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Development and Validation of Spectrophotometric methods for the Estimation of Mesalamine in Pharmaceutical Preparations

Srinivasa Rao Narala[1*] and K. Saraswathi[2]

[1]*Department of Chemistry, R.V.R. & J.C. College of Engineering, Guntur, A.P., India*
[2]*Department of Chemistry, S.V. University, Tirupathi, A.P., India*

ABSTRACT

Two new, simple and sensitive spectrophotometric methods (A and B) have been developed for the quantitative determination of Mesalamine in bulk drug and pharmaceutical preparations. Method A is based on the condensation of Mesalamine with p-hydroxy benzaldehyde to form schiff's base, which was an yellow colored chromogen exhibiting λ_{max} at 420 nm. Method B is based on the reaction of Mesalamine with Folin-Ciocalteu phenol's reagent under alkaline conditions forming a blue colored chromogen exhibiting λ_{max} at 750 nm. Beer's law is obeyed in the concentration range of 8-28 µg/ml for method A and 4-24 µg/ml for method B. These methods were extended to pharmaceutical formulations and there was no interference from any common excepients. The results of analysis have been validated statistically and by recovery methods.

Key words: Spectrophotometric determination, p-hydroxy benzaldehyde, Folin-Ciocalteu phenol's reagent.

INTRODUCTION

Mesalamine is chemically known as 5 – amino – 2 – hydroxy benzoic acid, is an anti-inflammatory drug used to treat inflammation of the digestive tract (crohn's disease) and mild to moderate ulcerative colitis. It is a bowl-specific amino salicylate drug that is metabolized in the gut and has its predominant actions there, thereby having fewer systemic side effects. The literature survey reveals that few analytical methods for this drug are reported, which include chromatographic[1-3] and spectrophotometric[4-7] methods. The present investigation has been undertaken to develop two simple and accurate spectrophotometric methods using p-hydroxy benzaldehyde and Folin-Ciocalteu phenol's reagent. Which are essential for routine quality control analysis of pharmaceutical products containing Mesalamine as active constituent.

EXPERIMENTAL SECTION

Apparatus:
A Tech-comp model UV-2301 UV-Visible spectrophotometer with 1 cm matched quartz cells were used for all spectral measurements. Pure drug was obtained from local pharmaceutical laboratory, Chennai and commercial formulations were procured from the market. All the chemicals used were of analytical grade.

Reagents :
P-hydroxy benzaldehyde (4.0 %)
4.0 gr of p-hydroxy benzaldehyde dissolved in 100 ml of methanol
Folin-Ciocalteu phenol's reagent
1: 2 ratio diluted solution with distilled water
Sodium Carbonate (10 %)
10 gr of sodium carbonate dissolved in 100 ml distilled water

Preparation of Standard solution :
Accurately weighed 100 mg of Mesalamine was dissolved in 100 ml of methanol in a volumetric flask to obtain a concentration of 1mg /ml. From this suitable dilutions were made to obtain the working standard concentration of 200 µg/ml for method A and method B.

Preparation of sample solution:
Accurately weighed tablet powder equivalent to 100 mg of drug was transferred in to a 100 ml volumetric flask containing 50 ml of methanol, sonicated for 10 min. and diluted to 100 ml with methanol. The resulting solution was filtered through a whatmann filter paper. This solution was further diluted with methanol to obtain the working standard concentration of 200 µg/ml for method A and method B.

Assay Procedure:
Method A
Aliquots of standard drug solution ranging from 0.5 to 5.0 ml (200 µg/ml) were transferred to a series of 25 ml volumetric flasks. To each flask 2.0 ml of p-hydroxy benzaldehyde was added, kept for 5 min. and the volume was made up to the mark with methanol. The absorbance of yellow colored chromogen was measured at 420 nm against a reagent blank. The amount of drug in the sample was computed from its calibration curve.

Method B
Aliquots of standard drug solution ranging from 0.5 to 5.0 ml (200 µg/ml) were transferred to a series of 25 ml volumetric flasks. To each flask 1.0 ml of Folin-Ciocalteu phenol's reagent and 1.0 ml of sodium carbonate were added, kept for 10 min. and the volume was made up to the mark with distilled water. The absorbance of blue colored chromogen was measured at 750 nm against a reagent blank. The amount of drug in the sample was computed from its calibration curve.

RESULTS AND DISCUSSION

The optical characteristics such as Beer's law limits, Molar absorptivity, Sandell's sensitivity and relative standard deviation were calculated and the results are summarized in Table 1. Regression characteristics like slope, intercept and correlation coefficient were calculated and are presented in Table 1.

Table-1 Optical characteristics of the proposed methods

Parameter	Method A	Method B
λ max	420	750
Beer'law limits (µg/ml)	8-28	4-24
Molar absorptivity (l mol^{-1} cm^{-1})	$0.4931X10^3$	$0.4104X10^3$
Sandell'sensitivity (µg cm^{-2})	0.3106	0.3731
Regression equation (Y= mX+b)		
Slope (m)	0.0351	0.0437
Intercept (b)	-0.0371	-0.0074
Correlation coefficient	0.9985	0.9999
R.S.D.(%)*	0.4807	0.4386
% Range of error (confidence limits)		
0.05 level	± 0.5968	± 0.5445
0.01 level	± 0.9898	± 0.9029

** Mean of five determinations*

Commercial formulations of Mesalamine were successfully analyzed by the proposed methods and the values are presented in Table 2. To evaluate validity and reproducibility of the methods recovery experiments were conducted and the results are summarized in Table 2.

Table-2 Amount of Mesalamine found in formulations by proposed methods

Fomulation	Labeled amount mg/tablet	Amount found *		% Recovery **	
		Method A	Method B	Method A	Method B
Tablet-1	400	399.93±0.046	399.90±0.080	99.88±0.108	99.94±0.074
Tablet-2	800	799.91±0.090	799.91±0.081	100.00±0.068	99.90±0.050

** Mean of five determinations*
*** Mean of three determinations*

CONCLUSION

The proposed visible spectrophotometric methods for the estimation of Mesalamine are simple, sensitive, accurate and can be used for the routine quality control of the drug in bulk as well as in pharmaceutical formulations.

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