Summary

The present thesis comprises three chapters:

Chapter (1)

Contains the introduction which includes two parts: the first part gives an idea about the drugs under consideration, a discussion about the definitions, actions, chemical structures and chemical names, characters of the studied drugs dequalinium chloride, tramadol hydrochloride, ambroxol hydrochloride, sertraline hydrochloride and clidinium bromide are also given. The second part gives a literature survey of the previous studies for the analysis of the studied drugs including spectrophotometric, ultra-violet spectrophotomtric, capillary electrophoresis, high-performance liquid chromatography, electroanalytical and chromatographic methods. Chemical structures, chemical names and a literature survey of the acid dyes used bromophenol blue (BPB), bromocresol green (BCG) and bromothymol blue (BTB) are also given.

Chapter (2)

Contains the experimental part which includes apparatus used for measurement, procedures for preparations of the drug solutions, reagents and October Pharma methods for the determination of the studied drugs in pure forms or in the pharmaceutical forms which are analysed. It also contains the proposed spectrophotometric methods for determination of the drugs under considerations in pure forms, in urine and in dosage forms. Also, it contains the official methods for analysis of the studied drugs.

Chapter (3)

Contains the results and discussion which include the spectrophotometric procedures for the determination of the studied drugs using acid dyes reagents bromophenol blue (BPB), bromocresol green (BCG) and bromothymol blue (BTB). The proposed methods are based on coloured ion-pair complex formation between the acid dyes and drugs which is extracted with organic solvents except for Deq, (chloroform, carbon tetrachloride, methylene chloride, hexane, benzene) and determination of the concentration by measuring the absorbance of the extracted complex against a blank prepared by the same way except addition of the drug. The following experimental variables were investigated.

1- Effect of pH.
2- Effect of time.
3- Effect of the extracting solvent.
4- Effect of reagent concentration.
5- Molecular ratio of the complex.
6- Suggested mechanism.
7- Interference.
8- Evaluation of the stability constants of the ion-pair complexes.

1- Using BPB

Beer's law is obeyed within the concentration ranges 1.0-20 μg/mL, 2.0-20 μg/mL, 1.0-20 μg/mL, 1.0-12 μg/mL and 2.5-20 μg/mL for Deq, Tram, Ambr, Sert and Clid, respectively in case of bromophenol blue (BPB). For more accurate results, Ringbom optimum concentration ranges are determined. Molar absorptivity, Sandell sensitivity, detection and quantification limits are calculated. The stoichiometric ratios of the studied

drugs with BPB are established using the mole ratio and continuous variation methods and found to be 1:1 for all the drugs under consideration with BPB. In order to determine the accuracy and precision of the proposed methods, solutions containing three different concentrations of the studied drugs are prepared and analysed in six replicates. The recovery, relative standard deviation, relative error and confidence limits are calculated. The proposed methods can successfully be applied to determine the pure form of the studied drugs and their dosage forms. The results obtained are compared statistically by Student's t-value and variance ratio F-test with the official methods at 95% confidence level. The results show that the t- and F-values are less than the critical value indicating that there is no significant difference between the proposed and official methods. Thus, the proposed spectrophotometric methods can be applied in determination of the studied drugs in pure form and in dosage forms. Also the studied drugs are determined in urine sample and the results showed that no interferences occur between the studied drugs and urine.

2- Using BCG

Beer's law is obeyed within the concentration ranges 1.0-18 μg/mL, 2.0-16 μg/mL, 2.0-20 μg/mL, 2.0-18 μg/mL and 2.5-20 μg/mL for Deq, Tram, Ambr, Sert and Clid, respectively in case of bromocresol green (BCG). For more accurate results, Ringbom optimum concentration ranges are determined. Molar absorptivity, Sandell sensitivity, detection and quantification limits are calculated. The stoichiometric ratios of the studied drugs with BCG are established using the mole ratio and continuous variation methods and found to be 1:1 for all the drugs under consideration with BCG. In order to determine the accuracy and precision of the proposed methods, solutions containing three different concentrations of the studied drugs are

prepared and analysed in six replicates. The recovery, relative standard deviation, relative error and confidence limits are calculated. The proposed methods can successfully be used to determine the pure form of the studied drugs and their dosage forms. The results obtained are compared statistically by Student's t-value and variance ratio F-test with the official methods at 95% confidence level. The results showed that the t- and F-values are less than the critical value indicating that there is no significant differences between the proposed and official methods. Thus, the proposed spectrophotometric methods can be applied for the determination of the studied drugs in pure and dosage forms. Also the studied drugs are determined in urine samples the results and calculations showed that no interferences between the studied drugs and urine can occur.

3- Using BTB

Beer's law is obeyed within the concentration ranges 1.0-15 µg/mL, 4.0-30 µg/mL, 1.0-21 µg/mL, 3.0-22.5 µg/mL and 4.0-30 µg/mL for Deq, Tram, Ambr, Sert and Clid, respectively in case of bromothymol blue (BTB). For more accurate results, Ringbom optimum concentration ranges are determined. Molar absorptivity, Sandell sensitivity, detection and quantification limits are calculated. The stoichiometric ratios of the studied drugs with BTB are established using the mole ratio and continuous variation methods and found to be 1:1 for all the drugs under consideration with BTB. In order to determine the accuracy and precision of the proposed methods, solutions containing three different concentrations of the studied drugs are prepared and analysed in six replicates. The recovery, relative standard deviation, relative error and confidence limits are calculated. The proposed methods can be successfully used to determine the pure form of the studied drugs and their dosage forms. The results obtained are compared statistically

by Student's t-value and variance ratio F-test with the official methods at 95% confidence level. The results showed that the t- and F-values are less than the critical value indicating that there is no significant differences between the proposed and official methods. Thus, the proposed spectrophotometric methods can be applied for the determination of the studied drugs in pure form and dosage forms. Also the studied drugs are determined in urine samples and the results showed that no interferences between the studied drugs and urine occur.