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UV Spectrophotometric Method Development and Validation of Mesalazine in Bulk and Solid Dosage Form

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Abstract
Objective: A new, simple, sensitive, precise and reproducible UV spectroscopic method was developed for the estimation of Mesalazine in bulk and Tablet Formulation. **Methods:** The UV spectrum of Mesalazine in Triflouroacetic acid (0.1%) showed λ max at 300nm. Beer's law is valid in the concentration range of 10-50 µg/ml. This method was validated for linearity, accuracy, precision, ruggedness, and robustness. **Results:** The method has demonstrated excellent linearity over the range of 10-50µg/ml with regression equation y = 0.017x + 0.0637 and regression correlation coefficient r^2= 0.9953. Moreover, the method was found to be extremely sensitive with LOD (5.67 µg/ml) and LOQ (17.19µg/ml). **Conclusion:** Depending on results the given method can be successfully applied for assay of Mesalazine in tablet formulation.

Keywords
Mesalazine, UV spectroscopy, method development and validation, Triflouroacetic acid water 0.1%, Tablet Formulation.

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INTRODUCTION:
Mesalazine is also called as mesalamine. It (5-aminosalicylic acid) is an anti-inflammatory agent, structurally related to the salicylates, which is active in inflammatory bowel disease and active ulcerative proctitis. It is a tan to pink crystalline powder, relatively insoluble in chloroform, ether, n-hexane and ethyl acetate and freely soluble in dil. HCl and alkali hydroxides. Mesalazine is available in tablet dosage forms (400 mg) and is an official drug of USP. The chemical structure of Mesalazine is shown in Figure 1.

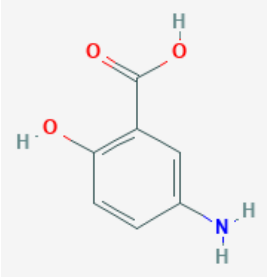

Fig 1: Chemical Structure of Mesalazine

Literature survey reveals that, some study about HPLC determination of Mesalamine and its degradation metabolite in plasma determination of 5-aminosalicylic acid in pharmaceutical formulation by differential pulse voltammetry. To the best of our knowledge, there is no UV method for the analysis of MSZ in pharmaceutical formulations has been reported in literature survey.

The aim of this study is to develop a fast, simple, reliable, selective, and inexpensive UV spectrophotometric method for the determination of MSZ in bulk drug & commercial pharmaceutical-formulations as tablet and its validation.

MATERIALS AND METHOD:

Materials

Mesalazine was taken as gift sample from Airis, Hyderabad. TFA 0.1% water was taken from local market. Other Analytical Grades Chemicals are used.

Instruments:

UV/Visible double beam spectrophotometer Systronic 2201. Standard cuvettes having 10 mm of path length are used for analysis. Ultra Sonicator (micro clean-103) was used to sonicate the formulation sample. Drug sample was weighed by using an electronic analytical balance (Shimadzu AY220). Sonicator (Microclean-1103), UV-Visible (Labman).

Experimental:

Preparation of standard stock solution:

Accurately weighed 10mg of Mesalazine transferred to 100ml volumetric flask. It was dissolved 5ml in 0.1% TFA water & sonicated for 5 minutes. The volume was made up to mark with same diluent to make up final strength.

Procedure for plotting calibration curve:

For calibration curve in a series of 10 ml volumetric flasks, 1-5 ml of standard solution was pipetted out separately. The volume was completed to the mark using 0.1% TFA water. The absorbance was measured at wavelength 300 nm against blank solution.

Analysis of Mesalazine in tablet Formulation:

10mg equivalent Mesalazine Tablet was weighed and transferred to the 100ml volumetric flask and dissolved in TFA water 0.1% as a solvent. After that sonicated for 5min and vortex for 2min. 4 ml of above solution was pipetted out and transferred to the 10ml volumetric flask and make up the volume upto the mark with same solvents and analysed at 300 nm. Calculate the % purity of Mesalazine.

RESULTS AND DISCUSSION:

The absorption spectrum shows λ max of Mesalazine at 300nm.

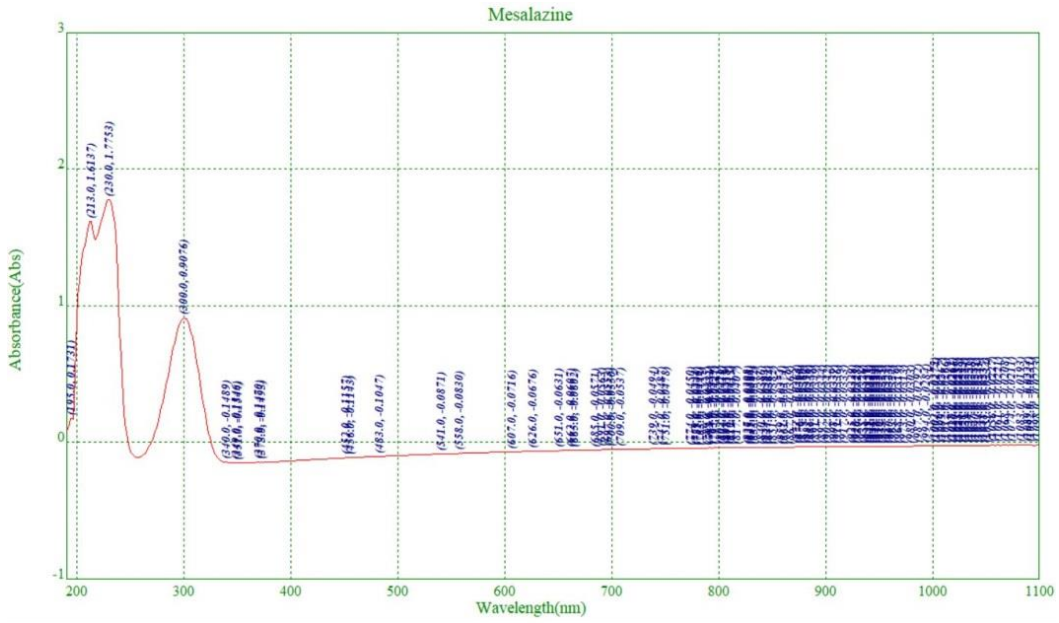

Figure 2: UV spectrum of Mesalazine

The proposed method was validated according to ICH Q28 R1 guidelines for validation of analytical procedure.[4-8]

Linearity:

Five different concentrations of Mesalazine were prepared and analysed at wavelength 300 nm. The regression coefficient was found to be 0.9953. The absorbance was found in limit i.e. 10-50. Hence the analysed parameter was found to be validated (table 1).

Table 1: Results of Linearity

Sr.no.	Concentration(µg/ml)	Absorbance
1	10	0.215
2	20	0.414
3	30	0.601
4	40	0.732
5	50	0.907

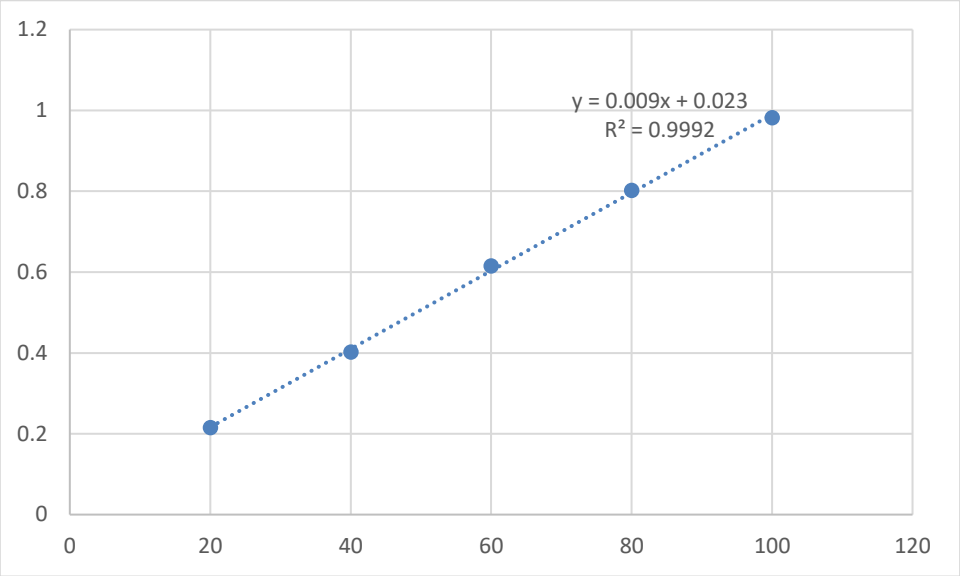

Figure 3: Calibration curve for Mesalazine (Conc. vs. Abs.)

Table 2: Optimization parameters of Mesalazine

Parameters	Method values
Maximum Wavelength	300nm
Beer's Law	10-50µg/ml
Correlation Coefficient (r^2)	0.9953
Regression Equation	y = 0.017x + 0.0637
Slope (m)	0.017
Intercept (c)	0.0637

Accuracy:

The concentration 40, 50, 60µg/ml was taken as 80,100,120% and % recovery was found to be in range 99%-101%. Hence the parameter was found to be validated.

Table 3: Results of Accuracy

Name of Drug	Recovery Level in %	Concentration	Amount Recovered	% recovery with SD
Pentasa	80	40µg/ml	40.02	100.02±0.25
	100	50µg/ml	50.03	100.03±0.7
	120	60µg/ml	59.01	99.01±0.29

Range:

Range is an interval between highest and lowest concentration limit of the analyte i.e. 10-50 µg/ml.

Precision:

In precision intra-day and inter-day precision were performed at concentration (50µg/ml). The obtained results were found within limit i.e., less than 2% RSD.

Table 4: Results of Intra-day Precision

Sr. no.	Concentration	Absorbance
1		0.9076
2		0.9137
3	(50µg/ml)	0.9135
4		0.9128
5		0.913
6		0.9122
	SD:	**0.002618**
	%RSD:	**0.29**

Table 5: Results of Inter-day precision

Sr.no.	Concentration	Absorbance (Day1)	Absorbance (Day2)
1		0.9076	0.9082
2		0.9137	0.9142
3	(50µg/ml)	0.9135	0.9135
4		0.9128	0.9135
5		0.913	0.9142
6		0.9122	0.9102
	SD:	**0.002618**	0.002503
	%RSD:	**0.29**	0.274339

Limit of Detection (LOD): The limit of detection (LOD) is the lowest concentration of an analyte in a sample that can be detected but not necessarily to be determined quantitatively under specified experimental conditions. The limit of detection, as calculated statistically for mesalamine was found to be to be 5.37µg/ml.

Limit of Quantification (LOQ):
The limit of quantification (LOQ) is the lowest concentration of an analyte in a sample that can be quantitatively determined within an acceptable level of accuracy and precision under the stated operational conditions of the method. The limit of quantification was found to be 17.19µg/ml.

Ruggedness
The change in analyst with same concentration and environmental condition did not affect the results.

Table 6: Results of Ruggedness

Concentration	Absorbance (Analyst1)	Absorbance (Analyst2)
20µg/ml	0.413	0.414
	0.412	0.413
	0.413	0.413
	0.411	0.414
	0.412	0.413
	0.412	0.412
Average:	**0.4125**	**0.4131**
SD:	**0.000753**	**0.000725**

Robustness:
The change in wavelength (300nm and 305nm) and concentration (30µg/ml) didn't affect the results.

Table 8: Results of Robustness

Wavelength	300nm	305nm
Concentration	30µg/ml	30µg/ml
Absorbance	0.601	0.611
	0.602	0.612
	0.602	0.612
	0.602	0.613
	0.601	0.611
	0.603	0.612
Average	**0.601833**	**0.611833**
SD	**0.000753**	**0.000735**

Assay:

The assay was performed by using Pentasa at concentration 40µg/ml. The % purity was found to be 99.57

Table 9: Results of Assay

Formulation	Labeled Amount	Amount obtained	% purity
Pentasa	500	499.55	99.57%

CONCLUSION:

An analytical UV spectrophotometric method was developed & validated thoroughly for quantitative determination of Mesalazine in bulk drug and tablet formulation. The presented method was found to be simple, precise, accurate, rugged, and reproducible and gives an acceptable recovery of the analyte, which can be directly easily applied to the analysis of pharmaceutical Tablet formulation of Mesalazine.

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