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QUALITATIVE AND QUANTITATIVE ANALYSIS OF LORNOXICAM

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ABSTRACT

Lornoxicam is a non steroidal anti inflammatory drug (NSAID). Modern spectroscopic methods are very effective and sensitive tool for the qualitative and quantitative analysis of many drugs and the results are well employed in the quality control laboratories of pharmaceutical firm. In the present work, the FTIR and Raman spectra of the drug was recorded in the solid state. The fundamental modes of vibration wereassigned based on the position, shape and relative intensity of the recordedspectra and in correlation with the vibrational bands of structurallyrelated molecules. The stability of the drug under different environmental conditions is one of the quality assurance method undertaken in the pharmaceutical laboratory. By employing FTIR spectral technique the qualityof the drug under various storage conditions has been studied. The assay of the tablet of the title drug was done using UV-visible spectroscopyand compared with the labelled amount.

Keywords *– FTIR, FT Raman, UV-Visible Spectroscopy, anti-inflammatory drug, stability, Drug assay.*

1. INTRODUCTION

Lornoxicam is a non steroidal anti inflammatory drug (NSAID).It belongs to oxicam class with analgesic (pain relieving), anti-inflammatory and antipyretic (fever reducing) properties. It is available in oral and parentral formulations. Lornoxicam differs from other oxicam compounds in its potent inhibition of prostaglandin biosynthesis, a property that explains the particularly pronounced efficacy of the drug.Lornoxicam is used for the treatment of various types of pain, especially resulting from inflammatory diseases of the joints, osteoarthritis, surgery, sciatica, and other inflammations[1]. The systematic IUPAC name of Lornoxicam is (3E)-6-chloro-3-[hydroxy(pyridin-2-ylamino)methylene]-2-methyl-2,3-dihydro-4Hthieno[2,3-e][1,2]thiazin-4-one 1,1-dioxide. Its molecular formula is $C_{13}H_{10}ClN_3O_4S_2$. The molecular mass of Lornoxicam is 371.8192 g/mol. Lornoxicam is a yellow crystalline substance.

It is very essential to design pharmaceutical products that consistently deliver the intended performance, which demands monitoring of their quality incessantly. Quality of drug plays a very vital role indicating the suitability of drug product for its intended use. In this work, a quality analysis of anti-inflammatory drug Lornoxicam has been carried out by employing FTIR and FT-Raman spectroscopic techniques. The change in quality of the drug when stored under various conditions has been studied. Hence the present study aims to

make use of FTIR, FT Raman and UV-Visible spectroscopic methods in the qualitative and quantitative analysis of this drug. The molecular structure of Lornoxicam is shown in Fig 1.

2. MATERIALS AND METHODS

The spectroscopically pure grade sample of Lornoxicam was procured from a reputed Pharmaceutical company, Chennai, India and was used as such for the spectral measurements. The FTIR and FT Raman spectra were measured using Perkin Elmer Spectrum1 spectrometer over the region 4000-400 cm^{-1} and The FT Raman spectra were measured using 1064 nm line of Nd: YAG Laser operating at 200 mW on BRUKER RFS 27spectrometer in the region 4000-50 cm^{-1}, respectively at Sophisticated Analytical Instrumentation Facility (SAIF), IIT, Chennai, India. The UV-Visible spectral measurements have been made using UV-1700 Series in the wavelength region 200-800 nm at pharma analytical Lab, Puduchery. The FTIR and FT Raman spectra of Lornoxicam are presented in Fig 2 and 3 respectively.

3. RESULTS AND DISCUSSION

3.1 Vibrational Spectral Analysis

Fourier transform infrared (FTIR) and Raman spectroscopic methods are being extensively used to identify the structural groups present in a compound. The vibrational band assignments of Lornoxicam have been made in accordance with their position, shape and relative intensity. Also the assignments have been made in analogy with the structurally related molecules. The qualitative investigation on the vibrational band assignments derived from FTIR and FT Raman spectra for Lornoxicam are presented in Table-1.

3.1.1 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[2]. Based on this, in the present investigation, N-H stretching vibrations are observed at 3436 cm^{-1}.

3.1.2 C-H stretching vibrations

The hetero aromatic compounds and its derivatives are structurally very close to benzene. The C-H stretching vibrations[3-6] of hetero aromatic structure occur in the region 3100-3000cm^{-1} for asymmetric stretching and 2990-2900cm^{-1} for symmetric stretching modes of vibration. Further, in this region, the bands are not much affected due to the nature and position of the substituents. Heterocyclic compounds C-H vibration absorption bands are usually weak for detection. Hence, in the present work, the FTIR bands observed at 3066 cm^{-1} have been assigned to C-H stretching vibrations. Also the corresponding Raman bands are observed at 3108, 3068, 3042, 3006 and 2926 cm^{-1}.

3.1.3 C-N stretching

The C-N stretching vibrations[7] generally occur in the region 1170-1040 cm^{-1}. In the present study, the weak and medium to strong intensity bands appearing at frequencies 1146, 1084 and 1039 cm^{-1} in IR spectrum and in Raman spectrum, weak band appearing at 1037 cm^{-1} have been assigned as C-N stretching vibrations for Lornoxicam.

3.1.4 Deformation vibrations

The C-C-C bending bands always occur below 600 cm^{-1}. Isopropyl benzenes[8] have a medium intensity absorption band in the region 560-480 $cm^{-1.}$ The bands at 291,109 cm^{-1} are due to C-C-C bending vibrations. N-C-C out of plane bending vibrations are observed at 766,632 and 576 cm^{-1} in IR spectrum and in Raman spectrum, 718 and 634 cm^{-1} for Lornoxicam.

Thus, a satisfactory vibrational band assignment has been made by observing the position, shape and intensity of the vibrational bands both in IR and Raman spectra of the chosen drug and hence studied their quality.

3.2 Qualitative Analysis using FTIR spectroscopy

Drug quality is a source of great concern worldwide as pharmaceutical products plays an important role in improving the health and promoting the well being of every individual. Use of poor quality drugs has serious health implications and wasted resources. Temperature is one of the important environmental parameter that plays a key role in maintaining the drug quality. Drug must be stored, handled and transported according to predetermined conditions as supported by stability data. Drugs which are not stored under the recommended temperature conditions might degrade even prior to the expiration date. Among the various methods for the analysis, spectroscopic techniques are a predominant tool used to analyze the quality of drugs under different storage conditions. In the present work FTIR spectroscopic method has been applied to check the nature and the quality of the drug sample Lornoxicam when it is exposed to sunlight, ice point etc.

The Indian Pharmacopoeia recommends that Lornoxicam should be stored in tightly closed, light-resistant containers[9]. The behaviour of the drug that was stored under the prescribed storage condition with those stored at altered conditions has been compared. The FTIR spectra of the sample has been recorded for the pure drug stored in (i) well-sealed light resistant container (ii) exposed to sunlight and (iii) at ice point. Fig 4 gives a comparison of the FTIR spectra of Lornoxicam at different storage conditions.

Table 3 compares the absorbance values of some selected specific modes of vibration for the chosen molecule. This table indicate change in the absorbance values with change in storage condition. The internal standard ratio is calculated among the various absorption modes of vibration of the drug and the results are tabulated in Table 3. The internal standard ratios evaluated clearly shows the deterioration in the quality of the sample due to alteration in the storage conditions.

3.3 Assay of Drug– UV-Visible spectroscopy

Tablets are the popular form of dosage because of their cost effective preparation, stability and convenience in packaging, transporting and dispensing. It is popular among patients for accuracy of dosage, compactness, portability, blandness of taste and ease of administration[10]. Quantitative spectrometry is an extension of calorimetry and many pharmacopical substances are assayed spectrophotometrically[11]. In the present work medicine of Lornoxicam in the form of tablet was subjected to quantitative estimation of the drug substance in the tablet using UV-visible spectroscopic technique. The tablet NOC-8 mg

Containing Lornoxicam as the active ingredient was obtained from a leading pharmaceutical company. The drug content is determined by preparing a stock solution of the test sample and the solution is diluted to the same concentration as that of the standard sample and the absorbance of the resulting solution under UV-visible radiation was measured[12]. The drug was found to obey Beer's law. The drug content of the tablet is calculated as given below.

$$\text{Drug content of the tablet or assay} = \frac{\text{Test absorption}}{\text{Standard absorption}} \times \frac{\text{Standard weight}}{\text{Test weight}} \times \text{Average weight of one tablet}$$

The UV spectral recording of the pure sample Lornoxicam and tablet NOC-8 mg was carried out for concentration of 17.5 mcg.The UV-Visible spectrum of the sample exhibits wavelength maximum at 259, 289 and 377 nm. The average weight of one tablet is found to be 193 mg mg. The UV-visible spectra are recorded and absorbance is noted for the pure and tablet form of the sample. Also the bar diagram in the Fig. 5 shows the variance of λmax with absorbance at pure and tablet form of the sample. The quantitative estimation or assay of the active substance Lornoxicam is estimated in tablet (NOC-8 mg) and is found to be 7.84 mg.

Table 1: Vibrational band assignments of Lornoxicam.

Frequency (cm^{-1}) FTIR	Frequency (cm^{-1}) FT Raman	Vibrational band assignment
3436		N-H stretching
	3108	C-H Asym.Stretching
3066	3068	C-H Asym.stretching
	3042	C-H Asym.Stretching
	3006	C-H Asym.Stretching
	2926	C-H sym.Stretching
1646	1633	C=O stretching
1621		C=O stretching
1595	1590	C=O stretching
1546		C=C stretching
	1502	N-H out of bending
1425	1428	C-H bending
1383	1383	C-C stretching
	1355	C-C stretching
1327		S=O stretching
1236		C-O stretching
1172	1180	C-H bending
1146		C-N stretching
1084		C-N stretching
1039	1037	C-N stretching
	999	H-C-N torsional vibration
	871	H-C-C torsional vibration
830	833	C-S stretching
791		C-N-C out of plane bending
766		N-C-C out of plane bending
	718	N-C-C out of plane bending
	690	S-C-C bending
632	634	N-C-C bending
613		N-O-S out of plane bending
576		N-C-C bending
545		N-O-S out of plane bending
	491	Cl-C-S out of plane bending
	433	N-C-S out of plane bending
	399	C-N-S bending
	291	C-C-C bending
	230	Cl-C-S out of plane bending
	152	S-C-N out of plane bending
	109	C-C-C out of plane bending
	77	S-C-N out of plane bending

Table 2 : Absorbance for certain modes of vibration under different conditions of storage for Lornoxicam

Frequency (cm^{-1})	Absorbance		
	Labeled Condition	Exposed to sunlight	At ice Point
3067	0.847	0.849	0.964
1595	1.163	1.246	1.344
1425	1.093	1.181	1.279
1327	1.022	1.072	1.210
1146	0.832	0.878	0.987
1040	0.653	0.670	0.768
791	0.589	0.603	0.703
544	0.413	0.427	0.481

Table 3 Internal standard ratio parameters for Lornoxicam

Condition of Exposure	Internal Standard of specific modes of vibration at 3067 cm^{-1}							
	$A_{3067/3067}$	$A_{1595/3067}$	$A_{1425/3067}$	$A_{1327/3067}$	$A_{1146/3067}$	$A_{1040/3067}$	$A_{791/3067}$	$A_{544/3067}$
Labeled condition	1.0000	1.3731	1.2904	1.2066	0.9823	0.7710	0.6954	0.4876
At sun light	1.0000	1.4676	1.3910	1.2627	1.0342	0.7892	0.7102	0.5029
At Ice point	1.0000	1.3942	1.3268	1.2552	1.0239	0.7967	0.7293	0.4990
	Internal Standard of specific modes of vibration at 1595 cm^{-1}							
	$A_{3067/1595}$	$A_{1595/1595}$	$A_{1425/1595}$	$A_{1327/1595}$	$A_{1146/1595}$	$A_{1040/1595}$	$A_{791/1595}$	$A_{544/1595}$
Labeled condition	0.7283	1.0000	0.9398	0.8788	0.7154	0.5615	0.5064	0.3551
At sun light	0.6814	1.0000	0.9478	0.8604	0.7047	0.5377	0.4839	0.3427
At Ice point	0.7173	1.0000	0.9516	0.9003	0.7344	0.5714	0.5231	0.3579
	Internal Standard of specific modes of vibration at 1425 cm^{-1}							
	$A_{3067/1425}$	$A_{1595/1425}$	$A_{1425/1425}$	$A_{1327/1425}$	$A_{1146/1425}$	$A_{1040/1425}$	$A_{791/1425}$	$A_{544/1425}$
Labeled condition	0.7749	1.0640	1.0000	0.9350	0.7612	0.5974	0.5389	0.3779
At sun light	0.7189	1.0550	1.0000	0.9077	0.7434	0.5673	0.5106	0.3616
At Ice point	0.7537	1.0508	1.0000	0.9461	0.7717	0.6005	0.5496	0.3761
	Internal Standard of specific modes of vibration at 1327 cm^{-1}							
	$A_{3067/1327}$	$A_{1595/1327}$	$A_{1425/1327}$	$A_{1327/1327}$	$A_{1146/1327}$	$A_{1040/1327}$	$A_{791/1327}$	$A_{544/1327}$
Labeled condition	0.8288	1.1380	1.0694	1.0000	0.8141	0.6389	0.5763	0.4041
At sun light	0.7920	1.1623	1.1017	1.0000	0.8190	0.6250	0.5625	0.3983
At Ice point	0.7967	1.1107	1.0570	1.0000	0.8157	0.6347	0.5810	0.3975
	Internal Standard of specific modes of vibration at 1146 cm^{-1}							
	$A_{3067/1146}$	$A_{1595/1146}$	$A_{1425/1146}$	$A_{1327/1146}$	$A_{1146/1146}$	$A_{1040/1146}$	$A_{791/1146}$	$A_{544/1146}$
Labeled condition	1.0180	1.3978	1.3137	1.2284	1.0000	0.7849	0.7079	0.4964
At sun light	0.9670	1.4191	1.3451	1.2210	1.0000	0.7631	0.6868	0.4863
At Ice point	0.9767	1.3617	1.2958	1.2260	1.0000	0.7781	0.7123	0.4873
	Internal Standard of specific modes of vibration at 1040 cm^{-1}							
	$A_{3067/1040}$	$A_{1595/1040}$	$A_{1425/1040}$	$A_{1327/1040}$	$A_{1146/1040}$	$A_{1040/1040}$	$A_{791/1040}$	$A_{544/1040}$
Labeled condition	1.2971	1.7810	1.6738	1.5651	1.2741	1.0000	0.9020	0.6325
At sun light	1.2672	1.8597	1.7627	1.6000	1.3104	1.0000	0.9000	0.6373
At Ice point	1.2552	1.7500	1.6654	1.5755	1.2852	1.0000	0.9154	0.6263
	Internal Standard of specific modes of vibration at 791 cm^{-1}							
	$A_{3067/791}$	$A_{1595/791}$	$A_{1425/791}$	$A_{1327/791}$	$A_{1146/791}$	$A_{1040/791}$	$A_{791/791}$	$A_{544/791}$
Labeled condition	1.4380	1.9745	1.8557	1.7351	1.4126	1.1087	1.0000	0.7012
At sun light	1.4080	2.0663	1.9585	1.7778	1.4561	1.1111	1.0000	0.7081
At Ice point	1.3713	1.9118	1.8193	1.7212	1.4040	1.0925	1.0000	0.6842
	Internal Standard of specific modes of vibration at 544 cm^{-1}							
	$A_{3067/544}$	$A_{1595/544}$	$A_{1425/544}$	$A_{1327/544}$	$A_{1146/544}$	$A_{1040/544}$	$A_{791/544}$	$A_{544/544}$
Labeled condition	2.0508	2.8160	2.6465	2.4746	2.0145	1.5811	1.4262	1.0000
At sun light	1.9883	2.9180	2.7658	2.5105	2.0562	1.5691	1.4122	1.0000
At Ice point	2.0042	2.7942	2.6590	2.5156	2.0520	1.5967	1.4615	1.0000

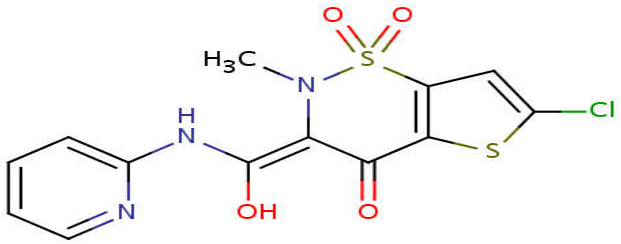

Fig.1 : Molecular structure of Lornoxicam.

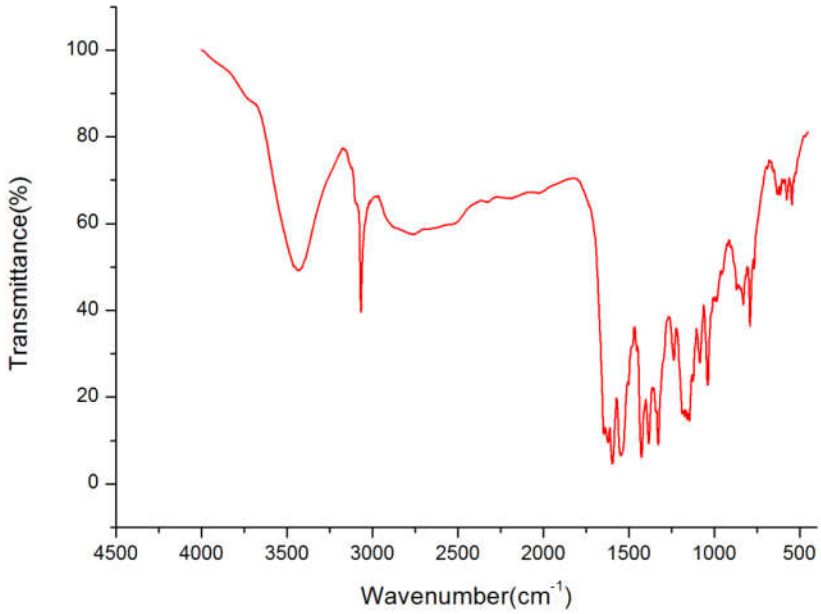

Fig.2 : FTIR spectrum of Lornoxicam.

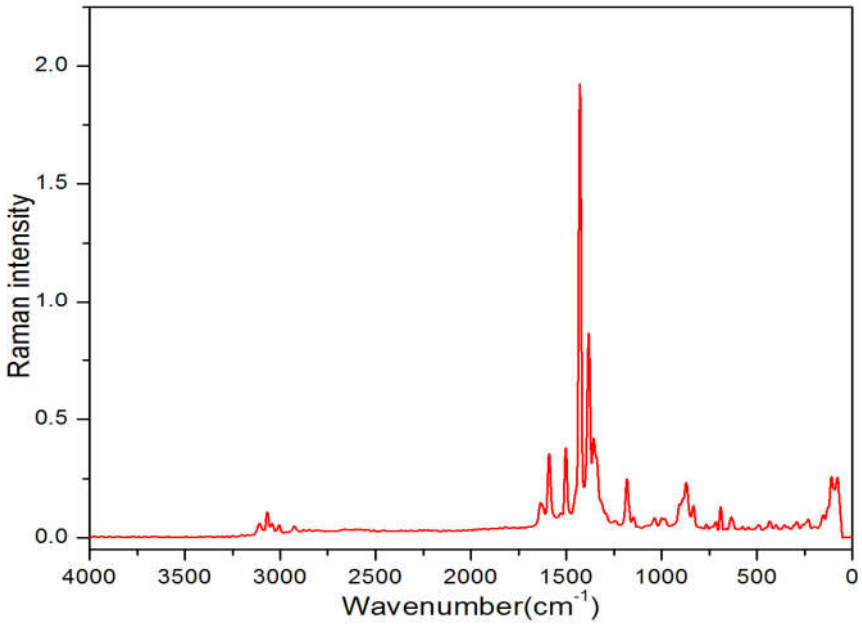

Fig.3 : FT-Raman spectrum of Lornoxicam.

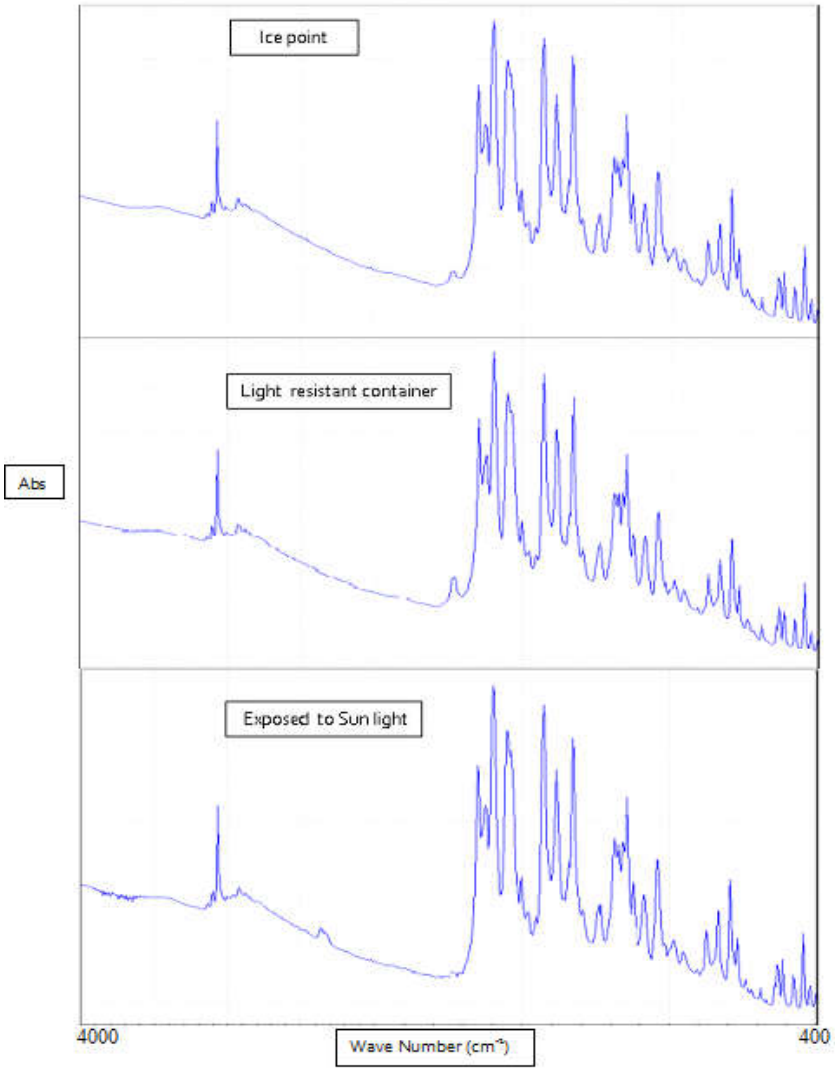

Figure 4 : Comparison of the FTIR Spectra of Lornoxicam at Different Environmental Exposure

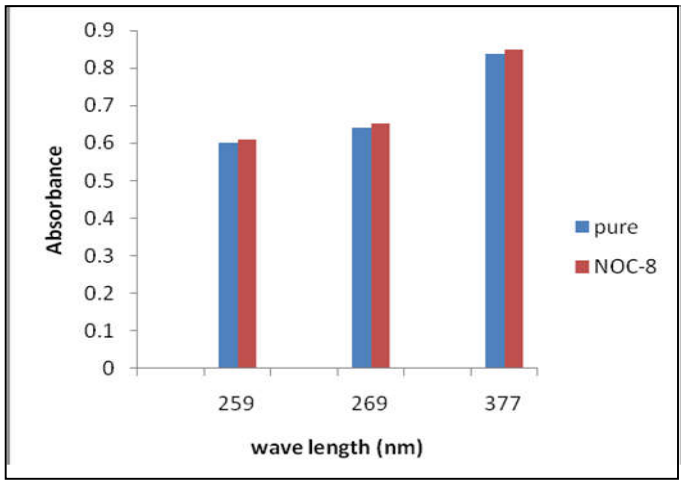

Fig 5 : Bar chart for the variance of λmax with absorbance of pure Lornoxicam and NOC tablet.

4. CONCLUSION

FTIR and FT-Raman spectroscopic technique have been employed for the qualitative analysis of the anti-inflammatory drug Lornoxicam. A satisfactory vibrational assignment of the drug has been done from the FTIR and FT-Raman spectral measurements. They confirm the basic functional groups present in the compound. The intensity ratio calculated among specific modes of vibrations clearly shows that some vibrational bands are more altered due to sunlight exposure and storage at ice point. This clearly denotes that a change in the quality of the drugs has taken place due to the change in storage condition. Therefore, the drugs should be stored or placed according to the storage condition of the particular drug otherwise its efficiency will be destroyed and it is dangerous to intake the drug. The UV-visible spectroscopic method was used to find the amount of drug present in tablet formulations. Tablet NOC-8 mg was found to contain 7.84 mg of Lornoxicam as the active substance.

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