

# Standoff Detection of Hazardous Molecules for Defense Applications

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### Abstract

The paper is focused on the development of measurement technique and processing of signal for the detection of chemical, explosive, biological agents and its simulants using Engineered Quartz Enhanced Laser Photoacoustic Spectroscopy (QE-LPAS) technique. Hazardous materials like Sarin, TATP (Tri acetone tri-peroxide) and their simulants like DMMP (Dimethyl Methyl Phosphonate), Acetone, and atmospheric species were detected at 7 to 11 µm wavelength band. QE PAS technique is developed and demonstrated from a standoff distance of up to 30 meters in gaseous/vapourous and aerosols based on retro-reflector in ~2.0 ppm concentration. Hazardous biomolecules like Tryphatophan and DPA were detected on diffused aluminum adsorbed surface plate. Explosive and its simulants, TNT on adsorbed surface and DMMP, Nitrobenzene, Acetone were detected in vapour/aerosol form. A dedicated single screen, single user, user friendly Graphical User Interface (GUI) for controlling the entire system, acquisition and processing of the incoming signal and demonstration of results has been developed with the help of Laboratory Virtual Instrument Engineering Workbench (LABVIEW).

**Keywords:** Phase sensitive detection; Lock-in amplifier card; Hazardous chemicals; Bio-molecules; Greenhouse gases and explosive detection system; Graphical user interface; Labview; Photoacoustic spectroscopy; Quantum cascade laser

## Introduction

The laser absorption spectroscopy has become rapidly growing area of research after the invention of Quantum Cascade (QC) laser [1]. It is emerging as potential alternative to other analytical methods such as FTIR for trace-gas spectroscopy. The QCL is a new type of mid-IR tunable semiconductor laser, based on inter-sub band transitions in a multiple-quantum-well heterostructure, designed by means of band-structure engineering grown by molecular beam epitaxy [MBE]. The QCL has attracted much attention in recent years due to its room temperature operation [2] and its superior output power and mode quality as compared to that of lead salt diode lasers. Many vapor phase chemical direct Photo acoustic sensors based on QCL have been demonstrated [3]. The transduction displacement has been studied using the photo acoustic spectroscopy [4]. Laser photo acoustic spectroscopy has been used for trace detection of vapors, gas and aerosols [5-8]. Photo acoustic spectroscopy has also been utilized for detection of explosives and other remote sensing applications [9,10]. Standoff Quartz Enhanced Laser Photo acoustic Spectroscopy (QE-LPAS) technique is emerging as a powerful technique for detection of hazardous chemicals, biological and explosive agents [11]. Aim of the present work is to develop a technique based on QE-LPAS for the detection of hazardous molecules for the forensic application and homeland security. The present technology has the potential to get converted into a compact product for the field operation having capability to detect explosives, hazardous chemicals and bio-molecules in the form of vapor, aerosols and samples adsorbed on solid surfaces. The sensitivity of the Quart Crystal Tuning Fork (QCTF) detector in vacuum condition has also been discussed.

## **Experimental Setup**

Standoff Laser photo acoustic spectroscopy experimental setup is shown in Figure 1. The commercially available QCL sourced from M/s. Daylight Solutions, model Uber Tuner UT-8, and UT-9 (tuning range 7-11 micrometer) with increment wavelength of 1 cm<sup>-1</sup> is used for low resolution, and 0.2 cm<sup>-1</sup> for high resolution. The QCL laser beam is modulated by pulses of 500ns duration at the frequency 33 kHz using a function generator, the maximum average power of the laser is 12 mW (at 40 kHz modulation) at 1275 cm<sup>-1</sup>, Tunable power of QCL (1-12 mW) in the 7-11micron band. Function generator card is used to modulate the laser and to give the reference signal to lock-in-amplifier. The laser is incident on target having adsorbed explosive species. The QCTF detector is having a narrow frequency response centered at 33 kHz. Amplified PA signal from the current amplifier is fed to a lock-in-amplifier card. The time constant of measurement of Lock-in-amplifier card was kept at 10 ms and sensitivity on 3 mV scales. The analog signal from lock-in amplifier card (USB 4716 Advantech ) and recorded on a computer having a LABVIEW based graphical user interface. The samples were prepared by dissolving in Acetone and allowed to dry on the target to form a uniform layer. The solutions were prepared to deposit 5-50



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microgram/cm<sup>2</sup> level quantity of analyte under examination, on the target. The target materials in our case were plastics and cloths. Standoff Photo-acoustic spectra were also recorded for DMMP and acetone in vapor/aerosol forms in open air from a distance of 25 meters. Figure 1, shows the block diagram of the standoff-LPAS detection setup consisting of the various electro-optical components that are required for successful detection of the biological and explosive agents, chemicals and pollutant gases. Tunable Quantum cascade laser radiations (QCL) beam is allowed to propagate to the target via the folding gold coated mirror. Alignment was done with guide beam visible diode laser beam co-aligned with the QCL beam. Mid-IR radiation is absorbed partly by the sample and rest of the radiation gets reflected back from the target. Back reflected radiations were captured by the receiving gold coated mirror optics and focused to the QCTF detector. Detector signals were processed using the pre-amplifier and phase sensitive lock in amplifier and Data acquisition system for detection and identification of the sample. The sensitivity is enhanced using QCTF detector in vacuum condition. A tripod mounted system based on this technique was developed (Figures 1 and 2).

# **Results and Discussion**

Various profiles (signal received by the detector as a function of wavenumber) viz. laser profile, explosive profiles for the detection of Acetone, DNT and DMMP are shown in Figures 3a-3c. Initially, the laser profile was recorded and then in the second run the explosive/ chemical profile was recorded. The laser profile has to be recorded only once at the starting of the experiment. Very frequent changes in the ambient atmospheric conditions in the near earth environment cause the laser profile to change. Therefore, it is compulsory to record laser profile every time we start an experiment. This recorded laser profile can be utilized for detection of different explosives. The laser profile graph has been denoted as Y1 graph and the explosive profile graph



Figure 2: Tripod Mounted Engineered Q E LPAS System.





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Figure 3(b): Standoff LPAS Spectra of DNT on adsorbed surface.



has been denoted as Y2 graph. Both these profiles were recorded and processed (normalized) to obtain a software compatible profile for the detection of dips in efficient way. This normalizes the explosive profile with reference laser profile to obtain the resultant graph and hence clearly indicates the dips that are generated because of the presence of explosive. The target was kept at a distance of 20 m from the Tripod mounted system. The explosives/chemicals have been detected in all the three states of matter. The data obtained indicates repeatability. The detection of hazardous materials has been performed by using both the online detection and the post processing methods. The false alarm rate has been found out to be approximately 5% on the detected explosive materials. The low false alarm rate indicates the very high accuracy of the developed data acquisition system. Moreover, the response time of the system is of the order of few ms. The data of the online detection was stored in various files for post detection analysis. The dip position of the various explosive materials depends on various factors like atmospheric conditions, distance of the target from the sensor, characteristics of the explosive material. Therefore, the dip values lie within a certain band of  $\pm$  5 (cm-1) from a central value. Therefore, there is a slight change in the value of dip positions every time an experiment is conducted. The software has been designed in such a way that a slight change of  $\pm$ 5 (cm-1) in the wave-number has been made accountable. Moreover, a 'zoom' factor has been added for increasing the amplitude in all the graphs. The total time taken for processing the information and for detection of hazard and displaying the hazard on the screen is less than 5 sec. The P A Spectra of Acetone, DMMP, and DNT are recorded in Figures 3a-3c. The vibrational bands of these hazardous molecules are assigned and peak positions are given in Table 1.

The High resolution photoacoustic spectral lines of HCHO,  $CH_4$ ,  $SO_2$ ,  $H_2O_2$ ,  $NO_x$ ,  $CO_2$  and  $O_3$  and special greenhouse effect gases  $CO_2$ ,  $CH_4$ , etc. were recorded in Figure 4. These spectra are recorded at

lower level (Earth) atmospheric condition in open path from distances up to 30 meter. The assignments of the lower Earth atmospheric/ environmental species are given in Table 2. High resolution photo acoustic Spectra are given in Figure 4. The Signal to noise ratio (SNR) of LPAS signal received from the samples for distances up to 20 meter was varied in the experiments. Therefore, the standoff detection range can be increased further. Further retro-reflector based standoff LPAS experiments will be carried out from distances up to 100 meter. Further enhancement in LPAS signal can be done by putting QCTF detector in evacuated enclosure with 25 Torr pressure. Figure 5 shows, 3 times enhancement in PAS signal at 25 Torr in comparison to that

Molecules	Peak Position ( cm <sup>-1</sup> )					
DMMP	1255					
Acrosol in open air	1325					
Aerosol in open all	1345					
	1210					
DNT adsorbed on Steel Surface	1227					
	1345					
Acetone	1240					
Aerosol in open air	1320					

Table 1: Vibrational bands of DMMP, DNT and acetone.



Molecules	нсно	CO2	$H_2O_2$	$CH_4$	<b>O</b> <sub>3</sub>	$N_2O$	SO <sub>2</sub>	HNO <sub>3</sub>	NO2
Peak Position(cm-1)	1065	1060	1330	1306	1110	1280	1360	1325	1320

Table 2: High Resolution Spectral lines of near Earth Atmospheric Molecules.



Figure 5: Resonance frequency of QCTF (1) in atmospheric pressure condition (2) low pressure condition.

at 760 Torr (atmospheric pressure). Thus reducing the pressure of the enclosure housing QCTF will help in achieving the standoff detection range up to 100 meter.

## Conclusions

QE-LPAS System is developed based on the phase sensitive data acquisition for detection of hazardous biological, chemicals and explosive materials. An engineered prototype of tripod mounted LPAS system has been developed and hazardous species were detected from standoff distances of up to 30 meter. The system is able to give alarms for hazardous biological, chemicals and explosive and pollutant gases in atmosphere (like greenhouse gases) using the developed software. In the tripod mounted system, lock-in amplifier and function generator have been replaced by their respective cards to reduce the volume, weight and size. In the system, gold coated ellipsoidal receiver mirror of aperture size 20cm has been used. The QE LPAS spectra were recorded in retro reflector based configuration for the detection of pollutant gases present in lower atmosphere. The technique has also been tested using evacuated the QCTF detector. The Q value of QCTF detector is enhanced nearly 10 times in vacuum condition. A shift of nearly 2 Hz in the resonant frequency was observed under vacuum condition. The technique can be used for detection of hazardous molecules from standoff distances of up to 100 meter in retro reflector based configuration. Standoff Quartz Enhance Quantum Cascade Laser Photoacoustic Spectroscopy is a highly selectivity, eye safe in mid IR wavelength region technique. The major advantage of this technique it will work in solar back ground. There is not interference of any ambient light/Solar light and any background electronic frequency. It is applicable in the laser spectroscopic technique. The QE-LPAS system can be used for the homeland security in present scenario.

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