



Experimental Raman Study of N-phenyl-3-para Nitrophenyl Isoxazolidine-5-Carbonitrile

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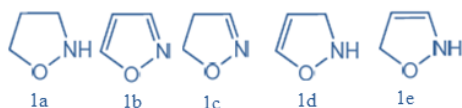
Isoxazolidine
Raman spectroscopy
Vibrational mode

Abstract:

N-phenyl-3-para nitrophenyl isoxazolidine-5-carbonitrile compound has been investigated by Raman spectroscopy at room temperature. Raman spectra have been measured at 647.5 nm excited laser lines in the frequency range 150–4000 cm^{-1} . The wave numbers, widths, and intensities of the Raman lines were determined by fitting using the LabSpec5 software with a combined Lorentzian–Gaussian band shape. The error range for the determination of the peak position was ± 0.5 after the accumulation of the data and the fitting procedure. The Raman spectrum is characterized by a series of bands in the 150–4000 cm^{-1} spectral range.

1. Introduction

Organic materials are frequently characterized by their various chemical properties, which could lead to technological innovations. Nitrogen containing heterocycles with an oxygen atom are considered as an important Class of compounds in medicinal chemistry because of their diversified biological applications. Isoxazolidine (1a) is a five membered heterocyclic compound containing oxygen and nitrogen atoms in the 1, 2 positions, it is the saturated relative of Isoxazole (1b); its partially saturated analogs are called isoxazolines (1c-e).



Isoxazolidines are very useful organic compound because of their application as medicines or insecticides; they are known to possess biological activities as antibacterial [1], anti-HIV [2], antifungal [3], anti-diabetic [4], etc. They form important families of heterocycle, as simple heterocycle or forming part of complex structures.

Besides, they provide a convenient path for the synthesis of natural base and natural compound containing N and O atoms in the molecules. Thus, they can be used as intermediates to synthesize natural base [5].

The vibrational spectroscopy applied to organic materials is one of the most broadly applied techniques for the identification and characterization of molecules, as well as for the determination of molecular structures [6-8]. Raman spectroscopy frequently offers additional information about molecular vibrations.

The purpose of this paper is to investigate by Raman spectroscopy the vibrational properties of N-phenyl -3-para nitrophenyl isoxazolidine-5-carbonitrile compound and determines reasonable vibrational frequencies. The various bands were attributed to their respective vibrational mode.

2. Experimental Details

Raman scattering investigations were performed using as excitation the 647.5 nm radiation of an Ar–Kr laser, and a T64000 multichannel Raman spectrometer (Horiba–Jobin–Yvon) equipped with

a cooled CCD detector. The laser power in the samples was limited to 7 mW. The spectra were measured from 150 to 4000 cm^{-1} with a 500 tr/mm grating and 200 s exposition times. The resolution (FWHM) was 2.1cm^{-1} . The measurements were made under a microscope in the backscattering geometry using an X-50 LF objective, on the (001) natural face of single crystals (z-axis) in z(xy)z polarization, where y stands for a birefringence direction.

The wave numbers, widths, and intensities of the Raman lines were determined by fitting using the LabSpec5 software with a combined Lorentzian–Gaussian band shape. The error range for the determination of the peak position was $\pm 0.5\text{ cm}^{-1}$ after the accumulation of the data and the fitting procedure.

3. Results and Discussions

3.1. Geometry optimization

N-phenyl -3-para nitrophenyl isoxazolidine-5-carbonitrile compound is formed by 35 atoms it has 96 fundamental vibrations; Gaussian view software package [9] was used for the geometry optimization represented in Figure 1.

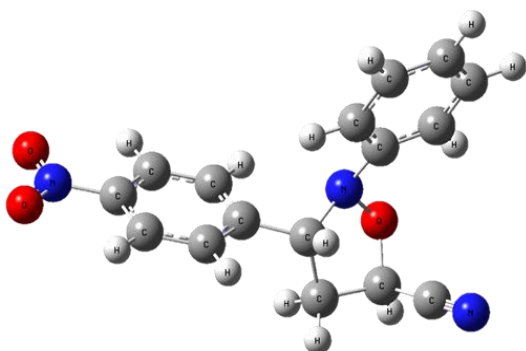


Figure 1. Optimized geometry of N-phenyl -3-para nitrophenyl isoxazolidine-5-carbonitrile compound

The C–C distances in the phenyl group are in the range 1.379 – 1.393 Å and the C–C–C angle values vary between 119.5 and 120.9. In the isoxazolidine the C–C distances are in the range 1.514 – 1.534 Å, the C–C–C angle values is 112.8 and the N–O distance equal to 1.448 Å. The C≡N bond distance equal to 1.137 Å. In the nitro group the N=O bond distance equal to 1.235 Å, and the O–N–O angle values is 123.87.

3.2 Vibrational frequencies

The Raman spectrum is characterized by a series of bands in the 150 – 4000 cm^{-1} spectral range (figure

2), the wave numbers of the Raman lines were determined by fitting using the LabSpec5 software with a combined Lorentzian–Gaussian band shape. (figure 3) shows the deconvolution of the Raman spectrum in the frequency range 150–4000 cm^{-1}

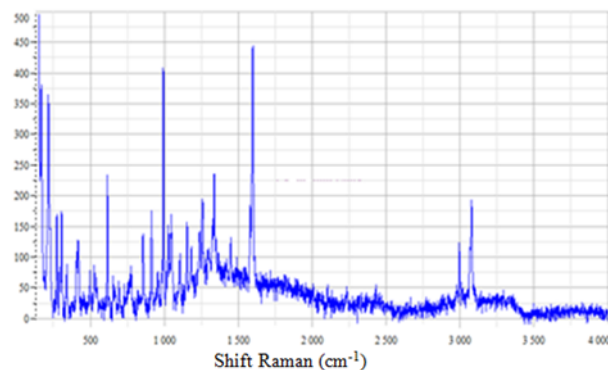


Figure 2. Raman spectrum of N-phenyl -3-para nitrophenyl isoxazolidine-5-carbonitrile compound

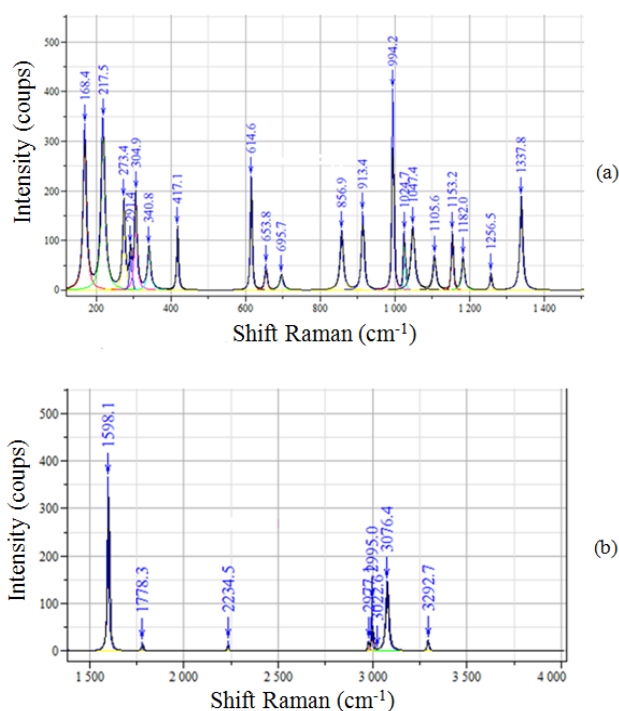


Figure 3. Deconvolution of the Raman spectrum in the frequency range (a), 150–1500 cm^{-1} ; (b), 1500–4000 cm^{-1}

3.2.1. C–H vibrations

The asymmetric and symmetric C–H stretching bands appear in the 3000–3200 cm^{-1} range, the in-plane C–H stretching bands in the 1000–1375 cm^{-1} range and the out of- plane C–H stretching bands in the 650–900 cm^{-1} range [10, 11]. Regarding the

experimental results, in the Raman spectrum the C–H stretching bands appeared in the 3022.6–3292.7 cm^{-1} range, in-plane and out-of-plane C–H stretching bands were observed in the 1024.7–1337.8 cm^{-1} and 614.6–994.2 cm^{-1} ranges, respectively.

3.2.2. C–C vibrations

C–C ring stretches in aromatic compounds appear in the 1400–1600 cm^{-1} region [9, 10]. In the experimental Raman spectrum, C–C ring stretches were assigned to 1598.1 cm^{-1} .

3.2.3. C \equiv N vibrations

A particular band at 2234.5 cm^{-1} characterizes the C \equiv N stretching vibration in the experimental Raman spectrum.

4. Conclusions

In this work, N-phenyl -3-para nitrophenyl isoxazolidine - 5- carbonitrile compound has been investigated by Raman spectroscopy and Raman spectra have been studied. The Raman spectrum is characterized by a particular band at 2234.5 cm^{-1} which was assigned to the C \equiv N stretching vibration. C–C ring stretches were assigned to 1598.1 cm^{-1} . All the results are in a fairly good agreement with the predictions.

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References

- [1] D.P.A. Iannazzo, V. Pistara, A. Rescifina and R. Romeo. *Tetrahedron*. 58, 581-587 (2002). Doi: 10.1016/s0040-4020(01)01161-9
- [2] B. Loh, L. Vozzolo, B. J. Mok, C.C. Lee, R.J. Fitzmaurice, S. Caddick, , A. Fassati. *Chem. Biol. Drug. Des.*, 2010, 75, 461-474. DOI: 10.1111/j.1747-0285.2010.00956.x
- [3] M. P. Sadashiva, H. Mallesha, N. A. Hitesh, K. S. Rangappa. *Bioorg. Med. Chem.*, 2004, 12, 6389-6395. DOI: 10.1016/j.bmc.2004.09.031
- [4] C. Parmeggiani, D. Martella, F. Cardona, A. Goti. *J. Nat. Prod.* 2009, 72, 2058-2060. DOI: 10.1021/np900435d
- [5] Éva Frank, Zoltán Mucsi, Mihály Szécsi, István Zupkó, János Wölfling and Gyula Schneider, *New J. Chem.*, 2010, 34, 2671–2681. DOI: 10.1039/C0NJ00150C
- [6] J. Neugebauer, M. Reiher, C. Kind, B.A. Hess, J. *Comput. Chem.* 23 (2002) 895–910.

- DOI: 10.1002/jcc.10089
- [7] K. Karoui, M. Ben Bechir, A. Ben Rhaïem, A. Bulou, F. Calvayrac, K. Guidara, *Phase Transit.* 87 (2014) 613–628. Doi.10.1080/01411594.2013.879588
 - [8] A. Oueslati, A. Bulou, F. Calvayrac, K. Adil, M. Gargouri, F. Hlel, *VibSpectrosc* 64 (2013) 10–20. DOI: 10.1016/j.vibspec.2012.10.002
 - [9] R. Dennington, T. Keith, J. Millam, *Gaussview Ver. 5*, Semichem, Shawnee Mission, KS, 2009.
 - [10] Dana W. Mayo, Foil A. Miller, Robert W. Hannah. *Course notes on the interpretation of infrared and Raman spectra* John Wiley & Sons 2003. DOI: 10.1002/0471690082.ch5
 - [11] Wilson, E. B.; Decius, J. C.; Cross, P. C. *Molecular Vibrations*; McGraw-Hill: New York, 1955. DOI: 10.1149/1.2430134