



Fast and accurate routine analysis of alcohol content in the presence of sugar concentrations in spirits and other alcoholic beverages.

Abstract

The spirits and beverage industry has a high demand for fast and precise quality tests of alcohol content even in the presence of a vast variety of other components in the produced products. The sensitivity of conventional methods suffers from either procedure complexity or from the sensitivity especially when spirits, liqueurs and other beverages have high sugar contents. NIR spectroscopy is known as a method for the determination of ethanol and methanol – even in small concentrations, but in the presence of sugars and some other components the accuracy of the detection decreases.

An important parameter, which is strictly controlled in alcoholic drinks, is the methanol content which naturally appears in many distilled spirit products. Similarly to the ethanol content detection, sugars bring complexity to the measurement of the methanol content using NIR methods.

The aim of this study was to validate NIR calibration models for a broad variety of distilled spirits and liqueurs that have been developed by ZEUTEC, Germany in cooperation with Landwirtschaftskammer Tirol, Innsbruck, Austria (U. Zeni) in 2020. Ulrich Jacob Zeni is the consultant in fruit processing and crop protection at the chamber of commerce of Tirol. The challenge of this work was to achieve the desired precision in ethanol and methanol measurements in presence of high sugar concentration in the respective samples.

Experimental setup

About 200 samples including commercially available products and trial blends were measured at the Landwirtschaftskammer Tirol using ZEUTEC's SpectraAlyzer SPIRITS. Samples were chosen to have different sugar and ethanol content (sample sets 1-3) and those which contain methanol, ethanol and sugar (sample set 4). Calibrations were validated using the analytical reference methods. The ethanol content was validated with electronic densitometry of the distillate. The methanol content was determined using gas chromatography. The sugar content was validated with an enzymatic method.

Conclusions

The chemometric model for determining the alcohol concentration in alcoholic beverages containing high amounts of sugar developed in this work has shown a high accuracy. The SpectraAlyzer SPIRITS provides an easy turnkey solution for all steps of alcoholic beverages and spirits production and it does not require any sample preparation or preprocessing for the measurement. The presented fast ethanol, methanol and sugar contents determination method has shown high accuracy in simultaneous valuation of all above mentioned parameters. Besides the scope of this poster accurate calibration models for sugar content determination were developed and validated.

Results and discussion

It could be validated that ZEUTEC's SpectraAlyzer SPIRITS performs quick and accurate determinations of the ethanol and methanol content with the presence of total sugar up to 350 g/l with 8% - 96% ABV. The solutions from the sample set 4 contained methanol in a concentration range from 550 ppm to 5350 ppm. ZEUTEC has performed the calibration test and determined a cross validated calibration error for ethanol of < 0.09% vol. (RMSECV) in the mixture of methanol, ethanol and sugar. The cross validated error of the calibration for ethanol without methanol and sugar presence in the mixture has been found to be < 0.04% vol. (RMSECV). These results are summarized in table 1. The multiple linear regression (MLR) prediction curves for these measurements are represented in the Fig. 1 - 4. For the determination of methanol in the mix, a calibration with a cross validated error of 70 ppm (RMSECV) for methanol could be developed (Fig. 4 and table 2).

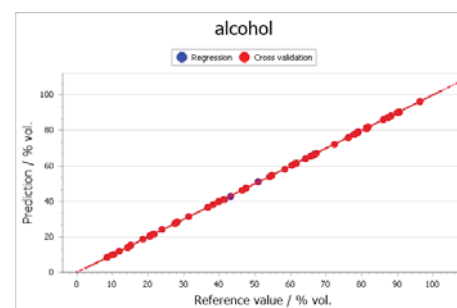


Fig 1. MLR(**) curve for measurements of ethanol content in samples without sugar (sample set 1 from the Table 1)

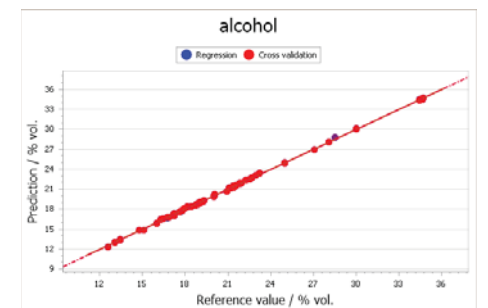


Fig 2. MLR curve for measurement of ethanol content in samples with high total sugar quantity (sample set 3 from the Table 1). Sugar content is 100 - 320 g/l

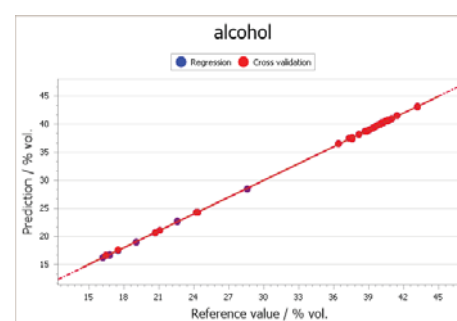


Fig 3. MLR curve for the measurements of ethanol quantity in samples which consist of ethanol, sugar and methanol (sample set 4 from the Table 1). Sugar content is 0 - 352 g/l

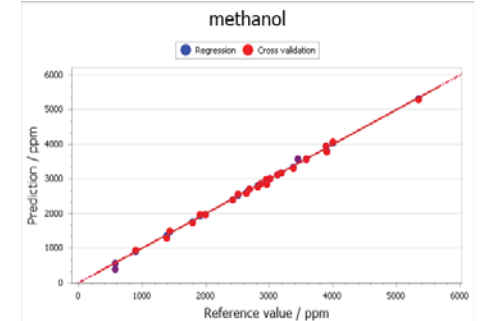


Fig 4. MLR curve for the measurements of methanol quantity in samples which consist of methanol, sugar and ethanol (sample set 4 from the Table 2). Sugar content is 0 - 352 g/l

Table 1. Calibration errors, ethanol content

		Ethanol, % vol.	Methanol, ppm	Total sugar, g/l	RMSECV(*), % vol.
Sample set 1	Fig. 1	8 - 96	0	0	<0.1
Sample set 2		8 - 96	0	0 - 21	<0.17
Sample set 3	Fig. 2	12 - 60	0	100 - 320	<0.1
Sample set 4	Fig. 3	16 - 43	550 - 5350	0 - 352	<0.09

Table 2. Calibration errors, methanol content

		Ethanol, % vol.	Methanol, ppm	Total sugar, g/l	RMSECV(*), ppm
Sample set 4	Fig. 4	16 - 43	550 - 5350	0 - 352	<70

(*) RMSECV – the root mean square error of cross-validation.

(**) MLR – the multiple linear regression