Application of a portable near infrared analyser for wheat measurement

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Introduction

After the large changes in the agricultural system in Hungary there was a great demand by small firms to analyse cereals, many of whom could not afford to maintain laboratories or buy expensive NIR analysers. This fact led us to develop an inexpensive, battery operated portable near infrared (NIR) analyser which does not require a laboratory environment or skilled personnel.

The main application of this analyser is the fast measurement of cereals at receiving stations, where the segregated storage of the cereals of different qualities is essential. Because the accuracy of the instrument is almost equivalent to a commercial infrared analyser, it can be used for analysing not only cereals but other solid and semi-liquid samples used in food industries worldwide.

Quality control in cereal and milling industries

Nowadays, raw materials of special quality are needed by the customers in the cereal and milling industries in Hungary. In modern production systems the planning of the quality of an end product begins in the fields—choosing the variety, agricultural techniques etc. Some firms buy the cereals in the market. In such cases, the specifications of the Hungarian National Standard are valid.[1]

The Hungarian wheat classification system is based traditionally on the knowledge of the wet gluten content, of the baking value and of the physical features. The measurement of wheat by conventional methods according to the Hungarian National Standard takes about three days.[1]

After the changes in the Hungarian agricultural system the quality of wheat varied widely. There are about 40 registered varieties in cultivation and the conditions for growing are very different. This is why a fast method is required in the harvest survey work.

After the harvesting, the separated lots are homogenized and their quality is determined by traditional methods. During this process a “quality map” is made, which is the basis of the uniform quality of end-products.

About the analyser

The developed analyser—named Mininfra-5—is a portable NIR spectrophotometer which has five interference filters in a filter wheel. The optical arrangement of the analyser has been restricted to a compact size and is field portable (Figure 1).

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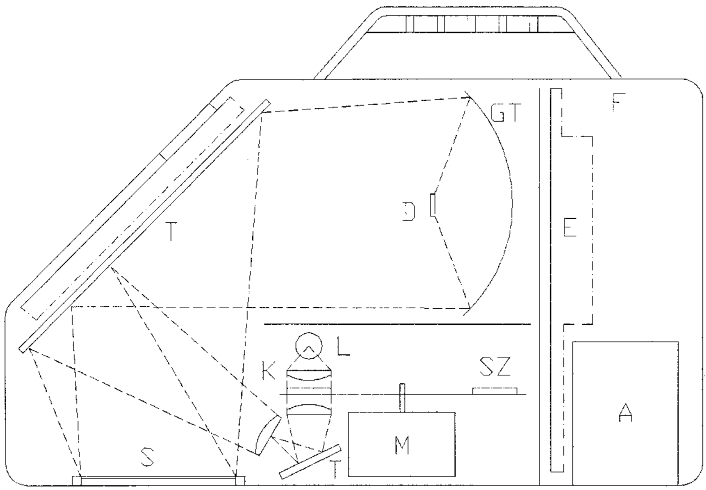

Figure 1.

The main parts of the instrument are a tungsten lamp, lenses, mirrors, a sample window (S), a detector and narrow band infrared filters. The filter wheel is rotated by a d.c. motor (M). The accumulator (A) and the electronic card (E) are located in the back part of the instrument, separated from the optical parts. In the front of the analyser there is a display with 2×16 characters and a keyboard (T) with 21 audio feed-back buttons. On the top of the instrument is a handle. The instrument has a sample holder unit which can hold a "Petri" dish filled with the sample. The analyser has to be lifted by a hand spike before the sample is put in. For transporting the analyser the sample holder may be attached to the bottom plate of the instrument.

Operation of the analyser

The analyser has five narrow band long-life NIR filters. The wavelengths of these are selected according to the absorption bands of the components to be measured. The optical system of the instrument forms a relatively enlarged light spot on the sample (diameter 70 mm) and the reflected energy is collected from this surface by the detector. The enlarged spot ensures that inhomogenous materials can also be measured with good accuracy.

Due to the special optical compensation system and the robust construction the instrument has excellent stability and insensitivity against ambient temperature changes.

Since the analyser is battery-powered and has no thermostatic parts, a built-in optical light compensation system eliminates the signal changes due to changes in the battery potential and the detector temperature. The interference filters used have a low temperature coefficient, so the wavelength shift of them is negligible in the range of 15–40°C. The measuring time is about 10 seconds and the complete measurement is microprocessor controlled.

During the measuring process three calibrations are active, that is three components of a sample can be measured simultaneously.

The first step during the measurement is the checking of the power of the battery and the temperature of the instrument. Then the microcomputer switches on the lamp and the filter motor and checks the optical signal. The measurement will start only when the measured signal becomes stable and the noise of this signal is better than a preset limit.

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Use of the instrument

Handling of the instrument is menu-driven, so it can be utilised in a very short time. There are five main menu items: MEASURE, PRODUCT, CHECK, CAL.MOD. and AUTOCAL.

After switching on the Mininfra, the software enters the MEASURE menu. To make measurements the user simply has to press the "MEAS" button only. The memory stores results of up to 100 measurements and these may be accessed later. After finishing a measurement, the results can be transferred to a printer or a computer via a RS232 line. Using the data stored, elementary statistics, average and standard deviation, can be measured and calculated.

In the PRODUCT menu the user can reach all calibration information. The instrument can store up to 16 different calibrations. The CHECK menu serves to read out measured optical data. The CAL.MOD. and the AUTOCAL menus serve to calibrate the instrument. The AUTOCAL menu holds complete multiple linear regression software to provide an automatic self-calibration function. With this the user can make calibrations himself. For this a sufficient number of known samples are required.

The calibration program can handle up to 100 measurements and calculates the calibration coefficients for up to five wavelengths. The user can switch on or off both measurements and wavelengths. After processing, the multiple correlation coefficient (R) and the standard error of calibration (SEC) will be displayed.

The program may run in automatic or manual mode. In the automatic mode, the outlier samples and the non-significant wavelengths will be erased. During the calibration a product identification matrix will be calculated on the basis of "Mahanalobis" distances. This matrix is used after the calibration for filtering samples not matching the calibration, that is to distinguish interpolation and extrapolation measurements.

Service functions

The instrument has many additional built-in service functions to provide easy use. One part informs the user about the status of the instrument. The most important of these is the power of the battery. The software checks the battery potential before every measurement and does not allow it to make measurements if the potential is under a certain limit. (In this case the battery must be changed or a charger has to be attached).

The instrument can measure more than 200 samples with a fully charged battery. Using the line charger the number of measurements is unlimited. Additional service functions are:

- sending of the results stored in the memory to a printer
- entry of date
- entry of a sample identification number
- language is optional, presently English, Dutch, or Hungarian are selectable.

Application of the instrument for measuring protein-, gluten- and the moisture-content of wheat

Sample preparation

We recommend the use of the QC-124 hammer type grinder with 1.3 mm sieve size for sample preparation of wheat.

The accuracy of the measurements is slightly better using a smaller sieve size, but moisture content diminishes due to the warm-up of the sample caused by the longer grinding time. Using the sieve size of 1.3 mm, and if the moisture content is <16%, the moisture loss is negligible.

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Filling the sample holder

A plastic "Petri" dish (diameter of 90 mm and height of 14 mm) is used as the sample holder for the instrument. After filling it with the ground sample, the surface of the sample has to be smoothed with a ruler to the exact level of the dish. A cover is not used on the sample holder.

The measuring result is influenced by the distance between the sample-surface and the optical window, therefore the same type of sample holder is needed both during calibration and measurement. The sample holder has to be always fully filled.

Calibration

Calibrations of the instrument are typically made by 70–80 samples and they are controlled by at least 20 additional samples. In selecting the samples, it is essential that the practical ranges have to be covered by their protein-, gluten- and moisture-content, and they should represent the usable kinds of wheat. Moreover, the selected samples which have about the same protein-, gluten- and moisture contents, should differ in baking quality. The samples are selected from the "wheat bank" of the Research and Development of the Flour Milling Company Ltd, which contains hundreds of samples, and they represent the diversity of wheat cultivated by small and big farms in Hungary today.

The laboratory analyses of the protein-, gluten- and moisture-content of samples are made according to the Hungarian National Standards.

The five term calibration equations are calculated by the built-in multiple linear regression program of Mininfra. Sometimes, in the case of the calibration for gluten, we use a more sophisticated program after sending the measured optical values from Mininfra to a PC.

Accuracy

The typical accuracies of Mininfra calibrations for measuring the protein-, gluten- and moisture-content of flour and ground wheat are presented in Table 1.

Unfortunately, the accuracy of a NIR gluten calibration is always worse than the protein calibration, because:

- The object of the wet gluten measurement is the flour made by a laboratory mill, moreover the NIR instrument measures the whole ground wheat. The traditional gluten result does not retain the removed parts of wheat (bran), but during the NIR measurement they are present.

Table 1.

Product/Component	Protein	Wet gluten	Moisture
Flour			
SEC	0.15	0.9	0.12
Range %	10–15	19–41	12–16
Wheat			
SEC	0.22	1.4	0.15
Range %	9–16	18–42	10–16

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- Before the wet gluten measurement, the wheat must be conditioned to a constant moisture level. The original wheat sample, the object of the NIR measurement, may have a very different moisture content. This can cause a non-linearity effect during the calibration.

Because of the circumstances mentioned above, the accuracy of the calibrations (e.g. the correlation coefficient, *R*) of the gluten content is always not as good as the protein calibration's accuracy based on the same series of samples. In practice however, the gluten calibrations with accuracy of *SEC* < 2.0 are applicable, because the main goal is the differentiation of parcels of different quality with the aim of separate stocking. Generally the parcels are classified by their measurements into 3–4 classes, for which the accuracy of *SEC* < 2.0 is suitable. Considering that in a buying station a lot of small producers can arrive at the same time, the measurement for the separate stocking may only be performed by a fast method. For this purpose the Mininfra instrument is widely found throughout many areas in Hungary.

Calibration by a computer

The instrument, using the built-in serial interface, can be calibrated by a computer as well. It can be necessary in the cases of some complex components e.g. gluten. In Figure 2 can be seen the results measured by the Mininfra and the results calculated by a MLR program used by us in case of gluten calibration of wheat. In the bottom right corner of this figure can be seen the *SEC* change in function of the number of samples. The diagram is started at $N = 7$ sample number - this is the minimal number of samples for running the MLR program in case of five wavelengths. The *SEC* is calculated at every new measurement, consequently the *SEC* is calculated first in case of $N = 7$ then $N = 8$, 9... up to the last number of samples. From the formation of the *SEC*, conclusions can be drawn about the quality of the calibration. It is noticeable that the *SEC* is increasing to about $N = 40$, after this it becomes stable and at the end decreases slightly. The calibration is acceptable if an addition of a new sample does not cause significant change in the formation of the *SEC*.

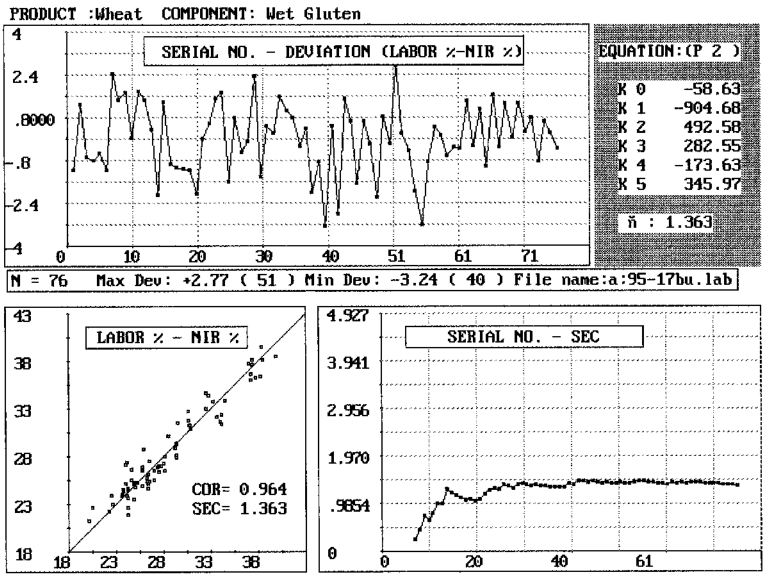

Figure 2.

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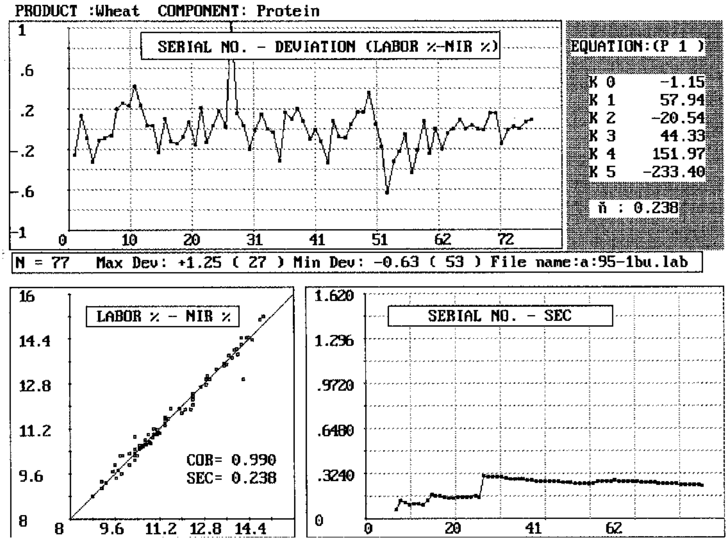

Figure 3.

In the bottom left of the diagram we have represented the results measured by the conventional method and the calculated NIR results by the calibration equation. In the upper diagram, can be seen the differences between labor % and NIR % in the case of every measurement. In Figure 3 we have represented the results of a protein calibration. In the diagram an outlier sample can be seen (serial number 27), the effect of this is a skip on the Serial N°—*SEC* diagram by $N = 27$.

A program built into the Mininfra detects the outlier measurements and they will be left based on the so-called "Outlier T-test". The "Outlier T-test" is calculated from the "Cook distances" from which a decision can be made upon whether a sample causes significant changes in calibration coefficients or not.

Foreign sample detection

For filtering defective measurements (e.g. in case of a not appropriately ground sample) the Mininfra contains a sample identification function based on calculation of "Mahanalobis distances". The calculation and storage related to this function is performed automatically in the AUTOCAL function. The effectiveness of identification depends greatly upon the range of the optical signal's changes. In case of calibrations made for a narrow range the threshold value is smaller, that is the identification is more rigorous.

Influence of season

The influence of the season cannot be disregarded in the case of gluten and protein calibrations. The calibrations made by the samples of one season should be updated using 30–40 samples of the new harvest. Although the coefficients of the calibration equations change greatly during updating, the accuracies of the calibrations do not increase after the modification. The common calibrations based on the samples of more seasons eliminate the influence of season.

Reference

1. MSZ 6383: Wheat, Hungarian National Standard (1979).

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