

Measurement of CO₂ concentration based on supercontinuum laser absorption spectroscopy*

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The concentrations of CO₂ were measured by the supercontinuum laser at normal temperature and variable temperature accurately in this paper. The absorption spectra of CO₂ at different concentrations (1.2%—9.0%) were measured in the wavelength range of 1 425—1 445 nm under the optical path of 26.4 m at 293 K and 1 atm. The experimental results showed that the positions of the primary and secondary absorption peaks (1 432 nm, 1 437 nm) were consistent with the HITRAN database. A linear model of concentration and signal intensity at 1 432 nm was established. The maximum relative error of the concentration measurement was 3.3%. The line intensities of 99.9% CO₂ in the 1 425—1 445 nm at different temperatures (298—373 K, interval of 15 K) were measured. The influence of temperature changes on the concentration measurement result was corrected and the relative error of the concentration measurement was reduced to 1.4%. Finally, the source of the uncertainty of the entire spectrum measurement system was analyzed and evaluated. This paper demonstrates that the supercontinuum laser can achieve the long-distance measurement of the CO₂ under normal temperature or variable temperature environment accurately, which provides an important reference for the long-distance gas detection on site and simultaneous detection of multi-component gases.

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CO₂ is considered as the main cause of global warming, which causes climate change and numerous ecological problems. Anthropogenic activities such as burning fossil fuels, and biomass burning are the major sources of CO₂. Therefore, the measurement of CO₂ concentration is of great significance. Nowadays the gas is measured mainly by the spectral technology, which is an effective detection technique with high sensitivity, wide detection range and strong practicability.

The common infrared spectroscopy technologies include tunable diode laser absorption spectroscopy (TDLAS), Fourier transform infrared spectroscopy (FTIS), photoacoustic spectroscopy (PAS), and supercontinuum laser absorption spectroscopy (SCLAS). TDLAS has the advantages of high sensitivity and fast response speed, but the wavelength tuning range of the light source is narrow, thus only a single type of gas could be measured and the application range is limited^[1,2]. FTIR technology has high measurement accu-

racy and it can be used to measure the multiple gas components at the same time, but the complexity of the system structure leads to low efficiency of the work^[3]. PAS is a detection technology which uses the acoustic resonance mode of operation in the measurement. It is susceptible to noise, airflow and other surrounding environments during measurement, and the working mode is not easy to maintain. Compared with these three technologies, SCLAS has the advantages of convenience, high sensitivity and strong stability. High-precision measurement of gas absorption spectra can be achieved by the laser light source, which has high brightness, easy collimation, wide spectral range, and strong directionality^[4].

As an emerging non-invasive optical detection technology, SCLAS has received extensive attention in the fields of biomedical, metrology and spectroscopy. Ji-hyung et al^[5] used the SCLAS to measure the near-infrared absorption spectrum of low-concentration

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hydrocarbon gas under the optical path of 30 cm. The experimental results were consistent with the HITRAN database, which demonstrated the ability of supercontinuum lasers to detect broadband absorption spectra. Stelmaszczyk *et al.*^[6] successfully applied supercontinuum laser to cavity ring-down spectroscopy and measured the absorption spectrum of 2 ppm NO₂. Amiot *et al.*^[7] used a mid-infrared supercontinuum laser of 3 000—3 450 nm combined with cavity enhanced absorption spectroscopy to detect multi-component gases such as acetylene and methane simultaneously. The experiment proved the feasibility of the method. Wagner *et al.*^[8] used SCLAS for combustion diagnostics to achieve the measurement of component concentrations and other gas phase combustion parameters. Zheng Lin *et al.*^[9] used the supercontinuum laser to study acetylene with a concentration of 4 000 ppm in a short optical path of 50 cm. Due to the influence of the light source noise, the absorption line cannot be detected directly, and the experimental results obtained were not satisfactory.

In summary, the above scholars have studied related hydrocarbon gases such as methane, ethylene and acetylene based on the SCLAS, but few research has been reported on the concentration detection of CO₂. At the same time, the accurate measurement of CO₂ has great practical significance for the current treatment of environmental pollution. Aiming at the problem that it is difficult to measure the CO₂ concentration accurately in environmental monitoring, this paper uses the supercontinuum laser to measure the CO₂. In addition, Wagner *et al.*^[8] and Zheng Lin *et al.*^[9] selected the optical path of absorption cell in the range of 41 mm—50 cm in the near-infrared spectroscopy study of gas. The long optical path can improve the accuracy of measurement and reduce the detection limit of the system directly and effectively. Therefore, this topic combines SCLAS with long-path absorption technology to study the absorption spectrum of CO₂. A super-continuous spectrum laser detection system with simple structure and high measurement accuracy was developed. The system realized the measurement of CO₂ concentration accurately and verified the characteristics of SC laser under long-distance transmission conditions. The measurement model of CO₂ concentration at normal temperature and pressure was obtained through experiments. The maximum relative error of concentration measurement was 3.3%. The effect of temperature on the absorption spectrum of CO₂ was researched. According to the evaluation method of uncertainty by Cordero *et al.*^[13] and Julicer *et al.*^[14], the sources of uncertainty in the experimental system were analyzed and evaluated. It provides a new method and new means for realizing the long-distance gas detection in the field by the SCLAS.

Spectral measurement techniques are divided into direct absorption spectroscopy (DAS) technology and wavelength modulation spectroscopy (WMS)^[15]. DAS is a direct measurement technique that does not require

calibration. It is convenient, fast and selective. It is also easy to use in the measurement of gas. When the laser passes through the gas to be tested, the light intensity will be attenuated, and the information about the gas concentration to be measured can be analyzed by the change of the light intensity before and after the attenuation. The Lambert-Beer law is the theoretical basis for the absorption spectroscopy^[16]. The schematic diagram is shown in Fig.1.



Fig.1 Schematic diagram of Lambert-Beer law

The Lambert-Beer law is shown below:

$$I_t(\nu) = I_0(\nu) \exp[-S(T)g(\nu - \nu_0)PNL], \quad (1)$$

where $I_t(\nu)$ and $I_0(\nu)$ are the frequency-dependent transmitted and incident intensities, respectively, $g(\nu - \nu_0)$ is the lineshape function of the spectrum profile, ν_0 is the central frequency of the absorption band; P is the gas pressure; N is the gas concentration to be measured; L is the absorption path length of the gas. The gas concentration N can be derived as:

$$N = \frac{-\ln\left(\frac{I_t}{I_0}\right)}{S(T)g(\nu - \nu_0)PL}. \quad (2)$$

The lineshape function in Eq. (2) satisfies

$$\int_{-\infty}^{+\infty} g(\nu - \nu_0) d\nu = 1. \quad (3)$$

Therefore, Eq.(2) is simplified as below:

$$N = \frac{\int_{-\infty}^{+\infty} -\ln\left(\frac{I_t}{I_0}\right) d\nu}{PS(T)L} = \frac{A}{PS(T)L}. \quad (4)$$

In the above Eq.(4), A is the integral of absorbance. So the absorbance of the gas to be tested can be expressed as below:

$$A = PS(T)LN. \quad (5)$$

The measurement range of the absorption spectrum is particularly important for the measurement of CO₂. The source of the supercontinuum laser has a spectral range of 400—2 400 nm. The wavelength range is 1 000—1 700 nm after passing through the laser line tunable filter (LLTF) and by avoiding the position of the background spectrum that has the peaks and valleys, the wavelength range becomes 1 280—1 700 nm. According to the HITRAN database, thus the wavelength 1 280—1 700 nm (wavenumber is 5 882—7 813 cm⁻¹) is selected to study the CO₂. The absorption spectrum of CO₂ in the 5 882—7 813 cm⁻¹ is shown in Fig.2(a) and 6 920—7 017 cm⁻¹ is shown in Fig.2(b). It can be found that the CO₂ has two distinct absorption peaks at the wavenumbers of 6 983.24 cm⁻¹ and 6 958.94 cm⁻¹ (the wavelength is 1 432 nm and 1 437 nm). Therefore, on the basis of avoiding the influence of other interfering gases,

the wavelength range of 1 425—1 445 nm is finally selected to measure the absorption spectrum of CO₂.

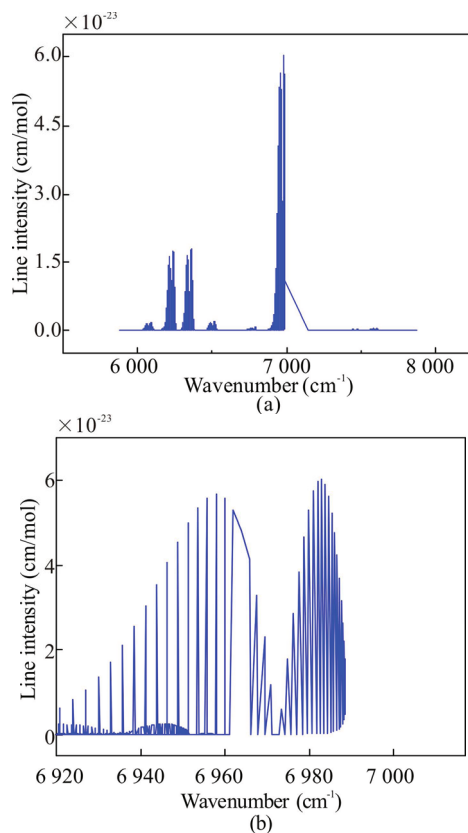


Fig.2 Absorption spectra of CO₂ within the ranges of (a) 5 882—7 813 cm⁻¹ and (b) 6 920—7 017 cm⁻¹

The supercontinuum laser detection system includes a supercontinuum laser, an LLTF, a diaphragm, a dynamic dilution calibrator, a white cell, a photodetector, a data acquisition card (DAQ), and a computer (PC). The supercontinuum picosecond pulse laser SC400-4 with wide spectrum range, high brightness and strong stability is selected as the laser light source, which is produced by the Fianium company in the United Kingdom. The supercontinuum laser (type SC400-4, Fianium, UK), which is a picosecond pulsed and with the wavelength between 400 nm and 2 400 nm and maximum output power of 8 W, was used and its wavelength filtered by the LLTF. The LLTF is used in the experiment, which is the light source's spectroscopic accessory in this system. The output spectral range is 1 000—2 300 nm, which has a high damage threshold and long lifetime. The spectral bandwidth of LLTF is 3 nm, the tuning step set by LLTF is 0.04 nm, the wavelength tuning resolution is 0.1 nm, and the wavelength scanning speed is 1 000 nm/s. The dynamic dilution calibrator employs two mass flow meters with high precision to control the gas flow rates, thus obtaining the desired concentration of the test gas. The flow measurement accuracy is $\pm 1.0\%$, the flow control repeatability is $\pm 0.2\%$, and the flow measurement linearity is $\pm 0.5\%$. The diaphragm is a GCM-5711M variable square aperture dia-

phragm, which can filter out the surrounding stray light, thereby adjusting the intensity of the passing beam. The photodetector is mainly used to detect the characteristic light signal absorbed by the gas. This system uses PDA50B germanium tube detector produced by American THORLABS company, with a sensitive spectral range of 800—1 800 nm, a gain range of 0—70 dB, a response time of 50 ns. It also has a high response coefficient, fast photoelectric conversion rate and low system noise.

In order to improve the sensitivity of the detection system, the gas absorption cell adopts the white cell structure. The white cell in the system is produced by American Infrared Analysis, Inc. The cell is 35-V-H and the material is borosilicate heat-resistant glass. The volume is 8.5 L and the optical path range is 2.2—35 m. The incident laser was reflected multiple times in the cell to increase the optical path length, thereby achieving the purpose of gas circulation absorption. The reflection process of the laser in the cell is shown in Fig.3, and the schematic of the experimental setup is shown in Fig.4.

The concentration experiment was carried out under the conditions of 293 K at room temperature and 1 atm. The supercontinuum laser was generated by a supercontinuum laser and filtered by the LLTF, so that the output laser beam wavelength was scanned at 1 425—1 445 nm. After the stray light around the beam was filtered by the diaphragm, the laser was into the white cell. The laser was reflected many times in the cell and then received by a photodetector. The optical path of the cell is 26.4 m. The photodetector convert optical signals into electrical signals and transfer it to the DAQ. The reception and storage of the data were performed by the DAQ and PC. During the experiment, high-purity nitrogen (99.999%) was first introduced into the white cell, and the measured signal was used as the background signal. Then, the CO₂ with concentrations of 1.2%, 2.2%, 3.0%, 5.0%, 7.0%, and 9.0% was sequentially introduced into the absorption cell through a dynamic dilution calibrator. To ensure the accuracy of the experimental results, the cell was purged with high-purity nitrogen gas after the concentration was measured each time, after which the gas to be tested was introduced.

The cell was equipped with a heating temperature control device for the temperature experiment. When the heating device was working, the gas and the window were heated, and the temperature of the adjusting device for optical path and the fixing device were kept unchanged. The cell and the optical path were separated by an insulating plate, and the cavity was heated by the heating sleeve, the temperature can be detected by the thermocouple in real time. The temperature could be controlled accurately by the intelligent temperature controller. In the heating process, the factors that affecting the absorption line of CO₂ were not only reflected in the changes of temperature, but also the influence of the pressure changes. In the experiment, the white cell had a pressure release valve, which was used to keep the internal pressure constant. The experiment was carried out at a

standard atmospheric pressure of 1 atm. Firstly, nitrogen (99.999%) was introduced into the cell to purge the absorption cell, and then high purity CO₂ (99.9%) was introduced into the cell. The white cell was heated by the thermostat, and the initial temperature was set to 298 K, and then the temperature was increased by a step value of 15 K until it was heated to 373 K. After the set temperature was reached each time and it was kept for 30 min to ensure that the gas was evenly heated. Data collection was performed after the heating was stabilized.

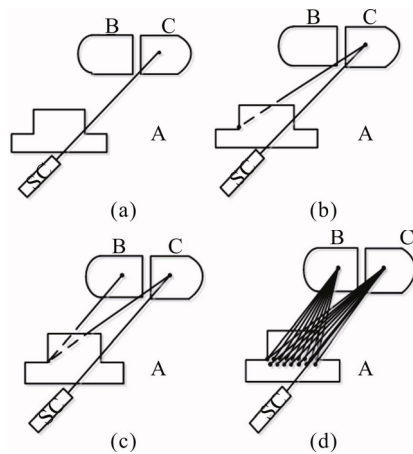
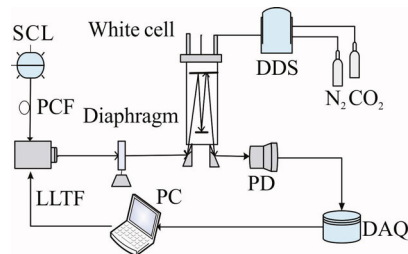


Fig.3 The reflection paths of laser in the white cell (A is the plane mirror, and B and C are the concave mirrors.)



SCL: supercontinuum laser; PCF: photonic crystal fiber; LLTF: laser line tunable filter; DDC: dynamic dilution calibrator; PD: photodetector; DAQ: data acquisition card

Fig.4 Schematic of the experimental setup

The data acquisition program of Labview was used to store data on the PC, and the sampling frequency was set to 300 Hz. The background subtraction method was used to eliminate the influence of low-frequency background noise such as baseline drift. After subtracting the background, the direct absorption signal of 1 425—1 445 nm CO₂ at different concentrations in Fig.5 was obtained. Fig.2 is the absorption line intensity of CO₂ derived from the HITRAN database at 296 K and 1 atm. Fig.5 shows the absorption signal of CO₂ gas molecule spectrum, which has subtracted the experimental background. The difference between the two figures is that Fig.2 is the theoretical absorption spectrum of gas molecules, and Fig.5 is the measured absorption spectrum. The experimental results showed two distinct absorption peaks corresponding to 1 432 nm and 1 437 nm, which were con-

sistent with the HITRAN database. Fig.6 shows the fitting results of the concentration and the signal peak at 1 432 nm. The R^2 was 0.999. The experimental results showed that the gas concentration and the signal intensity of absorption peak had a good positive correlation. The inversion model of concentration was obtained by linear fitting with the least squares method. The results of concentration measurement are shown in Tab.1 below, and the maximum relative error is 3.33%.

Compared with the CO₂ concentration measurement results currently reported, the method used in this paper is simple, fast and accurate, it has strong practicability. Zhang Jianfeng et al^[17] measured the concentration of CO₂ based the on-line calibration technology of atmospheric oxygen and the photoacoustic spectroscopy, and the relative error is less than 1%; Yue Jixing et al^[18] measured the concentration of CO₂ by the TDLAS, and the relative error measured was 5.336%; Gu Fang et al^[19] used a new type of infrared sensor of gas to detect the CO₂ concentration in the range of 0—2 000×10⁻⁶ accurately. The average maximum relative error was 5.2%; Gao Yun et al^[20] used the CFD technology to study the CO₂ concentration numerically in the piggery, and the relative error range of the CO₂ concentration obtained was 4.3%—24.2%.

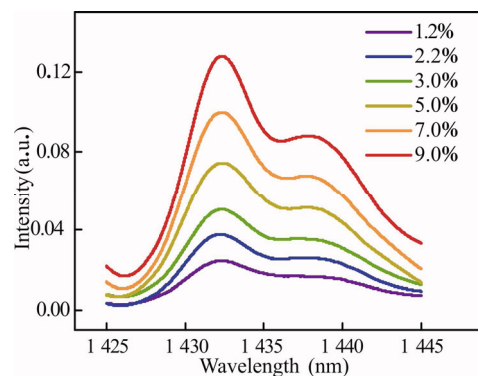


Fig.5 The absorption spectra of CO₂ at different concentrations (1.2%, 2.2%, 3.0%, 5.0%, 7.0%, 9.0%) in the range of 1 425—1 445 nm

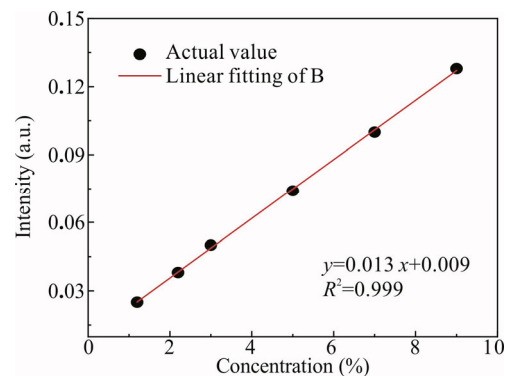


Fig.6 The linear fitting results between signal intensity of CO₂ and corresponding concentrations at 1 432 nm

Tab. 1 The results of concentration inversion

Gas concentration	Intensity of 1 432 nm	Linear fitting inversion concentration	Relative error
1.2%	0.025	1.19%	0.8%
2.2%	0.038	2.18%	0.9%
3.0%	0.050	3.10%	3.3%
5.0%	0.074	4.94%	1.2%
7.0%	0.100	6.92%	1.1%
9.0%	0.128	9.06%	0.7%

By keeping the pressure constant, the effect of temperature on the CO₂ absorption spectrum was studied. The absorption spectra of 99.9% CO₂ at 1 425—1 445 nm at different temperatures (298—373 K, interval of 15 K) was measured. The results are shown in Fig.7. The signal intensity of CO₂ at 1 432 nm of the main absorption peak at different temperatures was nonlinearly fitted to the temperature, and the results are shown in Fig.8. The experimental results showed that the temperature had a great influence on the absorption spectrum. As the temperature increases, the signal intensity of the gas gradually decreases. The reason is that the increase of temperature affects the spectral intensity of the gas and the shape of the absorption spectrum, which in turn causes the absorption measurement of CO₂ to decrease with increasing temperature. Therefore, the influence of temperature must be corrected to guarantee the accuracy of the measurement. In this paper, the method of temperature correction empirical equation was obtained by the least square method. The fourth-order polynomial was finally determined to correct the temperature by comparing the relative error of concentration measurement. The modified empirical equation is shown in Eq.(6). 298 K is used as the temperature T_0 to start measurement, the instantaneous temperature after the rise is T , the concentration measurement result without temperature correction is N_0 , and the concentration corrected is N_{CO_2} . As shown in Tab.2, the evaluation of the system temperature is as following. The maximum relative error of the concentration measurement was reduced to less than 1.4%, which improves the accuracy of the measurement system effectively.

$$N_{CO_2} = \frac{A_4 T^4 + A_3 T^3 + A_2 T^2 + A_1 T + A_0}{A_4 T^4 + A_3 T^3 + A_2 T^2 + A_1 T + A_0} N_0 \quad (6)$$

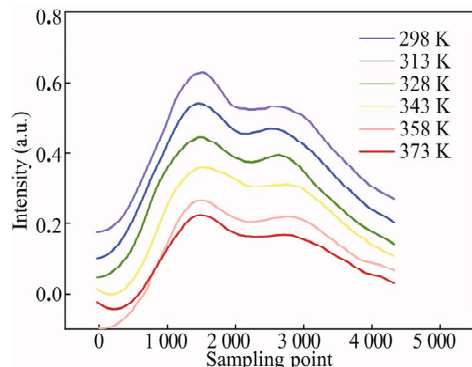


Fig.7 The 1 425—1 445 nm absorption spectra of 99.9% CO₂ in different temperatures (298—373 K, interval of 15 K)

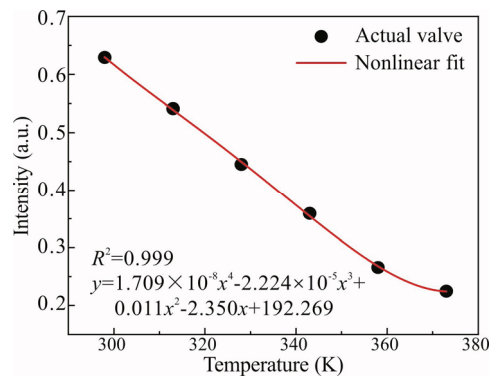


Fig.8 The nonlinear fitting results between signal intensity of CO₂ and corresponding temperatures at 1 432 nm

Tab.2 The results of measurement after correction at different temperatures

T (K)	Standard gas concentration (%)	Uncorrected concentration (%)	Corrected concentration (%)	Relative error (%)
298	99.9	99.9	99.9	0
313	99.9	85.9	100.4	0.5
328	99.9	70.6	98.8	1.1
343	99.9	57.0	101.3	1.4
358	99.9	42.1	99.3	0.6
373	99.9	35.7	100.4	0.5

The measurement of absorption spectrum is affected by various factors, and there will be certain errors inevitably. By assessing the measurement uncertainty, the main factors affecting the results of measurement can be analyzed, and it can improve the level or quality of the measurement results. The sources of measurement uncertainty in this experiment mainly include the following six aspects: the standard concentration gas, the laser, the LLTF, the photodetector, the DAQ, and the uncertainty caused by repetitive measurements.

The uncertainty of the gas is mainly derived from two aspects, namely the uncertainty introduced by the production deviation before the gas leaves the factory and the uncertainty caused by the two mass flowmeters in the dynamic dilution calibrator. The allowable production deviation of the gas is 0.1% by according to the certificates of CO₂ and N₂ used in the laboratory. According to the uniform distribution, the uncertainties of the two gases were:

$$u_{11} = 0.1/\sqrt{3} = 0.0577, u_{12} = 0.1/\sqrt{3} = 0.0577 \quad (7)$$

The dynamic dilution calibrator includes two mass flow meters. The measurement accuracy of the flow is $\pm 1.0\%$ FS. Wherein, the maximum throughput of the flow meter 1 is 10 LPM, then the reference error of the flow meter 1 was $Y_1 = (10 \times 1\%) / 10 = 1\%$. The maximum allowable error was $\delta = 1\% \times 10 = 0.1$.

According to the uniform distribution, the uncertainty is $u_{13} = 0.1/\sqrt{3} = 0.0577$. The maximum throughput of

the flow meter 2 is 1LPM, then the reference error of the flow meter 2 was $Y_2=(1\times 1\%)/1=1\%$. The maximum allowable error was $\delta=1\%\times 1=0.01$. According to the uniform distribution, the uncertainty was $u_{14}=0.01/\sqrt{3}=0.0058$. The four components are irrelevant. Calculated by the method of square and root, the uncertainty caused by the dilution of the gas was: $u_1=\sqrt{u_{11}^2+u_{12}^2+u_{13}^2+u_{14}^2}=0.1731$.

The laser is the core part of the system. The wavelength of the laser and the intensity of the light source directly affect the accuracy of the system measurement results. The repeatability of the supercontinuum laser is $\pm 5\%$. Calculated by the uniform distribution, including factor is $k=\sqrt{3}$, the uncertainty of the laser was $u_2=0.05/\sqrt{3}=0.0289$.

The uncertainty of the LLTF is derived from its resolution of wavelength tuning. The resolution D of the filter is 0.1 nm, the resolution is taken as the entire interval width, and half of the resolution as the half-width interval. According to the uniform distribution, the uncertainty generated by the filter was $u_3=D/2\sqrt{3}=0.0289$.

The sensitivity of the detector will have an impact on the results of measurement. The deviation percentage of the detector is 0.35%. The value of including factor is $k=\sqrt{3}$. The uncertainty of the photodetector was: $u_4=0.35/\sqrt{3}=0.2021$.

The main function of the DAQ is to realize the A/D conversion of the data. The accuracy of the conversion will have an impact on the experimental results. The A/D conversion accuracy of the DAQ is 0.003%. Full scale is ± 1 . The range selected in the experiment is $E_r=30\text{ V}$ (The scope is $-15\text{ V}\sim+15\text{ V}$). The bits digit (BD) is 16 bits, thus the quantitative error is $Q=E_r/2^{BD}=0.0458\%$. The maximum deviation caused by the A/D conversion accuracy of the DAQ varies within $[-a, a]$, so we can calculate $\alpha=0.003\%\times E_r+O=0.0014$. The uncertainty caused by the DAQ was $u_5=0.0014/\sqrt{3}=0.0008$.

The 9% of CO_2 was selected, and we repeated the measurements 10 times in a row to examine the repeatability of the measurements. The scanning wavelength is 1425—1445 nm. The signal intensity value at 1432 nm was obtained by the experiment, and the average value \bar{x} of the signal intensity is 0.1284. The experimental standard deviation of a single measurement calculated by the Bessel equation was

$$S(x_i)=\sqrt{\frac{\sum_{i=1}^n(x_i-\bar{x})^2}{n-1}}=0.0085. \quad (8)$$

Therefore the standard uncertainty caused by repetitive measurement was: $u_6=S(x_i)/\sqrt{n}=0.0027$.

The experimental uncertainty includes the above six aspects. The first five aspects are mainly the uncertainty of the system itself, which belong to the class B uncertainty. Uncertainty caused by the repetitive measurement is class A uncertainty. They are independent of each other.

The synthetic uncertainty was:

$$u_c=\sqrt{u_1^2+u_2^2+u_3^2+u_4^2+u_5^2}=0.2691.$$

According to $p=95\%$ and $\nu=8$, and the inclusion factor is $k_p=2.31$, then the extended uncertainty was

$$U_p=2.31\times u_c=0.6216. \quad (9)$$

In summary, it can be concluded that the uncertainty caused by the DAQ is the smallest, and the uncertainty created by the photodetector is the largest. Therefore, the overall uncertainty of the system can be reduced by reducing the u_4 , and it will improve the reliability of the measurement result.

In this paper, a supercontinuum laser absorption spectroscopy system was designed and built. The SCLAS was used to measure the concentration of the CO_2 accurately. It proved that the supercontinuum laser can be used to detect the concentration of CO_2 . The absorption spectra of different concentrations of CO_2 near-infrared 1425—1445 nm were measured. The maximum relative error of concentration measurement was 3.3%. The absorption spectra of 99.9% CO_2 at different temperatures (298—373 K, interval of 15 K) were studied. The temperature compensation equation was obtained by using the fourth-order polynomial, which reduced the influence of temperature changes on the results of concentration measurement. The uncertainty source of the whole spectrum detection system was analyzed and evaluated, and the system synthesis standard uncertainty was 0.2691, and the extended uncertainty was 0.6216. The experimental results show that the SCLAS has greater advantages in avoiding on-site measurement of environmental interference, and it provides theoretical support and technical support for other gases and the simultaneous detection of multi-component gases.

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