CHAPTER 7

SUMMARY OF THE PRESENT INVESTIGATIONS
Introduction

Quality assurance and control of pharmaceutical and chemical formulations are essential to ensure the availability of safe and effective drugs to consumers. Hence, Pharmaceutical analysis is an important to secure statutory certificates for drugs either to the industry, or for perusal by the regulatory authorities. The complex problems encountered in pharmaceutical analysis are in achieving the selection, speed, cost-effectiveness, simplification, precision and for ensuring accuracy of results. New methods of analysis are being quickly adopted by pharmaceutical industries and chemical laboratories depending upon the facilities available. Formulations containing various combinations of drugs for potentiating and complementing one another are key in therapy. Quite often the reported procedures need improvements or changes in view of the latest advances.

Among the several instrumental techniques eg., HPLC, GC, Fluorimetry, NMR, IR, UV and Visible spectroscopy available for the assay of drugs, spectrophotometric technique is simple and cost-effective.

UV and Visible spectrophotometric methods are highly versatile, sensitive and reproducible. An attempt is made here to develop new spectrophotometric methods for estimating the selected drugs from pharmaceutical preparations.

The contents of the thesis have been divided into seven chapters with appropriate references placed at the end of the last chapter.
Chapter-1 is a brief account of (a) drugs, (b) dosage forms (c) drug quality (d) the role of Analytical chemistry in pharmacy (e) drug validation and (f) spectroscopy.

Chapter-2 describes the survey of literature on the determination of the selected drugs and objectives of the present investigation.

Chapter-3 is divided into two sections.

Section 3.1 discusses the preparation of various drug solutions and reagents.

Section 3.2 depicts as brief profile of the selected drugs.

Chapter-4 of the thesis is divided into four sections

Section 4.1 comprises of the results obtained in a simple ion pair complex method for the estimation of ramipril by using spectrophotometry. The proposed method is based on the formation of chloroform extractable complex of ramipril with bromocresol green at pH 3.5. The absorbance of the extracted complex is measured at 420 nm. The amount of ramipril present in the sample is analysed by means of a calibration curve. The results are statistically validated.

Section 4.2 proposes a simple ion pair complex method for the estimation of esomeprazole magnesium by spectrophotometric method. The proposed method is based on the formation of chloroform extractable complex of
esomeprazole magnesium with bromocresol green at pH 3.5. The absorbance of the extracted complex is measured at 420 nm. The amount of esomeprazole magnesium present in the sample is read from calibration curve. Statistical validity of the results is tested.

Section 4.3 describes a simple ion pair complex method for the estimation of tenofovir difoproxil fumarate by spectrophotometric method. The proposed method is based on the formation of chloroform extractable complex of tenofovir difoproxil fumarate with bromocresol green at pH 3.5. The absorbance of the extracted complex is measured at 415 nm. The amount of tenofovir difoproxil fumarate present in the sample is represented by a calibration curve that enables an analyzing of results.

Section 4.4 analyses a simple ion pair complex method for the estimation of pramipexole by spectrophotometric method. The proposed method is based on the formation of chloroform extractable complex of pramipexole with bromocresol green at pH 3.5. The absorbance of the extracted complex is measured at 425 nm. The amount of pramipexole present in the sample is analyzed and the statically evaluating results are carried out.

In the proposed method, a systematic study of the effects of various relevant parameters, concentration of reagents, order of addition, time and temperature required for reaction, pH of buffer, nature of solvents for final dilution and the stability of reagents of the coloured species are studied by varying one parameter at a time and controlling all other
parameters to get a maximum development of colours, reproducibility and reasonable period of stability. Thus, the final coloured species is obtained.

Chapter-5 of the thesis is divided into four sections.

Section 5.1 contains a simple condensation method for the estimation of pramipexole by spectrophotometric method. The method is based on the condensation of amino group containing drugs with vanillin under acidic conditions to produce yellow coloured Schiff's base. The absorbance of the coloured species is measured at the 400 nm against reagent blank and the amount of the pramipexole is measured from the calibration curve. The results are statistically validated and incorporated in the thesis. The obtained colored solutions are utilized in the development of simple, rapid, and accurate spectrophotometric methods for the analysis of pure forms as well as in capsules. The proposed method is superior to the previously reported UV-based spectrophotometric methods, as the measurements are performed in the visible region. In addition, the proposed method uses a spectrophotometer, which is available in all quality control laboratories, and they involve very simple procedures.

The method is simple precise, reproducible and it can be adopted for the routine analysis of drugs in pharmaceutical formulations.

Section 5.2 discusses a simple condensation method for the estimation of lamotrigine by spectrophotometric method. The method is based on the condensation
of amino group containing drugs with vanillin under acidic conditions to produce yellow coloured Schiff's base. The absorbance of the coloured species is measured at the 390 nm against reagent blank and the amount of the lamotrigine is calculated through the calibration curve. The results are statistically validated and incorporated in the thesis. The obtained colored solutions are utilized in the development of simple, rapid, and accurate spectrophotometric methods for the analysis of pure forms as well as in capsules. The proposed method is superior to the previously reported UV-based spectrophotometric methods, as the measurements are performed in the visible region. In addition, the proposed method uses a spectrophotometer, which is available in all quality control laboratories, and involves very simple procedures.

The method is simple precise, reproducible and it can be adopted for the routine analysis of drugs in pharmaceutical formulations.

Section 5.3 reports on a simple condensation method for the estimation of mesalamine by spectrophotometric method. The method is based on the condensation of amino group containing drugs with vanillin under acidic conditions to produce yellow coloured Schiff's base. The absorbance of the coloured species is measured at the 395 nm against reagent blank and the amount of the mesalamine is calculated through the calibration curve. The results are statistically validated and incorporated in the thesis. The obtained colored solutions are utilized in the development of simple, rapid, and accurate spectrophotometric methods for the analysis of pure forms as well as in capsules. The
The method is simple precise, reproducible and it can be adopted for the routine analysis of drugs in pharmaceutical formulations.

Section 5.4 describes a simple condensation method for the estimation of amiloride by spectrophotometric method. The method is based on the condensation of amino group containing drug with vanillin under acidic conditions to produce yellow coloured Schiff's base. The absorbance of the coloured species is measured at the 405 nm against reagent blank and the amount of the amiloride is made through the calibration curve. The results are evaluated and incorporated in the thesis. The obtained colored solutions are utilized in the developing simple, rapid, and accurate spectrophotometric methods for the analysis of pure forms as well as in capsules. The proposed method is superior to the conventional UV-based spectrophotometric methods because the measurements are performed in the visible region. In addition, the proposed method uses a spectrophotometer, which is available in all quality control laboratories, and involves very simple procedures.

The method is simple precise, reproducible and it can be adopted for the routine analysis of drugs in pharmaceutical formulation.
Chapter 6 of the thesis is divided into four sections

Section 6.1 deals with the spectrophotometric assay of mesalamine by diazotization method. The mesalamine is treated with sodium nitrite in acidic medium for diazotization. After completion of diazotization, the diazotized drug is coupled with a coupling reagent, resorcinol, in basic medium to produce orange colour. The absorbance of the orange colour is measured at 460 nm against the reagent blank. This is a new method, found to be simple and sensitive. The colour development is instantaneous and stable for 24 hours.

Section 6.2 interprets the method of spectrophotometric assay of dapsone by diazotization. The dapsone is treated with sodium nitrite in acidic medium for diazotization. After completion of diazotization, the diazotized drug is coupled with a coupling reagent, resorcinol, in basic medium to produce orange colour. The absorbance of the orange colour is measured at 450 nm against the reagent blank. This is a new method, found to be simple and sensitive. The colour development is instantaneous and stable for 24 hours. The results are statistically validated and incorporated in the thesis.

Section 6.3 deals with the spectrophotometric assay of alfuzosin hydrochloride by diazotization method. The alfuzosin hydrochloride is treated with sodium nitrite in acidic medium for diazotization. After completion of diazotization, the diazotized drug is coupled with a coupling reagent,
resorcinol, in basic medium to produce orange colour. The absorbance of the orange colour is measured at 400 nm against the reagent blank. This is a new simple and sensitive method. The colour development is instantaneous and stable for 24 hours.

Section 6.4 describes the method of spectrophotometric assay of mosapride by diazotization. The mosapride is treated with sodium nitrite in acidic medium for diazotization. After completion of diazotization, the diazotized drug is coupled with a coupling reagent, resorcinol, in basic medium to produce orange colour. The absorbance of the orange colour is measured at 470 nm against the reagent blank. This is a new method, found to be simple and sensitive. The colour development is instantaneous and stable for 24 hours.

Chapter 7 of the thesis summarizes the present investigations. Thus, it is concluded that the proposed method has greater advantage over the several analytical methods like titrimetric methods, classical analytical methods and the several instrumental methods such as conductometric and potentiometric methods. The above methods are simple, specific, and easy to perform and require short time to analyze the samples. Low limit of quantification and limit of detection make these methods suitable for use in quality control. These methods were found to be accurate, precise, linear, stable and useful.
In conclusion, the author is of the opinion that the proposed methods by spectrophotometry offer definite advantages and novelty over conventional methods.