CONCLUSION

The subject matter of this thesis is to develop some new methods for analysis of pharmaceuticals by using HPLC and HPTLC techniques. These techniques are preferred above other modern analytical techniques because of their versatility in terms of application, selectivity, sensitivity and speed. In today’s regulated pharmaceutical industry it is only too essential to use very specific, sensitive, simple and accurate techniques, such as HPLC and HPTLC to ascertain the quality of these pharmaceuticals. Method development for various classes of drugs, such as Antibacterial, Sympathomimetic, Antihistaminic, Analgesic, Antipyretic, β-adrenergic bloker, Antianginal, Expectorant, β-adrenoceptor agonist, H₁ receptor and Dopamine antagonist has been attempted. The work was carried out in two parts namely:

1. HPLC technique has been used for determination and separation of some fixed drug combinations and for bulk drugs in presence of impurities.
2. HPTLC technique has been used to obtain better chromatographic data in terms of separation, sensitivity and speed for some fixed drug combinations.

The conclusion of the work is summarized as follows:

1. A new, simple, precise, rapid and selective RP–HPLC method has been developed for the simultaneous determination of Ciprofloxacin and Tinidazole in tablet dosages using 0.1% triethylamine in water. Acetonitrile (78:22 v/v), pH adjusted to 2.6 with phosphoric acid (1% v/v) as mobile phase and C₁₈ Intertial ODS–3V column (5µm, 25 cm × 4.6 mm; id), as stationary phase. Retention times of Ciprofloxacin and Tinidazole were 4.21 and 8.21 min. respectively.
2. A new HPLC method for the determination of Phenazopyridine Hydrochloride and related impurities in tablet and in bulk drug, using C\textsubscript{18} Inertsil ODS–3V column (5 \( \mu \)m, 25 cm \( \times \) 4.6 mm; id), as stationary phase and water: Acetonitrile (1:1 v/v) as mobile phase. Retention time was 9.80 min. Using the proposed chromatographic procedure and LC–MS technique two different process impurities namely, 2,6-diamine (5-phenazo, 2-aminopyridine) pyridine and 2,6 diamino diphenazo dianiline pyridine have been identified and confirmed.

3. A simple, precise and rapid reversed phase high performance liquid chromatographic method (HPLC) has been developed for the simultaneous determination of Nalidixic acid (NAL) and Phenazopyridine hydrochloride (PHE) in tablet. Chromatography was carried out on Nucleosil C\textsubscript{18} column (10\( \mu \)m; 250mm \( \times \) 4.0 mm; id) buffer: methanol (40: 60; v/v). The buffer was prepared by dissolving 7.73 gms of citric acid and 5.35 di-basic sodium orthophosphate in 1 liter of water. The pH of this buffer was 3.45. Retention times of NAL and PHE were 5.20 and 8.44 min. respectively.

4. A new simple, precise and rapid reversed phase high performance liquid chromatographic method (RP–HPLC) has been developed for the simultaneous determination of Phenylephrine hydrochloride (PHE), Chlopheniramine maleate (CPM), Paracetamol (PARA) and Caffeine (CF) in tablet. Chromatography was carried out on Inertsil ODS–3V column (5\( \mu \)m; 250mm \( \times \) 4.6 mm; id) developed mobile phase consisting of 0.1% Triethylamin in water. The pH was adjusted to 3.0 by using dilute phosphoric acid (1%; v/v) and Acetonitrile (75:25; v/v). Retention times of PHE, CPM, PARA and CF were 2.3, 3.78, 4.67 and 5.25 min. respectively.
5. A simple, rapid and precise HPTLC method has been developed for the simultaneous determination of Sulbutamol Sulphate (SS) and Bromhexine Hydrochloride (BH) by using methanol: chloroform: triethylamine (5.04.5:0.05 v/v) as the mobile phase and Merck precoated 60F<sub>254</sub> silicagel on aluminium sheet (0.25 mm thickness) as the stationary phase. Retention factors of SS and BH were 0.45 and 0.82 respectively.

6. A new simple, rapid and precise HPTLC method has been developed for the simultaneous determination of Cinnarizine (CINN) and Domperidone Maleate (DoM) by using methanol dichloromethane, formic acid (1:9:0.05; v/v) as the mobile phase and Merck HPTLC plates (0.2 mm thickness) precoated with 60F<sub>254</sub> silica gel on aluminum sheet as stationary phase. Retention factors of CINN and DoM were 0.48 and 0.80 respectively.

7. A simple, precise, rapid and selective high performance thin layer chromatography (HPTLC) method has been developed for the simultaneous determination of Diloxanide Furoate (DF) and Thinidazole (TZ) in tablets by using methylene chloride: methanol (9.6:0.25; v/v) as mobile phase and Merck HPTLC plates (0.2 mm thickness) precoated with 60F<sub>254</sub> silica gel on aluminum sheet as stationary phase. Retention factors of DF and TZ were 0.45 and 0.28 respectively.

8. A new simple, precise, rapid and selective high performance thin layer chromatographic (HPTLC) method has been developed for the simultaneous determination of Atenolol (ATL) and Amlodipine (AMLO) in tablets by using methylene chloride: methanol: ammonia solution (25% NH<sub>3</sub>), (8.8:1.3:0.1; v/v) as mobile phase and Merck HPTLC plates (0.2 mm thickness) precoated with 60F254 silica gel on aluminum sheet as stationary phase. Retention factors of ATL and AMLO were 0.33 and 0.75 respectively.
9. A simple, precise, rapid and selective high performance thin layer chromatography (HPTLC) method has been developed for the simultaneous determination of Bromhexine hydrochloride (BH) and Orciprenaline Sulphate (OS) in syrup by using methanol: chloroform: diethylamine (7:3:0.1; v/v) as mobile phase and Merck HPTLC plates (0.2 mm thickness) precoated with 60F254 silica gel on aluminum sheet as stationary phase. Retention factors of BH and OS were 0.72 and 0.32 respectively.

10. A simple, rapid and precise HPTLC method has been developed for the determination of Cinnarizine using methanol: chloroform: formic acid (1:0:0.05; v/v) as the mobile phase and Merck HPTLC plates (0.2 mm thickness) precoated with 60F254 silica gel on aluminum sheet as stationary phase. Retention factor of Cinnarizine is 0.8. Forced degradation study was carried out and the proposed method was applied for the determination of Cinnarizine. Cinnarizine was found to be degrading under oxidizing conditions.

11. The derivative spectrophotometric method was based on the determination of both the drugs at their respective zero crossing point (ZCP). The first order derivative spectra was obtained in methanol and the determinations were made at 233.8 nm (ZCP) of cefpodoxime proxetil for dicloxacillin and 321 nm (ZCP) of dicloxacillin) for cefpodoxime proxetil. The linearity was obtained in the concentration range of 10–80 µg/ml for dicloxacillin and 4–32 µg/ml for cefpodoxime proxetil. The mean recovery was 100.70 ± 0.38 and 99.90 ± 0.36 for dicloxacillin and cefpodoxime proxetil respectively.