

# Pressure Calibration by the Infrared Absorption Spectrum of Mineral Oil

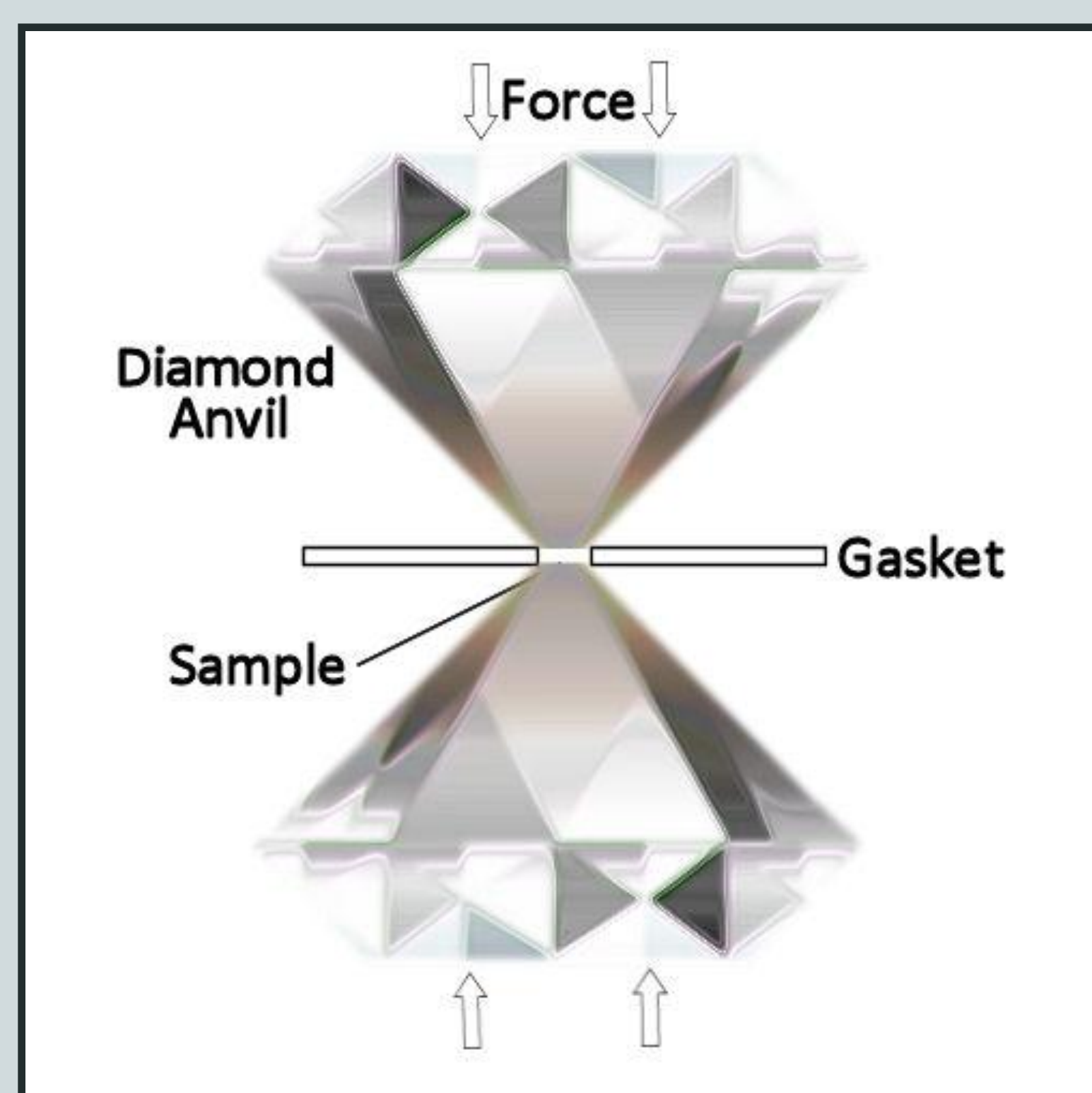
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## Introduction

The diamond anvil cell places large pressures on small samples. An infrared pressure calibration would be useful for vibrational spectroscopy of solids under pressure.



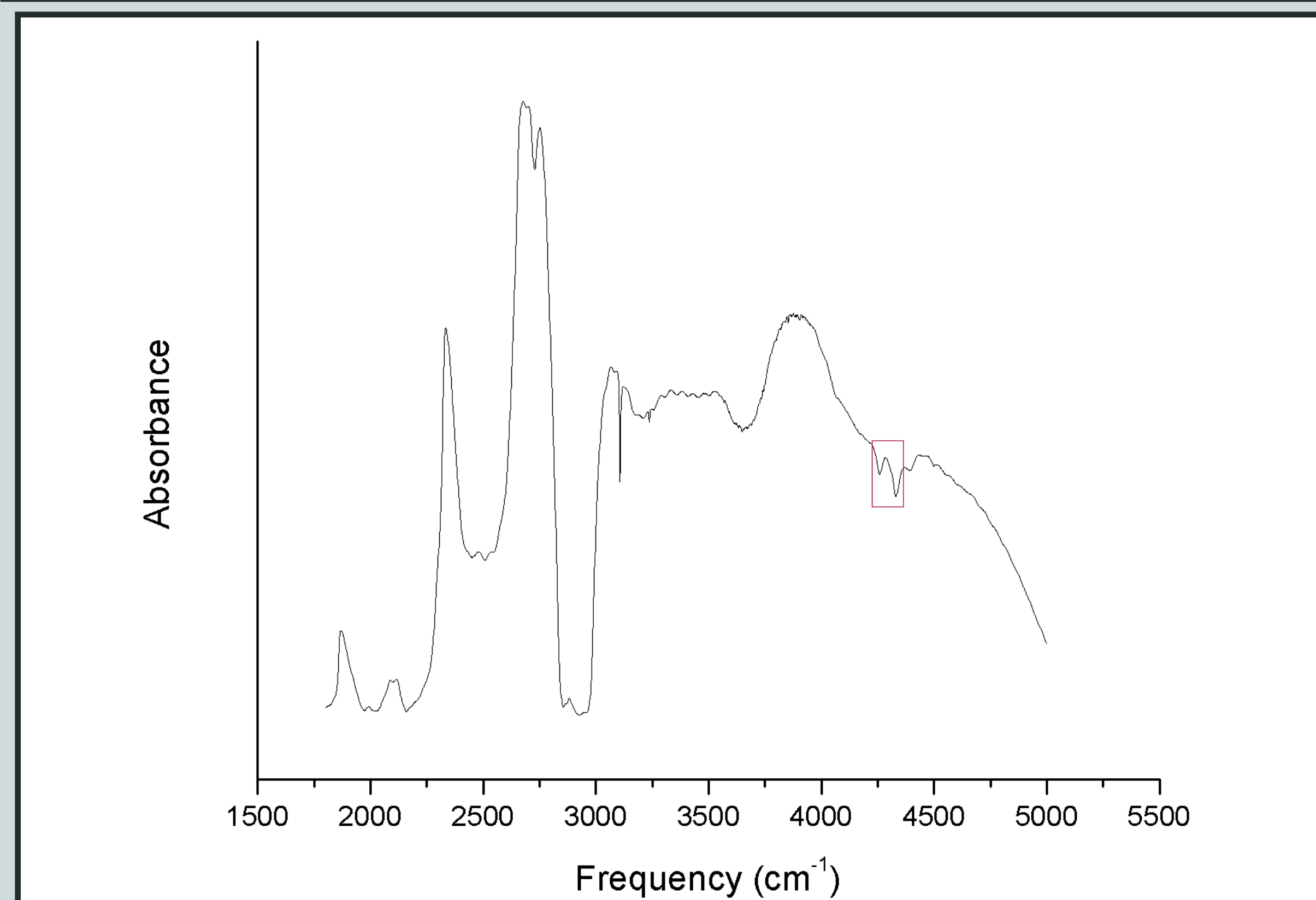
Ruby fluorescence is the most common technique used to measure pressures with more accuracy. When a ruby is placed under pressure, a peak in its red fluorescence spectrum shifts to a higher wavelength. The peak location is measured for a reference ruby at atmospheric pressure and for a ruby chip included in the cell, and the difference in wavelength is used to calculate the pressure within the cell.

A similar effect is seen in the infrared absorption spectrum of mineral oil. The frequency of two peaks increases as pressure is applied to the mineral oil.

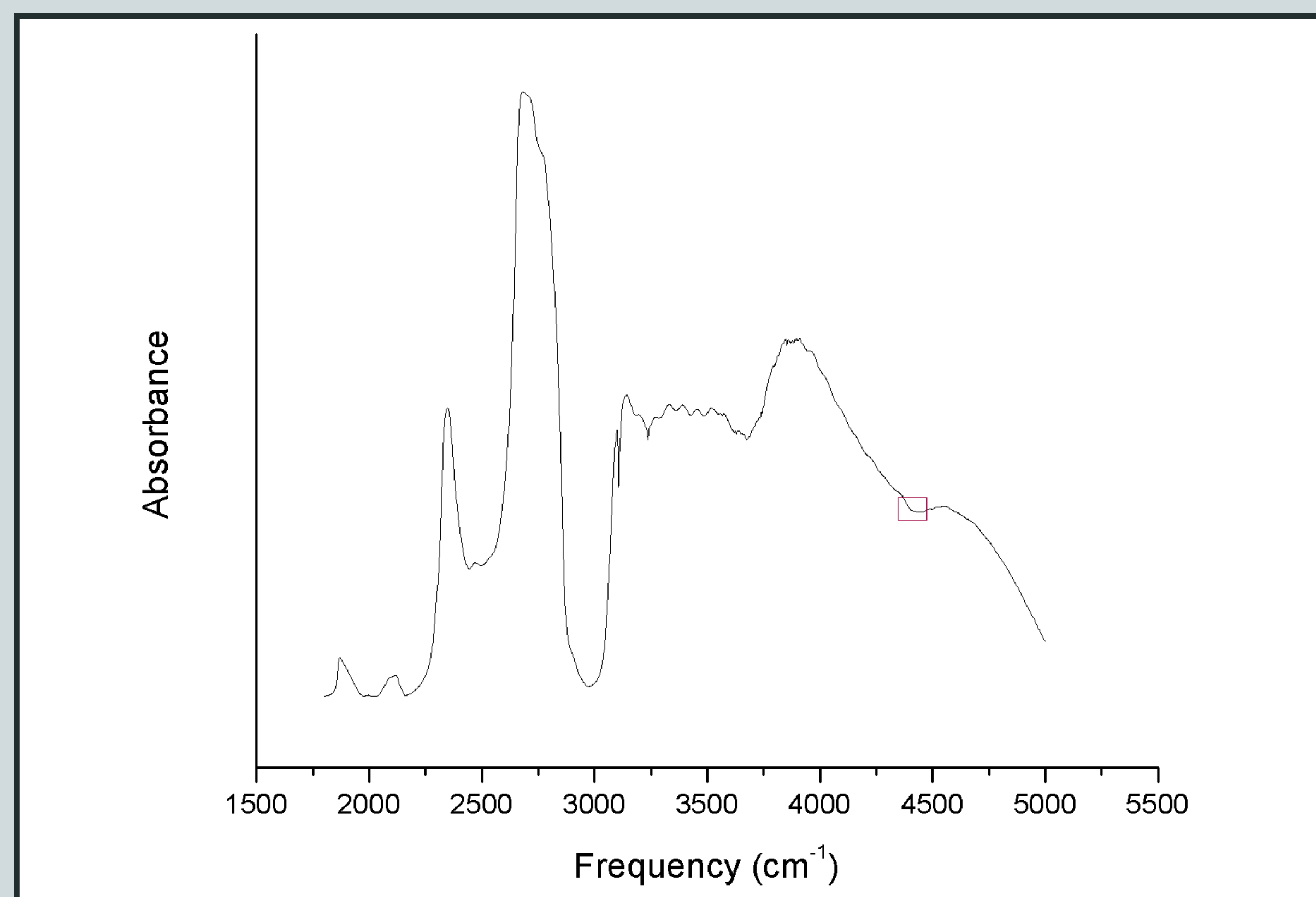
## Method

A diamond anvil cell was loaded with a sample of mineral oil (Fluka brand mineral oil for IR spectroscopy, density 0.85 g/mL at 20 °C) and a ruby chip. Moissanite anvils were used with most of the samples at lower pressures (less than 15 kbar), but all higher pressures were placed on the samples by diamond anvils. The pressure on the sample was calculated by the ruby fluorescence method, and a Fourier transform infrared spectrometer was used to measure the absorption spectrum of the sample between 1800 and 5000 wavenumbers. The pressure was then increased by several thousand atmospheres, and the spectrum was taken again. When pressure reached about 60 kbar, it was slowly decreased and spectra were again taken every several thousand atmospheres.

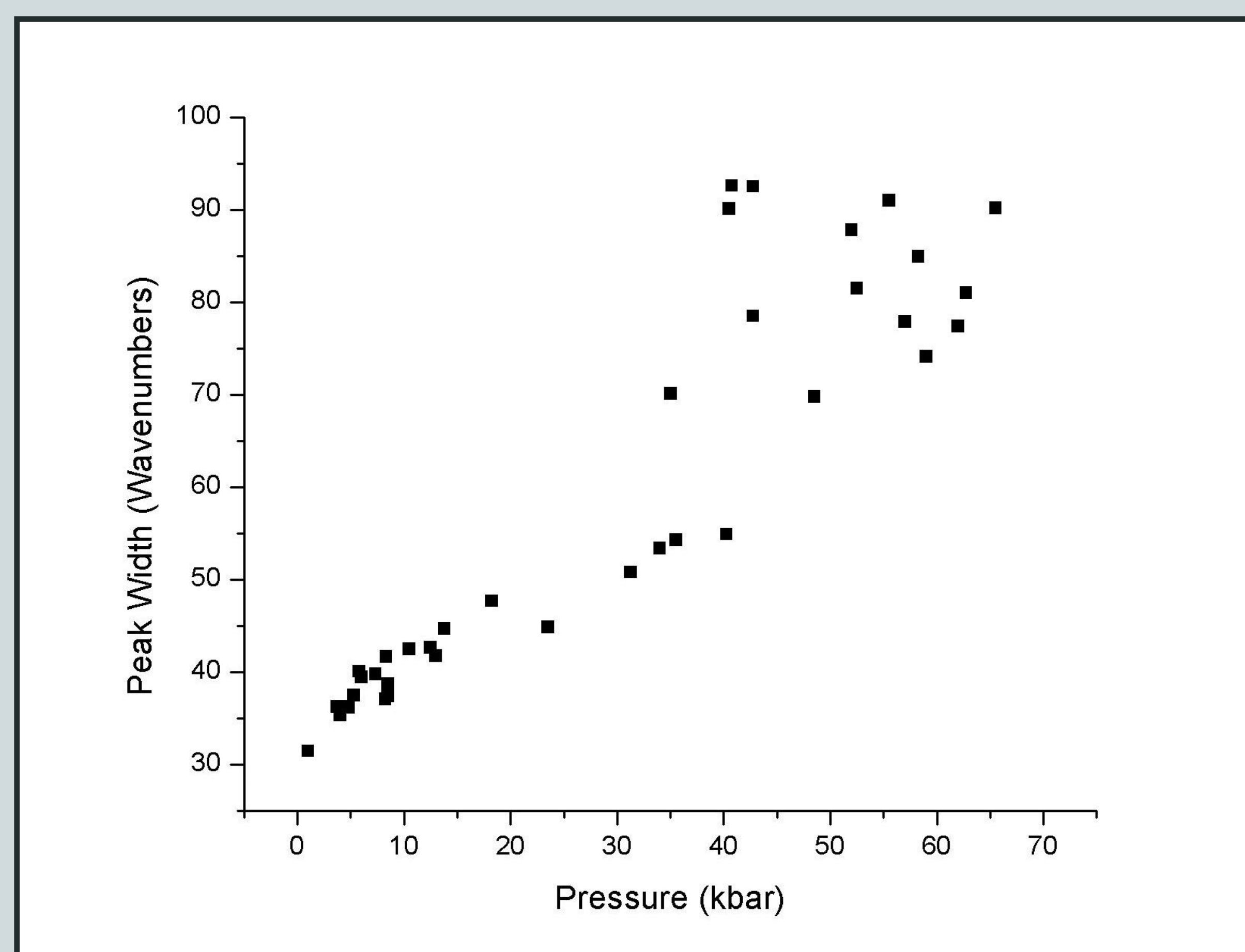
At a pressure of about 1 kbar (100 MPa), both peaks are distinct:



At 65 kbar (6500 MPa), the upper peak is much more broad, but still present:



Both peaks broaden with increasing pressure, though there is significant uncertainty at high pressures:



## Results

The position of the upper peak in each spectrum was plotted as a function of pressure. A linear relationship was found between the pressure in the cell and the location of this peak.

The frequency ( $\nu$ , in wavenumbers) where the peak is found can be calculated from pressure ( $P$ , in kbar) by the equation

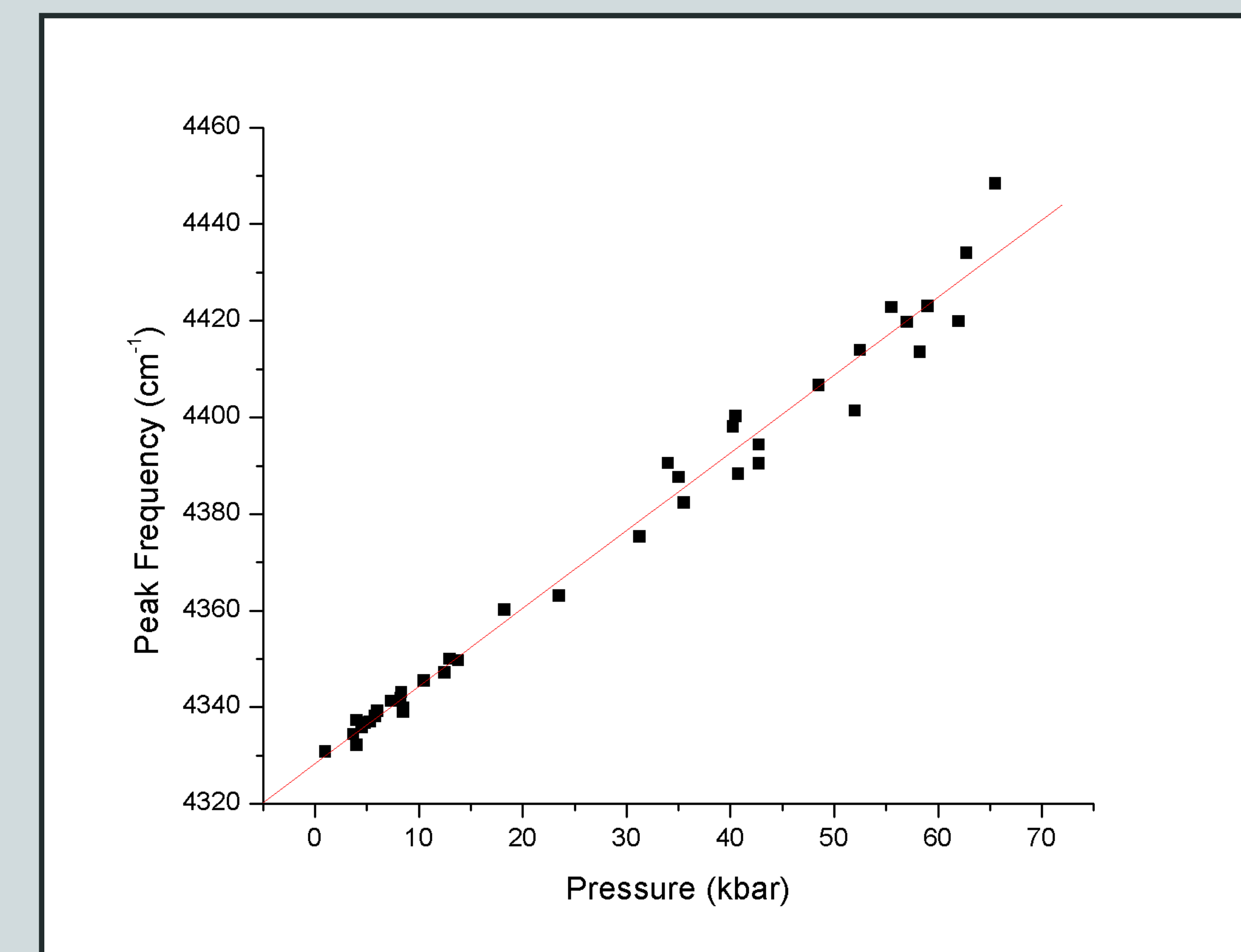
$$\nu = a + bP$$

Where

$$a = [4328 \pm 1] \text{ cm}^{-1}$$

$$b = [1.61 \pm 0.03] \text{ cm}^{-1}/\text{kbar}$$

The linear fit is best at relatively low pressures, but the relationship appears to hold up to the highest pressure where data was taken, about 65 kbar.



The position of this peak in the infrared absorption spectrum of mineral oil appears to be a reasonably accurate indicator of the pressure in the cell. More data are needed to improve the equation and the accuracy of calculations. By observing the location of the peak in many more spectra to better establish the linear relationship, it should be possible to determine the pressure on diamond anvil cell samples that contain mineral oil, without the need to include a ruby chip.

## References

Piermarini, G. J., S. Block, J. D. Barnett, and R. A. Forman. "Calibration of the pressure dependence of the R1 ruby fluorescence line to 195 kbar." *Journal of Applied Physics* 46.6 (1975): 2775-80.