Cross-interference correction and simultaneous multi-gas analysis based on infrared absorption*

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In this paper, we present the simultaneous multiple pollutant gases (CO_2, CO, and NO) measurements by using the non-dispersive infrared (NDIR) technique. A cross-correlation correction method is proposed and used to correct the cross-interferences among the target gases. The calculation of calibration curves is based on least-square fittings with third-order polynomials, and the interference functions are approximated by linear curves. The pure absorbance of each gas is obtained by solving three simultaneous equations using the fitted interference functions. Through the interference correction, the signal created at each filter channel only depends on the absorption of the intended gas. Gas mixture samples with different concentrations of CO_2, CO, and NO are pumped into the sample cell for analysis. The results show that the measurement error of each gas is less than 4.5%.

Keywords: environmental pollution measurements, optical measurement technology, non-dispersive infrared technique, gas analysis

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1. Introduction

In China, especially in densely populated and highly industrialized areas, where the damage of harmful gases to human beings is drawing the growing attention of people, many facilities that generate or potentially generate harmful gas emissions need to monitored by employing multi-gas analyzer systems to ensure that the emitted pollutant concentrations are compliant with the corresponding regulations.[1,2] Non-dispersive infrared (NDIR) multi-gas analyzers measure the concentrations of the target gases by measuring the infrared radiation absorbed by these gases.[3,4] The NDIR gas analyzer was invented and first used in Germany in the 1930's.[4,5] Since then, many thousands of these analyzers with different structures have been produced and used in the fields of combustion, pollution, medical, automotive, chemical, refining, and others.[5] Continuous improvements to the NDIR technique over the years have made it one of the most successful and versatile techniques for gas analysis. They have been used to measure more than 100 different gases with concentrations ranging from ppm to percent. The NDIR instruments are rugged, easy to maintain and operate, and less expensive than the other infrared spectrometers.[6–8]

In the middle infrared waveband (between 1 μm and 10 μm), which is a specific region for NDIR gas analyzers, lots of gases show absorption interferences with each other, including the interferences from the unintended gases (especially H_2O) presented in the operating environment and the cross-interferences among the target gases.[9] Besides, any additional cause of attenuation (scattered by the dust, effect of impurities deposited on the instrument windows) and any uncontrolled variation of the source spectral luminance due to aging, voltage fluctuation, etc. will produce interferences in the NDIR gas analyzers. All these interferences have to be corrected during the analysis.[10] Most NDIR analyzers on the market, including spectral correlators (e.g., gas filter correla-

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tors (GFCs) and interferometric correlators (INCs)) and broadband analyzers, usually use a reference filter to adjust the latter interferences and correct the interference due to water vapor by preprocessing the sample gas. However, efficient correction methods for correcting the cross-interferences among target gases have seldom been used in the disclosed NDIR gas analyzers.[11,12] Galais *et al.* proposed that the cross-interference could be corrected by using the spectral correlation.[10] However, it was only applied to spectral correlators for one gas analysis. Appel and his coworkers assumed that the total signal generated in one filter channel was the sum of the responses generated by each individual gas. Thus, the cross-interferences were corrected by setting up a series of simultaneous equations using the interference matrix.[12] It gives ideal correction results if every filter channel has a linear response. However, this method might not work if the interference is not only spectral overlap or there is saturated absorption in any filter channel. Tyson *et al.* treated the spectral-peak overlap of three classes of lipids (triglyceride, phospholipids, and cholesteryl) with both the least-square and the cross-correlation methods, concluding that the cross-correlation method could scale the degrees of overlap by the relative intensity of each reference at a particular wavelength.[13] However, a similar algorithm used for broadband-spectrum overlap has not been reported.

In this paper, we present a cross-interference correction and the concentration retrieval using the NDIR multi-gas analyzer. We investigate the feasibility of simultaneous high accuracy multi-gas measurement of the pollutant emission. Our work focuses on developing a compact instrument that is suitable for either *in situ* systematic pollution monitoring or process control. Accuracy, reliability, and long-term stability are more concerned than the high sensitivity.

2. Cross-interference correction algorithm

The correlation is an operation which quantifies the degree of similarity between two functions of time. They can be correlated by the displacement of one relative to the other along the time axis and simultaneous integration of the products of the functions.[14,15] The correlation is defined as

$$z(t) = \int_{-\infty}^{+\infty} x(\tau)h(t+\tau)\,\mathrm{d}\tau, \tag{1}$$

where $z(t)$ is the correlation function, and τ is the displacement of the function.

Similarly, if the correlation function is modified and used to quantify the cross-interference between two gases, the degree of interference of gas A to gas B is defined as[13]

$$\Gamma_{\mathrm{AB}}(x) = \frac{1}{C_{\max} - C_{\min}} \int_{C_{\min}}^{C_{\max}} F_{\mathrm{A}}(\tau)F_{\mathrm{B}}(\tau+x)\,\mathrm{d}\tau, \tag{2}$$

where $F_{\mathrm{A}}(x)$ is defined as the response function of gas A at filter channel #A, which quantifies a relationship that relates the absorbance to the corresponding concentration,[16−20] $F_{\mathrm{B}}(x)$ is the response function of gas A at filter channel #B, $C_{\min}$ and $C_{\max}$ represent the lowest and the highest detecting limits of the analyzer for gas A, respectively. The modified cross-correlation function $\Gamma_{\mathrm{AB}}(x)$ is also called the interference function of gas A to gas B.[21−23]

By way of a non-limiting example, assume that there are three gases for measurement, and they interfere with each other. Filter channels #A, #B, and #C (and a reference filter to adjust the instrument drift or random noise) are designed to measure gases A, B, and C, respectively. In such a case, the signal detected on any specific channel caused by any given gas mixture is dependent on the concentrations of the gases and the sensitivities of the specific channel to the gases. For instance, assume that the interfering gases are present in a sample cell, the absorbance of filter channel #A would actually be composed of one strong signal caused by the absorption of gas A plus a series of weak signals caused by the other gases absorptions. In order to determine the concentration of each gas in the mixture simultaneously, the analyzer should take the total signal generated by a given channel and subtract out the signals caused by the interfering gases, leaving just the signal or the value representing the absorption by the intended gas for the specific filter channel.

Assume that the total absorbances of filter channels #A, #B, and #C are A_{total}, B_{total}, and C_{total}, respectively. The pure absorbances of gases A, B, and C are X, Y, and Z, respectively. The response functions of gas A at filter channels #A, #B, and #C

are $F_{\rm A}(x)$, $F_{\rm B}(x)$, and $F_{\rm C}(x)$, respectively. The response functions of gas B at filter channels #A, #B, and #C are $G_{\rm A}(x)$, $G_{\rm B}(x)$, and $G_{\rm C}(x)$, respectively. The response functions of gas C at filter channels #A, #B, and #C are $H_{\rm A}(x)$, $H_{\rm B}(x)$, and $H_{\rm C}(x)$, respectively. Thus, the interference function $\Gamma_{\rm AB}(x)$ of gas A to gas B can be determined by the correlation of $F_{\rm A}(x)$ and $F_{\rm B}(x)$. Accordingly, $\Gamma_{\rm AC}(x)$ can be determined by the correlation of $F_{\rm A}(x)$ and $F_{\rm C}(x)$, $\Gamma_{\rm BA}(x)$ by the correlation of $G_{\rm B}(x)$ and $G_{\rm A}(x)$, $\Gamma_{\rm BC}(x)$ by the correlation of $G_{\rm B}(x)$ and $G_{\rm C}(x)$, $\Gamma_{\rm CA}(x)$ by the correlation of $H_{\rm C}(x)$ and $H_{\rm A}(x)$, $\Gamma_{\rm CB}(x)$ by the correlation of $H_{\rm C}(x)$ and $H_{\rm B}(x)$. Thus, three simultaneous equations are set up by using all these interference functions

$$\begin{cases} X + \Gamma_{\rm BA}(Y) + \Gamma_{\rm CA}(Z) = A_{\rm total}, \\ \Gamma_{\rm AB}(X) + Y + \Gamma_{\rm CB}(Z) = B_{\rm total}, \\ \Gamma_{\rm AC}(X) + \Gamma_{\rm BC}(Y) + Z = C_{\rm total}. \end{cases} \tag{3}$$

By solving the above equations, the pure absorbance of each gas can be obtained. The pure absorbance is then used for concentration retrieval with the calibration curves. The calibration curve of each gas is the inverse function of the response function at the intended filter channel, it can be obtained by using the least-square fitting method, and the total absorbance of each filter channel can be calculated as[20,21]

$$T_{\rm total} = \ln\left(\frac{I_T^0/I_{\rm ref}^0}{I_T^{\rm s}/I_{\rm ref}^{\rm s}}\right), \tag{4}$$

where I_T^0 and $I_T^{\rm s}$ represent the incident and the emergent intensities at the target filter channel, respectively, $I_{\rm ref}^0$ and $I_{\rm ref}^{\rm s}$ represent the incident and the emergent intensities at the reference filter channel, respectively, all of them can be measured by a detector. Here T refers to $\#A$, $\#B$, or $\#C$.

Such a method applies to the multi-gas simultaneous analysis as well with the same principle of cross-interference correction.

3. Instrument description

Figure 1 shows the structure of a NDIR multi-gas analyzer, the instrument includes an infrared (IR) light source, an optical filter wheel, a sample cell, a detector, and a data processor. The IR light source is a glowing metal filament heated upto 1200 K, generating an infrared radiation in the wavelength range from 1.5 μm to 10 μm. The optical filter wheel is controlled by a chopper motor, which can filter and modulate the optical signal simultaneously. The filter wheel has four filters that filter the optical signal at certain frequency bands. Different optical filters are selected in sequence to modulate the optical signal (at a rotating speed of 25 r/s). The parameters of the filters are shown in Table 1. The reference filter used for adjusting the instrument drift has a pass band, in which there is no (or negligible) absorption for the emission caused by any gas considered. Besides, the relative weaker absorption waveband of 4.84 μm (compared to 4.3 μm) is chosen for CO_2 because of the high concentration of CO_2 presented in the industrial emission. The sample cell has three spherical mirrors ($f = 396.3$ mm) to increase the optical path length of the optical signal when it passes through the sample gas ($\sim$ 12 m optical path length within the 60 cm long multi-pass cell), resulting in higher sensitivity and accuracy. The sample gas is pumped into the sample cell at a constant flow rate ($\sim$ 1 L/min.). The capillary and the pump are used to ensure the stability of the pressure in the cell. The temperature in the sample cell is stabilized at 343 K by using a temperature controller. The gas flow path is controlled by a magnetic valve. The standard gas path is opened during calibration, and the sample gas path is opened for the rest of the time. The detector is a lead-selenide (PbSe) detector from Sens-Array Infrared, USA. It offers good responses in the wavelength range from 1 μm to 10 μm.

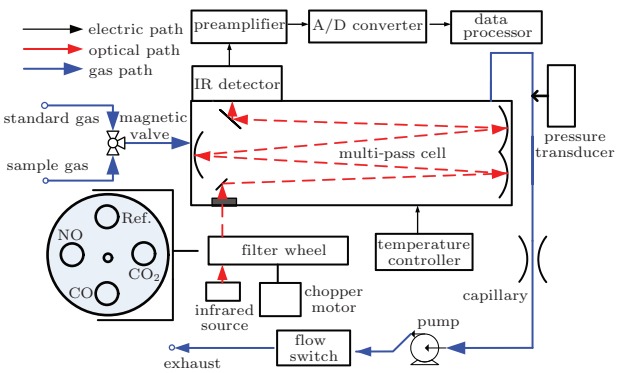


Fig. 1. (colour online) Functional structure of the self-made NDIR multi-gas analyzer.

By using other filter combinations, the instrument can also be used to measure other pollutant gases, such as SO_2, NO_2, CH_4, N_2O, HC, H_2O, and H_2S.

Table 1. Parameters of each band pass filter.

Channel	#1	#2	#3	#4
Filter	Ref.	CO	NO	CO_2
Center wavelength/μm	3.73	4.61	5.25	4.84
Transmission/%	76	80	80	93

4. Experiments and discussion

4.1. Calibration curves

In order to obtain the calibration curves of different gases, we design the following experiments (in the following discussion, CO_2, CO, and NO are corresponding to A, B, and C mentioned in Section 2, respectively). We take CO_2 as an example, and switch the magnetic valve to open the standard gas path. Then CO_2 with various concentrations are pumped into the sample cell in turn (all gas concentrations mentioned in this study are in units of volume mixing ratio). The voltages measured at the CO_2 filter channel are in ratio against the voltages measured at the reference filter channel to correct for the hardware instability, and then are converted to the absorbance. The least-square method based on a third-order polynomial is adopted to treat the data array $(X^i,\ C^i_{CO_2})$. Thus, the calibration curve of CO_2 is obtained.[24,25] Here, $C^i_{CO_2}$ is the concentration of CO_2 pumped into the sample cell for the i-th time $(i = 1, 2, \ldots, n)$, X^i is the corresponding absorbance taken at the CO_2 filter channel when CO_2 concentration is $C^i_{CO_2}$. The calibration curves of CO and NO are obtained in the same way. Figures 2(a), 2(b), and 2(c) show the calibration curves of CO_2, CO, and NO, respectively. The fitting parameters are also listed in the figures. It shows that the correlation coefficients R are all greater than 0.999. The polynomials of the calibration curves of CO_2, CO, and NO are $C_{CO2}(X) = 0.40622 + 111.62446X + 476.51356X^2 + 214.81723X^3$, $C_{CO}(Y) = 10.93293 + 268.23827Y + 11935.61756Y^2 - 8851.46091Y^3$, and $C_{NO}(Z) = 11.12413 + 2463.03665Z + 12917.31925Z^2 + 15491.96741Z^3$, respectively.

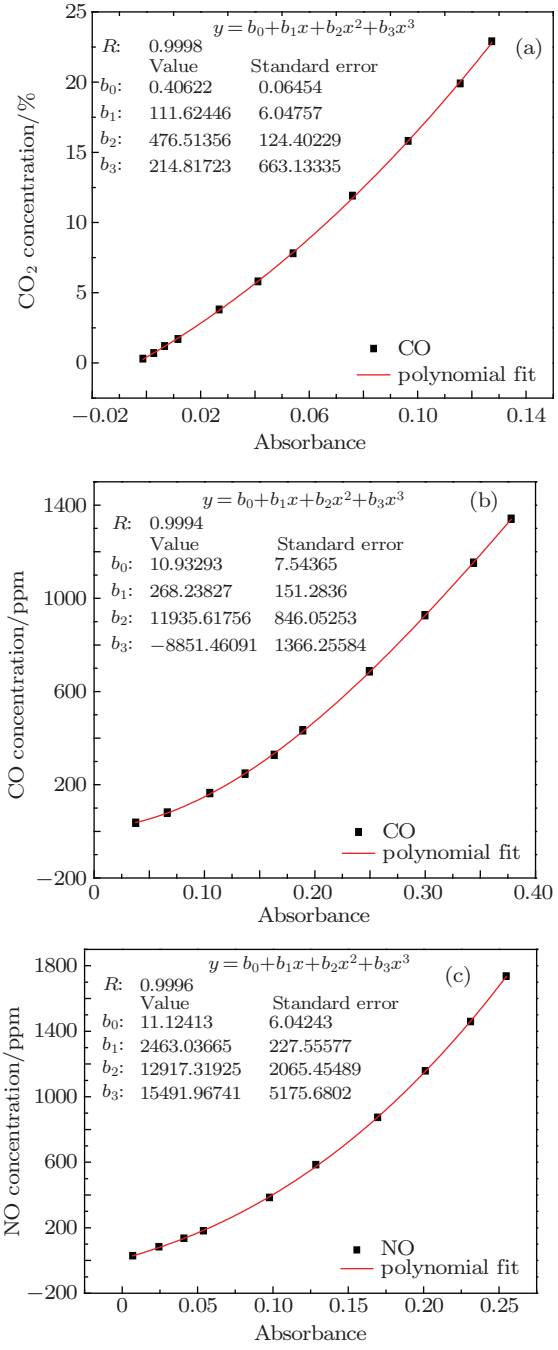


Fig. 2. (colour online) Least-square fitted calibration curves of (a) CO_2, (b) CO, and (c) NO with the third-order polynomial model. The fitting parameters and the fitting correlation coefficients are also shown.

4.2. Interference functions

We take CO_2 as an example as well, and switch the magnetic valve to open the sample gas path. High concentration CO_2 (25%) is diluted into CO_2 of different concentrations, which then are pumped into the sample cell in turn. The procedure is as follows. Firstly, 1 L CO_2 of 25% concentration is pumped into the sample cell. Secondly, 0.95 L 25% CO_2 and 0.05 L

pure N_2 (99.999%) are mixed and then pumped into the cell. Thirdly, the mixture of 0.9 L 25% CO_2 and 0.1 L pure N_2 is pumped into the cell, etc. Finally, 1 L pure N_2 is imported. We make sure that the time interval of the operations is longer than the response time of the analyzer (60 s). The data processor takes the voltages measured at CO_2, CO, and NO filter channels and then converts them to absorbances every 5 s. The interference functions of CO_2 to CO and NO can be obtained by linear least-square fitting data arrays (X^i, Y^i) and (X^i, Z^i), respectively. Here X^i, Y^i, and Z^i ($i = 1, 2, \ldots, n$) represent the absorbances at CO_2, CO, and NO filter channels for the i-th time, respectively. The other interference functions, CO to CO_2, NO and NO to CO, CO_2, can be obtained in the same way. Figures 3(a)-3(f) show the interference functions of CO_2 to CO and NO, CO to CO_2 and NO, and NO to CO_2 and CO, respectively.

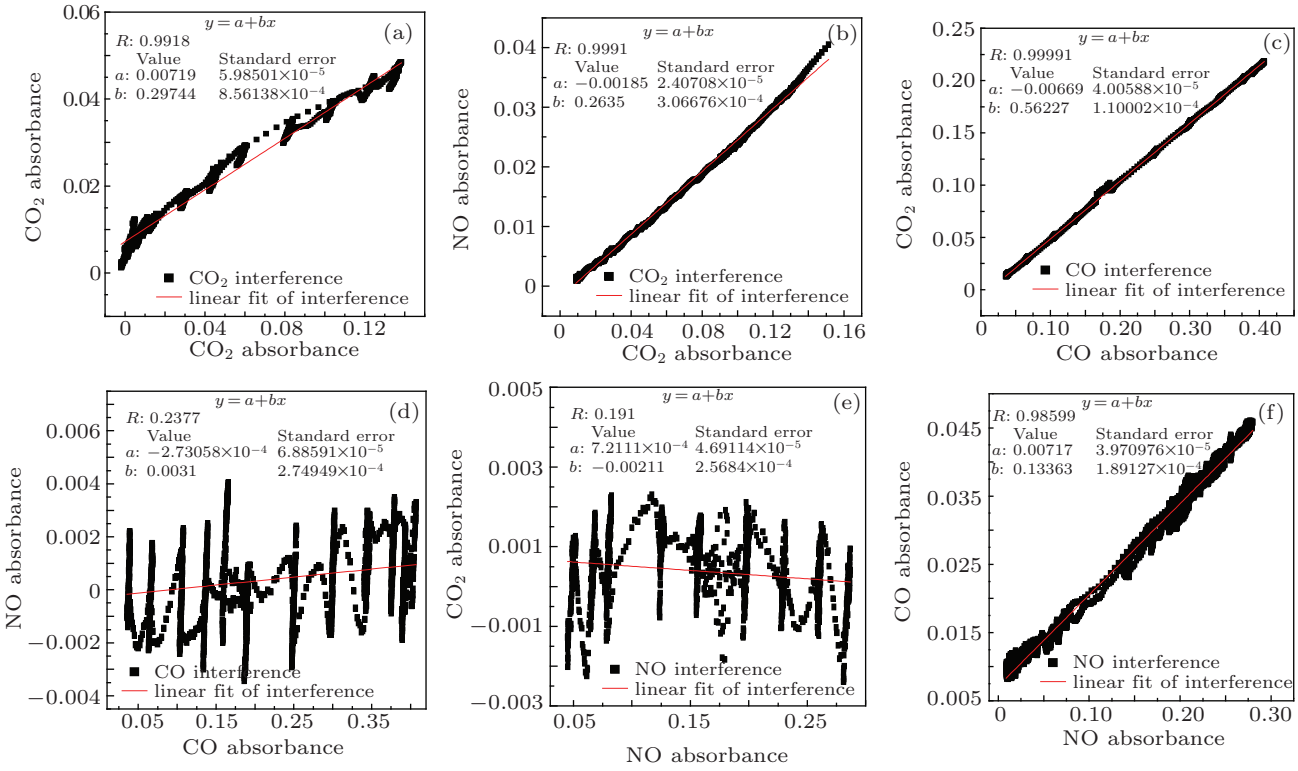


Fig. 3. (colour online) The least-square fitted interference functions with a linear model. Panels (a)–(f) show the interference functions of CO_2 to CO and NO, CO to CO_2 and NO, and NO to CO_2 and CO, respectively. The fitting parameters and the fitting correlation coefficients are also shown.

Interference functions with low fitting correlation coefficients (< 0.3) can be neglected, i.e., the cross-interferences are negligible. As shown in Figs. 3(d) and 3(e), the fitting correlation coefficients are only 0.2377 and 0.191, respectively. So it is considered that CO has negligible interference with NO, and so is for NO to CO_2. In fact, both plots show that the absorbance of the interference filter channel (the NO filter channel in Fig. 3(d) and the CO_2 filter channel in Fig. 3(e)) fluctuates around zero when the absorbance of the intended filter channel (the CO filter channel in Fig. 3(d) and the NO filter channel in Fig. 3(e)) changes from none to saturation. The fluctuation may be caused by a system drift and is not from the interferences of the other target gases.

Slope b in the interference function $y = bx + a$ is defined as the response coefficient or the cross-correlation coefficient. In a way, it describes the overlap extent of the absorption spectra of two gases in a specific waveband. The intercept a is defined as the interference coefficient, which represents the interference beside the overlap spectra, such as the imperfections of the detector's frequency response. It varies from detector to detector, but can be measured during the calibration. The auto-correlation coefficient is 1.0, i.e., each filter has a response of 1.0 to the intended gas. The response and the interference coefficients of CO_2, CO, and NO filter channels to each gas are sum-

marized in Table 2.

Table 2. Response and interference coefficients, where a and b are interference and response coefficients, respectively.

Channel		# CO_2	# CO	# NO
CO_2	b	1	0.29744	0.2635
	a	0	0.00719	–0.00185
CO	b	0.55227	1	0
	a	–0.00669	0	0
NO	b	0	0.13363	1
	a	0	0.00717	0

These assumptions and approximations made for interference functions are validated by the theoretical calculation. Table 3 shows the absorption coefficients of CO_2, CO, and NO at each filter channel. These coefficients are obtained by line-by-line (LBL) integral of the line strength provided by HITRAN database over the relevant wavelength range.[26,27] The ratio of any two absorption coefficients at a specific filter channel describes the overlap extent of the absorption spectra of the two gases. So it can be concluded from Table 3 that CO_2 has remarkable absorption interferences with CO ($\sim$ 0.2) and NO ($\sim$ 0.15), and the same for CO to CO_2 ($\sim$ 0.48) and NO to CO ($\sim$ 0.008). However, the absorption interferences of CO to NO ($\sim 10^{-7}$) and NO to CO_2 ($\sim 10^{-6}$) can be neglected. It is in good agreement with that suggested by the fitted interference functions.

Table 3. Absorption coefficients through theoretical calculation (in units of cm^2).

Channel	#1	#2	#3	#4
Filter	Ref.	CO	NO	CO_2
CO_2	1.016×10^{-25}	1.2642×10^{-21}	9.17×10^{-22}	6.3606×10^{-21}
CO	0	9.3513×10^{-18}	5.5277×10^{-25}	4.4884×10^{-18}
NO	0	2.3546×10^{-20}	2.907×10^{-18}	8.5623×10^{-24}

4.3. Interference correction and concentration retrieval

CO_2, CO, and NO of different concentrations are pumped into the sample cell respectively to validate the efficiency of the proposed method for cross-interference correction. The experimental results demonstrate that the interferences with the unintended gases are corrected with the fitted interference functions. Figure 4 shows the absorbance variation at each filter channel before and after the cross-interference correction. Figures 4(a), 4(c), and 4(e) show that both the intended and the interfering filter channels respond to the sample gases before the interference correction. However, after the correction, the variations of the sample gas concentrations occur only at the intended filter channels, while the absorbances of the interfering filter channels are corrected to zero, as shown in Figs. 4(b), 4(d), and 4(f). For example, figures 4(a) and 4(b) show the results of the sample cell filled with 28%, 21.2%, 15%, 8.2%, and 3.3% CO_2 in turn. Figure 4(a) shows that the absorbances of CO_2, CO, and NO all vary obviously with the CO_2 concentration, decreasing as its concentration reduces. However, with the interference correction, figure 4(b) shows that only the CO_2 filter channel indicates the variation of CO_2 concentration, while the absorbances of the unintended gases CO and NO are corrected to zero. Similar results are shown in Figs. 4(c)–4(f). Figures 4(c) and 4(d) show the results of the sample cell filled with 1600 ppm (1 ppm=10^{-6}), 1200 ppm, 800 ppm, 475 ppm, and 220 ppm CO in turn. Figures 4(e) and 4(f) show the results of the sample cell filled with 1275 ppm, 955 ppm, 627 ppm, 374 ppm, and 155 ppm NO in turn. A conclusion can also be drawn from Figs. 4(c) and 4(e) that the interferences of CO to NO filter channel and NO to CO_2 filter channel are negligible, suggesting that it is reasonable to neglect the fitted interference functions with low fitting correlation coefficients.[28–30]

The accuracy of the concentration retrieval by the analyzer after the cross-interference correction is examined. Nominal gases (gases with known concentrations) with different concentrations (mixtures of CO_2, CO, NO, and N_2) are pumped into the sample cell following the calibration. Then, the concentration readings of the analyzer are compared with the nominal values. Three simultaneous equations would be set up for every concentration retrieval using the coefficients in Table 2.

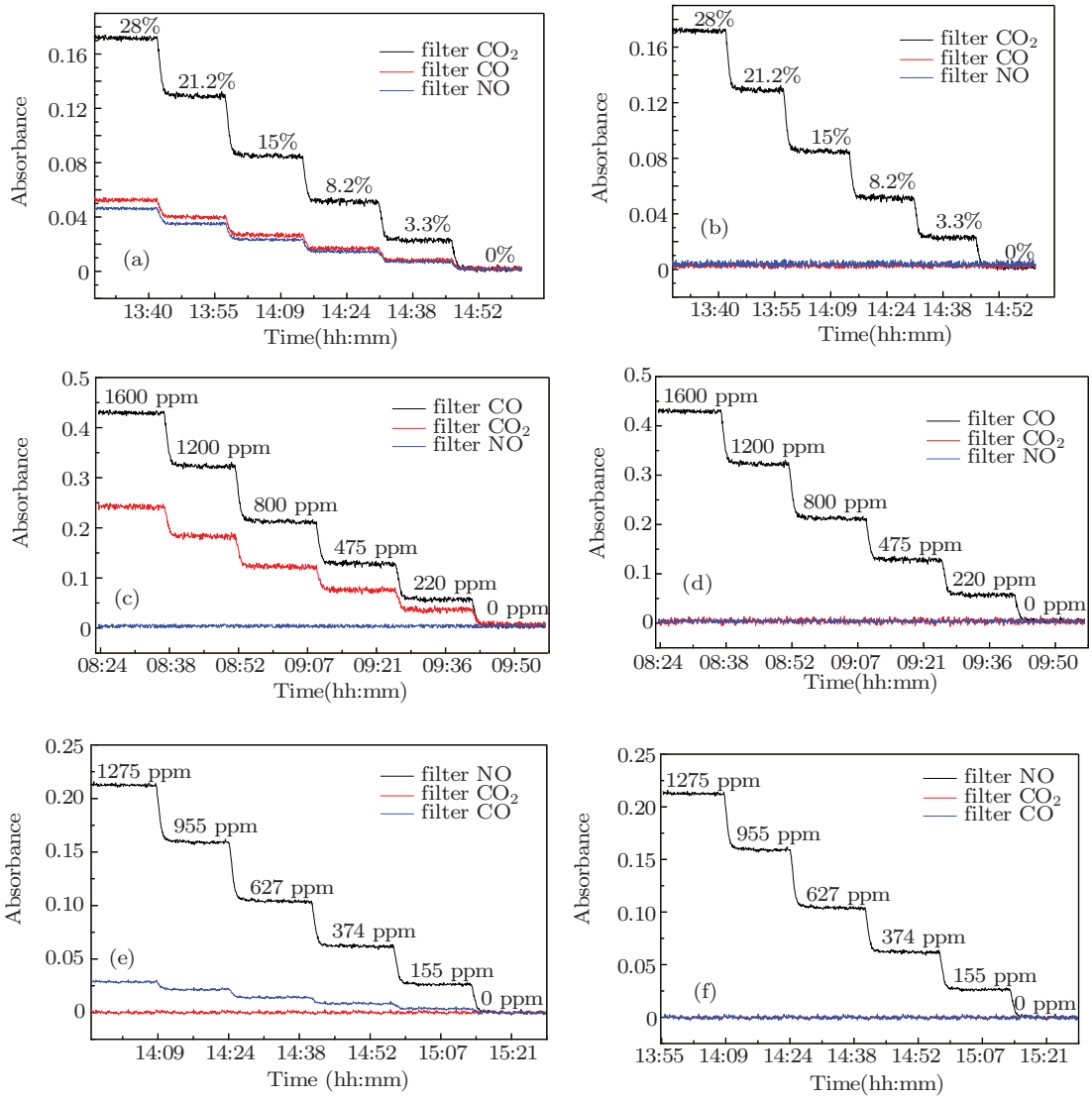


Fig. 4. (colour online) Absorbance variations at each filter channel before and after cross-interference correction, panels (a), (c), and (e) show the cases before the interference correction, panels (b), (d), and (f) show the cases after the interference correction. Panels (a) and (b) are for the sample cell filled with different concentrations of CO_2 in turn. Panels (c) and (d) are for the sample cell filled with different concentrations of CO in turn. Panels (e) and (f) are for the sample cell filled with different concentrations of NO in turn. The data are averaged over 32 times.

$$\begin{cases} 1 \times X + (0.55227 \times Y - 0.00669) + 0 \times Z = A_{\text{total}}, \\ (0.29744 \times X + 0.00719) + 1 \times Y + (0.13363 \times Z + 0.00717) = B_{\text{total}}, \\ (0.2635 \times X - 0.00185) + 0 \times Y + 1 \times Z = C_{\text{total}}, \end{cases} \tag{5}$$

where A_{total}, B_{total}, and C_{total} denote the total absorbances taken at CO_2, CO, and NO filter channels, respectively. The unknown variables, X, Y, and Z, represent the pure absorbances of CO_2, CO, and NO, respectively. Table 4 summarizes the results from the concentration retrieval of each gas. Experiments conducted for several times show that the measurement error for each gas is less than 4.5%, as shown in Table 4. The measurement error is calculated by

$$\text{Error} = \left| \frac{C_{\text{measured}} - C_{\text{nominal}}}{C_{\text{nominal}}} \right| \times 100\%. \tag{6}$$

Table 4. Experiment results with CO_2, CO, and NO.

No.	Nominal value			Measured value			Measurement error		
	CO_2/%	CO/ppm	NO/ppm	CO_2/%	CO/ppm	NO/ppm	CO_2/%	CO/%	NO/%
1	24.5	1970	1966	24.9	1944	1997	1.7	1.3	1.6
2	21.3	1765	1783	21.8	1733	1827	2.3	1.8	2.5
3	18.6	1547	1564	18.2	1515	1601	2.4	2.1	2.4
4	15.1	1331	1346	15.5	1365	1385	2.8	2.6	2.9
5	12.3	1172	1156	12.7	1205	1119	3.1	2.9	3.2
6	10.5	877	943	10.1	846	976	3.4	3.6	3.5
7	8.4	674	631	8.1	653	643	3.3	3.1	2.0
8	5.7	423	413	5.47	441	395	4.1	4.3	4.4
9	2.8	199	223	2.67	207	213	4.4	4.2	4.2
10	0	0	0	0.01	3	3	1	3	3

5. Conclusion

The cross-interference correction and the simultaneous multi-gas analysis based on the NDIR absorption have been investigated. The calibration curves are used to quantify the relationship between the concentrations and the corresponding absorbances. The cross-correlation correction functions are introduced to quantify the cross-interferences among the target gases. Based on least-square fittings, the calibration curves and the cross-correlation correction functions are approximated by three-order polynomials and linear curves, respectively. The cross-interferences are corrected, and the pure absorbance of each gas is obtained by solving three simultaneous equations using the fitted interference functions. After the interference correction, the signal detected at each filter channel only depends on the absorption of the intended gas. Gas mixture samples with different concentrations of CO_2, CO, and NO are pumped into the sample cell for analysis. The result shows that the measurement error of each gas is less than 4.5%.

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