Sample presentations for near infrared analysis of intact fruits, single grains, vegetable juice, milk and other agricultural products

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Indroduction

Sample presentation is very important for near infrared (NIR) spectroscopy. Figure 1 shows the four major modes of spectral data acquisition: (1) transmittance, (2) reflectance, (3) transflectance and (4) interactance. In the transmittance mode [Figure 1(a)], incident light illuminates one side of the sample and an unabsorbed portion of the incident light is collected by the detector on the opposite side. This mode is widely used for liquids and low density solids. Incident light, in the case of the reflectance mode [Figure 1(b)], illuminates the surface of a sample, some of the light enters the sample and is diffusely reflected to the detector. In this mode, the sample is usually opaque, like a powdered sample having more than 1cm depth. Transflectance [Figure 1(c)], originally developed by Technicon (now Bran+Luebbe, Germany) for the InfraAlyzer, was designed to study liquids in an instrument designed to measure reflectance. Hence, the liquid cuvette is "backed" with a diffuse reference. The incident light is transmitted and reflected through the sample. Interactance [Figure 1(d)] was developed by Karl

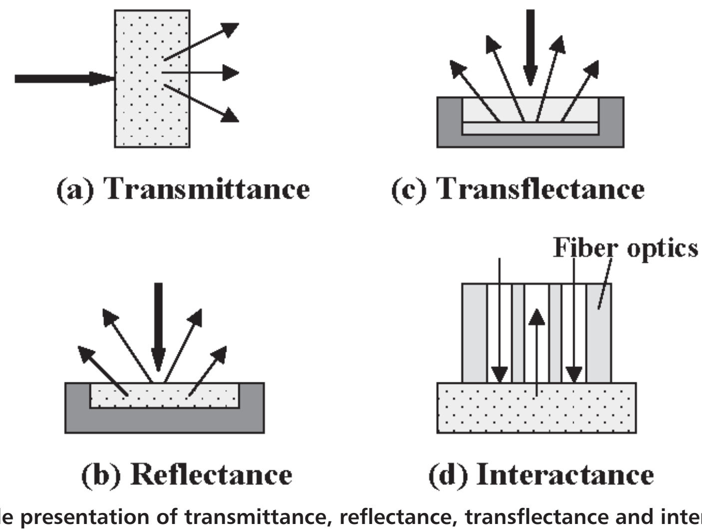

Figure 1. Sample presentation of transmittance, reflectance, transflectance and interactance.

Norris as a means for studying living plant and human tissue. Usually undertaken with concentric fibre optics, as shown in Figure 1(d), the sample-to-fibre optics is such that the incident light is forced into the sample to interact with the sample before making its way to the detector optics. Therefore, only the light transmitted through, or interacting with, the sample can be detected.

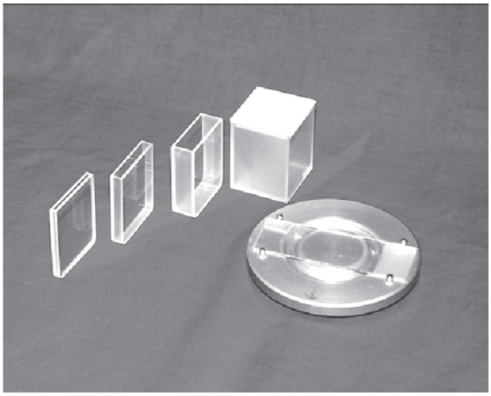

Figure 2. Sample cells for liquids; cuvettes and aluminium cell.

Sample cell for liquids

A typical cuvette made of quartz is shown in Figure 2. It is the popular mode for collecting spectra of liquids such as water, liquors and juices. The thickness (path length) of the cell depends on the wavelength region and samples used. In the case of water, 2–3 cm thickness is suitable for measurements at 960 nm, 2–3 mm thickness is suitable for measurements at 1450 nm and cells with a thickness less than 1mm can be used at 1930 nm. Sample thickness is very important when conducting spectral–transmittance measurements.

In a reflectance instrument, the aluminium cell specially designed for the InfraAlyzer 500 (right in Figure 2) is widely used. A small amount of the sample is taken and dropped onto the central portion. The cell covered with a glass plate may now be placed in the sample drawer of the instrument. The usual path length of the cell is 0.1 mm.

Sample cell for pastes

Spectra of pastes, such as dough, ground meat and fermented soybean paste (called Miso) can be acquired using the sample cell shown in Figure 3. Figure 3 illustrates the two types of sample cells for obtaining reflectance spectra (shown at the left in Figure 3, so-called “Open cup”) and transmittance spectra (right, so-called “transport cell”, or TC). The open cup, designed specially for the InfraAlyzer 500, may be positioned without a glass cover into the drawer of the instrument. The surface of the sample in the open cup should be smoothed. The TC was designed specially for the NIRS 6500. Samples are usually packed into polyethylene bags for measurements. Closing the cell forces the sample to a constant thickness of 1 cm. While the spectrum is continuously scanned, the sample is transported through the NIR beam.

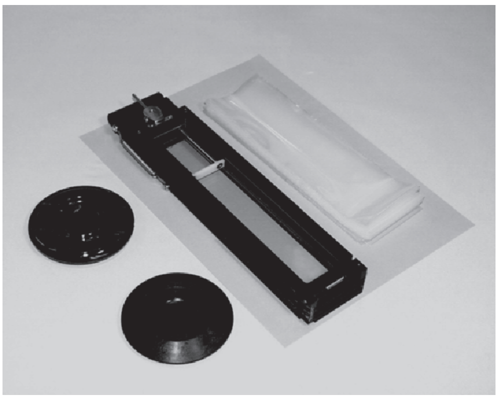

Figure 3. Sample cells for pastes; open cups and high fat/high moisture transport cell.

Sample cell for a powdered sample

Powdered samples can be measured in a sample cup like that shown in Figure 4. The cell has a specially designed rubber pad to help produce a reproducible packing density. Density does affect scattering and scattering should be carefully

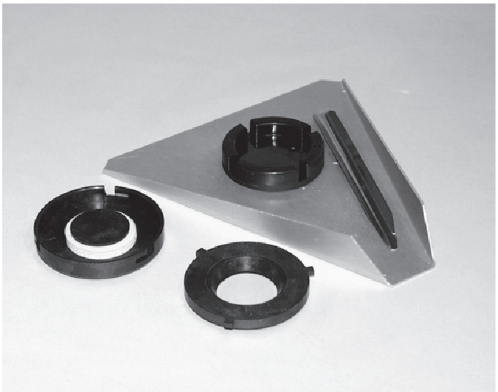

Figure 4. Sample cell for powdered sample.

Figure 5. Sample cells for whole grains.

controlled during any given NIR experiment. The cell in Figure 4 was designed for the InfraAlyzer 500. It has a circular quartz window, 3.5 cm in diameter, mounted in black plastic. The thickness of a powdered sample in this cell is approximately 1 cm. The reflectance cell in Figure 4 is shown with a cell holder and scoop.

Sample cell for whole grains

Whole grain samples can be scanned in the sample cells shown in Figure 5. The sample cell on the left was developed by NIRSystems for the NIRS6500 instrument. The sample cell has a loading funnel input that makes it easy to load. It is a "transport type" cell, mounted vertically with the NIR instrument which moves up and down slowly during NIR measurement to compensate for the heterogeneity of the samples. A single spectrum using this cell is usually the average of 30–50 scans. The circular cell, shown to the right in Figure 5, was designed for the InfraAlyzer 500. The cell, 85 mm in diameter and 12 mm in depth, is rotated quickly during acquisition of NIR spectra, another means of averaging heterogeneity.

Sample holder for fruits

Acquisition of NIR spectra of large samples such as fruit requires a specially designed "sample holder." Figure 6 is a typical of holder needed to make interactance measurements of fruits with fibre optics. Bifurcated fibre optics are required to isolate the incident and interactance energy. A cushion made of urethane foam is pasted on the end of the probe to hold the sample and exclude stray light. The holder requires only that the sample be positioned on the cushion. The sample holder in Figure 6 has been used to study the rela-

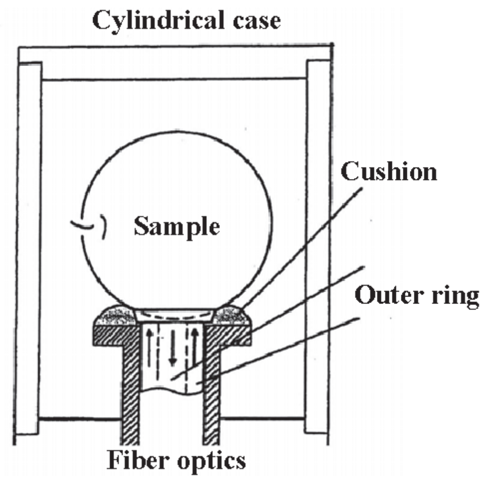

Figure 6. Sample placement for NIR interactance measurements using fibre optics.

tionship of NIR spectra to the Brix values of intact peaches.[1]

Useful interactance measurements are difficult to obtain from oranges because peel thickness inhibits interactance of NIR energy with orange meat. The transmittance mode is for more effective for this type of measurement. A schematic of a piece of apparatus for making measurements in the transmittance mode is given in Figure 7. The top of the sample is illuminated with monochromatic light through a fibre optic. The light passes through the sample and is measured with a silicon detector located below the sample. In transmittance spectroscopy, the spectra are affected by the variation in diameter of the samples measured. In order to reduce the sample size effect, $d^2\log(1/T)$ at each wavelength was divided by $d^2\log(1/T)$ at 844 nm, a wavelength having a high correlation only to fruit size. The corrected spectra, called "normalised 2nd derivative spectra", are nominally not affected by sample size. Multiple linear regression (MLR) based on normalised 2nd derivative spectra and Brix value, produced very good results (R = 0.989, SEC = 0.28°Brix, SEP = 0.32°Brix).[2]

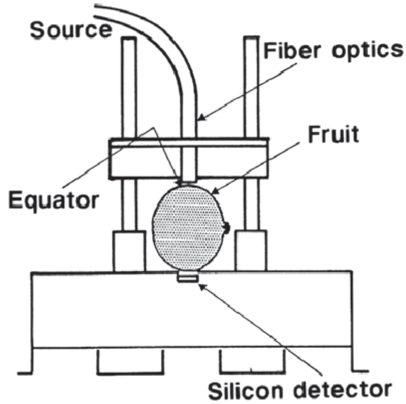

Figure 7. Sample placement for NIR transmittance measurement using fibre optics.

The other sample cells

Conventional liquid cuvettes are difficult to wash and dry. This laboratory has studied the use of test tubes for sample cells. Studies of milk, rumen juice and urine of milch cows have been conducted using test tubes.[3] Test tubes have been used to study spectral properties of spinach juice for determination of undesirable constituents. This laboratory has also worked to develop special sample holders for making measurements of single kernels of grain.

Conclusion

Recording of useful spectra requires many parameters to be monitored. However, selection of the most appropriate sample holder (cell) is absolutely essential.

Reference

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