SWIR absorption spectroscopy based on broadband source for the gas sensing

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Abstract—The paper presents research on short wavelength infrared (SWIR) absorption spectroscopy for gas sensing. The study focuses on detecting methane and ammonia, significant gaseous analytes, using a broadband light source and optical spectrum analyzer. The research demonstrates the potential for detecting methane and ammonia gas. The study addresses the current needs of industrial gas metrology and emphasizes the importance of detecting methane and ammonia due to their environmental and industrial implications.

Infrared spectroscopy is an essential and powerful tool used to detect various gaseous analytes [1-3]. It is mainly used in the mid-infrared range using the NDIR technique and thermal light sources. Recently, we have increasingly encountered systems based on QCL laser sources. Laser detectors are effective, but unfortunately, they are also expensive. The research carried out in this work contributes to the design of near-infrared gas detection systems and constitutes a real answer to the current needs of industrial gas metrology. A measurement system was designed to detect two types of gases: methane and ammonia. The choice of types of gas analytes is not accidental. Methane is a dangerous, explosive gas and the second most important greenhouse gas after CO_2. Methane is also a product of the biomass decomposition reaction in anaerobic conditions. Improperly organized and old landfills may be a source of CH_4 emissions into the environment. Ammonia (NH_3) is one of the most essential substances encountered daily. The major industrial application is in agriculture, specifically in the production of fertilizers. Ammonia is also a necessary indicator in the food industry. Therefore, detecting and measuring its concentration is very important in many industries.

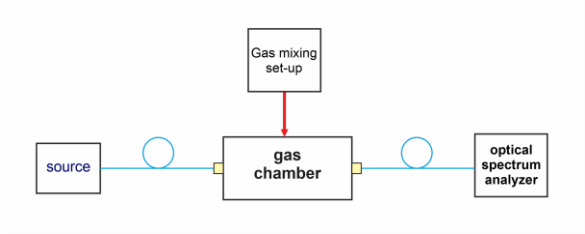

Fig 1. Scheme of the measurement set-up.

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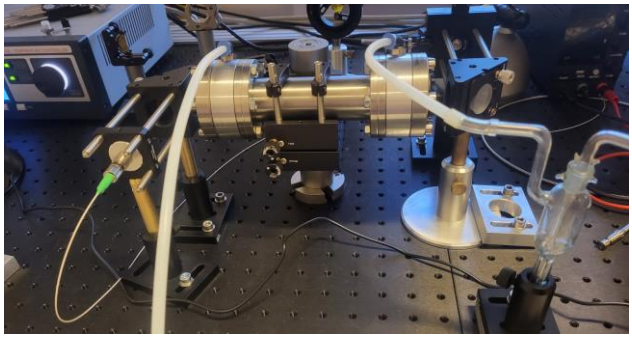

Fig. 2. Measuring gas chamber.

The research used the SWIR absorption spectroscopy method. The measurement system utilized a broadband SLED (SLD1550S-A2, ThorLabs Inc.) light source and an optical spectrum analyzer AQ6370D (Yokogawa Inc.). The measurement system diagram is shown in Fig. 1.

The research was undertaken near the optical telecommunications C band due to the relatively cheap and readily available optoelectronic components. A broadband IR SLED source with a CWL wavelength of 1550 nm and a FWHM of 90 nm was used. In this spectral band of the source, there are several distinct absorption lines of methane and ammonia. These are not lines with strong absorption (approx. 100 times weaker than in the mid-IR band), but the ease of construction of the measurement system and the availability of optoelectronic and optical components can compensate for these limitations.

The measurement gas cell designed and constructed as part of the research (Fig. 2) was built based on a stainless-steel pipe with CF vacuum flanges. The flanges are equipped with specially designed and manufactured reductions containing gas quick connectors for connecting the tested analytes. The whole is closed by flanges with sapphire windows with a diameter of 1 inch. The effective length of the chamber is 181 mm.

Before measuring the specific gas analytes, a procedure was conducted to calibrate the measurement system and determine the relevant spectral ranges in the SWIR band for methane and ammonia gas. The studies of spectral absorption of radiation in the SWIR band were planned in such a way as to measure the change in optical power of

the light beam passing through the measurement cell. This change resulted from the absorption of radiation energy by the gas being studied. The studies were conducted with a constant carrier gas (pure N_2) flow of 200 sccm (standard cubic centimetres per minute) and at a constant temperature of 22°C.

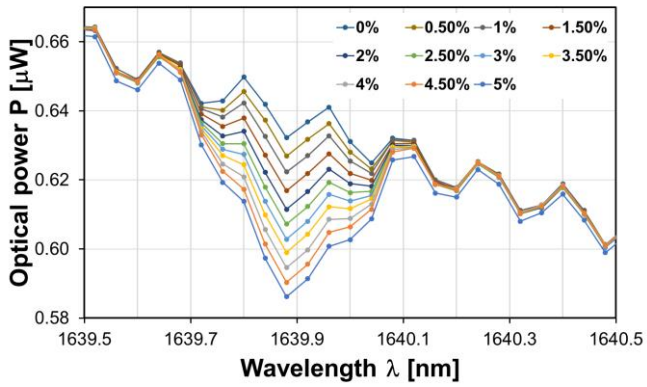

Fig. 3. Methane absorption line spectrum at 1639.88 nm for different concentrations.

As mentioned in the first paragraph, the studies were conducted for two specific gas analytes. The first was methane, which was introduced into the measurement cell in varying volumetric concentrations from 0.5% to 5% vol. in nitrogen. Fig. 3 shows the methane absorption line spectrum at 1639.88 nm for various concentrations ranging from 0.5% to 5%. These measurements were made with a resolution of 0.04 nm.

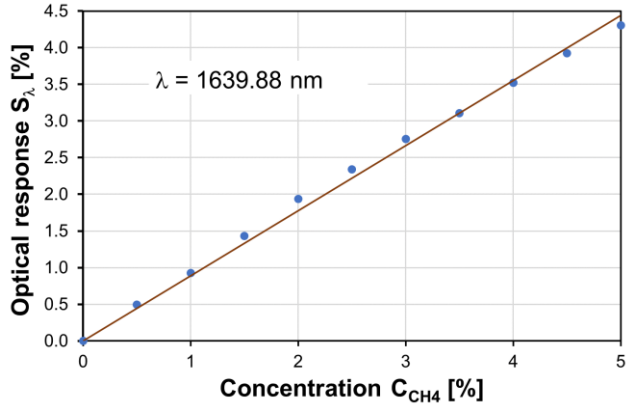

Fig. 4. Optical response for methane absorption line 1639.88 nm depended on concentration.

The results presented show a clear response at a concentration of 0.5%. As shown, this method has a clear potential for detecting methane at concentrations below 0.5% vol/vol. Using the data presented in Fig. 3, the sensor's optical response signal was determined. The measure of the optical signal is the relative change in optical power, determined by the characteristic wavelength. The reference was the optical radiation power recorded under pure nitrogen flow conditions. The optical response signal was determined by:

$$S_\lambda = \frac{P_\lambda^{N2} - P_\lambda^{Analite}}{P_\lambda^{N2}} * 100\% \qquad (1)$$

Figure 4 shows the change in the sensor's optical response S at the wavelength of 1639.88 nm as a function of methane concentration at the range 0.5–5% vol.

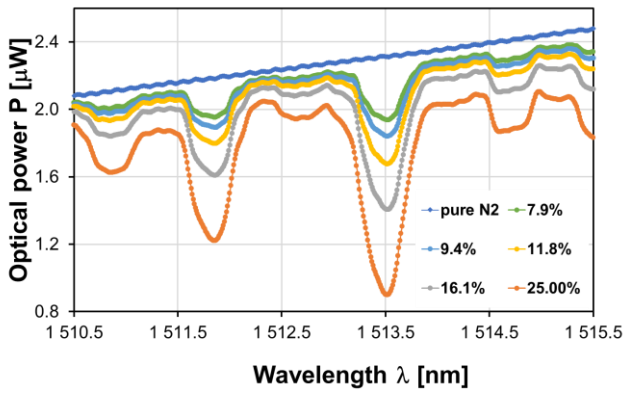

Fig. 5. Ammonia solution vapor absorption for a few lines and different concentrations.

The second analyte was ammonia. The source of gaseous ammonia was ammonium hydroxide, with an initial weight concentration of 25% wt. Under standard thermodynamic conditions, ammonium hydroxide spontaneously releases gaseous ammonia above the free surface of the solution.

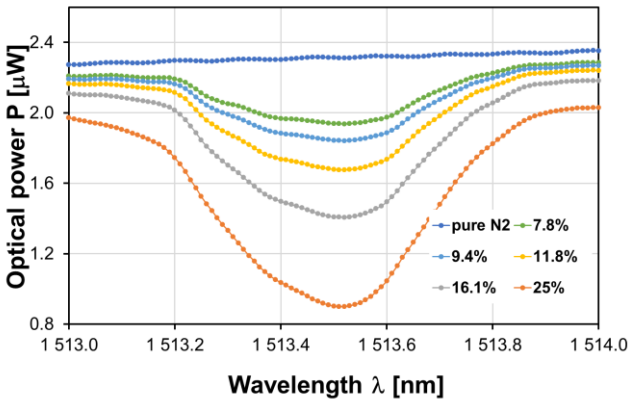

Fig. 6. Ammonium hydroxide vapor (ammonia) absorption line spectrum at the 1513–1514 nm wavelength for different concentrations.

Therefore, to dose varying ammonia concentrations, an ammonia solution was sealed in a glass ampoule through which the carrier gas, nitrogen, flowed. The ammonium hydroxide in the ampoule was diluted with deionized water to several different weight concentrations.

Ultimately, ammonium hydroxide solutions with varying weight concentrations from 7.8% to 25% wt. were used as the source, generating gaseous ammonia (and water vapor) in the carrier gas stream at varying concentrations. The ammonia water vapor test results can be seen in Fig. 5. Figure 6 shows an enlarged section of the characteristic in the region of the strongest absorption around the 1513.5 nm band. These measurements were made at the maximum resolution of 0.02 nm allowed by the AQ6370D

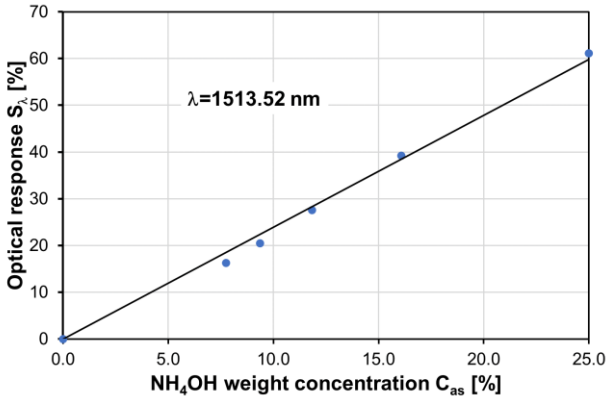

Fig. 7. Optical response S ammonia solution vapor absorption line 1513.52 nm vs. wt. concentration ammonium hydroxide.

Using Eq. (1), similar to the methane case, to determine the optical response of the sensor, Fig. 7 shows the relative change in the optical signal as a function of the weight concentration of the ammonia solution, which was the source of ammonia in a constant nitrogen flow. The signal S was determined for the wavelength of 1513.52 nm.

In summary, the study has demonstrated the potential and feasibility of constructing relatively low-budget sensor solutions for detecting gas analytes in the SWIR band. This was achieved by leveraging solutions available in fiber optic telecommunications technology, adapted to operate near the C-band. Future research will focus on reducing the concentrations of the measured gases and expanding the research setup to include commercial gas sensors. These sensors will be utilized to validate the measured gas concentration levels.

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