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Abstract

Keywords: HPTLC; silica gel G60 - F254; Tablet; Stationary phase (SP)

Introduction

BAL (BAL; 5-[4-carboxyethylcarbamoyl phenylazo] salycylic acid; Figure 1) is a widely used for Ulcerative colitis [1-4].

Literature survey revealed that various analytical methods and pharmacological methods like spectrophotometric [4], Studies of two Novel Sulfasalazine Analogs, Ipsalazide and Balsalazide [5], Sulphasalazine and balsalazide have membrane-stabilizing e ects and cytoprotective action on ethanol-treated rat rectocolon [6], A Meta-Analysis of the E cacy (SSZ) of Sulfasalazine in Comparison with 5-Aminosalicylates (5-ASAs) in the Induction of Improvement and Maintenance of Remission in Patients with Ulcerative Colitis [7], Low dose balsalazide (1.5 g twice daily) and mesalazine (0.5 g three times daily) maintained remission of ulcerative colitis but high dose balsalazide (3.0 g twice daily) was superior in preventing relapses [8] have been reported for the determination of BAL and either individually or combination with some other drugs, but no HPTLC method was reported for estimation estimation of BAL in dosage forms. e review of literature prompted us to develop an accurate, selective and precise estimation method for the estimation of dosage forms.

Experimental

Chemicals and materials

Methanol (A.R. grade), Water (HPLC Grade), Hydrochloric acid (A. R. Grade), Potassium di-hydrogen Phosphate (A. R. Grade), Sodium hydroxide (A. R. Grade), Hydrogen peroxide (A. R. Grade) and Ortho phosphoric acid (A. R. Grade) were used as solvents to prepare the mobile phase.

Chromatographic conditions

e samples were spotted in the form of band width 6 mm with

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standard stock solution of 5000 g/mL was applied on TLC plate with the help of microlitre syringe, using Linomat V sample applicator. The plate was developed and scanned in the above established chromatographic conditions. Peak area was recorded for each concentration of drug; the observations are reported in and calibration curve was plotted as concentration peak area.

Specificity: The peak purity of BAL was tested by correlating the spectra of BAL at the peak start (S), peak apex (A) and at the peak end (E) positions. Correlation between these spectra indicates purity of BAL peak. Thus, it can be concluded that no impurities or degradation products were found with the peaks of standard drug solutions.

Accuracy (% Recovery): The accuracy of the method was determined by calculating recoveries of BAL by method of standard additions. Known amount of BAL (80, 100 and 120%) were added to the pre quantified sample solution, and the amount of BAL was estimated by measuring the peak areas and by fitting these values to the straight-line equation of calibration curve.

Method precision (Repeatability): Standard solutions of BAL (500, 1000 and 1500 ng/spot) were prepared and spectrums were recorded. Absorbance was measured at 289 nm using methanol as a blank. The absorbance of the same concentration solution was measured six times and %RSD was calculated.

Intermediate precision (Reproducibility): Variation of results of three different concentrations (500, 1000 and 1500 ng/spot) within the same day (intra- day), variation of results between days (inter- day) of 500 - 3000 ng/band. The calibration curve for BAL was prepared by plotting area versus concentration. The following equations for straight line were obtained for BAL: Linear equation for BAL: Y = 2.9944x + 41.057; Slope = 2.9944, Intercept = 41.057. Coefficient of correlation = 0.999. The linear range, correlation coefficient, detection limit and standard deviation for BAL are by HPLTC method (Table 1, Figure 3).

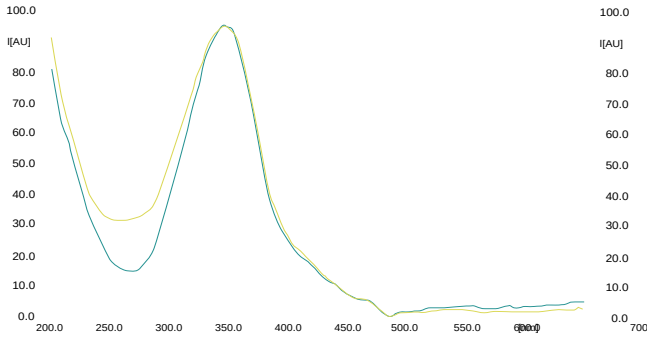
Specificity: The specificity study was carried out to check the interference from the excipients used in the formulations by preparing synthetic mixture containing both the drugs and excipients. The chromatogram showed peaks for the drug without any interfering peak and the recoveries of the drug were above 99% (Figure 4).

Accuracy: Accuracy was determined by calculating the recovery. The method was found to be accurate with % recovery 99.99% - 100.04% for BAL (Table 2).

Precision

a) Repeatability: The % RSD < 2 for BAL which indicate that the method is precise.

b) Intra and inter day precision: Variation of results within the same day (intra- day), variation of results between days (inter- day)



were analyzed. e method was found to be precise with % RSD 0.91-1.02 for inter-day study (n=3) and % RSD 0.49-0.63 for inter-day study (n=3). e % RSD < 2 for BAL indicates that the method is precise. (Table 3).

Limits of detection (LOD) and Limits of Quanti cation (LOQ): Under the experimental conditions used, the lowest amount of drug that could be detected (LOD) for BAL was found to be 0.19 g/ml. e limit of quanti cation (LOQ) for BAL was found to be 1.19 g/ml, with an RSD <2%.

RobustnessAcceptable %RSD values obtained a er making small deliberate changes in the developed. Stability indicating HPLC method indicates that the method is robust for the intended purpose (Table 4).

Solution stability: e sample preparations were analyzed by HPTLC system at regular intervals for 24 hrs as per test procedure. e method is also rugged as there was no change in absorbance up to 24 hours of preparation of solution in Methanol.

Method application

e proposed, developed and validated method was successfully applied to analysis of BAL in their marketed formulation. ere was no interference of excipients commonly found in tablets as described in speci city studies. e assay results obtained were satisfactory, accurate, and precise as indicated by the good recovery and acceptable standard deviation values (Table 5). e good performance of the method indicates that it can be used for the determination of BAL in pharmaceutical formulation.

Conclusion

is developed and validated method for analysis of BAL in pharmaceutical preparations is very rapid, accurate, and precise. e method was successfully applied for determination of BAL in its pharmaceutical tablet formulation. Moreover it has advantages of short run time and the possibility of analysis of a large number of samples, both of which signi cantly reduce the analysis time per sample. Hence this method can be conveniently used for routine quality control analysis of BAL in its pharmaceutical formulation.

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