

Introduction:

Raman Spectroscopy has been shown to be useful in the study of explosives (1). One of the problems associated with the study of explosives by Raman spectroscopy is that samples are often too dilute for direct measurement. This problem can be overcome using the SpectRIM™ Slide.

This application guide demonstrates how even dilute solutions of explosives can be studied by using the SpectRIM Slide and the Drop Coated Deposition Raman (DCDR) technique invented in the lab of Dr Dor Ben-Amotz (2).

Raman Instrumentation

Any moderate to high resolution micro-Raman system can be used with this application guide.

Materials and Reagents

- Tienta SpectRIM Slide
- Explosive sample 1mg/ml in solvent
- Micropipette

Preparation of Explosive Solution

Stock solutions of the explosives were prepared in various solvents, as indicated in the Table 1 below, at concentrations of 1 mg/ml.

Procedure

Samples aliquots of 5 µL samples were then deposited on a SpectRIM slide and allowed to dry at room temperature.

Spectra, in this application guide, were collected with Almega XR Raman Spectrophotometer (Thermo Electron). The beam of the 785 nm laser was focused with 50X objective onto a selected area of a spot of explosive deposited on SpectRIM Slide explosive, and a Raman spectrum was collected in the broad range from 100 to 3400 cm⁻¹ in the high resolution mode of acquisition, with the slit set at 25 µm. The laser power was ~ 30 mW (and ~20 mW at the sample under these conditions) according to the manufacturer. Depending on the strength of Raman signals spectra were acquired either in 4 or in 2

accumulations of 20 s integration time each accumulation. All presented spectra were corrected for dark background noise.

Compound	Solvent
2,4,6-trinitrotoluene (TNT)	acetonitrile
Pentaerythritoltetranitrate (PETN)	acetonitrile
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	acetonitrile
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazine (HMX)	acetonitrile
Triacetone triperoxide (TATP)	acetonitrile
Hexamethylenetriperoxidediamine (HMTD)	acetonitrile
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	acetonitrile
Ethylene glycol dinitrate (EGDN)	acetonitrile
Triaminetrinitrobenzene (TATB)	acetonitrile
3-nitro-1,2,4-triazol-5-one (NTO)	acetonitrile
CI-20	acetonitrile
Nitrocellulose (NC)	acetonitrile
1,3,3-trinitroazetidine (TNAZ)	acetonitrile
Nitroglycerin (NG)	methanol
Picric acid (PA)	methanol
Ammonium nitrate (AN)	water
Ammonium perchlorate (AP)	water
Ammonium dinitramide (ADN)	water
Potassium nitrate	water
Potassium perchlorate	water

Table 1: Stock solutions of explosives prepared at a concentration of 1mg/mg, studied for the application of SpectRIM in detection of explosives.

Samples that displayed a broad spectral feature presumably due to fluorescence were subjected to a 5 to 10 minutes exposure to the light beam prior to spectral measurements. This exposure known as ‘fluorescence burning’ resulted in an appreciable decrease in the intensity of the broad band presumably due to photo-decomposition of fluorescence material. After such an exposure, this decrease in the intensity of the broad band levels off. In further support of the fluorescence origin of the band, change of the 785 nm light source to the 633 nm laser typically resulted in a more pronounced band of a different shape.

Raman bands were not affected by the change of light source or by the exposure

Results and Discussion

Figure 1 shows a photomicrograph of 2,4,6 trinitrotoluene deposited on the SpectRIM slide. The 5 μ l deposition at 1mg/ml in acetonitrile resulted in a circle with a diameter of approximately 1-2 mm.

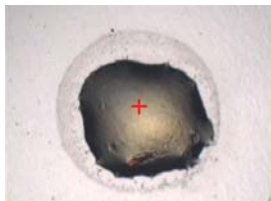


Figure 1: Photomicrograph of a 5 μ L droplet of 2,4,6-trinitrotoluene (TNT) dried on a Tienta SpectRIMTM Slide. The total amount of dried material was 5 μ g.

Figure 2 shows the Raman spectrum of 2,4,6-trinitrotoluene (TNT).

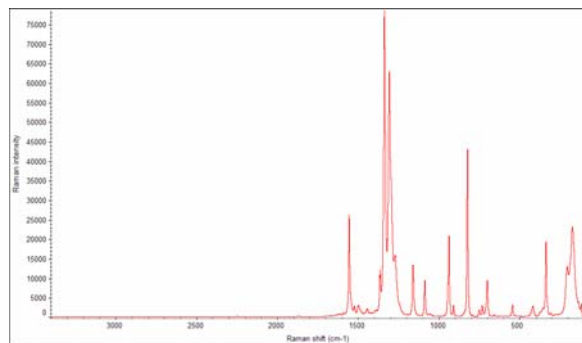


Figure 2: The Raman spectrum of 2,4,6-trinitrotoluene (TNT) taken on a Nicolet AlmegaTM XR Raman Spectrophotometer made by Thermo Electron. The spectrum was obtained using a 785nm laser, 50x.

Figure 3 shows the photomicrographs and Raman spectra of 5 μ g of hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) and Methyl-2,4,6-trinitrophenylnitramine (Tetryl) deposited on a SpectRIM slide.

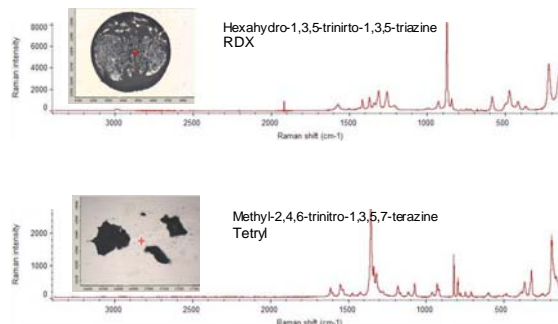


Figure 3: Samples deposited on Tienta Sciences SpectRIM Slide. Spectra collected on a Nicolet AlmegaTM XR Raman Spectrophotometer made by Thermo Electron. The spectrum was obtained using a 785nm laser, 50x .

The present work demonstrates that DCDR may be used to obtain high-quality normal (non-enhanced) Raman spectra from small quantities of explosives. The resulting spectra are highly reproducible.

Results may vary depending on instrumentation, illumination wavelength, integration time, and the thickness and purity of the sample.

References

1. Explosives Detection: A challenge for Physical Chemistry. Jeffrey I. Steinfeld, Jody Wormhoudt. *Ann. Rev. Phys. Chem.* 1998, 49 203-232
2. Raman Detection of Proteomic Analytes. Dongmao Zhang, Yong Xie, Melissa F. Mrozek, Corasi Ortiz, V. Jo Davisson, and Dor Ben-Amotz. *Anal. Chem.* 2003, 75 5703-5709.

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