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Item Type	Article
Authors	Hawe, Eamonn;Fitzpatrick, Colin;Chambers, Paul;Lewis, Elfed
Citation	Journal of Physics: Conference Series;76, 012041
Publisher	IOP Publishing
Download date	2026-05-13 00:31:26
Item License	https://creativecommons.org/licenses/by-nc-sa/1.0/
Link to Item	https://hdl.handle.net/10344/9102

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To cite this article: E Hawe *et al* 2007 *J. Phys.: Conf. Ser.* **76** 012041

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Ozone detection using an integrating sphere as an optical absorption cell

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Abstract. This paper presents a multipass optical absorption cell that is based on a spherical cavity. Ozone concentrations have been detected in the visible region at 603 nm, in the Chappuis band. A 2-inch diameter (50.8 mm) integrating sphere has been modified for use as an optical absorption cell. A method of calculating the effective optical path length of the integrating sphere is also presented. It is reported that the effective optical length of the sphere is reduced as the ozone concentration is increased as predicted by the effective path length formula.

1. Introduction

Ozone is a pollutant which is becoming increasingly prevalent in urban environments. Ozone is hazardous to human health as it reacts with the lining in the lungs causing inflammations which result in a weakening and thus premature aging of the lungs. Much of the ozone in urban area is formed by the photolysis of nitrogen dioxide. Ozone is also being increasingly used for industrial purposes primarily in the sterilisation of water supplies and in the environmental packaging of food products. As a result it is essential that ozone levels be monitored.

In this experiment an integrating sphere was modified for use as an absorption test cell in order to monitor ozone concentrations. The integrating sphere used has four ports, two of which were used for input and output gas and the other two allowed for the input and output of optical radiation. The sphere has a 2 inch (50.8 mm) diameter and a highly reflective internal coating. The internal coating is made from SpectralonTM which is over 95% reflective between 300 nm and 2100 nm. The sphere offers many advantages over conventional multipass sensors as it can operate in the ultra violet, visible and near infrared regions and it also allows the use of broadband and low power optical sources which can significantly reduce the overall cost of the system.

2. Theory

2.1. Integrating sphere theory

The theory of light propagation in integrating spheres has been extensively reported in the literature [1]. An important parameter regarding the operation of an integrating sphere is the so called sphere multiplier which is the average number of reflections a single photon will undergo before it exits the sphere. The multiplier is denoted by M and is calculated according to equation 1:

$$M = \frac{\rho}{1 - \rho(1 - a(\nu))(1 - f)} \quad (1)$$

where ρ is the reflectance of the sphere's internal surface, f is the ratio of the area of the port openings to the total internal sphere surface area and $a(\nu)$ is the level of attenuation of the optical radiation over a single pass through the sphere due to absorption and scattering by the test gas [2].

As the sphere is constructed from a diffuse and highly reflective surface, the average path length that a single photon will travel within the sphere is the arithmetical product of the multiplier and the average distance travelled by a photon for a single pass through the sphere. A close approximation for this is that the average path length is two-thirds of the diameter of the sphere [3]. Thus the average path length, L_{eff} , travelled by a photon within an integrating sphere containing an absorbing gas is found in equation 2.

$$L_{\text{eff}} = \frac{2}{3} \cdot M \cdot D \quad (2)$$

Where M is the sphere multiplier and D is the diameter of the sphere [2].

There are therefore three variables which can alter the effective optical path length within the sphere: the port fraction, the reflectance of the sphere's internal surface and the level of attenuation due to the gas contained within the sphere. The latter is an important parameter in understanding the mechanism of light propagation through the gas within the sphere. Clearly the gas can give rise to non-absorptive attenuation e.g. scattering which will limit the average path length as rays diverted into longer path lengths experience sufficient attenuation as to not allow them to be detected above the noise limit of the detector. In addition to this, light which is absorbed will result in a greater attenuation through the Beer-Lambert law and this also decreases the average path length. The value of the path length is introduced to the Beer-Lambert law and the attenuation calculated on the basis of this value. At higher concentration values (e.g. several percent for the gas concerned) this is manifested as a departure from a simple Beer-Lambert law calculation using a single path length value.

2.2. Ozone absorption theory

Ozone absorbs radiation in the visible region due to electron transitions within the ozone molecule. The visible photoelectron absorption spectrum is shown in figure 1 and stretches from 410 nm to 700 nm, the Chappuis Band, with the peak absorption occurring at 603 nm [4]. Ozone is also strongly absorbent at 254 nm. However the reflectivity of the integrating sphere's internal coating is much lower (95%) at this point and as a result any potential advantages gained by ozone's higher absorptivity in this region are negated by the reduction in the potential optical path length that can be achieved. Thus experiments to detect ozone were conducted in the visible region where the sphere's reflectivity was over 99%.

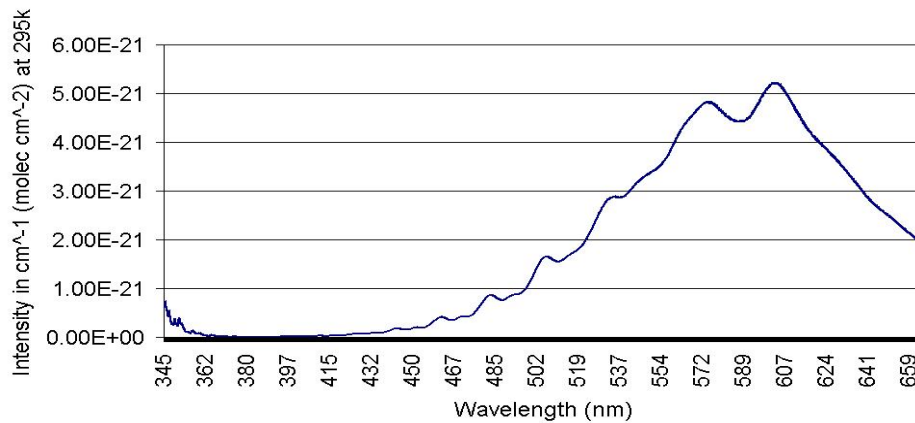


Figure 1: Ozone visible absorption spectrum [4].

3. Experimental Set-up

The sphere used in this investigation is a 2-inch (50.8 mm) diameter integrating sphere manufactured by Labsphere. This sphere is fitted with four optical ports. Two of these were used to input and output optical radiation. The remaining two were adapted to allow for the input and output of gas. The internal surface of the sphere was coated with Spectralon, a type of Polytetrafluoroethylene (PTFE). This coating is highly reflective (over 99%) between 350 nm and 1650 nm [5]. Spectralon's high reflectivity makes it possible for this sensor to detect a wide range of gases which exhibit absorption characteristics within this range. A photograph of the integrating sphere is shown in figure 2.



Figure 2: Integrating sphere sensor.

Light was launched into the sphere from a deuterium/halogen light source (DH-2000 from Ocean Optics) as the optical source and a UV/VIS spectrometer was used as the detector (SD2000 from Ocean Optics). The source had an emission spectrum ranging from 200 nm to 2000 nm while the spectrometer had a detection range of 200 nm to 1100 nm and a resolution of 1.5 nm which allowed it to be used to detect and spectrally resolve ozone absorption centred at 603 nm. The optical source and detector were connected to the integrating sphere using UV/VIS optical fibres from Ocean Optics (P400-2-UV-VIS). The fibres have a 400 μm core and 200 μm cladding. Ozone was produced using a commercial ozone generator from Opticslab (OL80A/DLS). Oxygen was input to the generator which was then converted into ozone by an electrical discharge process. The ozone concentration was varied by adjusting the oxygen flow into the generator. The concentration of ozone in the sphere was

simultaneously monitored using a Mini Hican ozone analyser from IN USA Inc. This analyser detects ozone concentrations using ultra violet absorption techniques and can operate up to 900 g/m^3 of ozone. Two of the sphere's optical ports were adapted to allow the input and output of ozone from the sphere. The experimental set-up is shown in figure 3 below.

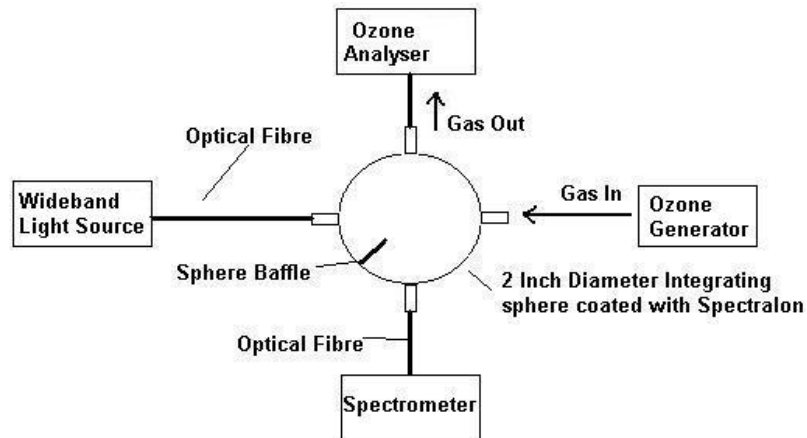


Figure 3: Ozone detection experimental set-up.

4. Results

A test was conducted to measure the amount of absorption exhibited by various ozone concentrations at 603 nm. The effective path length for ozone at 603 nm was determined using equation 5. At 603 nm the reflectivity of the sphere was found to be 0.989 (from the manufacturer's data sheet). Since optical fibres were used to connect both the source and the detector to the sphere the port fraction is reduced and was measured to be 0.01. The attenuation coefficient was found using the absorption data presented in figure 1 [4]. This data was used to determine the level of attenuation for a single pass through the sphere using the Beer-Lambert law and was found to be 0.026 for concentrations of ozone up to 20,000 ppm. This resulted in an average effective optical path length of 70 cm.

As can be observed from figure 4, the measured absorption closely matches the calculated absorption level at concentrations as high as 20,000 ppm. At concentrations above this amount, there is a significant divergence between the measured and the calculated absorption level. This is due to an increase in the attenuation coefficient for higher ozone concentrations. If equation 2 is recalculated for an ozone concentration of 55,000 ppm, the attenuation coefficient is found to be 0.034 and this results in a reduction of the average integrating sphere path length to 60 cm. As the ozone concentration is further increased to 70,000 ppm, the attenuation coefficient increases to 0.045 and this results in an average effective optical path length of 50 cm. The calculated absorption for each of the three path lengths is shown in figure 4. As can be observed the measured absorption corresponds favourably with the 70 cm calculated line until the ozone concentration becomes 20,000 ppm. Between 35,000 and 55,000 ppm the measured absorption matches the calculated absorption for 60 cm and above 70,000 ppm the measured absorption further decreases to a level close to the calculations for a 50 cm path length.

This indicates that the average effective path length formula is correct, as with increases in the ozone concentration, the measured increase in absorption is less than would be expected from a simple calculation of absorption based on a single path length. The difference between the two sets of data exists because of the reduction in the average effective optical path length of the sphere.

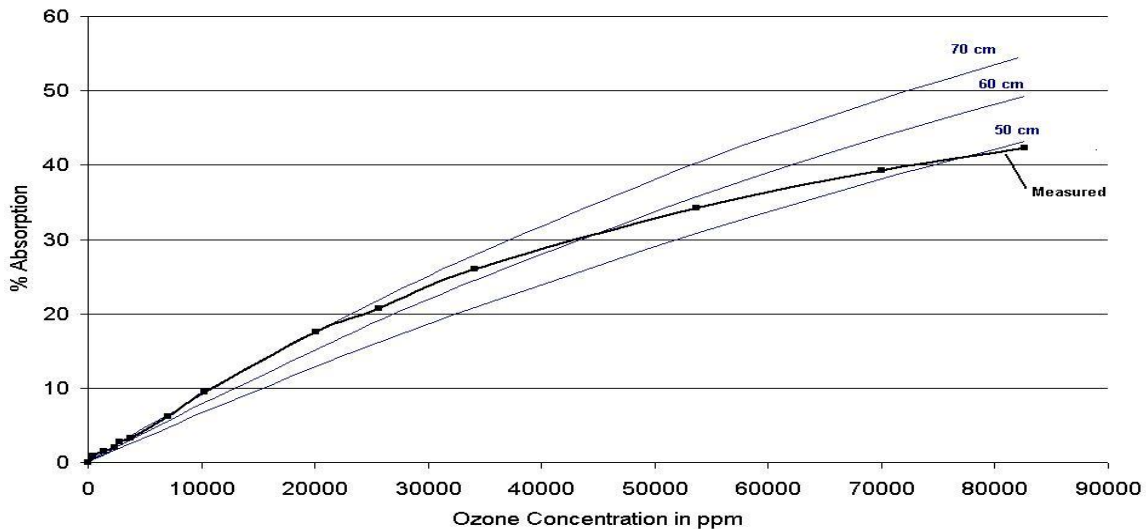


Figure 4: Measured ozone absorption compared to calculated absorption over optical path lengths of 70 cm, 60 cm and 50 cm.

5. Conclusion

A multipass absorption cell based on a 2-inch diameter integrating sphere capable of operating in the ultra violet and visible regions of the spectrum has been demonstrated. A method of calculating the effective optical path length of the sphere has also been presented. Optical path lengths as high as 70 cm have been demonstrated in the visible region. It has been shown that the effective optical path length of the integrating sphere is reduced as the concentration of ozone is increased as is predicted by the effective optical path length formula.

Future work will concentrate on developing a similar sensor using a larger diameter integrating sphere. This should generate longer effective optical path lengths which will allow the detection of much lower concentrations of ozone (10^3 's of ppm).

Acknowledgements

The authors would like to acknowledge the support of the EC for the FP6 project Opto-Emi-Sense (Contract number: TST3-CT2003-506592) for providing funding for this work.

Also the authors would like to thank the staff of Electronic and Computer Engineering Department of the University of Limerick, the University of Liverpool and Fachhochschule Wismar for their assistance and patience during testing and the Irish Research Council in Science, Engineering and Technology

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