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Photoacoustic spectroscopy for remote detection of liquid contamination

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ABSTRACT

The remote detection and identification of liquid chemical contamination is a difficult problem for which no satisfactory solution has yet been found. We have investigated a new technique, pulsed indirect photoacoustic spectroscopy (PIPAS), and made an assessment of its potential for operation at stand-off ranges of order 10m. The method involves optical excitation of the liquid surface with a pulsed laser operating in the 9-11μm region. Pulse lengths are of order 3μs, with energy ~300μJ and repetition rates ~200Hz. Rapid heating of the liquid by the laser pulse produces acoustic emission at the surface, and this is detected by a sensitive directional microphone to increase the signal-to-noise ratio and reduce background clutter. The acoustic pulse strength is related to the liquid's absorption coefficient at the laser wavelength; tuning allows spectroscopic investigation and a means of chemical identification. Maximum coverage rates have been examined, and further experiments have examined the specificity of the technique, allowing a preliminary assessment of false-alarm and missed-signal rates. The practical aspects of applying the technique in a field environment have been assessed.

Keywords: remote-sensing, stand-off, photoacoustic, spectroscopy, chemical, liquid

1. INTRODUCTION

Photoacoustic spectroscopy is a sensitive technique widely used for the detection of trace contamination and the measurement of low absorption coefficients in both liquids and gases [1,2]. The sample is conventionally confined within a closed cell; by contrast we have developed the concept of PIPAS (pulsed indirect photoacoustic spectroscopy) for the examination of free surfaces of liquids, and applied this to the problem of stand-off liquid chemical detection and identification. The technique is based upon the fact that when a pulse of laser energy is incident upon a liquid it heats a thin surface layer, sometimes to a considerably elevated temperature. Some portion of this heat is conducted to the air adjacent to the liquid causing it to expand and create an acoustic wave. The magnitude of the acoustic wave is proportional to the amount of heat transferred to the air, which is closely proportional to the absorption coefficient of the liquid at that wavelength.

Interrogation of the material at several wavelengths builds up a photoacoustic spectrum, which corresponds closely to the optical absorption spectrum allowing in principle the identification of the substance. Overall, we have obtained an excellent understanding of the basic physical processes through a series of experiments on CO_2 laser excitation of simulant liquids, and these are reported fully in references [3] and [4]. In this paper we report further recent developments of the technique, and assess some of the practical difficulties involved when the technique is applied in real-life situations.

2. THE PIPAS TECHNIQUE

2.1 Physical process

This section briefly summarises earlier work [3] in which we reported theoretical and experimental (short-range) investigations of the technique in the laboratory. The tunable Q-switched CO_2 laser source emitted pulses of up to 100μJ energy at a pulse repetition frequency of ~1kHz. The pulse duration was of order 3μs, and it was tuned by rotating an intracavity diffraction grating permitting pulsed output on over 20 lines of the CO_2 spectrum in the 9-11μm region. The laser pulses illuminated samples of simulant liquid (e.g. ethanol, DMMP, diffusion pump oil) that were

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either smeared on a glass substrate or placed in a small container; an electret microphone positioned close to the sample detected the acoustic emissions. Typical PIPAS signals from diffusion pump oil are shown in Fig. 1.

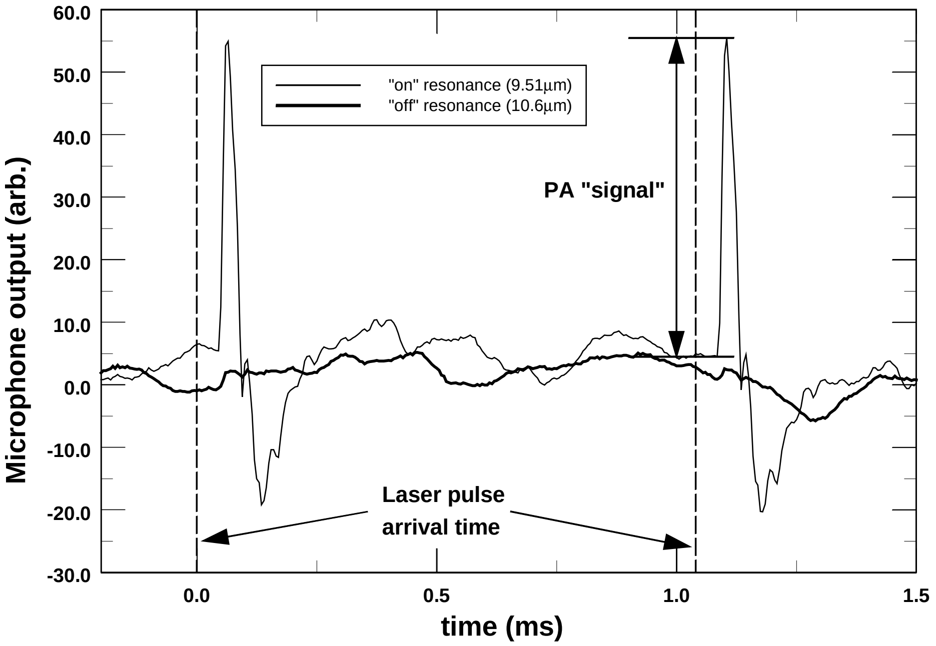

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Figure 1. Measured photoacoustic pulse profiles. The two pulses shown here are separated by 1.04ms, corresponding to a pulse repetition frequency of 961Hz, and are the result of averaging 256 measurements. The PIPAS pulse is detected 6.6μs after the laser pulse, corresponding to the acoustic time-of-flight over the 2.2cm range. We define the PIPAS signal as the height of the initial acoustic peak, shown here for the second on-resonance pulse. Note that there is still an observable signal for the off-resonance illumination.

Investigations of the PIPAS signal properties revealed that the amplitude of the acoustic pulse is proportional to laser pulse energy, implying a more effective conversion of optical to acoustic energy for larger pulses. The amplitude also reduces inversely with the distance between target and microphone, indicating a good approximation to emission of a spherical wave. Experiments in which the sample was translated through a focus of the laser beam showed the acoustic pulse amplitude to be independent of illuminated area. This behaviour was shown to break down only when the focusing was sufficiently tight to induce localised boiling of the liquid surface, leading to a substantial increase in acoustic emission. Experiments reported here have all been carried out at pulse energy densities substantially below the threshold for the onset of this non-linear behaviour. Theoretical analysis predicted that the amplitude of the PIPAS response should be roughly proportional to the absorption coefficient of the material at the illumination wavelength. Fig. 2 shows good agreement between the PIPAS spectrum and the measured absorption spectrum of the test material (diffusion pump oil), suggesting that remote chemical identication is feasible, at least in principle, via measurement of photoacoustic spectra.

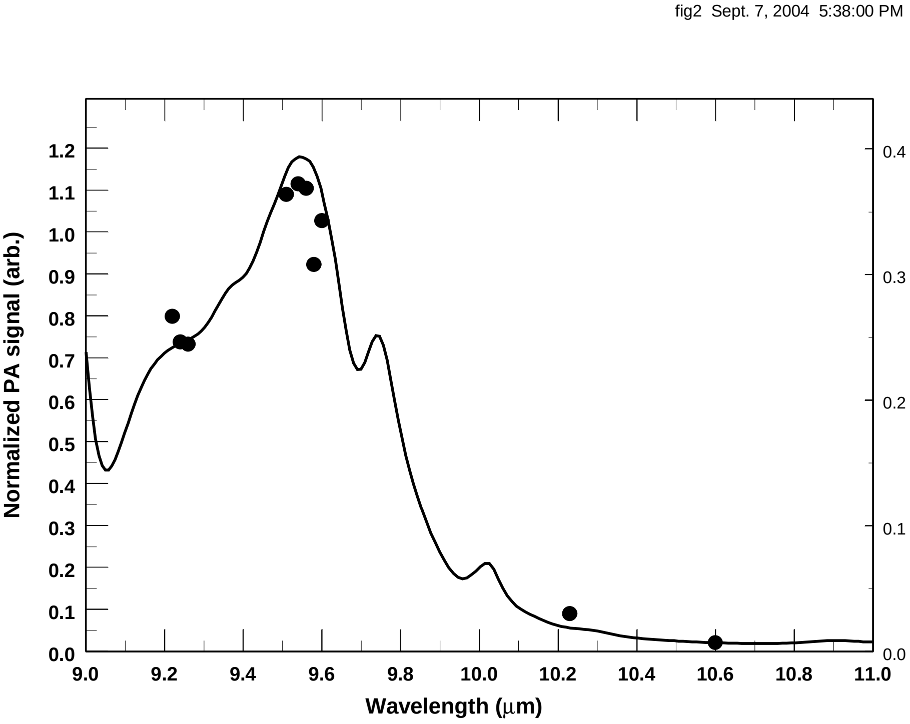

Figure 2. PIPAS spectrum (discrete circles) for type 704 pump oil obtained with the emission lines of the CO_2 laser source. The continuous curve is an absorption spectrum measured with a conventional absorption spectrometer.

2.2 Acoustic detection

Stand-off chemical identification requires acoustic detection of sound pulses emitted from a single point about 10m away, with high sensitivity and good directionality for elimination of background clutter. Different detection methods were compared, and it was established that a reflector microphone (using a combination of a parabolic reflector dish and a cardioid microphone) is the best practical way of achieving the necessary directionality and sensitivity [4].

Environmental effects were examined via ray-tracing simulations and by a series of outdoor trials that investigated the propagation and detection of pre-recorded PIPAS acoustic pulses. Temperature gradient effects were shown to be negligible at the proposed operating range of 10-20m. Windy conditions can lead to some fluctuation in acoustic signal strength, but a partially-defocused detection scheme was shown to mitigate this effect.

Background clutter levels were investigated for a variety of locations. These recordings were analysed in order to gain greater understanding of the nature of environmental noise; typically this follows a crude 1/f frequency dependence, with frequencies beyond ~10 kHz being rapidly attenuated by the atmosphere. It was found that excellent clutter rejection can be achieved by applying a 5-15kHz bandpass to the photoacoustic pulses [4]. This eliminates the majority of background noise while retaining a significant fraction of the PIPAS signal. However, the close proximity of high-intensity high-frequency noise sources (such as power tools) will cause significant difficulties.

2.3 Enhancement of PIPAS sensitivity for stand-off operation

Effort has been directed towards improving the sensitivity to allow demonstration of the technique at the maximum range attainable in our laboratory of 8m. Three principal methods were used to enhance the sensitivity of the PIPAS technique, compared with that achieved in the initial studies [3] carried out at a few cm range. These are: 1) Increased CO_2 laser pulse energy (300 μJ per pulse) leading to increased strength of the generated photoacoustic signal; 2)

Increased microphone sensitivity, and improved directionality by the use of a 60 cm diameter parabolic dish; 3) Signal processing that allows improved discrimination of the signal from noise levels through prior knowledge of the pulse shape and pulse repetition frequency. Each of the above three enhancements was implemented in the laboratory system; the results are fully documented in [4].

Experiments were carried out on standard simulants such as methyl salicylate, DMMP and diffusion pump oil, which have similar volatility, thermal properties and absorption coefficient to the liquids of interest. The work demonstrated that detection of the PIPAS signal produced from low-volatility liquids in the laboratory at a range of ~10m is straightforward. Further increases in laser pulse energy will result in operation at longer ranges, or reduced averaging times. A pulse energy in excess of 1mJ should create PIPAS pulses at a stand-off range of 10m that have sufficient SNR to require no further averaging.

2.4 Laser source requirements

Currently, the carbon dioxide (CO_2) laser, whose output is line tunable between 9 µm and 11 µm, offers the only viable and mature laser technology operating in the 8 to 14 µm far infrared (FIR) atmospheric transmission window. CO_2 lasers are capable of providing pulsed energy at the single milli-Joule level at up to the 1 kHz pulse repetition frequency (PRF) believed to be required for stand-off detection of the majority of liquids of interest.

Previous work has investigated the use of a carbonyl sulphide (OCS) gas laser, a FIR optical parametric oscillator (OPO), and difference frequency generation to produce a tunable 8 - 12 µm source. The conversion efficiency of all three techniques was low (~1%), but the power produced was adequate for the requirements of the PIPAS system (~1mJ). Current research has concentrated on a solid-state route to the FIR. Recent developments in crystal growth techniques have significantly improved the optical quality of the non-linear materials $AgGaSe_2$ and CdSe, which allows a two-stage OPO route to an 8 - 12 µm source [5]. A compact device (25 kg) that would satisfy the requirements of the PIPAS technique has been demonstrated in the open literature operating at 300 Hz with 300 µJ pulse energies [6].

3. COVERAGE RATES

3.1 Achievable interrogation rate

We have assumed that the contaminated surface to be investigated will consist of hazardous droplets of diameter 1-3mm distributed over the ground consisting of (e.g.) tarmac, grass or concrete. The surface may also be contaminated with non-hazardous spillage such as oil or diesel. In practice, the contaminant droplets will reside on a surface whose spectral properties are unknown. It is essential that the system pixel size does not exceed the droplet size, so that the contaminant spectrum is not corrupted by the background spectrum. With such a small pixel area of a few square millimetres, about 10^5 pixels/m^2 would need to be accessed for complete interrogation of the surface. Hence it is impractical to achieve 100% interrogation of a realistic surface area with PIPAS. This requirement will inevitably limit surface interrogation rates and it is of interest to consider whether acceptable performance can be achieved by sampling the surface less densely.

3.2 Improved coverage by undersampling

Since the contamination can realistically be assumed to be spread randomly over the surface, it is apparent that an undersampling pattern which is itself anywhere between the extremes of totally regular and completely random will yield the same result. An otherwise ideal detection system is assumed having a zero probability of false alarm (FAP) and of missed signal (MSP) when directed to interrogate every pixel in the surface. Clearly when the surface is undersampled the FAP will remain at zero while the MSP will rise. This critical parameter of MSP will now be evaluated, under the assumption that a single laser pulse is sufficient to provide a PIPAS signal of the necessary signal-to-noise ratio.

We define the surface coverage as c ($0<c<1$). For an ideal detection system, the probability of no contaminant being detected in any given pixel is therefore (1-c). The probabilities of numbers of detections as a function of the total number of interrogations n of different parts of the surface is given by the binominal distribution; in particular the probability of no detections is $(1-c)^n$. In the situation of small c and large n, this is excellently approximated by MSP = exp(-nc). Thus the MSP is a function of the product nc alone; for a value of MSP of 10^{-4}, nc = 9.2; for 10^{-3}, nc = 6.9; for 10^{-2}, nc = 4.6; and for 10^{-1}, nc = 2.3. This functional form is such that the actual value of MSP chosen has little impact on the corresponding nc product; three orders of magnitude change in MSP alters the value of nc by only four times. Hence the conclusions are rather insensitive to the actual value of MSP chosen. However, for any particular value, the number of pixels which must be interrogated is inversely proportional to the surface coverage c, or more accurately to the coverage at which the MSP is defined. So for a MSP of 5 x 10^{-5}, and spherical liquid drops of radius 2mm, with a mass loading of 0.1 gm/m^2, c is found to be about 10^{-4} and n = 10^5 interrogations must be carried out. Increasing the acceptable mass loading to 1 gm/m^2 decreases n to 10^4, and at 10 gm/m^2 only 1000 interrogations are needed. Clearly it is important to put much more thought into setting values of mass loading than MSP.

In the above illustrations, a rough conversion has been used between mass loading expressed in gm/m^2 and coverage expressed in likelihood of pixel contamination. This conversion factor is dependent on droplet density ρ, radius r (assumed here to be monodisperse for simplicity), droplet rheology and interrogation geometry. An approximation for this conversion factor is obtained by assuming droplets which are spherical caps of contact angle θ ($\theta \rightarrow 0$ associated with good surface wetting) observed at angle α to the plane of the surface, where $\alpha << \theta$. Inserting a typical liquid density of 1 gm/cm^3, and droplet radius of 2mm, we find c = 1.6 x 10^{-3} $f(\alpha,\theta)$ for an assumed mass loading of 10 gm/m^2. Contact angles are not well known, but for large values of θ, $f(\alpha,\theta)$ assumes a value of about 2.5, giving c = 4×10^{-3}. This result suggests that the number of interrogations n ~ 1000 may be enough to achieve an acceptably low MSP. If the surface is well wetted then c could be an order of magnitude larger, with n reduced by the same factor.

From the value of n calculated by the method described above, it is straightforward to evaluate the required interrogation prf as Knv/R for a given detection range R and vehicle speed v in order that a roving vehicle can avoid contamination. It is assumed K different wavelengths are required to achieve successful identification of the chemical species, and signal processing is instantaneous. Setting K equal to the minimum of 2, v equal to 1 ms^{-1}, R equal to 10m, and n equal to 10^3, we find a prf requirement of 200 Hz; with K = 4 wavelengths this increases to 400 Hz. These are realistic targets for a photoacoustic system. With the assumption of uniform random surface coverage, this could be achieved with any swath width exceeding 2rKn/R drop diameters; when 2rKn/R <1, scanning may be unnecessary except to allow the revisiting of pixels at different wavelengths from the moving platform. Note, however, that in situations where the assumption of random coverage does not apply, the MSP can increase significantly above that calculated here.

4. REAL-TIME CHEMICAL IDENTIFICATION

A LabView programme was developed, involving calibration of grating position against laser wavelength, to allow automated scanning and data saving. An automated scan consisted of progressively varying the laser wavelength and acquiring data from the output of the microphone system. At each point in the scan the peak-to-peak height of the PIPAS signal was measured from within a narrow time window. After completion of the scan, a set of these values was viewed and stored. Additionally, the laser pulse height was recorded to enable the PIPAS signal to be scaled appropriately. A slow servo system was implemented where an analog output card in the computer was used to supply a voltage to a piezo-controlled output coupler in the laser. The laser output was monitored with the data acquisition system and the piezo voltage varied until a local maximum was found. Scan acquisition then proceeded as normal and this process was then repeated for each new wavelength. Although a slow solution to the cavity tuning problem, this approach does demonstrate the principles, and it ensures that all the possible wavelengths can be obtained. A deployable system would employ a much more rapidly tunable laser system. There are well-established methods to achieve the necessary frequency agility.

The signal processing contained several phases. Filtering was applied with a high cut off frequency of 50kHz. Additional improvements were achieved using the techniques discussed in previous reports and publications [3,4]. The final aspect of signal processing comes from selecting a time window where the signal can be expected to be found. In the current configuration this window was selected manually around an observable signal and was typically a few hundred µs wide. In a fieldable system this timing information would be obtained from a range finder, which would also dispel issues of range ambiguity caused by the time taken for the acoustic signal to propagate from the target to the microphone. A typical reference PIPAS spectral scan is shown in Figure 3. This shows the PIPAS signal (peak-to-peak voltage) for a sample of DMMP, along with the laser pulse height and the PIPAS signal normalised to laser pulse energy. This spectrum was taken with the microphone mounted a distance of about 4cm away from the point where the laser strikes the sample. The scan proceeded in grating steps of 10µm which is sufficient to ensure 2-3 points within each lasing line. Each point arises from the average of 10 data acquisitions, no signal processing was used and the scan took 300s. DMMP contains a fairly strong absorption feature around 9.5µm; this is immediately apparent from the normalised PIPAS spectrum.

The absorption spectrum of a material is in principle a unique identifier of that material. However the spectrum must have sufficient spectral range and resolution to be useful. The use of our current tunable CO_2 laser allows 4 wavelength regions to be assessed for their spectral absorption. This approach was taken for several sample materials and the normalised PIPAS values in the 4 bands were stored. A correlation coefficient was then calculated for new data using the stored data. The Pearson correlation coefficient is given by:

$$r = \frac{\sum xy}{\sqrt{\sum x^2 \sum y^2}}$$

where x and y are the PIPAS signals for the two data sets (reference and test), and the summation is carried out over the different wavelengths. Comparing values of the identification coefficient $1/(1-r^2)$ for each material and plotting on a bar graph allowed the best match to be obtained.

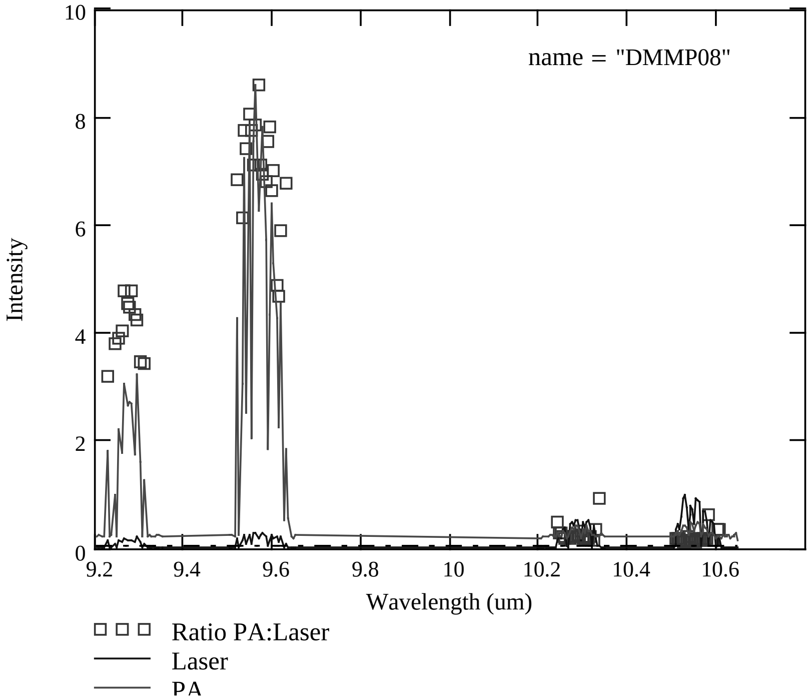

Figure 3. Results from a scan over CO_2 laser wavelength showing the PIPAS signal, the laser pulse height and the scaled PIPAS:laser ratio.

As an example of the correct identification of a specific chemical, repeated scans were taken for a DMMP sample and the correlation coefficient calculated for each of the reference materials. The results plotted in Figure 4 show clearly that DMMP was always correctly identified out of the group of materials used. For materials such as sand with intermediate values of absorption, identification can be more difficult although still achievable. This highlights the fact that reliable identification should be based on as many spectral points as it is possible to obtain. Other techniques such as template matching could be employed to identify mixtures of materials. The need to sample at many wavelengths is one drawback of the use of PIPAS for materials identification. However there is scope for minimising the number of wavelengths needed by choosing the wavelengths judiciously so as to enhance absorption features unique to the materials/liquids of interest.

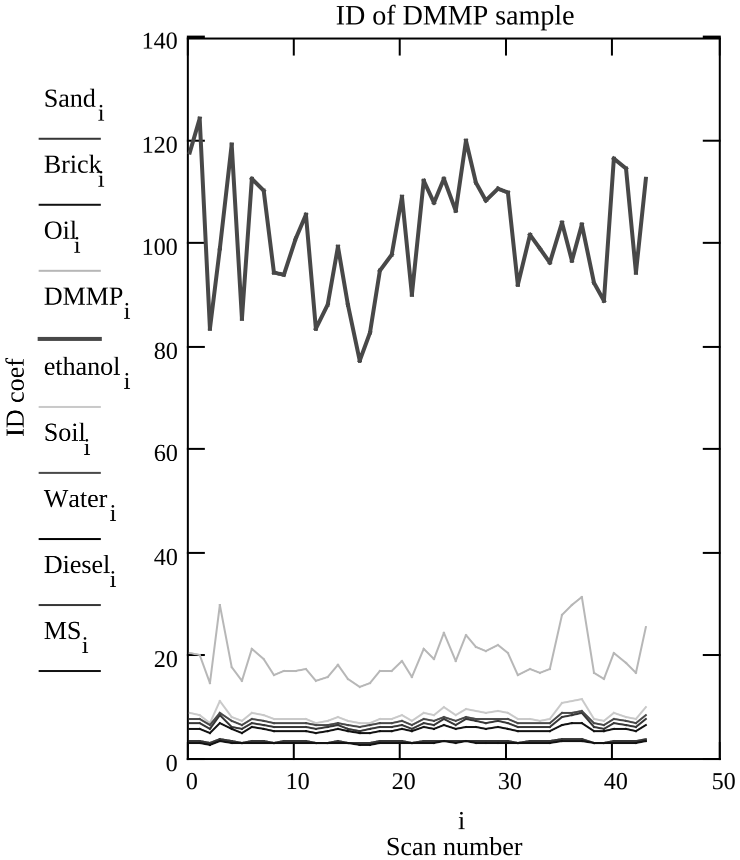

Figure 4. Identification results for a series of 44 scans using a DMMP target. The ID coefficient clearly allows a successful and unambiguous identification to be made in every scan.

In summary: an automated system for generating, collecting and analysing photoacoustic signals has been constructed. This system should be considered a prototype system that has proved the principle of using PIPAS to identify non-volatile chemicals at approximately 10m range. The current system functions well in the laboratory and significant improvements could be made to speed up the processing rate. In particular the major bottleneck is the tuning speed of the laser. The slow method of grating tuning and the GPIB interface to the controller account for the majority of the time taken.

5. PLATFORM MOTION COMPENSATION

Chemical remote sensing techniques must rely on spectroscopic methods, in which more than one interrogating wavelength is required to sample the target spectrum. For differential reflection or absorption measurements these various wavelengths may in principle be emitted simultaneously, since photons of different wavelength can be separated at the receiver by, for instance, dichroic beamsplitters. Photoacoustic and photothermal methods do not receive photons emitted by the source, but monitor effects of target heating; in the first case by emitted sound, and in the second by emitted thermal broadband radiation. Consequently the interrogating wavelengths must be emitted serially, with sufficient spacing to ensure that the slowly changing acoustic or thermal signals from adjacent wavelengths are well distinguished. There is a serious implication for sensing from a moving platform, since the same spot must be targetted at a succession of times. For military sensing from a reconnaissance vehicle there could be substantial pitch, roll, and heave components to its motion and these need to be accurately measured and corrected by a fast-acting mirror assembly. Fortunately such mirror technology is well developed; it is the purpose of this section to examine the feasibility of determining the vehicle motion with sufficient precision to allow photoacoustic methods to be used.

It has been shown (section 3.2) that for a contaminated surface the best that can be done is to randomly pick a target area no larger than a typical drop size (of a few millimeters), determine its composition, and move on to the next target area. Thus the precision with which a region must be revisited must not exceed about 1mm. This must be done at as great a range ahead of the recce vehicle as possible; a range of 10m is assumed useful and has been demonstrated in the laboratory. Thus the angular pointing precision needs to be 100μr or better. Vehicle motion even over smooth tarmacadam or concrete will result in angular excursions significantly greater than this. In addition translational motions larger than 1mm, especially normal to the direction of travel (e.g 'heave'), will also result in missing the target on revisit and must be compensated for. Both rotational and translational compensation can be achieved by pointing corrections applied by the mirror. The question is whether it is possible to provide inputs to the mirror pointing system with sufficient accuracy to achieve this goal. A critical parameter is the time T_r between revisits to the target. Pointing errors will increase with time due to instrumental noise, and so the smaller the value of T_r the easier the task becomes. It is also desirable to minimise T_r in order to sample the ground at a useful rate. However the minimum period is limited by technological constraints. These include the available prf of the laser source, the number of wavelengths needed for identification, and the finite duration of the photoacoustic signal. The analysis of section 3.2 indicates that in order to achieve a useful coverage rate the total time will be of order 10ms. Here we assume a worst-case value for T_r of 1 second in order to assess the overall feasibility.

Because the angular position at sensing initialisation is random, angular motion only has to be measured over the period of about 1s. Commercially-available inertial measurement units (IMUs) can achieve the desired angular precision with ease [7]. More problematic is the determination of heave and allied motion. IMUs contain precision accelerometers which can give the position change via double integration of their outputs. There are two constants of integration associated with this process, corresponding to the initial velocity and position of the system. The position constant is immaterial since only positional *changes* are required. However the initial velocity must be known, with a precision of better than the 1mm position accuracy over the 1 second observation time, i.e. 1mm/s. The only way this can be dealt with inertially is to set the initial velocity at zero when the vehicle is stationary and calculate position over the entire transit time of the vehicle rather than during a sensing period of a second. Since the duration period is likely to be measured in hours, the current state of the art in IMU technology does not allow the velocity to be measured to anything close to the required precision [7].

Hence other methods must be sought to provide the velocity at initiation of the measurement cycle. GPS is an obvious contender but typically delivers an inadequate velocity accuracy of only about 30mm/s [8]. Alternatively, laser Doppler velocimetry (LDV) provides sightline velocity. When used in conjunction with the earth-referenced pointing directions provided by the IMU, a three-axis LDV will provide the necessary velocity information. The sightline Doppler shift is given by $2v_{los}/\lambda$ where λ is the operating wavelength; the precision with which this can be measured is set by the speckle bandwidth, itself about equal to v_f/d, where v_f is the forward velocity of the vehicle and d is the spot size on the ground. Hence the error in the sightline velocity amounts to about $\lambda v_f/2d$. The corresponding positional error over the observation time is thus roughly $\lambda v_f T/2d$ for a signal-to-noise ratio near unity. Under these conditions, and using a wavelength of 600nm for the LDV, with an interrogating spot size of 1mm on the ground and an observation time of 1s, the positional accuracy associated with the velocity error is 1mm for a forward velocity of 3m/s. In practice it is straightforward to achieve a high signal-to-noise ratio, leading to substantially improved velocity accuracy.

The range to the target must also be known in order that transverse positional movement can be corrected by mirror rotation. This can be measured with a laser rangefinder, using similar technology to the LDV. The remaining error in position is equal to the product of the transverse distance moved and the fractional error in range. Taking a representative transverse heave of 10cm, the final precision of 1mm is met for an accuracy of 10cm in a range of 10m. This performance is straightforwardly attained by pulse timing or stereoscopic methods. In conclusion, this study verifies the feasibility of illuminating a target droplet with successive pulses over a period of a second at a range of ten metres from a ground platform in motion. The required one millimetre revisit precision can be attained using inertial measurement units combined with laser Doppler velocimetry and rangefinding functions to control a fast acting pointing mirror. This capability is a critical part of a system for the remote detection of hazardous liquids by photoacoustic (or, indeed, photothermal) techniques.

6. CONCLUSIONS

The PIPAS technique has been demonstrated at a range of 8 metres in the laboratory. Algorithms have been developed to process the PIPAS signals that succesfully identify the presence of simulant liquids. This result gives confidence in the viability of the technique in a realistic scenario, and allow the remote detection of condensed phase liquids in the field, with the potential for positive identification of persistent hazardous liquids from ranges of ten metres or greater. This would represent a significant improvement on the current contact detection techniques which risk exposure of the operators. There are several issues requiring further development in order to achieve a fieldable system. These include platform motion compensation and the provision of a laser source with the required wavelength, pulse energy, repetition rate, and frequency agility.

The biggest drawback identified with the technique is the low coverage rate. However, it has been shown that with an undersampling strategy, an acceptable compromise may be reached between coverage rate and missed signals. While coverage rates are limited for PIPAS, we have shown that appropriate sampling could permit interrogation of a road surface at speeds of several km/h in favourable conditions. The PIPAS technique compares favourably with other methods proposed for remote chemical detection such as Raman scattering, differential reflectivity (DR) and photothermal emission [9] which suffer from low specificity and/or low sensitivity.

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