Interlaboratory comparison tests as a tool for quality improvement in fertilizer analysis methods and laboratory performance

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In 2008, a yearly European wide interlaboratory comparison (ILC) program for fertilizers ("VDLUFA EU Fertilizer Ring Test") was established by the Association of German Agricultural Analytic and Research Institutes (VDLUFA) via its section 'fertilizer analysis'. The aims of this program are to (1) continuously review the reproducibility of lab test results, (2) to ensure quality assurance for laboratories offering fertilizer analysis and (3) to validate and (if proven necessary) revise analytical methods.

This ILC specially addresses laboratories notified by the EU Commission for fertilizer analysis, but is open for all laboratories offering fertilizer analysis worldwide. In the past years, up to 48 laboratories from 21 different countries participated. Based on EU fertilizer regulation methods (EC No 2003/2003) or other standardized methods (CEN, ISO) different analytes have to be determined depending on the fertilizer type. The statistical evaluation of the results from the participating labs is carried out using the software package 'ProLab' (QuoData GmbH, Dresden, Germany). Based on IUPAC guidelines, a robust method is applied (DIN 38402 A45, Q-method, Hampel estimate) and Zu-scores (tolerance limit |Zu| = 2.0) are calculated as a bias estimate to enable participating laboratories to evaluate their analytical performance.

In the recent ILC conducted in 2016, a magnesium calcium carbonate ("dolomitic limestone") fertilizer was sent to the labs and 13 different analytes had to be determined. Comparing the results for total Mg content for the participating laboratories reveals that equal Mg concentrations were detected using the official EU-methods based on complexometry or flame AAS (overall mean 10.87 % Mg) compared to an ICP-OES based method (overall mean 10.75 % Mg) with standard deviations of reproducibility of 0.37 (EU method) and 0.48 (ICP-OES). Based on these results that prove a successful validation of the ICP-OES method, which is an established procedure for Mg analysis in many laboratories all over Europe, a discussion on establishing this analytical procedure as new international standard can be initiated.

Suggestions for modifications of the analytical procedures resulting from this ILC are directly communicated to and discussed with German (DIN), European (CEN) and

international standardization organizations (ISO). Furthermore, fertilizer material from this program is offered as 'non-certified but validated' reference fertilizer material for daily lab quality assuring routines. Due to upcoming changes in EU regulations, organic and organomineral fertilizers will be included in forthcoming ILC organized by VDLUFA.