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# Colorimetric Estimation of Mesalamine in Bulk and Pharmaceutical Dosage Forms.

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## Abstract
Three simple and sensitive colorimetric methods (A, B and C) have been developed for the quantitative determination of Mesalamine in bulk drug and pharmaceutical preparations. Method A, B are based on the oxidation followed by complexation of iron with 1, 10, phenanthroline, 2 2' bipyridine to form red colored chromogens and exhibiting absorption maxima at 510.5, 522.5 nm. Method C is based on oxidative coupling reaction of Mesalamine with 3-methyl benzothiazolinone hydrazone (MBTH) reagent under alkaline conditions forming a green colored chromogen and exhibits absorption maxima at 586.8 nm. Beer's law was obeyed in the concentration range of 2-10 and 4-20 µg/ml for method A, B and C respectively. These methods were extended to pharmaceutical formulations and there was no interference from any common excipients usually present in the pharmaceutical formulations. The results of analysis have been validated statistically and by recovery methods.

## Key Words
Colorimetry, 1, 10 phenanthroline, 2.2'bipyridine, MBTH, chromogen.

## 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 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[5-6]. The literature survey reveals that few analytical methods for this drug are reported, which include chromatographic[7], and spectrophotometric methods[8-9]. The present investigation has been undertaken to develop three simple and accurate calorimetric methods using1, 10 Phenanthroline, 2.2'bipyridine and MBTH reagent. Which are essential for routine quality control 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 cells were used.

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

### Reagents:
Alcoholic solution of 1, 10 phenanthroline (0.1 %w/v). Alcoholic solution of 2 2' bipyridine (0.1 %w/v). Aqueous solution of MBTH (0.5 %w/v). Aqueous solution of Ferric chloride (0.1 and 1% w/v). 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 the working standard concentration of 100µg/ml.

### Preparation of sample solution:
Two brands of commercially 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 0.2 to 1.0 ml (1ml=100µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 0.5ml of $FeCl_3$(0.1%w/v), 0.5ml of 1,10 phenanthroline(0.1%w/v)were added and heated for10min at $40^oc$ and the volume was made up to the mark with 0.1N HCl. The absorbance of red colored chromogen was measured at 510.5 nm (Fig 1) against a reagent blank. The amount of drug present 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=100µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 0.5ml of $FeCl_3$(0.1%w/v), 0.5ml of 22' bipyridine were added and heated for 10min at $40^oc$ and the volume was made up to the mark with 0.1N HCl. The absorbance of red colored chromogen was measured at 522.5 nm (Fig 3) against a reagent blank. The amount of drug present in the sample was computed from its calibration curve (Fig 4).

**Method C**

Aliquots of standard drug solution ranging from 0.4 to 2.0 ml (1ml=100µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 1ml of $FeCl_3$(1%w/v), 0.8ml of MBTH were added, kept aside for 10 min to develop the color and the volume was made up to the mark with distilled water. The absorbance of blue colored chromogen was measured at 586.8 nm (Fig 5) against a reagent blank. The amount of drug present in the sample was computed from its calibration curve (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 are presented inTable 1. Commercial tablets of Mesalamine were successfully analyzed by the proposed methods and 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 methods recovery experiments were conducted and the results are summarized in Table 2. The other active ingredients and excipients usually present in pharmaceutical dosage forms did not interfere.

## Conclusion

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

## Acknowledgements

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## References

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

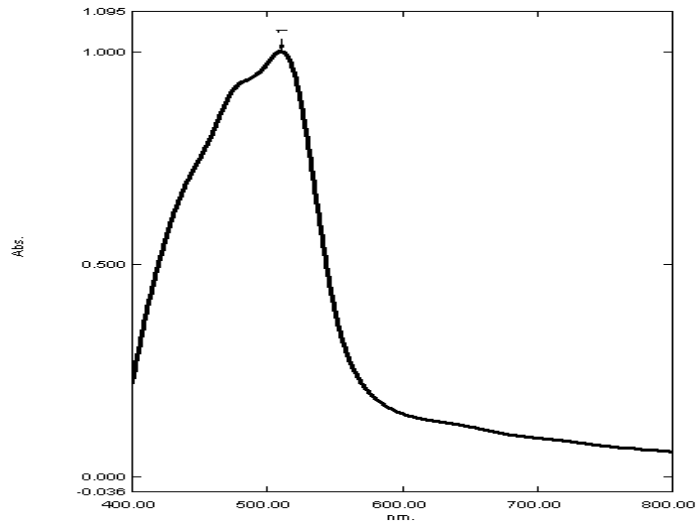


**Fig 1:** Absorption Spectrum of MES by 1, 10 PTL.

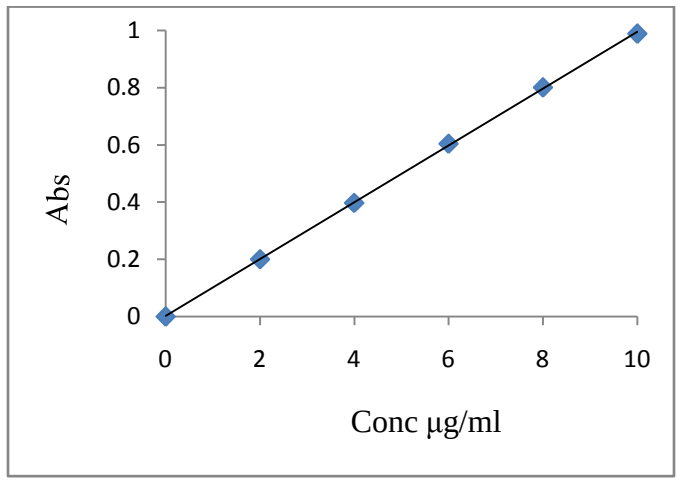


**Fig 2:** Calibration Curve of MES by 1, 10 PTL.

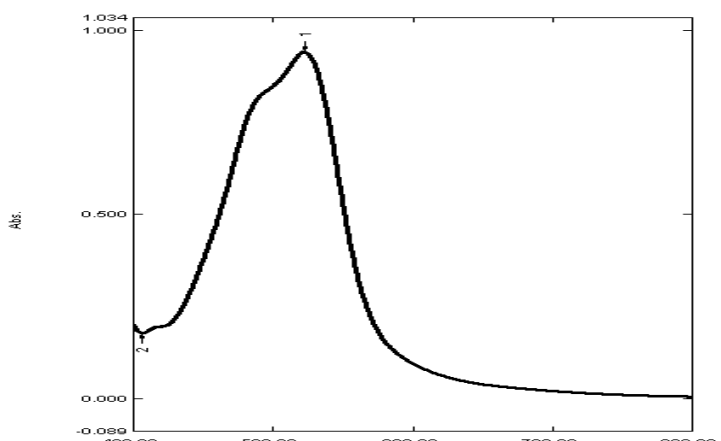


**Fig 3**: Absorption Spectrum of MES by 2.2.BPD

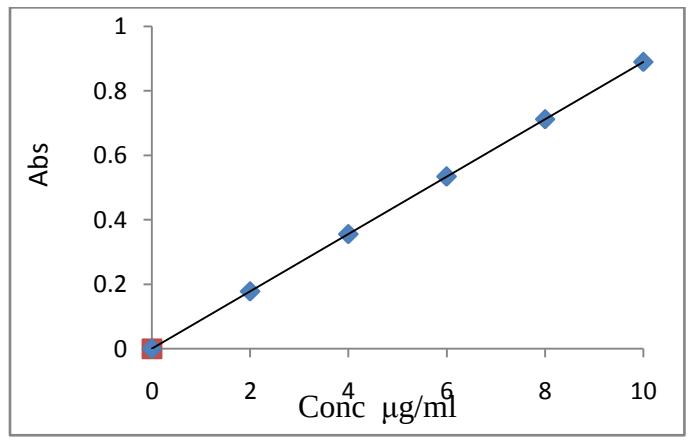


**Fig 4:** Calibration Curve of MES by 22' BPD.

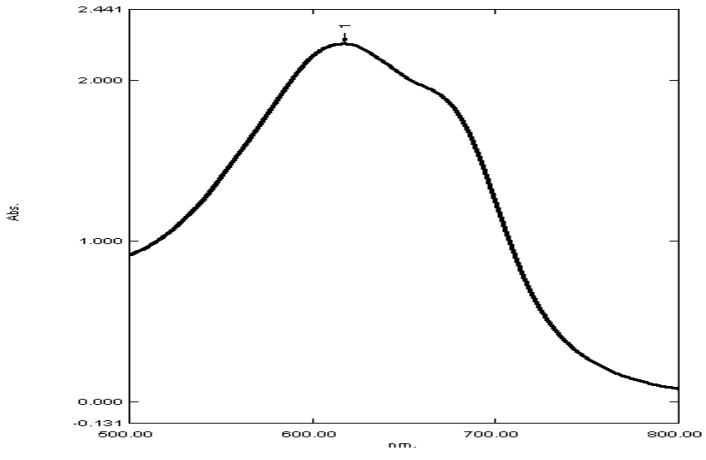


**Fig 5:** Absorption Spectrum of MES by MBTH.

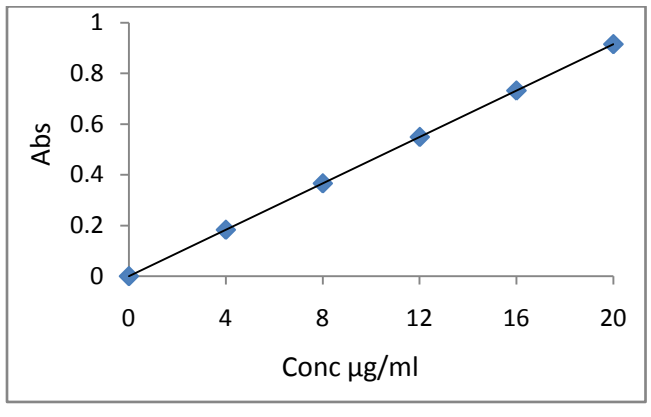


**Fig 6:** Calibration Curve of MES by MBTH.

**Table 1:** Optical characteristics and Precision.

| Parameters | Method A | Method B | Method C |
|---|---|---|---|
| λ max (nm) | 510.5 | 522. 5 | 586.8 |
| Beer's law limits (μg/ml) | 2-10 | 2-10 | 4-20 |
| Molar absorptivity (L $mol^{-1}$ $cm^{-1}$) | 7.0025 X $10^3$ | 1.5433X$10^4$ | 1.3622X$10^4$ |
| Regression equation (Y = a+bc) | | | |
| Slope (b) | 0.0081 | 0.0989 | 0.0197 |
| Intercept (a) | 0.0218 | 0.0054 | 0.0890 |
| % R S D | 0.1824 | 0.1655 | 0.0001 |
| Correlation co-efficient (r) | 0.9972 | 1.0007 | 1.0028 |
| Limit of Quantitation (LOQ) | 1.2295 | 0.1011 | 0.0600 |
| Limit of Detection (LOD) | 0.4057 | 0.0333 | 0.0198 |
| Range of errors** | | | |
| Confidence limit with 0.05 level | 1.0576 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=a+bc were C is the concentration of Mesalamine in μg/ml and Y is absorbance unit. ** For eight measurements.

**Table 2:** Evaluation of Mesalamine in Tablet Dosage formulations.

| | **Label Claim (mg)** | **Amount of drug obtained by proposed methods (mg)** | | | **Reference method UV**** | **% Recovery*** | | | **Reference method**** |
|---|---|---|---|---|---|---|---|---|---|
| | | **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 |

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