

Summary

This thesis consists mainly of three chapters as follows:-

Chapter (1); Introduction

Contains two parts:

The first part:

Represents general introduction about fluoroquinolones and gives short notes about the physical and chemical properties of the drugs under investigation besides their mode of action and uses. Also give great attention on the basics, theory and applications of spectrophotometry, elemental analyzer (C, H, N, S and O analyzer), Infrared spectroscopy and Thermal Analysis Techniques.

The second part:

Includes a literature survey on the previous works carried out for the determination of the drugs; ciprofloxacin hydrochloride (CIP), levofloxacin (LEV), norfloxacin (NOR) and enrofloxacin (ENR), including spectrophotometric, titrimetric, electrochemical and chromatographic methods of analysis.

Chapter (2); Experimental

Contains two parts:

The first part:

Experimental part for determination of the studied drugs in liquid form through describing the procedures used throughout the study so as to get the

optimum conditions favoring colored complex formation between the drug molecules as electron donors and various electron acceptor molecules [rose bengal (RB), bromocresol purple (BCP), erythrosine (ERY) and ferric chloride (FeCl_3)]. Also describes the UV-Visible instrument used in the work and explaining the factors affecting the formation of these complexes such as pH, sequence additions, the effect of time, temperature, concentration of reagents and additives.

The second part:

Contain solid state study of the ion –pair complexes through explaining the procedure which used to syntheses of these complex between the drugs under investigation and trivalent transition element (Iron Fe(III)) and tetravalent element (uranium U(IV)) with describing the instruments which used in characterization and identification of these complexes, such as a elemental analyzer for carbon, hydrogen, oxygen and nitrogen elements, infrared spectrophotometer and a thermal analysis (TGA and DTA).

Chapter (3); Results and discussion

Reviewing and analyzing the results and is divided into two parts:

The first part:

Includes the representation of results obtained throughout the spectrophotometer work on the ion-pair complexes in liquid form and the explanation of them; the optimum conditions for the formation of the complexes were extensively studied. These conditions were then chosen for the micro determination of the drugs under investigation which is:

(I) Britton and Rhobinson universal buffer solution was found to be the best media for complexation process this series of buffer solution has the

advantage of wide range of PH (2-12) and its components do not seem to interfere with the drugs or reagents used.

- (II) An evidence for complex formation between drug and reagent molecules is observed in the maximum wave length (λ_{\max}) of coloured complexes formed under the optimum conditions. It possesses a red shift from that of the pure reagents or drugs under the same conditions.
- (III) Study of the effect of time and temperature showed that complexes are formed simultaneously and remain stable for about two hours. Also, the obtained complexes are stable to heating up to 45 °C.
- (IV) The sequence of addition was found to be of significant importance where it was found that the best sequence of additions is Reagent – Drug – Buffer in the majority of cases
- (V) The stoichiometry of the complexes formed in solutions was detected using the mole ratio and Continuous variation methods. The stability constant of the drug-reagent complexes were calculated from spectral data of the mole ratio and Continuous variation methods.
- (VI) The optimum concentration of drugs which can be successfully determined using the reagents under study, were detected by Beers law.
- (VII) Another way for detecting the lower and higher limits of concentrations of drugs could be determined is the Ringbom method.
- (VIII) The presence of additives and excipients such as sodium acetate, bicarbonate, magnesium stearate, talc powder, starch, glucose, fructose and lactose was studied where it was found that they do not interfere up to 10 %.
- (IX) The possibility of application of Beer's law and comparing the results with those obtained from the methods adopted; where possible to estimate the concentration of drugs in the pure form. It was also the application of the proposed methods of drugs under study in the pharmaceutical form and compare the results statistically with the results from the methods adopted and the results show that there is no clear difference between them which

confirms the possibility of applying the methods proposed to set the drugs under study in the pure form and pharmaceuticals.

The second part:

Includes the representation of the results obtained throughout the work on the solid ion-pair complexes which been isolated and analyzed through:

- (I) The molecular composition was first determined by elemental analysis and the proposed tentative formula was determined.
- (II) The IR spectra of ion-pair complexes were studied. The stretching vibrations of C-X, OH, C-O and C=O groups suffer dramatic changes in positions and intensity. The explanations in previous studies are used for proving the formation of complexes and understanding the nature of bonding between the donors and acceptors.
- (III) The thermal analysis (TGA and DTA) explain the percent of weight loss of the complexes by increasing the temperature.

These measurements have been used to prove the forming of the complexes and the molecular structure for it and understanding there nature as well as to consolidate the results of studies on the spectral solutions of the complexes. Also show that the interaction of the drugs with the metals to form ion-pair complexes are suggested to be $[\text{Fe}(\text{D})_2(\text{H}_2\text{O})_2]\text{Cl}_2 \cdot 6\text{H}_2\text{O}$ and $[\text{UO}_2(\text{D})_3](\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$.