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Recent Experiences with NIRS-Analysis of Rapeseed

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SUMMARY

According to the findings presented here, NIRS-analysis in a network is well suited to complete the official method in those applications where accurate and fast analysis of several quality parameters of rapeseed is required. Additionally the integration of new instruments into the network can easily be done by the standardisation procedure.

With the setup of a network of NIRS-instruments the management of the same becomes important. The network manager will be the person in charge to cope with problems arising in connection with rapeseed analysis at all sites. All validation procedures should be governed centrally on a regular basis. A single reference laboratory accepted by all network participants has to be determined and controlled. Networks of more than 100 NIRS-instruments (e.g. BÜCHMANN, 1995) show the potential of NIRS-analysis in a network.

The first use of near infrared spectroscopy (NIRS) to analyse oilseeds dates back to the work of BEN-GERA AND NORRIS (1968). Another early work on ground oilseed samples was published by HYMOWITZ *et al.* (1974). An important step towards practicability and speed of analysis was the work on whole seed analysis by TKACHUK (1981). With his results sample preparation could be reduced to a minimum.

In 1984 parts of the German seed industry and research institutes started a joint project to utilize NIRS-analysis for quality control of rapeseed in Germany. The aim was to establish a technique that was able to give an accurate analysis of the quality in the short period between harvest and sawing of rapeseed at a moderate price. The ability of an undestructive analysis of several quality parameters simultaneously is of great importance for use in the seed industry. E.g. the work of KÖSTER (1989) and REINHARDT (1992) resulted in the establishment of NIRS-analysis at almost any rapeseed breeding companies in Germany.

In NIRS-analysis it is generally accepted that in order to get most accurate and reproducible results in a number of laboratories it is useful to connect all NIRS-instruments involved to a network and manage them centrally. Another important advantage of such a network is the possibility of quick know-how transfer to all sites and an enormous saving of cost and labor to calibrate additional instruments. Such a network of NIRS-instruments is based on a calibration which is developed on the master instrument and then used on all the other satellite instruments in the network.

In a project initiated by the Union for the Furtherance of Oil- and Protein plants (UFOP), Bonn, the feasability of such a centrally managed network for accurate quality assessment was investigated and a collaborative trial was done. In a preliminary study

several strategies to build a network of NIRS-instruments were analysed. Because of the results of other studies and the spread of instruments in Germany this study focused on NIRS-instruments working in reflectance mode.

Strategies to get accurate results from several NIRS-instruments

In most cases, NIRS-analysis is conducted in a site specific way, i.e. calibrations are developed individually at each site often using a local reference laboratory. This procedure may be satisfying where e.g. a breeder desires a relative ranking of his materials. But differences in NIRS-analyses between laboratories are often determined by several factors and can seldom be resolved satisfactorily. The next step towards reproducible NIRS-analyses is the use of a uniform calibration set; the corresponding calibration of each instrument specifically has been demonstrated by REINHARDT (1992) and REINHARDT AND TILLMANN (1993) for rapeseed analysis. In this way differences between reference laboratories – the most important ones – can be minimised as can be differences in the representativity of the calibration set.

A further step to obtain reproducible results from all NIRS-instruments is the use of the same calibration on all instruments (Figure 1). Then instrument specific differences have to accounted for which was usually done by *bias* correction (OSBORNE AND FEARN, 1983). A newer approach is the standardisation of instruments as proposed by SHENK *et al.* (1985) which ahs the advantage of using the outlier detection on the satellite instruments and the ease of integrating additional instruments into the network.

In the own work (TILLMANN, 1997) several methods of building a network of NIRS-instruments were investigated. It turned out that instruments of one brand could easily be connected by a single sample standardisation with the help of a repeatability file.

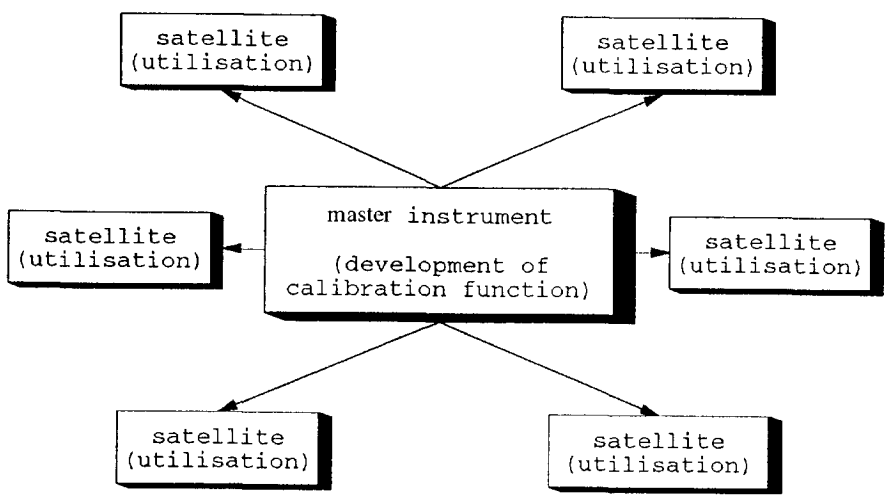

Figure 1: Network of NIRS-instruments using a single calibration function

Collaborative trial of NIRS-analysis

In a next step of the project NIRS-analysis of rapeseed was tested in an international collaborative trial using the official methods of oil and glucosinolate determination as a reference. From three differnt countries 29 laboratories participated, participation was voluntary, so that the distribution to the different methods was rather unequal.

The "repeatability standard deviation" (σ_r) describes the method-specific difference between two single analyses in one laboratory. The "reproducibility standard deviation" (σ_R) describes the method-specific difference between two single analyses in two laboratories using the same method. Accordingly the reproducibility tells how close the results of one sample analysed in two laboratories with the same method can be.

No differences between the methods of oil determination were detected regarding the reproducibility standard deviation as weren't between the methods of glucosinolate determination regarding the repeatability standard deviation (Table 1). But the repeatability standard deviation of the widely used nuclear magnetic resonance method (NMR) was half that of NIRS-analysis for the oil determination, which was little worse than the oil determination by SOXHLET extraction.

The reproducibility standard deviation for the glucosinolate determination was lowest for the HPLC method followed by NIRS-analysis. The result for the x-ray fluorescence method (XRF) was little reliable because of the few numbers of laboratories participating with this method in the trial. All results except the reproducibility standard deviation of the XRF method compared favorably to the specifications in the appropriate international norms (ISO 5511, 1992; ISO 659, 1996; ISO 9167-1, 1992; ISO 9167-2, 1994).

Table 1: Repeatability (σ_r) and reproducibility standard deviation (σ_R) of different methods for the quality determination of rapeseed

Parameter	Scale	Range	Method	N	p	σ_r	σ_R
oil	% DM	45,36 – 50,44	SOXHLET	15–20	4–5	0,28	0,71
			NMR	35–40	9–10	0,19	0,91
			NIRS	51–52	13	0,36	0,66
GSL	µmol/g DM	7,89 – 16,70	HPLC	15–20	4–5	0,86	1,09
			XRF	11–12	3	0,77	3,37
			NIRS	50–52	13	0,83	1,96
protein	% DM	18,33 – 22,86	NIRS	51–52	13	0,28	0,40

N = number of single determinations of any sample, p = number of labs
σ_r = repeatability standard deviation, arithmetic average of 10 samples
σ_R = reproducibility standard deviation, arithmetic average of 10 samples

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