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NOTE

Spectrophotometric Determination of Mesalamine in Pure and Pharmaceutical Formulations

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Two simple and sensitive visible spectrophotometric methods for the assay of mesalamine (MA) have been developed. In Method-A, mesalamine forms a violet coloured complex in alkaline medium with 1,2-naphthaquinon-4-sulphonic acid sodium salt reagent. Method-B is based on the oxidation of mesalamine with iron(III), and subsequent chelation of iron(II) with 1,10-phenanthroline (Phen) to form a red coloured complex (λ_{max} 515 nm); the colour so produced is perfectly stable for 24 h. Method A and B obey the Lambert-Beer's law in concentration ranges employed for this method. The results of analysis of both the methods have been validated statistically and by recovery study. The proposed methods are selective, simple and economical for the determination of mesalamine.

Mesalamine (MA) is chemically 5-amino-2-hydroxybenzene-1-carboxylic acid or 5-amino salicylic acid which acts on colon and rectum (anti-inflammatory). It is commercially available in tablets, capsules as well as rectal solution and suspension. Mesalamine ($C_7H_7O_3N$) is official in USP-23-NF-18 (Supplement-VII)[1] and is estimated by HPLC method[1]. Literature survey[1–4] indicates that only HPLC method is used for assay. We have developed simple colorimetric methods for the estimation of MA in its dosage form, since these methods are found to be much reliable, precise and accurate techniques for the assay and have not been used so far for this purpose. The development of these methods and their applications for routine assay of MA in pure and pharmaceutical formulation are described. In method-A, MA forms a violet coloured complex in alkaline medium with 1,2-naphthaquinon-4-sulphonic acid sodium salt reagent, whereas method-B is based on the oxidation of MA by Fe(III) to Fe(II) which subsequently reacts with 1,10-phenanthroline to form a red coloured complex. This paper presents a simple, economical method for routine analysis of MA in its formulations.

A Shimadzu UV/VIS recording spectrophotomet (model UV 2100) with 1 cm (quartz) cells and wavelength accuracy of ± 0.5 nm was used for measurements of absorbance. All reagents and chemicals used were of A.R. grade.

Aqueous solutions 1,2-napthaquinon-4-sulphonic acid sodium salt (0.1%), sodium hydroxide (1 N), hydrochloric acid (0.1 M), ferric chloride (0.003 M), 1,10-phenanthroline (0.01 M), orthophosphoric acid (0.025 M) were prepared.

Preparation of standard drug solution: Stock solution of mesalamine (1 mg/mL) was prepared by dissolving 100 mg in 100 mL volumetric flask in 0.1 M HCl for methods A and B. The working standards were prepared by suitable dilutions of the stock solution with distilled water, 100 μg/mL (Method A) and 20 μg/mL (method B).

Preparation of sample solution: Twenty tablets were crushed well separately, and the analytical sample was made into fine powder. Powder or suspension equivalent to 100 mg was weighed for each method, and the solutions for these methods (A and B) were prepared as under standard solution preparation and filtered if insoluble residue present.

Procedures

Method A: Aliquots of standard mesalamine solution 0.5–1.5 mL (100 μg/mL) were placed in a series of 10 mL volumetric flasks. A volume of 2 mL (0.1%) 1,2-naphthaquinon-4-sulphonic acid sodium salt was added in each and finally diluted up to the mark with sodium hydroxide (1 N) shaken for 2 min and absorbances were measured at λ_{max} 540 nm against a reagent blank. The amount of mesalamine present was calculated from calibration graph. A plot of absorbance *vs.* drug concentration obeys Lambert-Beer's law in the concentration range of 10 to 50 μg/mL.

Method B: Aliquots of the standard solution 0.25–2.0 mL (20 mg/mL) of mesalamine, 1.5 mL of $FeCl_3$ [Iron(III)] and 2.0 mL of 1,10-phenanthroline solution were successively added to a series of 20 mL graduated test tubes. The contents of each tube were mixed well and heated on a boiling water bath for 30 min; the tubes were cooled to room temperature and 2 mL of orthophosphoric acid was added to each tube and the volume was brought to 20 mL with distilled water. The absorbance was measured against a reagent blank at 515 nm. The coloured species was stable for 24 h. The amount of mesalamine present was calculated from calibration graph. A plot of absorbance *vs.* drug concentration obeys Lambert Beer's law in the concentration range 1.0 to 10.0 μg/mL.

Literature survey indicates that only HPLC is used for assay of MA. The proposed methods were found to be simple, accurate and rapid for routine simultaneous estimation of mesalamine in formulation. For the calibration plot for these two methods, the precision and accuracy were found by analysing six replicate samples containing known amounts of the drug and the results are summarised in Table-1 and 2.

Tests for accuracy of the methods and recovery experiments were performed by adding known amounts of pure drug to preanalysed formulation and the present recovery values obtained are listed in Table-2. Recovery experiments indicated the absence of interferences from the commonly encountered pharmaceutical additives and excipients. The values of standard deviation were satisfactorily low and recovery was close to hundred per cent indicating the reproducibility and accuracy of the method.

Thus the proposed two methods are simple and selective with reasonable precision and accuracy. These can be used for the routine determination of mesalamine in Quality Control Analysis. The results are given in Tables-1 and 2.

TABLE-1
ANALYSIS OF PHARMACEUTICAL FORMULATIONS BY PROPOSED METHODS

Pharmaceutical Preparation	Labelled amount MA (mg)	Amount found by the proposed method MA (mg)		Standard Deviation	
		A	B	A	B
Tablet					
*T_a	400	398.12	398.75	±0.8628	±0.9291
*T_b	400	401.20	398.02	±0.7718	±0.6549
Suspension					
*S_a	4	3.80	4.25	±0.7319	±0.8932
*S_b	4	4.10	4.15	±0.5123	±0.9907

MA = Mesalamine; T = Tablets; S = Suspension; * Mean of six readings

TABLE-2
RECOVERY STUDY DATA

Pharmaceutical Preparation of MA	Amount Labeled MA (mg)	Amount Added MA (mg)	Amount found of MA proposed method (mg)		% Recovery of MA	
			A	B	A	B
Tablet						
*T_a	400	40	438.10	441.82	99.57	100.41
*T_b	400	40	437.96	438.38	99.54	99.63
Suspension						
*S_a	4	2	5.78	5.91	96.33	98.50
*S_b	4	2	5.85	6.32	97.67	105.33

*mean of six readings.

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