Available online at www.derpharmachemica.com

Scholars Research Library

Der Pharma Chemica, 2011, 3(2): 342-346
(http://derpharmachemica.com/archive.html)

ISSN 0975-413X
CODEN (USA): PCHHAX

Development and Validation of Visible Spectrophotometric methods for the Estimation of Mesalamine in Pharmaceutical Dosage Forms

Venugopal R Darak, Aravind B Karadi*, MD Arshad and SajjaAppalraju

Department of Pharmaceutical Analysis, H.K.E.S's College of Pharmacy, Gulbarga(India)

ABSTRACT

Threesimple and sensitive spectrophotometric methods (A, B and C) have been developed forthe quantitative determination of Mesalamine in bulk drug and pharmaceutical preparations.Method A, B are based on the condensation of Mesalamine with p-dimethyl aminobenzaldehyde and p-dimethyl amino cinnamaldehyde to form schiff's bases, to yield a yellow, red colored chromogen and exhibits absorption maxima at 440, 523.5 nm. Method C isbased on the reaction of Mesalamine with Folin-Ciocalteu (phenol's) reagent under alkaline conditions forming a blue colored chromogen and exhibits absorption maxima at 616 nm. Beer's law was obeyed inthe concentration range of 50-250, 20-100 and 10-50µg/ml for method A,B and C respectively. Thesemethods were extended to pharmaceutical formulations and there was no interference from any common excepients. The results of analysis have been validated statistically and by recoverymethods.

Key words: Spectrophotometry, p-dimethyl amino benzaldehyde, p-dimethyl amino cinamaldehyde, FC reagent, Schiffs base.

INTRODUCTION

Mesalamine is chemically known as 5 – amino – 2 – hydroxy benzoic acid[1-4], is an anti-inflammatory drug used to treat inflammation of the digestive tract (crohn's disease) and mild tomoderate ulcerative colitis. It is a bowl-specific amino salicylate drug that is metabolized in thegut and has its predominant actions there, thereby having fewer systemic side effects[5-6]. Theliterature survey reveals that few analytical methods for this drug are reported, which include chromatographic[7],and spectrophotometric methods[8-9]. The present investigation has beenundertaken to develop three simple and accurate spectrophotometric methods using p-dimethyl amino benzaldehyde, p-dimethyl amino cinamaldehyde and Folin-Ciocalteu reagent.

Which are essential for routine qualitycontrol analysis of pharmaceutical products containing Mesalamine as active constituent.

MATERIALS AND METHODS

Instrument

All spectral measurements were made on Shimadzu 1800 UV-Visible spectrophotometer with 1 cm matched quartz cellswere used.

Materials

Pure drug of Mesalamine was obtained from Cosmo pharmaceutical Pvt Ltd, Goa and commercial formulations were procured from local market. All thechemicals used were of analytical grade.

Reagents

Alcoholic solution of P dimethyl amino benzaldehyde (0.5 %w/v)
Alcoholic solution of P-dimethyl amino cinamaldehyde (0.5 %w/v)
Folin-Ciocalteu reagent (1N) was diluted to 2N with distilled water
Aqueous solution of Sodium Hydroxide (1N)
Aqueous solution of Hydrochloric acid (0.1N)

Preparation of Standard solution :

Weigh accurately 100 mg of Mesalamine and transferred in to 100 ml volumetric flask and dissolve in 100 ml of 0.1N HCl to obtain a concentration of 1mg /ml. From this suitable dilutions were made to obtain theworking standard concentration of 100µg/ml.

Preparation of sample solution:

Two brands ofcommercially available tablets were taken, twenty tablets each weighing 400mg were weighed and powered. A tablet powder equivalent to 100 mg was weighed accurately and transferred in to 100 ml volumetric flask containing 50 ml of 0.1N HCl, the flask was sonicated for 5 min, the volume was made up to mark with 0.1N HCl, and the solution was filtered through whatmann filter paper 41, from the above stock solution, working standard solution of 100mg/ml were prepared by further dilution with 0.1N HCl, the above procedure was applied for analysis.

Assay Procedure:

Method A

Aliquots of standard drug solution ranging from 0.5 to 2.5 ml (1ml=1000µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 1 ml of P- dimethyl amino benzaldehyde, 1ml of concentrated HCl were added and kept for 20min heating at 60^{o}c and the volume was made up to the mark with water. The absorbance of yellow colored chromogen was measured at 440 nm (fig 1) against a reagent blank. The amount of drugpresent in the sample was computed from its calibration curve (fig 2).

Method B

Aliquots of standard drug solution ranging from 0.2 to 1.0 ml (1ml=1000µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 1 ml of P-dimethyl amino cinamaldehyde,

1ml of concentrated HCl were added and kept for 20min heating at 40^{o}c and the volume was made up to the mark with water. The absorbance of red colored chromogen was measured at 523.5 nm (fig 3) against a reagent blank. The amount of drugpresent in the sample was computed from its calibration curve (fig 4).

Method C

Aliquots of standard drug solution ranging from 1 to 5 ml (1ml=100µg/ml) were transferred in to aseries of 10 ml volumetric flasks. To each flask 1.0 ml of Folin-Ciocalteu reagent and1.0 ml of 1N sodium hydroxide were added, kept for 10 min to develop the color and the volume was made up to themark with distilled water. The absorbance of blue colored chromogen was measured at 616 nm(fig 5) against a reagent blank. The amount of drug present in the sample was computed from its calibrationcurve (fig 6).

RESULTS AND DISCUSSION

The optical characteristics such as Beer's law limits, Molar absorptivity, 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 arepresented in Table 1.Commercial tablets of Mesalamine were successfully analyzed by the proposed methodsand the results are presented in Table 2. Comparison of the results obtained with the proposed and UV methods for dosage forms (Table 2) confirms the suitability of these methods for Pharmaceutical dosage forms. To evaluate validity and reproducibility of the methodsrecovery experiments were conducted and the results are summarized in Table 2. The other active ingradients and excipients usally present in pharmaceutical dosage forms did not interfere.

Table-1 Optical characteristics and Precision

Parameters	Method A	Method B	Method C
λmax (nm)	440	523.5	616
Beer's law limits (µg/ml)	50-250	20-100	10-50
Molar absorptivity (L mol^{-1} cm^{-1})	5.1454 X 10^{2}	1.3093X10^{3}	2.4042X10^{3}
Regression equation (Y = a+bc)			
Slope (b)	0.0033	0.0084	0.0197
Intercept (a)	0.0218	0.0044	0.0001
% R S D	0.1992	0.1953	0.2025
Correlation coefficient (r)	0.9998	0.9999	0.9999
Limit of Quintitation (LOQ)	0.3350	0.0084	0.6067
Limit of Detection (LOD)	0.1105	0.1848	0.2002
Range of error**			
Confidence limit with 0.05 level	1.0573 X 10^{-3}	1.0576 X 10^{-3}	4.8960 X 10^{-4}
Confidence limit with 0.01 level	1.5648 X 10^{-3}	1.5648 X 10^{-3}	7.2436 X 10^{-4}

Y=bC+a were C is the concentration of Mesalamine in µg/ml and Y is absorbance unit
*** for eight measurements*

Table-2 Evalution of Mesalamine in Tablet Dosage formulations

	Label Claim (mg)	Amount of drug obtained by proposed methods (mg)			Reference method UV	% Recovery*			% Recovery
		A	B	C		A	B	C	UV
M_1	400	398.14	398.64	399.92	398.64	99.49	99.37	99.46	99.37
M_2	400	399.92	399.58	398.74	398.74	99.46	99.54	99.28	99.28

**mean of six determinations, M_1= Mesacol (UnipharmaPvt Ltd),M_2= Walasa(Wallace PvtLtd)*

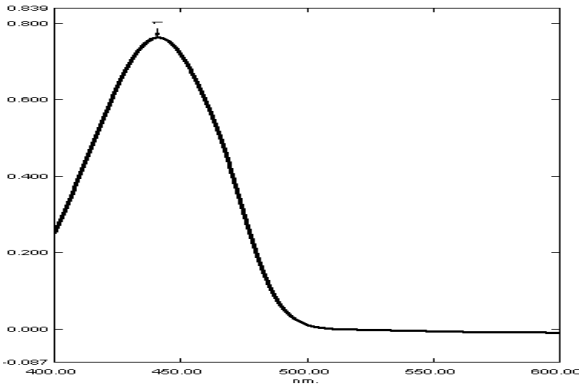

Fig 1 Absorption Spectrum of MES by PDAB

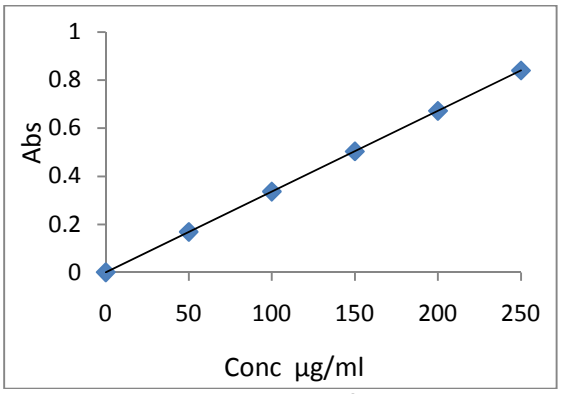

Fig 2Calibration Curve of MES by PDAB

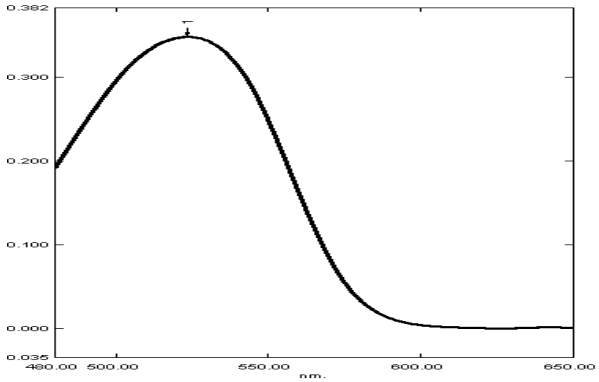

Fig 3 Absorption Spectrum of MES by PDAC

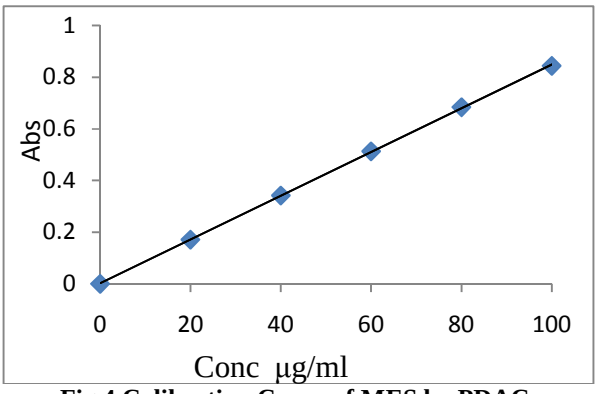

Fig 4 Calibration Curve of MES by PDAC

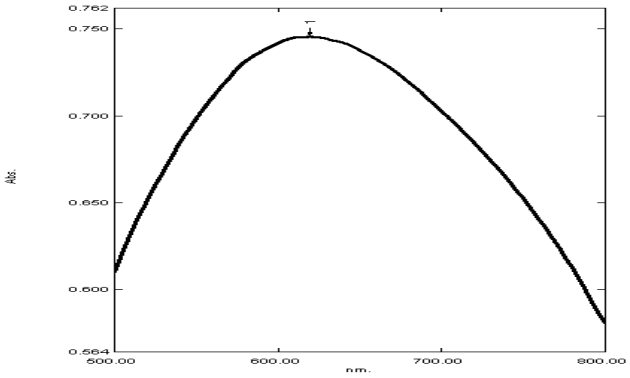

Fig 5 Absorption Spectrum of MES by FC

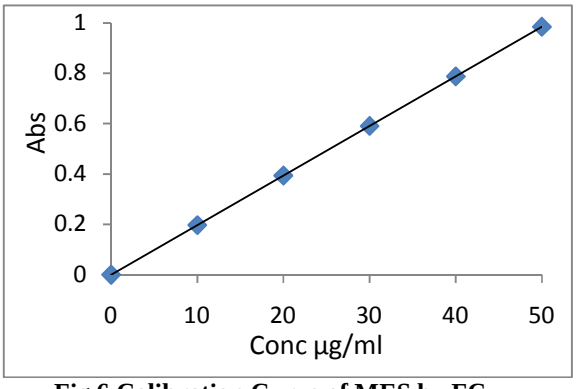

Fig 6 Calibration Curve of MES by FC

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.

Acknowledgements
The Authors are thank full to Cosmo Pharma Pvt Ltd, Goa for providing gift sample of drug for research and Principal, Management, HKES's College of Pharmacy, Gulbarga Karnataka (India) for providing necessary laboratory facilities to carry out the present work.

REFERENCES

[1] O' Neil. M.J;The Merck Index. An Encyclopedia of Chemicals, Drug and Biologicals. Merck& Co. Inc, **2006**, 14, 613.
[2] Sweetman.S.C; Martindale the complete drug reference.Pharmaceutical press, London (U.K), **2007**, 35, 1573.
[3] Aleka K. Dashand Harry G; Analytical profile of drug substances and excipients, brittain, **1998**, 25, 209-242.
[4] The United State Pharmacopeial Convection, (Ed.), United State Pharmacopeia. NF, Asian Edition, **2007**, 3, 2584.
[5] Bertam G Katzung; Basic and Clinical Pharmacology, Mc. Graw Hill, Singapore **2007**, 9, 1030.
[6] K.D.Tripathi; Essentials of Medical Pharmacology,Jaypee Brothers Medical Publishers (P) Ltd, New Delhi, **2004**, 5, 620-621.
[7] J.A. Rafael, R.J. Jose, R. Casagrande, R.S. Georgetti, M.F. Borin, M.J. Vieira Fonseca, *Brazilian Journal of Pharmaceutical Sciences*., **2007**, 43(1), 97.
[8] K.M .Patel, C.N. Patel , B.Panigrahi, A.S. Parikh , H.N.Patel, *J Young Pharmacists*.,**2010**, 2, 284-288.
[9] Navyasloka. S, B.M.Gurupadayya and C.H. Aswinkumar, *Der PharmaChemica*., **2010**, 2(4), 389-396.