

# OPTICAL TESTING THE COMPLIANCE WITH REGULATORY STANDARDS OF UREA CONCENTRATION IN AdBlue®

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**Abstract** This paper presents an absorption-based optical approach to check the compliance of urea concentration in AdBlue® with regulatory standards. The implemented low-cost and compact measuring system exploits two LEDs that emit in the SWIR spectral band, a glass capillary and an amplified photodiode to detect the intensity of the radiation transmitted through the fluid under test. The functionality of the system has been demonstrated on urea–water solutions with urea concentrations from 0 up to 525 mg/mL. Thanks to the attained calibration curve, we retrieved the urea concentration of different AdBlue® samples, found in agreement with HPLC results. The proposed optical method allows to easily verify if the estimated urea concentration falls within the required range for AdBlue®.

**Keywords:** Absorption measurement, AdBlue®, LED, Optical sensing, Photodiode, Urea

## 1. INTRODUCTION

Contactless and label-free detection of urea concentration in solution is of significant relevance in chemical, biomedical, industrial, and automotive sectors [1-2]. In automotive applications, accurate monitoring of urea concentration in diesel exhaust fluid, also known by the registered trademark AdBlue®, is essential. This solution ideally consists of 32.5% w/w of urea and 67.5% w/w of demineralized water [3]. Adding this fluid in new generation diesel engines with selective catalytic reduction (SCR) technology, the emissions of polluting gases, such as nitrogen oxides, are reduced. This work introduces a compact and low-cost instrumental configuration for label-free, reagent-free, and contactless detection of urea concentration in AdBlue®, for checking its compliance with regulatory standards, exploiting the absorption properties of urea in the short-wave infrared region (SWIR).

## 2. METHODS AND PROCEDURES

The functionality of the implemented optoelectronic measuring system has been demonstrated on urea–water solutions with urea concentrations from 0 up to 525 mg/mL (the latter corresponding to 34.4% w/w of urea) and then applied for testing the composition of two samples of AdBlue®, purchased from two different gas stations. The intensity of the radiation transmitted through the fluid under test, contained in a rectangular hollow glass capillary with an optical pathlength of 1 mm, is detected in two spectral bands. Two low-cost, low-power LEDs with peak emission wavelength at  $\lambda_1 = 1450$  nm (LED1450, model LED1450L by Thorlabs, Newton, NJ, USA) and  $\lambda_2 = 2350$  nm (LED2350,

model LED2350P by Thorlabs, Newton, NJ, USA) driven by an ON-OFF modulated current, were used as readout sources. Detection of the transmitted light was achieved using an amplified InGaAs photodiode (DET10D2 by Thorlabs, Newton, NJ, USA) placed on the other side of the capillary, in front of the LEDs, maintaining a source-to-detector distance below 1 cm, to minimize optical losses, as shown in Figure 1.

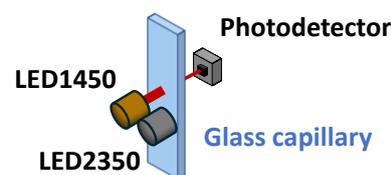


Figure 1. Instrumental configuration scheme.

## 3. RESULTS AND DISCUSSION

The actual transmitted power contribution was determined as the difference between the output voltages provided when the LEDs were ON and OFF. Figure 2 shows 20 ON-OFF cycles of the signals acquired during the absorption analysis of two AdBlue® samples, as well as urea–water solutions with urea concentrations  $C$  (in mg/mL) of 0 (water), 200, and 400, illuminated with both LED sources.

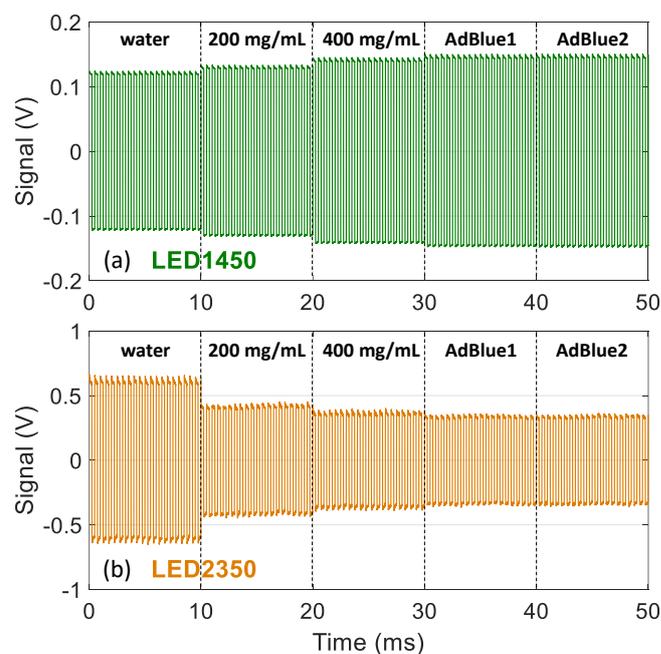


Figure 2. Signals acquired during the absorption analysis of two AdBlue® samples, as well as urea–water solutions with urea concentrations  $C$  (in mg/mL) of 0 (water), 200, and 400, illuminated with: (a) LED1450 (green trace); (b) LED2350 (orange trace).

Transmittance of light generated by LED2350 significantly decreases as the urea concentration in solutions increases, due to the higher absorption coefficient of urea around  $\lambda_2 = 2350$  nm compared to that of water [4]. As water (solvent of all tested solutions) also absorbs in the same spectral region, its contribution was accounted for by measuring the transmittance in the spectral region around  $\lambda_1 = 1450$  nm, where water absorbs, but not urea. Figure 3 presents a magnified view of the signals shown in Figure 2, when water is filling the capillary (solid line) and when AdBlue1 sample is filling the capillary (dashed line).

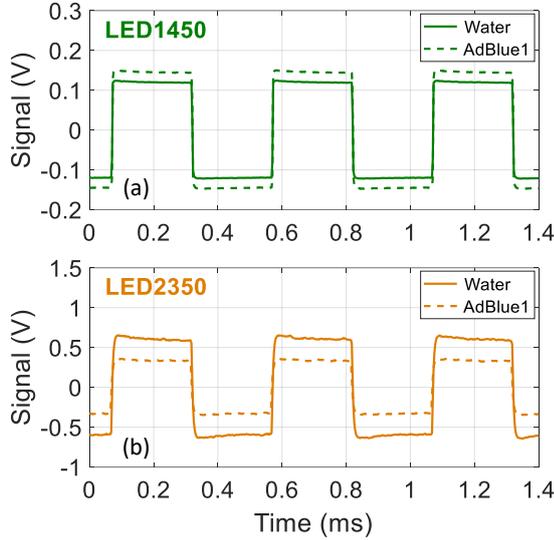


Figure 3. Magnified view of the signals shown in Figure 2, when water is filling the capillary (solid line) and when AdBlue1 sample is filling the capillary (dashed line). (a) LED1450 (green traces); (b) LED2350 (orange traces).

The transmissivities at  $\lambda_1$  and  $\lambda_2$ ,  $T_{1450}$  and  $T_{2350}$ , respectively, were obtained by normalizing the average transmitted intensity over 20 ON-OFF cycles in presence of each tested sample, to the average transmitted intensity in presence of pure water, considered as reference fluid. Figure 4 reports the scatter plot of  $T_{1450}$  and  $T_{2350}$  obtained by testing urea-water solutions (green circle markers) and two samples of AdBlue® (yellow and magenta diamond markers).

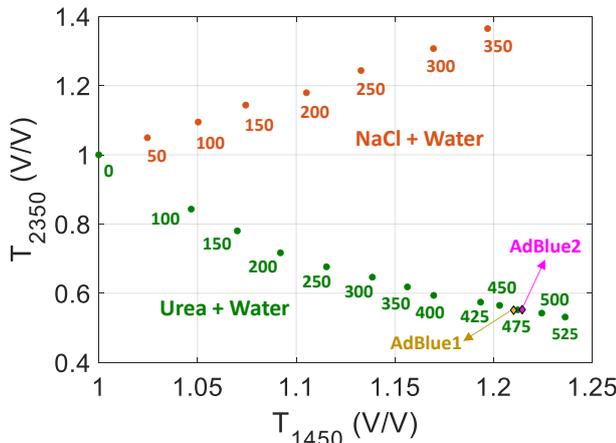


Figure 4. Scatter plot of  $T_{1450}$  and  $T_{2350}$  obtained by testing urea-water solutions (green circle markers), two AdBlue® samples (yellow and magenta diamond markers) and NaCl-water solutions (red circle markers) where the numbers next to each marker refer to the solute concentration in mg/mL.

To highlight the specificity of the measures, we also compared the results with optical measurements performed on NaCl-water solutions (red circle markers). The numbers next to each marker refer to the solute concentration in mg/mL. We also demonstrated that the ratio  $Q(C) = T_{1450}(C)/T_{2350}(C)$ , experimentally obtained, is a significant output variable, specific for urea detection, with good linearity in a wide range of urea concentration  $C$ .

Using the linear equation shown in Figure 5 as calibration curve, we successfully estimated the urea concentration of two AdBlue® samples. The estimated values fell within the expected range for AdBlue®, that is 31.8 – 33.2% w/w of urea, or approximately 466 – 497 mg/mL, in compliance with regulatory standards [3]. The sensitivity of the system is  $S = \Delta Q(C)/\Delta C \approx 2.5 \times 10^{-3}$  mL/mg, and the limit of detection  $LoD = 3 \times \sigma/S \approx 5$  mg/mL, where  $\sigma$  is the standard deviation of  $Q$  obtained in presence of water. Results of the optical measurements were also found in good agreement with data provided by more expensive and complex HPLC investigations on the same samples.

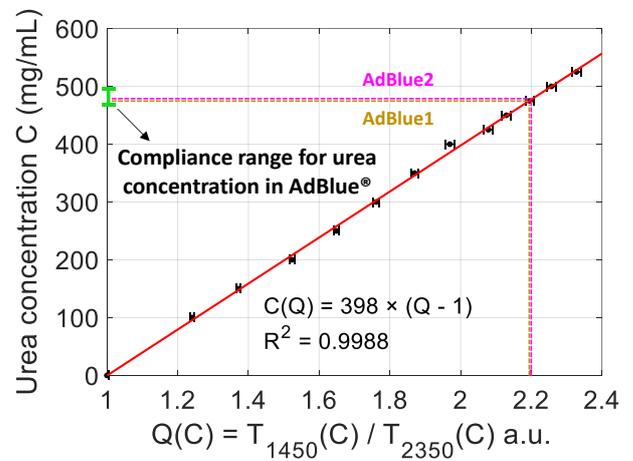


Figure 5. Ratio  $Q(C) = T_{1450}(C)/T_{2350}(C)$  (black markers) and the corresponding calibration curve (red trace) exploited to estimate the urea concentration for each AdBlue® sample.

#### 4. CONCLUSIONS

This work introduces a compact and low-cost instrumental configuration for label-free, reagent-free, and contactless detection of urea concentration in AdBlue®, for checking its compliance with regulatory standards, exploiting the absorption properties of urea in the SWIR spectral band.

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