WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 7.523
Volume 6, Issue 14, 320-327. Research Article ISSN 2277– 7105

SPECTROPHOTOMETRIC ESTIMATION OF SULPHASALAZINE IN PHARMACEUTICAL FORMULATIONS

Mallapu E. Rani

Associate Professor and H.O.D. of Chemistry, Rayalaseema University, Kurnool 518002, A.P. India.

Article Received on
11 Sep. 2017,
Revised on 02 Oct. 2017,
Accepted on 22 Oct. 2017
DOI: 10.20959/wjpr201714-9682

***Corresponding Author**
Dr. Mallapu E. Rani
Associate Professor and H.O.D. of Chemistry, Rayalaseema University, Kurnool 518002, A.P. India.

ABSTRACT

The present validation of a developed a new spectrophotomctric estimation of sulphasalazine in pharmaceutical Formulations In this method, the oxidation of sulphasalazine drug by a known excess amount of cerium IV sulphate in acid medium, the unreacted cerium IV sulphate was treated with Iron II Sulphate, After 5 minutes the result ant Iron III slphate solution was treated with (1M) Ammonium thiocyanate, immediately it forms blood red colour of Iron III sulphate thiocyanate drug complex. This blood-red coloured complex formed under standardized conditions was measured at 545 nm against reagent blank Beer's law was obeyed for 50-250 μg/ml of sulphasalazine Results of analysis were validated statistically and recovery studies. The procedures described were successfully applied to the estimation of sulphasalazine in pharmaceutical formulations.

KEYWORDS: Sulphasalazine spectrophotometric cerium IV sulphate, Iron III sulphate (ammonium ferrous sulphate Solution) Ammonium thiocyanate solution.

INTRODUCTION

Sulphasalazine chemically (3E)-6-OXO-3 [(4-pyridin 2-sulfamoyl) phyenyl] hydrazinylidene) cyclohexa 1,4 diene 1 carboxylic acid, sulphasalazine is soluble in distilled water.

It is commercially available in different trade names in the market such as

1. SAAZ, 500 mg Tablets
2. SAAZ –DS 1000mg Tablets

3. SALAZAL 500 mg – Tablets
4. SAZO-EN 500 mg Tablets etc.

It is used in treatment of anti inflammatory action in the colon.

Survey of literature reveals that various methods were reported for the estimation of sulphabalazine in tablets and bulk drug powder. This drug was describes analysis of liquid chromatographic method, High speed liquid chromatograhic method and FTRaman Spectroscopy (FTRS) method ELISA METHOD, This method is based on polyclonal antibodies and characterization.

The present investigation was under taken with the aim of developing new, simple, rapid and accurate method. Hence this spectrophotometric method based on a charge transfer complexation reaction.

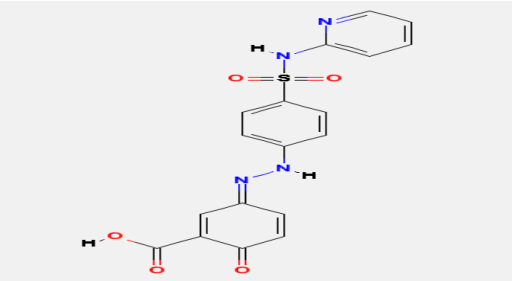

Fig: 3.2.1: Structure of sulphasalazine.

EXPERIMENTAL

APPARATUS

ELICO UV Visible double beam spectrophotometer with 10 nm matched quartz cuvettes used for absorbance values of the drug solution. This instrument provides a unique monochromatic design and a varieties of microprocessor controlled features to give fast and accurate, spectrophoto metric measurements.

Reagents

All chemicals were of analytical reagent grade. Double distilled water was used through out the investigation.

1. Standard solution of sulphasalazine solution

An accurately weighed 50 mg of pure sulphasalazine solution is dissolved in double distilled water and then volume is adjusted to 50 ml with double distilled water and then volume is adjusted to 50 ml with double distilled water. The stock solution was further diluted to get working concentration of 50 μ g/ml.

2. Cerric Ammonium Sulphate (0.05M): 2.9826 g of AR cerric Ammonium Sulphate is dissolved in double distilled water and the resulting solution is made up to the mark in the 100 ml standard flask with double distilled water.

3. Ammonium Ferrous Sulphate Solution(0.02M): 0.7842 g of AR Ammonium ferrous sulphate is dissolved in distilled water and the solution is made up to the mark in the 100 ml standard flask with distilled water.

4. Ammonium thio-cyanate (1M): 7 g of AR Ammonium thio-cyanate is dissolved in double distilled water and the resulting solution is made up to the mark in the 100 ml standard flask with double distilled water.

5. Hydrochloric Acid Solution (5N): Hydrochloric acid solution (5N) is prepared by diluting the requisite volume of concentrated AR hydrochloric acid (Ranbaxy make) with distilled water.

6. Spectrophotometry

The estimation of sulphasalazine by cerimetric method. In this method is based on the oxidation of the drug by a known excess amount of cerric IV sulphate in acid and oxidation product. When unreacted Ce IV sulphate, it oxidized from Iron II sulphate to Iron III sulphate After '5' minutes the Iron III sulphate solution was treated with (1M) ammonium thiocyanate, immediately, it forms blood-red colored complex. i.e, Iron III sulphate thiocyanate complex solution and this coloured solution was more stable for more than 24 hrs. This sample solution was measured at 545 nm against a blank solution. The blank were prepared for this study, the reagent blank containing optimum concentrations of the reagents expect drug. The absorbance was found to decrease linearly with increasing concentrations of sulphasalazine and this forms the basis for the determination of drug. Finally the estimation of the drug was made through the calibration curve.

Reaction scheme showing formation of measured color

Drug + Ce(IV) ⟹ oxidation product + unreacted Ce(IV) + Ce(III)

$$\text{Unreacted Ce(IV)} + \text{Fe(II)} \xrightarrow{H^+} \text{Ce(III)} + \text{Fe(III)}$$

$$\text{Fe(III)} + \text{SCN}^- \Longrightarrow \text{Fe(SCN)}^{2+}$$

Blood red coloured complex)

PROPOSED ASSAY PROCEDURE

A Series of 25ml of volumetric flasks 0.5ml, 1ml, 1.5 ml, 2 ml, 2.5 ml of the working standard solution of the drug was pipetted into each flash 1ml of 0.05N cerric ammonium sulphate IV solution and 1 ml of 5 N Hydrochloric acid solution and requisite volume of double distilled water are added. The flasks were let stand for 5 minutes with occasional shaking subsequently. 1 ml of 0.02 N ammonium of ferrous sulphate was added to each flask and the contents were mixed Well, unreacted Ce IV sulphate Iron II to Iron III. Then after '5' minutes this solution was treated with 3 ml of (1M) ammonium thiocyamate solution. It forms blood red colored solution of Iron III sulphate thiocyanate complex. The absorbance of the blood red colored solution in each flask was measured at 545 nm by using spectrophotometer against a blank solution. The absorption spectrum of sulphasalazine is presented in figure -2. The sulphasalazine curve was obtained by plotting absorbance values against the amount of standard drug. The amount of sulphasalazine present in the sample was computed from the calibration curve are presented in figure -3 and the result is given in Table -1.

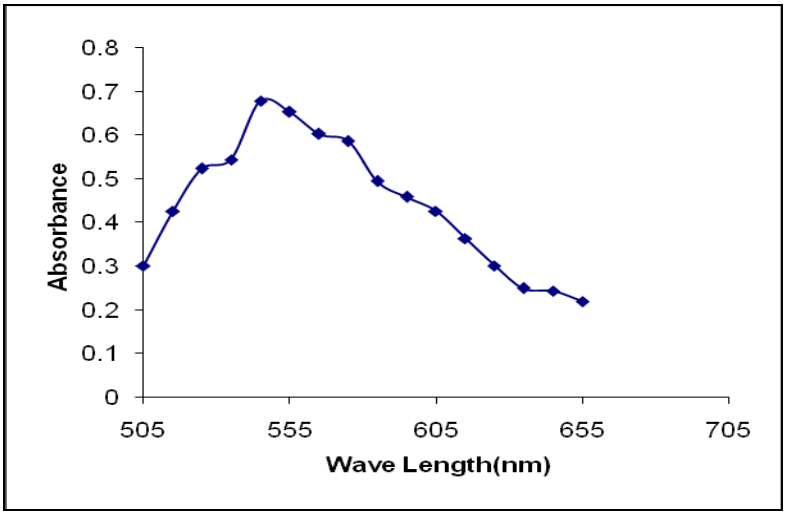

Fig: 2 Absorption Spectrume of sulphasalazine.

Table 1: Spectral data for calibration Curve.

Amount of Drug Solution	Absorbance at 545nm
0.5ml	0.285
1ml	0.632
1.5ml	0.905
2ml	1.235
2.5ml	1.521

Fig: 2: spectrum of sulphasalazine

The drug calibration graph was obtained by plotting absorbance values against the concentration of sulphasalazine drug solution. The calibration graph was found to be linear over the concentration range of 50-250 µg/ml for sulphasalazine. The linearity of the curve indicates that it obeys Beer's law. The amount of sulphasalazine present in the sample was read from the calibration graph. The results are present in the fig – 3.

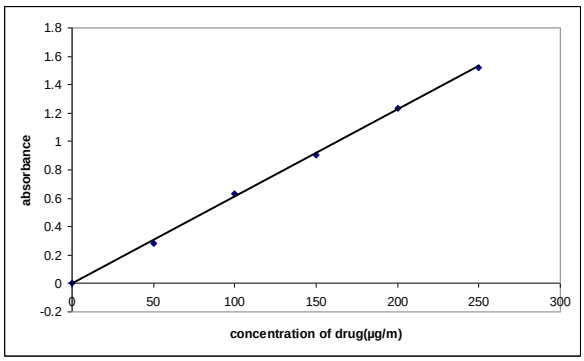

Fig: 3: Calibration curve of sulphasalazine.

Validation of the Method

This method was validated in terms of linearity, accuracy, precision, specificity and reproducibility of the sample applications the linearity of this method was investigated by serially diluting the stock solutions of sulphasalazine and measured the absorbance value at 545 nm by spectrophotometer calibration curves were constructed by plotting the absorbance difference values against the amount of drug in µg/ml.

Statistical analysis

A statistical analysis was performed on the statistically significant variables using the statistical software. The following parameters were determined standard deviation (SD), Relative deviation (RSD) Student t- test, F – test.

The standard deviation (SD) and Relative standard deviation (RSD) student t test and F- test of sulphasalazine was calculated from five measurements of replicate samples.

Table. 2: Assay of sulphasalazine in tablets.

Sample	Labelled amount (mg)	*Amount found by proposed method ± S.D*	% of Label claim	RSD%*	t^*_{cal}	F*	*Amount found by Reference method ± S.D*
Tablet1	500	499.483 ± 0.0580	99.89	0.0116	1.1467	1.6842	499.527 ± 0.0752
Tablet2	500	499.470 ± 0.0678	99.89	0.0135	1.3816	1.2319	499.527 ± 0.0752
Tablet3	500	499.510 ± 0.0754	99.90	0.0157	0.3867	0.9948	499.527 ± 0.0752

***Average of six determinations based on label claim.**

The values of standard deviation (SD) and Relative standard deviation (RSD) and Relative standard deviation are low, indicated high accuracy and reproducibility of this method. The data of assay values of commercial formulations was subjected to statistical evaluation for student ‘t’ test to study the proposed method. The calculated ‘t’ values are less than ‘t’ theoretical values with 4(n-1=5-1) degrees of freedom at 5% level of significance indicate that there is no significant difference between proposed method and standard method.

RESULTS AND DISCUSSION

The method is based on the oxidation of sulphasalazine with a known excess of cerium(IV) sulphate and the determination of the unreacted oxidant by spectrophotometry. This method is based on the oxidation of sulphasalazine by a measured excess of cerium (IV) sulphate in HCl medium, reduction of the residual oxidant by a fixed amount of iron (II) and subsequent formation of iron (III)-thiocyanate complex, which is measured at 545 nm. When a fixed concentration of cerium (IV) sulphate is reacted with increasing concentrations of

sulphasalazine, there will be a proportional increase in the concentration of the oxidant. The unreacted oxidant, when treated with a fixed concentration of iron (II) accounts for a proportional increase in the iron (III) concentration. This is observed as a proportional increase in the absorbance of iron (III)-thiocyanate complex with the drug concentration, which formed the basis for the assay of drug. The Standard deviation, RSD% and t_{cal} and F test of the sulphasalazine is calculated from six measurements of replicate samples. The values of Standard deviation, RSD%, $t_{cal,}$ F test, were shown in Table.2. The values of standard deviation and RSD% are low, indicates high accuracy and reproducibility of the method. The data of assay values of commercial formulations is subjected to statistical evaluation for student 't' test to study the proposed method. The calculated't' values are less than't' theoretical values with 4 (n-1= 5-1) degrees of freedom at 5% level of significance indicate that there is no significant difference between proposed method and standard method. Commonly encountered excipients such as starch, talc, glucose, alginate and stearate did not interfere in the proposed methods. The described method is rapid and reliable and hence can be used for routine analysis.

REFERENCES

Bibilography

1. Qi M, Wang P, Cong R and Yang J., J Pharm Biomed Anal., 2004; 35(5): 1287-91.
2. Zhao G.Z and Li H.K. Guang Pu Xue Yu Guang Pu Fen Xi., 2003; 23(1): 157-9.
3. Suhagia B.N, Shah S.A, Rathod I.S, Patel H.M and Doshi K.R, Parmar VK., Indian Journal of Pharmaceutical Sciences, 2006; 68(4): 543-546A.
4. Lim J, Jang B, Lee R, Park S and Yun H., J Chromatogr B Biomed Sci Appl., 2000; 746(2): 219-25.
5. Feng Y.C and H.u C.Q., J Pharm Biomed Anal., 2006; 41(2): 373-84.
6. Egli K.L., J Assoc Off Anal Chem., 1985; 68(4): 803-6.
7. Bighley L.D, Mc Donnell J.P., J Pharm Sci., 1975; 64(9): 1549-53.
8. Watts P.J, Tudor A, Church S.J, Hendra P.J Turner P, Melia C.D and Davies M.C., Pharm Res., 1991; 8(10): 1323-8.
9. Pastor-Navarro N, Gallego-Iglesias E, Maquieira A and Puchades R., Anal Chim Acta., 2007; 583(2): 377-83.
10. Mennickent S, Pino L, Vega M, Godoy CG and de Diego M., J Sep Sci., 2007; 30(5): 772-7.
11. Mennickent S, Pino L, Vega M and de Diego M., J Sep Sci., 2008; 31(1): 201-6.

12. Trabelsi H, Bouabdallah S, Bouzouita K and Safta F., J Pharm Biomed Anal., 2007; 29(4): 649-57.
13. Fang J and Gorrod J.W.J Chromatogr., 1993; 614(2): 267-73.
14. Igarashi K and Castagnoli N., J Chromatogr., 1992; 579(2): 277-83.
15. Lea A.R, Hailey D.M and Duguid P.R., J Chromatogr., 1982; 250: 35-42..
16. Ouanês S, Kallel M, Trabelsi H, Safta F and Bouzouita K., J Pharm Biomed Anal., 1998; 17(3): 361-4.
17. Vujic Z, Radulovic D and Zivanovic L., Farmaco., 1995; 50(4): 281-4.
18. Rahman N, Rahman H and Azmi S.N., Chem Pharm Bull., 2005; 53(8): 942-8.
19. Gupta K.R, Tajne M.R and Wadodkar S.G., Indian Journal of Pharmaceutical Sciences, 2008; 70(4): 511-513.
20. Garg G, Saraf S and Saraf S.J., AOAC Int., 2008; 91(5): 1045-50.
21. Liu H, Ren J, Hao Y, Ding H, He P and Fang Y., J Pharm Biomed Anal., 2006; 42(3): 384-8.
22. Chawla S, Ghosh S, Sihorkar V, Nellore R, Kumar T.R and Srinivas NR. Biomed Chromatogr., 2006; (4): 349-57.
23. Horai Y, Ishizaki T, Kusaka M, Tsujimoto G and Hashimoto K., Ther Drug Mnit., 1988; 10(4): 42833.
24. Winkler H, Ried W and Lemmer B., J Chromatogr., 1982; 228: 223-34.
25. Lea A.R and Hailey D.M, Duguid P.R., Journal of Pharmaceutical and Biomedical Analysis, 2005; 38(3): 543-550.
26. Colbourne P.D, Baker G.B and Coutts R.T., J Pharmacol Toxicol Methods., 1997; 38(1): 27-32.