

# The use of the InfraAlyzer 260 Whole Grain in the oil industry

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## Introduction

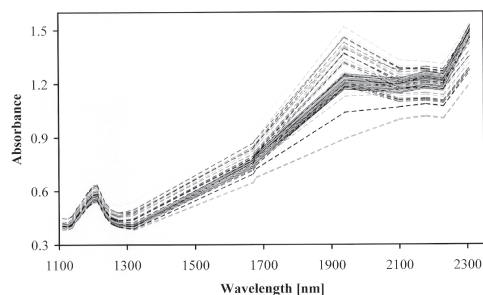
The main oil raw material is still double low rapeseed. The main area of development is perceived to be in the oil mills. These oil mills will be based on improved fullpressing and extraction technologies. The oil and water were the constituents selected for investigation on the grounds of their importance in the oils industry. The estimation of the oil and water content is normally carried out by time-consuming methods.

Reflectance properties of whole rapeseed and rapeseed oil meal are of great interest because they offer the possibility of providing the most convenient analytical procedure.<sup>1,2</sup> Such a procedure has ultimate benefits in terms of convenience, speed and cost.

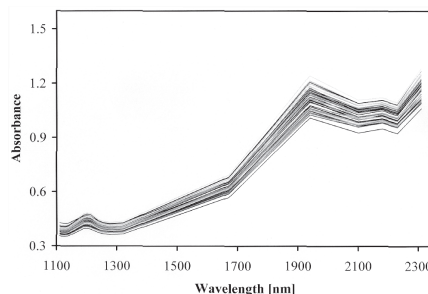
The study describes an investigation into utility of the filter near infrared (NIR) reflectance instrument—InfraAlyzer 260 Whole Grain (Bran+Luebbe GmbH, Norderstedt, Germany) for the routine analysis of the raw whole rapeseeds and their side product—the rapeseed oil meals.

## Materials and methods

The rapeseed and rapeseed oil meal samples were obtained from NZPT Co. Ltd—Brzeg. A sets of 103 samples of rapeseed and 50 samples of rapeseed oil meal were analysed for water and oil contents according to the ISO-procedures.<sup>3</sup> The selected samples were scanned with a whole grain cell in the wide-angled moving drawer using all 19 filters of an InfraAlyzer 260 Whole Grain with wavelengths between 1110 and 2310 nm. The NIR measurements of raw whole samples of rapeseed and rapeseed oil meal were carried out in the reflectance mode. The calibrations for water and oil were performed by stepwise multiple linear regression (MLR) analysis by using SESAME v. 2.10 software



**Figure 1.** NIR spectra of the whole rapeseed samples (IA 260WG).



**Figure 2.** NIR spectra of the raw rapeseed oil meal samples (IA260WG).

(Bran+Luebbe GmbH, Norderstedt, Germany) on an interfaced PC computer. Each sample was measured in three replicates and the mean of the replicate spectra obtained was used in the calibration.

Figures 1 and 2 show the NIR spectra of the rapeseed and rapeseed oil meal samples measured on an InfraAnalyzer 260 Whole Grain, respectively. These calibrations were then applied to a separate set of 30 samples of whole rapeseed and 15 samples of raw rapeseed oil meal which, for validation purposes, were also analysed by classical laboratory methods (water: drying at 105°C for 3 h; oil: extraction with petroleum ether in a Soxhlet apparatus).

Multiple linear regression (MLR) equations of raw spectral data ( $\log 1/R$ ) were evaluated on separate samples of validation sets by simple linear regression analysis of NIR predicted v. laboratory values.

## Results and discussion

Table 1 shows the characteristics of the sample sets used in the study determined by the reference methods. The ranges and mean values of the samples for calibration and validation were very close.

**Table 1. Parameters (%) of the rapeseed and rapeseed oil meal samples for the calibration and validation sets.**

Rapeseed						
Constituent	Calibration ( $n = 103$ )			Validation ( $n = 30$ )		
	Range	Mean	SD	Range	Mean	SD
Water	1.42–19.56	7.21	3.73	2.87–18.88	7.65	2.98
Oil	42.71–47.50	45.12	1.41	43.08–46.95	44.97	1.57
Rapeseed oil meal						
Constituent	Calibration ( $n = 50$ )			Validation ( $n = 15$ )		
	Range	Mean	SD	Range	Mean	SD
Water	11.01–15.63	13.36	1.11	11.45–15.09	12.87	1.24
Oil	2.12–12.41	6.50	2.76	2.97–11.76	6.33	2.54

**Table 2. Calibration statistics and wavelengths used to predict parameters of rapeseed and rapeseed oil meals samples.**

Rapeseed			
Constituent	$R$	$SEC$ (%)	Selected wavelengths (nm)
Water	0.996	0.29	1276, 1320, 1940, 2100, 2180
Oil	0.955	0.40	1128, 1238, 2180, 2310
Rapeseed oil meal			
Constituent	$R$	$SEC$ (%)	Selected wavelengths (nm)
Water	0.971	0.25	1940, 2180, 2230
Oil	0.997	0.18	1188, 1212, 2100, 2230

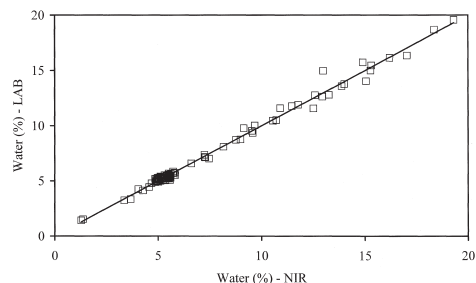


Figure 3. NIR calibration equation developed for water in whole rapeseed samples.

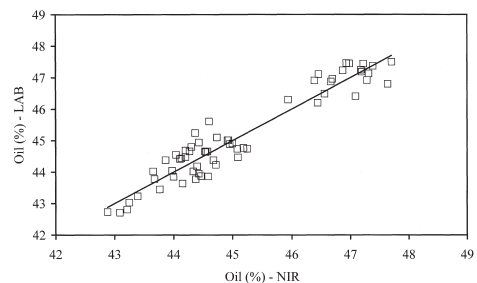


Figure 4. NIR calibration equation developed for the oil in whole rapeseed samples.

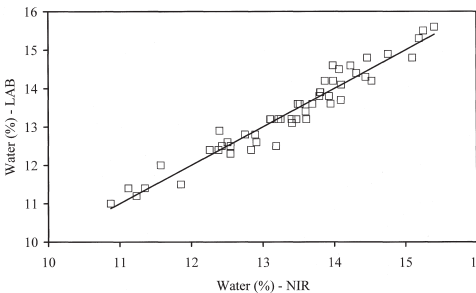


Figure 5. NIR calibration equation developed for water in raw rapeseed oil meal samples.

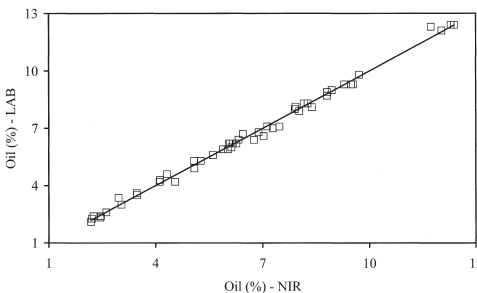


Figure 6. NIR calibration equation developed for the oil in raw rapeseed samples.

Table 3. Statistical parameters obtained in the validation of the calibration.

Rapeseed			
Constituent	<i>r</i>	<i>SEP</i> (%)	Bias (%)
Water	0.991	0.34	−0.007
Oil	0.950	0.43	0.004
Rapeseed oil meal			
Constituent	<i>r</i>	<i>SEP</i> (%)	Bias (%)
Water	0.965	0.27	−0.005
Oil	0.989	0.20	0.003

Table 2 contains the NIR results for calibrations, together with the selected wavelengths. Figures 3 and 4 show NIR calibration equations developed for water and oil in the rapeseed, Figures 5 and 6, in the rapeseed oil meal samples.

Possibly, the seed coats affected the absorptions but performance of the calibration obtained was confirmed by using prediction samples. Validation statistics from simple linear regression analysis with those predicted from NIR analysis, are shown in Table 3.

The best equation for each constituent was chosen by the optimal combination of the statistics parameters from the equation development: high multiple correlation coefficient ( $R$ ), low standard error of calibration ( $SEC$ ) and high  $F$ -values in the calibration set. The accuracy of the equations in prediction was expressed as high simple correlation coefficient ( $r$ ), low standard error of prediction ( $SEP$ ) and bias. The calibrations used in practice were obtained using three to five terms for each constituent.

## Conclusion

The results of NIR analysis on water and oil contents in raw whole rapeseed and rapeseed oil meal samples, indicate that InfraAlyzer 260 Whole Grain with a wide-angled moving drawer and whole grain cell offers possibilities for the rapid determination of these constituents. This spectrophotometer offers rapid and accurate analysis during the different manufacturing stages in the oil industry.

## Acknowledgements

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## References

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2. R. Tkachuk and F.D. Kuzina, *Can. J. Plant. Sci.* **62**, 875 (1982).
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