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Photo-Acoustic Spectroscopy of Chemical Warfare Agents Using Optical Fibre Delivery of a Distributed Feedback Laser

Final Report

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Abstract

The detection of acetylene has been demonstrated at concentrations of 1% and 100 ppm using a photo-acoustic spectroscopy (PAS) test bed based upon a distributed feedback (DFB) laser in combination with a high power erbium-doped fibre amplifier as an excitation source. Photo-acoustic (microphone) signal strengths have been characterized as functions of power, laser centre frequency, and the applied modulation frequency and amplitude. The present PAS DFB system used to detect acetylene might serve as a test bed for the development of a compact, efficient, and lightweight unit for the detection of chemical warfare agents.

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Table of contents

1. Background 5

2. Aims, Objectives and Tasks 5

3. Experimental Apparatus 6

4. Experimental Results 8

4.1 Sinusoidal Frequency Modulation with temperature tuning of the laser centre frequency 8

4.1.1 Principles of signal generation 8

4.1.2 Variation of Signal amplitude with modulation current 9

4.1.3 Variation of first and second harmonic signal amplitudes with laser centre frequency 10

4.1.4 Variation of acoustic signal amplitude with laser modulation frequency 12

4.1.5 Variation of signal amplitude with amplifier power output and laser input 13

4.2 Ramp generated acoustic signals 13

4.2.1 Long Ramp Period 13

4.2.2 Short Ramp Period 15

4.2.3 Tests using 100ppm Acetylene 17

5. Conclusions 18

List of figures

Figure 1. Gas cell interrogation system 6

Figure 2. Gas cell 6

Figure 3. DFB Laser characteristics 7

Figure 4. Generation of an acoustic signal through laser frequency modulation 9

Figure 5. Variation of acoustic signals with laser modulation current 10

Figure 6. Temperature scanning with laser bias current of 60mA 11

Figure 7. Temperature scanning with laser bias current of 100mA 11

Figure 8. Variation of signal amplitude with modulation frequency 12

Figure 9. Variation of acoustic signal amplitude with amplifier output 13

Figure 10. Acoustic signal (upper trace) generated as a result of ramping the laser drive current (lower trace) 14

Figure 11. Signal amplitude as a function of frequency scan rate 15

Figure 12. Acoustic signal generated with a Short Ramp (Upper trace) and the modulation applied to the laser 16

Figure 13. Acoustic (Microphone) signal obtained using 100ppm Acetylene 18

List of tables

Table 1. Oscilloscope signals compared to lock-in measurements for different positions along the ramp. 17

1. Background

The combination of Distributed Feedback (DFB) lasers and high power fibre amplifiers provides flexible (both frequency and amplitude) modulation capability; narrow band output (less than the line width of individual gas vibration – rotation absorption lines); precise frequency / wavelength control through temperature tuning; excellent beam quality and high output power and efficiency in a compact, robust and low cost system. Such capabilities may bring significant benefits to the field of photo-acoustic spectroscopy, particularly in terms facilitating deployment in the field. This project was intended as a preliminary investigation of using such a system for photo-acoustic spectroscopy.

2. Aims, Objectives and Tasks

The overall aim of the proposed project was to construct a test bed to enable an experimental investigation of photo-acoustic spectroscopy (PAS) for trace gas detection using a fibre coupled DFB laser source, an erbium doped fibre amplifier, optical fibre beam delivery to the PAS cell and signal recovery via a lock-in amplifier. Acetylene was chosen as the target gas for convenience. Specific objectives included a study of signal strengths as a function of gas concentration, incident laser power (up to 1 Watt), applied modulation frequency and various signal recovery parameters. It was also intended that some preliminary evaluation of signal to noise ratios and detection sensitivity could be achieved.

Specific tasks built into the programme were as follows:

Task 1. Design, construction and testing of the PAS test bed including measurement and validation of beam properties such as wavelength and power characteristics.

Task 2. Design and construction of a gas handling system to enable the PAS cell to be filled with known concentrations of Acetylene.

Task 3. Experimental investigation of PAS signals as a function of acetylene concentration, excitation power (up to 1 Watt), signal modulation frequency and signal recovery parameters such as integration time / bandwidth, amplifier gain etc.

Task 4. Analysis of results to enable conclusions regarding detection sensitivity in terms of minimum detectable absorbance units so that the results may be used to assess the technique for application to any gas with known absorption strength.

Task 5. Preparation and submission of the Final Report.

3. Experimental Apparatus

The experimental system is shown in Figure 1. A DFB laser is amplitude / frequency modulated using a signal generator connected to the laser driver that delivers a bias current. The temperature controller is used to vary the output centre wavelength of the laser. In order to generate the required optical power, the output of the laser is connected to an erbium doped fibre amplifier (EDFA) whose output power can be controlled in the range 20-30dBm (100mW to 1W). The acoustic signal generated by the micro-phone is recovered by a lock-in amplifier.

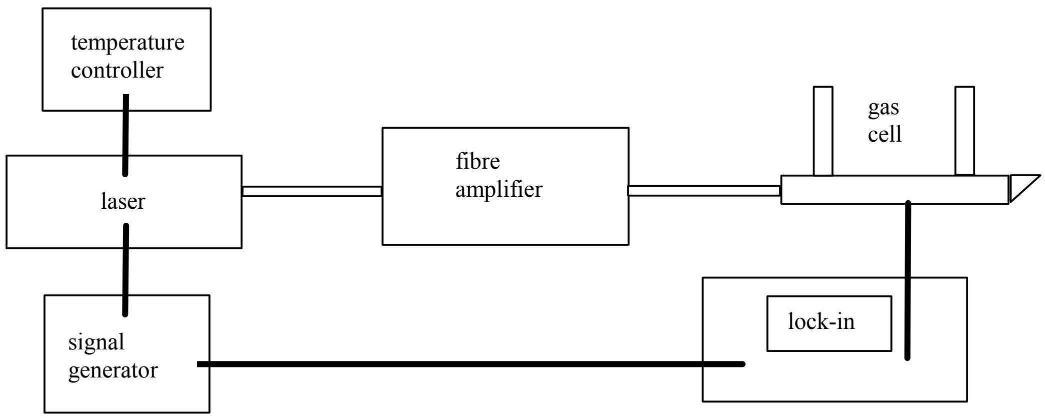


Figure 1. Gas cell interrogation system

The gas cell (figure 2) has been modified from its as-received condition. One end has been cut off and a brass ferrule inserted to hold the fibre pigtailed GRIN lens used to deliver a light beam. This ensures that a collimated narrow beam (<500µm in diameter) is consistently launched down the centre of the cell. The original microphone has also been replaced with a new EK3132 device. For safety and stability the cell is mounted in a machined grey nylon block that also acts as a beam dump for the laser.

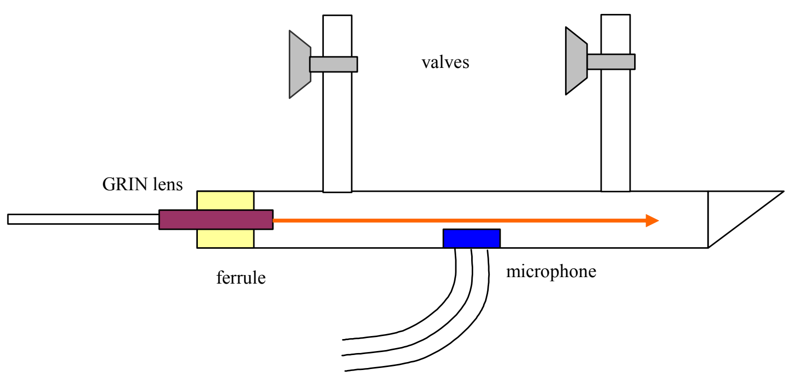
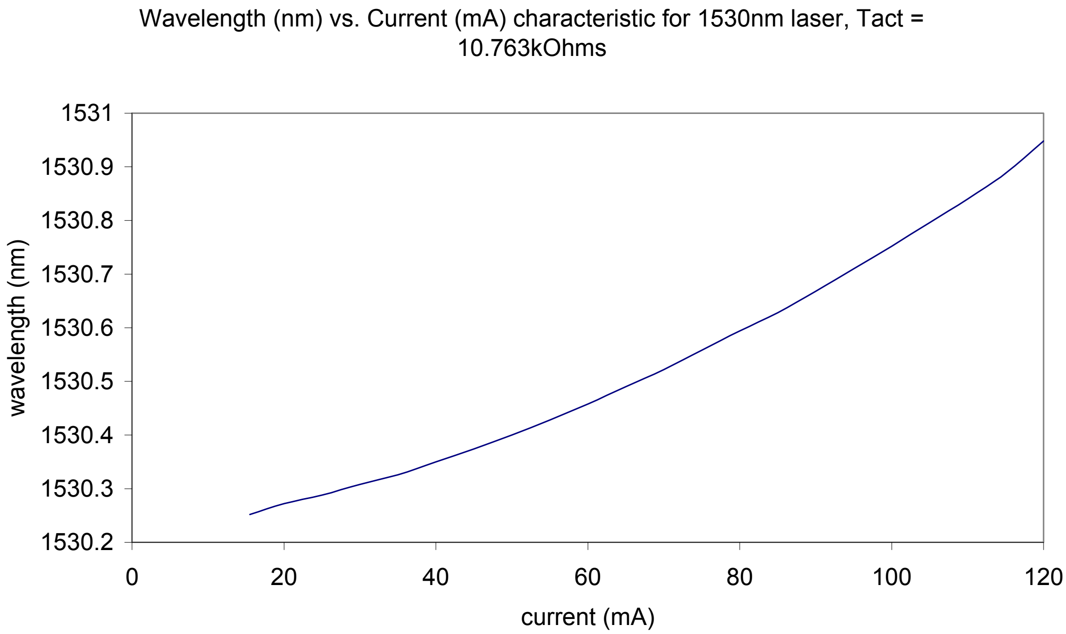


Figure 2. Gas cell

The DFB laser used during these experiments had the following output wavelength and power characteristics as a function of drive current.



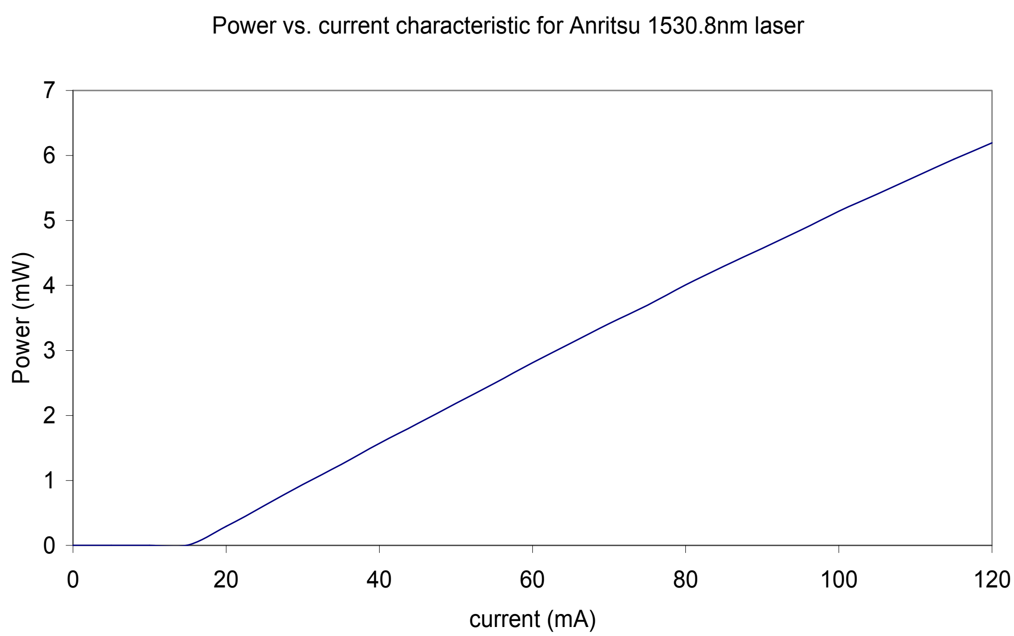


Figure 3. DFB Laser characteristics

As can be seen from the above graphs, the laser output power and wavelength will increase simultaneously as the drive current is increased. Note that the current / wavelength relationship shown above applies to a specific laser temperature (as determined by the temperature controller). Altering the laser temperature will cause this curve to shift, this being the method used to control the centre frequency. The acoustic signals generated during the following experiments are the result of the laser frequency / wavelength being swept through all or part of the gas absorption line, with a smaller contribution resulting from the changing output power.

4. Experimental Results

The first results described here were carried out using a mixture of 1% acetylene in nitrogen. The cell was thoroughly flushed through and the valves closed before measurements were taken. Two interrogation techniques were investigated. The first uses a sinusoidally modulated laser drive current. This in turn sinusoidally modulates the frequency / wavelength of the laser output around a centre frequency that is thermally tuned to a point on a single gas rotation-vibration absorption line. The second uses a ramped laser current and hence output frequency, with thermal tuning used to control the range of frequencies swept relative to the absorption line frequency (i.e. the position of the absorption peak on the ramp).

4.1 Sinusoidal Frequency Modulation with temperature tuning of the laser centre frequency

4.1.1 Principles of signal generation

In these experiments the laser output was modulated about a centre frequency whose value is determined by thermal tuning (figure 4). The amplitudes of the acoustic signal at both 1f (the fundamental modulation frequency) and 2f (the second harmonic of the modulation frequency) were then measured by the lock in amplifier as a function of the centre frequency of the laser as it was tuned through the absorption line. The DFB laser was modulated at 33Hz with a modulation current of 9mA during these two tests, with the laser bias current set to either 60mA or 80mA. Modulation of the laser drive current has two simultaneous effects; it modulates the output intensity and it modulates the laser frequency about the centre frequency set using the temperature controller. The drive currents described above resulted in depths of amplitude modulation of approximately 18% and 12% when applied to laser bias currents of 60mA and 80mA respectively. This means that the signal amplitudes obtained during these experiments were largely the result of frequency scanning, but with an additional component due to amplitude modulation that manifests itself as an asymmetry of the amplitudes of the signals obtained on either side of the absorption curve. The amplitude of the laser modulation current was chosen such that the frequency modulation would scan the full width of the acetylene absorption line (0.03nm FWHM). The optimum modulation of 9mA corresponds to a wavelength modulation of around 0.1 nm according to measurements previously carried out on this laser source. In all of these experiments the amplifier output was set to 27dBm (500mW).

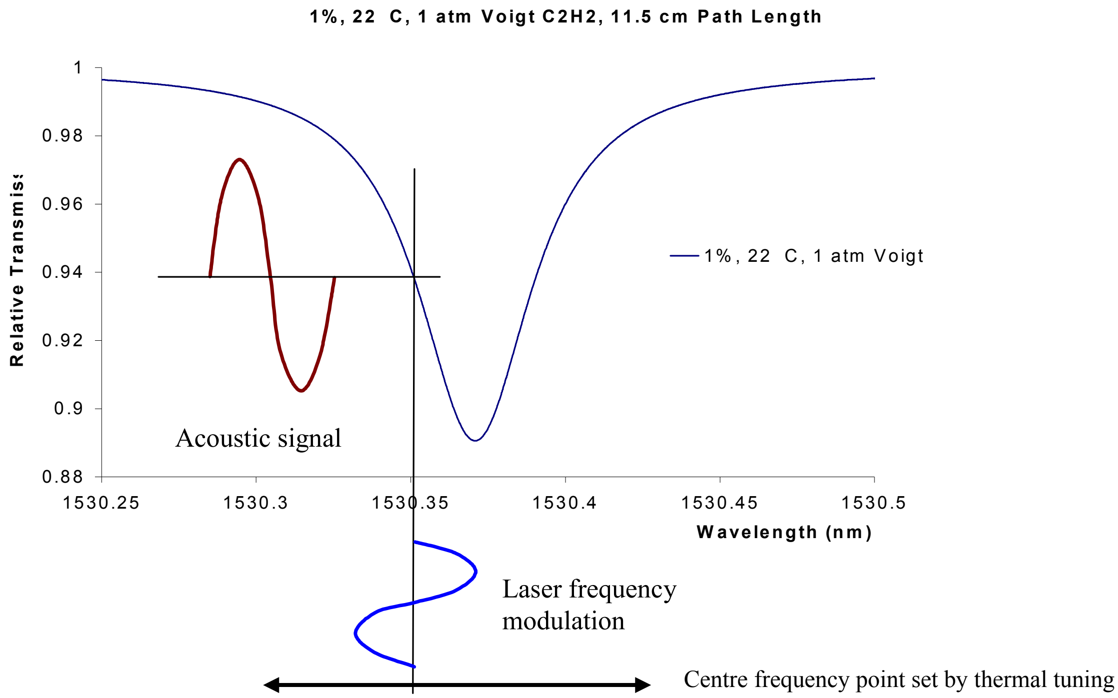


Figure 4. Generation of an acoustic signal through laser frequency modulation

4.1.2 Variation of Signal amplitude with modulation current

Firstly, we wished to determine the optimum applied modulation to achieve the maximum signal output. This was done by applying a modulation signal and then tuning the laser centre frequency to obtain the maximum acoustic signal output measured firstly at 1f. The output 1f signal amplitude was then measured as a function of the applied signal amplitude which was varied 1-20mA (Figure 5). This was repeated for the 2f output signal (Figure 5). Figure 5 clearly shows that an amplitude of 9mA results in the optimum peak output signal for both 1f and 2f detection and this was then used in further experiments of this nature.

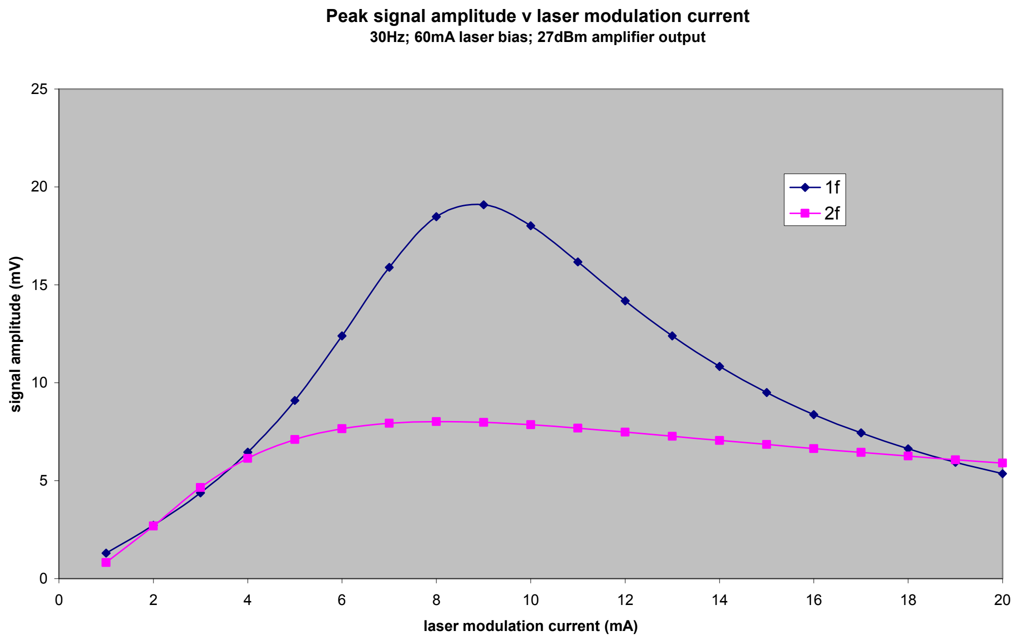


Figure 5. Variation of acoustic signals with laser modulation current

4.1.3 Variation of first and second harmonic signal amplitudes with laser centre frequency

With the applied modulation signal set to the optimum value of 9mA, the centre wavelength of the DFB was then tuned through the absorption line by altering the temperature of the laser using a controller that defines the temperature through the resistance of a thermistor. The amplitudes of both the fundamental (1f) and the second harmonic (2f) components of the output signal were then measured as a function of the centre frequency / wavelength of the laser and the results are given in figures 6 and 7. As would be expected, the second harmonic shows a maximum at the point where the modulation is symmetrical about the peak of the absorption curve and the fundamental component shows a minimum.

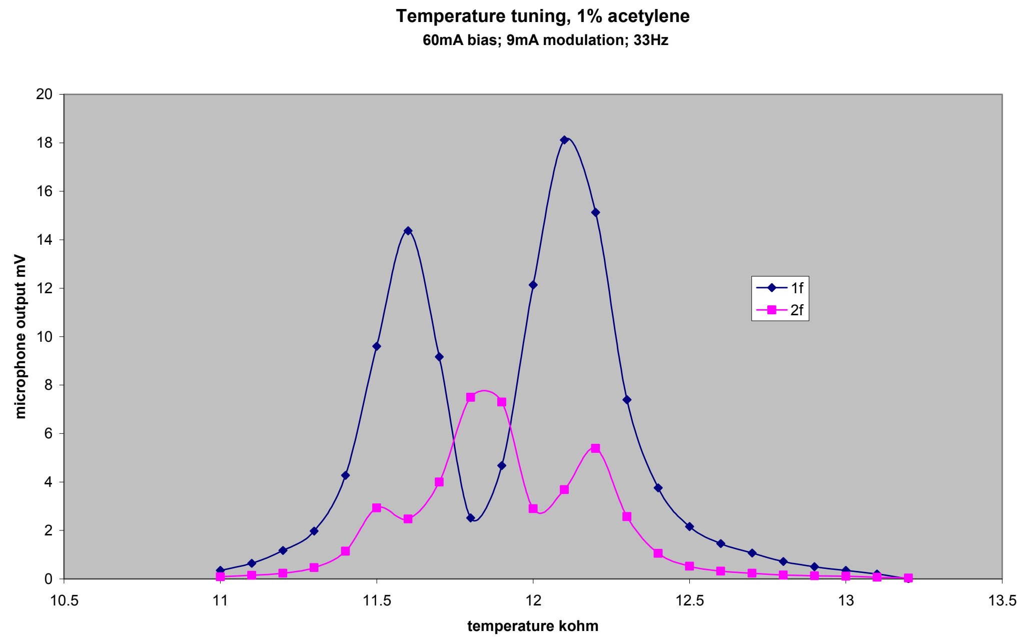


Figure 6. Temperature scanning with laser bias current of 60mA

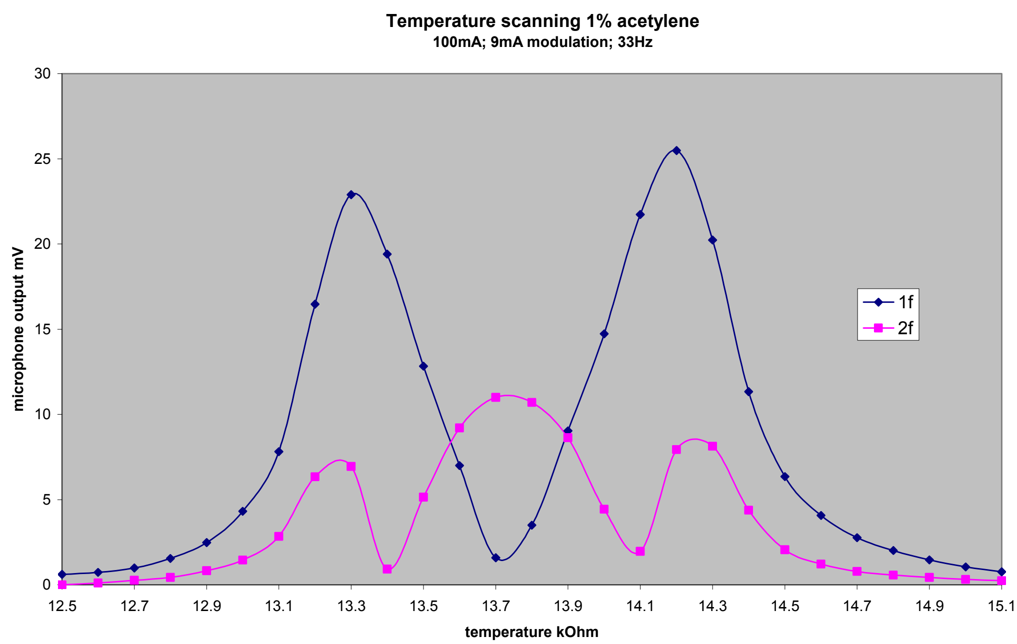


Figure 7. Temperature scanning with laser bias current of 100mA

The peak signal amplitudes of 25mV occurred for the 1f signal.

4.1.4 Variation of acoustic signal amplitude with laser modulation frequency

Figure 8 shows the effect of laser modulation frequency on the acoustic signal amplitude. The signal amplitude increases dramatically as the modulation frequency is reduced, presumably due to the increasing energy per cycle.

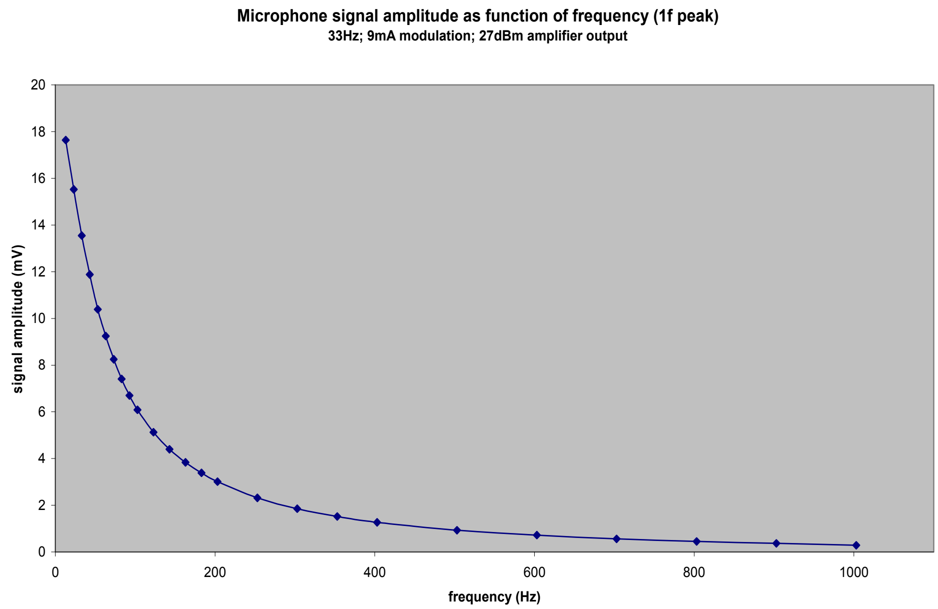


Figure 8. Variation of signal amplitude with modulation frequency

Note: It was observed that these high amplitude, low frequency, signals were only observed when the cell was sealed (ie the valves were closed). If the valves were opened the signals became unmeasurably small, though this was not due to leakage of the acetylene mixture as was shown by the recovery of the signal when the valves were closed.

4.1.5 Variation of signal amplitude with amplifier power output and laser input

Figure 9 shows the variation of the 1f signal amplitude with amplifier output power.

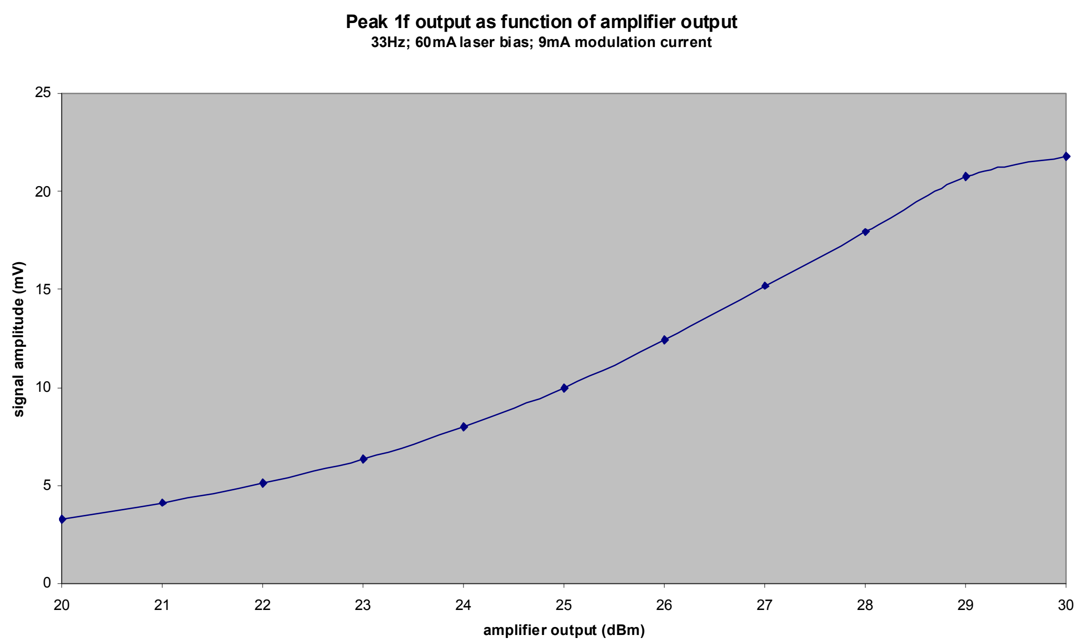


Figure 9. Variation of acoustic signal amplitude with amplifier output

4.2 Ramp generated acoustic signals

4.2.1 Long Ramp Period

An alternative method of generating an acoustic signal was investigated, which was to ramp the laser drive current and hence frequency using a sawtooth signal. The laser was biased with a dc current as in the previous experiment. Thermal tuning was required firstly to bring the absorption line within the frequency range of the ramp and secondly to tune it such that it coincided with laser output frequencies near to the maximum of the ramped current. Although a signal can be obtained at any point on the ramp slope, its amplitude will increase as the maximum current value is approached, since this will correspond to the maximum laser output power.

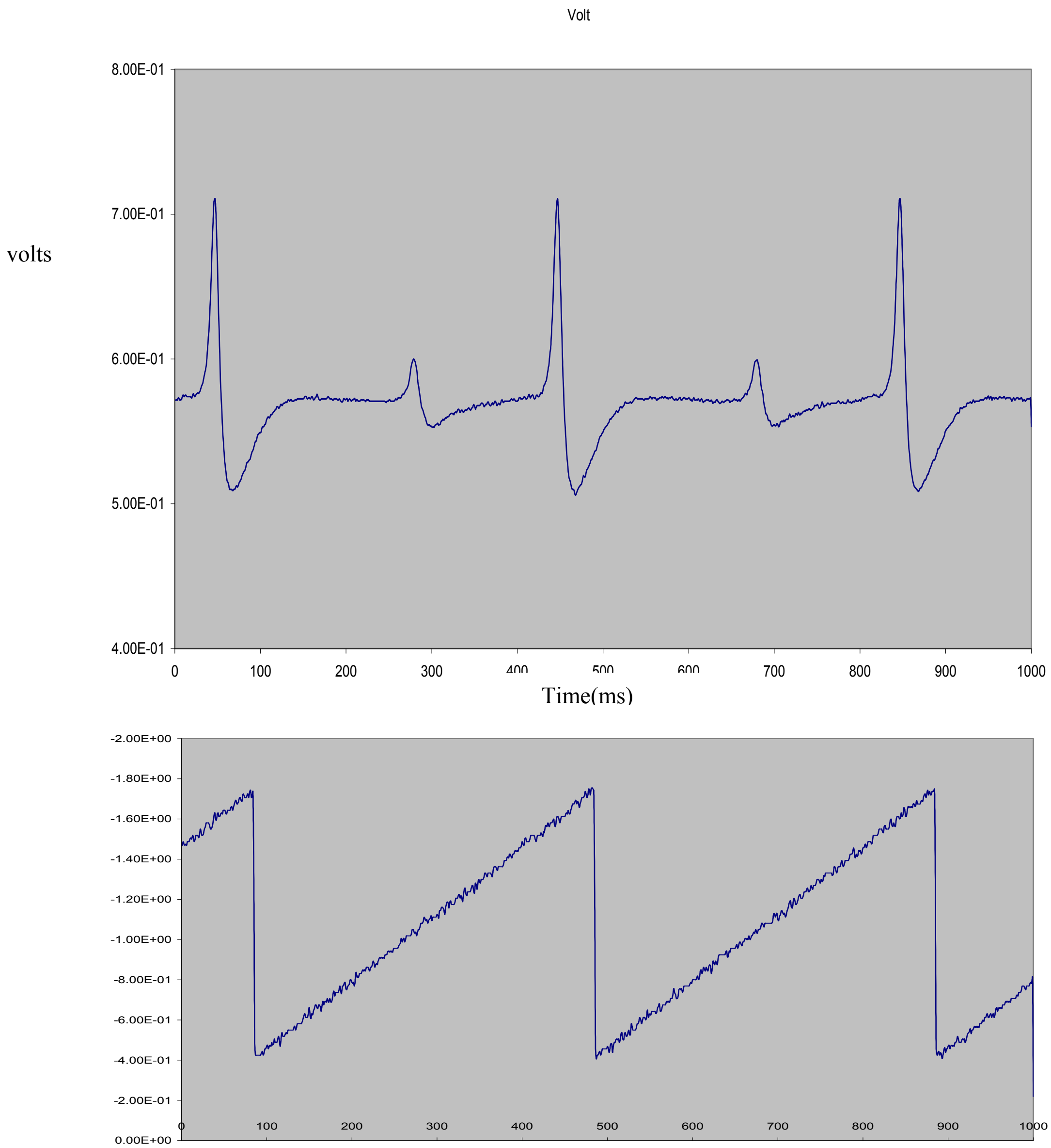


Figure 10. Acoustic signal (upper trace) generated as a result of ramping the laser drive current (lower trace)

Figure 10 shows the resulting acoustic signal (upper trace) and the corresponding ramped current applied to the laser (lower trace) on a synchronised time frame. It clearly shows the acoustic signal occurring at the top end of the current ramp coinciding with the high end of the laser output power. The signal trace presented was taken directly from an oscilloscope with no averaging and shows a peak-to-peak amplitude of 200mV. A dc offset from the microphone means that the true zero is actually at around 0.58V. The laser was biased at 40mV and ramped over 700mV (=70mA) over a period of 800ms, whilst the amplifier output was set to 27dBm. Presumably the second peak is due to a weaker absorption line.

The effect of varying the ramp period was also investigated. Again, the maximum signal amplitudes are obtained at low scan rates in agreement with results obtained in the previous section (Figure 11). Experiments are restricted by the frequency response of the microphone, which is not specified below 50Hz and probably tails away to little or no response to dc pressure.

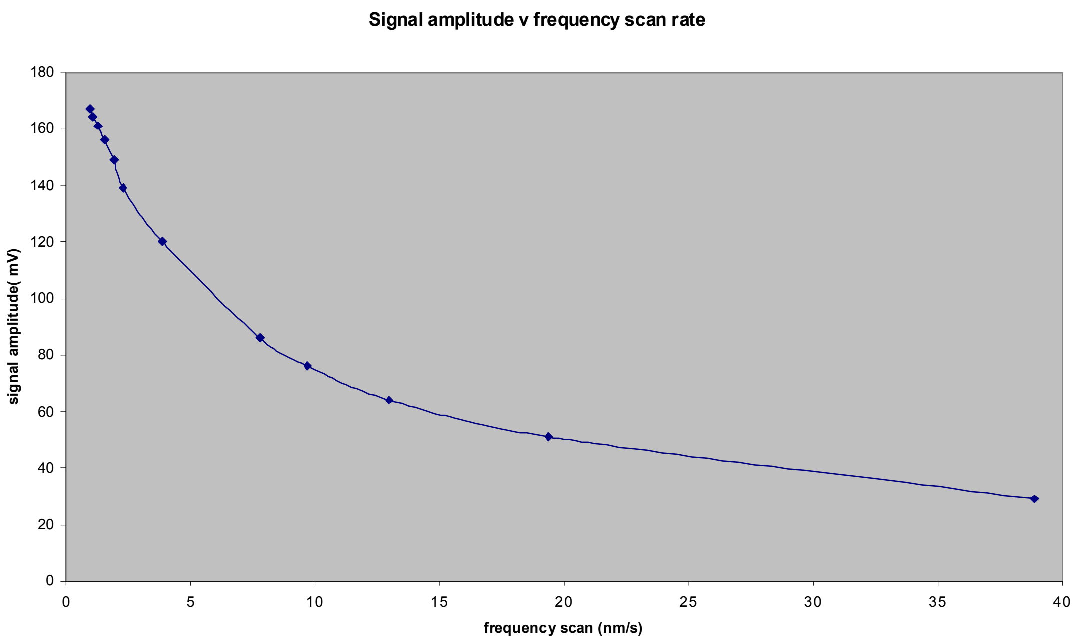


Figure 11. Signal amplitude as a function of frequency scan rate

4.2.2 Short Ramp Period

Clearly it is not necessary to use as long a scan period as that shown in figure 10. Shortening the scan period to 100ms with a 150mV (=15mA) amplitude using a bias current of 80mA results in the trace shown in figure 12. This gives a signal that can be analysed using a lock-in amplifier to measure the fundamental and second harmonic components of the modulation frequency. These are shown in table 1 as a function of the laser temperature as given by the thermistor resistance. The conditions for the experiment were: laser bias 80mA; modulation current 15mA; ramp period 100ms.

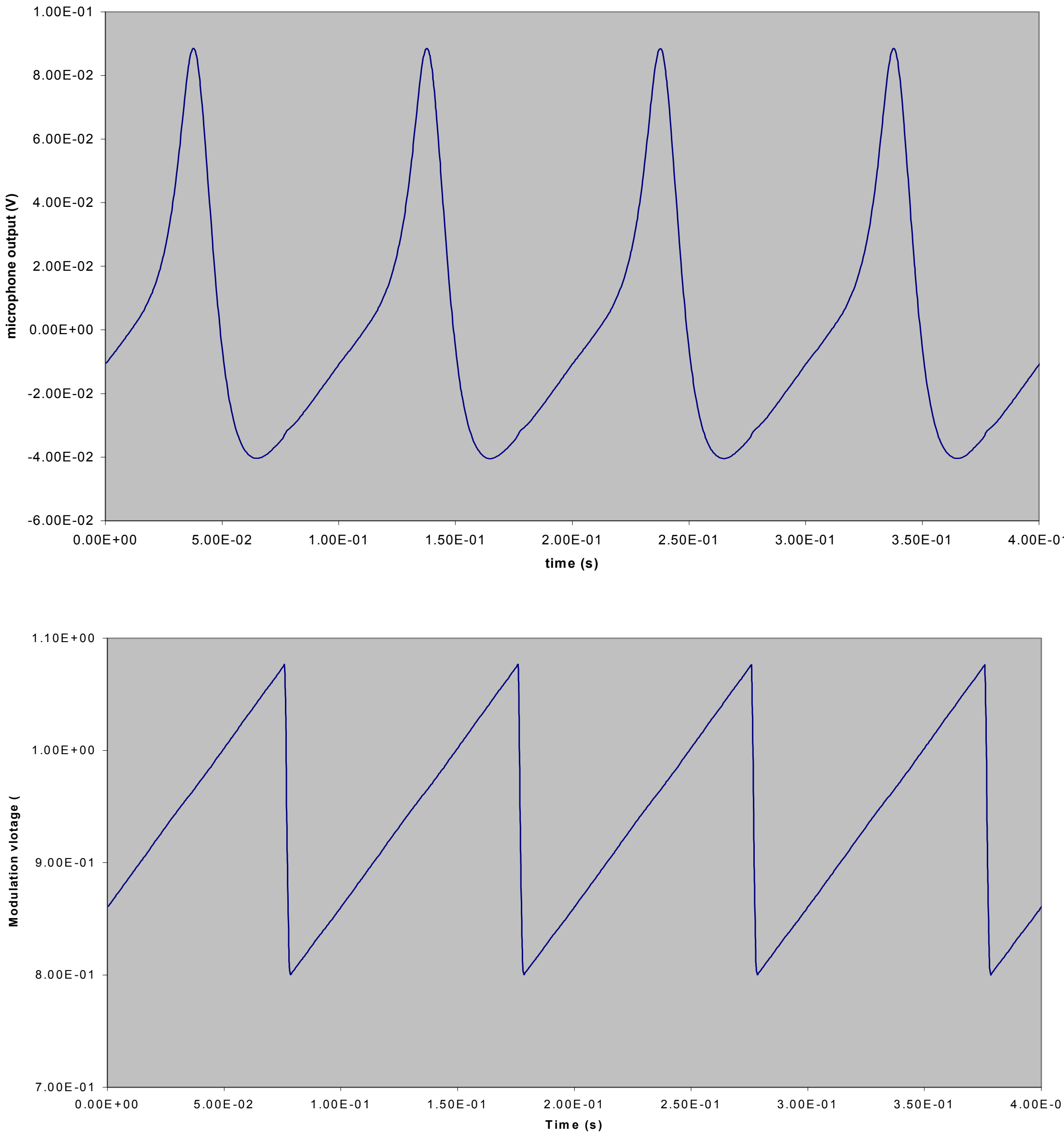


Figure 12. Acoustic signal generated with a Short Ramp (Upper trace) and the modulation applied to the laser

Laser temperature (thermistor kΩ)	Oscilloscope Signal pk-pk (mV)	Lock-in Amplifier (mV)	
		1st harmonic	2nd harmonic
7.614	132.0	32.5	17.5
7.564	130.1	32.2	17.3
7.514	128.0	32.1	17.0
7.464	127.0	32.2	16.5
7.414	125.2	32.4	15.8
7.364	123.5	32.9	15.5
7.314	122.6	33.6	16.3
7.264	123.3	33.7	18.6

Table 1. Oscilloscope signals compared to lock-in measurements for different positions along the ramp.

The above table shows that all measurements are stable with respect to position along the ramp. Slight discrepancies in terms of the behaviour of the harmonics compared with the raw signal amplitude appear to be due to the down slope of the ramp causing changes to the shape of the signal.

4.2.3 Tests using 100ppm Acetylene

Since a sample of 100ppm acetylene in nitrogen was available, a test was carried out using a sample of this mixture. The results are shown in figure 13. The laser parameters were the same as for those used to obtain figure 10 except that an average trace of 8 samples was used to reduce the noise level. The amplitude is around 2 mV which scales as expected (linearly) with respect to that obtained from 1% acetylene (200mV). Mains pickup is the most probable explanation for the presence 50Hz ripple.

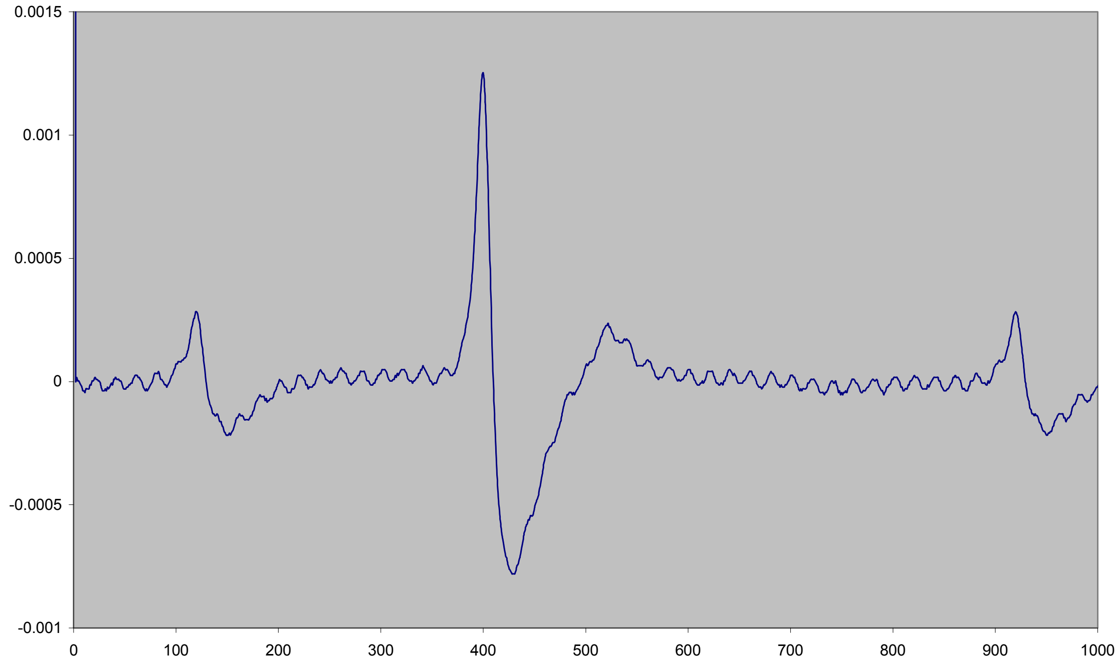


Figure 13. Acoustic (Microphone) signal obtained using 100ppm Acetylene

5. Conclusions

A PAS test bed has been established using a DFB laser in combination with a high power EDFA as the excitation source (Task 1) and a suitable gas handling system has been built to enable the PAS cells to be filled with any required gas concentration (Task 2). Acoustic (microphone) signal strengths have been measured as a function of excitation power, laser centre frequency and the applied modulation frequency and amplitude, using both sinusoidal and ramped modulation of the laser current / frequency (Task 3+). Two concentrations of acetylene were used in the study: 1% and 100ppm.

With 1% acetylene (10% absorption on peak), maximum signals from the microphone of 200mV were observed for 0.5 Watts of incident power in the linear ramp scan experiments. The estimated energy absorbed per cycle is 3.6mJ. The corresponding results for 0.01% (100ppm) acetylene (0.1% peak absorption) was 2mV for 0.5W incident power (0.036mJ absorbed energy per cycle). As expected the signals scale approximately linearly with absorbance and with incident power.

In the sine wave modulation experiments with lock-in amplifier signal recovery, maximum signals of around 25-30mV were observed for 1% acetylene (10% peak absorption) for 0.5W of incident light. The absorbed energy per cycle was a little more difficult to estimate here but is probably around 1-2mJ.

Given the above we believe that this project has demonstrated the potential of this type of system for PAS applications in compact, power efficient, light weight, robust low cost units.

With the above results, it is a trivial task to estimate the minimum detectable absorption (Task 4) if the noise characteristics of the microphone were available – to date we have been unable to measure them or obtain figures from the manufacturer in the required units of $mV/Hz^{1/2}$. Hence, we leave this simple calculation to the sponsoring scientist or to future investigations.

One of the limitations of traditional PAS techniques, that use amplitude modulation, is the residual signal arising from absorption at the cell windows. In the above approach, the signals are generated largely by frequency sweeping across an absorption line thus diminishing the problem somewhat. Residual signals from the windows still exist, due to the concomitant amplitude modulation on the laser, but to a much lesser extent.

A further benefit of the system concept and configuration reported here arises from the beam quality of the output giving diffraction limited, narrow and collimated beams over a long path length. This means that significant signal enhancements over what is seen above may be obtained by designing cells of substantially smaller volume to just match the beam dimensions used.

Future work should address the following:

- a full study of signal sensitivity as regards system noise and decreasing gas concentration.
- minimisation of amplitude modulation in the excitation beam to eliminate residual signals from spurious absorption in the beam path.
- a full investigation of alternative cell designs and beam propagation optics to minimise the cell volume to that allowed by the smallest beam dimensions, thus optimising the signal
- a study of Raman amplifiers to enable extension of the wavelength range of operation and hence the species that can be addressed.

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The detection of acetylene has been demonstrated at concentrations of 1% and 100 ppm using a photo-acoustic spectroscopy (PAS) test bed based upon a distributed feedback (DFB) laser in combination with a high power erbium-doped fibre amplifier as an excitation source. Photo-acoustic (microphone) signal strengths have been characterized as functions of power, laser centre frequency, and the applied modulation frequency and amplitude. The present PAS DFB system used to detect acetylene might serve as a test bed for the development of a compact, efficient, and lightweight unit for the detection of chemical warfare agents.

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