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Phase-shift Cavity Ring Down Spectroscopy Set-up for NO_2 Sensing : Design and Fabrication

Cherry Dhiman, Mohd. Shahid Khan*, and M.N. Reddy#

Dept. of Physics, Jamia Millia Islamia, New Delhi -110 025, India
#Laser Science and Technology Centre, Delhi-110 054, India
**E-mail: mskhan@jmi.ac.in*

ABSTRACT

An indigenously designed cavity ring down spectroscopy cell of 80 cm length of mild steel material was fabricated by attaching two 1″ diameter high reflecting concave mirrors with reflectivity 99.997 % at 405 nm and radius of curvature was 1 m in specially designed mirror holding assemblies to the cell at two ends. Fine alignment of the resonator is facilitated with three tip-tilt adjusting screws to the mirror-mounting plate assembly. The PS-CRDS experimental set-up is evaluated by measuring the phase shift values corresponding to the absorption of NO_2 gas filled at low pressures in the cell. The limit of detection of pure NO_2 using the set-up under given conditions of Ar @ 50 mbar is estimated to be 1.50×10^{11} cm^{-3} and @ 60 mbar as 2×10^{11} cm^{-3}.

Keywords: Phase-shift cavity ring down spectroscopy (PS-CRDS), high reflectivity, laser diode, broadband

1. INTRODUCTION

Being one of the anthropogenic NO_x (NO and NO_2) species, nitrogen dioxide (NO_2) plays very important role in the atmospheric chemistry. It serves a vital role in balancing the production of ozone in troposphere layer of the atmosphere. Nitrogen dioxide (NO_2) is well known as the main precursor for the toxic molecules like nitric acid (HNO_3), peroxy aceyl nitrates (PAN's) and Ozone (O_3) etc and the direct impact of which leads to the chronic results for the human organs[1-2]. In addition to its environmental importance, NO_2 can also be used in the detection of explosives traces. Various techniques like laser-induced fluorescence (LIF), photo-acoustic spectroscopy (PAS), frequency modulation spectroscopy (FMS) and cavity ring down spectroscopy (CRDS) have been employed in the past for the sensitive detection of molecular species up to ppb level concentration[3-6]. Among all these techniques, cavity ring down spectroscopy (CRDS) is one of the most sensitive absorption-based techniques through which frequency-dependent absorption cross section of molecules can be extracted. The CRDS-based experimental schemes; both with pulsed lasers and continuous wave modulation based are well established for studying the weak absorption features of the molecules in ultraviolet to far infrared regions of the electromagnetic spectrum of light[7]. The phase shift cavity ring down (PS-CRDS) with the laser diode modulation method has proven to be more versatile. Herbelin[8], *et al* introduced the phase shift -based CRDS technique and used it to measure the reflectivity of HR mirrors. Further, Anderson[9], *et al.* also performed a similar experiment by abruptly switching off the CW light source for the determination of reflectivity of HR mirrors. Later, O'Keefe and Deacon[10] used the pulsed laser to carry out CRDS experiments for the spectroscopic measurements. Since then, this technique has attracted various research groups and has been employed for the detection of traces of molecular species in various environments including ambient air.

Among different types of spectroscopic techniques, PS-CRDS has gained the tremendous interest of various research groups because of its applications in different areas including solids, liquids, and gaseous samples[11-12]. The PS-CRDS is one of the categories of cavity ring down laser spectroscopy (CRDS) in which the phase difference between input and the output intensity signal is measured. This technique is extremely sensitive due to high signal-to-noise ratio and can be used to study the weak absorption features, pre-dissociation stages, and overtones in the molecular species[13,14]. Several experiments have been reported in the area of detecting and studying the thermodynamics, chemical kinetics, and spectroscopy of the various toxic gases[15-17].

In the present work, the sealed 80 cm cavity ring down cell is designed, fabricated in mild steel material and tested for sensing of NO_2 gas by employing phase shift cavity ring down experiment. The PS-CRDS experimental set-up has been used to perform the studies for the quantitative analysis of concentration of nitrogen dioxide (NO_2) at ppm level by employing 405 nm diode lasers. The absorption coefficient and concentration limit of the NO_2 at 405 nm has been calculated by optimizing the cavity parameters.

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2. THEORETICAL SECTION

In an optical resonator, the frequency of the mode having transverse and longitudinal modes is given by:

$$\nu_{qmn} = \frac{\omega_{qmn}}{2\pi} \tag{1}$$

$$= \frac{c}{2L}\left[q + (n+m+1)\frac{\cos^{-1}\left(\pm\sqrt{g_1 g_2}\right)}{\pi}\right] \tag{2}$$

where *qnm* are indexes and g_1 and g_2 are the cavity parameters:

$$g_1 = 1 - \frac{L}{r_1},\; g_2 = 1 - \frac{L}{r_2} \tag{3}$$

'L' spacing between two mirrors and r_1 and r_2 as radii of curvature. For, 80 cm cavity with ROC as 100 cm, mode frequency is defined as:

$$\nu_{qmn} = 46.852 \times 10^{8}\,\text{Hz}$$

The modulated intensity in the form of either sine wave or square wave when coupled into the high finesse cavity and reflected back and forth in the cavity produces the output CRD signal with same period as the input laser signal[8].

$$\tan(\varphi) = \omega\tau \tag{4}$$

Equation (4) gives the phase-shift signal for the first harmonics, representing the single pass absorption.where, 'ω' modulating frequency and 'τ' cavity decay time. The decay time constant of the optical cavity with the absorbing species is expressed as:

$$\tau = \frac{L}{c(1 - R + \alpha L)} \tag{5}$$

where '*c*' is the velocity of light, $R = (\sqrt{R_1 R_2})$ R_1 and R_2 are the reflectivity of the two cavity mirrors and 'α' is the intra cavity loss, including the absorption and scattering loss inside the cavity[18].

$$\alpha = \frac{1}{c}\left[\frac{1}{\tau_\lambda} - \frac{1}{\tau_0}\right] \tag{6}$$

where σ = absorption coefficient, τ_λ = decay time on conc. with sample,

τ_0 = decay time without sample, *c* = velocity of light

$$N_{\lim} = \frac{(1-R)\Delta\tau}{\sigma L \tau_0} \tag{7}$$

N_{lim} = minimum concentration detection or sensitivity, 'σ' absorption cross-section and Δτ is the difference between decay time with and without the sample.

3. EXPERIMENTAL SECTION

The PS-CRDS experimental set-up was established by designing and fabricating an 80 cm length high finesse sealed cavity ring down cell and a lock-in-amplifier for measuring the phase-shift values corresponding to the absorption of NO_2 gas by a broadband diode laser which is emitting at 405 nm wavelength. The experimental schematic of PS-CRDS is shown in Fig. 1.

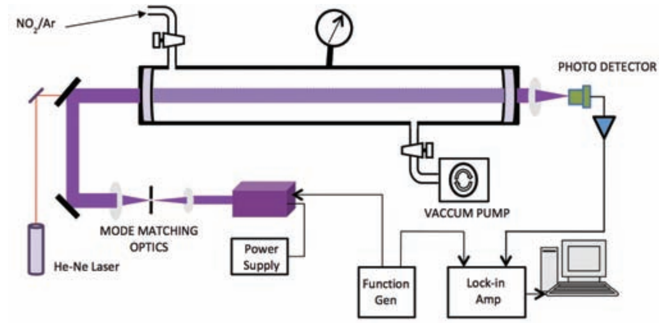

Figure 1. Schematic diagram of the Phase shift cavity ring down spectroscopy (PS-CRDS) set-up.

A unique leak-proof CRDS cell of 80 cm length was designed and fabricated to form a resonant cavity with excitation laser at 405 nm wavelength. Powder coating was used to avoid any reaction of NO_2 gas from the wall surfaces of sealed CRDS cell in mild steel material. A 50 mm diameter cylindrical tube of mild steel was welded with two circular flanges at the ends and specially designed mirror holder assemblies are attached at two ends as shown in Figs. 2 and 3.

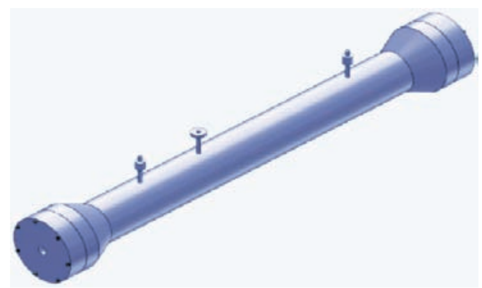

Figure 2. A CAD design of sealed CRDS cell of 80cm cavity length with safety cover.

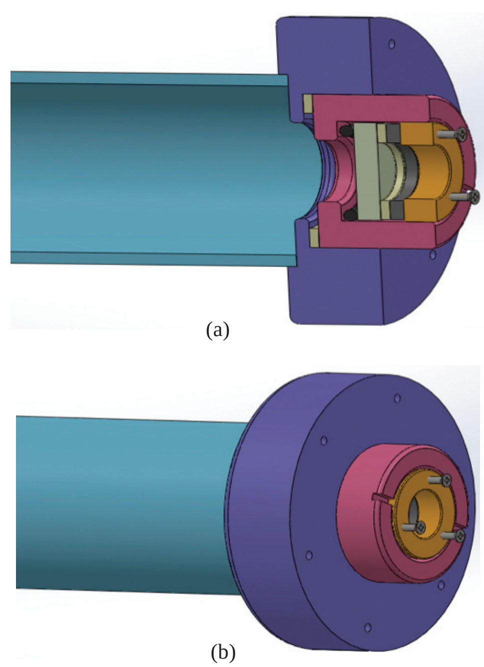

Figure 3. Section and detailed view of cavity cell. (a) Section view of CRDS cell without safety cover, and (b) detailed view of CRDS cell without safety cover.

Mirror holding assemblies were designed for tight holding of 1″ diameter high reflecting mirrors in the centre with 4 mm thick silicon o-rings at inner sides and 3 mm thick Teflon gaskets at outer sides. The mirrors along with o-rings and gaskets on either side were firmly sandwiched between outer and inner stainless steel mounting rings of the mirror holder. The mirror holder assembly was attached to the flange by means of fine threading arrangement. The optical cavity was fine-aligned by tip-tilt movement of the mirror mounting plates with the help of three screws attached to the inner mounting ring of the mirror-holder assembly. A safety cover with glass window was also provided at both ends to avoid the environmental contamination of the expensive high reflecting mirrors. The three welded ports on the cylindrical CRDS cell, two ports for gas inlet and outlet piping, and the third for pressure gauge with KFC clamp were given on the cylindrical CRDS cell. The CRDS cell hardware was fabricated on the basis of the CAD designs. Laboratory set-up of the cavity with gas and vacuum tubing of stainless steel 316 material which was fitted through a needle valve.

The optical resonating cavity was formed with a pair of high-reflectivity (R = 99.997%) 1″ diameter concave mirrors of radius of curvature (r = 1 m) which were AR coated at 405 nm and procured from CRD-optics. The spacing between the two mirrors d was chosen 80 cm so as to satisfy the criterion of a stable cavity ($0 < d < r$ and $r < d < 2r$). A 5 mW *He-Ne* laser was used for initial alignment of the two mirrors for aligning the optical cavity with the excitation laser diode lasing at 405nm. The sealed cell was mounted on the optical bread board of size 4×3 feet which was also designed and fabricated with a honeycomb arrangement within two stainless steel 316 sheets. The pressure gauge of scale between 50 mbar to 250 mbar was mounted on the CRD cell for measuring the gas pressure of the cell. The needle valves were connected with the nozzles to control the flow of the gas inside the cell. The nozzle was used to evacuate the residual gas by a rotary pump. Stainless steel tubing was used for the gas flow from the cylinder to CRDS cell and from CRDS cell to rotary pump.

The experiment was conducted by modulating the laser light intensity using a square wave drawn from a function generator model: Rigol, DG100. The computer-controlled laser diode was operated at 60 mW output power. The square pulses of laser light were generated with a duty cycle of 50 %. These square pulses of laser were injected in to the optical cavity after cleaning the beam shape. The output PS-CRDS leak signal was detected using a silicon photo detector procured from Thorlabs Model:DET 10A. The detector output was fed to the voltage input of the SRS 830 lock-in amplifier through a 50Ω terminator. The output digital data of lock-in amplifier was taken to the computer through a RS-232 for data reduction and calculations. The phase shift signals were measured at the first harmonics of the laser modulation frequency.

In PS-CRDS experimental set-up, the CRD cell was initially purged with pure *Ar* gas to remove if any aerosols presents within the cavity. Subsequently, pure NO_2 gas in small quantities was introduced in the cell to measure the absorption coefficients of NO_2 at 405 nm wavelength. Initially, the reference PS-CRDS signal in argon is recorded by measuring the phase values in lock-in amplifier at modulating frequencies of the excitation laser diode between 30-50 kHz. Similarly, the PS-CRDS signals of NO_2 gases at 50 mbar and 60 mbar pressures were recorded by measuring the respective phase-shift values in the lock-in amplifier in the same frequency ranges.

4. RESULTS AND DISCUSSIONS

Cavity ring down technique is widely studied by the tuneable pulsed lasers to calculate the spectrum of molecules under different temperature and pressure conditions. However, since the development of laser diodes, this technique is widely studied as the phase shift cavity ring down spectroscopy (PS-CRDS). There are several groups which are working on the development of the phase shift technique for the development of field-based detection system or the continuous monitoring of NO_2 gas in the environment and reactions of some toxic molecules with a single wavelength of light. The source of light used for such systems was either laser diode or LED. The LED-based phase shift CRDS experiments are known as CAPS that is cavity attenuated phase shift spectroscopy. In last few years, CAPS-based systems have been reported for the detection sensitivity of the instrument for the detection of NO_2 down up to ppbv level. However, in our studies our main emphasis was to check the design of the CRDS cell in MS material and its feasibility to check its detection limit in ppm concentration of NO_2 [19-21]**.** NO_2 molecules have four electronic states in the visible region as $^2A_1$, $^2B_2$, $^2B_1$ and $^2A_2$. The maximum oscillator strength of NO_2 comes from the X $^2A_1$-$^2B_2$ transition. The excitation wavelength thus chosen as 405 nm to detect NO_2 in its trace level concentrations diluted by mixing with *Ar* inert gas[22]. The nitrogen dioxide (NO_2) gas has a strongest absorption band around 405 nm which coincides with one of the electronic transitions of NO_2 [22-24]. The chosen wavelength of 405 nm of laser light hence provides high detection sensitivity for NO_2. In an optical resonating cavity, light intensity is coupled when one of the laser modes coincides with the cavity mode. In this case, several modes will always couple with the resonator as the spectral width of the excitation laser is as large as 3nm. The resonator formed by HR mirrors allows the light to travel inside the high finesse cavity up to several kilometres by retracing the path of the light beam circulating between the two high reflecting (HR) mirrors. The approximate effective path length of the light travelling inside the cavity resonator is given by $d/1\text{-}R$ where d is the distance between the mirrors which is taken as 80 cm and R is the reflectivity of the mirrors considered as 99.99 %. With such configuration for the optical resonating cavity the effective path length is 8000 m.

The laser diode was modulated between 30 kHz - 50 kHz frequencies to avoid the low frequency 1/f noise. The PS-CRDS experimental set-up was initialised first by modulating the excitation laser diode at 30 kHz and adjusting the phase to zero in the lock-in amplifier. The CRD cavity leak out signals were measured by measuring the phase-shift values with a time constant of 100 ms.

Decay time constant was calculated using Eq. (4). To calculate the absorption coefficient and concentration limit of the detection of the CRDS instrument, the phase shift signals were recorded for different modulating frequencies between 30 kHz - 50 kHz. The decay time of the light was measured in *Ar* gas as a reference signal is shown in Fig. 4.

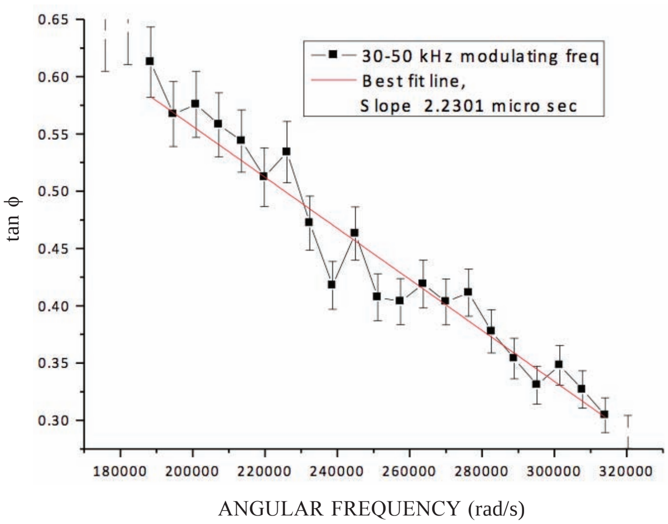

Figure 4. Reference signal in argon measured between 30-50 kHz modulating frequency of the laser diode.

On purging the pure NO_2 at 50 mbar and 60 mbar, modulating intensity signals were recorded and represented in Figs. 5 and 6. The bars in the graph represent the standard deviation in the data points from the mean value. For calculating the absorption coefficients and concentration limits, expressions in Eqn. (6) and (7) respectively were used. The results of the absorption coefficients obtained are well matched with the reported results on the NO_2 by Holc[23], *et al.* The results are presented in the Table 1 which shows the absorption coefficients of the order 10^{-6} cm^{-1}. The absorption cross-section considered for NO_2 in the calculations is taken as $5.5\times10^{-19}cm^2$ to find out the concentration limit. With the present PS-CRDS set-up, concentration measurement limits can be achieved as low as ppb levels. The results show that for NO_2 detection, the achievable concentration limit of 10^{11} cm^{-3} which corresponds to ppb level detection.

Table 1. PS-CRDS experimental results for pure NO_2 gas in argon

Gas	Decay time (μs)± standard deviation	Abs. Coeff. (cm^{-1})	Conc. limit (cm^{-3})
Ar (ref.)	2.2280 ± 1.26×10^{-7}	-	-
Ar+NO_2 @50mbar	1.7390 ± 0.986×10^{-7}	4.21×10^{-6}	1.50×10^{11}
Ar+NO_2 @60mbar	1.7207 ± 1.132×10^{-7}	4.41×10^{-6}	2×10^{11}

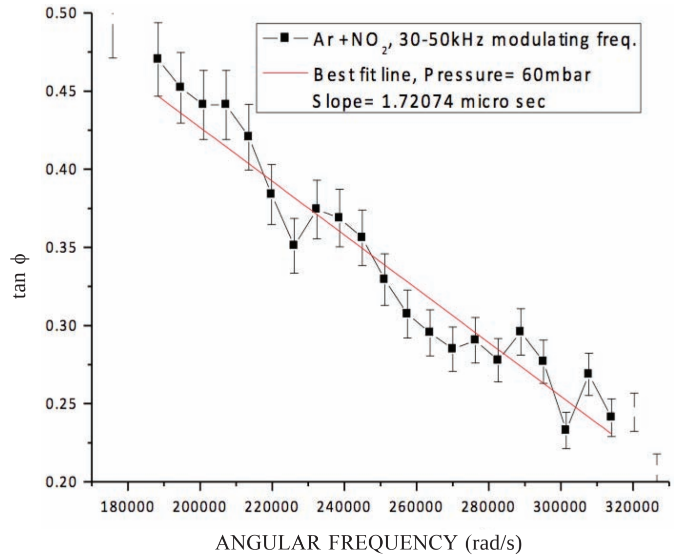

Figure 5. Reference signal in argon and pure NO_2 gas at 50 mbar measured between 30-50 kHz modulating frequency of the laser diode.

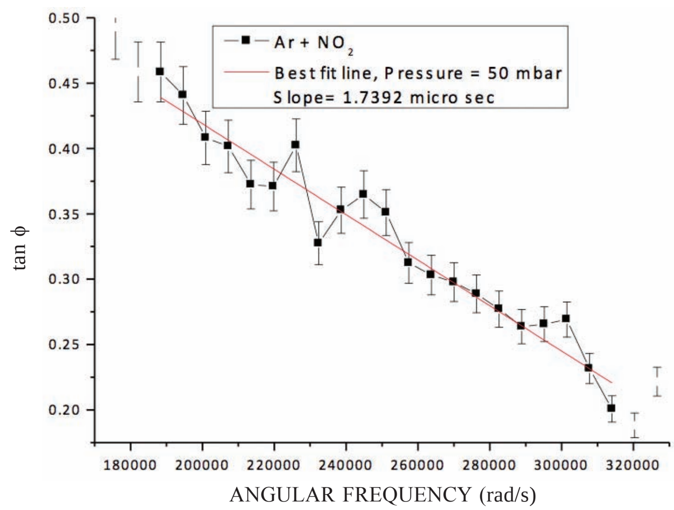

Figure 6. Reference signal in *Ar* and pure NO_2 gas at 50 mbar measured between 30 kHz - 50 kHz modulating frequency of the laser diode.

5. CONCLUSIONS

A broadband CW laser diode was modulated to achieve the phase shift cavity ring down spectroscopy (PS-CRDS) experimental set-up. The design of the CRDS cell was well tested by fabricating the CRDS cell in mild steel material for the detection of trace level concentration of toxic gasses. The CRDS cell was further tested for the NO_2 in argon environment. The decay time constants were measured from the slope of the phase shift graphs versus angular frequency of laser modulation for the pure NO_2 in argon gas environment at 50 mbar and 60 mbar pressure. Thus, the corresponding absorption coefficients values were deduced. Using the given parameters, the detection limit of the instrument can be calculated and its minimum detection limit achieved is in the ppb level per cubic centimetres. A simple PS-CRDS set-up like this can be installed on the mobile platforms for sensing and accurate measurement of trace concentrations of toxic NO_2 gas emissions in metropolis and industrial complexes.

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CONTRIBUTORS

Ms Cherry Dhiman received her BSc (H) Physics from Deen Dayal Upadhayaya College, Delhi University, and MSc (Physics) from Punjabi University, Patiala. She is currently pursuing her PhD from Jamia Milia Islamia, New Delhi. She has worked as a project assistant on a DRDO funded project on 'Detection of traces of toxic gases/Explosives using cavity ring down laser spectroscopy' at Jamia Milia Islamia (JMI), New Delhi.

Dr Mohd. Shahid Khan did his MSc Physics (Material Science) from Jamia Millia Islamia, New Delhi, in 1992. He completed his PhD (Physics) from JMI in 2002. He has been teaching Physics at JMI since 2004 and is presently working as Assistant Professor. His areas of research are laser spectroscopy, non-linear optics and computational molecular and nanoscience. He has authored 40 research publications.

Dr M.N. Reddy is Scientist 'G' and Divisional Head of Advanced S&T and Photonics Division at LASTEC, Delhi. He has done his MSc and MPhil in Physics from Central University of Hyderabad, Hyderabad, and PhD in Laser High Resolution Spectroscopy of Rydberg Atoms from IIT, Kanpur. His present R&D activities include development of laser based sensors for stand-off detection of explosives and mines and high power fibre lasers for directed energy systems.