Near Infrared Reflectance Spectroscopy (NIRS): A Method of Rational Multicomponent Analysis

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Abstract. With the introduction of chemometric evaluation software combined with diffuse reflectance measurements, near infrared spectroscopy has been launched into a new era of analytical applications. In the present contribution the basic principles of NIRS and its potential for chemical multicomponent analysis are discussed.

Key words: near-infrared spectroscopy, chemometrics, multicomponent analysis.

The NIRS method has been used for the analysis of agricultural and food products for around a decade. Technological progress with regard to optics and computers has developed to a point where this technique can now also be used in the area of chemical and pharmaceutical multicomponent analysis [1, 2]. In principle, the method belongs to the discipline of chemometrics, which generates correlations between experimental data and the chemical composition or physical properties of the tested samples by application of mathematical and statistical procedures.

The principle of the procedure for quantitative analysis is based on recording of the NIR transmittance or diffuse reflectance spectra of around 25—30 standard samples of known composition and storing these spectra with the levels of the constituents determined by independent, conventional analytical methods in the dedicated computer system. Using a given program, the absorption intensities which correlate with these concentration levels are determined, and standardization curves for the individual constituents subsequently constructed by means of linear regression. Using their NIRS spectrum and this existing standardization, unknown samples are then evaluated with regard to the levels of their components. When the standardization has once been performed, the analysis time required for an unknown sample is almost independent of the number of components to be determined, and only amounts to a few minutes in comparison to several hours previously. Alternatively, the NIRS technique can also be used for qualitative identification in the course of an identity check.
Promising applications for this technique have been reported for the following product groups:

- analysis of the content of active ingredient and byproducts in liquid and solid pharmaceutical and plant protection formulations,
- analysis of polymers of widely varying morphology including the determination of characteristic chemical and physical parameters of synthetic fibers.

**Experimental and Results**

The experimental results were obtained on a Technicon 500 system equipped with a Hewlett Packard 1000 computer. The optical measurement of solids is based on the intensity of diffusely reflected radiation from the sample. The radiation directly reflected from the gold-plated inner surface of the integrating sphere serves as reference intensity (Fig. 1). Around two minutes are required to take a spectrum in the wavelength range of 1100-2500 nm. Liquids were measured using the so-called transflection method, which consists in coating the base of the cell with a diffusely reflecting layer and passing the measuring beam through the sample twice.

The NIRS spectra are recorded using a set of standard samples with varying levels of the components (which have been determined by means of independent, conventional analytical methods), and the individual levels are provided to the computer. The relationship between the intensity at a definite wavelength (or at different wavelengths) characteristic for a

![Fig. 1. Simplified optical scheme of a scanning NIRS instrument](image-url)