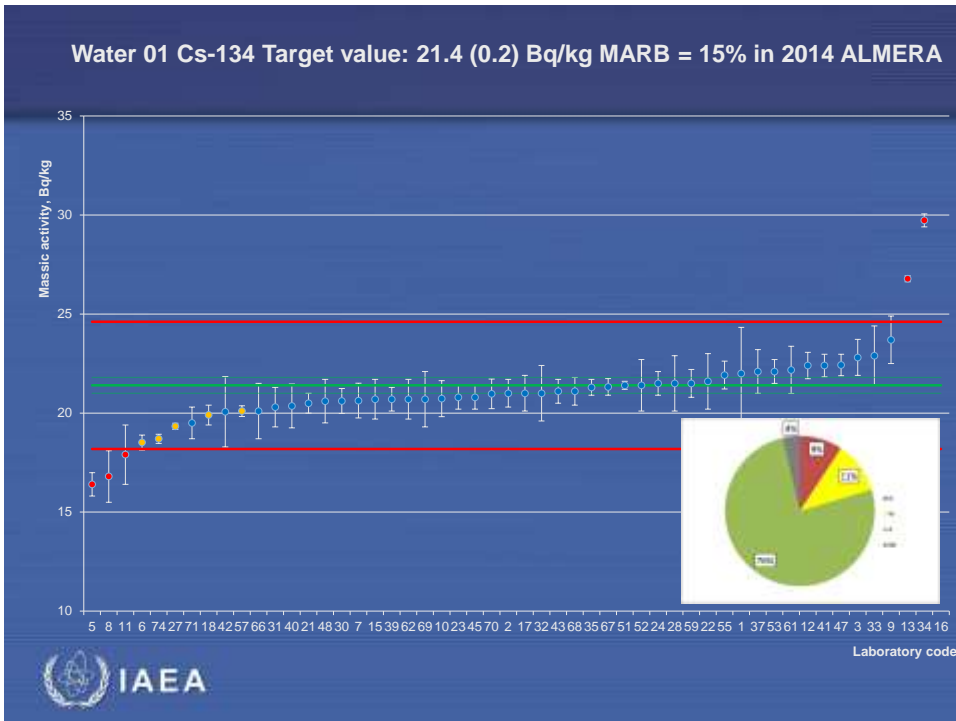
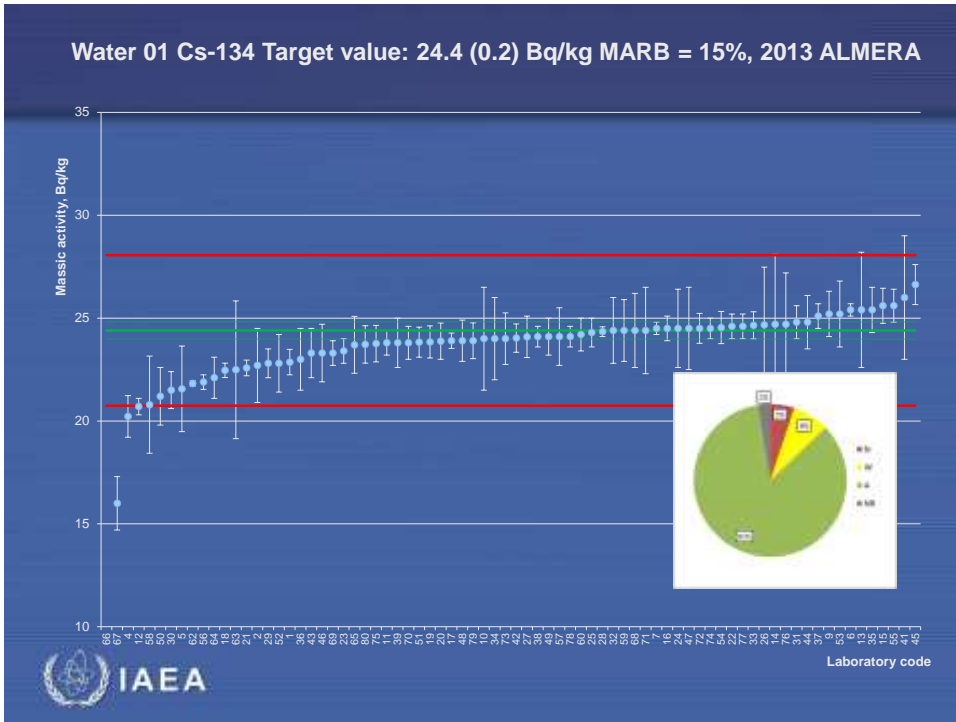
# Data interpretation

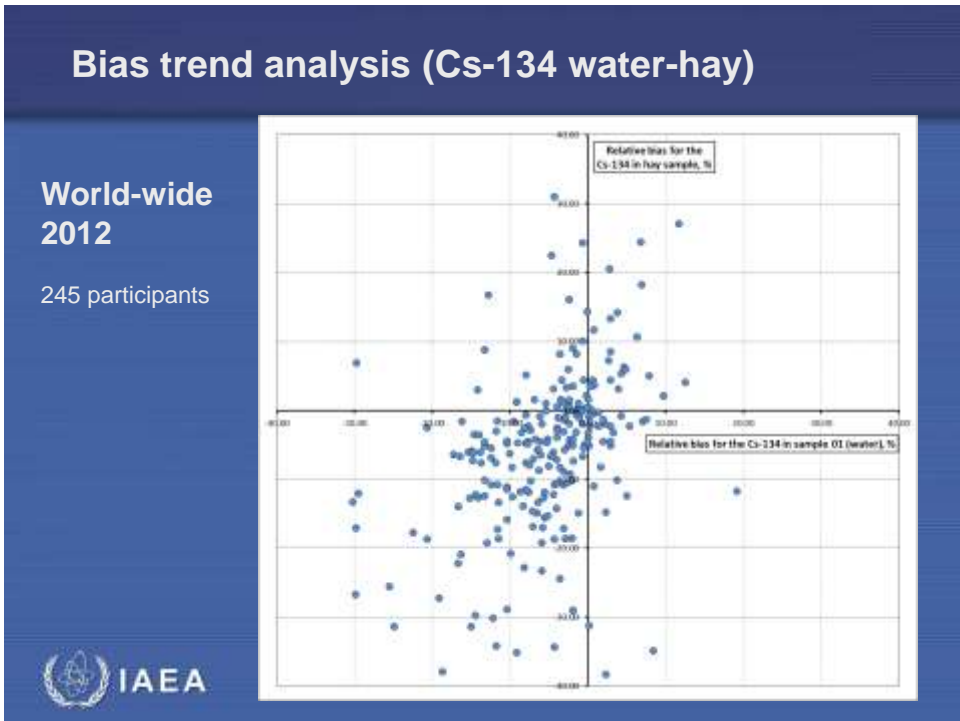
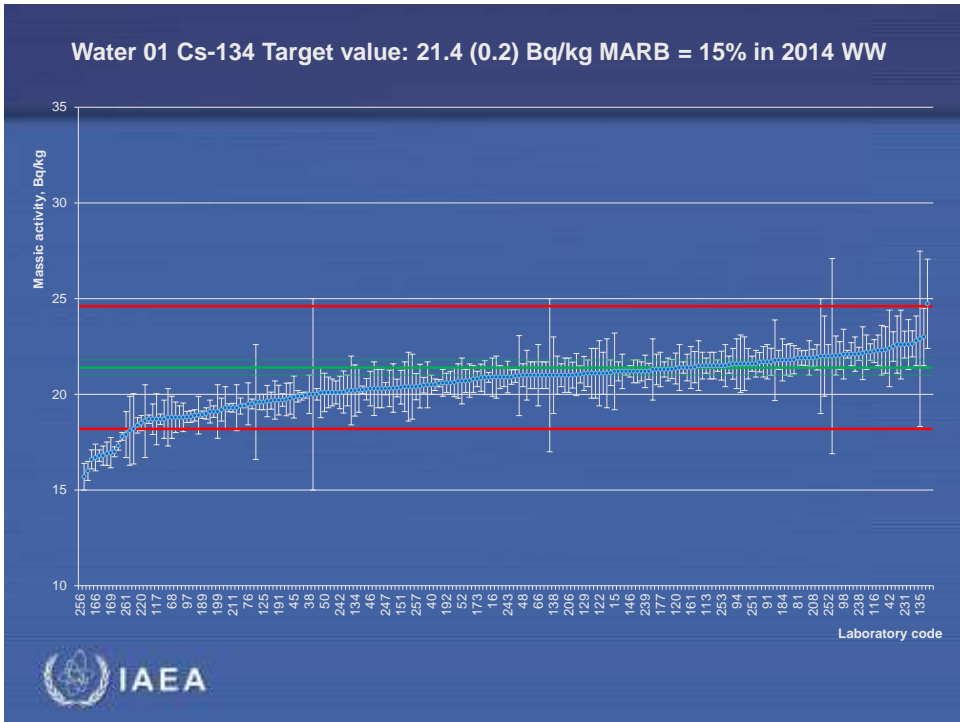
(different case studies, othehand waht the organiser want to see)



Water 01 Cs-134 in target value: 82,6 (0,7) Bq/kg MARB=15%, 2012 ALMERA



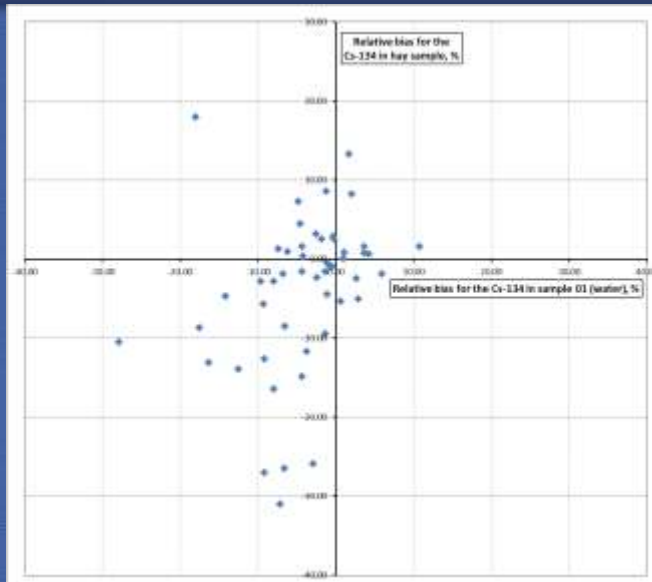




## Bias trend analysis (Cs-134 water-hay)

ALMERA  
2012

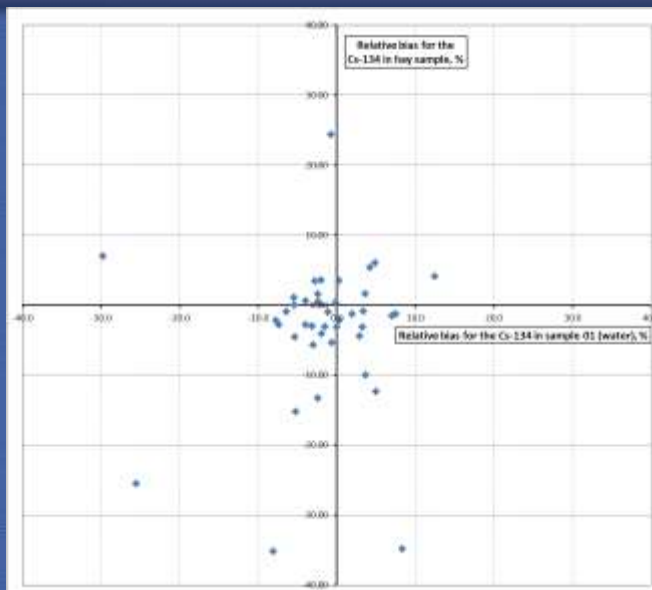
52 participants



## Bias trend analysis (Cs-134 water-hay)

Japan  
2012

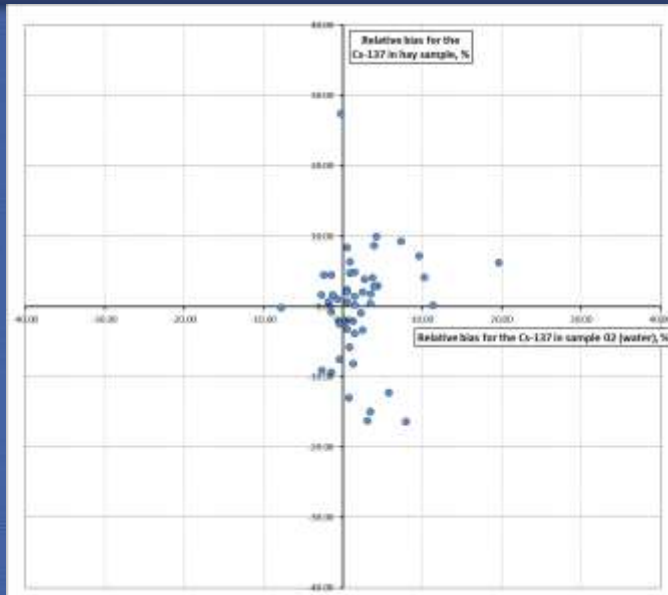
47 participants



## Bias trend analysis (Cs-137 water-hay)

ALMERA  
2012

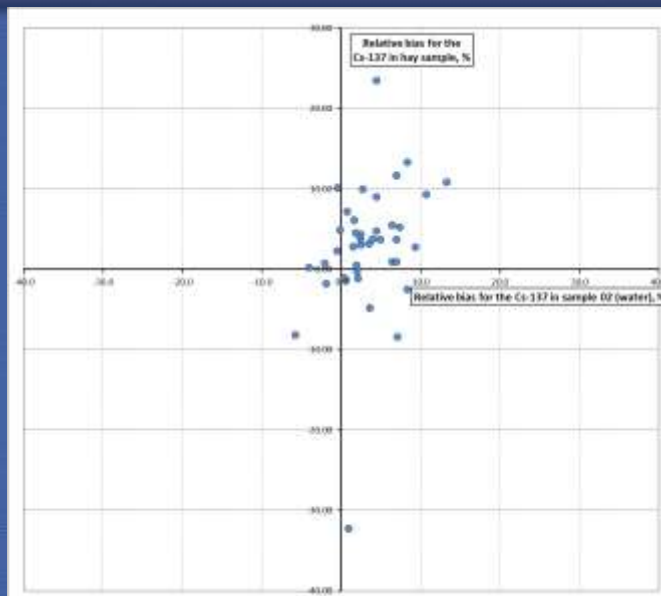
52 participants



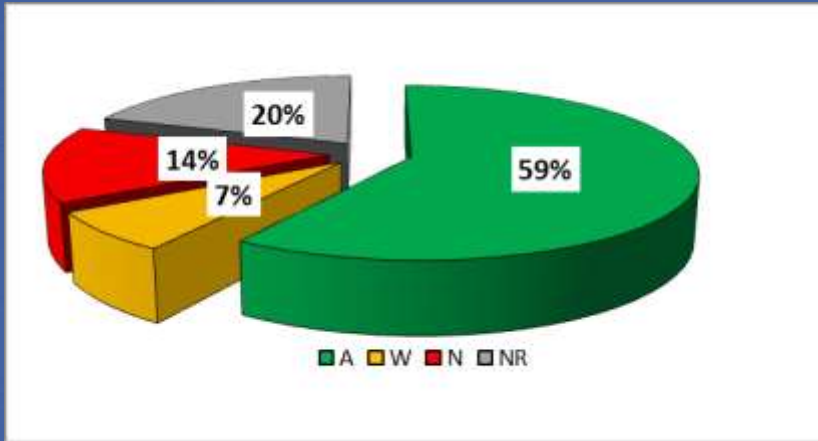
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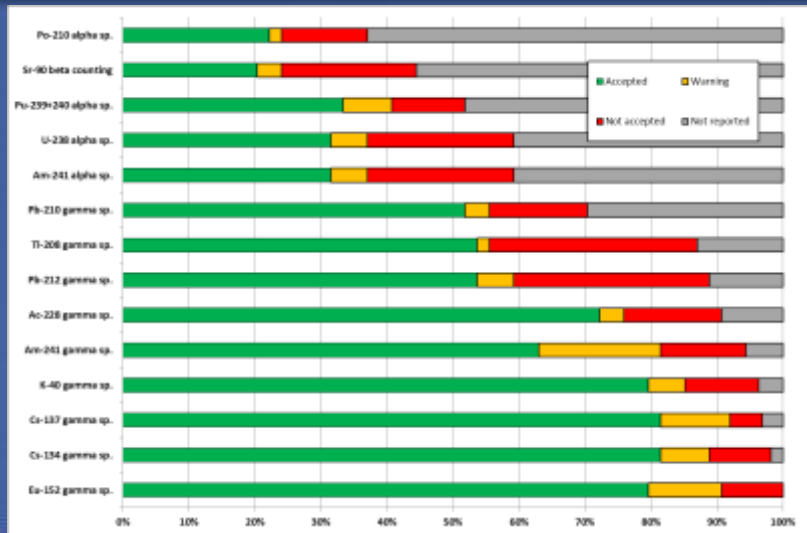
47 participants



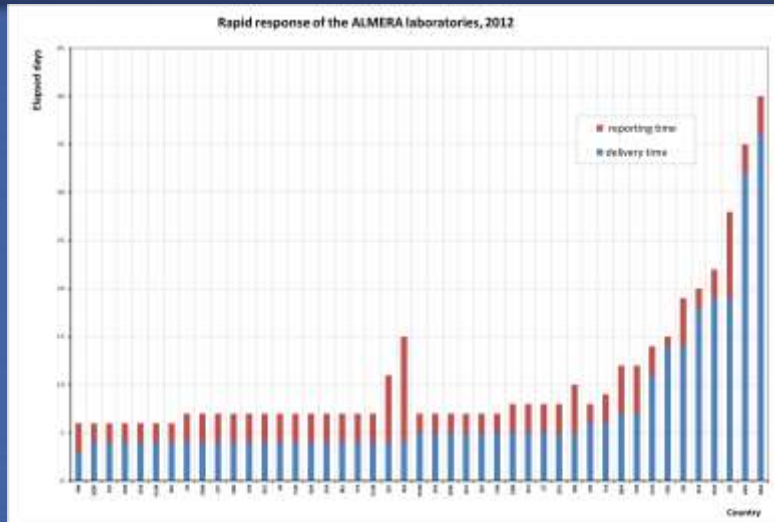
## Overall performance including all analytes



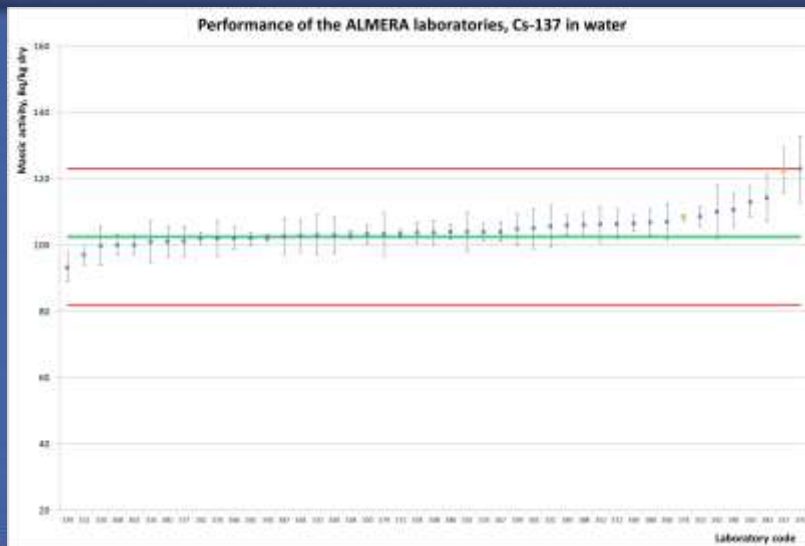
## Gap analysis



## Response time



## Performance due to the rapid reporting





## Conclusions, experiences

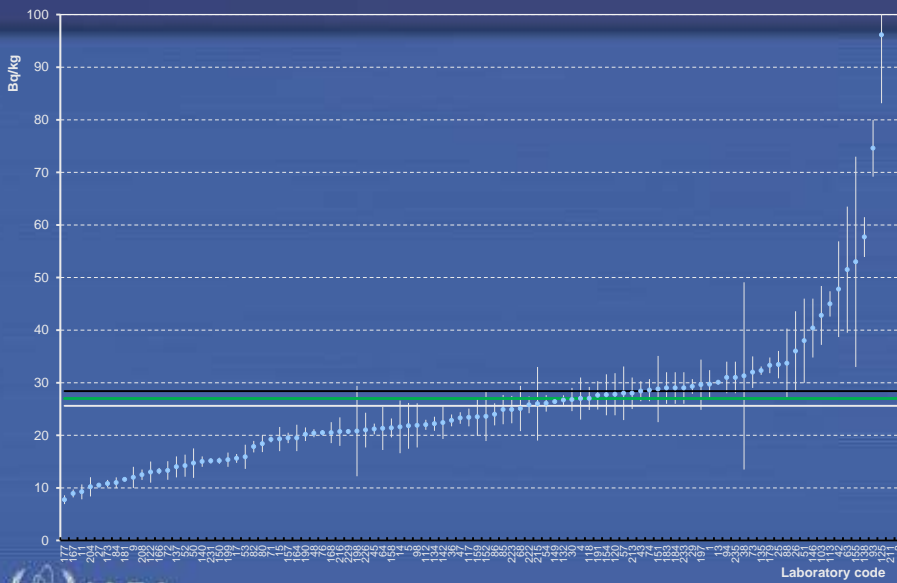
- The laboratories pay more attention for the perfect analysis and corrections if they participating in several consecutive PT
  - true coincidence summing effect
  - self attenuation of the sample
  - dry content (moisture content)
- The proper uncertainty estimation still a challenge
- There is a significant difference in the performance between the ALMERA and WW group
- The BIAS trend analysis is a powerful tool to demonstrate the significant analytical difficulties
- The method related performance evaluation definitely shows the areas to be improved



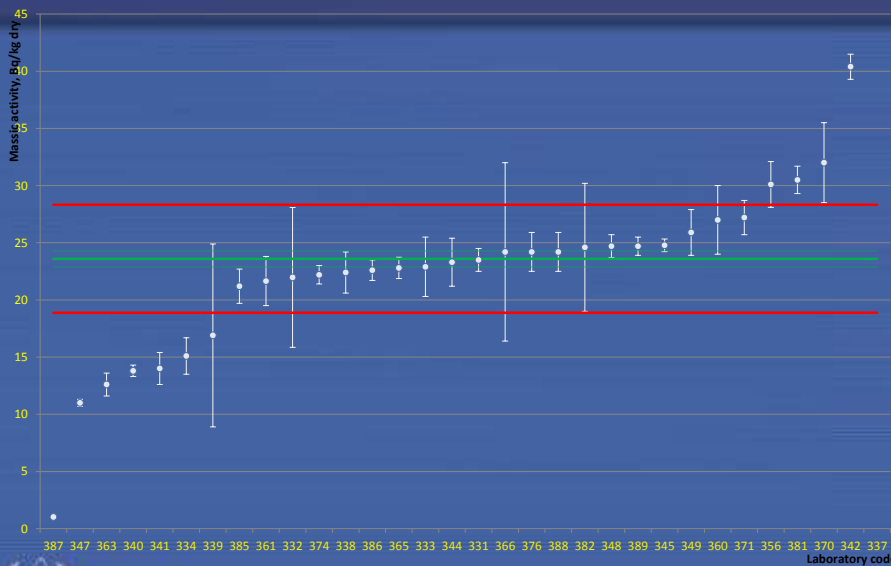
Thank you  
for your  
attention!



## U in soil 2011

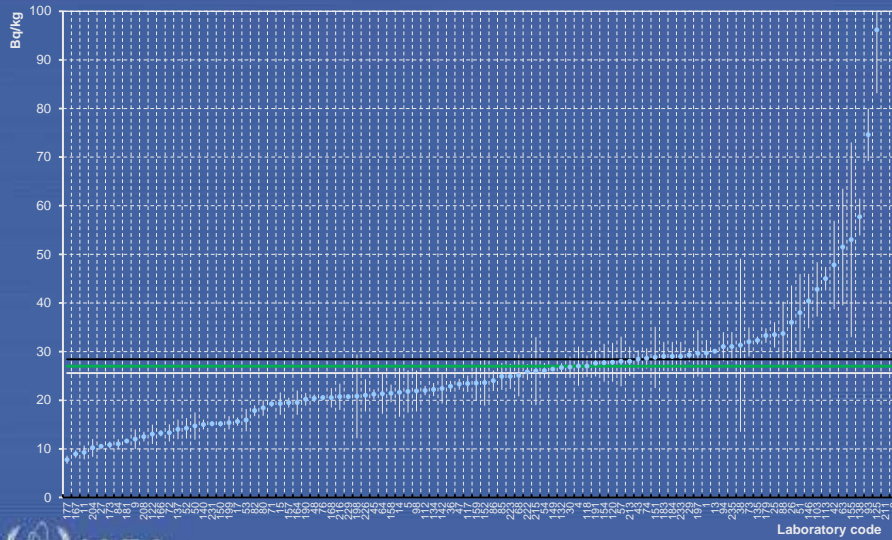


## U in soil 2012



# U in soil 2011 WW

Sample-04 Soil, <sup>238</sup>U



Laboratory code