Full Paper

Near Real-Time Standoff Detection of Explosives in a Realistic Outdoor Environment at 55 m Distance

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Abstract

Standoff identification of explosives at distances of up to 55 m has been performed by applying spontaneous Raman spectroscopy. This work has been focused on detection in a realistic environment, using an outdoors test field and performing experiments under varying weather conditions such as rain- or snowfall or bright sunshine. The instrumentation, based on a 532 nm pulsed laser source combined with gated detection, proved the performance insensitive to weather variations. Investigated HMEs and precursors were TATP, HMTD, HP, MEKP, NM, NB, and IPN; all in bulk quantities. The time needed for acquiring spectra was typically between single pulse (5 ns) and 10 s. Detection through green and brown glass bottles and PET bottles were tried and found viable.

Keywords: Explosives Detection, HME, IED, Raman Spectroscopy, Standoff

1 Introduction

Improvised explosive devices (IEDs) are a common and growing threat to civilian society as well as military operations. Detecting and identifying explosive's charges and IEDs in real environments (indoors or outdoors) is a very challenging task and, in many cases, also a very hazardous task. From the perspective of the first responders and military personnel, much would be gained if detection and identification could be performed reliably from safe distances. This drives the research of possible standoff detection methods.

IEDs are unique in nature because the IED manufacturer has to improvise with the available materials. For this reason, the threat varies in different parts of the world; it varies over time and evolves, as countermeasures are taken for protection against them. An IED will contain any explosive that is available; this means commercial, or military explosives, or home made explosives (HMEs). The diverse and varying nature of IEDs pose great challenges to the methods for their detection. The developed methods will need to be both sensitive and selective in order to detect an explosive material in gas phase and at low concentrations, or as trace amounts of particles on a surface, among an unknown number and composition of possible interfering substances, and to do this at a distance of some tenths to a few hundreds of meters. Techniques that are anticipated to fulfil these requirements are the spectroscopy based ones.

One spectroscopic method that has gained interest for its possible use in standoff measurements is Raman spectroscopy. Raman is an instantaneous process; an inelastic scattering of photons where some energy is lost to (or gained from) the target molecule, returning scattered light with a different wavelength, the difference corresponding to an energy shift in the molecule. In this way, Raman spectroscopy probes the vibrational modes of the target molecules. These vibrational modes can be regarded as a fingerprint that uniquely identifies the substance or substances in a sample. Also complex mixtures can often be analyzed using algorithms for pattern recognition.

Standoff Raman spectroscopic systems have been developed for the characterization of planetary surfaces [1 – 5] at distances between 5 and 65 m, using doubled Nd:YAG lasers at 532 nm. Carter et al. [6, 7] have studied the identification of 4 – 8% solid explosives in dry sand at up to 50 m using 532 nm from a Nd:YAG laser. Sharma et al. [8, 9] have made a portable remote Raman system for monitoring environmental pollution and gases on planetary surfaces up to

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100 m, using the second harmonic (532 nm) from a Nd:YAG laser. They also measured standoff spectra of explosives at 10 m distance as well as of various liquid explosives and precursors at distances up to 100 m [8].

A disadvantage with Raman spectroscopy is the small cross-sections as compared to infrared (IR) absorption spectroscopy or laser induced fluorescence (LIF). Raman scattering (inelastic scattering) is weaker than Rayleigh scattering (elastic scattering) by four to six orders of magnitude. However, just as Rayleigh scattering, Raman scattering is proportional to $1/\lambda^4$, giving a significantly higher Raman signal in the ultraviolet (UV) region of the spectrum. Several authors have taken advantage of this fact using lasers in the UV region. Brookhaven National Laboratory has developed a Mobile Raman Lidar Van (MRLV) [10] for the identification of bulk chemical spill (surface contaminations, ~ 500 g m^{-2}) at distances of 0.5 km or more using quadrupled Nd:YAG lasers at 266 nm and up to 60 s integration time. They have also performed point UV Raman measurements on some explosives [2] in 0.5 – 1% concentration, using 248 nm laser radiation. At this wavelength they noted a significant near resonance enhancement of some of the vibrational modes. Gaft and Nagli [11] have also pursued UV Raman detection and report reduced background interference as well as near resonance enhancement for a number of explosives on fluorescing background. The Raman cross-section can be significantly enhanced (by a factor of 100 – 10^6) [12] by resonance effects when the laser excitation frequency matches or nearly matches an electronic transition in the molecule.

Another standoff technique of interest is photo fragmentation with LIF detection (pf-LIF), where the detection targets sensitive identification of vibrationally excited NO-fragments produced in the fragmentation process of NO_2-containing explosives [13 – 16].

Efforts have also been made on using laser induced breakdown spectroscopy (LIBS) for standoff explosives detection [17 – 19]. The LIBS technique works by creating plasma from the targeted material and, thereafter, applying emission spectroscopy to identify the explosive by its elemental composition. Standoff measurements of 10 – 20 m distance have been reported.

Recently, standoff detection from 20 m, using photoacoustic spectroscopy of explosives on surfaces, has been reported. Using a tuneable quantum cascade laser as an optical source and a quartz crystal tuning fork for detection, a detection limit of 100 ng cm^{-2} is achieved in a laboratory experiment [20].

This paper aims at proving the applicability of Raman spectroscopy for the standoff detection of explosives under realistic field conditions. We have, for this purpose, done a large number of Raman measurements of IED substances performed at our outdoor testing site and at standoff distances (20, 30, and 55 m). This is done utilizing gated Raman spectroscopy with a pulsed Nd:YAG laser source of 532 nm wavelength. The measurements were initially done using equipment available in the laboratory, save the telescope. Various measurements from 30 m standoff distance are presented; these include nitromethane (NM), nitrobenzene (NB), and isopropyl nitrate (IPN) in single pulse to 100 pulse measurements. The possibility to detect low concentrations of hydrogen peroxide (HP) at standoff distances was investigated, concluding that a concentration of 5% in water is detectible in 100 laser pulses. Measurement through colored glass and PET material was successfully performed using 10 laser pulses at 30 m. Also included in the standoff measurements are the peroxides triacetone triperoxide (TATP), methyl ethyl ketone peroxide (MEKP), and hexamethylene triperoxide diamine (HMTD).

In all cases, the spectra show good agreement with the acquired reference spectra, using a bench top Bruker FT-Raman instrument; all vibrational features are identifiable in the standoff spectra.

2 Experimental Part

The instrumentation was kept indoors in a laboratory during measurements. The laboratory was placed adjacent to an explosives approved test field, which allowed the sample to be placed outside in an open environment. The test field was covered with grass during the green period of the year. Indoor instrumentation consisted of a pulsed laser and optics to direct out the laser beam into the test field, a fiber coupled telescope for detecting the Raman signal, and a spectrometer with gated ICCD for detection, all described in detail below. Since the experiments reported in this paper were conducted using an experimental setup under development, some components have been changed during the course of the work. Liquid samples were contained in 25 or 100 mL Pyrex glass laboratory bottles, if not otherwise stated. NM, NB, and IPN were of 100% concentration; HP had a maximum concentration of 30% and MEKP was of 40% concentration. TATP and HMTD samples were neat. For these samples, a small amount of water was added to the bottle to act as heat buffer, making the sample float on top.

The TATP and 20 m measurements were performed using a Quantel YG581C-10 Nd:YAG-laser with 12 ns pulse length, 280 mJ pulse energy, and 10 Hz frequency of operation. This laser was temporarily replaced by a borrowed Quantel Brilliant B Nd:YAG-laser with 5 ns pulse length, 450 mJ pulse energy, and 10 Hz frequency of operation, used for the 55 m measurements. In the process of improving the setup, the Quantel YG581C-10 laser was replaced by an Ekspla Nd:YAG laser NL303HT with a pulse length of 5 ns, a pulse energy of 320 mJ, and a pulse frequency of 10 Hz. The Ekspla laser is better suited for field experiments, mainly due to size, cooling and power requirements. All lasers are Q-switched and were operated at the second harmonic wavelength of 532 nm.

At a distance of 2 m from the laser, the laser beam was deflected at $\sim 90°$ by a prism or by a mirror coated for 532 nm. The laser was placed in an adjacent room at 2 m distance from the prism/mirror used to direct the beam. The prism was mounted in a coaxial position. When the mirror was used (TATP, 20 and 55 m measurements), an oblique

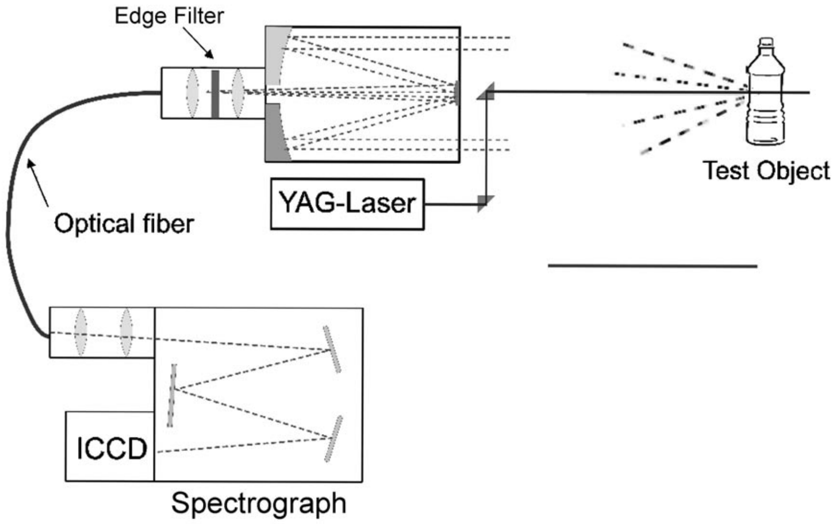

Figure 1. Experimental setup in croaxial geometry.

configuration was utilized. The distance between the telescope and the test object was either 20, 30, or 55 m.

The telescope was a C6-S Celestron of compact Schmidt–Cassegrain configuration. It had a 150 mm (f/10) aperture, peak light transmission of 89% at 520 nm, and an adjustable focus. This allowed for easy coupling into a 10 m UV fiber with a 400 μm core diameter. An interface, designed by Thorlabs, was used for this purpose. The interface was composed of two achromatic doublets (BK7, $f = 45$ mm, broadband AR coating centered at 587 nm), mounted in a stackable lens tube with an interchangeable long-wave-pass filter in between. The filter used was an LP03-532RU-25 from Semrock with OD 6 at the laser wavelength but transmitting over 99% for wavelengths above 538.9 nm (corresponding to a Raman shift of $\sim$241 cm^{-1}). A spectrometer from Jobin Yvon, SPEX 500 M with a SPEX 500F fiber coupling was used. The grating was from Richardson Grating Laboratory with 600 grooves mm^{-1} and blazed for 300 nm. Spectra were calibrated using an Oriel Hg pencil lamp. The setup is depicted in Figure 1.

The 20 and 55 m measurements and the TATP measurement were recorded using a gated ICCD (Jobin Yvon CCD-3000i) controlled by SpectraMax software. The detector had an air-cooled thermoelectric housing and a maximum dynamic gain of 18000. The chip measures 1024 × 256 pixels, each being 26 μm × 26 μm in size. The detector was mounted in the spectrometer so that the Raman spectra were dispersed horizontally along the chip. When depicting the spectra in image mode, it could be seen that the vertical extension of the spectra was covering no more than 30 pixels in the center of the chip. These pixels were binned together and the rest discarded to reduce electrical noise. This was an old detector, not fully operating according to specifications and with overall poor performance. The shortest possible gate width was used in the experiments and was estimated to be between 500 ns and 1 μs (100 ns according to specification).

All other measurements were made using an Andor fast gated ICCD iStar 740i-18F-03 with 5 ns gate width and controlled by the Solis software. The detector had an air cooled thermoelectric housing and a maximum dynamic gain of 60000. The active part of the chip measures 1300 × 512 pixels, each being 13 μm × 13 μm in size. The detector was run either in full vertical binning (FVB)-mode or single track (binning the active pixels vertically). The alternation between settings did not influence spectral quality to a noticeable extent. The gate width used in measurements was 10 ns.

The measured signal intensity of the Raman acquisitions is given in arbitrary units; data in the combined diagrams have been rescaled and offset for the purpose of clearer presentation. For the standoff measurements, the scaling factor for traces within the same figure is given in the figure description.

Reference spectra were obtained using a tabletop Bruker 55 FT-IR instrument with an FRA 106 Raman extension. The instrument used a continuous YAG laser source of 1064 nm with a maximum power of 1 W. Spectra were acquired with a resolution of 4 cm^{-1} and laser power ranging from 100 to 350 mW depending on sample characteristics (e.g., stability, aggregation state, and Raman cross-section).

3 Results and Discussion

The focus of the efforts on Raman standoff detection of explosives is to prove its applicability in a realistic environment. From this follows that special emphasis has been put on testing the detection capabilities outdoors under the influence of varying weather conditions, from snowy winter days to sunny summer days. For the area in Sweden where the test field is situated the averaged maximum day temperature in July (hottest month of the year) is +22 °C, while the averaged minimum day temperature in February (coldest

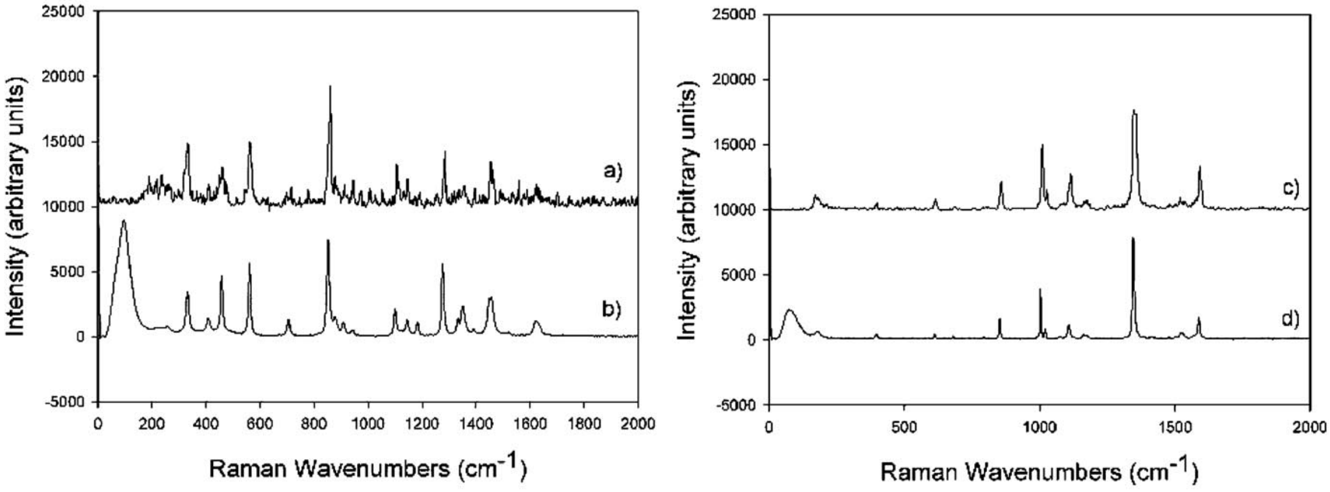

Figure 2. Raman spectra measured at 55 m distance with 100 laser pulses (10 s) and 100 μm slit width. The left diagram is from the IPN standoff measurement (a) and its Bruker instrument reference (b). The right diagram is the NB standoff measurement (c) and Bruker instrument reference (d). Note that the intensity of the reference spectra do not compare with the intensity of the standoff spectra.

month of the year) is −8 °C; all according to the Swedish Metrological and Hydrological Institute (SMHI) statistics of the Swedish weather for the years 1961–1990. During the winter time, measurements were occasionally performed several hours after sunset. The averaged relative air moisture content varies between 50 and 85% over the year according to the same source.

Other factors of interest are to assess the possibility to use Raman spectroscopy for detecting actual threat substances, thus including peroxides often encountered in terrorist contexts, and to investigate the possibility to use this method of detection if the HME is placed in a bottle made of colored glass or polyethylene terephthalate (PET).

3.1 55 m Measurements

Figure 2 shows the spectra of NB and IPN at 55 m distance. The quality of these standoff spectra is good enough to allow for longer distances or fewer laser pulses, without compromising the ability to identify the content of the bottle. Longer distances were not attempted in the described efforts since this would preclude keeping the laser beam within the perimeters of the test field. The 55 m measurements were performed early in the development process using the Jobin Yvon camera and the Coherent Brilliant B Nd:YAG-laser. The spectra were acquired at noon on a sunny winter day with snow covering the ground and, therefore, with relatively high ambient background illumination.

Single pulse measurements have, since, been accomplished. A comparison of spectral qualities for single pulse and 10 pulses spectra can be seen in the NM measurements presented in Figure 3.

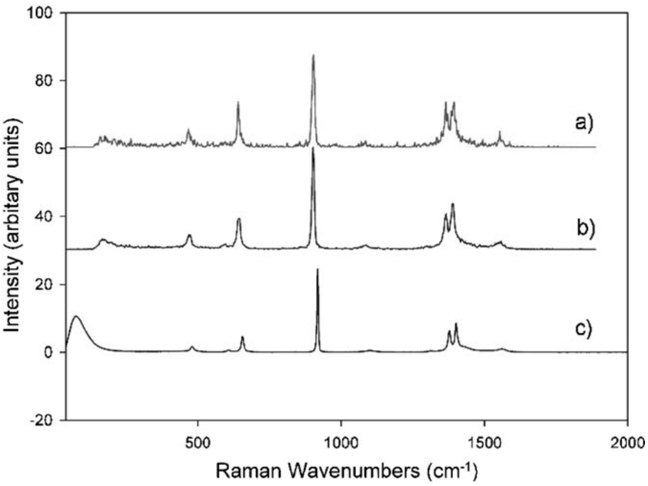

Figure 3. Raman spectra of NM standoff measurement at 30 m distance. From above: single pulse (5 ns) measurement (a); scaling factor = 10, trace 2:10 laser pulse (1 s) measurement (b); scaling factor = 1. Trace 3: reference spectrum (c); scaling factor is not relevant.

3.2 Influence of the Environment

Measurements have been made with the equipment indoors and the samples outdoors. This means that the measurement path and the bottle are exposed to all kinds of weather conditions including snow, rain, and sunshine. None of these conditions have been found to influence the results in any significant way.

Snow and rain could be expected to compromise the measurements by absorption and scattering of both laser light and the Raman shifted return signal. Even with heavy snowfall, almost so that the target is not visible to the naked eye, acquiring Raman spectra of good quality has been unproblematic. A subjective opinion is that detection is not

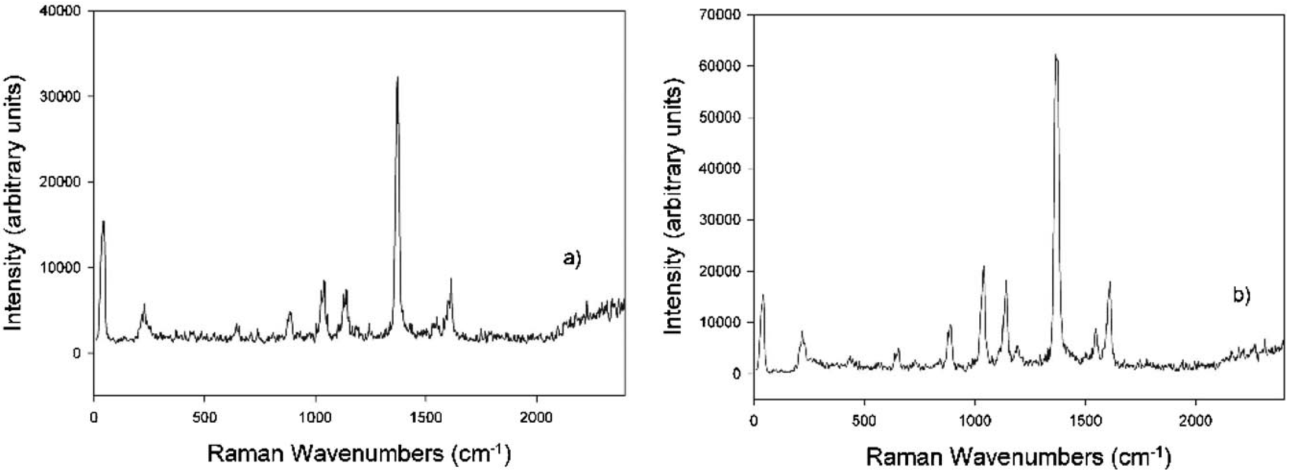

Figure 4. Raman spectra of NB. Both spectra are acquired in 100 laser pulses, or 10 s, at 55 m distance. The spectrometer slit width was 100 μm. The left spectrum (a) was acquired with the laser beam transversing a pile of snow that extended ~50 cm in the beam direction. The right spectrum (b) was acquired after removal of the snow.

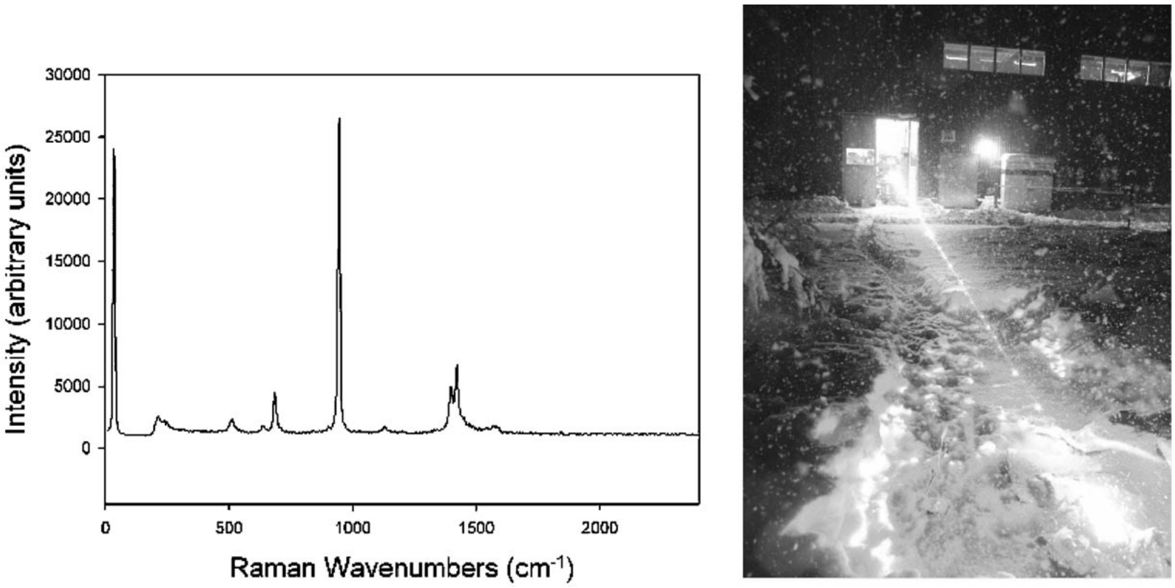

Figure 5. To the left is a Raman spectrum of NM. The spectrum is acquired at 20 m distance in 100 laser pulses, or 10 s, using a spectrometer slit width of 100 μm. The right picture illustrates the weather conditions during the measurement – the photo is taken during the measurement session.

influenced to any significant amount. Quantitative measurements of this phenomenon were never attempted. The insensitivity can instead be illustrated with Figures 4 and 5. In Figure 4, the spectra from NB were acquired from 55 m distance. In the first recording, the laser beam passed through a pile of snow, which extended ~50 cm in the beam direction. The snow had recently fallen and was, therefore, loosely packed. As soon as the snow pile was discovered, it was removed to increase the signal intensity. Although the snow did influence the Raman signal intensity, spectral quality is still surprisingly good. Figure 5 exemplifies one of the early measurements made of NM at 20 m distance; snow was falling quite abundantly that day as is shown in the photo taken during the measurement session. The chosen wavelength of 532 nm has good transmission through air and water; this contributes to the observed lack of sensitivity toward the prevailing weather condition. Under opposite weather conditions, i.e., when the sample is illuminated by bright sunshine, the use of gated detection contributes to a preserved spectral quality.

3.2.1 Hydrogen Peroxide Measurements

The precursors NB and IPN could easily be obtained in their concentrated form in the open market, as is also the

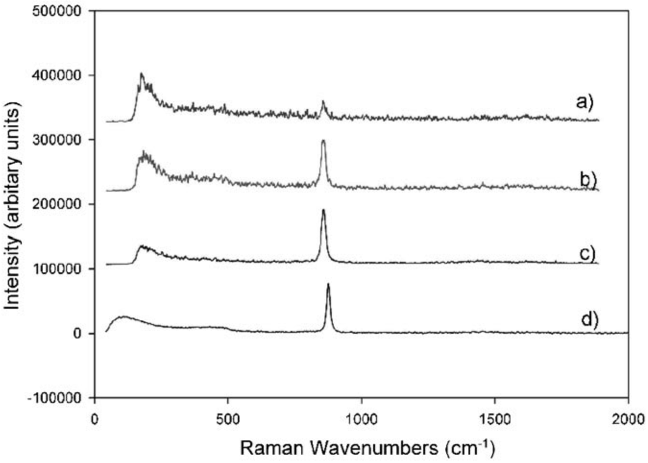

Figure 6. Raman spectra of HP. Standoff measurement from 30 m using 100 laser pulses (corresponds to 10 s) of Pyrex bottles of 50 mL containing solutions of HP diluted in water. From above: 5% HP (a); scaling factor = 4. Trace 2: 10% HP (b); scaling factor = 3. Trace 3: 30% HP (c), scaling factor = 1. Lowest trace: reference spectrum of 30% HP (d), scaling factor not relevant.

case for NM. Peroxides are typically acquired in a diluted form because of their explosive properties. This motivated the standoff measurements of diluted HP. As is seen in Figure 6, a concentration of 5% HP in water still shows the spectral feature associated with HP (O–O stretch at 877 cm^{-1}). The Raman signature from water lies beyond 2000 cm^{-1} and does not interfere with the HP signature, nor with the signatures from the other liquid samples in our experiments.

3.3 Bottle Variations

Laboratory-based experiments are typically designed to have the best possible prerequisites for optimal performance. In the case of detecting liquid samples, this is exemplified with our choice of Pyrex glass laboratory bottles, which are expected to have excellent optical transmission at 500 – 600 nm wavelengths. To perform Raman spectroscopic measurements of a liquid, the bottles or containers need to be transparent in the spectral region of interest. This obviously rules out bottles or containers made of opaque materials. In a realistic scenario, however, there are numerous possible bottle materials that fall in the category between laboratory bottle and opaque packing. These materials include bottles of colored glass and PET. In Figure 7, measurements of NM in colored glass bottles are presented. One brown bottle and one green bottle were used. Both the brown and the green bottles are standard 33 cl recycled glass bottles used in Sweden to bottle beer. In the case of the brown bottle, the signal to noise ratio is somewhat poorer, but the colored glass does not, in either case, pose a significant obstruction. The same positive result was achieved for the PET soda bottle measurement in Figure 8.

3.4 Peroxides – TATP, HMTD, and MEKP

To be implemented in realistic scenarios, standoff detection of versatile HMEs should be viable. A selection of HMEs was investigated for the possibility of their Raman standoff detection. These insensitive energetic materials

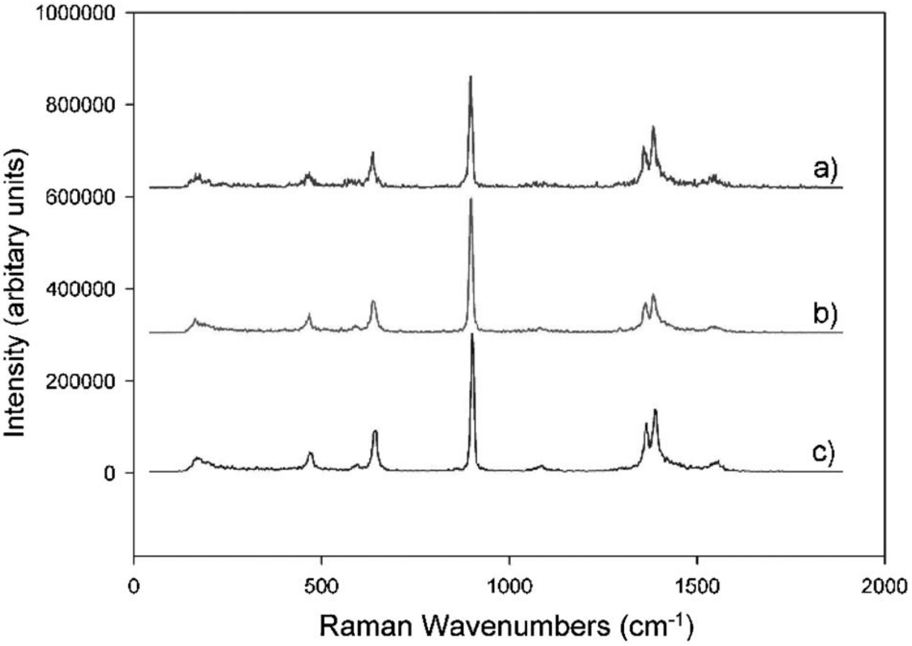

Figure 7. Bottle variations. Raman spectra of NM measured at 30 m distance with 10 laser pulses (1 s) and 100 μm slit width. Traces from above: 33 cl brown glass beer bottle (a), scaling factor = 6, trace 2: 33 cl green glass beer bottle (b), scaling factor 1.5. Lowest trace: 25 mL laboratory Pyrex bottle (c), scaling factor = 1.

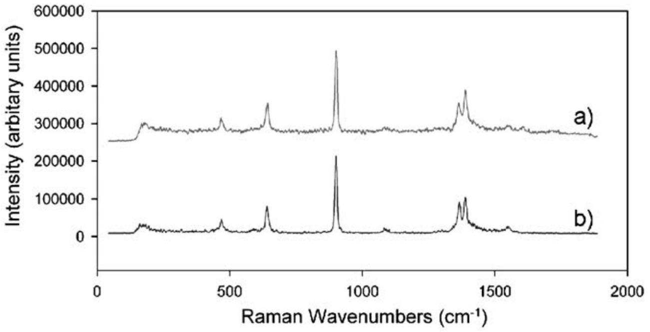

Figure 8. Raman spectra of NM in two different bottle materials measured at 30 m distance with 10 laser pulses (1 s) and 100 µm slit width. Reduced pulse frequency (2 Hz) and pulse energy (110 mJ) was used. Upper trace, 50 cl PET soda bottle (a), scaling factor = 1. Lower trace, 25 mL laboratory Pyrex bottle (b), scaling factor = 2.5.

(IEMs) are all peroxides and were chosen on the basis that they are easily produced out of publicly available materials and are commonly discussed in conjunction to terrorist bombings. An early 30 m standoff spectrum of TATP can be seen in Figure 9; 2000 laser pulses were used in this acquisition. The safety regulations associated with the TATP experiment made it impracticable to align the laser spot onto the TATP sample bottle. This fact in combination with an unstable and adjustment sensitive setup did most likely contribute to a somewhat poorer quality of the TATP spectrum than expected, although the TATP Raman signature is fully recognizable in the spectrum. TATP detection using the improved standoff setup was not attempted.

Of significantly better spectral quality are the HMTD measurements presented in Figure 10. The figure compares a 10 laser pulses spectrum with a 100 laser pulses spectrum.

MEKP of commercially available concentrations is a liquid and was used in its diluted form with 40% MEKP and 60% water. 10 and 100 laser pulse spectra are compared in Figure 11. As with the HMTD measurements, spectral quality is good.

3.5 Discussion

The choice of the Nd:YAG second harmonic wavelength of 532 nm for the Raman measurements is convenient from an optical and instrumental point of view. Falling within the visible spectral region, there are numerous optical components that are easily obtainable, even an off the shelf amateur astronomer's telescope has been found to perform very well in this experimental setup. In addition to these practical aspects, additional advantages exist: this wavelength has high transmission through air and water, making it plausible that standoff distances could easily be extended to hundreds of meters without seriously compromising spectral quality and acquisition time. It also has the advantage that the laser and the Raman shifted light can be transmitted through bottles of colored glass and PET. A possible drawback is that interferences from fluorescence often fall within the visible spectral region, making detection in a highly fluorescing matrix or background problematic.

By changing the incident Raman wavelength into the UV region, higher spectral quality is expected since the Raman signal intensity I is proportional to $1/\lambda^4$. Less influence of interfering fluorescence background is also expected and

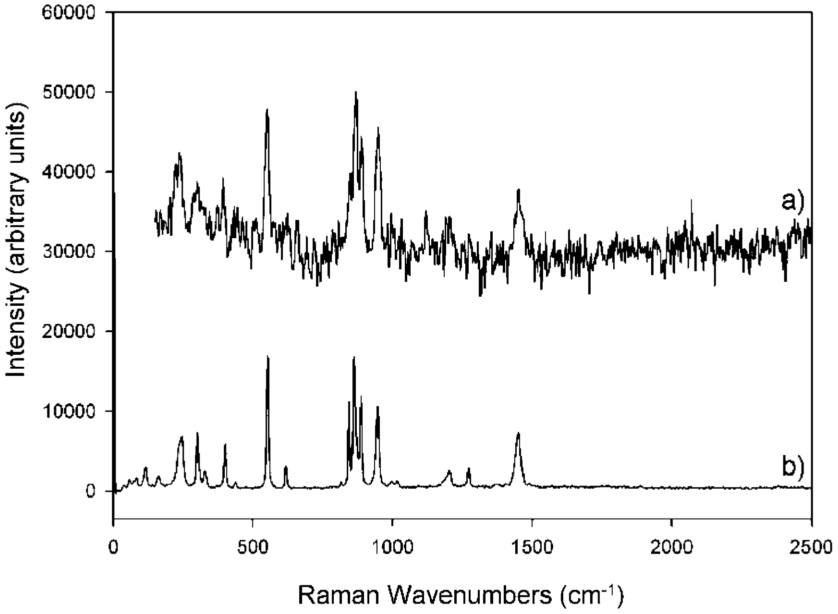

Figure 9. Raman spectra of TATP. Standoff measurement at 30 m using 2000 laser pulses (200 s) and 200 µm spectrometer slit width (a), scaling factor = 10. The lower trace is a reference spectrum from the Bruker bench top instrument (b), scaling factor is not relevant.

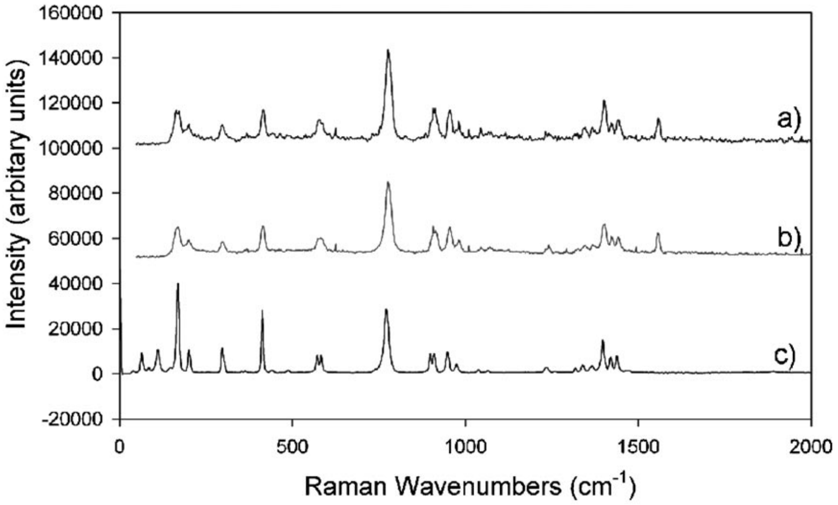

Figure 10. Raman spectra of HMTD. Standoff measurement at 30 m. Traces from above: using 10 laser pulses (1 s) (a), scaling factor = 10, using 100 laser pulses (10 s) (b), scaling factor = 1, and the Bruker instrument reference spectrum (c), scaling factor is not relevant.

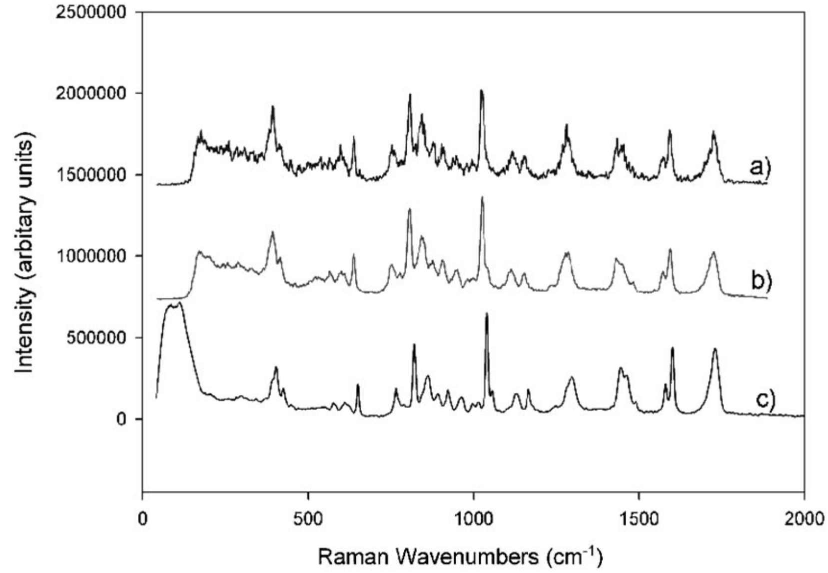

Figure 11. Raman spectra of MEKP. Traces from above: Standoff measurement at 30 m using 10 laser pulses (1 s) (a), scaling factor = 10 and 100 laser pulses (10 s) (b), scaling factor = 1. Lowest trace: reference spectrum from the Bruker instrument (c), scaling factor is not relevant.

has been reported by Lacey et al. [21] and by Gaft and Nagli [11], but at longer distances, some problems with absorption from molecular oxygen and water may occur, especially at wavelengths approaching VUV near 200 nm. Container materials other than UV grade quartz glass will not transmit the light satisfactorily. A conclusion from this is that it may, in fact, be beneficial to design an instrumentation which could be used in both the visible and the UV spectral region depending on the application at hand.

The issue of trace detection has not been covered in this paper. Experiments have been directed toward measuring appreciable amounts of the materials of interest. Spectral qualities so far have made it plausible that increasing the distance or decreasing the concentration of the sample materials is viable. To attempt trace detection, the influence of interfering Raman signatures must be considered, since they, under these circumstances, may be present in the same or a higher order of magnitude. Reduction of fluorescence background motivates the change of Raman wavelength into UV, despite possible losses from transmission through the air.

According to Svanberg [12], a further increase in sensitivity of $10^2 - 10^6$ times could be achieved by applying resonance Raman spectroscopy. Maximum resonance amplification could be expected on or close to electronic resonance transitions. This would imply the use of a laser source tunable from ~200 to 400 nm. This again leads to the conclusion that the potential for UV Raman, spontaneous as well as resonance, must be fully explored in the pursuit of detection of explosives.

4 Conclusion

Spontaneous Raman spectroscopy for the standoff detection of HMEs and precursors has been shown to have potential for detection and identification applications in realistic outdoor environments. This paper predominantly presents measurements of bulk amounts in tenths of seconds or shorter times. The high quality spectra vouch for the potential of further improvements, targeting either longer standoff distances or lower substance concentrations. A paper on Raman standoff detection at 470 m distance is under preparation by the FOI explosives' detection group [22]. No signal processing has so far been applied on the acquired spectra, applying algorithms for spectral recognition and background subtraction will further the detection capabilities.

Although many advantages can be expected when changing laser wavelength from 532 nm into shorter wavelengths (e.g., 355 and 266 nm), there are many situations where staying within the visible spectral region is beneficial. The possibility of performing detection through colored glass or PET bottles as well as measurements in rain and snow are examples hereof. It has also been observed that measurements in this spectral region are insensitive to weather variations such as rain- or snowfall.

The possibility to gain sensitivity through resonance Raman processes and also to suppress fluorescence background calls for the implementation of a UV-Raman instrumentation, but to act as a complement to the 532 nm instrumentation rather than to the sole solution.

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Symbols and Abbreviations

HME	Home made explosive
HMTD	Hexamethylene triperoxide diamine
HP	Hydrogen peroxide
IED	Improvised explosive device
IEM	Insensitive energetic material
IPN	Isopropyl nitrate
LIBS	Laser induced breakdown spectroscopy
MEKP	Methyl ethyl ketone peroxide
MRLV	Mobile Raman Lidar Van
NB	Nitrobenzene
NM	Nitromethane
PET	Polyethylene terephthalate
pf-LIF	Photo fragmentation with laser induced fluorescence
SMHI	Swedish Metrological and Hydrological Institute
TATP	Triacetone triperoxide