INTERNATIONAL VALIDATION STUDY OF A NIRS SCREENING METHOD

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Near infrared reflectance spectroscopy (NIRS) is a very fast non-destructive and environmentally friendly analytical technique. This method has proved to be very effective for a simultaneous determination of some soil constituents in soils, with practically no sample pre-treatment. Calibration equations reflect the relationship between the constituents of the sample as determined by a suitable reference method and the NIRS spectral information. The Central Institute for Supervising and Testing in Agriculture (UKZUZ) has developed and optimized the NIRS method for determining oxidizable carbon (Cox), total carbon (Ctot) and total nitrogen (Ntot) in soils and prepared this method for international standardization in ISO TC 190 Soil quality.

The main task in the validation study was to avoid as much as possible the influence of the variability of the reference methods and to determine only the NIRS variability. Therefore, not only samples for a validation study, but also samples for the NIRS calibration were send to the participants. The calibration samples were provided with the results received by reference methods in one laboratory. Soil samples were prepared for a direct measurement. Two calibration sample sets (60 soil samples from arable land and grasslands and 60 forest soil samples) were selected to obtain an evenly distributed coverage of the basic soil properties and most of the possible spectral variability. Six soil samples were selected for the validation study. Mineral soils with lower content of organic matter (three samples) and soils with higher content of organic matter (three samples). All soil samples were air dried and sieved (<2mm fraction). Finally, 126 boxes with soil samples were shipped to each participant.

The participants prepared their own calibrations for both concentration ranges by using the two calibration sample sets and the results from the reference methods provided by the organizer. Then they determined the measurands in the six validation samples. The NIRS measurement method, calibration procedure and instrument conditions were flexible.

Ten independent result data sets from nine participating laboratories from five countries were reported. The data were obtained from five different types of instruments.

There were very few outliers reported by the participants. Repeatability was very good in both concentration ranges. Reproducibility of the method was fairly good for higher concentrations of the measurand and acceptable also for low concentrations. The number of samples in the calibration set was found to be acceptable for a screening method. Nevertheless the results could be improved if more samples were used for calibration of the instruments.

The results of the validation study were accepted by ISO TC 190, they were included into the text of the standard and the final standard was published (ISO 17184:2014, Soil quality – Determination of carbon and nitrogen by near-infrared spectrometry).