

Infrared spectroscopic application using an integrating sphere for measuring vapor Ethanol

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Summary: We demonstrate a near infrared (NIR) spectroscopic application for measuring vapor ethanol within the internal of an integrating sphere. The optical setup uses a NIR distributed feedback laser (DFB) at 2274 nm wavelength, where Ethanol presents high absorption coefficient. The measurements show a discrimination of different concentration of vapor ethanol with remarkable low noise level.

Keywords: near infrared, integrating sphere, distributed feedback laser, vapor ethanol.

1. Introduction

Integration spheres are known to be beneficial in some spectroscopic applications, especially in scattered reflectance or transmittance measurements [1]. Moreover, integrating spheres are beneficial in gas sensing applications, as they easily increase the effective optical path length from the light source to the detector. Hence, the interaction length between light and gas sample becomes higher [2]. In the work presented here, an integrating sphere is used to measure ethanol vapor in air, with a distributed feedback laser (DFB) at 2274 nm wavelength, which is close to an absorption band of ethanol.

2. Measuring vapor Ethanol

In order to measure different concentrations of vapor ethanol that exist within the internal an integrating sphere, the setup shown in Fig. 1 was used. Cotton wool samples immersed in ethanol solutions of defined concentrations (0.00%, 9.38%, 18.75%, 37.50%) were used, one at a time, to provide the vapor ethanol within the sphere. During the sample preparation, ten minutes were needed until the cotton wool was fully soaked with liquid. To clean the integrating sphere from remaining ethanol vapors after each sample mounting, the integrating sphere was flushed with fresh air after each measurement. This was done using a little air pump for several minutes through the sample port, while no sample was mounted. Specifically, the procedure protocol for the measurement of different ethanol concentrations was as follows: 1.sample port closed for 5 min, 2.sample port open and measurement of reference for 3min, 3.sample mounted and measurement of vapor ethanol for 5 min, 4.ventilation of integrating sphere with air pump for 3 min, 5.repetition of step 2-4 for all ethanol concentrations, 6.sample port open for 3 min to get an idea of the overall drift, 7.sample port closed (>5 min).

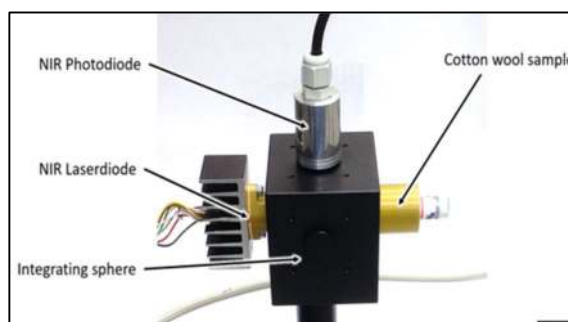


Fig. 1. Optical setup for measuring different vapor ethanol concentrations using NIR.

Before running any test with this setup, an Allan-Werle measurement has been conducted to confirm that the setup works and is sufficiently stable. For the Allan-Werle measurements all ports of the integrating sphere were closed except those for the Laser and the detector. The used light source was the Sacher Laser (DFB-2274-002) because ethanol shows high absorption at its wavelength (2.274 μ m). The achieved Allen-Werle plot is depicted in Fig. 2. The minimum can be found at roughly 4 sec of integration time, where the noise level in general is acceptable low.

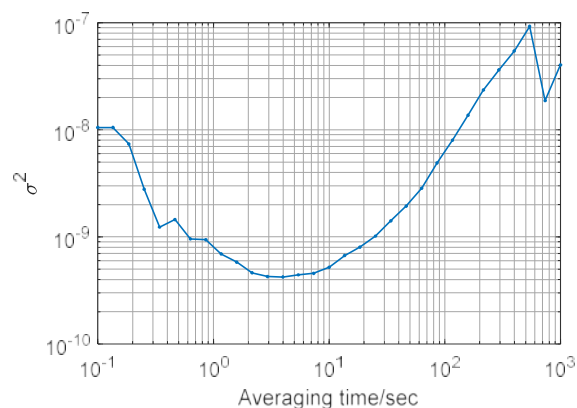


Fig. 2. Allan-Werle variance of the optical setup.

The result of the experiment that follows the measurement procedure stated above, is shown in Fig.3. It is noticeable, that there is almost no difference between the sample port opened and the sample mounted. Thus, it can be concluded that the sample absorbs almost all of the light, no matter if ethanol is present or not, this is something that verifies the absorption peak at $\sim 2.3\mu\text{m}$ of cotton sample [3]. However, with increasing ethanol concentration, a difference in the signal shape over time can be determined.

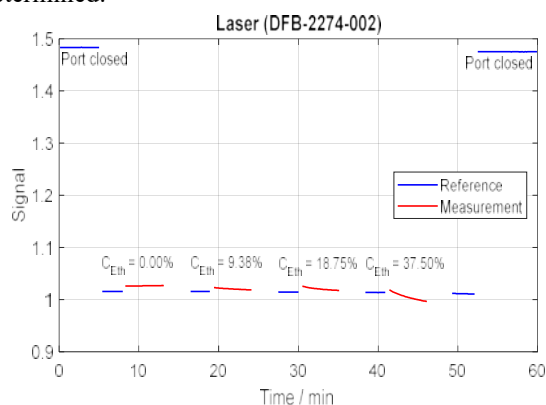


Fig. 3. Measurement result for different vapor ethanol concentrations.

Fig. 4 shows the excerpt of each concentration measurement (red lines in Fig. 3) referenced to the reference level (blue lines in Fig. 3) taken right before. It can be seen that the signal level right after the sample has been mounted does not give any information on the actual ethanol concentration of the sample. However, over time, the signal level decreases. This effect is stronger for samples with higher ethanol concentration, where ethanol vapor fills the integrating sphere and leads to higher light absorption due to multiple reflections within the integrating sphere, resulting to a decrease of the detected signal level.

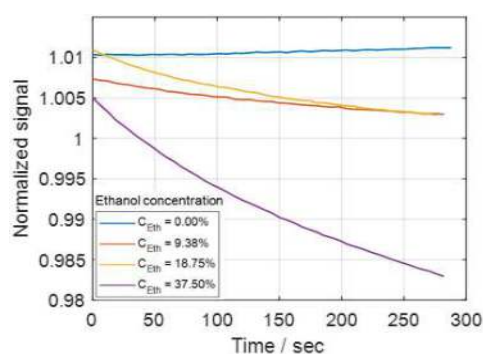


Fig. 4. Excerpt graph from measurement results in Fig. 3

As discussed, the initial signal value after the sample is mounted does not give any information on the ethanol concentration on the sample. However, the initial value of the graphs in Fig. 4 have been used as baseline and shifted to 1 (Fig. 5). It is possible to distinguish between different ethanol concentrations and it is remarkable, that the noise level is quite low. The slight signal increase of the 0% ethanol

measurement originates either from any kind of drift in the setup or from the fact, that humidity in the integrating sphere increases over time. The former however appears more reasonable, because there is no good explanation why increasing humidity should increase and not decrease the light intensity. From that, we conclude, that the optical setup, the used electrical components, and the signal processing software [4] are well suited for this type of application. From the graphs in Fig. 5 one can estimate that the limit of detection for this method is in the range of 5%vol ethanol concentration.

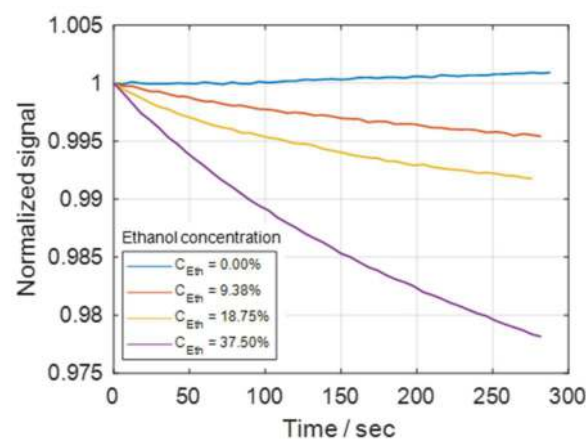


Fig. 5. Measurement results from Fig. 3, normalized and baseline corrected.

3. Conclusions

It has been demonstrated that is possible to distinguish between different vapor ethanol concentrations existing within the internal of an integrating sphere, with a limit of detection at around 5%vol ethanol.

Acknowledgements

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