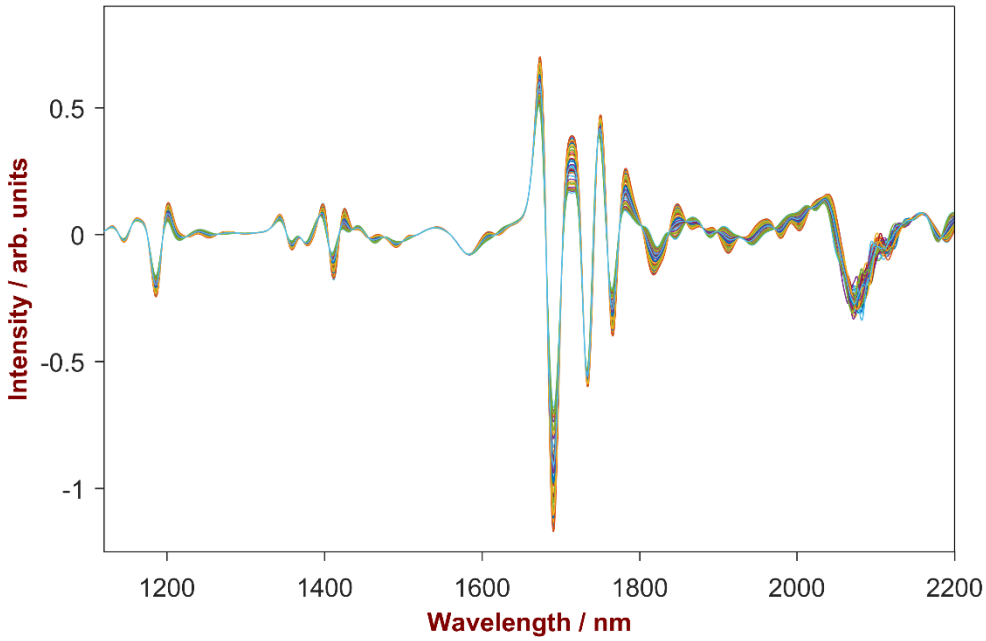
NIR Application Note NIR-061

Determination of aliphatic alcohols in alcohol mixtures using visible near-infrared spectroscopy



This Application Note describes a fast, nondestructive, and reliable method for the determination of the chemical composition of alcohol mixtures exemplified by ethanol/isopropanol mixtures. With visible near infrared spectroscopy (VIS-NIRS), results are available in real-time, thus making NIRS highly suited for fast quality control.

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Method description

Introduction

Aliphatic alcohols are used as ingredients for a wide range of antiseptic products. Disinfectants are usually a mixture of two or three components such as ethanol, propanol, isopropanol, and water. The ratio of the used ingredients is usually a product-specific quality parameter, which needs to be controlled. Therefore, a fast, nondestructive method for determining the alcohol content is essential to monitor product quality. A possible solution is the use of visible near-infrared spectroscopy, which enables simultaneous determination of multiple quality parameters within a minute.

Experimental

A total of 44 liquid customer-specific mixtures of ethanol and isopropanol were used in the present study. The content of both alcohols ranged from 0 to 100%. The NIR spectra were measured in transmission mode using a Metrohm XDS RapidLiquid Analyzer with a spectral range from 400 to 2500 nm. The samples were placed into 8 mm disposable glass vials. Sample temperature was kept constant at 30 °C during measurements. The software package Vision Air 2.0 Complete was used for data acquisition, data management, and development of the method (Tab. 1, Fig. 1).

Tab. 1: Used equipment and software

Equipment	Metrohm part number
NIRS XDS RapidLiquid Analyzer	2.921.1410
NIRS 8 mm disposable glass vials	6.7402.000
Vision Air 2.0 Complete	6.6072.208

Fig. 1: The NIRS XDS RapidLiquid Analyzer was used for spectral data acquisition over the full range from 400 to 2500 nm.

For method development 33 samples were used, whereas 11 residual samples were used for the validation. The spectra were pre-treated using 2nd derivative and a partial least squares regression (PLS) was performed over specific spectral regions.

Results and discussion

Fig. 2 shows the Vis-NIR spectra of all analyzed samples. A variation of the O-H combination band near 2100 nm and that of the first O-H overtone near 1460 nm is observable. This signal variation is related to alcohol concentration changes. Fig. 3 shows the second derivative spectra for these samples, which was calculated to compensate for any baseline variations.

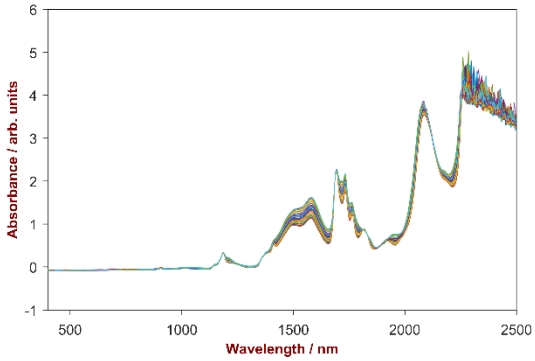

Fig. 2: Vis-NIR spectra of 44 ethanol/isopropanol mixtures.

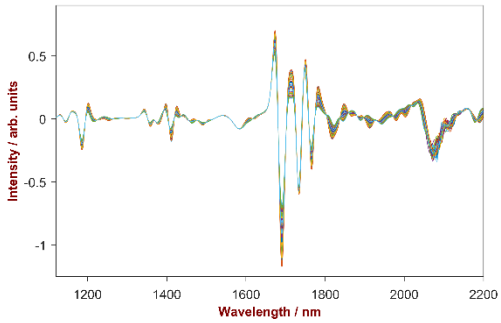

Fig. 3: 2- derivative spectra of 44 ethanol/isopropanol mixtures in the region of 1100–2200 nm.

The correlation plot in Fig. 4–5 shows high correlation between the reference values provided by the costumer (x-axis) and the predicted values (y-axis) from Vis-NIR spectroscopy. Furthermore very low standard errors of calibration and cross-validation (SEC, SECV, SEP) were achieved.

Ethanol

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Method description

Tab. 2: Setting and results of the quantitative method development for ethanol

Concentration range	0–100%
Method	PLS
Number of factors	3
Wavelength range	1300–1650 nm
Pretreatment	2nd derivative
SEC	0.34%
SECV	0.37%
SEP	0.44%
R^2	0.9998

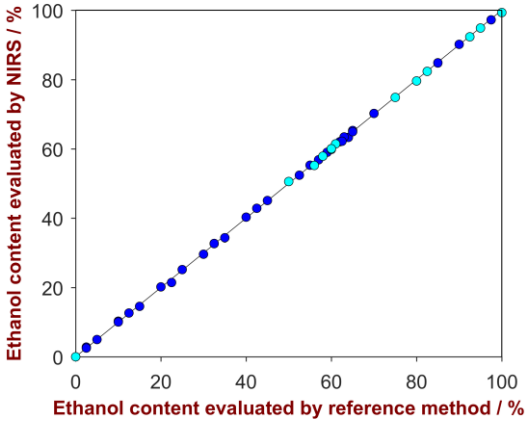

Fig. 4: Correlation plot of ethanol content predicted by NIRS versus reference values. A high correlation is observable. Displayed are the calibration (blue) and the validation data (turquois).

Isopropanol

Tab. 3: Settings and results of the quantitative method development for isopropanol

Concentration range	0–100%
Method	PLS
Number of factors	3
Wavelength range	1300–1650 nm
Pretreatment	2- derivative
SEC	0.38%
SECV	0.41%
SEP	0.45%
R^2	0.9998

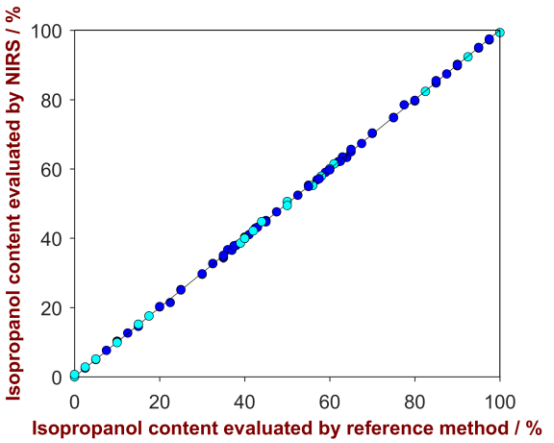

Fig. 5: Correlation plot of isopropanol content predicted by NIRS versus reference values. A high correlation is observable. Displayed are the calibration (blue) and the validation data (turquois).

Summary

NIR spectroscopy can be used to accurately and rapidly determine the content of very similar alcohols in mixtures used in antiseptic products. Liquid samples were successfully analyzed using a Metrohm NIRS XDS RapidLiquid analyzer. The developed methods were successfully validated using a independent sample set. Therefore, near-infrared spectroscopy is uniquely suited for fast analysis of such mixtures without sample preparation. The use of NIR for the quality control of antiseptic agents can be further extended through the development of quantitative methods for moisture, colorant, or additive content.

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