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FT-Raman Spectroscopy of Some Energetic Materials and Propellant Formulations

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13. ABSTRACT *(Maximum 200 words)*

Fourier transform Raman (FTR) spectroscopy employing near-IR laser radiation at 1.06 μm as the scattering source has been used to obtain Raman spectra of some neat energetic materials and several propellant formulations containing those energetic materials. FTR spectroscopy is shown to be a useful tool in determining the principal ingredient in many noncolored propellant formulations.

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1. INTRODUCTION

Identification and characterization of energetic materials and propellant formulations are usually accomplished by using a combination of techniques. In the U.S. Army Research Laboratory (ARL), pyrolysis mass spectrometry (MS), gas chromatography-MS (GC-MS), liquid chromatography-MS (LC-MS), pyrolysis GC-FTIR, photoacoustic Fourier transform-infrared (FT-IR) spectroscopy, infrared (IR) reflectance and transmittance microscopy, and x-ray fluorescence techniques have been used for this purpose. More recently, Fourier transform Raman (FTR) spectroscopy employing near-IR excitation has been used to investigate energetic materials and propellant formulations.

FTR spectroscopy employing near-IR laser light has been shown to be useful for identifying energetic materials in commercially available explosives (Akhavan 1991; Hodges and Akhavan 1990). This report documents initial efforts by ARL to compile a data base of Raman spectra of pure energetic materials and of propellant formulations. Also, the report is the first in a series about FTR spectra of energetic materials and propellant formulations.

2. EXPERIMENTAL

Figure 1 shows the ray-diagram of the irradiation and collection optics of the experimental setup. Spectra were recorded on a Bomem DA-8.02 FT-IR spectrometer equipped with a CaF_2/quartz beam splitter and a liquid nitrogen-cooled InGaAs detector. Samples were irradiated with 1.06 μm (9,394.5 cm^{-1}) laser radiation using a Quantronix series 100 Nd:YAG laser which has a maximum continuous wave output of 6 watts. Back-scattered radiation was focused onto an aperture at the emission port of the spectrometer. A low pass interference filter was used to exclude the Rayleigh line.

All spectra were obtained at a resolution of 4 cm^{-1} using coaddition of between 100 and 500 scans. The total recording time for a 500-scan spectrum was approximately 18 minutes. Powdered samples were placed in a 1-mm ID glass capillary tube. The Nd:YAG laser beam was focused on the front surface of the tube with the focus adjusted to maximize the DC output of the detector. Cylindrical propellant grains were examined by focusing the laser at the center of the flat ends of the grain. No attempt was made to assure that the positioning of the grain was repeatable from run to run, although some effort was made (visual check only) to assure that the flat end surface of the grain was normal to the direction of the laser beam. After repositioning, spectra recorded on the same grain varied in the absolute intensity of the peaks but were virtually superimposable when normalized to the most intense peak in each spectrum.

Samples of energetic materials and propellant formulations were obtained from in-house sources. No purification of any energetic materials was attempted, so these are referred to as neat (not diluted or mixed with other substances) rather than pure in the text.

None of the spectra were corrected to account for the sensitivity of the detector or for filters used to exclude the laser radiation at 1.06 μm from the interferometer. The response of the liquid nitrogen-cooled InGaAs detector used in these experiments falls off rapidly at lower frequencies (<6,500 cm^{-1}), making Raman shifts unreliable at wavenumbers greater than approximately 2,900 cm^{-1}. Raman spectral frequencies reported here refer to the shift in wavenumbers from the exciting radiation at 9,394.5 cm^{-1}.

3. RESULTS AND DISCUSSION

3.1 Comparison With Other Techniques. It is useful to compare Raman spectra with other types of vibrational spectra for a given compound. Figure 2 shows the FTR spectrum of a neat sample of the energetic nitramine cyclotetramethylene tetranitramine (HMX). Table 1 lists assignments of some localized and quasilocalized vibrations in HMX. A notable feature of the FTR spectrum of HMX is the low intensity (relative to the IR absorption spectrum for HMX) of the band due to the NO_2 asymmetric stretch near 1,500 cm^{-1}. Figures 3, 4, and 5 are, respectively, FT-IR transmission (reported as absorption), reflectance, and photoacoustic spectra of neat HMX powder. In contrast to the FTR spectrum, the absorbance near 1,500 cm^{-1} is among the most intense in each FT-IR spectrum.

For HMX and for crystalline nitramines in general, FTR gives the best characteristic spectrum with the least amount of band overlap. The relatively low Raman intensity of the asymmetric NO_2 stretch makes it easier for other features of the spectrum to be observed (Hodges and Akhaven 1990). However, as discussed below, for amorphous or polymeric materials and formulations, FTR does not always give the "best" spectrum.

3.2 Neat Energetic Materials. Table 2 summarizes the energetic materials and formulations discussed in this and the following section. Figure 6 is the FTR spectrum of cyclotrimethylene trinitramine (RDX), the most common energetic nitramine and the six-membered ring homolog to HMX. Figure 7 is the FTR

Table 1. Assignments of Localized and Quasilocalized Vibrations in HMX

Raman Shift (cm^{-1})	Type of Vibration
200-500	Ring deformation
760	NO_2 wag
830-950	Ring stretch
1,080-1,200	Ring stretch
1,230-1,320	Symmetric $N-O_2$ stretch N-N stretch
1,400-1,500	C-H deformation
1,520-1,580	Asymmetric $N-O_2$ stretch
2,990-3,040	C-H stretch

Source: Iqbal et al. (1974).

Table 2. Summary of Spectra

Energetic Material	Figure Number	Laser Power (W)	Composition
HMX	2	0.8	neat
RDX	6	0.4	neat
β-HNIW	7	0.4	neat
Nitroguanidine (NQ)	8	0.4	neat
DMNA	9	0.6	neat
Nitrocellulose (NC)	10	1.5	neat
PETN	11	0.4	neat
TNT	12	0.3	neat
HMX2	14	0.4	80% HMX, 20% polyester
XM39	15	0.6	76% RDX, 12% CAB, 8% plasticizer, 4% NC
M43	15	0.4	same as XM39
M9	16	2.5	58% NC, 40% NG
M30	17	0.4	40% NQ, 28% NC, 22% NG (sample ignited)
C4	18	0.75	
CAB	19	1.5	neat

Note: NG = Nitroglycerin.

spectrum of hexanitroisowurtzitane (β-HNIW), a recently developed caged nitramine. Figure 8 is the FTR spectrum of nitroguanidine. The N-H stretch in this molecule occurs beyond the detector range (>3,200 cm^{-1}). Figure 9 is the FTR spectrum of dimethylnitramine (DMNA). Although not an energetic nitramine, DMNA is included here because it is a simple nitramine fragment model. Figure 10 is the FTR spectrum of nitrocellulose. Nitrocellulose was the poorest scatterer of all the neat energetic materials examined for this report and the only non-crystalline neat energetic material examined. Figure 11 is the FTR spectrum of the nitrate ester pentaerythritol tetranitrate (PETN), one of the principal ingredients in the commercial explosive Semtex C.

Figure 12 is the FTR spectrum of trinitrotoluene (TNT). The TNT used in these experiments consisted of light yellow needle-like crystals. Because of the coloration, some of the incident radiation gets absorbed, resulting in sample heating. The spectrum in Figure 12 is the FTR spectrum of TNT superimposed on the emission spectrum of the heated crystals. This "greybody emission" is seen in the slight lifting of the spectrum from the baseline. For TNT, it was necessary to use low laser energy to prevent the sample from combusting. It should be noted that decomposition products of neat samples increase thermal sensitivity to the Nd:YAG laser radiation.

As in IR reflectance and transmittance spectra of neat energetic materials (Iqbal et al. 1974; Goetz and Brill 1979) strong spectral features attributable to the asymmetric and symmetric stretches of the NO_2 group appear in the FT-Raman spectra near 1,500 cm^{-1} and 1,200 cm^{-1}, respectively. Unlike IR spectra, the features in the Raman spectrum, attributable to the symmetric NO_2 stretch near 1,200 cm^{-1}, are more intense than those for the asymmetric stretch.

In each of the FTR spectra shown, lines appear as the result of the Kr lamp used to pump the Nd:YAG laser. These lines are excluded from entering the interferometer by an interference filter. Figure 13 shows a spectrum collected with the laser turned on, no sample present, but with the Kr plasma filter removed so that spectral features in sample spectra, attributable to the Kr lamp, may be identified.

3.3 <u>Propellant Formulations</u>. Figure 14 shows the FTR spectrum of HMX2, a propellant composed of 80% HMX and 20% polyester, and the FTR spectrum of neat HMX. Although the samples were different in appearance (HMX2 was in the form of an extruded cylindrical strand or "grain" approximately 3 cm long by 1.5 cm in diameter, neat HMX is a white powder), the two spectra are almost superimposable. This points out both a strength and weakness of the FTR technique. From the spectra in Figure 14, HMX may easily be identified as a main component of HMX2. However, there is little or no evidence of the polyester binder that comprises 20% of the formulation.

Figure 15 shows the FTR spectra of XM39 and M43. Both formulations consist of approximately 76% RDX, 12% CAB (cellulose acetate butyrate, a polymer), 8% plasticizer, and 4% nitrocellulose. For comparison, Figure 15 also shows the FTR spectrum of neat RDX powder. The spectra are almost superimposable even though the propellant formulations were in the form of grains when the spectra were obtained. Again, there is very little spectral evidence of the noncrystalline components.

Figure 16 shows the FTR spectrum obtained from a grain of M9 (58% nitrocellulose and 40% nitroglycerin). A spectrum of neat nitrocellulose is shown for comparison (neat nitroglycerin was not available for these experiments). The M9, which contains no crystalline material, required 2.5 watts of incident laser power to obtain a satisfactory spectrum (roughly six times the power for a spectrum of RDX). Other researchers have noted that M9 is almost transparent to 1-µm radiation (Beyer, private communication). Still, it is apparent from Figure 16 that a principal ingredient in M9 is nitrocellulose.

Figure 17 shows the FTR spectrum of a grain of M30 (40% nitroguanidine, 28% nitrocellulose, 22% nitroglycerin). The FTR spectra of nitroguanidine and nitrocellulose are shown for comparison. M30 is dark grey in color, and combusts during the data collection at the lowest laser power that gives a satisfactory signal. From the spectrum of M30 in Figure 17 it appears that the principal ingredient is nitroguanidine. Evidence of amorphous nitrocellulose is only apparent in the peak in the spectrum of M30 near 800 cm^{-1}. Figure 18 shows the FTR spectrum of the explosive C4 and of RDX. It may be seen that the principal ingredient in C4 is RDX.

Nitramine propellant formulations used here typically contain about three-fourths crystalline energetic material, with the remainder of the formulation being comprised of polymers and plasticizers. The FTR spectra of the propellant samples show little evidence of components other than crystalline energetic material. This is because most of the crystalline energetic materials are much better Raman scatterers than are the amorphous plasticizers or polymers. As an example, the FTR spectrum of cellulose acetate butyrate (CAB), a polymeric binder used in XM39 and M43, is shown in Figure 19. To obtain this spectrum, it was necessary to use almost four times the power used to obtain a similar quality spectrum of RDX.

3.4 Miscellaneous Applications. Recently, we began using FTR spectroscopy to investigate the demilitarization of propellant using supercritical fluid extraction. Figure 20 shows the FTR spectrum of a grain of unextracted M43 (upper spectrum) and the FTR spectra of the exterior (bottom spectrum) and interior (middle spectrum) of a grain of M43 after extraction with supercritical CO_2 modified with acetonitrile (CH_3CN). The CH_3CN modifier is observed in the FTR spectrum of the exterior of the extracted grain (bottom spectrum) at around 2,200 cm^{-1}. A small peak at the same frequency may also be seen in the FTR spectrum of the interior of the extracted grain (middle spectrum), indicating penetration by supercritical fluid into the interior of the grain.

FTR spectroscopy has also been used to characterize unknown propellant samples. Figure 21 shows the FTR spectrum of neat RDX (lower spectrum) and the spectrum of an unknown compound suspected to be a plastic explosive (upper spectrum). From these two spectra, it is apparent that a principal ingredient in the unknown sample is RDX. Figure 22 shows the FTR spectrum of M43 propellant, along with the FTR spectra of HMX and RDX. Typically, most RDX manufactured in this country contains a small amount of HMX (typically from 5 to 20%), a by-product of RDX synthesis. The arrows in Figure 22 point to features in the FTR spectrum of M43 attributable to HMX. Figure 23 is an expanded portion of the FTR spectrum of the unknown plastic explosive, along with the FTR spectra of M43 and neat RDX. No HMX is in the RDX used in the unknown sample. For this reason, it may be reasonable to suspect that the unknown sample was not domestically manufactured.

4. CONCLUSIONS

It has been shown that FTR spectroscopy is a useful tool in identifying the principal crystalline ingredient(s) of some propellant formulations. Unfortunately, the technique may not be very useful in the quantitative analysis of other propellant ingredients, such as polymers and plasticizers, because these other ingredients are poor scatterers of the near infrared (NIR) radiation. Also, darkly colored samples are difficult to analyze because they absorb the incident laser radiation and pyrolyze or ignite.

The main advantages of FTR spectroscopy for characterizing energetic materials are the ease of sample preparation, the relative insensitivity to sample positioning when obtaining spectra of propellant grains, and the ease with which crystalline energetic materials in propellant formulations may be identified, mainly because the neat energetic materials are usually good Raman scatterers. FTR spectra of crystalline energetic materials are usually "cleaner" than the corresponding spectra using absorption spectroscopy. (However, ATR and thin film transmission spectroscopy often give excellent results (Bartick, private communication). Finally, although most difficulties occur when examining colored samples, we have recently examined a brilliant white rocket propellant that began pyrolyzing at the lowest setting of the Nd:YAG laser, preventing collection of the FTR spectrum.

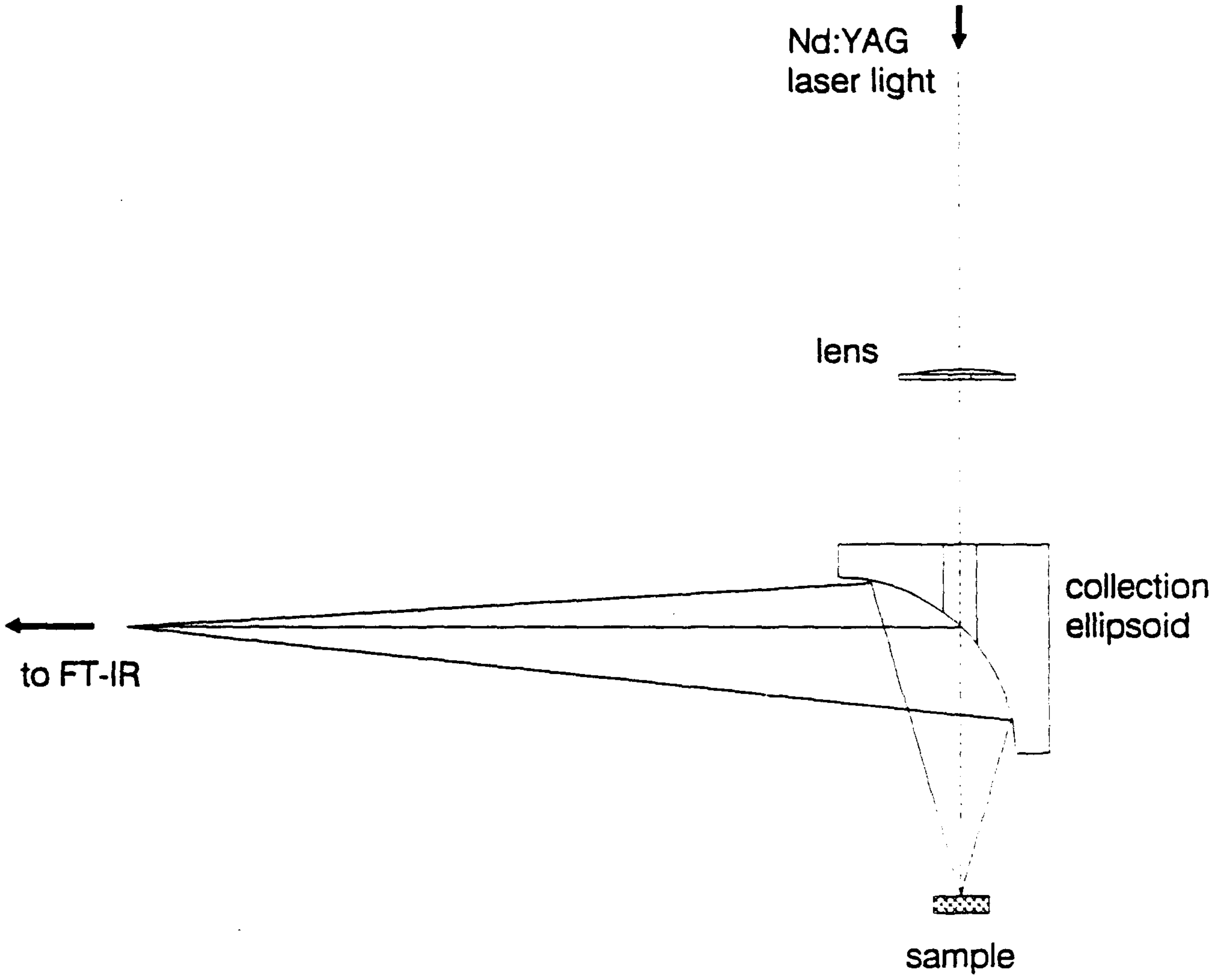

Figure 1. A simple diagram of the collection optics used on the Bomem Raman accessory.

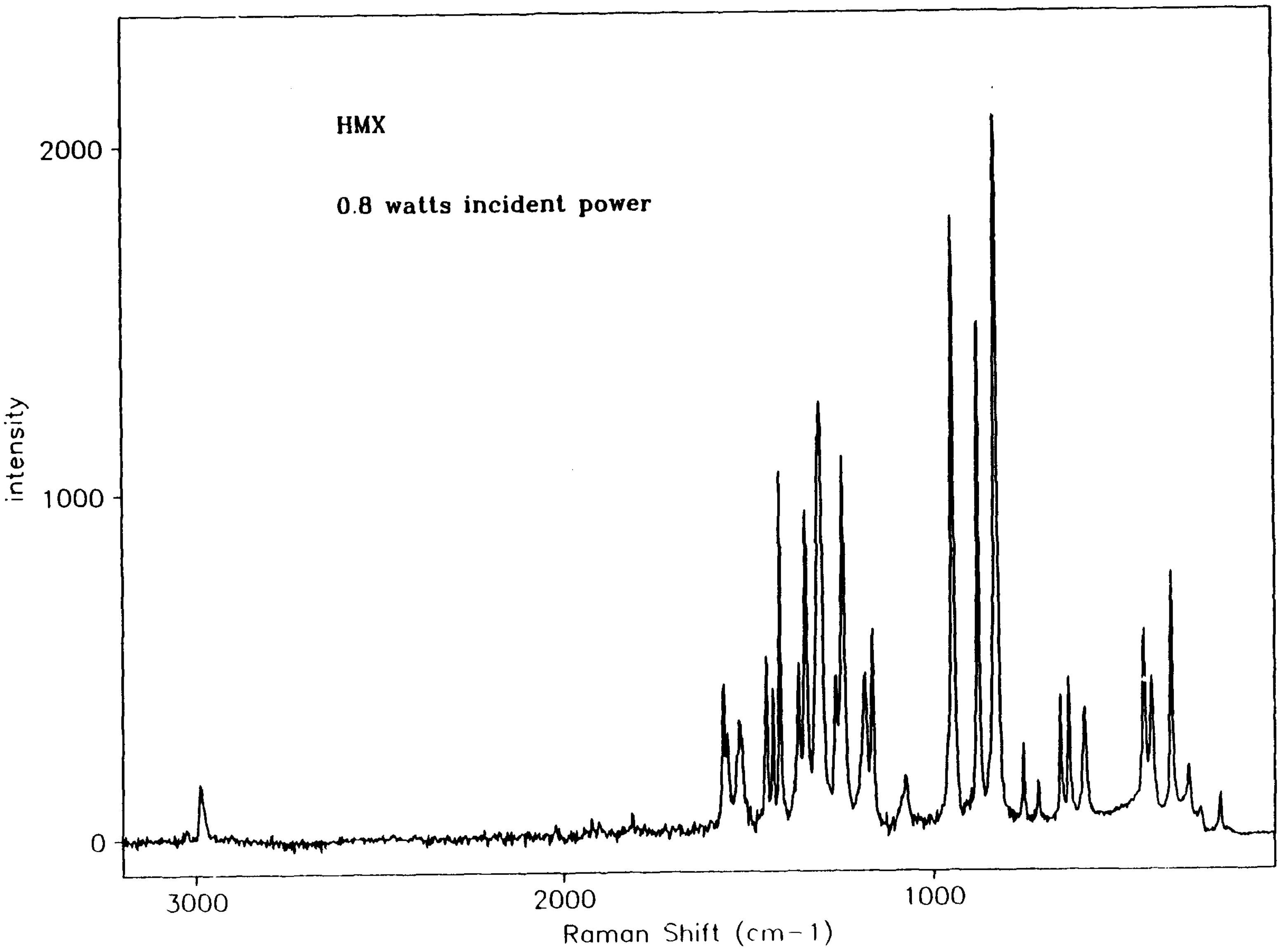

Figure 2. The FTR spectrum of HMX.

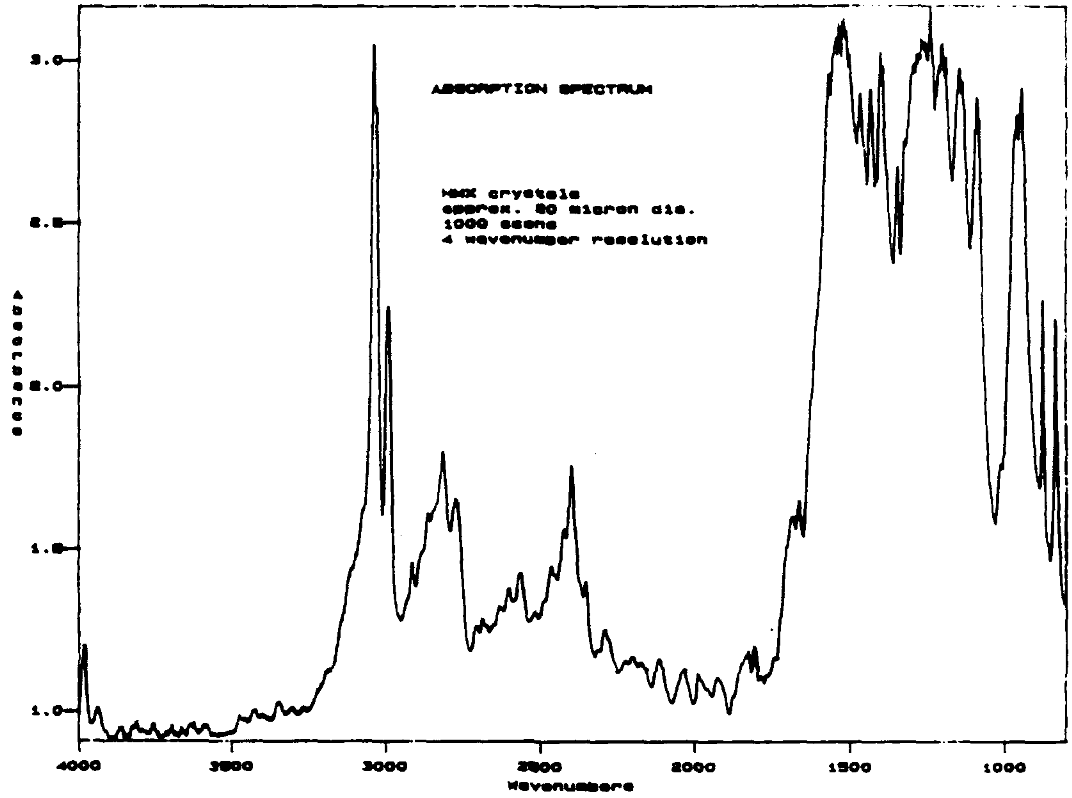

Figure 3. The FT-IR microscope absorption spectrum of neat HMX.

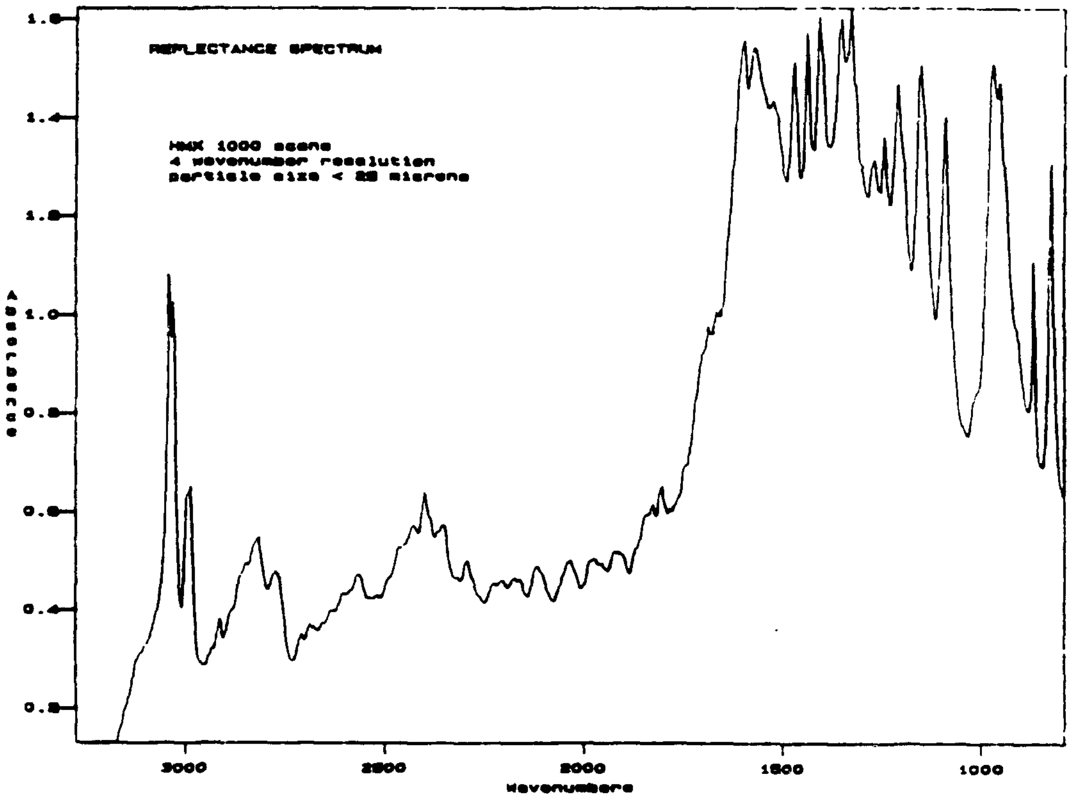

Figure 4. The FT-IR microscope reflectance spectrum of neat HMX.

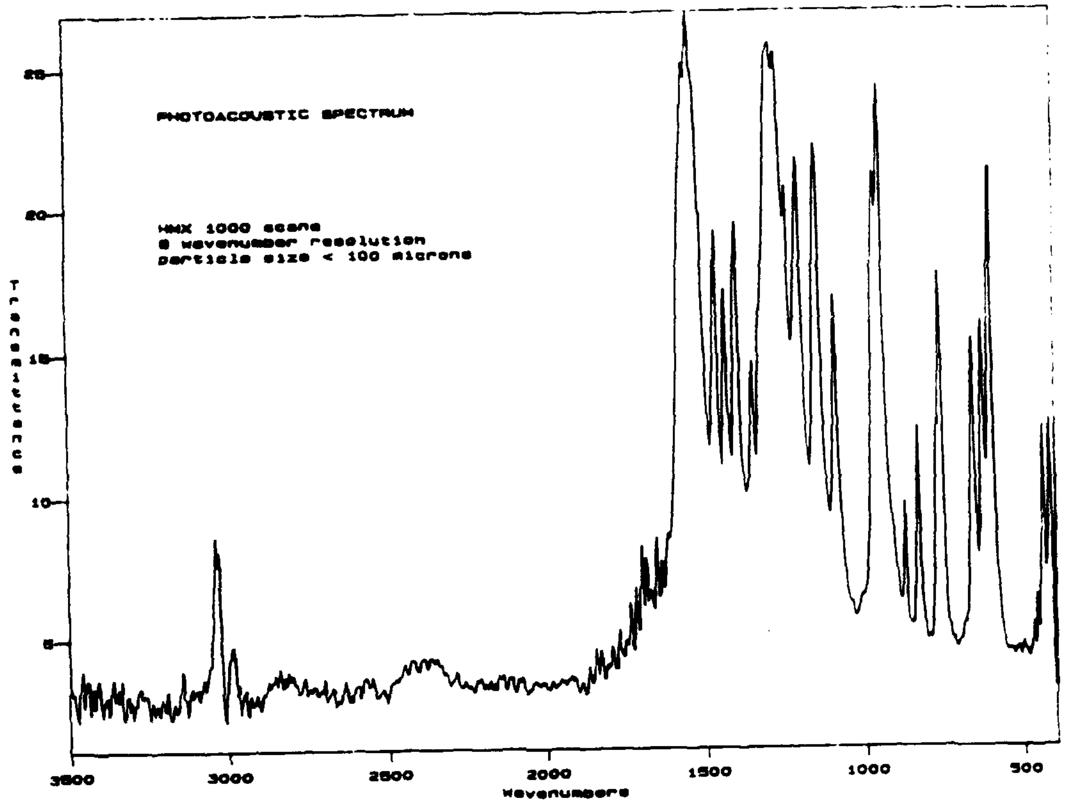

Figure 5. The FT-IR photoacoustic spectrum of neat HMX.

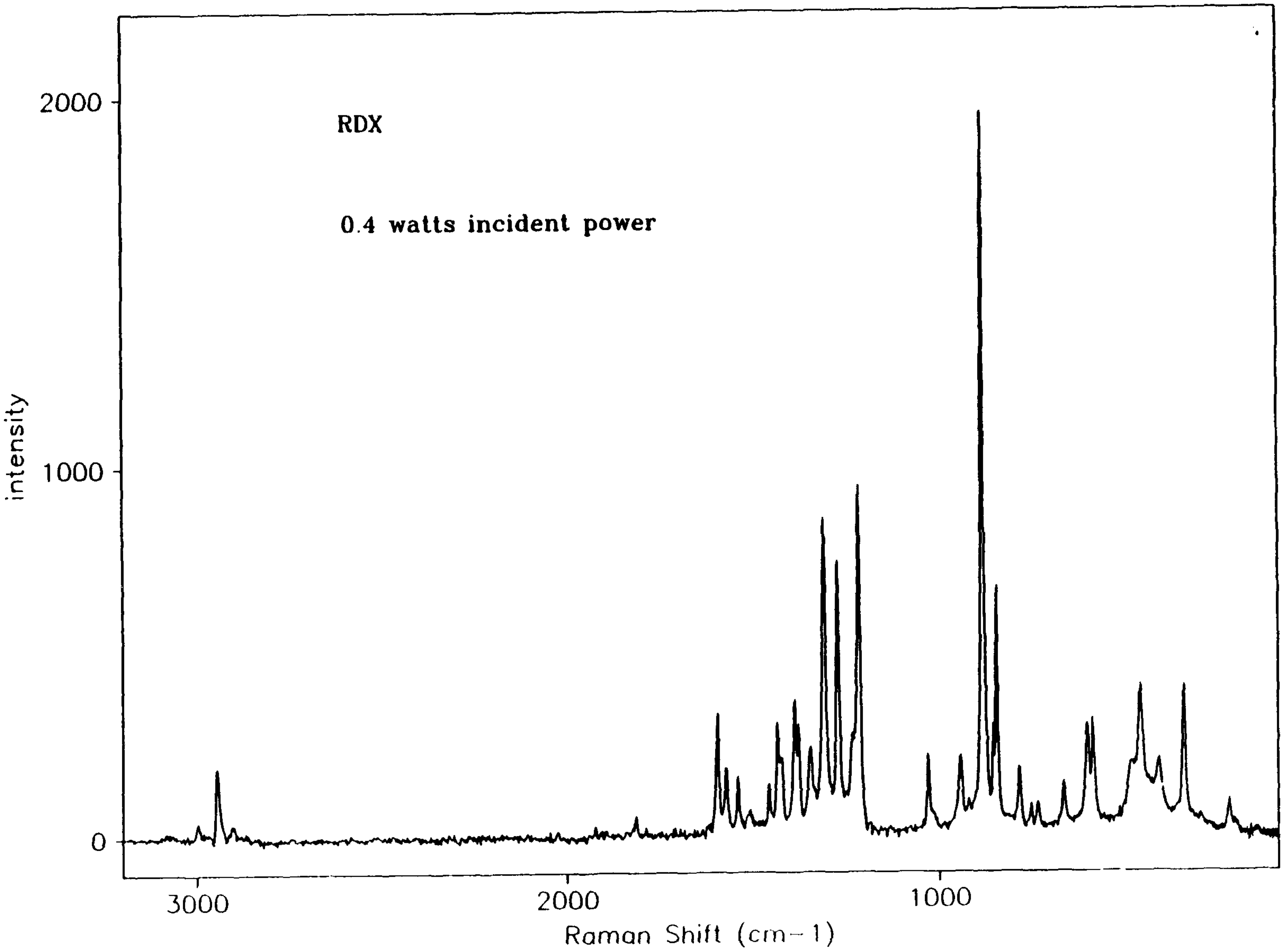

Figure 6. The FTR spectrum of RDX.

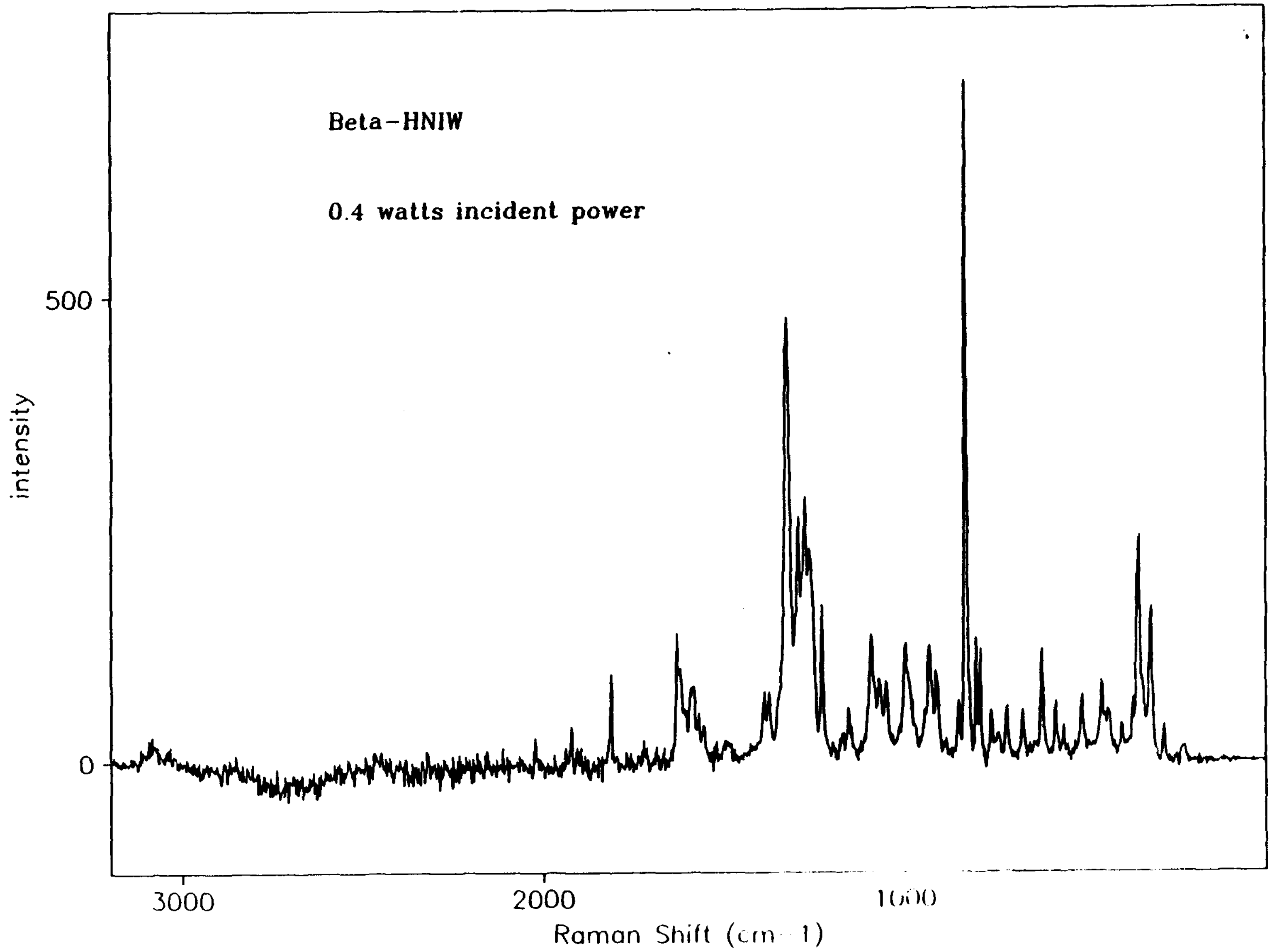

Figure 7. The FTR spectrum of β-HNIW.

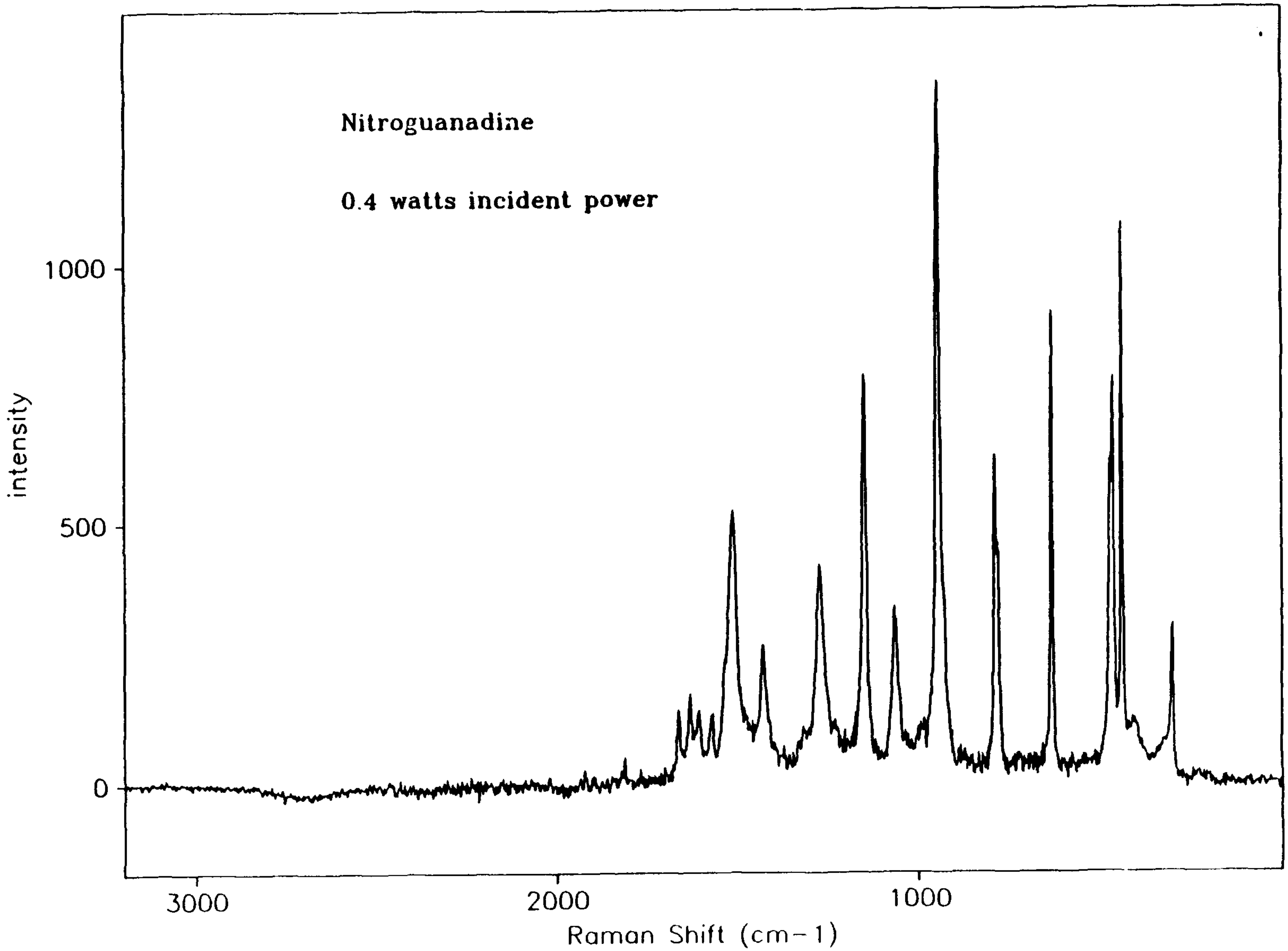

Figure 8. The FTR spectrum of nitroguanidine.

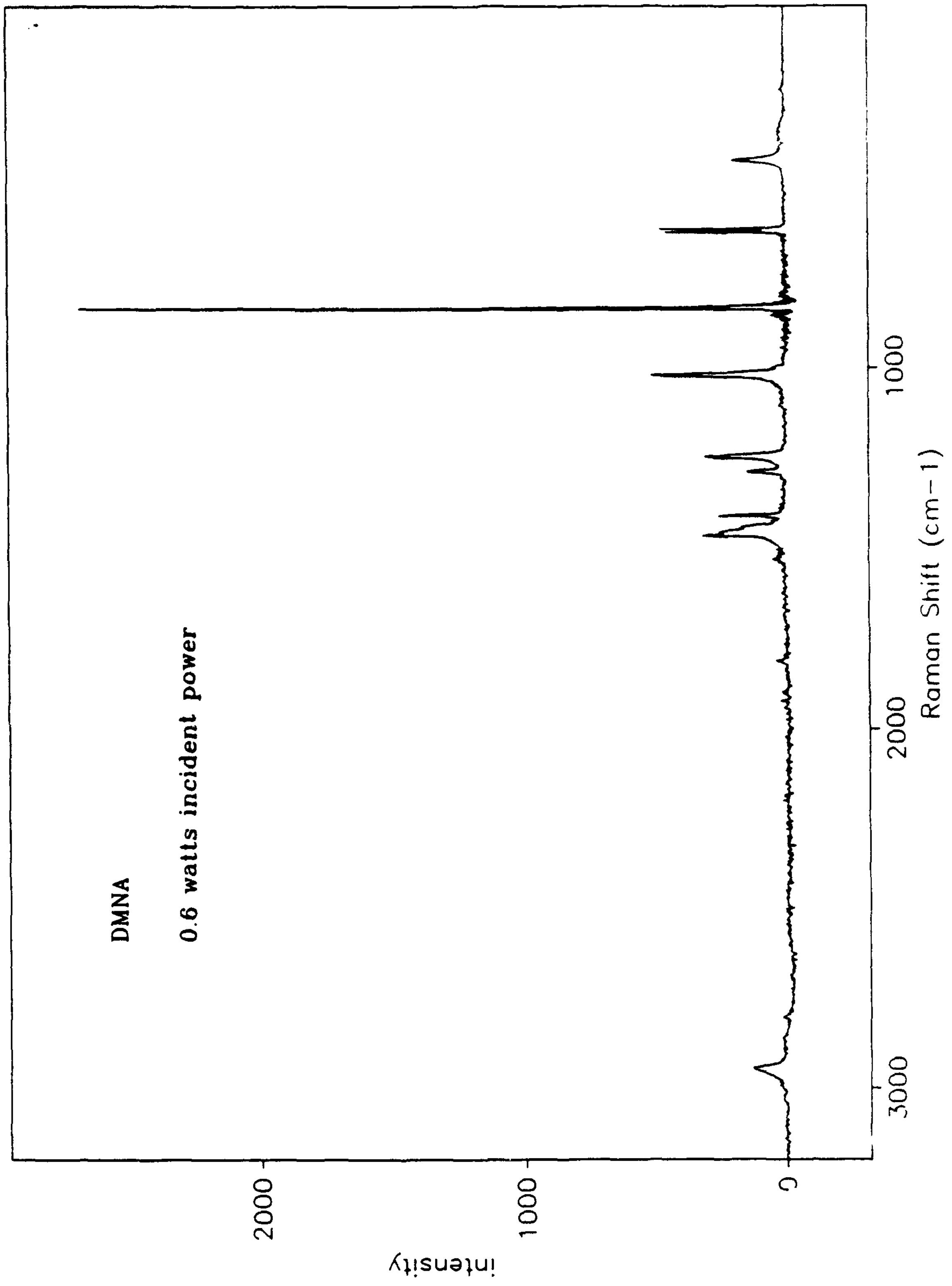

Figure 9. The FTR spectrum of DMNA.

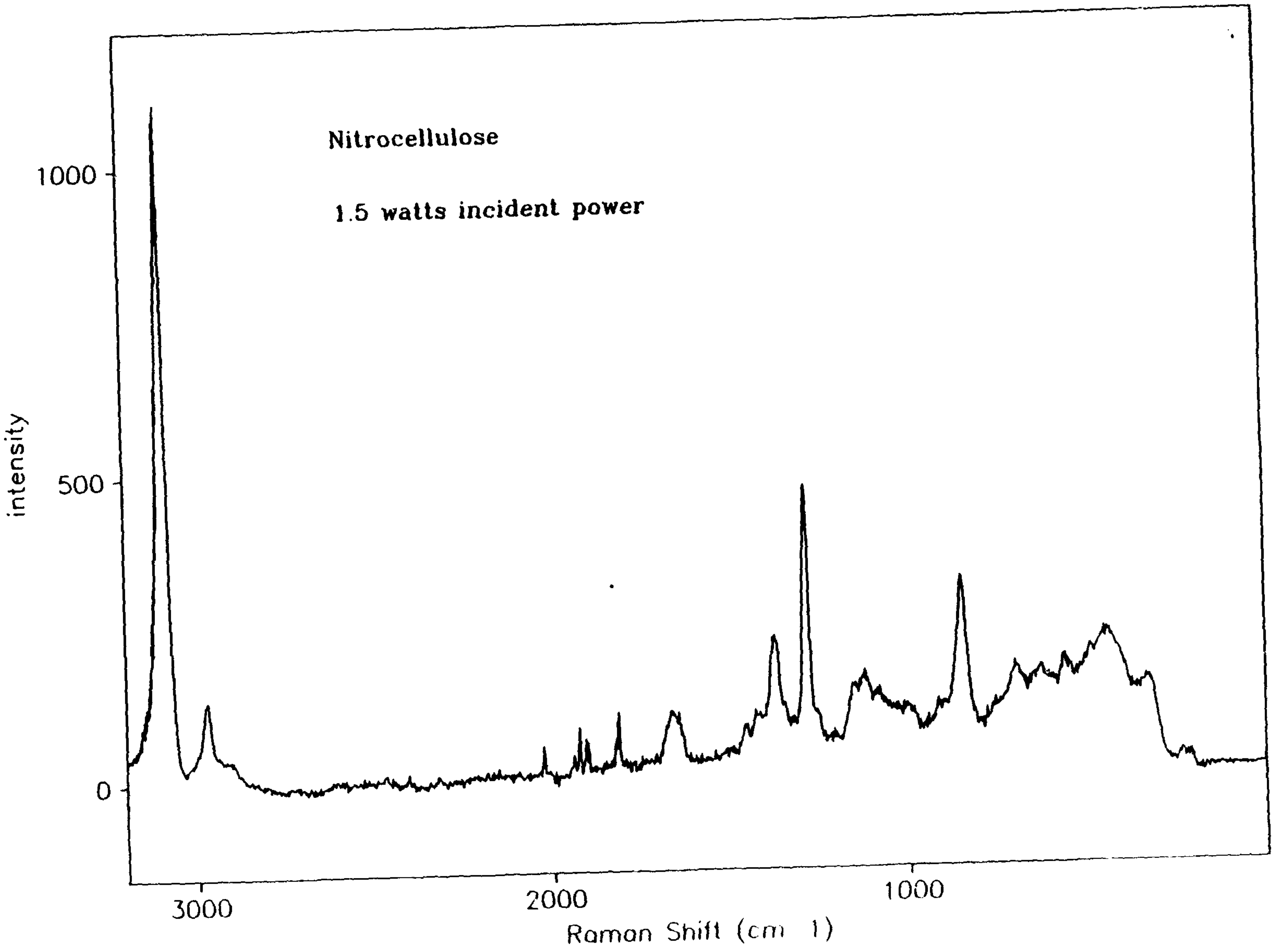

Figure 10. <u>The FTR spectrum of nitrocellulose</u>.

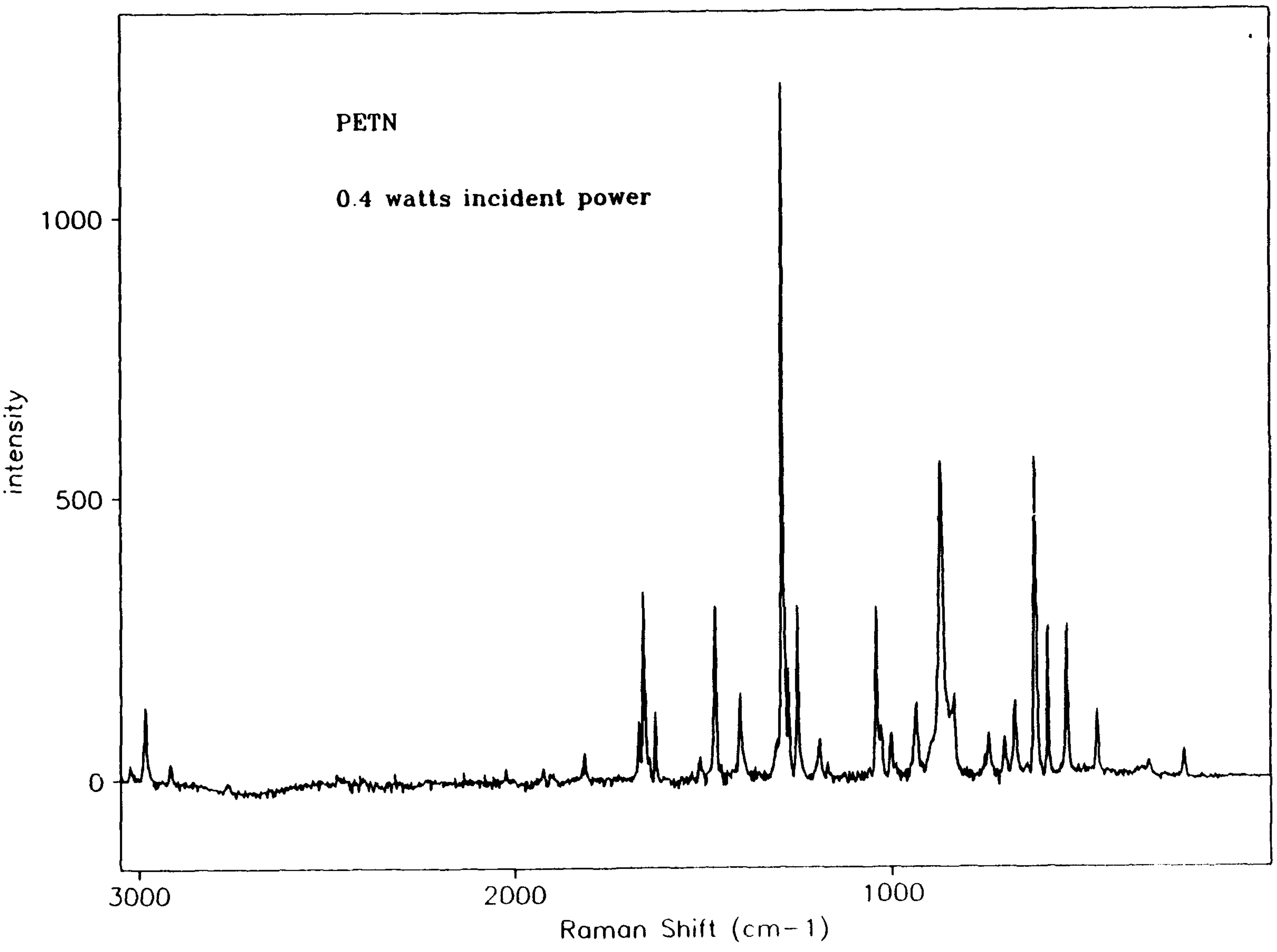

Figure 11. The FTR spectrum of PETN.

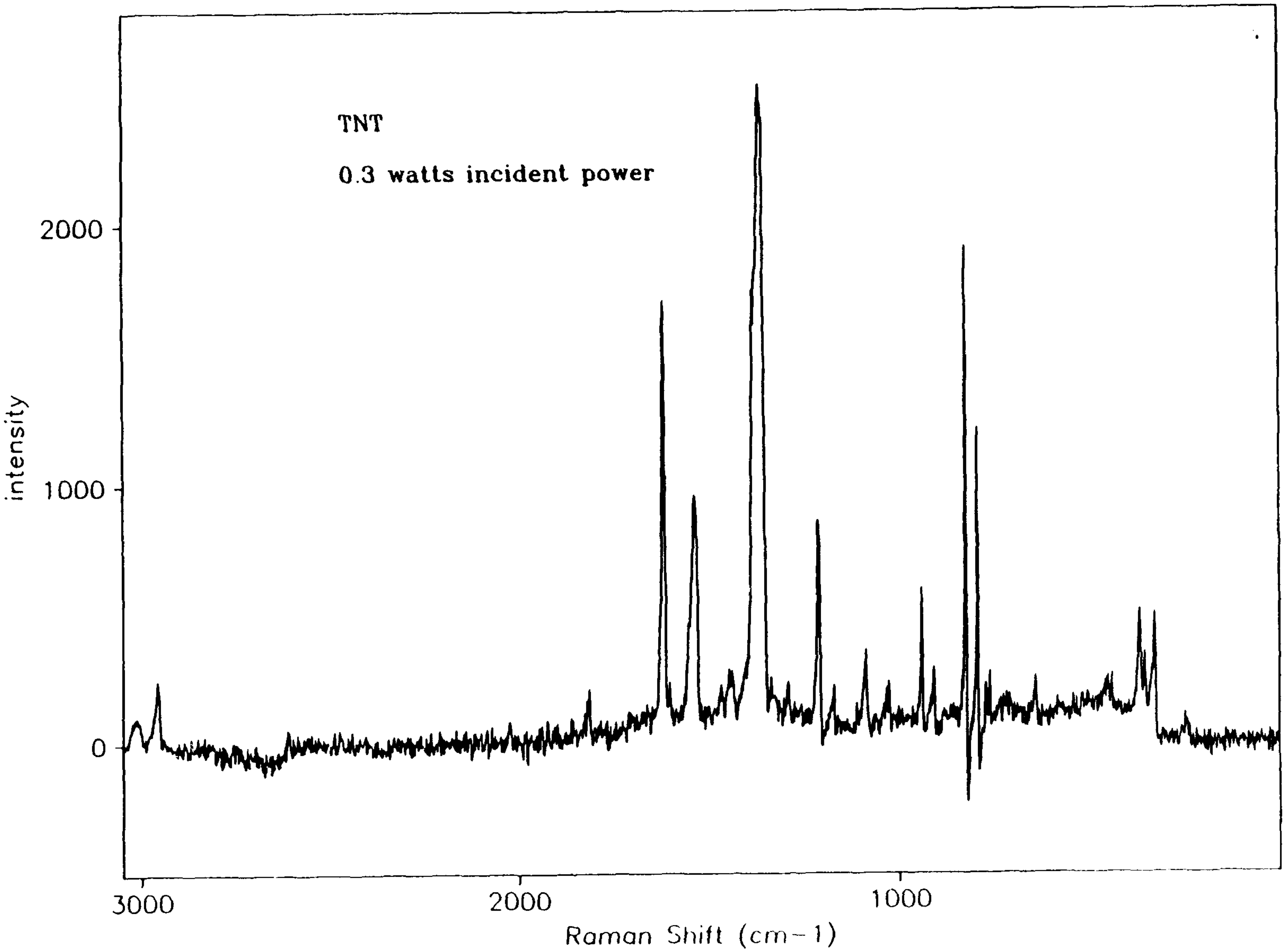

Figure 12. <u>The FTR spectrum of TNT</u>.

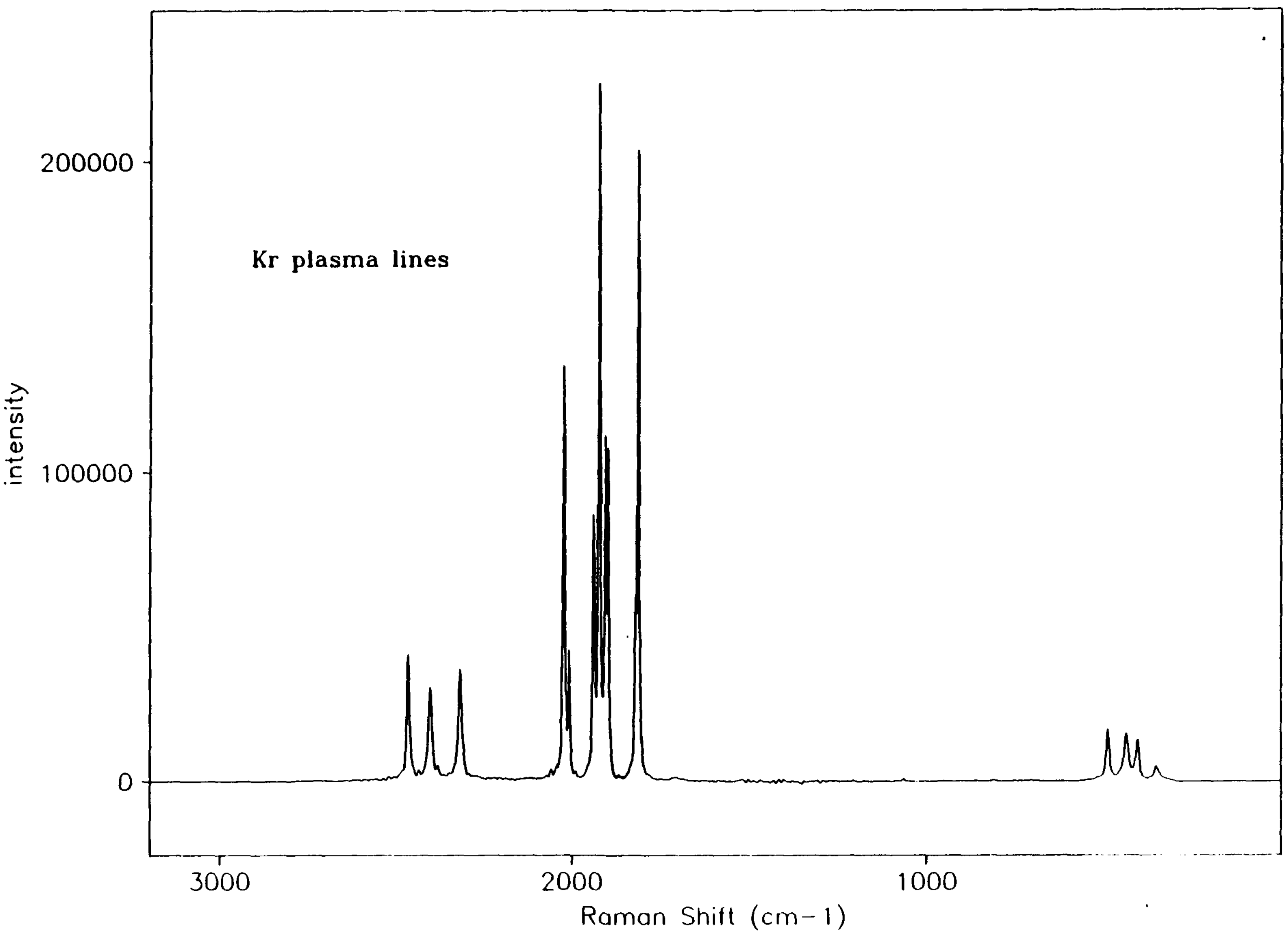

Figure 13. The spectrum of the Kr lamp used to pump the Nd:YAG laser; the plasma line filters have been removed.

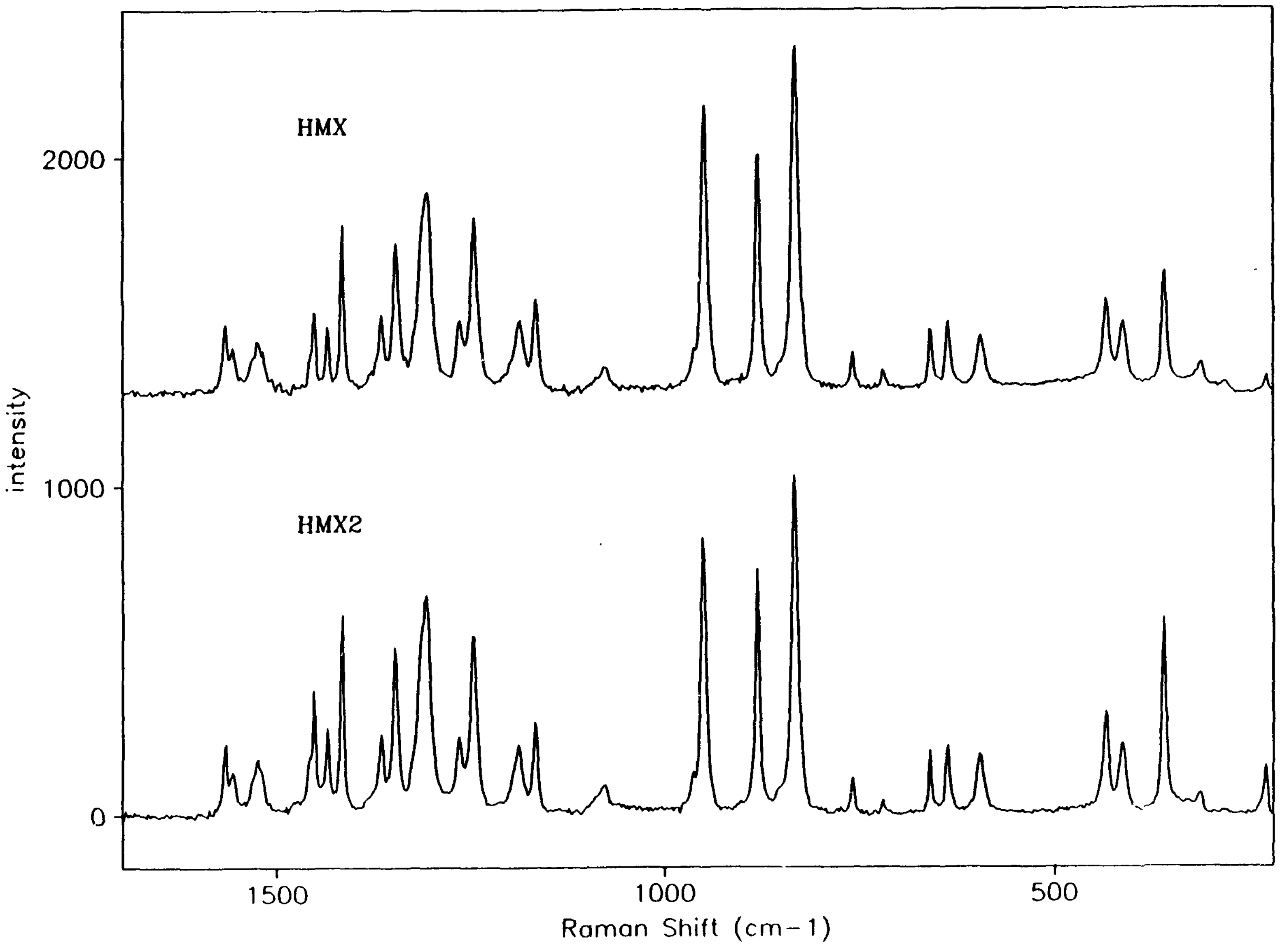

Figure 14. The FTR spectra of HMX (top), and HMX2 (bottom).

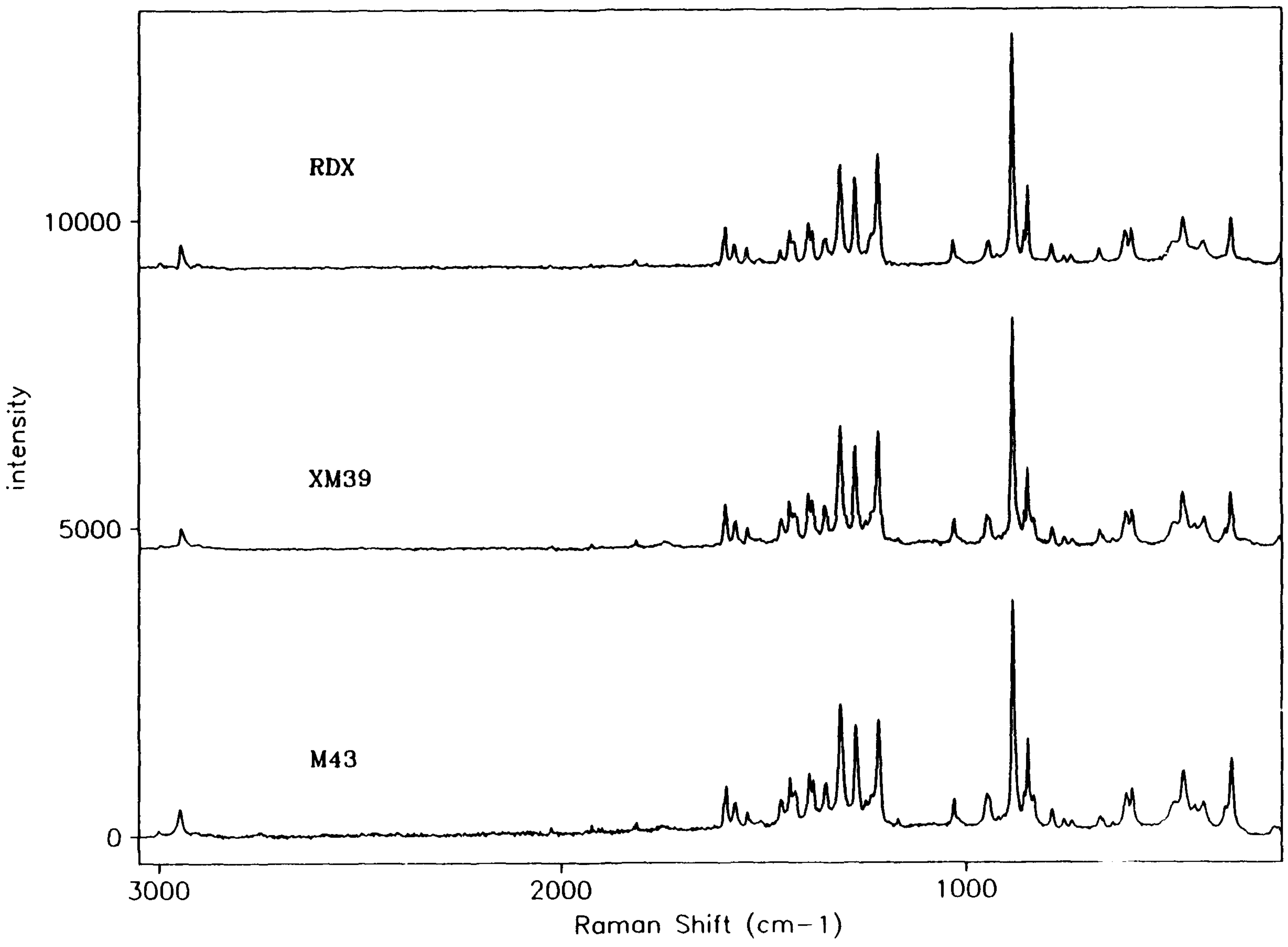

Figure 15. The FTR spectra of RDX (top), XM39 (middle), and M43 (bottom).

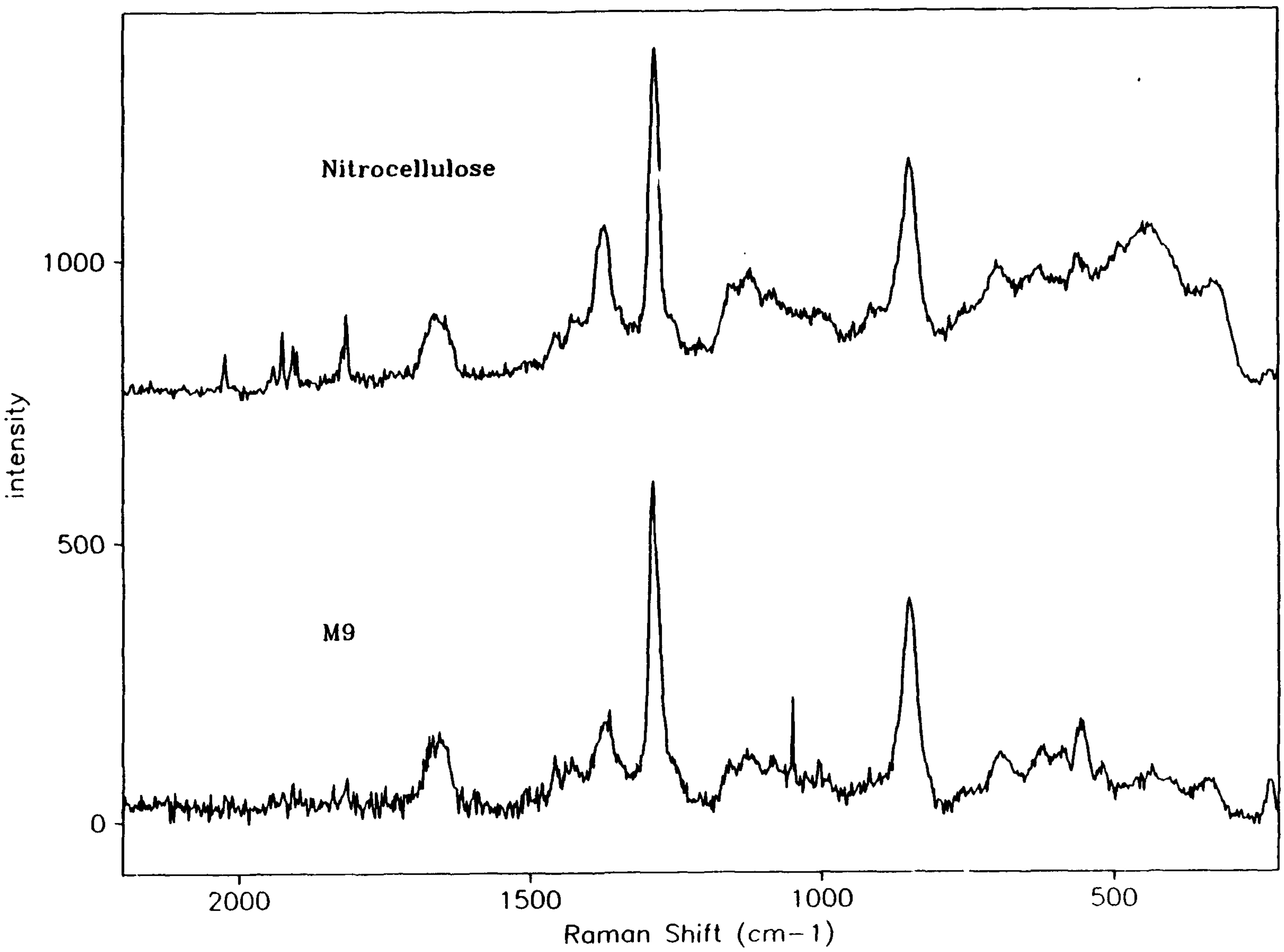

Figure 16. The FTR spectra of nitrocellulose (top) and M9 (bottom).

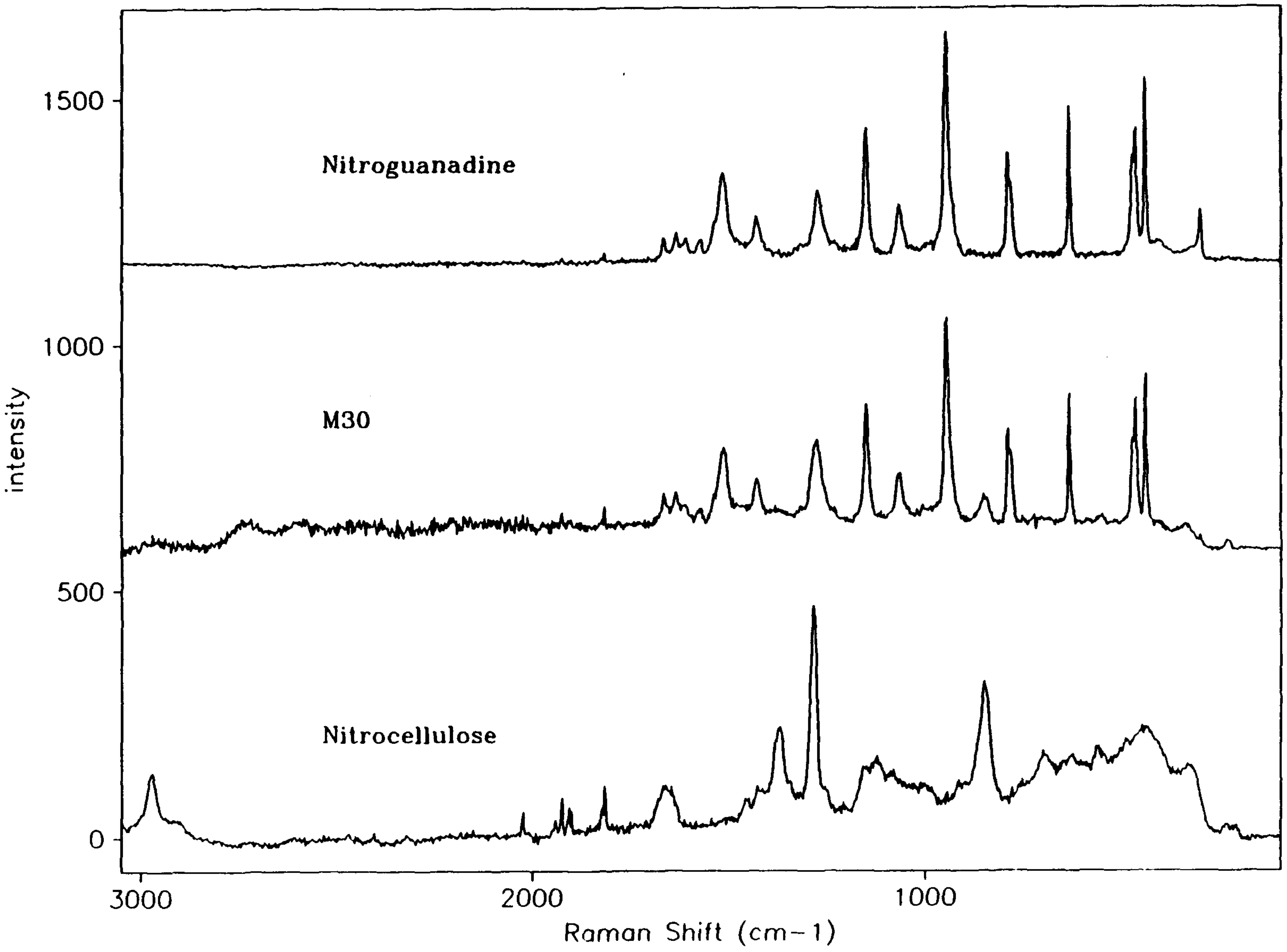

Figure 17. The FTR spectra of nitroguanidine (top), M30 (middle), and nitrocellulose (bottom). The M30 combusted during the data collection.

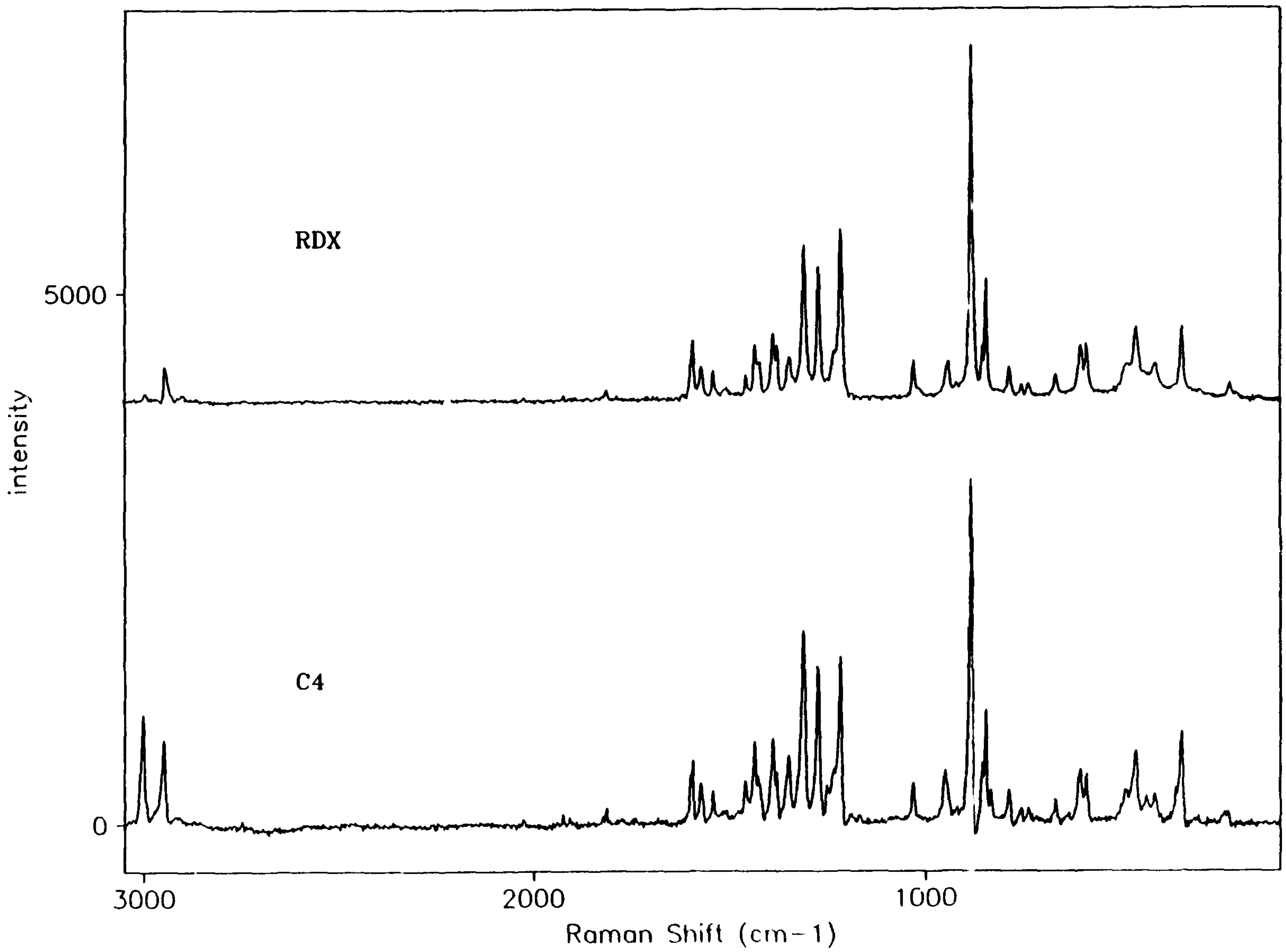

Figure 18. The FTR spectrum of the explosive C4 (bottom) and RDX (top).

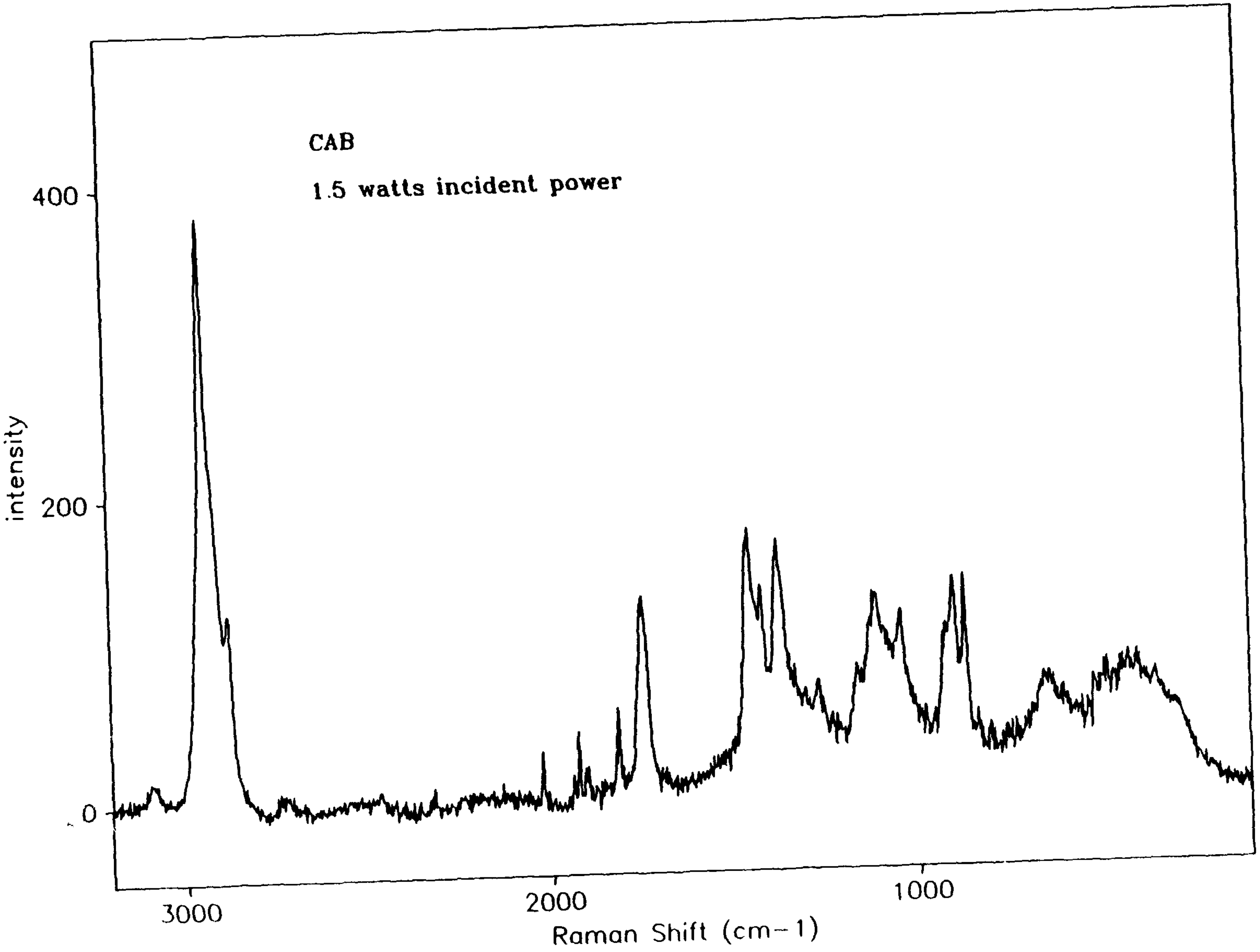

Figure 19. <u>The FTR spectrum of CAB</u>.

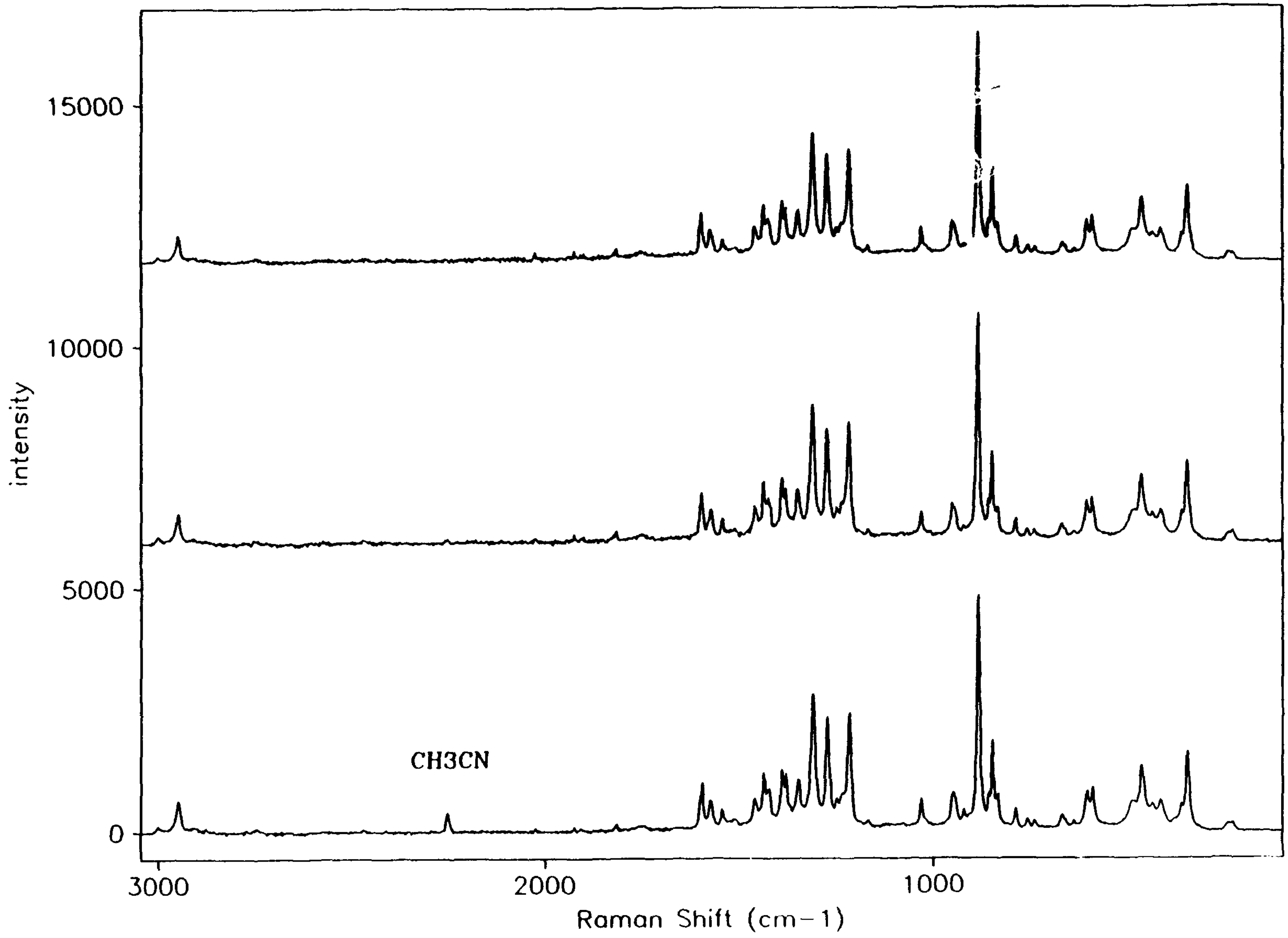

Figure 20. The FTR spectra of a grain of M43: before extraction with supercritical CO_2 modified with CH_3CN (top); interior of grain after extraction (middle); exterior of grain after extraction (bottom).

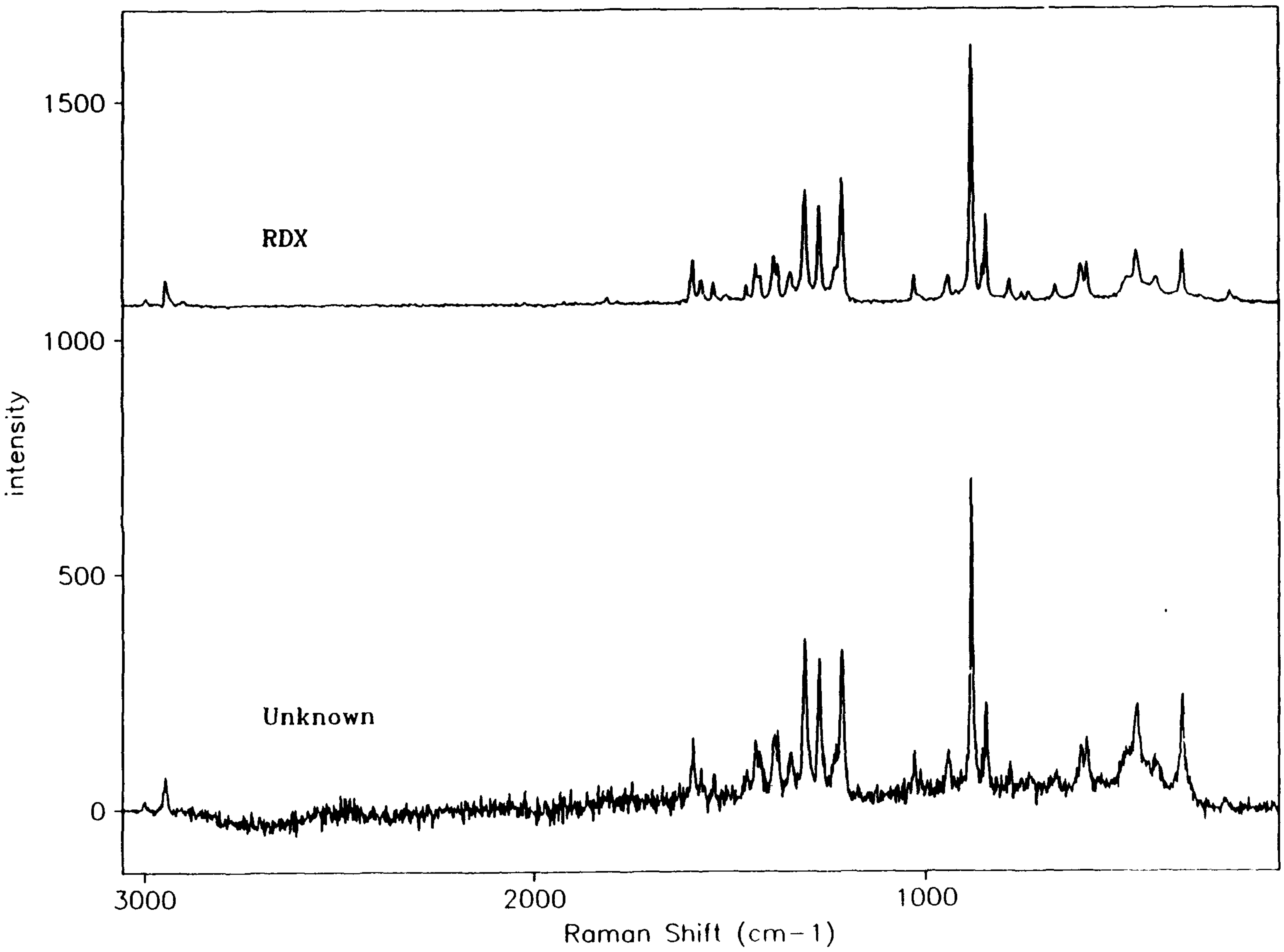

Figure 21. The FTR spectra of RDX (top) and of an unknown sample thought to be a plastic explosive (bottom).

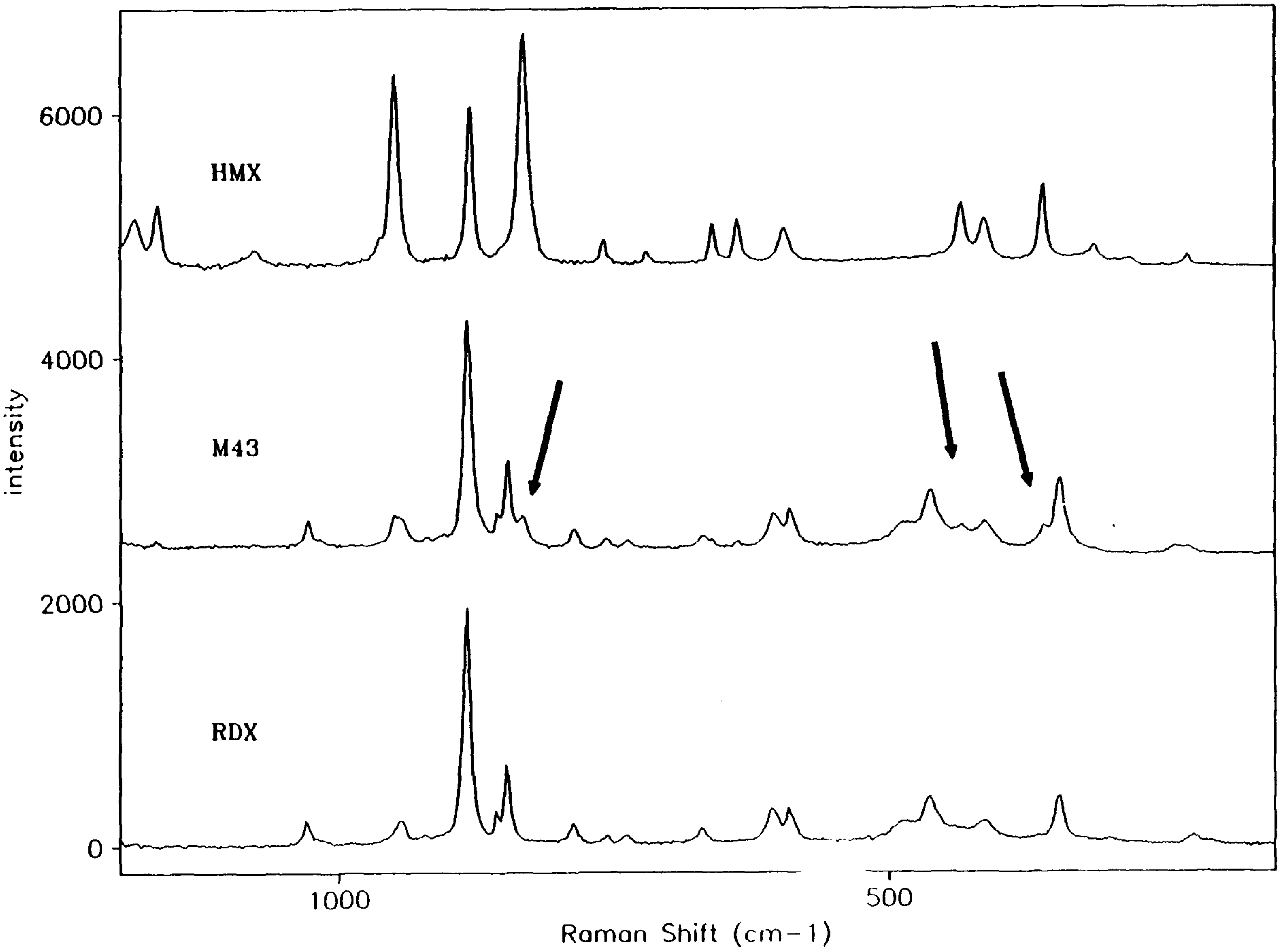

Figure 22. The FTR spectra of HMX (top), RDX (bottom), and the exterior of a grain of M43 (middle). The arrows in the middle spectrum indicate features attributable to HMX impurities in domestically produced RDX.

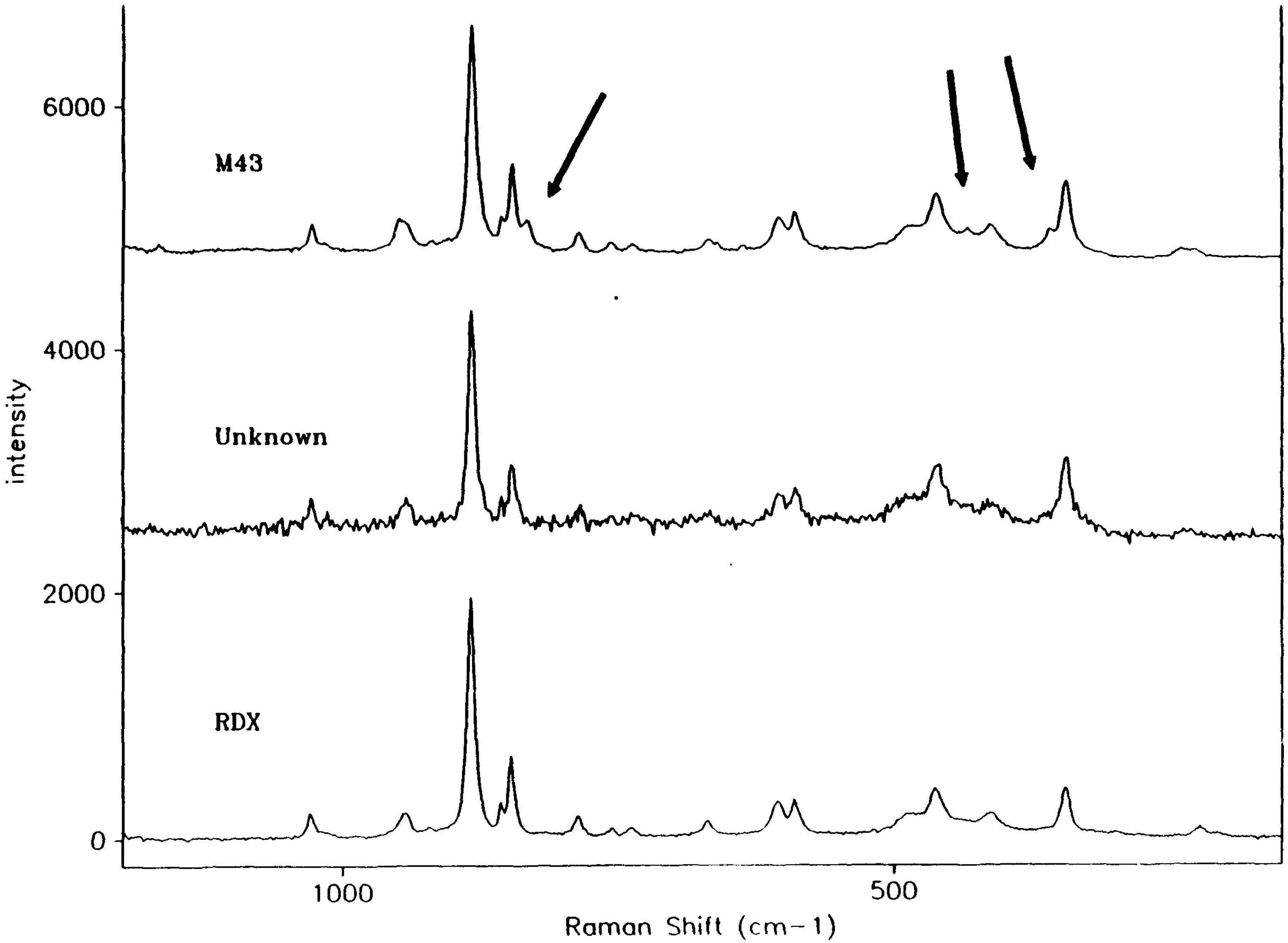

Figure 23. The FTR spectra of the exterior of a grain of M43 (top), an unknown sample thought to be a plastic explosive (middle), and RDX (bottom). Arrows in the top spectrum indicate HMX impurities in domestically produced RDX. Middle spectrum does not show any features attributable to HMX.

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