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FTIR, FTR and UV-Vis Analysis of Carbamazepine

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ABSTRACT

Spectroscopy is the use of absorption, emission, or scattering of electromagnetic radiation by matter to qualitatively or quantitatively study the matter or to study physical processes. Vibrational spectroscopy has significant contributions towards the studies of structure and physico chemical properties of crystals and molecular system. Recently spectroscopic studies of benzene and its derivatives have been motivated by their biological and pharmaceutical importance. This article deals with spectroscopic investigation of Carbamazepine of pharmaceutical and biological interest using FTIR, FTRaman and UV-Vis spectral measurements. To study the structure these pharmaceutical compounds, the FTIR spectrum of the compound has been recorded over the region 4000-400 cm^{-1}. A satisfactory assignment of the fundamental vibrations has been made according to the position, shape, nature and relative intensity. The quality analysis of the chosen compound has been analyzed using UV-Vis spectroscopy.

Keywords: Carbamazepine FTIR, FTRaman and UV-Vis spectroscopy.

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INTRODUCTION

FTIR and Raman spectroscopic methods are being extensively used to identify the structural groups present in a compound [1-4]. Many research suggested an interesting method of assigning group frequencies observed in vibrational spectra. During the course of our investigation on the samples of pharmaceutical active compounds, our attention has been turned towards the Carbamazepine drug. It is an anticonvulsant drug. It is white (or) almost white, crystalline powder, almost odorless, exhibits polymorphism. It is used for controlling certain types of seizures and relieving pain in patients with nerve pain in the face, jaw, tongue or throat. The FTIR and Raman spectra of Carbamazepine have been thoroughly investigated and detailed vibrational band assignments have been made. The assignment of the fundamental frequencies is made on the basis of magnitude and relative intensities of the observed bands. Also the variations of the absorbance of maximum wavelength of the drug subjected to different temperature conditions have been analyzed using UV-Visible spectral measurements. In the present work, Fourier Transform Infra-Red, FT Raman, UV-Visible spectroscopic methods have been employed successfully in the present investigation and the results obtained are discussed.

MATERIALS AND METHODS

The sample of Carbamazepine was procured from a reputed pharmaceutical firm, Chennai, India and used as such. The FTIR spectrum of the compound are recorded over the region on 4000-400 cm^{-1} using ABB-BOOMEN series and UV-Visible spectral measurements have been made using Shimadzu spectrophotometer in the wavelength region 200-4000 nm. at Dr.Ceeal analytical Lab, Chennai. All sharp bands observed in the spectra are expected to have an accuracy of ±1cm^{-1}. The FT Raman spectrum is analyzed in the range 50-3500cm^{-1} at Central Electrochemical Research Institute (CECRI), Karaikudi.

The molecular structure of Carbamazepine and pharmaceutical data are presented in Fig.1 and Table 1 respectively. The FTIR and FT Raman spectra of the compound are presented in Fig.2 and 3 respectively.

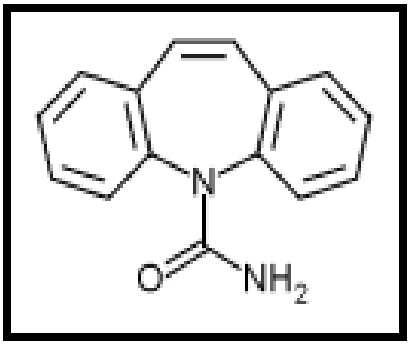

Fig.1 Structure of Carbamazepine

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Table 1: Pharmaceutical data of Carbamazepine

Name	Carbamazepine I.P
Formula	$C_{15}H_{12}N_2O$
Molecular weight	236.27
IUPAC Name	Carbamazepine is 5H-dibenz (b,f) azepine-5-carboxamide
Category	Anti convulsant
Dose	200mg daily
Description	White, Crystalline powder, Odorless
Solubility	Freely soluble in dichloromethane, sparingly soluble in ethanol (95%) and in acetone, insoluble in water
Storage	Store in well closed containers

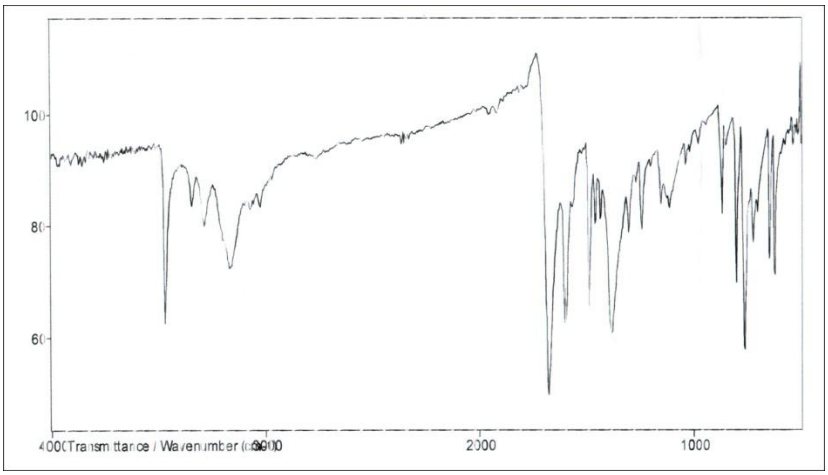

Fig.2 FTIR spectrum of Carbamazepine

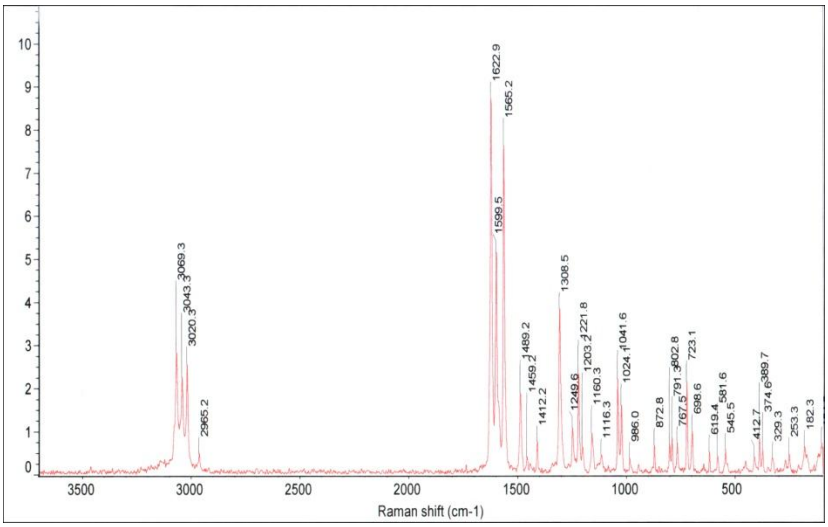

Fig.3 FTR spectrum of Carbamazepine

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RESULTS AND DISCUSSION

Vibrational analysis

Fourier transform infrared (FTIR) and Raman spectroscopic methods are being extensively used to identify the structural groups present in a compound. The aim of the present work is to make thorough investigation on vibrational frequencies of Carbamazepine. In analogy with the vibrational band assignments of related compounds and the magnitudes and relative intensities of the bands. The vibrational band assignments of the compound are presented in Table 2. A qualitative discussion on the specific modes of vibration is discussed as follows.

Table 2: Vibrational band assignments of Carbamazepine

Frequency (cm^{-1})		Band Assignment
FTIR	FT Raman	
507 (vw)		C-N-C bending
514 (vw)		C-N-C bending
537 (vw)	546 (vw)	C-C-C in plane bending
624 (m)	619(vw)	NH_2 Wagging
647 (w)		(C=O) in plane bending
704 (w)	697(vw)	C-H out of plane bending
724 (w)	723 (w)	C-H out of plane bending
764 (s)	766 (vw)	C-H out of plane bending
801 (m)	791(w)	C-H out of plane bending
851 (vw)		N-H out of plane bending
870 (w)	872 (vw)	N-H out of plane bending
989 (vw)	986 (vw)	H-C-N bending
1020(vw)	1024 (w)	C-H in plane bending
1044(vw)	1041 (w)	C-H in plane bending
1113(w)	1116 (vw)	C-H in plane bending
1152(w)	1160 (vw)	C-H in plane bending
1245(w)	1250 (w)	C-N stretching
1307(w)	1309 (m)	C-N stretching
1384(s)		NH_2 rocking
1436(w)		C=C stretching
1462(w)	1459 (w)	C=C stretching
1489(m)	1489 (w)	N-H in plane bending/C=C stretching
1594(s)		C=C stretching
1605(m)	1600 (m)	C =N stretching
1677(vs)		NH_2 scissoring/C=O stretching
3023(w)	3020 (m)	C-H stretching
3162(m)		C-H stretching
3283(w)		N-H symmetric stretching
3340(w)		N-H asymmetric stretching
3466(m)		O-H stretching

vw-very weak, w-weak, m-medium, s-strong, vs-very strong

N-H stretching vibrations

N-H stretching frequencies corresponding to the symmetrical and asymmetrical NH stretching vibrations for dilute solutions are occur near 3520 cm^{-1} to 3480 cm^{-1}. In the spectra of solid samples are observed near 3350 cm^{-1} to 3180 cm^{-1} because of Hydrogen bonding [5]. Based on this, in the present investigation, N-H symmetric stretching vibrations are observed at 3283 cm^{-1} and N-H asymmetric stretching occurred at 3340 cm^{-1}.

C-H stretching vibrations

These bands occur in the region 3010 cm^{-1} – 3200 cm^{-1}. Two moderately intense bands are frequently occurs in this region [62]. The appearance of two bands is attributed to Fermi resonance between the fundamental aldehydic C-H stretch and the first overtone of the aldehydic C-H bending vibration that usually appears near 1390cm^{-1}. Based on this in the present investigation, C-H stretching vibrations occur at 3023 and 3162 cm^{-1}. The corresponding Raman band is observed at 3020 cm^{-1}.

C-N stretching vibrations

Medium to weak absorption bands for the unconjugated C-N linkage in primary, secondary and tertiary aliphatic amines appear in the region of 1250-1020 cm^{-1}. Aromatic amines display strong C-N stretching absorption in the 1342-1266 cm^{-1} region [7]. Hence in the present case the FTIR bands observed at 1245 and 1307 cm^{-1} and corresponding Raman bands observed at 1250 and 1309 cm^{-1} are assigned to C-N stretching vibrations.

C=C stretching vibrations

Benzene has two doubly degenerate modes and two non-degenerate modes of vibrations due to stretching of C=C bonds. The C=C stretching vibrations occur in the region 1625-1430 cm^{-1}. In the present work, the FTIR bands observed at 1436, 1462, 1489 and 1594 cm^{-1} have been assigned to C=C stretching vibrations. Also the corresponding Raman bands are identified at 1459 and 1489 cm^{-1}.

C=O stretching vibrations

The C=O absorption of lactams, 6 membered rings or larger is near 1650 cm^{-1}. The five membered ring lactams absorb in the range 1750-1700 cm^{-1}. The four membered ring lactams, unfused absorbs at 1760-1730 cm^{-1} [8]. In the present investigation the FTIR band for C=O stretching occur at 1677 cm^{-1} at very strong region.

O-H vibrations

Alcohols and phenols, in vapour state or in dilute solution in non-polar solvents exhibit a sharp rather weak O-H stretching absorption due to non-bonded or free OH groups. These non-bonded O-H stretching bands appear near 3650 cm^{-1} in alcohols and near 3600 cm^{-1} in phenols. Inter - molecular hydrogen bonding increases as the concentration of the solute in solution increases and additional bands start to appear at lower frequencies near 3550-3200 cm^{-1} at the expense of the free hydroxyl band. In case, where intramolecular bonding occurs the hydroxyl group (O-H) band appears at 3590 - 3400 cm^{-1} [9]. Based on this in the present investigation the band observed at 3466 cm^{-1} is assigned to O-H stretching vibrations.

NH_2 Group vibrations

The NH_2 group has two (N-H) stretching vibrations, one being symmetric and other asymmetric. The frequency of asymmetric vibration is higher than that of symmetric one. It has frequency range of 3300 cm^{-1} to 3700 cm^{-1}. In addition, NH_2 group has scissoring, rocking, wagging and torsion modes. The NH_2 scissoring mode has been suggested to lie in the region 1590 cm^{-1} to 1650 cm^{-1} in benzene derivatives containing an NH_2 group. In the present work NH_2 scissoring band occur at 1677cm^{-1}. It is noted that frequency 1677 cm^{-1} is in very strong region. Similarly NH_2 rocking vibration occurs at 1384 cm^{-1} and NH_2 wagging vibrations occur at 624cm^{-1} and corresponding Raman band is observed at 619 cm^{-1}.

Bending vibrations

The in plane, out of plane bending of a ring hydrogen atom is strongly coupled to adjacent hydrogen atoms. The position of absorption of the out of plane bending band is, therefore, characteristic of the number of adjacent hydrogen atoms on the ring. The bands are frequently intense and may be used for the qualitative determination of the relative concentrations of isomers in mixtures. The absorption band that frequently appears in the spectra of substituted benzenes near 710-675 cm^{-1} is attributed to out of plane bending [10, 11]. In the present investigation the C-H out of plane bending bands occurs at 704, 724, 764 and 801 cm^{-1}. The IR bands observed at 1020, 1044, 1113 and 1152 cm^{-1} are assigned to C-H in plane bending vibrations. The corresponding FTRaman bands identified at 1024, 1041, 1116 and 1160 cm^{-1}.

In the present work, the FTIR band observed at 1489 cm^{-1} is referred as N-H in plane bending and bands occurs at 851 and 870 cm^{-1} have been assigned to N-H out of plane bending vibrations respectively. Similarly the other vibrations are observed in the characteristic range.

Quality Analysis

Quality assurance plays a central role in determining the safety and efficacy of medicines. Highly specific and sensitive techniques hold the key to the design, development, standardization and quality control of medicinal products. UV-Visible spectrophotometry is a satisfactory method employed in the quality analysis of drugs.

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The method is most often used in a quantitative way to determine concentrations of an absorbing species in solution, using the Beer-Lambert law:

$$A = -\log_{10}\left(\frac{I_o}{I}\right) = \varepsilon . c.L$$

where A is the measured absorbance, I_o is the intensity of the incident light at a given wavelength, I is the transmitted intensity, L the path length through the sample, and c the concentration of the absorbing species. The Beer-Lambert Law is useful for characterizing many compounds but does not hold as a universal relationship for the concentration and absorption of all substances.

For various concentrations, the spectra have been recorded and the absorbance values of wavelength maximum (λ_{max} = 286 nm) have been identified. 100 mg of Carbamazepine is dissolved in 100 ml of ethanol. For various concentrations the absorbance values are summarized in Table 3. It is confirmed that the absorbance values and concentrations are good with Beer's relationship. Verification of Beer's law is presented in Fig.4.

Table 3: Variation of absorbance with concentration

Concentration (□ g/ml)	Absorbance (□$_{max}$=286nm)
5	0.2517
10	0.6161
15	0.7509
20	1.2386
25	1.5513

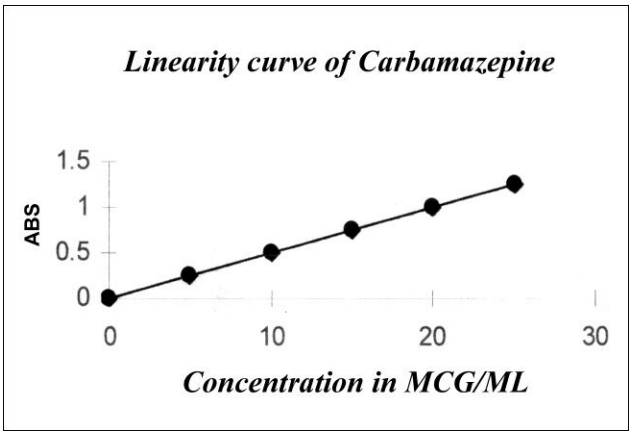

Fig.4 Verification of Beer's law

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Study of storage condition

Modern methods of quality analysis are extremely sensitive, providing precise and detailed information from small samples of material. For these reasons, they are in widespread use in product development in the control of manufacture and formulation as a check on stability during storage and in monitoring the use of drugs and medicines. The drugs used in the present study should be stored in light resistance containers. The storage condition plays a vital role for the good quality of the drug. Light resistant container protects the contents from the effect of incident light by virtue of the specific property of material of which it is composed of including any coating applied to it. UV-Visible spectrophotometry is a method used for checking the quality of the drug under different storage conditions.

Sample of Carbamazepine (100 mg) is dissolved in 100 ml of ethanol. Equal amount of solution is taken and kept under LRC (light resistance container), Ice point, Sunlight and exposed to Infrared light for two hours. The UV-Vis spectrum of Carbamazepine stored at ice point is presented in Fig.5 and variation of absorbance with different storage conditions are given in Table 4. Hence it is concluded that the best storage condition of the drug is LRC to retain its pharmaceutical properties.

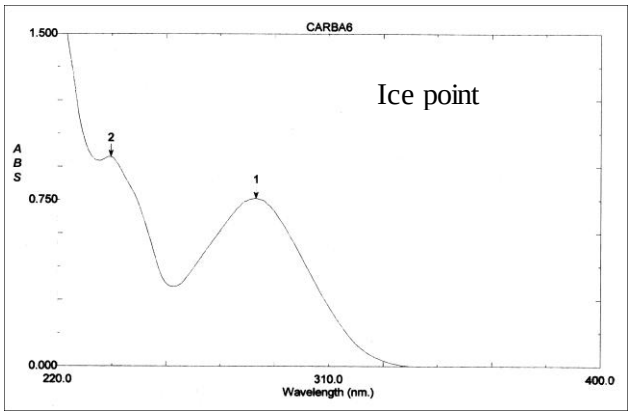

Fig.5: UV-Vis spectrum of Carbamazepine stored at Ice point

Table 4: Carbamazepine stored at different conditions

Storage Condition	Absorbance
Sun light	0. 7368
Ice point	0.7576
LRC	0.7509
IR Exposed	0.7162

CONCLUSION

The spectroscopic methods have been employed in the analysis of drug Carbamazepine. Using FTIR and FTRaman spectra the vibrational band assignments have been made in analogy

with the related molecules. Also the light absorption characteristics and storage condition of the compound has been made using UV-Visible spectral measurements. The best storage condition of the drug is light resistance container. Hence it has been established that the sophisticated spectrophotometers are used as a powerful tool for quality control in pharmaceutical laboratories.

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