MOISTURE MEASUREMENT

Near infrared transmittance for measuring the moisture content of grains

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Introduction

Determining the moisture content of grain and oleaginous foods is just as important as determining their protein content prior to their sale. If the moisture content is too high, the grain must first be dried to achieve a moisture content that is low enough for the grain to be stored - this is a costly and time-consuming process. In addition, comminution of grain demands a specific moisture content and this requirement must be complied with as closely as possible. The moisture content of grain and oleaginous foods thus has a considerable influence on the sale price which can be obtained; consequently rapid and exact determination of the moisture content during harvesting, storage and processing is of utmost economic importance.

In Germany, hygrometers used in official or commercial transactions must be verified before they can be used in the field - in fact these instruments must be type approved by the Physikalisch-Technische Bundesanstalt (PTB) for the grains and oleaginous foods they are intended to measure.

In accordance with the procedure applied, hygrometers are classified into measuring instruments used:

- to determine the moisture content by drying; and
- to measure a moisture-dependent physical quantity such as electrical resistance, capacitance or reflection, or absorption of near infrared radiation.

Measurements whereby the moisture content is determined by drying the grain and subsequently determining the loss of mass are generally too expensive and time-consuming for trade in cereals: results are obtained more rapidly by devices which determine the moisture content by measuring a physical quantity.

Near infrared transmission spectral analyzer

In April 1998, a device which works on a new measuring principle was approved in Germany for the measurement of the moisture content of wheat, rye, barley and triticale in the range from 10 % to 20 % (See Fig. 1). Further approvals have been granted in the USA (FGIS), Canada (CGC), Argentina (I.A.S.C.A.U), Denmark (Plant Directory) and South Africa (Wheat Board).

Description of the measurement principle

The functional principle of an N.I.T. (Near Infrared Transmission) device is depicted in Fig. 2:

- Light from a halogen lamp is directed onto a monochromatic mirror.
- This mirror generates monochromatic radiation in the wavelength range between 800 nm and 1100 nm.
- With the aid of an electronic-mechanical control technique, the wavelength range from 850 nm to 1050 nm is applied to the sample at wavelength steps of 2 nm.
- Part of the light is reflected or absorbed by the sample; the other part, the transmitted light, is received by a detector.

The absorption of light varies as a function of the composition of the different sample components, such as moisture, protein, fat and fiber structure, the absorbance being decisively determined by the layer thickness of the sample. Measuring vessels with a layer thickness of 18 mm are used for wheat, rye, barley and spelt and a measuring cell with variable layer thickness is used for other grains to be measured. The required layer thickness is set in line with the grains to be measured with the help of a servomotor.

Fig. 1 The Foss Infratec 1229 Grain Analyzer

Measurement is started with a scan, without the sample, carried out as a reference measurement over the whole wavelength range. The detector system thus determines the light intensity furnished by the system. Subsequently, after having been filled into the sample funnel, the sample is automatically transported to the measuring vessel. In the course of the measurement, the intensities of the 100 selected wavelengths are determined. Then the absorbance values are calculated by the computer system. The values obtained furnish a spectrogram with peaks which are characteristic of the sample measured.

From Fig. 3, the moisture and protein values can then be determined with the aid of a calibration transcribed via the network (see below) or copied from a floppy disk.

The calibrations for the individual components are defined by the instrument manufacturers, in terms of suitable laboratory reference procedures, by an adjustment calculus according to the least squares method. The PTB then checks these calibrations and, if correctness has been proved, grants an approval. From the verification law point of view, only the moisture value is of significance though various interested parties also request a verified determination of the protein content. This is why the possibility is being considered of evaluating the calibrations with respect to the protein content within the scope of a special test.

The advantages of the N.I.T. procedure over the other methods of measurement are the following:

- the sample must not be bruised for the analysis;
- prompt measurement results;
- the measurement results are independent of sample temperature and ambient temperature;
- simultaneous determination of several quality characteristics (e.g. moisture and protein);
- measuring devices with networking capability.

During a single measurement process the device carries out ten individual measurements. The sample quantity (which varies between 300 g and 500 g) is fed to the device without having been bruised (see Fig. 4). The measuring cycle then takes approximately 40 seconds, following which the measurement result is indicated on the digital display. In addition, the standard deviation of the ten individual measurements can be retrieved, which allows conclusions to be drawn concerning sample homogeneity.

The NITNET network

The analyzers can be used most efficiently when they are interconnected by a network. At present, four N.I.T. networks exist in Germany:

- Doemens Calibrierdienst (DOEMENS-NITNET) near Munich;
- Raiffeisen HG Nord (RHG-NITNET) in Hanover;
- Network Rhineland-Palatinate (RLP-NITNET) in Leideneck;
- VDLUFA Network (VDLUFA-NITNET) in Kassel.

Approximately 180 N.I.T. analyzers are interconnected within these four networks which are independent of each other. They are connected as satellites to their network operator in a star pattern via a modem and transcription to, or modification of, the calibrations on the individual devices is possible only via this modem from the central processing unit of the network operator. This prevents manipulation of the calibrations by the measuring instrument user.

Verification

In contrast to the laboratory reference procedure and the electrical hygrometers, the N.I.T. devices determine only the water fraction which is molecularly bound in the grain and not the water possibly adherent to the

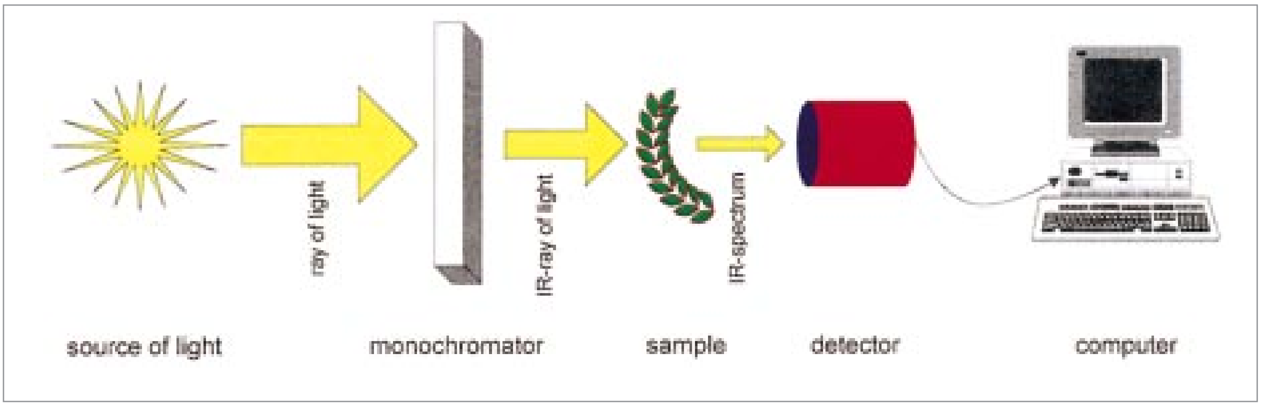


Fig. 2 Functional principle of an N.I.T.

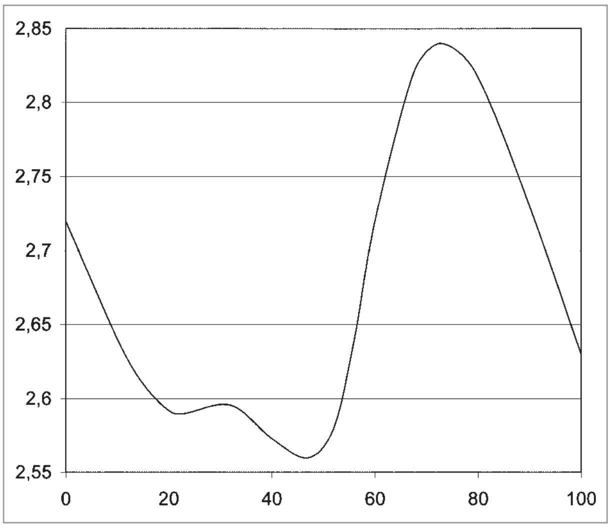


Fig. 3 Graphical representation of a spectral analysis of wheat with the following component values:

Moisture:	20.4 %
Protein:	14.0 %
Spectral measuring range:	850 nm to 1050 nm
Points of measurement:	100 (corresponding to a resolution of 2 nm)

Fig. 4 Filling of the grain sample to be measured

dish. It was, therefore, necessary to open up new paths for the verification of these measuring instruments.

Foss submits the calibrations developed to the PTB for examination and approval. These calibrations are transcribed to the PTB's master device and checked for correctness. After that, the three submasters available at the verification authorities of Bavaria, Lower Saxony and Rhineland-Palatinate are compared with the PTB's master (see Fig. 5). A maximum deviation of ± 0.2 % is permissible. With the aid of these submasters, the users' measuring instruments are then verified in accordance with the approval.

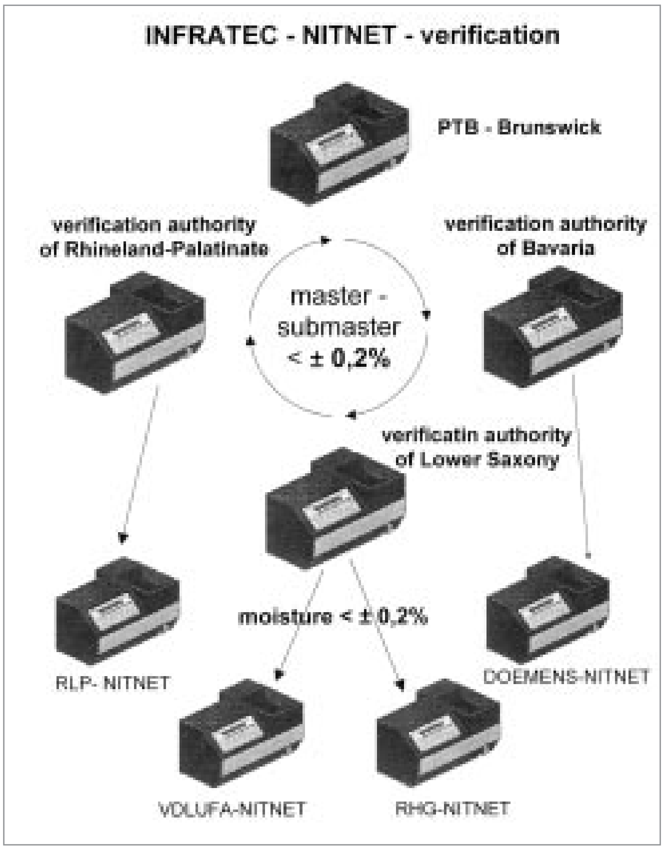


Fig. 5 Connection of submasters to PTB's master device

The first verification of grain analyzers in Germany took place in Rhineland-Palatinate in July 1998. To date, about 80 devices have been verified in Rhineland-Palatinate and if subsequent verifications are included the total result is 125 verifications.

Verification test

The verification technological test comprises:

(i) Functional test with granulate

Three measurements are carried out in succession with a granulate specially produced for this purpose. The indication must lie in the interval 100 ± 0.5. If this condition is not complied with, testing of the device is stopped.

(ii) Comparison of the device tested with the submaster

Comparisons between the device tested and the master are carried out with two samples of each type of grain approved. Any commercial grain or seed can be used as sample material. The moisture content of one of the samples must be between 11 % and 12 %, that of the other between 14 % and 15 %. The grain may also be moistened; care must, however, be taken that the sample material is thoroughly moistened. This can be achieved by ensuring a sufficiently long mixing time (about three days).

Each sample must be measured three times by the submaster and three times by the device tested. The mean values of the measurement results obtained by the submaster and the device tested must not deviate by more than ± 0.2 %.

A problem encountered upon verification, in particular on hot days in rooms without air-conditioning, is that samples with higher moisture values may dry out during measurement. Practical applications have shown that the moisture value may change by 0.1 % during a test cycle involving a sub-master and the device tested. When several devices are verified it is, therefore, necessary to recheck the moisture value on the master after each measurement comprising three individual measurements. The time and effort required for this procedure are such that only four to six devices can be verified each day. Solutions are therefore being sought which will allow the annual subsequent verification to be carried out in future together with the network operator in the form of an intercomparison.

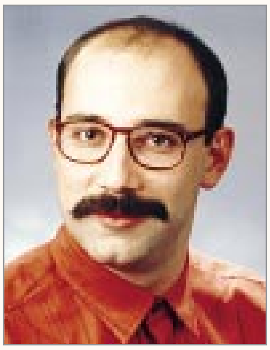
It is difficult to furnish reliable figures about measurement stability due to the fact that all the devices which were verified were subsequently also calibrated for various grains - this was carried out just prior to the subsequent verification. Furthermore the devices will be maintained and if necessary repaired. Therefore, the measurement error during verification cannot be compared to the measurement error of moisture determination after the device has been in operation for one year. However, the reproducibility with a deviation of ± 1 digit (0.1 %) measurement results is very good.

Summary and conclusions

The new N.I.T. devices offer the possibility to determine the important quality characteristics of grain and oleaginous foods quickly and without the risk of operator errors. Devices operated in a network furnish results with very small deviations not only for moisture measurements, but also for other parameters such as protein.

Practical application will still have to show to what extent the correctness is guaranteed, in particular in the case of grain whose biological characteristics deviate from those serving as a standard, where measurement stability is higher compared to hygrometers which measure electrical resistance or capacity.

Furthermore it has been shown that N.I.T. network instruments considerably improve relations between producers and traders, since producers are certified and every trader who is connected to the network can supply and certify a certain product quality. ■



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