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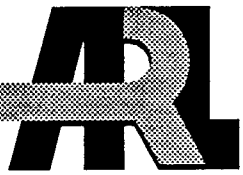
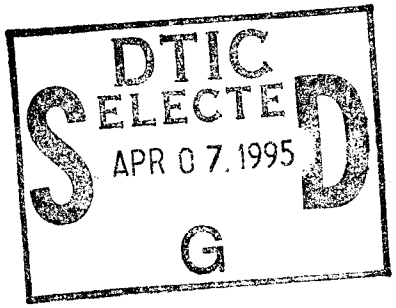


Fourier Transform (FT) Raman Spectroscopy of Nitrogen at High Pressure

K. L. McNesby
J. B. Morris

ARL-TR-718 March 1995



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13. ABSTRACT *(Maximum 200 words)*

Ro-vibrational and rotational Raman spectra of nitrogen gas at 300 K over the pressure range 3 MPa to 24 MPa (500 to 3,500 psi) have been measured using a Fourier transform (FT) spectrometer employing 1,064 nm laser radiation as the scattering source. It is shown that the pressure-induced narrowing and peak shift of the Raman spectrum of N_2 observed in these experiments agrees within experimental error with data obtained by other investigators using different techniques.

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1. INTRODUCTION

The narrowing of ro-vibrational Raman spectra of some gases with increasing pressure was first observed for CO in 1959 (May 1959) and for N_2 and O_2 a few years later (Mikhailov 1964; Varghese 1967). Theoretical explanations of the narrowing of ro-vibrational Raman spectra with increasing pressure for some species also appeared around this time (Alekseev and Sobel'man 1968; Fiutak and Van Kranendonk 1963). Alekseev and Sobel'man (1968) showed that molecular collisions may lead to the narrowing of a group of lines in a manner similar to that previously observed for nuclear magnetic resonance spectra (Bloembergen and Pound 1948). These early experiments used the mercury vapor line at 404.7 nm as the scattering source (May, Stryland, and Varghese 1970).

With the advent of lasers as scattering sources, more detailed studies of the effect of sample gas density on Raman scattering became possible (Wang and Wright 1973). The development of nonlinear scattering techniques (CARS, four-wave mixing [Hall and Eckbreth 1981; Hall, Verdieck, and Eckbreth 1980; Roland and Steele 1980]) and the potential applications to combustion diagnostics brought renewed interest in the narrowing of ro-vibrational Raman spectra observed for some species at elevated pressure (Hall and Eckbreth 1981; Hall, Verdieck, and Eckbreth 1980; Roland and Steele 1980).

We report here the first study of Raman spectra of nitrogen at elevated pressure using a Fourier transform (FT) spectrometer employing Nd:YAG laser radiation at 1,064 nm as the scattering source (hereafter referred to as FT-Raman spectroscopy). We have found that ro-vibrational Raman spectra of N_2 measured using FT-Raman spectroscopy over the pressure range from 3 to 24 MPa (500 to 3,500 psi) agree within experimental error with data collected by other methods over similar pressure regions (May, Stryland, and Varghese 1970; Roland and Steele 1980; Wang and Wright 1973).

2. EXPERIMENTAL

The Raman spectrometer system used in this experiment has been described in detail in McNesby, Wolfe, Morris, and Pesce-Rodriguez (1994). Briefly, the system consists of a Bomen/Hartmann & Braun Inc. Model DA-8 FT spectrometer to which a Raman accessory has been added. Laser radiation from a Quantronics Model 100 Nd:YAG laser focused onto the sample cell containing high pressure nitrogen. Back-scattered radiation is focused onto the entrance port of the FT spectrometer. The scattered radiation

is filtered (to minimize the Rayleigh line) before and after modulation by the interferometer, and the modulated radiation is detected using a liquid nitrogen cooled Indium-Gallium-Arsenide detector.

Power incident on the sample was 1 W. Laser cavity apertures were adjusted to give the "best" Gaussian beam profile. The full width at half maximum of the Nd:YAG laser was approximately 0.4 cm^{-1}. Although an effort was made to achieve single mode operation, it was found that for our system, the best beam shape was achieved when the laser was running multimode. No polarizing filters were used in the experiment reported here.

The sample cell is a Harrick Corp. high pressure gas cell. The cell has an internal volume of 0.3 cm^3 and is equipped with 3-mm-thick sapphire windows. Gas pressure is maintained by an Isco Corp. Model 50 syringe pump. Ultra high purity nitrogen supplied by Matheson, Inc. was used without further purification.

The Q-branch spectra were measured at 0.5 cm^{-1} resolution for 100 coadded scans. The pure rotational spectra were measured at 1 cm^{-1} resolution for 500 coadded scans. Spectra were zero-filled prior to being Fourier transformed to minimize any error in peak shape or intensity caused by instrument resolution. The maximum error in linewidths reported here is estimated to be ± 0.125 cm^{-1}. This estimate is based on a FT spectrometer operating at 0.5 cm^{-1} resolution (prior to zero-filling) reporting intensity every 0.25 cm^{-1}. All spectra were recorded with the sample gas at room temperature (300 K ± 5 K).

3. RESULTS AND DISCUSSIONS

Figure 1 shows the Q-branch of the Raman spectrum of nitrogen from 10 to 24 MPa (1,500 to 3,500 psi) measured at a resolution of 0.5 cm^{-1}. Two features may be distinguished. First, there is a red shift of the peak of the Q-branch with increasing pressure. Second, and less noticeable, the full-width at half maximum (FWHM) of the Q-branch decreases with increasing pressure.

Figure 2 shows the FWHM for the Q-branch of the Raman spectrum of nitrogen as a function of pressure, along with data obtained at similar pressures using other Raman techniques (e.g., CARS [Hall and Eckbreth 1981; Hall, Verdieck, and Eckbreth 1980; Roland and Steele 1980], Hg lamp excitation

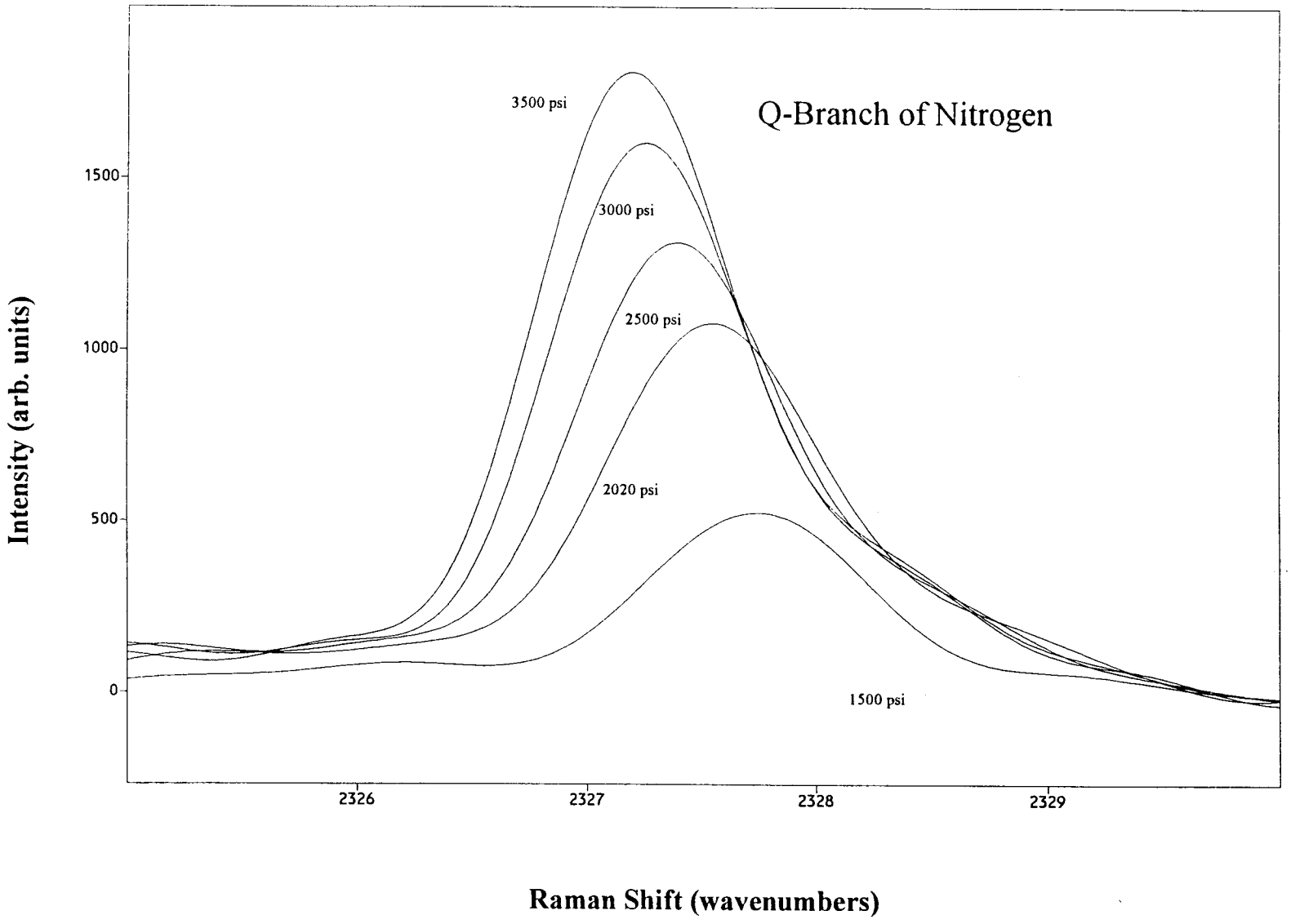

Figure 1. The FT-Raman spectra of nitrogen from 10 MPa (1,500 psi) to 24 MPa (3,500 psi) at 300 K. Incident laser power was 1 W.

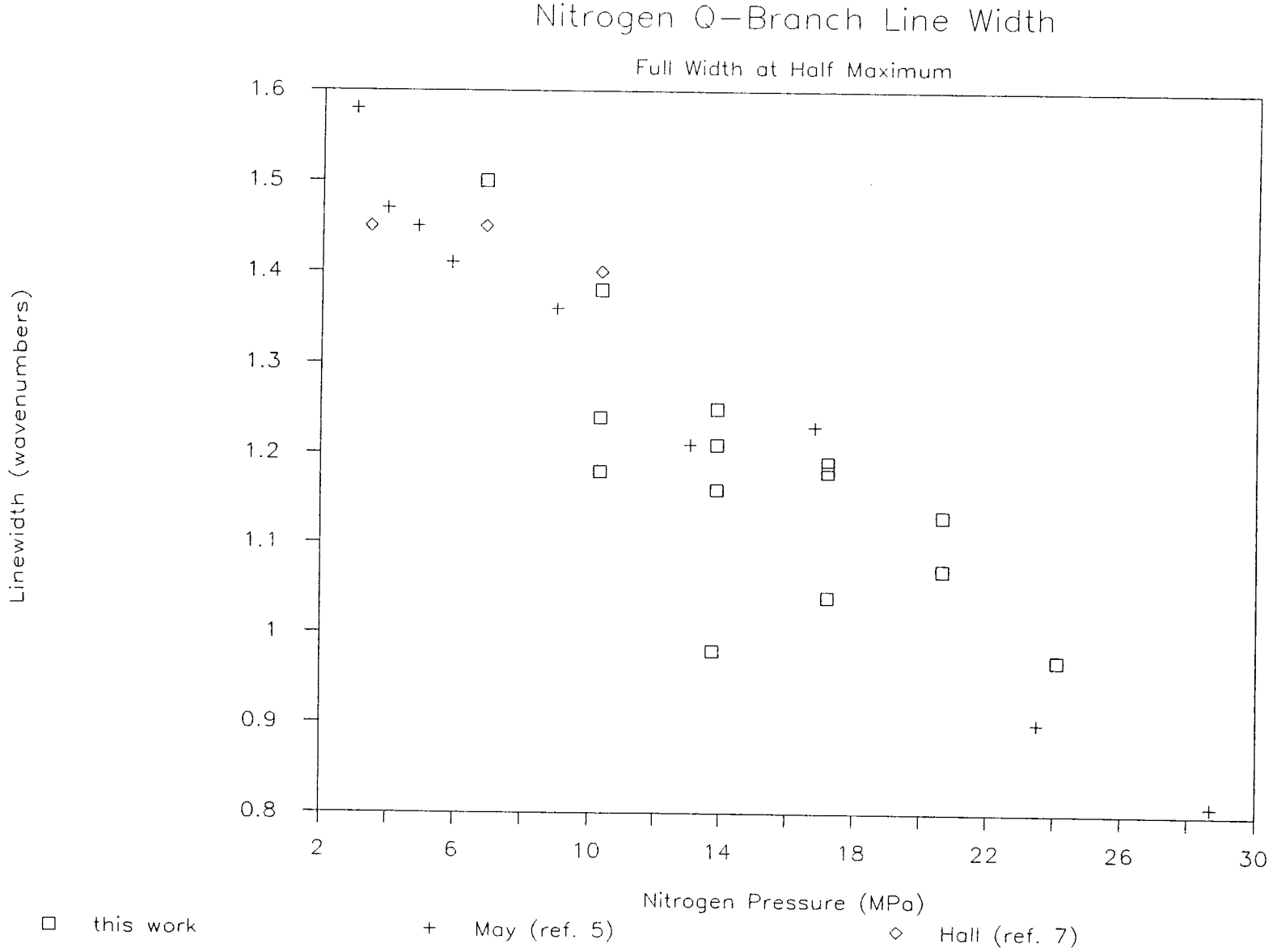

Figure 2. The change in FT-Raman Q-branch linewidth (full width at half maximum) for nitrogen over the pressure range studied in these experiments. Maximum error is estimated to be +/–0.125 cm^{-1}

(May, Stryland, and Varghese 1970). As seen from Figure 2, the present results agree well with those obtained by other investigators, including those techniques (e.g., CARS) for which the dependence on electric susceptibility is nonlinear.

Figure 3 shows the peak of the Raman shift for the Q-branch over the pressure range studied. The best linear fit through these data is

$$(\text{Raman shift } [\text{cm}^{-1}]) = -0.0495*(\text{pressure in MPa}) + 2{,}328.138 \text{ cm}^{-1}.$$

The value for the Q-branch peak extrapolated to zero pressure (2,328.138 cm^{-1} [$\pm$0.066 cm^{-1}]) agrees well with the value obtained by using Ar laser excitation (2,328.2 cm^{-1}) (Wang and Wright 1973).

The shift and narrowing of the Q-branch occurs because rotationally inelastic collisions approach a frequency comparable to that of the separation of the individual transitions that comprise the Q-branch. The rotation-vibration interaction, normally dependent on $e_v J(J + 1)$ in which e_v is the rotation-vibration interaction constant and J is the rotational quantum number, is replaced by a value indicative of the mean rotational state (May, Stryland, and Varghese 1970).

Figure 4 shows a portion of the pure rotational spectrum of nitrogen at 17 MPa (3,000 psi) obtained using FT-Raman spectroscopy. The portion of the spectrum shown is that covered by the short pass filter supplied by the instrument manufacturer (Bomen/Hartmann & Braun, Inc.). This spectrum has been corrected for instrument and filter response functions. The Raman scattering cross sections for the pure rotational transitions are larger than the scattering cross sections for the O- and S-branches of the ro-vibrational transitions (Bendtsen 1974) because the scattering intensity is dependent on the fourth power of the frequency of the scattered radiation (Herzberg 1950). This may be seen by the absence of O- and S-branch transitions in Figure 1, even though the spectra shown in Figures 1 and 4 were collected using the same incident laser power. The intensity of the Q-branch transition (relative to the O- and S-branches) in the ro-vibrational Raman spectrum is also a manifestation of the so-called J-dependent trace-scattering (Herzberg 1950) term in the expression for Q-branch line strength.

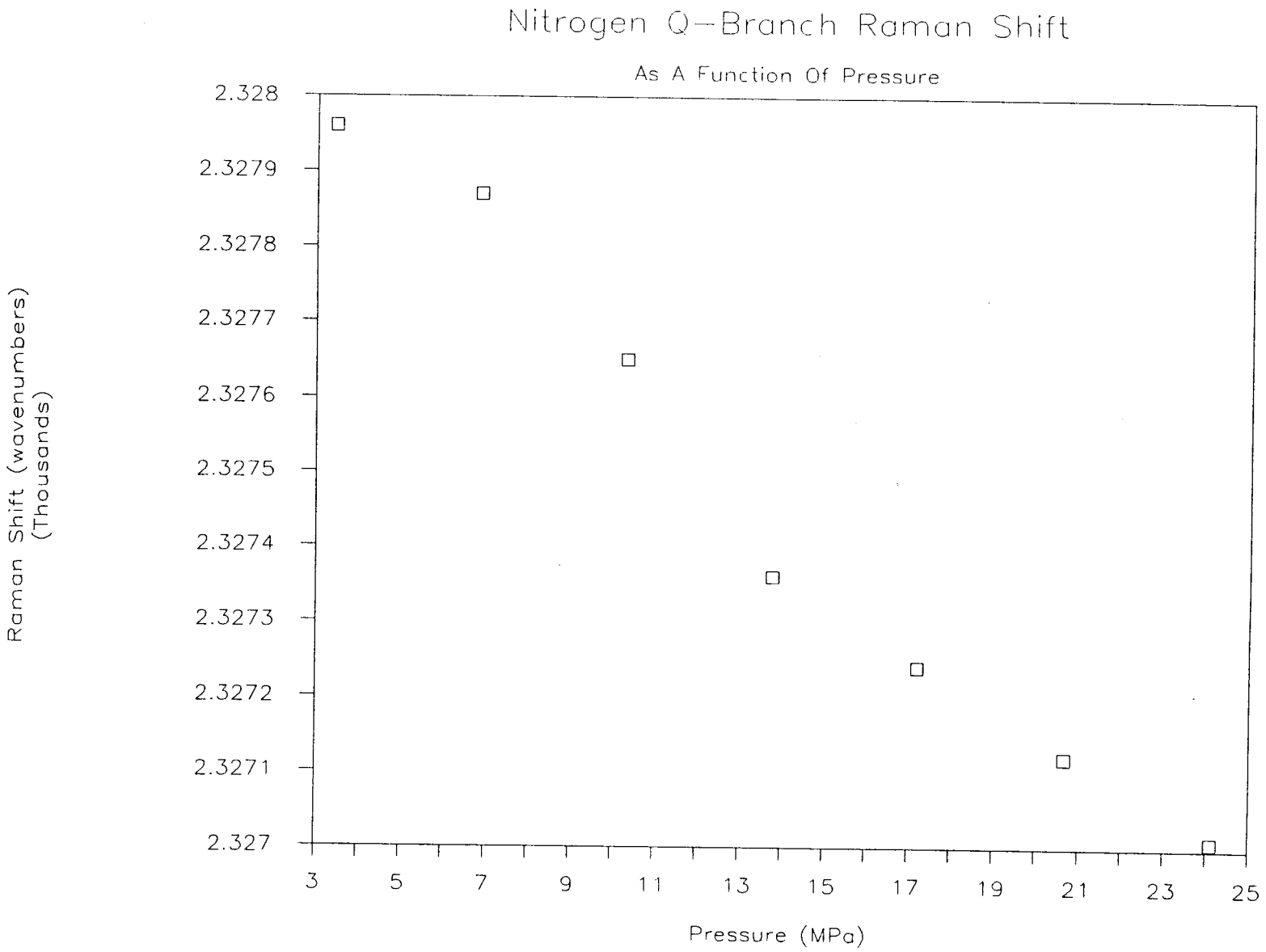

Figure 3. The shift in the peak position of the Q-branch of the FT-Raman spectra of nitrogen as a function of pressure at 300 K

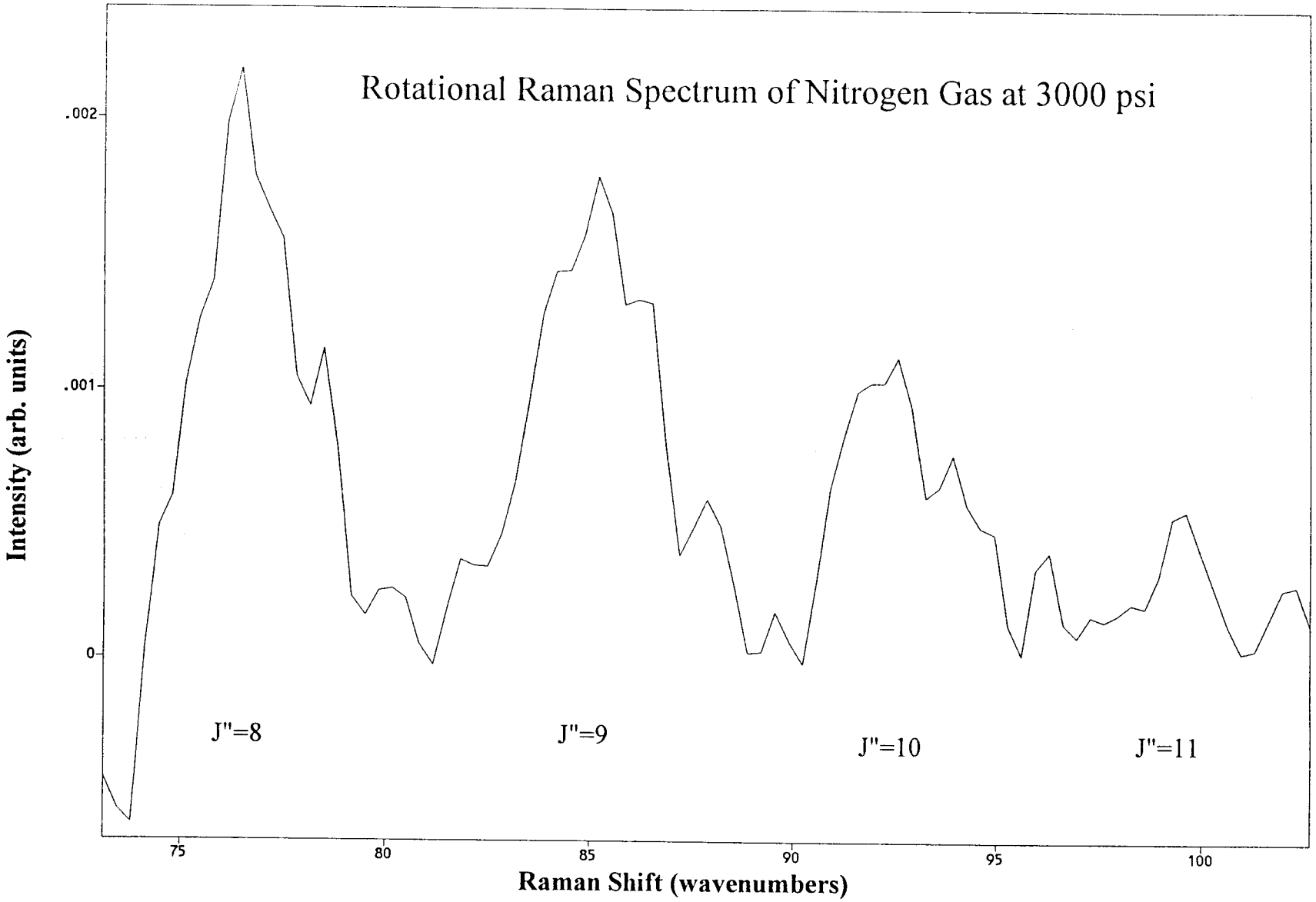

Figure 4. A portion of the pure rotational FT-Raman spectrum of nitrogen at 21 MPa (3,000 psi) and 300 K. "J" values indicate the rotational level from which the transition occurs

For FT-Raman spectroscopy, collecting high-quality rotational or ro-vibrational FT-Raman spectra of nitrogen below 500 psi is difficult. It should be noted that Raman spectra of nitrogen using higher frequency light sources (e.g., the Ar laser line at 514.5 nm) routinely show well resolved O- and S-branches (Bendtsen 1974).

4. CONCLUSION

It has been shown that motional narrowing of the Raman Q-branch in high pressure nitrogen is observed using a FT spectrometer when the scattering radiation is at a wavelength of 1,064 nm. The narrowing and shifting of the Q-branch transition observed in these experiments agrees within experimental error with measurements obtained by other workers using Ar laser excitation and using nonlinear (e.g., CARS) techniques. We are presently extending FT-Raman spectroscopy to the study of supercritical fluids.

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