Hazardous gas detection with an integrating sphere in the near-infrared

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Abstract. This paper describes an optical fibre near infrared Carbon Dioxide sensor. Using an integrating sphere as a test cell, increased path length is achieved due to multiple reflections within the sphere. Carbon dioxide is input into the sphere via an unused optical port. Light from a C + L band ASE source is transmitted through the sphere and any absorption is detected using a near infrared spectrometer. A change in optical intensity was observed at a wavelength of 1.59 \mu m when carbon dioxide was present from which the carbon dioxide concentration can be determined.

1. Introduction

The ability to test for hazardous gases is becoming more and more important. Environmental concerns mean harmful emissions from industrial sources, vehicles, etc need to be accurately monitored. This has led to much work being done in optical sensing techniques. Optical fibre sensors are well suited to emissions monitoring because of their robust nature and low susceptibility to noise or interference. Two gases of particular interest are Carbon Dioxide and Carbon Monoxide. Both gases are emitted from a wide range of sources and a multitude of optical sensors have been developed to detect them [1-3]. Both gases have absorption lines in the near infrared region around the 1570 nm region, which allows similar sensors to monitor each gas.

Different approaches have been taken in the past including luminescence [1,3] and fluorescence [2-3] based sensors. These sensors have long delay times and are not therefore particularly suited to applications where gas levels are rapidly fluctuating and need frequent monitoring. Intrinsic fibre sensors using the evanescent field have also been investigated [4-5] but these sensors are also limited as only a small portion of the light (the evanescent field) comes into contact with the gas. Some of the best results to date have come from extrinsic or “open-path” sensors [6-8]. In these sensors an optical signal is transmitted through a gas sample. This offers the advantage of all light coming into contact with the gas sample, which can result in a greater amount of absorption resulting in an increase in sensitivity. The main problem with this type of sensor is that to increase sensitivity the path length, i.e. the distance the light is transmitted through the gas sample, must be as high as possible, sometimes up to several metres to achieve 1% sensitivity. A long path length is often difficult to achieve given that many applications require small non-intrusive sensors. One possible solution would be to use highly polished mirrors to reflect light beams and so to increase the path length. These have been demonstrated by Mihalcea et al [8]. However the main disadvantage of this system is the presence of
standing waves, which can produce interference fringe effects that can cause a significant modulation of the baseline.

A potential solution to the above problems is the use of a hollow sphere with a highly reflective internal coating. No interference fringes can be present since there would be an infinite number of optical paths present. If such a sphere could be implemented then all input light would be reflected inside the sphere several times increasing the path length with each reflectance. An integrating sphere provides a ready-made and commercially available solution and can easily be adapted and used as a test cell [9].

2. Theory

An integrating sphere is a simple device. It consists of a hollow sphere of which the internal surface is coated with a highly reflective material. A sphere has several ports (usually 2 to 4) through which light can be input and output. The sphere used in this investigation has 4 ports and the coating used is spectralon. The advantage of using spectralon as a coating is that it has a very high reflectance (over 97% [10]) between 250nm and 2500nm. This high reflectance also results in an increase in radiance.

One of the most important parameters in any sphere is the multiplier. The sphere multiplier, which is a dimensionless quantity, is a measure of the increase in radiance in the sphere due to multiple reflections. Its value depends on two factors: the reflectance of the sphere and the size of the port fraction. The ports are the only openings in the sphere and as a result most light lost in the sphere is lost through these. The port fraction is a measure of the area of the port openings divided by the total internal surface area of the sphere. The multiplier is calculated from the following formula [11]:

\[
M := \frac{\rho}{1 - \rho \cdot (1 - f)}
\]

Eqn. 1

Where \( \rho \) is the surface reflectance of the sphere and \( f \) is the port fraction. For the sphere used in this experiment surface reflectivity was supplied by the manufacturer and was found to be 98% at 1590nm. The port fraction was calculated to equal 0.05. This results in a value of approximately 14 for the multiplier.

Much work has been done to determine the optical path length within an integrating sphere (Tranchart et al [9]). They estimate the equivalent absorption path length from the following formula

\[
L_{eq} := \frac{2}{3} \frac{D}{(1 - \rho)}
\]

Eqn. 2

Where \( D \) is the diameter of the sphere. The diameter of the sphere used in our experiment is 2 inches or 5.08 cm. This means that the equivalent path length for our spheres is of the order of 168 cm.

The expected absorption is found from Beer’s law:

\[
\frac{I(\lambda, L)}{I_0} := e^{(-\alpha(\lambda)nL)}
\]

Eqn. 3

Where \( I(\lambda, L) \) is the transmitted intensity, \( I_0 \) is the incident intensity, \( L \) is the optical path length, \( n \) is the concentration and \( \alpha(\lambda) \) is the absorption co-efficient. Replacing \( \alpha(\lambda)nL \) with \( x \) gives the following Maclaurin series:

\[
e^{-x} := 1 - x + \frac{1}{2!}x^2 - \frac{1}{3!}x^3 + \ldots
\]

Eqn. 4
Since the absorption co-efficient and concentration are small, Equation 4 can be re-written:

\[ e^{-x} = 1 - x \quad \text{Eqn. 5} \]

Equation 5 gives a linear response. The expected absorption has been calculated from equation 3 and the results presented in Figure 1. As can be observed a near linear response is observed over the range of interest.

![Graph](image)

**Figure 1.** Expected absorption for CO\(_2\) through the integrating sphere (path length 168 cm)

### 3. Experimental Set-up

The experimental set-up is shown in Figure 2 below. Light is input to the sphere from a C+L band ASE source (OLS15CL) from optolink and detected by an NIR spectrometer (NIR512) from ocean optics. The source and detector are linked to the sphere by NIR fibre (AS 125/150 IRSE) from fiberware. As can be seen the source and detector are placed at 90 degrees to each other and a baffle is also located between them. The baffle’s purpose is to prevent first reflections from entering the spectrometer, however it has a minimal affect on transmission through the sphere.
Figure 2. Experimental Setup for measurement of CO₂ concentration in this experiment

Gas is input into the sphere via one of its ports. Plastic piping carries the gas to the sphere and the piping can be attached to the SMA connectors, which were supplied with the sphere. Similar piping is used to output the gas from the sphere to the gas analyser. The percentage of gas present is monitored using a quintox gas analyser from Kane-May (KM9106).

The light source has a range of 1530 to 1610 nm that allows monitoring of several CO₂ near infrared overtone and combination bands. The spectrometer has a range 900 to 1700 nm. Its resolution is 2.5 nm, which makes it difficult to monitor narrow absorption lines. However several weaker but wider lines exist in the 1590 to 1600 range and these were monitored in this experiment.

4. Results

The results obtained in this experiment are shown below. The first chart shows the drop in recorded intensity with 50% CO₂ present. As can be seen the biggest change is between 1590 and 1605 nm. This is due to several weaker absorption lines that are present in this region. These lines are weaker but occur in greater numbers than the stronger lines present around 1573 nm. The intensity drop recorded at 1594 nm is 2.7% for 50% CO₂ present. This compares favorably with theory where a similar drop is expected (figure 1). Figure 4 shows the recorded intensity decay with increasing amounts of CO₂. Again this compares favorably with theory. To eliminate noise and so improve result efficiency a ratio was calculated of the intensity at those points at which absorption occurred and those where there was none. This ratio is shown in figure 4 for different concentrations of CO₂. The ranges chosen were the average intensity between 1590 and 1600 nm divided by the average between 1555 and 1565 nm. Since the intensity is higher between 1590 and 1600 any drop in recorded intensity should result in a corresponding drop in the value of the calculated ratio. This can be seen from the graph of figure 4.
Figure 3. Recorded drop in intensity across source spectrum for 0% CO₂ and 50% CO₂

Figure 4. Recorded drop in intensity ratio for 10% increase in CO₂ present.

5. Conclusion
From the experiments conducted in this investigation we conclude that by using an integrating sphere as a gas cell, CO₂ levels can be monitored. Path lengths of up to 40 times the sphere diameter can be achieved. The sphere also offers the advantage of being easy to setup. Fibre coupled connectors are readily available making the coupling of the source and detector simpler and eliminates the need for mirrors/lenses. The durability of the spectralon coating offers the added advantage of making the sphere suitable to use as a test cell over a long period of time. Its wide spectral range means that this setup can also be used in the UV. A further increase in path length can be obtained by cascading several spheres. This arrangement would undoubtedly increase sensitivity however losses through the spheres would be accumulating thus increasing the impact of noise in the system and thus reducing the
overall sensitivity gain. It has been demonstrated that an integrating sphere may be used as a test cell
to measure CO\textsubscript{2} levels in the near infrared and thus can be used to test for other gases such as carbon
monoxide, ammonia and hydrogen sulphide which occur in the same wavelength range.

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