Internal quality control of the measurement of heavy metals in organic soil improvers and urban sewage sludges

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To develop a strategy for the internal quality control of the determination of aqua regia extractable elements, Zn, Pb, Cu, Cr, Cd and Ni, in organic soil improvers and urban sewage sludges following EN 13650:2001 and ISO 22036:2008 standards.

Objective

1. Calibration
   a. Calibration curve check:
      - Criterion: slop of the regression line is significant at 0.01 (by F test)
   b. Analysis of standard solutions (STD) of heavy metal for controlling ICP-AES performance:
      - Criterion: standard deviation of replicate measurement of each concentration level is not greater than 10% of the corresponding concentration level.

2. Reproducibility
   - Criterion: relative standard deviation of each concentration level is not greater than 10% of the corresponding concentration level.

3. Measurement Uncertainty
   - Criterion: the expanded uncertainty (k = 2) calculated for each concentration level is not greater than 20% of the corresponding concentration level.

Measurement Procedure Validation

- Validation of the linear regression model
- Limit of quantification
- Sensitivity
- Evaluation of the measurement uncertainty
- Evaluation of the intercomparison uncertainty
- Evaluation of the intermediate precision

Evaluation of the Measurement Uncertainty

- Limit of Quantification (LoQ)
  - The limit of quantification was estimated through the analysis of blanks in intermediate precision conditions and by using regression model parameter from a calibration curve built from signals collected in different days, Equation 1.4 and 1.5 respectively.

- Intermediate Precision
  - For the determination of measurement intermediate precision, 3 samples of reference materials were analysed in 3 weeks, equation 1.6

- Trueness
  - The trueness was assessed by comparing known and measured concentration of 6 reference materials. Then, the analyte recovery is estimated through equation 1.7.

- Internal Quality Control
  - To develop a strategy for the internal quality control of the determination of aqua regia extractable elements, Zn, Pb, Cu, Cr, Cd and Ni, in organic soil improvers and urban sewage sludges following EN 13650:2001 and ISO 22036:2008 standards.

Results

- Limit of Quantification (LoQ)
  - The limit of quantification was estimated through the analysis of blanks in intermediate precision conditions and by using regression model parameter from a calibration curve built from signals collected in different days, Equation 1.4 and 1.5 respectively.

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Conclusion

The developed quality control strategy allows identifying the cause of deviations in measurement results (x contamination or ICP-AES linearity response deviations) and involves checking the quality of the estimated measurement uncertainty.

The sensitivity of ICP-AES is checked through the analysis of a standard solution with a concentration near the LoQ.