TUNABLE DIODE LASER ABSORPTION SPECTROSCOPY OF GASES FOR CALIBRATION-FREE MEASUREMENT OF CONCENTRATION AND PRESSURE

NISHANK JAIN

DEPARTMENT OF ELECTRICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY GANDHINAGAR

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Nishank Jain

Tunable Diode Laser Absorption Spectroscopy for Gas Sensing

Tunable Diode Laser Absorption Spectroscopy (TDLAS) is a technique used to measure the concentration of a gas or a mixture of gases such as Carbon Dioxide, Methane, Carbon Monoxide etc. using laser diodes, which are tunable over a particular wavelength range, and performing laser absorption spectrometry using these special laser diodes.

Absorption spectrometry involves radiating a sample (gas in our case) with light (Laser in our case). The sample absorbs energy (photons) and the intensity of absorption varies as a function of frequency, and this variation is the absorption spectrum. Absorption Spectroscopy is performed across the Electromagnetic Spectrum. When the source of radiation is a Tunable Laser Diode, it is called TDLAS.

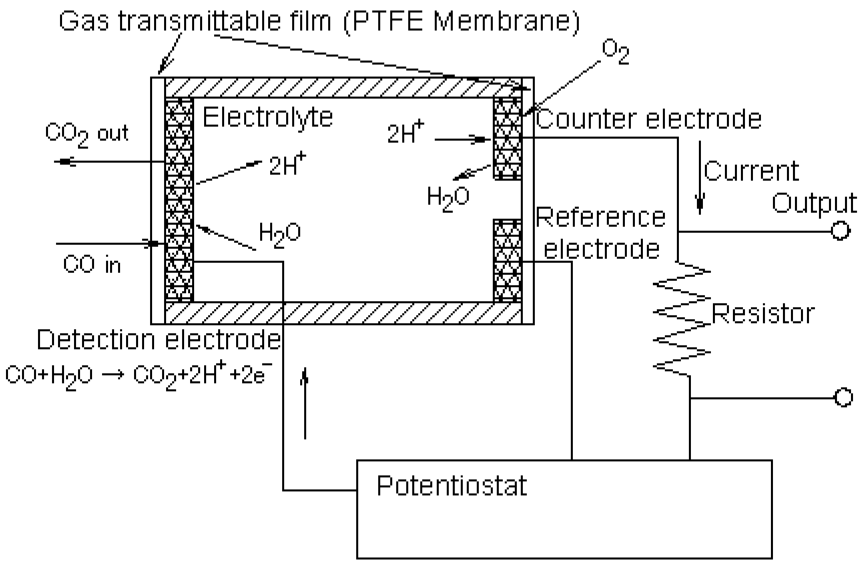
TDLAS can also makes it possible to determine the pressure and temperature of the gas under observation.

Need for TDLAS

Conventional gas detection systems have certain drawbacks when it comes to operational time and response time as well as sample consumption. Some of the common methods are described below:

1. Electrochemical Gas Sensors
   These are gas detectors that measure the concentration of the target gas by oxidising or reducing it at an electrode and measuring the resulting current.
   The major disadvantage of using this system is the problem of cross-sensitivity. Some gases which are very easily oxidised such as alcohol or CO interfere with the detection of the target gas and also give a response unless a chemical filter is used. Also this process is applicable only for those gases which are electrochemically active. Inert gases do not give results.

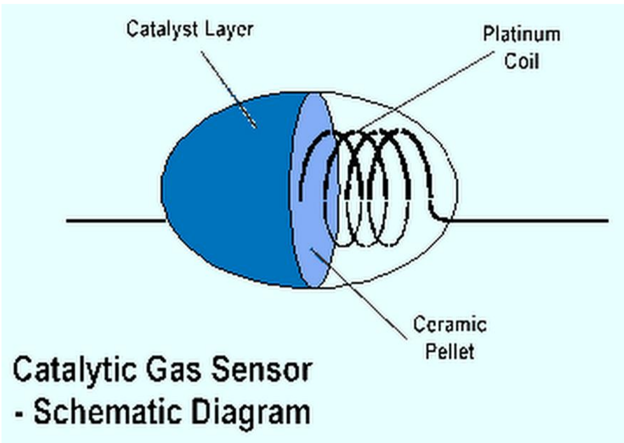
   The figure below is an example of the Electrochemical Gas Sensor for the Carbon Monoxide Gas. Another drawback of this method is that it is destructive in nature which means that it uses up part of the sample of the gas under observation which might be crucial if gas detection is done in remote areas where its availability is very less.



2. Pellistor

   Pellistor is made up of two words – pellet and resistor. It is a solid-state device used to detect gases which are either combustible or have a significant difference in thermal conductivity to that of air. The detecting element consists of small pellets of catalyst loaded ceramic whose resistance changes in the presence of gas.

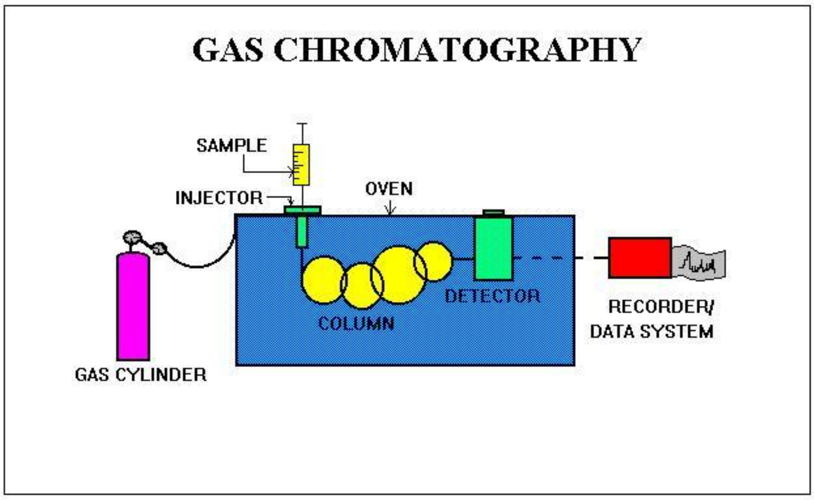
   Again this has the disadvantage of being destructive in nature.



3. Gas Chromatography

   A common type of chromatography used in analytical chemistry for separating and analysing compounds that can be vaporized without decomposition. The mobile phase is a carrier gas, usually an inert gas and the stationary phase is a microscopic layer of liquid or polymer of an inert solid support. The gas interacts with the walls of the column, which is coated with different stationary phases which causes the compound to elute at a different time, known as the "retention time" of the compound. The comparison of retention times gives analytical usefulness to Gas Chromatography.

   This has a disadvantage of being bulky in nature and having a slow response.



So, what is demanded from a modern gas sensor is:

1. High Sensitivity
2. Real-time Detection
3. Low Cost
4. Non-Destructivity
5. Remote Detection
6. Portability

This is where the principle of TDLAS comes into frame. Apart from the cost factor, it satisfies each and every condition or necessity as mentioned above. And the cost factor is one which can be taken care of once the technology is in mass production.

WHY GAS SENSING?

Gas is something which is found everywhere, whether it's low or high in concentration is a different case altogether, but in most cases some or the other type of gas is present even in traces. This makes it useful to develop a system which can detect gas and make some useful measurements pertaining to temperature, pressure and concentration. Such type of system finds great applications in varied fields some of which are mentioned below:

1. Pollution Control System
   Air pollution is a major cause of concern to humans and animals and plants alike causing them harm or discomfort and thereby causing damage to the natural environment. Major primary pollutants produces by human activities include:
   Sulphur Oxides, Nitrogen Oxides, Carbon Monoxide, Carbon Dioxide, Methane and other Hydrocarbons, Particulate Matter, Chlorofluorocarbons, Ammonia etc.

An independent control system to monitor the amount of harmful gases in the atmosphere is quite useful to detect and warn in advance of the effects it may cause to life using remote detection and non-destructive techniques.

2. Process Control Industry
   Process control enables mass production of continuous processes such as oil refining, paper manufacturing, chemicals, power plants and others. It enables automation of certain complex processes saving a huge amount of human labour. But here also there arises a need for a system which can remotely monitor real-time changes in the gas temperature or pressure and thereby helping in accurate process control.

3. Security
   Some explosive such as the Trinitrotoluene (TNT), Research Department Explosive (RDX) etc. emit gases which can be picked up by highly sensitive gas sensors and which can help in improving the security systems vastly.

4. Healthcare
   A human inhales and exhales a certain amount of different gases in the process of respiration. The characteristic quantities of the gases present in the breath provide a great insight into the extent of a disease (if it's there) in the human body. For each value of a concentration of a gas in the breath, there are Biomarkers which point to the normal value of some gas in the average healthy human breath sample. If the levels are abnormally high or low, it can be said easily which disease it corresponds to. Here the non-invasive technique of the laser-based gas detection system proves to be highly efficient and comfortable.

Principle of TDLAS

A basic TDLAS setup includes the following things:

1. A Tunable Diode Laser Light Source (DFB Laser)
2. Transmitting Optics (Optical Fibres and Couplers)

3. Optically Accessible Absorbing Medium (Gas Cell)
4. Receiving Optics (Optical Fibres and Couplers)
5. Detector (Photodiode)

Each gas has characteristic absorption lines of particular wavelengths in the Electromagnetic Spectrum. So first a tunable diode laser is found which has a wavelength that lies in the absorption region of the target gas, then the emission wavelength of the laser is tuned over the characteristics absorption lines of the target gas lying in the path of the laser beam. This causes a reduction in the measured signal intensity detected by a photodiode, which is used to determine the concentration, temperature etc. of the gas.

So, here a RAMP signal is generated through a function generator or a circuit and it is fed to a CURRENT CONTROLLER which controls the wave-shape and the amount of light emitted by the DFB (Distributed Feedback) Laser Diode. This light is passed through a GAS CELL containing the target gas and the Laser Diode's wavelength is tuned to the absorption line of the target gas using the Current Controller. When the wavelengths match, there is absorption of light by the target gas and the intensity of the output light coming out of the other side of the gas cell is reduced. This intensity is detected by the PHOTODIODE and it converts the light signal into a voltage signal and feeds it to an OSCILLOSCOPE which may be further controlled by a PC or may provide data to it.

Different laser diodes are used based on the application and range over which the tuning is to be performed. What is used in our lab is a 1650 nm DFB laser diode as well as a VCSEL laser diode and an InGaAs photo-detector. These lasers can be tuned both by adjusting their temperature and by changing the injection current using the TEC (Temperature Controller) and LDC (Laser Diode Controller) respectively.

DIRECT ABSORPTION SPECTROSCOPY

The absorption follows the Beer-Lambert Law:

$$\mathbf{I_t\,(\lambda)=I_o(\lambda)e^{-A(\lambda)l}}$$

$I_t\,(\lambda)$=Transmitted Intensity

$I_o(\lambda)$=Incident Intensity

$A(\lambda)$=Absorbance of the Medium

If the photodiode signal vs the time is plotted, a plot similar to that in figure 1 is obtained.

If the absorbance vs the tuned wavelength is plotted, a plot similar to that in figure 2 is obtained.

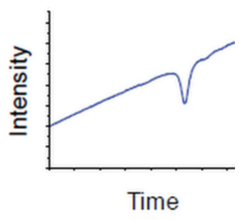


Figure 1

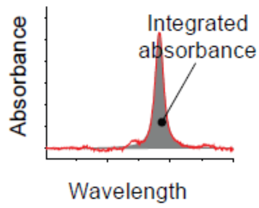


Figure 2

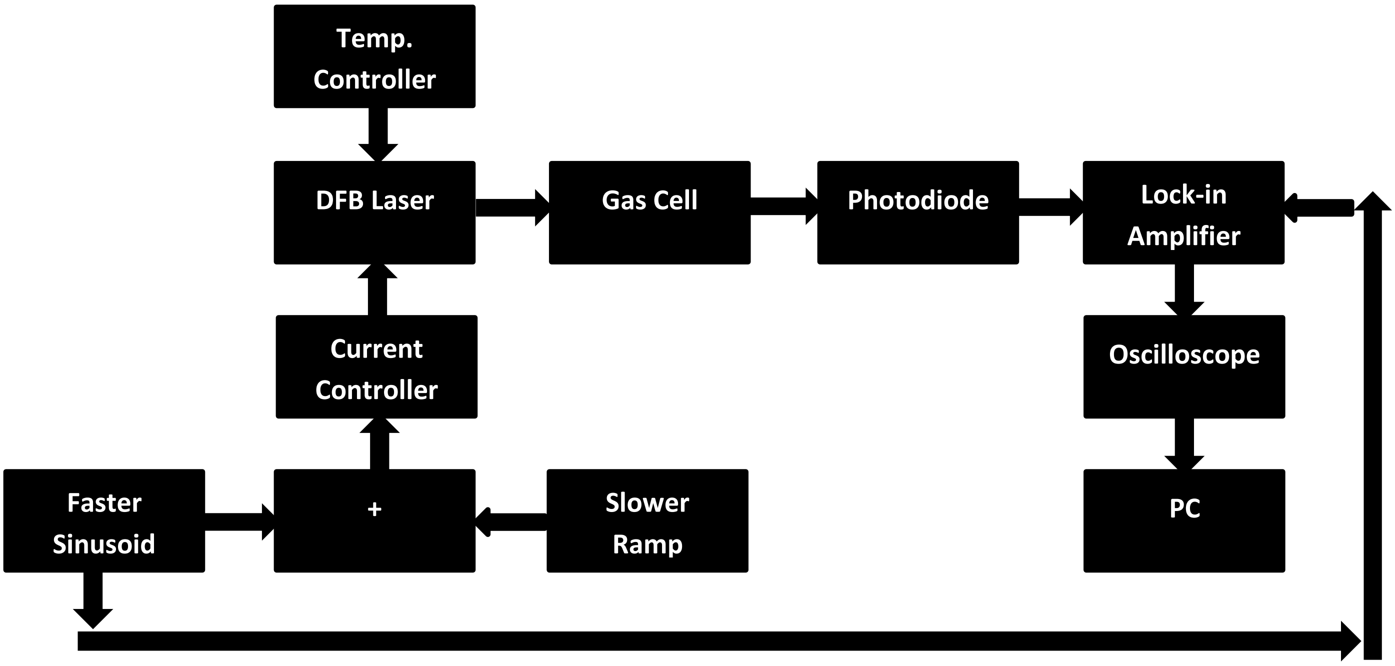
Direct Absorption Spectroscopy has limited sensitivity because of the noise introduced by the optical system or the light source. There are two ways to handle this situation:

1. Reduce the noise level in the signal – using Wavelength Modulation Spectroscopy
2. Increase the absorption – using a multi-pass cell to increase the interaction length.

Wavelength Modulation Spectroscopy

Here Wavelength Modulation Spectroscopy (WMS) is used to reduce the noise level in the circuit and improve the signal-to-noise ratio. The absorption signal is detected at a high frequency where the noise level is low. In WMS, the wavelength is continuously scanned across the absorption profile of the target gas and the signal is detected at a harmonic of the modulation frequency like 1f or 2f etc. Here a fact has been used which states that technical noise reduces with increase in the frequency, which is the reason for it being referred to as the 1/f noise.

When the laser diode is modulated with the TEC or the LDC, there is a change in wavelength (wavelength modulation) which can be interpreted as a change in frequency (frequency modulation), but what is less obvious is an accompanying change in the Intensity of the light being emitted by the laser diode. The intensity vs current characteristics of a laser diode is a non-linear increasing curve which implies that as the current is changed using the LDC to modulate the wavelength of the laser, it is accompanied by a parallel change in the intensity of the laser light which is undesirable as it contains no information and it also affects the dynamic range of the signal that is obtained after the lock-in amplifier. This means that if it is desired to amplify a small signal and the undesirable DC level creeps in then there is a limitation due to the saturation level of the photodiode and it would not be possible to obtain the full range of the signal from the photodiode. This is called **Residual Amplitude Modulation (RAM)**. The technique to reduce this "residual amplitude" is called **RAM Nulling**. The circuit used in Wavelength Modulation Spectroscopy is shown below.



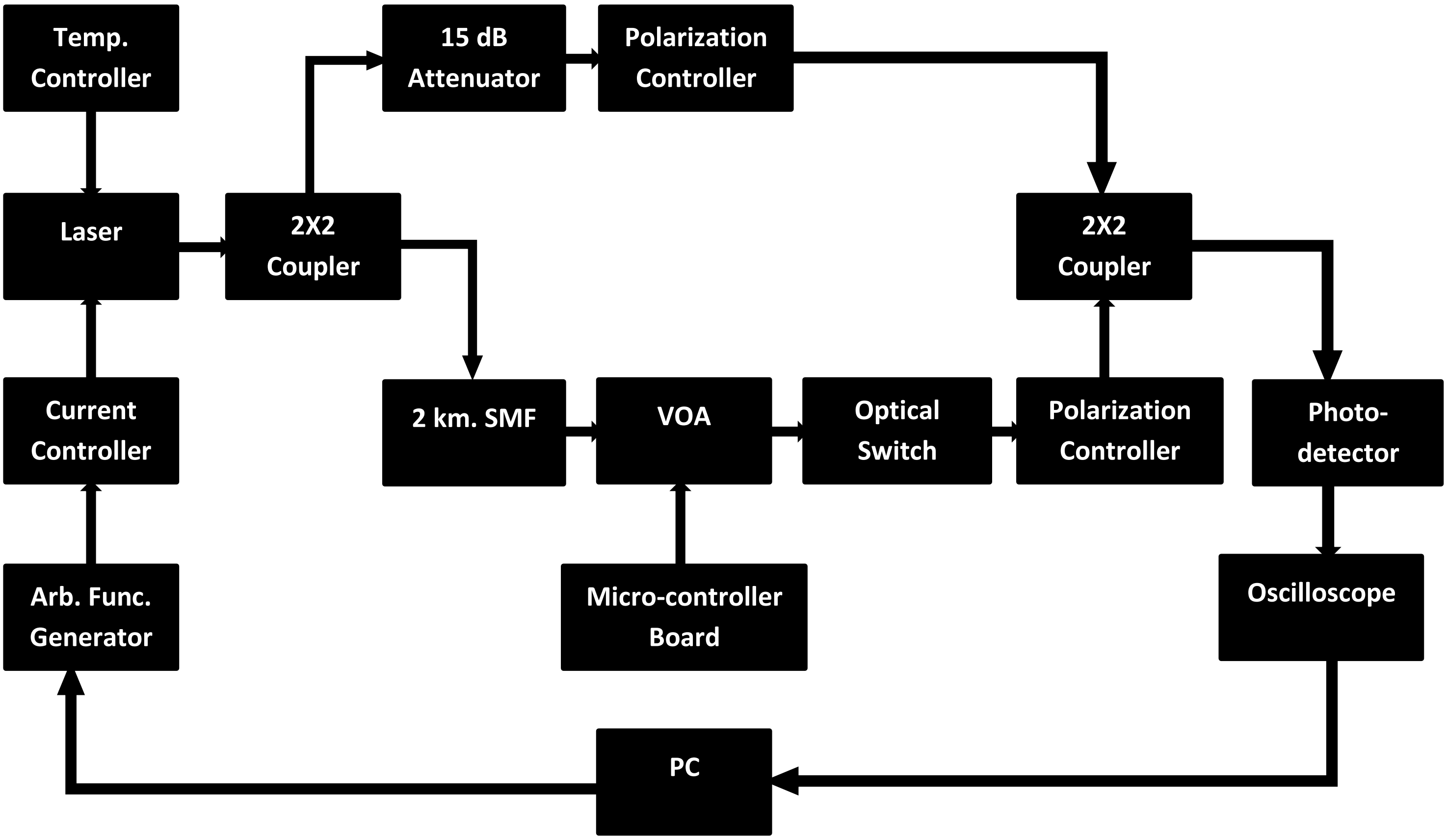
Circuit used for Wavelength Modulation Spectroscopy

A high frequency sinusoid is superimposed on a low frequency ramp signal and give it as an input signal to the Current Controller which modulates the DFB Laser Diode which is also controlled by a Temperature Controller. The laser radiation is passed through a gas cell containing the target gas and the resulting output radiation is collected by a photodiode which gives it as an input signal to the Lock-in Amplifier. The Lock-in Amplifier removes the ramp signal by normalizing it and converts the absorption dips into 1f, 2f etc. signals. The output is observed on an oscilloscope which is synched to a PC. The Lock-in Amplifier is triggered by the same sinusoid used at the start.

But now the question arises, where does the sinusoid go because it does not appear in the Lock-in Amplifier output? The answer is RAM Nulling. To avoid the effect of the residual amplitude in the form of increased intensity level, the laser output is split into two equal halves as shown in the figure below. The sinusoid also therefore splits into two equal halves (half the intensity each).

One signal is passed through the gas cell and other is passed through a 2km. Single-Mode Fiber, VOA and an Optical Switch. Finally both the signals are combined into a coupler and detected by a photo-detector whose output is observed on an oscilloscope and synched to a PC.

RAM Nulling Circuit



Experimental Setup for LD-1665-0010-DFB-1 Diode Laser

The Arbitrary Function Generator (controlled by a PC or synched to it) gives a superimposed signal consisting of a Low-Frequency Ramp and a High-Frequency Sinusoid to the Current Controller which in-turn modulates the laser diode whose temperature is maintained at a constant level by a Temperature Controller.

The Laser beam is split into two equal halves (intensity) using the 2X2 coupler because it cancels the effect of residual amplitude on both sides.

One arm contains the simulator of a gas cell in the form of a 15 dB attenuator and a polarization controller which absorbs light and gives reduced intensity at the other 2X2 coupler.

A 2X2 Coupler

The other arm contains a 2km. long Single-Mode Fiber (SMF) which produces a phase difference of 180^{0} with respect to the first arm which is required to destructively interfere with the amplitude of the sinusoid in the first arm resulting in a straight DC line with only prominent absorption lines of the target gas.

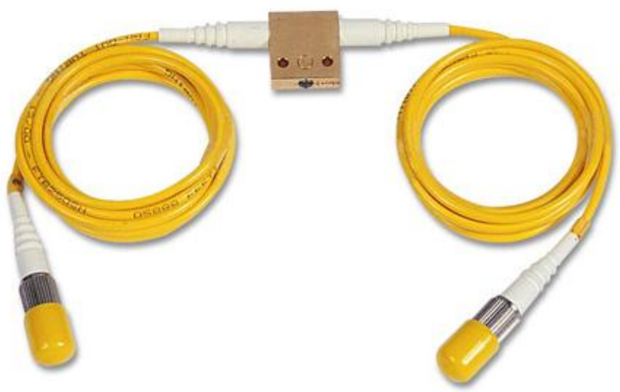
A 2km. SMF

The amplitude of the sinusoid is reduced when it passes through the first arm, so it is required to decrease the amplitude in the second arm too with an accurate and precise technique for which a VOA is used.

VOA is short for Variable Optical Attenuator, which is used to reduce the amount of light passing through it by decreasing the voltage across it's positive and negative terminals. The output light of the VOA is passed through an Optical Switch which is used to decide whether to pass on the light further to the polarization controller to complete the second arm and the process of destructive interference or to lay the second arm dysfunctional and observe the output of the first arm only.

The final outputs are coupled through a 2X2 coupler where destructive interference takes place and the output is detected using the photo-detector. This output is observed on the oscilloscope in the form of a voltage signal and is further synched to a PC.

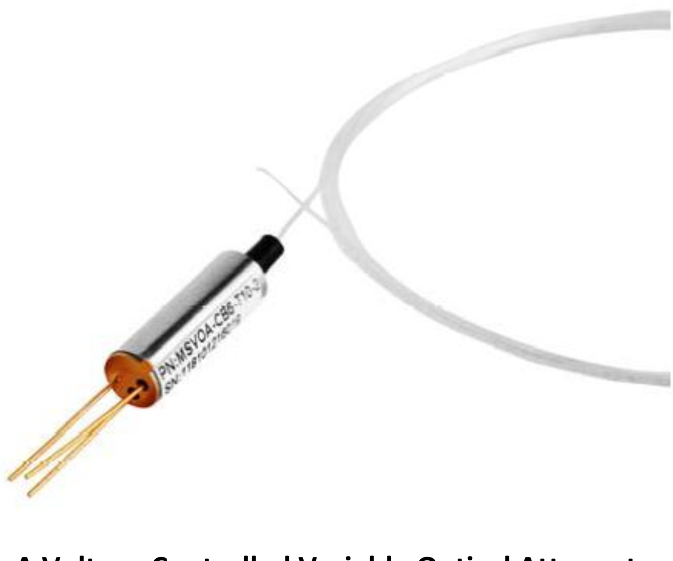
The whole process needs to be made compact and automated so that field measurements can be made. The first step in the control of a part of the circuit is taken in the form of manual control of VOA.



A Manual VOA

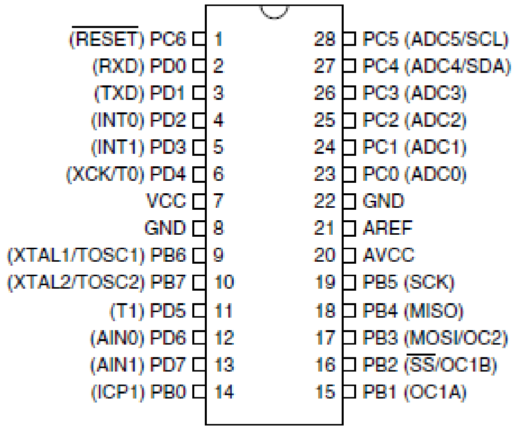
The manual VOA is used to control the amount of light (intensity) through the VOA by rotating a knob (which is a manual change in the resistance) using a screwdriver which makes it less accurate and undesirable to use in terms of field measurements because whatever feedback it receives (whether to increase the intensity or decrease it) is actually coming through human intervention. So, it is best to replace this Manual VOA with an automated system that controls the light passing through it by applying a controlled variable voltage across it to change the resistance.

The voltage across the terminals of the VOA can be controlled by breaking it down into fine steps (large number of steps). More the number of steps, better is the resolution. The voltage that appears across the VOA is analog voltage but to break it into finer steps a digital system is needed to carry out the division. Therefore, an ADC (Analog to Digital Convertor) is used which is present in a microcontroller to divide the voltage appearing across the VOA into steps.



A Voltage Controlled Variable Optical Attenuator

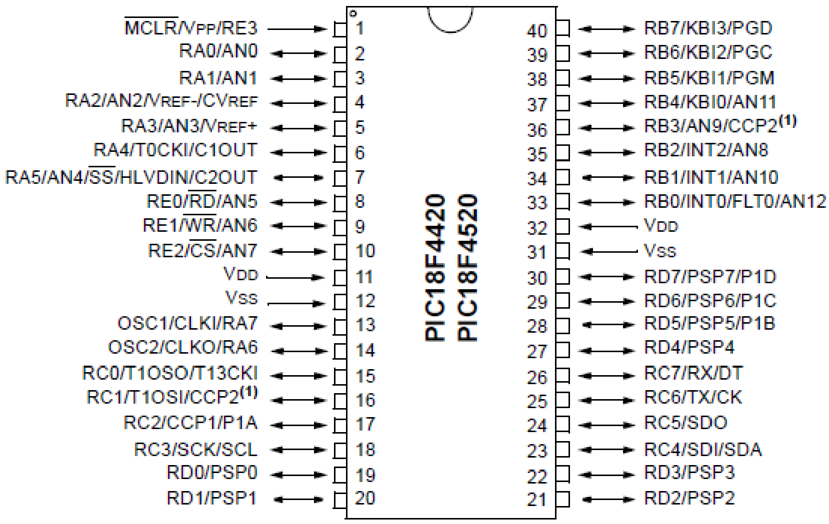
The microcontroller used can be an Arduino MC. The Arduino MC has different types of ADCs built into it – 8 bit, 10 bit, 12 bit. The one which was being used was a 10 bit ADC of which 8 bits were being used. By 8 bits, it is implied that if 5V is supplied to the terminals of the VOA, it would appear in 2^8 steps i.e. 256 steps of 5/256 V each. This is what is meant by the resolution of ADC.



ATmega 8 Microcontroller used in an Arduino

The Arduino has a limitation of the clock frequency which affects the speed with which one can operate the system and on a larger scale it can be seen in terms of effects on real-time monitoring strategies. So it is required to turn to another class of microcontrollers – the PIC MC.

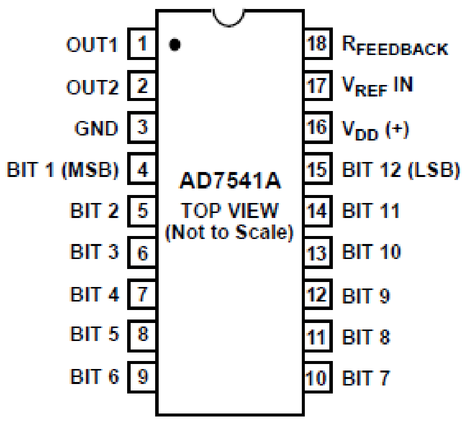
PIC MC works on a higher clock frequency and therefore has higher operational speed and large memory space too which increases the number of bits (resolution). The PIC 18F4520 MC has been used for this purpose. It consists of a LCD display too which can be programmed to display the voltage across the VOA as well as the resolution of the MC.



PIC 18F4520 Chip Configuration

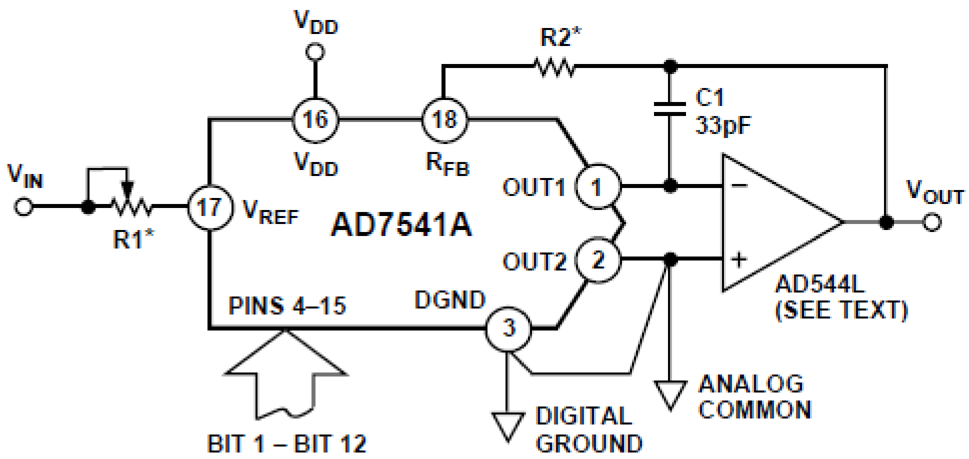
But the question arises, if the analog voltage is converted into digital voltages of some magnitude, then how does the VOA respond to it when it is known that it only responds to analog input voltages?

The answer is the use of a DAC (Digital to Analog Convertor). The DAC, as the name itself implies, converts the digital voltage from the ADC of the MC to an analog voltage for the VOA to operate correctly.



AD7541A 12-bit DAC

The circuit used for the same purpose is as shown below.

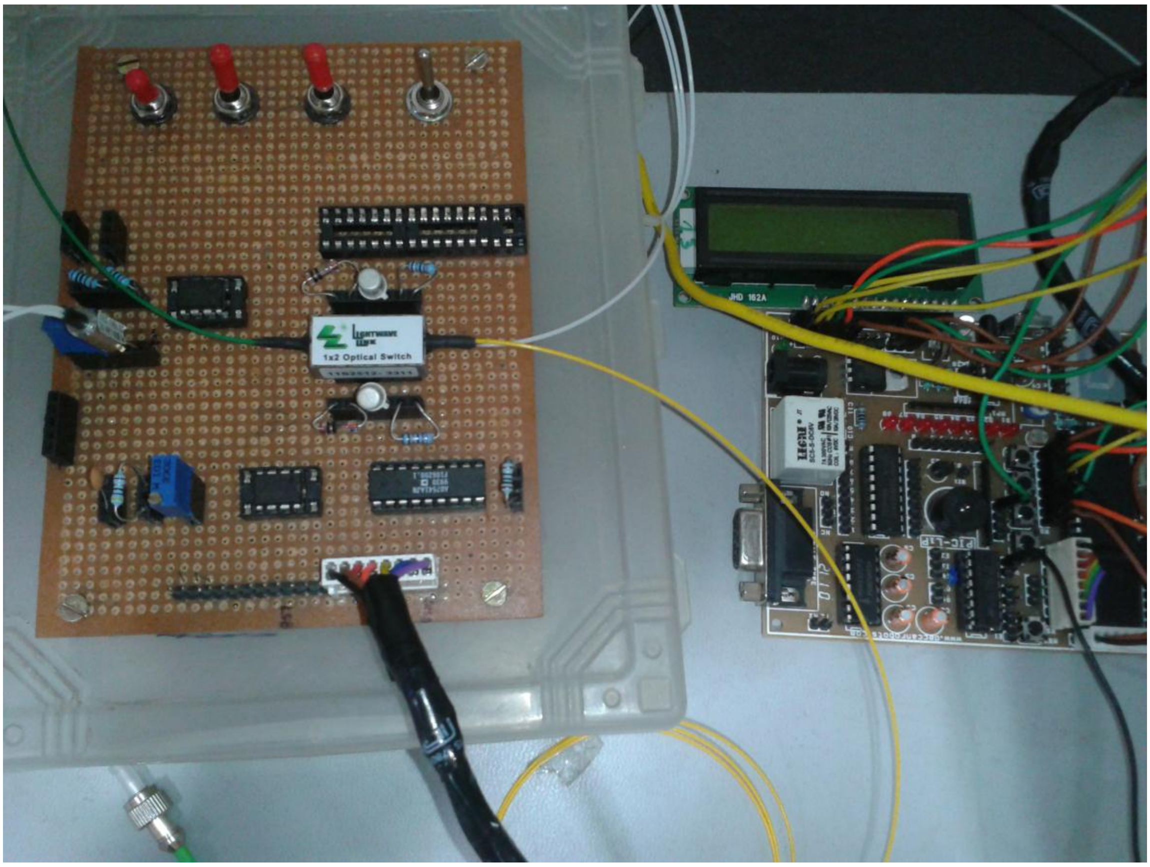


Bits 1-12 are provided from the ADC of the PIC MC and the analog voltage is taken from the 6th pin of the AD544L OpAmp and fed to the VOA. Rest all the parameters are set as mentioned in the datasheet and according to the requirements of the circuit.

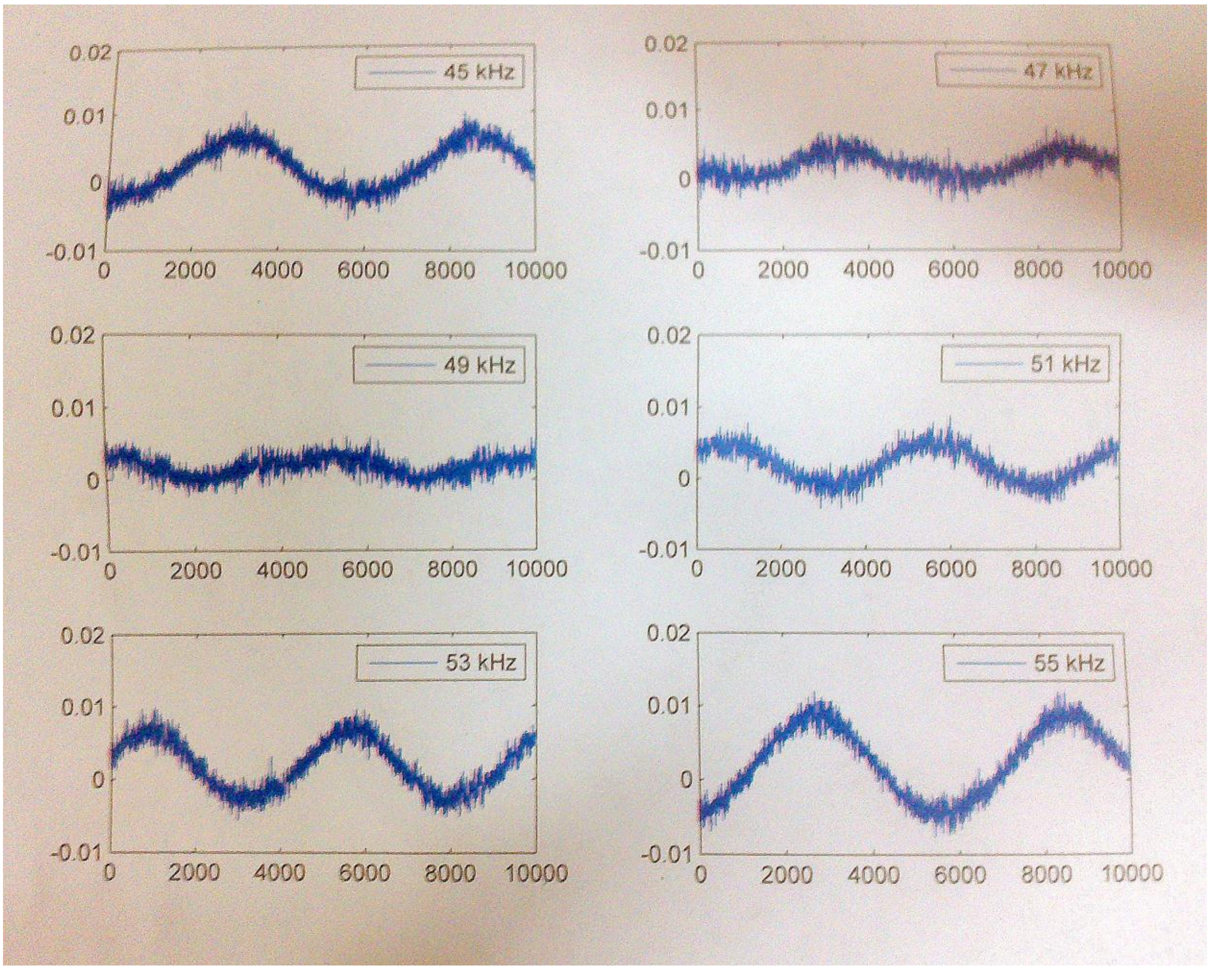
It is a 12-bit DAC and it follows the following conversion table.

Binary Number in DAC MSB		LSB	Analog Output, V_{OUT}
1 1 1 1	1 1 1 1	1 1 1 1	$-V_{IN}\left(\frac{4095}{4096}\right)$
1 0 0 0	0 0 0 0	0 0 0 0	$-V_{IN}\left(\frac{2048}{4096}\right) = -1/2\ V_{IN}$
0 0 0 0	0 0 0 0	0 0 0 1	$-V_{IN}\left(\frac{1}{4096}\right)$
0 0 0 0	0 0 0 0	0 0 0 0	0 Volts

The control system designed using the PIC MC also contains a feedback mechanism included in the program embedded into the chip. This mechanism has been designed to provide the MC with data from the final nulled signal to determine whether to increase the voltage across the VOA or to decrease it in accordance with the amount of intensity still needed to accurately obtain the nulling.



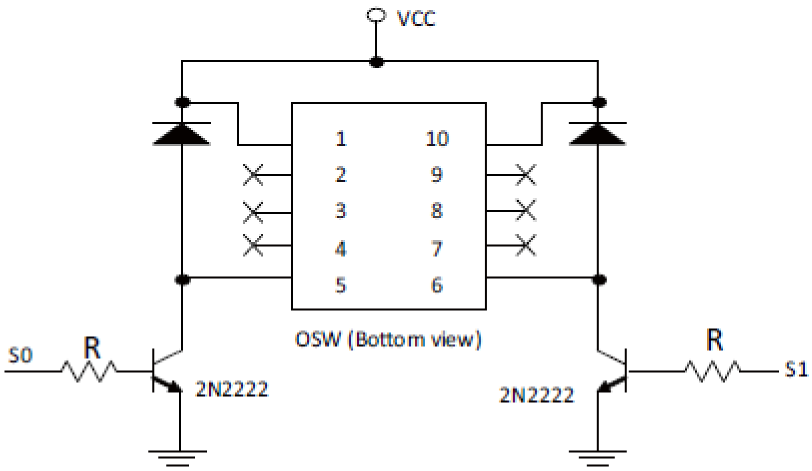
The PIC MC with all the components of the RAM Nulling Circuit on the PCB



<u>Plots showing the amount of nulling obtained at different frequencies of applied sinusoid</u>

The output of the VOA is given to a 1X2 Optical Switch.

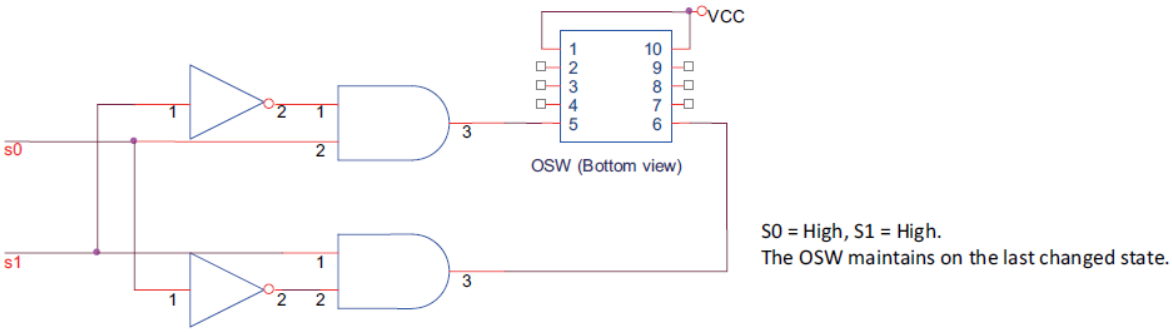
The circuit used for the Optical Switch is as shown below. It consists of two 2N2222A power transistors to supply the required amount of power needed to operate the OSW. It is a 1X2 optical switch which means that it has single input and two output channels which are fiber-coupled to connectors.



The Optical Switch (OSW) is used for two purposes:

1. Obtaining just the signal from the Gas Arm on the oscilloscope.
2. Obtaining the nulled signal using both the arms on the oscilloscope.

For operating it with a switch, it has been recommended in the datasheet to use the following circuit.



This circuit is needed to avoid the situation in which both S_0 and S_1 stand at high level simultaneously which again should be avoided for the Optical Switch. This case arises when the OSW is operated with a switch to alternate between its two channels.

But on experimentation, it was found out that the current output of the AND and NOT Gates is not sufficient to drive the whole circuit and therefore the circuit does not work. On further experimentation, it was found out that the OSW can be operated directly with a switch because the switching time is very less and therefore it is not affected by the alteration of the power switch and gives the output correctly.

FUTURE WORK

OBTAINING NI DATA ACQUISITION CARDS

Work is going on to obtain NI (National Instruments) cards which will help in making the whole process automated through a software - LAB View, which substitutes for the DSOs and Function Generators. Everything is operable using a computer with fast processing capabilities and real-time monitoring can be done.

DSPIC REPLACES PIC 18

The next step in the advanced automation of the system is using a MC capable of signal processing and filtering in place of the PIC 18 MC. This is easily done by a dsPIC MC which even has a large memory space as an added advantage which is useful for increasing the resolution further. Filtering is needed to eliminate the noise coming in from the power supply as well as the noise from the Optical Circuit. Signal Processing takes the signal closer to accuracy and makes it easier to measure different parameters of the target gas.

16-BIT DAC

The dsPIC MC controller supports a resolution up to 16-bits, so it is necessary to include in the new design a DAC with a resolution of 16-bits for which the AD5547 IC is to be used which is currently in the development phase of the project.

COMPACT CIRCUIT

The final aim of the whole project is to perform field measurements using a compact portable system, so it is necessary to pack the whole system in a small volume which is easy to carry and operate but without compromising on the quality of the results. Work is to be done in this regard as the number of instruments keep increasing on a monthly basis, so LABView can prove to be of great use here.

SOFTWARE LOCK-IN AMPLIFIER

Currently, hardware lock-in amplifier has been employed to eliminate the ramp signal and give the 1f and 2f signals by acting as a narrow band-pass filter. The same thing can also be achieved using software lock-in amplifier by employing the dsPIC to carry out the band-pass filtering through programming.

CONCLUSIONS

This project is basically a research and development project with final target being to develop a relatively inexpensive (for some specific purposes) product which can be used to perform gas sensing in any kind of surroundings possible.

Some of the important conclusions that have been drawn from the project are as follows:

1. A dsPIC gives better resolution and operational capability as compared to other equivalent microcontrollers and therefore finds greater use in the final product if the situation is such that the final product contains a PIC MC to operate the VOA and not a PC.
2. The lesser the hardware, more is the processing speed and accuracy as well as ease of operation. The aim is to build a panel which contains all the operational knobs and switches and screens etc. so that the system is almost completely computer operated with minimal necessary hardware and compact design.
3. Electrostatic Sensitive devices such as the 1650 nm DFB laser diode or the 16-bit AD5547 DAC etc. should be touched only when the human body is grounded with the power supply’s ground so that there is no flow of charge from the body to the device which may otherwise result in permanent damage to the device, which most of the times are pretty expensive.
4. The pressure sensor installed on the gas cell is not very accurate as it does not actually makes sure whether the pressure being measured in the cell is over the atmospheric pressure. So, a new pressure sensor needs to be developed for this purpose.
5. The temperature sensor also gives inaccurate readings as it is not completely immersed in the gas. Any physical contact with the body of the gas cell may result in incorrect temperature readings of the gas inside the cell which leads to incorrect calculations of other parameters.

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