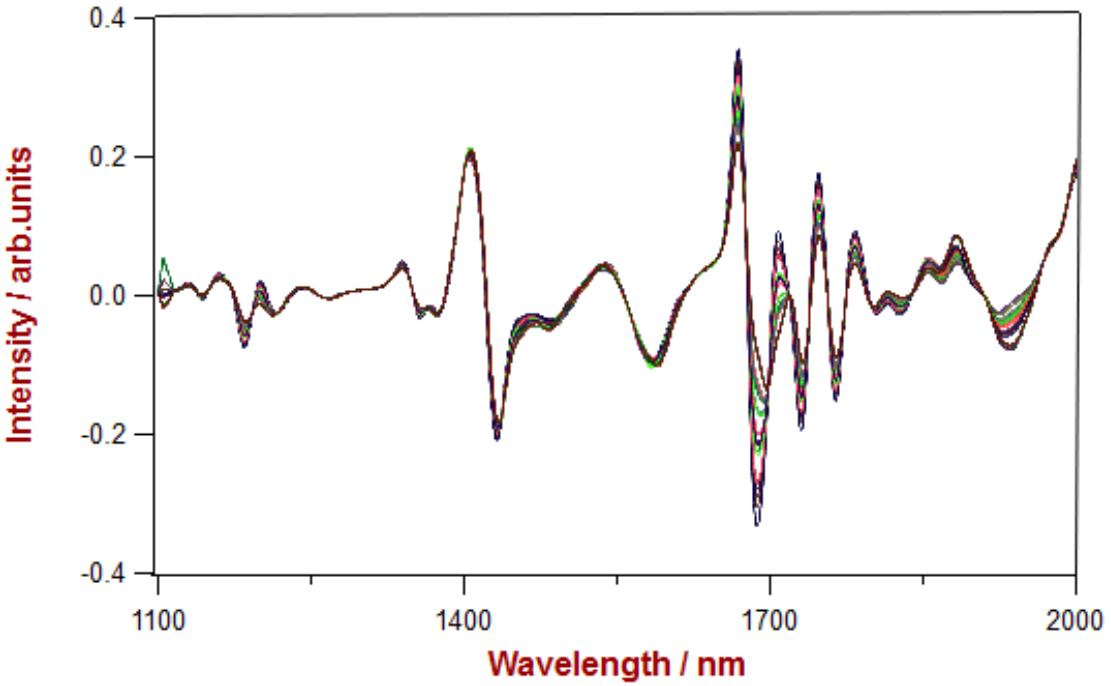
NIR Application Note NIR–070

Quantification of nicotine and glycerin in e-liquids using visible near-infrared spectroscopy



This Application Note describes a fast method for the simultaneous quantification of nicotine and glycerin in liquid mixtures used for electronic cigarettes. With visible near infrared spectroscopy (VIS-NIRS), results are available without sample preparation, thus making VIS-NIRS highly suited for fast quality control.

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Method description

Introduction

An electronic cigarette is a device that heats and vaporizes a solution called e-liquid that contains nicotine, in order to imitate the act of smoking.

The market of e-cigarettes has grown rapidly in the last eleven years since it was introduced in the United States of America in 2007. At first, many small manufacturers emerged, however in recent years the tobacco industry has taken interest and now international companies are the main distributors for these products. The e-liquid solution usually contains a mixture of glycerin of vegetable origin, propylene glycol, nicotine and additives used as flavorings. The ratio between propylene glycol and glycerin is commonly 70/30. Regulation of e-cigarettes varies from country to country, ranging from no regulations at all to complete bans. To give an example in 2014 the European Union has set an upper limit of 20 mg/ml nicotine in e-liquids. E-cigarettes are a relatively new product and considering the booming market, it is to be expected that stricter regulations will be applied in the future. Therefore, a fast method for determining the content of the ingredients is necessary, to ensure the product quality. A very suitable solution is the use of visible near-infrared spectroscopy, which enables simultaneous determination of multiple quality parameters within a minute.

Experimental

24 samples with varying concentrations of nicotine, propylene glycol, vegetable glycerin, and aroma were provided by a customer. The content of glycerin ranged from 5.00–50.00%, whereas the content of nicotine ranged from 0–20 mg/ml. The VIS-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 and kept at a constant temperature of 30 °C during measurements. Data acquisition and method development was carried out with the software package Vision Air 2.0 Complete (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 nm to 2500 nm.

Insignificant spectral regions were excluded during the method development process by the selection of specific wavelength ranges and using 2nd derivative as a pre-treatment. Internal cross-validation was applied on the data set to verify the performance of the PLS model.

Results

Fig. 2 shows the VIS-NIR spectra of all analyzed samples. Already the raw spectra displays significant differences between the samples in the wavelength range 1400–1900 nm. Fig. 3 shows the second derivative spectra for these samples, which was calculated to compensate for any baseline variations.

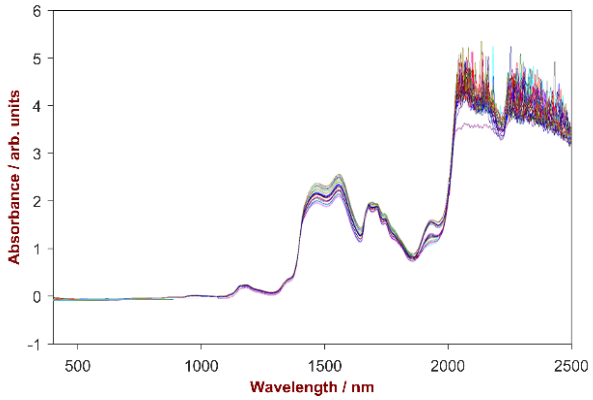

Fig. 2: VIS-NIR spectra of 24 e-liquid mixtures.

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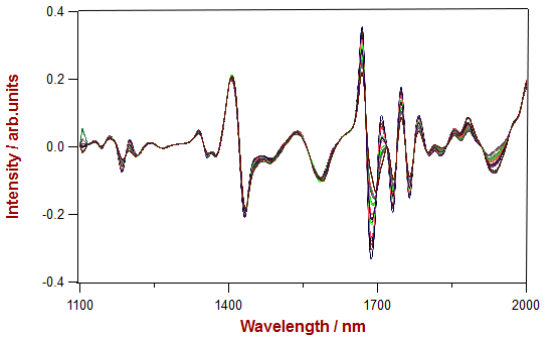

Fig. 3: 2nd derivative spectra of 24 e-liquid mixtures in the wavelength region of 1100-2200 nm.

The correlation plots in **Fig. 4–5** show high correlation between the reference values provided by the customer (x-axis) and the predicted values (y-axis) from VIS-NIR spectroscopy. Furthermore low standard errors of calibration and cross-validation (SEC, SECV) were achieved.

Nicotine

Tab. 2: Settings and results of the quantitative method development for *nicotine*

Range	0.00–20.00 g/ml
Regression model	PLS
Number of factors	3
Wavelength range	1892–1994 nm
Pretreatment	2nd derivative
SEC	0.58 g/ml
SECV	0.69 g/ml
R^2	0.9893

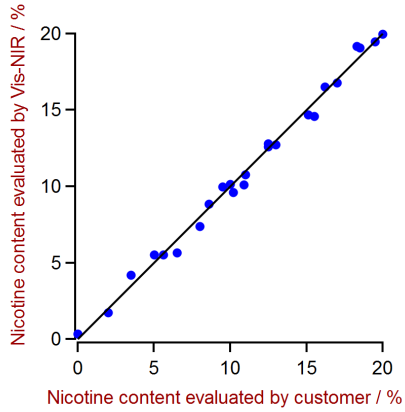

Fig. 4: Correlation plot of nicotine content predicted by VIS-NIRS versus reference values.

Glycerin

Tab. 2: Settings and results of the quantitative method development for glycerin

Range	5.00–50.00%
Regression model	PLS
Number of factors	3
Wavelength range	1120–1994 nm
Pretreatment	2nd derivative
SEC	1.53%
SECV	1.63%
R^2	0.9877

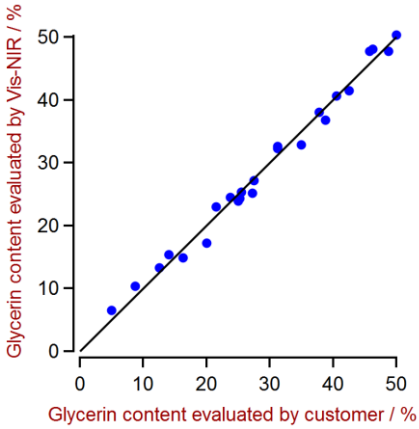

Fig. 5: Correlation plot of glycerin content predicted by VIS-NIRS versus reference values. A high correlation is observable.

Summary

The results presented in this Application Note show the feasibility of using VIS-NIR technology for quality control of e-liquids. The NIRS XDS RapidLiquid Analyzer in conjunction with Vision Air 2.0 Complete enables simultaneous determination of nicotine and glycerin. Therefore, near infrared spectroscopy appears to be uniquely suited for fast analysis of such mixtures without sample preparation.

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