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Synthesis and Spectral Characterization of Fluoroquinolone Drug-Norfloxacin

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ABSTRACT: FTIR study provides the most direct and definitive identification of fluoroquinolone and offer a means for qualitative analysis of newly synthesized fluroquinolone drug-Norfloxacin. The vibrational spectroscopy, such as FTIR has been used to measure the vibrational modes of fluoroquinolones, provides information about structural differences of its individual members. Form the interpreted spectral data norfloxacin has been distinguished by the presence of different substituents in their parent nucleus.

Keywords: Fluoroquinolones; Norfloxacin; Gonorrhea and Spectrum.

INTRODUCTION: Fluoroquinolones have been associated with a significant number of serious adverse drug reactions such as tendon damage and peripheral neuropathy, such as reactions may manifest long after therapy had been completed and in severe cases may result in lifelong disabilities. They are associated with severe psychiatric adverse reaction. The reaction was detailed within Stephen fried's book Bitter Pills (1999). Hepatotoxicity has also been reported with the use of some fluoroquinolones.

Norfloxacin is a synthetic antibacterial agent (antibiotic) that belongs to the class of fluoroquinolone antibiotics. It is used to treat urinary tract infections gynecological infections, inflammation of the prostate gland, gonorrhea and bladder infections.

Norfloxacin is a first generation fluoroquinolone that has been available for treatment of bacterial infections for many years. Like other fluoroquiolones, Norfloxacin is active against a wide range of aerobic gram positive and gram negative organism and is believed to act by inhibition of bacterial DNA gyrase and tropoisomerase IV that are required for synthesis of bacterial mRNA (transcription) and DNA replication. In contrast DNA gyrases are not present in human and other eukaryotic cells and the equivalent tropoisomerases are not sensitive to fluoroquinolone inhibition

MATERIALS AND METHODS: All chemicals are of analytical grade and purchased from CDH and Merck. Melting points are determined in an open capillary tube and are uncorrected. An infrared spectrum has been recorded in KBr on Perkin-Elmer RXI spectrometer at CDRI, Lucknow. The ^{1}H-NMR were measured in $CDCl_3$ solution on a brucker DRX-300 MHz spectrometer using tetramethyl silane (TMS) as an internal reference and chemical shift in ppm at CDRI, Lucknow. Elemental analyses were carried out with elementar vario El, III elemental analyser at department of biotechnology IIT, Kharagpur.

Synthesis of Norfloxacin: The compound was refluxed in autoclave at 150° C for two hours. Sodium hydroxide was with continuous stirring for 10 minutes. Thereafter, 10 g of piperazine was added and the mixture was added and the mixture was refluxed for 30 hrs in a 250 ml of three necked flask equipped with a reflux condenser and mechanical stirrer. Sodium carbonate was added until the mixture was alkaline. The excess solvent was removed by steam distillation. The residual solution was kept in a refrigerator until crystallization was completed. The solution was filtered on a Buchner funnel and washed with 10 ml of saturated sodium chloride solution.

Now, the solution of 40 g of sodium hydroxide and 160 ml of water were added in a one liter round bottomed flask equipped with a reflux condenser. The mixture was heated to boil until the compound was disappeared. The reaction mixture was diluted with an equal volume of water. When cold, the reaction product was poured with vigorous stirring into 125 ml of concentrated hydrochloric acid. Then, it was allowed to cool at room temperature. The compound was filtered at the pump and washed with a little water and characterized. It is crystalline in nature.

Table 1: Elemental analysis (in %).

	C	H	N
Found	60.12	5.64	13.15
Calculated	59.94	5.58	13.01

Pure sample of Norfloxacin ($C_{16}H_{18}FN_3O_3$) were obtained from external agency. The drug was 99.8% to 98.0% pure. FTIR spectroscopy is an important analytical technique which detects various characteristics of functional groups in molecule on interaction of an infrared light with the matter chemical bonds would, stretch, contract and bond as a result each chemicals functional group tend to absorb infrared radiation in a specific wavelength range regardless of the structure of the rest molecule. Based on this principle functional groups present in composite materials are identified. It is performed in a FTIR spectro photometer interfaced with Infra Red (IR) microscope operated in reflectance mode. IR spectrum of fluoroquinolone drug-Norfloxacin has been recorded in the range 4000-400 cm^{-1}. The spectrum was interpreteted considering few main peaks observed.

RESULTS AND DISCUSSION: The Norfloxacin was white to pale yellow crystalline powder in nature. It was gradually coloured by light. It was hygroscopic in nature.

IR spectra were recorded as KBr pellets in the region of 4000-400 cm^{-1} on a Perkin-Elmer Spectrophotometer.

IR spectrum of fluoroquinolone drug-norfloxacin has been recorded in the range 4000-400 cm^{-1}. The spectrum was interpreteted considering few main peaks observed.

The ^{1}H-NMR spectra were recorded on a Hitachi FT–NMR model R-600 spectrometerusing $CDCl_3$ as the solvent. The chemical shifts are given in ppm relative to tetramethylsilane (TMS).

Mass spectra of compound showed molecular ion peak $(M/Z)^+$ at 319 and other peak at 277,275,237 which corresponds to the molecular formula $C_{16}H_{18}FN_3O_3$ which is the molecular formula of Norfloxacin

The IR spectrum of norfloxacin shows an absorption band at 3032 cm^{-1} indicating the presence of aromatic C=C-H proton. It shows a characteristics band at 1045 cm^{-1}, which indicates the presence of C–F group in the molecules. The compound showed a characteristic band at 1615 cm^{-1} and 1495 cm^{-1}, those are responsible for benzene ring. The band at 1630 cm^{-1} indicates the presence of olefinic C=C. It shows a band at 3035 cm^{-1} indicating the presence of C=C–H linkage47. It also shows a frequency at 1670 cm^{-1}, which indicates the presence of conjugated C=O group. The absorption bands appear at 1210 cm^{-1} and 1080 cm^{-1} indicating the presence of carboxyl (COOH) group. The band appears at 1049 cm^{-1} indicating the presence of –O– linkage. The compound showed absorption bands at 2905 cm^{-1}, 2895 cm^{-1} and 1437 cm^{-1}. These bands indicate the presence of CH_3, CH_2 and CH group respectively.

The compound norfloxacin shows singlet peak in ^{1}H-NMR spectrum at δ7.2, which indicates the molecule contains aromatic CH proton. The spectrum of compound contains a singlet peak at 6.8 indicating the C=CH group. . It shows a singlet signal at δ6.8 ppm for the NH proton. It shows a triplet peak at δ4.3 ppm indicating the presence of two equivalent protons at its adjacent position in the molecule. Its spectrum shows a singlet peak at δ1.2 ppm for CH_3 group. It also shows a doublet peak at 3.15 ppm for CH_2 group. The spectrum of norfloxacin shows two singlet peaks respectively at 3.0 and 2.2 ppm for CH_2 and CH_3. NMR signal of –OH group appears at δ 11.3 indicating its highly deshielded nature.

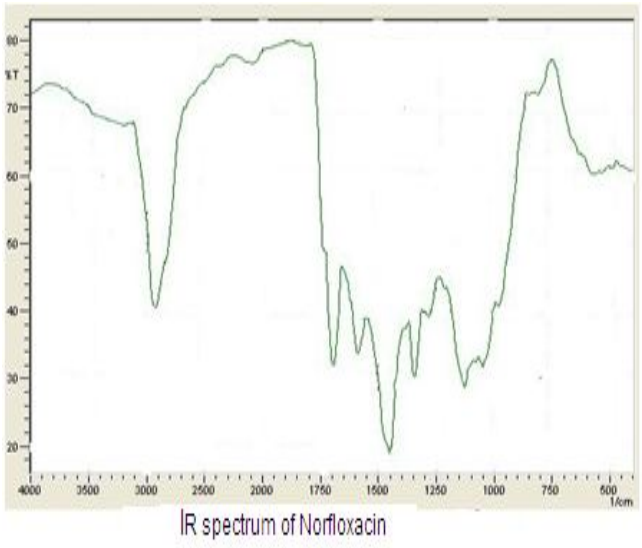
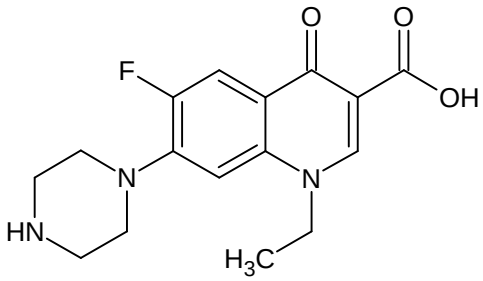


Figure 1: IR spectrum of Norfloxacin.

CONCLUSION: On the basis of FTIR spectral analysis of Norfloxacin, the structure of Fluoroquinolone-Norfloxacin may be drawn as:



Norfloxacin

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