



A portable laser-based sensor for detecting H₂S in domestic natural gas

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ABSTRACT

Hydrogen sulfide (H₂S) is a highly toxic gaseous component of natural gas that poses a significant hazard during the use of natural gas for domestic purposes; therefore, a high-sensitivity, *on-line* detection method is extremely important to ensure its safety for domestic use. In this work, a portable sensor was developed based on near-infrared tunable diode laser absorption spectroscopy (TDLAS). A special Herriott multipass cell (MPC) combined with a home-made embedded electronics system allowed for the fabrication of a compact sensor with a size of 50 × 20 × 10 cm³. A rugged movable cell was used for detecting contaminated gas samples. An embedded electronic system with a diameter of 8 cm was implemented to control the laser, acquire and process electronic signal. To reduce external interference, the calibration-free TDLAS (wavelength modulation frequency-2f/direct sine signal) was employed to suppress common field measurement noise. Based on the results of Allan deviation analysis, the limit of system detection could reach 0.14 ppm using the H₂S absorption line at 6336.61 cm⁻¹. It is suitable for *on-line* measurement because of its rapid response time. Its feasibility is validated for monitoring H₂S in a domestic natural gas of pipeline. This work demonstrates a system for *real-time* and *field* measurement of H₂S, which can be used to address the security risks associated with using natural gas.

1. Introduction

Natural gas, whose consumption has been growing steadily over the past two decades, is an important domestic fuel that is used for cooking and heating because it has high efficiency and is clean and safe [1]. Hydrogen sulfide (H₂S), a naturally occurring component of crude natural gas, is colorless, flammable, explosive, and highly toxic. The US Occupational Safe and Health Administration has set a concentration limit of 20 ppm for chronic exposure and a peak limit of 50 ppm for no longer than 10 min, while the International Standards Organization regulates the maximum allowable H₂S concentration of domestic natural gas as 13 ppm. Therefore, it is essential to be able to monitor H₂S in natural gas in *real time* and *in situ* for both industrial and domestic applications [2–6].

A common method for detecting H₂S is reflectometry, wherein the concentration of this gas is indicated by the reflected colors [7]; however, this method is only valid in the case of high concentrations. For low concentrations, a chemical sensor system is used in which H₂S is converted to SO₂, and the concentration of H₂S can be indirectly determined by measuring the SO₂ concentration via UV fluorescence

[5,8]. Additionally, sulfur chemiluminescence, flame photometry, and potentiometry are alternative methods used for measuring H₂S concentration; however, these methods are generally time-consuming and suffer from aging effects, which make them unsuitable for applying in the natural gas industry [9]. Therefore, optical methods which are compact, robust, and highly sensitive are necessary for *in situ* and *real-time* H₂S measurements, in particular for long-distance transmission of natural gas.

In recent years, tunable diode laser absorption spectroscopy (TDLAS) has been successfully employed for monitoring industrial processes, and it is also a promising method for detecting H₂S in natural gas for domestic use [10–12]. Weldon et al. first utilized a near-infrared distributed feedback (DFB) laser for detecting H₂S gas based on the $\nu_1 + \nu_2 + \nu_3$ absorption band [13]. Modugno et al. employed two-tone frequency modulation spectroscopy to realize a sensitivity of 4 ppm and investigate the self and air-broadening of the $\nu_1 + \nu_2 + \nu_3$ band of H₂S [14]. A compact, rugged, and portable fiber-optic evanescent-field laser sensor operating at 1.57 μm was developed for H₂S detection in the gas streams of volcanic fumaroles and had a detection limit of 100 ppm [15]. Chen et al. realized DFB laser off-axis integrated cavity output

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spectroscopy with a sensitivity of 0.67 ppm [16]. Recently, Xia et al. implemented a near-infrared TDLAS combined with a multi-pass cell to measure H₂S and achieved a sensitivity in the ppm range and Tian et al. achieved a sensitivity as low as 60 ppb based on a compact dense-pattern multipass cell [17,18]. A compact photoacoustic spectroscopy setup with a high sensitivity using a quartz enhanced photoacoustic sensor was applied for detecting H₂S in the 1.5 and 2.6 μm region by Varga and Viciani [5,9,19]; however, photoacoustic spectroscopy suffers from gas adsorption and corrosion of the system with use. Additionally, a high-power laser source was required to obtain strong acoustic signals, and the setup is sensitive to disturbances during the monitoring of H₂S in natural gas, such as those caused by mechanical vibrations in harsh industrial environments. There have been a few reports outlining the successful development of H₂S sensors based on TDLAS for *field* measurements. Chen and Song developed the open path H₂S detection system for environmental monitoring and industrial production [20,21]. You and Zhang used near-infrared TDLAS to continuously monitor the H₂S concentration of a desulfurizing device and for natural gas purification [22–24]. However, most previously reported H₂S sensors were bulky and did not consider suppression of external interference such as mechanical vibration, optical-electrical gain, and laser transmission variations [25].

In this work, a portable H₂S sensor was developed based on infrared TDLAS. The WMS-2f/DS-sine method was employed to suppress noise from the circuit, the optical path, and the harsh environments. A special Herriott structure with two reflective mirrors separated from the sampling cell was designed to avoid contamination of the mirrors and allow for easy cleaning of the cell windows. For compactness, a self-made embedded electronics system was developed and served as the DFB laser controller, the lock-in amplifier, the data acquisition and signal processing system, the display, and for serial communication. For further verification, this sensor was deployed for the *in-field* detection of H₂S in natural gas.

2. Experiments

2.1. Measurement principle

To avoid cross interference from other species in domestic natural gas, it is necessary to choose a discrete spectral line with strong absorption intensity. Based on the HITRAN 2016 database, the special lines of H₂S, as well as the main perturbing absorption lines from CH₄, H₂O, and CO₂ are all presented in Fig. 1. Other main components of natural gas, such as C₂H₆ and C₃H₈, have no absorption lines in the observed spectral region. It illustrates that the influence of the perturbing lines can be neglected under the assumed measurement

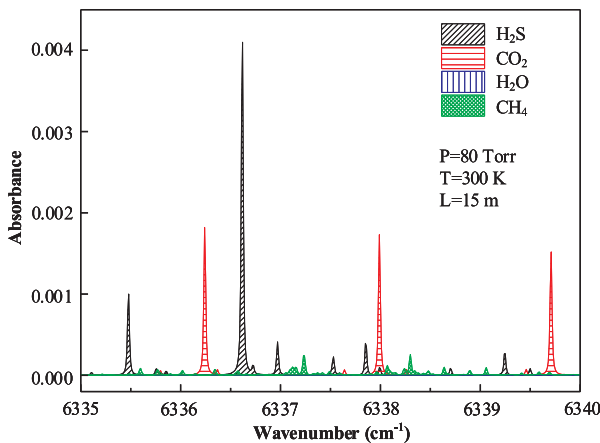


Fig. 1. Calculated spectra for 100 ppm H₂S, 1500 ppm CO₂, 1% H₂O and 85% CH₄ in the range of 6335–6340 cm^{-1} .

conditions. Therefore, the H₂S rotational line at 6336.61 cm^{-1} of the $\nu_1 + \nu_2 + \nu_3$ vibrational band was selected for concentration measurements. Based on the Beer-Lambert law, the transmission coefficient τ is expressed as [26]:

$$\tau(\nu) = \frac{I}{I_0} = \exp[-\alpha(\nu)] \approx 1 - \alpha(\nu), \quad (1)$$

where I and I_0 are the incident and transmitted laser intensities, respectively, and α is the absorption coefficient with respect to laser frequency. For wavelength modulation spectroscopy (WMS), the injection current of DFB laser is modulated with an angular frequency ω [27].

$$\nu(t) = \bar{\nu} + a \cos(\omega t), \quad (2)$$

$$I_0(t) = \bar{I}_0 [1 + i_1 \cos(\omega t + \psi_1) + i_2 \cos(2\omega t + \psi_2)], \quad (3)$$

where $\bar{\nu}$ is the center laser frequency and a is the modulation depth. \bar{I}_0 is peak-peak value of modulation signal detected by the photodiode, termed as DS-sine [28]. The parameters such as i_1 , i_2 , ψ_1 and ψ_2 describe the characteristics of the laser. The spectral absorption α is a periodic even function in ωt and can be expanded in a Fourier cosine series:

$$-\alpha\left(\bar{\nu} + a \cos(\omega t)\right) = \sum_{k=0} H_k\left(\bar{\nu}, a\right) \cos(k\omega t), \quad (4)$$

The H_k coefficient is the Fourier expanding coefficient:

$$H_0\left(\bar{\nu}, a\right) = -\frac{PxL}{2\pi} \int_{-\pi}^{\pi} \sum_j S_j \varphi_j\left(\bar{\nu} + a \cos\theta\right) d\theta, \quad (5)$$

$$H_k\left(\bar{\nu}, a\right) = -\frac{PxL}{2\pi} \int_{-\pi}^{\pi} \sum_j S_j \varphi_j\left(\bar{\nu} + a \cos\theta\right) \cos(k\theta) d\theta, \quad (6)$$

For WMS detection, a lock-in amplifier is used to get harmonic signals based on a quadrature demodulation scheme. For convenience, we matched the condition when the in-phase component signal was maximum. The 2f harmonic signal is approximately represented as:

$$X_{2f} = \frac{GI_0}{2} \left[\frac{i_1}{2} (H_1 + H_3) + \frac{i_2}{2} \left(\frac{H_4}{2} + H_0 \right) + H_2 \right], \quad (7)$$

where G is the optical-electrical gain of the detection system.

At fixed pressure, temperature, optical path length, and laser parameters, the line shape is insensitive to the sample concentration at low absorption conditions (typically less than 5%). Thus, the concentration relationship with 2f harmonic peak can be derived as $C \propto X_{2f}$. To reduce laser instability and the interference of the optical path, a simple normalized method is applied and given by:

$$C \propto \frac{X_{2f}}{I_0}. \quad (8)$$

Thus, all non-absorption transmissions can be ignored during the measurement.

2.2. Experimental details

A schematic diagram of the laser-based H₂S sensor is depicted in Fig. 2(a). A fiber-coupled DFB laser emitting at 1.578 μm was used as a light source. The laser was connected to an optical fiber-coupled GRIN lens for beam collimation; then, the collimated laser was coupled into a home-made Herriott-type multipass cell. The effective optical path-length was 15 m with a cell length of 0.3 m. To protect the reflective mirrors, the sampling cell was separated from both mirrors by two windows with a distance of 5 mm, and the windows of the sampling cell were coated with a dielectric anti-reflective film. Wedged-shaped 2° windows were adopted to suppress the optical interference fringes. The

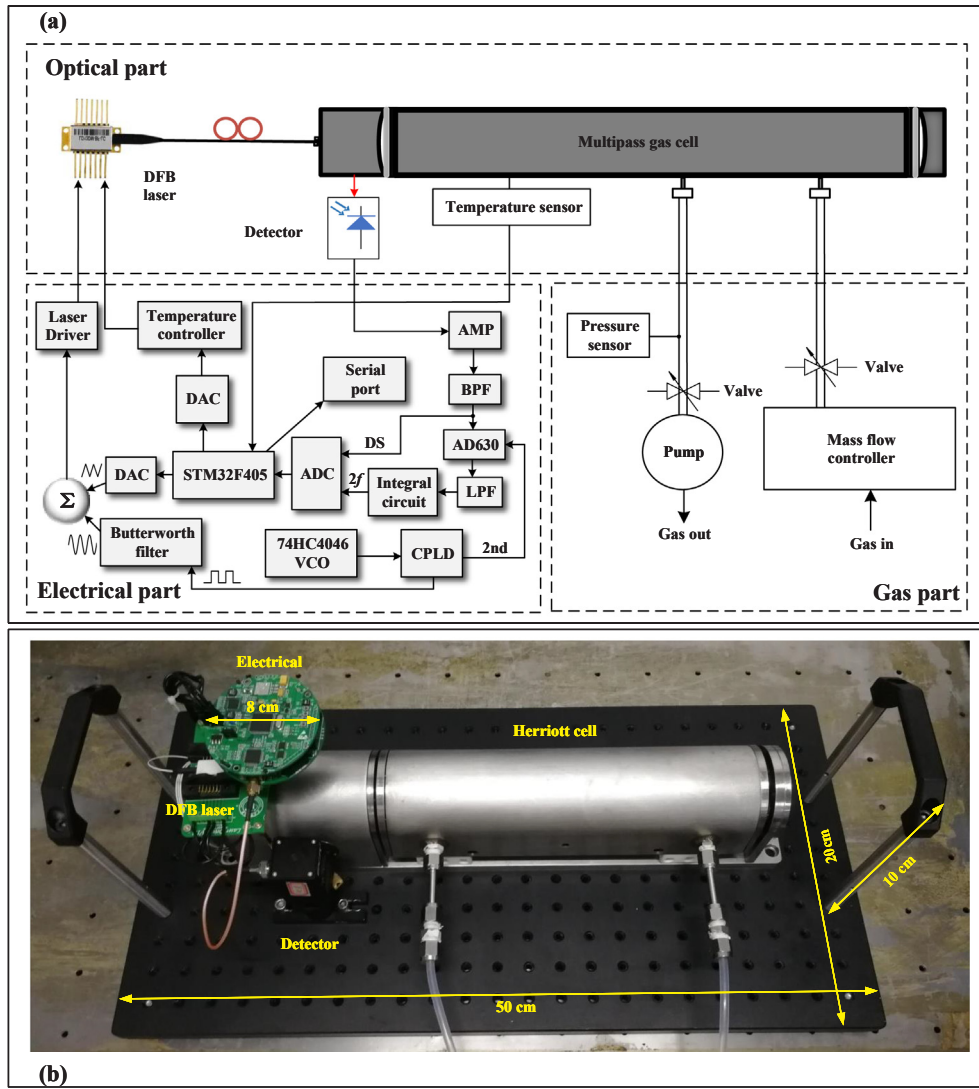


Fig. 2. (a) Schematic of the laser-based H₂S sensor system; (b) photograph of the real instrument.

cell was made of stainless steel to inhibit adsorption and corrosion by H₂S. The internal surface of the cell was polished, and the inlet and outlet pipes were made of polytetrafluoroethylene to suppress adsorption. Gas pressure was measured by Testo552 which was installed at the outlet of gas pipe and temperature was obtained by a DS18B20 sensor embedded at the bottom of the cell, as presented in the gas part of Fig.2(a). Furthermore, the valves were used for auxiliary pressure control of system.

The electrical portion of the system was divided into two parts, one part for the DFB laser drive circuit and the other was the analog to lock-in amplifier circuit. Both PCB boards were 8 cm in diameter (shown in Fig.2(b)), and the main control chip was a Microcontroller Unit (MCU, STM32F405). The current driver circuit based on a Hall-Libbrecht design and the temperature controller was implemented with an ADN8834. The laser wavelength characteristics are $0.014 \text{ cm}^{-1}/\text{mA}$ in the range of 20–70 mA and $0.378 \text{ cm}^{-1}/^{\circ}\text{C}$ in the range of 22–28 $^{\circ}\text{C}$, respectively. The stabilities of the current and temperature controllers were $4 \mu\text{A}$ and $0.0067 \text{ }^{\circ}\text{C}$, respectively. The controller is a home-made instrument. These two parameters were obtained by measuring the error signal with ESCORT-3136A multimeter and the details could be found from our previous published literature [29]. Triangular wave signals were generated by a 12-bit digital to analog converter (AD5683) controlled by MCU, and its amplitude was determined by the spacing of the selected absorption line and the relationship between driving

current and the wavelength. The amplitude of the scanning wave signal was 300 mV, corresponding to a scanning wavelength range of 6336 cm^{-1} – 6337 cm^{-1} ; the repetition frequency was set to 5 Hz; the modulated signal at 7.572 kHz (1f) was generated via a high-speed logic phase-locked-loop with VCO (74HC4046A), and was then sent to the CPLD (Altera, EPM7064AE) for frequency-doubling (2f) and phase-shift. The 1f TTL signal of the CPLD output was filtered into a sine signal using a 5th Order Butterworth low-pass filter. The final sine signal was superimposed on the triangle laser-scanning signal using an adder. The laser intensity after passing through the multipass cell was converted into an electronic signal using a high-speed InGaAs detector (ET-3010). Then, it was amplified by a pre-amplifier and filtered using a band-pass filter (BPF). The analog phase-locked amplifier circuit was implemented using a balanced modulator and demodulator (AD630). Both the 2f signal of the CPLD output as a reference signal and half the BPF output as a modulation signal were sent to the AD630 for synchronous demodulation (i.e., WMS-2f). The output of the AD630 was further filtered using a low-pass filter (LPF). Finally, the filtered and DS-sine signals were simultaneously acquired by two 16-bit analog-to-digital converters (AD7606) and stored in the FLASH of the MCU. The digital signals were processed and calculated using the STM32F405 MCU, and the DS-sine normalized signal was used to determine the concentration of the H₂S gas. Finally, the obtained concentration results were displayed using an LCD and transmitted to the host computer

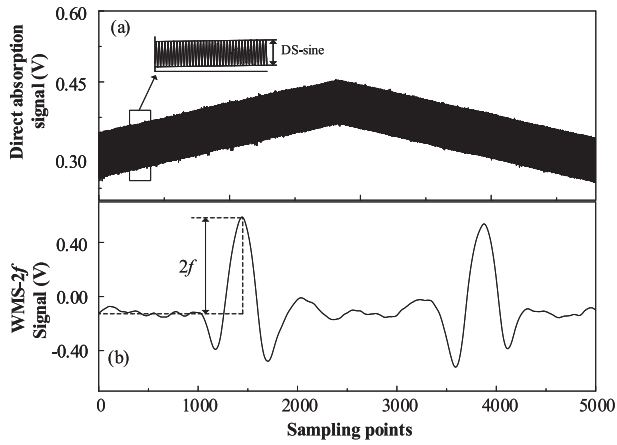


Fig. 3. Observed signals for 100 ppm H_2S is mixed with CH_4 at 80 Torr at a concentration of 100 ppm at room temperature. (a) Direct absorption signal with modulation and DS-sine value (insert figure); (b) WMS-2f signal of H_2S .

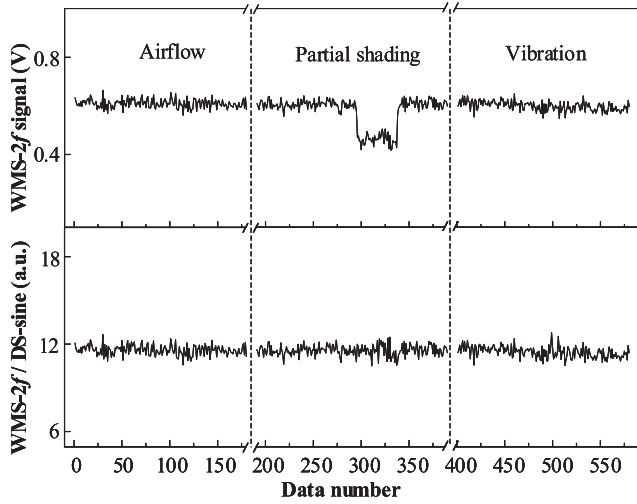


Fig. 4. Variations of WMS-2f and 2f/DS-sine signals with external interference.

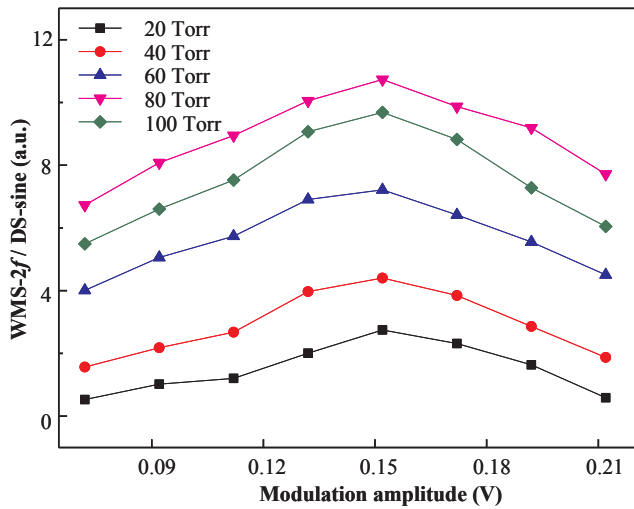


Fig. 5. Observed WMS-2f/DS-sine signal amplitude for 100 ppm H_2S with different modulation amplitudes at five different pressure values.

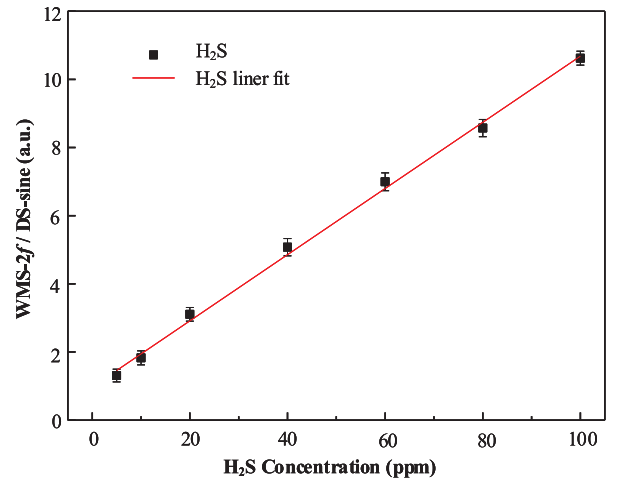


Fig. 6. The WMS-2f/DS-sine signal amplitude of H_2S and fitting curve as a function of H_2S concentration.

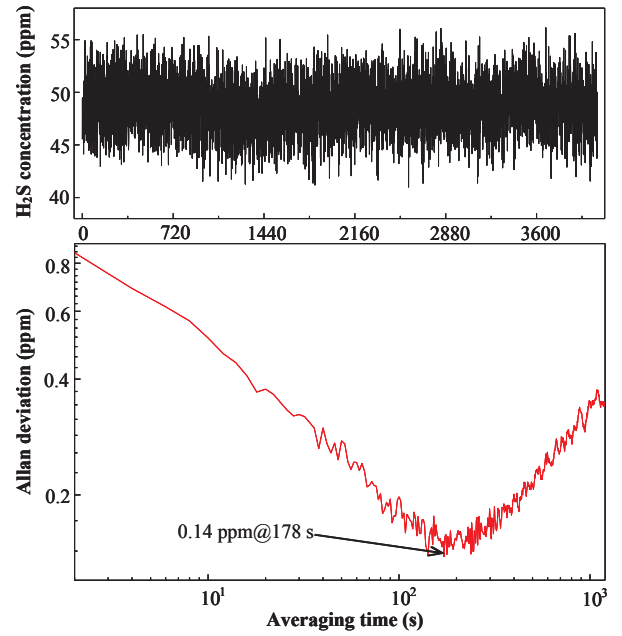


Fig. 7. Obtained signal for a 50 ppm H_2S sample at 80 Torr and 25 °C for a duration of more than an hour and its Allan deviation analysis.

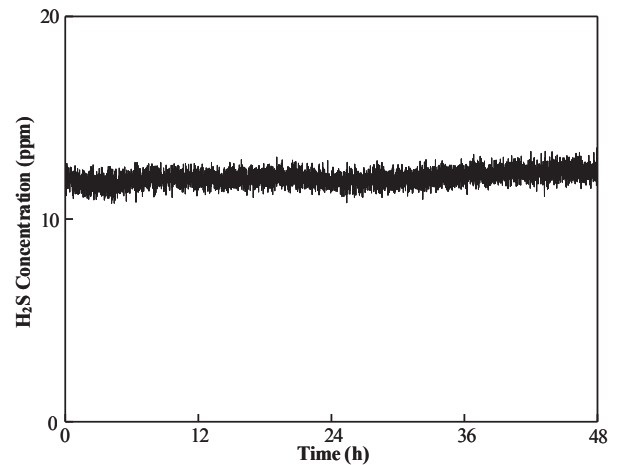


Fig. 8. Continuous measurement of hydrogen sulfide in civil gas for 48 h.

through the serial communication port.

3. Results

The measured DS and WMS-2f signals for a 100 ppm H₂S gas mixture that was pure H₂S mixed with the pure N₂ with the uncertainty of ± 1 ppm sample are shown in Fig. 3(a) and (b), respectively. The results show that the WMS-2f is more sensitive than direct absorption spectroscopy due to the significant reduction of 1/f noise and zero baseline stability [30]. In this experiment, the WMS-2f/DS-sine approach was applied to reduce fluctuations in the optical intensity because of gas flow and mechanical vibrations. The airflow means varying the sample velocity of flow and gas flow was changed from 0.5 L/min to 0.025 L/min using the mass flow controller. Mechanical vibration was generated by knocking the optical breadboard with a hammer, and the partial shading was accomplished by inserting a piece of lens paper. Three sections of interference occurred at the mid-duration lasting 2 min. The corresponding standard deviations of WMS-2f signal for gas flow, partial shading and mechanical vibration are 0.018, 0.016 and 0.020, but they are reduced to 0.017, 0.015 and 0.019 for WMS-2f/DS-sine approach. The results, as shown in Fig. 4, validate the stability and robustness of the sensor with this strategy. While the amplitude of the WMS-2f signal varies significantly, especially for beam blocking, the WMS-2f/DS-sine method is not affected by variation of the light intensity. Therefore, the technique shows promise for *in situ* measurement applications.

The amplitude of the wavelength modulation signal is relevant for both the modulation index and the pressure, so it is necessary to select optimal conditions [31]. Fig. 5 shows a series of WMS-2f/DS-sine signals for H₂S at different pressures and modulation amplitude values. The measured sample H₂S blending with pure N₂. By recording the H₂S spectra at 100 ppm of different pressures, the maximum signal for H₂S was realized at 80 Torr and 0.152 V. The amplitude of wavelength modulated harmonic signal depends on the modulation depth relevant with modulation amplitude and spectral width. The width of spectral line, herein, is dominated by Doppler broadening and pressure broadening, so the signal amplitude drops at 100 Torr and higher pressure. A similar phenomenon could be found in our previous publication [26].

The H₂S gas sensor was calibrated using a similar method to the reported in Ref. [32]. In calibration process, the CO₂ concentration was 15 times more than H₂S, mixture was used for verifying whether H₂S adsorbs on the inner cell and gas tube or not. This is because CO₂ absorption line close to that of H₂S and it is without adsorption characteristic. It demonstrated that the adsorption of H₂S could be neglected in measuring the flow H₂S sample according to the observed CO₂ signals. A series of samples with H₂S concentrations in the range of 1 to 100 ppm were measured using the WMS-2f/DS-sine method. The obtained data was plotted as a function concentration of H₂S mixed with pure N₂ at 80 Torr and 25 °C, as shown in Fig. 6. The linear relation between concentration (C) and the WMS-2f/DS-sine can be given by $C = (\text{Peak}(2f/\text{DS-sine})) - 0.97198)/0.09710$, where the coefficients are determined by fitting. The R² of the fitting is 0.997, and the adsorption of flowing H₂S gas can be neglected in the measured range. The error bar was given through calculating the average of obtained signals and their responding standard deviation. To estimate the detection limit and the system stability, 50 ppm H₂S was continuously measured for more than one hour. Fig. 7 presents the Allan deviation analysis of the measurement. The results indicate that the optimum integration time is 178 s and the corresponding detection limit was 0.14 ppm. In addition, the durations for the rise and fall processes while exchanging the gas sample were 6.4 s and 9.2 s, respectively.

The laser-based sensor was validated by monitoring the H₂S concentration of domestic natural gas provided by Taiyuan Gas Co. The signals, depicted in Fig. 8, were averaged 15 times and their temper interval was 3 s, the measured data for each 3 s was 12 ppm and the variation of the H₂S concentration was less than 2 ppm. The obtained

results demonstrated that values derived by continuous and *real-time* measurement satisfied the standards set by document No.9 of the national standards management committee of the People's Republic of China in 2012. Therefore, the developed laser-based H₂S sensor with a high sensitivity and rapid response can be used to improve safety during the transportation and domestic use of natural gas.

4. Conclusions

In this paper, a laser-based H₂S gas sensor was constructed based on near-infrared TDLAS. A compact sensor of 50 × 20 × 10 cm³ could be realized using a home-made embedded electronics system and a Herriott cell. The WMS2f/DS-sine free calibration method was employed for reducing external interference. Allan deviation analysis demonstrated that the sensitivity could reach 0.14 ppm with an integration time of 178 s. Finally, the sensor was used for continuous measurement of domestic natural gas for 48 h. The results demonstrated that the sensor had a high sensitivity and fast response. Therefore, it is suitable for application to the continuous *on-line* detection of H₂S in domestic natural gas.

Declaration of Competing Interest

None.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at <https://doi.org/10.1016/j.infrared.2019.103153>.

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