Portable near infrared transmission analyser for qualification of grains and flour

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Introduction

Making a portable near infrared analyser has been disregarded by major instrument manufacturers up to now. The possible reason for this is that even if these systems should be sold on a low price, they are expected to fulfil the high accuracy requirements of the customers. A few years ago we reported our fixed-filter type portable near-infrared reflectance analyser.[1] This device - named Mininfra-5 – is widely used today in Hungary and the neighbouring countries by grain receiving stations and by grain traders. The success of the instrument is due to its easy handling, good accuracy and moderate price. To enjoy these benefits the customers are supposed to accept the inconvenience of grinding. To avoid grinding made us to develop a new portable whole grain analyser, which can measure flour too.

Expectations for a portable whole grain analyser

A portable whole grain analyser should have to fit to the following demands:

- - Low weight
- - Low power consumption
- - Fast measuring time
- - No warm-up time
- - Wide ambient and sample temperature tolerance (at least between 10-40 C^0)
- - Good accuracy (better than 0, 3 SEP for wheat protein)
- - Good repeatability (better than 0, 1 SD for wheat protein)
- - Simple handling
- - Low price

Optical arrangement of the analyser

The optical system of our new analyser (Mininfra Scan-T) – consists of three parts:

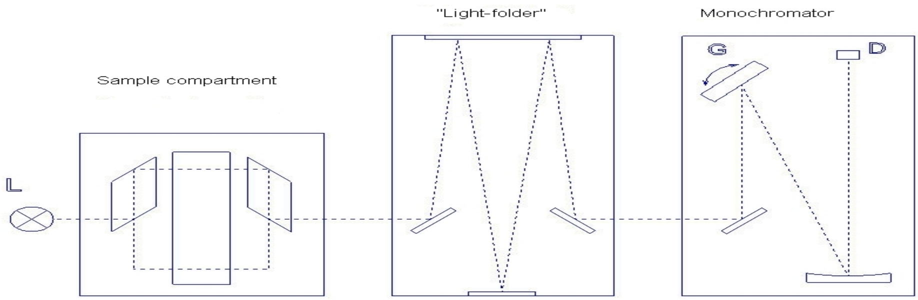

Figure 1. Optical layout of Mininfra Scan-T.

1. Sample compartment

Because of the different and inhomogeneous character of seeds the grain samples have to be measured on large surface. There are two ways: applying a large sensing size, or measuring and averaging in more position of the sample. The general solution is to move the sample in the light path. This can be done by pouring the sample into a shaft or to move it in a sample holder. Both solutions need complicated and expensive mechanical construction. We are using a unique and unusual, patented solution for sample presentation: A 20 mm diameter light spot turns quickly along a circle performing the scanning of the sample. During this scanning process the transmitted light intensity is measured at 8 or 16 (depending on the type of the sample holder) equidistant positions of the sample surface. These measurements represent subsamples, which will be averaged later and

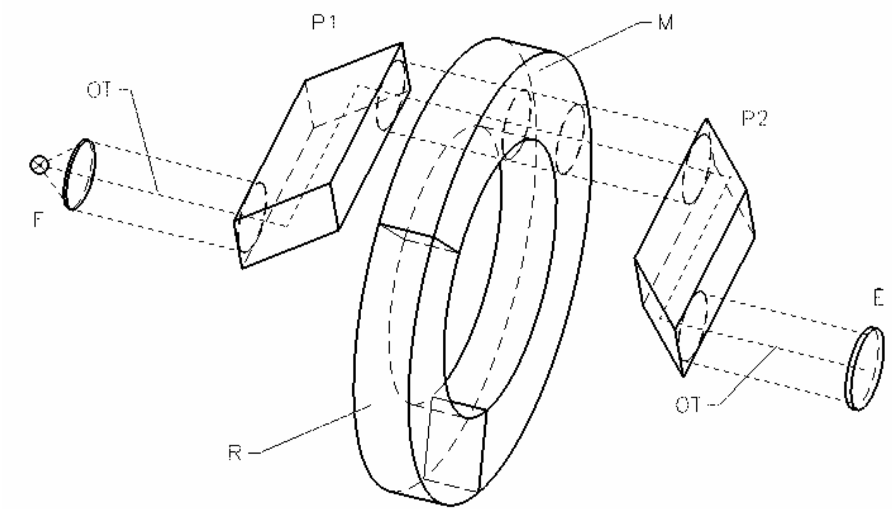

Figure 2. Sample compartment of Mininfra Scan-T.

one measurement on the standard can be done during every revolution of prism-wheel. In this arrangement all optical parts are common in both the sample and the reference light path. As elapsed time between sample and reference measurements is only a few milliseconds, so the lamp, optical parts and detector instabilities can be compensated. As a consequence, the long term stability depends only on the stability of the standard and on the wavelength stability of the monochromator, but no other part of the optical system has any influence on it. Additional advantage of this solution is that a simple, low cost rotating part serves for both sample-reference changing and subsample measurements.

Two types of sample holder are used depending on the type of sample:

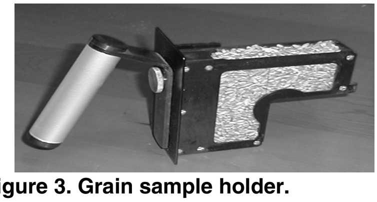

Figure 3. Grain sample holder.

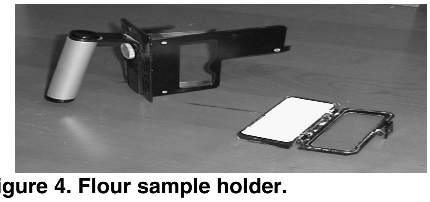

Figure 4. Flour sample holder.

The path length is 18 mm for grain and 6 mm for flour measurements respectively.

2. “Light-folder” part

The middle part of optics is a so called “light-folder” which consists of a few mirrors mounted on appropriate angle. The limited dimensions of a portable device make necessary the use of this part. The total light path of the instrument is 1400 mm. This path must be compressed into a space, as small as possible.

3. Monochromator part

We have chosen a monochromator which ensures great light intensity and brings together the compact size and the good resolution: it does not use slits but applies very long optical path. Applying 1200 rules/mm grating can be achieved a bandwidth of 9 nm in the 800-1080 nm range. The grating is driven by a stepping motor.

Operation of the analyser

Routine operation

The instrument routinely measures the composition of the sample using twelve wavelengths. The measurement starts after turning on the lamp and the prism wheel motor. Some control functions are used to ensure reliable results: the measurement can only start when the lamp has warmed up and the prism wheel has reached its normal rotation speed. It takes 4-5 seconds. After having moved the grating to the angle according to the first wavelength, the logarithm of the ratio of the light-intensities measured on the sample and the built-in standard is calculated. While the prism wheel turns around several times at every wavelength, the measured intensities are averaged in case of every sub-sample measurement. This process is repeated at each wavelength.

Scanning mode

The instrument can measure the entire near infrared spectrum in 0,02 nm steps, but it needs a long time. In practice we record spectra in about 2 nm steps, which lasts 3 minutes. Sub-sample spectra from a typical sample of wheat are shown in Figure 5.

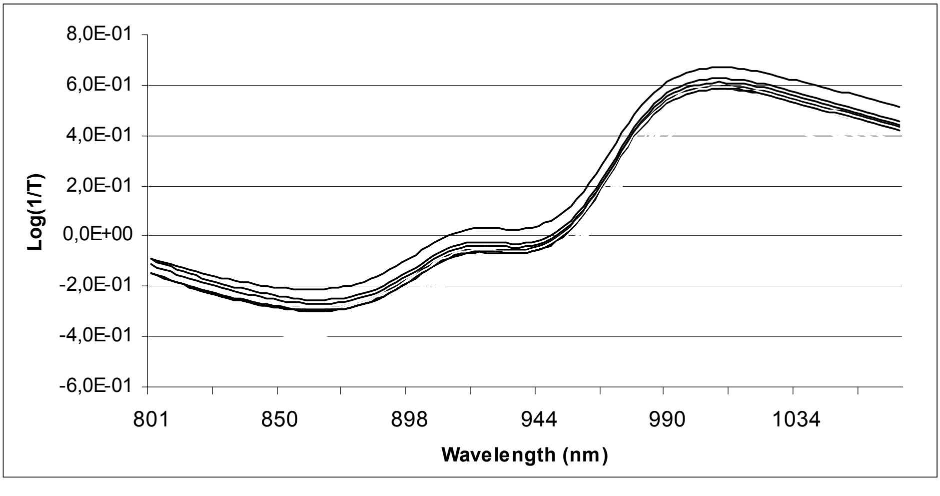

Figure 5. Sub-sample spectra from a typical sample of wheat.

A spectrum of a near infrared transmittance wavelength-etalon is shown in Figure 6. This etalon is a mixture of different rare earth-oxides in spectrally neutral matrix. This wavelength-etalon was produced by Metrika Kft, Hungary.

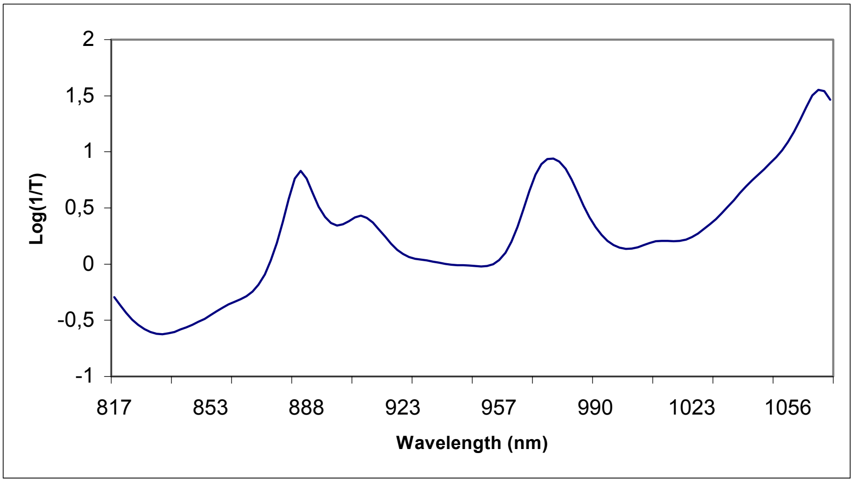

Figure 6. Spectrum of a rare earth-oxide transmittance wavelength etalon.

Application of the analyser

Measuring the protein and wet gluten content of wheat

The main application area of Mininfra Scan-T is measuring of the protein, moisture and wet gluten content of wheat. The analytical values for % protein in wheat using chemical method and Mininfra Scan-T are shown in Figure 7.

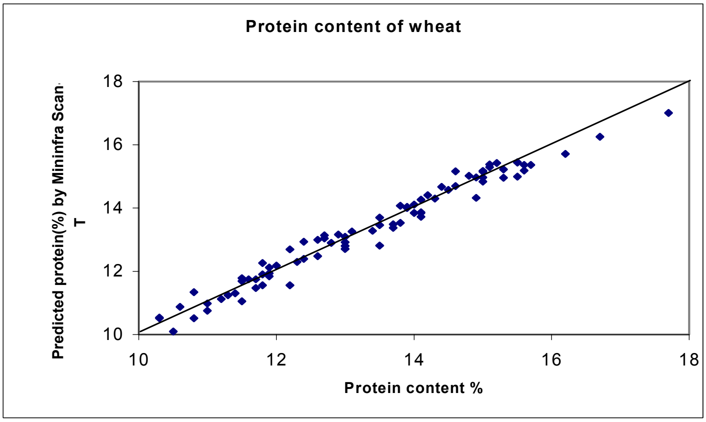

Figure 7. Analytical values for % protein in wheat and by Mininfra Scan-T. (Number of samples= 83, R=0,98, SEP=0,29).

The analytical values for wet gluten content of wheat using chemical method and Mininfra Scan-T are shown in Figure 8.

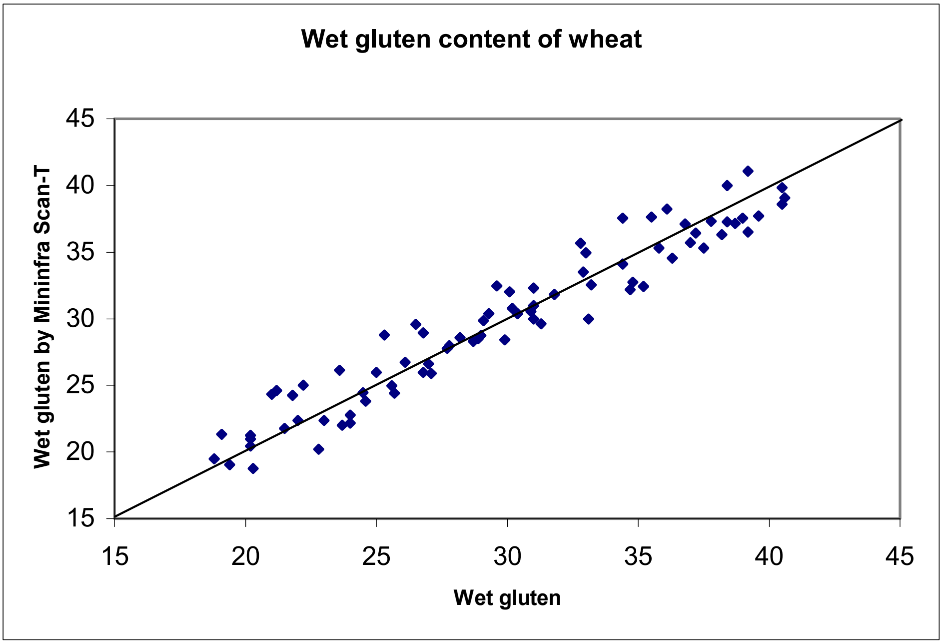

Figure 8. Analytical values for wet gluten content of wheat and by Mininfra Scan-T. (Number of samples= 83, R=0,96; SEP=1,6).

Measuring the ash-content of flour.

The instrument can be used to measure flour or grinded samples as well. Correlation plot of measurement of ash content in flour are shown in Figure 9.

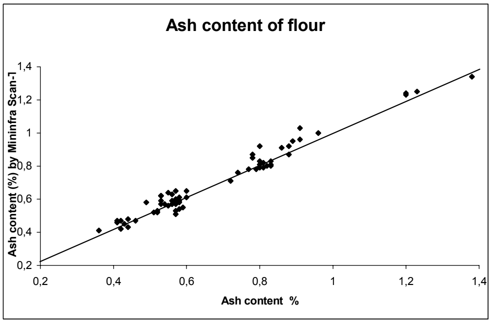

Figure 9. Analytical values for ash content in flour and by Mininfra Scan-T. (N = 64, R=0,98; SEP=0,035).

The electronic part of Mininfra Scan-T is a compact PCB, which controls the measuring process, stores the calibrations and communicates with the keyboard, the printer or the external computer. The instrument can be powered from mains by a 12 V/ 35 VA adaptor or from a car cigarette lighter plug-in.

In spite of the novelties applied in the optical part of the analyser, we kept the old-fashioned MLR calibration method. To develop a new application, the entire near infrared spectrum of calibration sample set has to be recorded. Usually the measured spectra consist of data measured at 120 wavelengths. The routinely used twelve wavelengths are selected by a reverse stepwise linear regression method. After the determination of the optimal wavelength set, the calibration is done according to the general rules of NIT / NIR calibration.

Acknowledgements

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References

1. G. Takács and T. Pokorny, in *Near Infrared Spectroscopy: Proceedings of the 7th International Conference,* Ed by A. M.C. Davies and Phil Williams. NIR Publications, Chichester, UK, pp. 92-97 (1996).