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DEVELOPMENT OF VISIBLE SPECTROSCOPIC METHODS FOR DETERMINATION OF NIMESULIDE IN PHARMACEUTICAL FORMULATIONS

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ABSTRACT

Three simple spectrophotometric methods A, B, C as here described for the assay of nimesulide in bulk and pharmaceutical dosage formulations. Method A based on reduction of nimesulide with zinc and hydrochloride and reacting the reduced nimesulide with sodium nitrate to give diazonium compound with resorcinol to give yellow coloured chromogen at 402 λmax. Method B is based on dissolving nimesulide in 0.1N Sodium hydroxide which gave a yellow coloured chromogen with λmax 392nm. Method C based on reaction of ferric alum with MBTH and reduced ferrous ions forms a complex with reduced drug which gives a light blue coloured chromogen with λmax 432nm.

KEY WORDS

MBTH, Sodium nitrate, Sodium hydroxide and Hydrochloride.

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INTRODUCTION

Nimesulide is chemically N- (4-Nitro 2-phenoxy – phenyl methane sulfonamide) used for anti inflammatory activity. It is prostaglandin synthetase and platelet aggregation inhibition[1,2]. It is not yet official in Indian pharmacopoeia. A survey of literature revealed that there are few reports on visible spectrophotometry. Therefore there is a need for the fast, low cost and selective methods are obviously especially for the routine quality control analysis of pharmaceutical formulation containing Nimesulide.

This paper describes three visible spectrophotometric methods for the determination of nimusulide by making the use of the reported procedure. Sodium nitrite and resorcinol (Method A) are commonly use for determination of amines[3,4]. Method A is based on formation of orange coloured species (λ_{max}:402 nm) on treating Nimesulide with sodium nitrite and resorcinol in alkaline medium. In method B due the alkaline pH of sodium hydroxide yellow colour was intensified (λ_{max}:392 nm). In method C MBTH and Ferric alum is used for determination of primary amines MBTH also used for determination of phenolic compounds (λ_{max}:432nm)[5, 6].

EXPERIMENTAL

Instrument

A systronics pc based UV- Vis double beam spectrophotometer (Model: 2202) with 1 cm matched quartz cells was used for all spectral measurements.

Reagents

All the chemicals used were of analytical grade and all the solutions were prepared with double distilled water. Aqueous solution of sodium nitrite (01% w/v), aqueous solution of Resorcinol (01% w/v) were prepared for Aqueous solution of sodium hydroxide (0.1 N) was prepared for Method B. 01% w/v Ferric alum was prepared in 1%v/v nitric acid, aqueous solution of MBTH (0.2% w/v) were prepared for method C.

STANDARD SOLUTIONS

Method A

Stock solution of Nimesulide (1 Mg/1 ml) was prepared by dissolving 100 mg of Nimesulide in 100 ml 0.1 N sodium hydroxide (1000 µg/ml). The working standard was prepared by dilution to 100 ml with 0.1 N sodium hydroxide (10 µg/ml).

Method B

Stock solution of Nimesulide (1 mg/ml) was prepared by dissolving 100 mg of Nimesulide in 100 ml of methanol (1000 µg/ml), the working standard was prepared by dilution to 100 ml with 0.1 N sodium hydroxide (10 µg/ml).

Method C

Methanolic stock solution of 5 ml Nimesulide was taken into 100 ml volumetric flask, to that add 5 g of zincdust and 4 ml of concentrated hydro chloric acid. Then kept it aside for 30 minutes, then the drug will get reduced, after that filter the solution the filtrate should be make up to 100 ml with 0.1 N hydro chloric acid. This solution can be used as the working standard (50 µg/ml).

Sample Solutions

Tablets of four brands were used for the purpose of analysis 20 tablets were powdered and powder equivalent to 100 mg of Nimesulide was weighed and the solutions were prepared as under standard solution preparation and filtered if insoluble portion present.

Assay Procedures

Method A

Aliquots of standard drug solution 2.5-12.5 µg/ml of Nimesulide were taken into five test tubes. To each test tube add 1 ml of sodium nitrite solution and 1 ml of resorcinol solution. The contents of each test tube were mixed well and the volume was made up to 10 ml with distilled water. The absorbance was measured against the reagent blank at λ_{max} 402 nm. The amount of the drug present in the sample solution was deduced from the calibration curve.

Method B

Aliquots of standard Nimesulide solution representing 2-10 µg/ml were transferred to 5 test tubes, then make up the volume with 0.1N Sodium hydroxide. The absorbance was measured at λ_{max} 392 nm against reagent blank. The amount of the drug in the sample was computed from beer-lambert's plot.

Method C

Aliquots of reduced drug solution representing 1-5 µg/ml were taken into 5 test tubes. To each test tube add 5 ml of Ferric Alum and 1 ml of MBTH. The solutions were mixed well and heated on a water bath for 10 minutes. After that the test tubes were cooled to room temperature. Then made up to 10 ml with distilled water. The absorbance was measured at λ_{max} 432 nm against reagent blank. The amount of

Nimesulide present in the sample solution was calculated from calibration graph.

RESULTS AND DISCUSSION

The optimum conditions for each method were established by varying one parameter at a time and keeping the others fixed and observing the effect produced on the absorbance of the coloured species and incorporated in the procedure. The optical characterstics and figures of merit are given in table 01, together with the regression equation (obtained by linear least squares treatment) for the calibration plots[7]. The precision and accuracy were found by analyzing 6 replicate samples containing known amount of drug and results were summarized in Table No.1.

Commercial formulations (tablets) containing Nimesulide were successfully analysed by the proposed method[8]. The ingredients usually present in the formulation of Nimesulide did not interfere with the proposed analytical methods.

Chemistry of Coloured Compounds

The formation of orange coloured species in method A with sodium nitrite and resorcinol suggest that Nimesulide under go hydrolysis under alkaline conditions to give a free amino group which is diazotized by sodium nitrite followed by coupling with resorcinol[3, 9].

The formation of yellow coloured species (method B) in methanolic solution of Nimesulide in the presence of sodium hydroxide is due to the alkaline nature of Nimesulide which gives yellow colour at alkaline pH[3].

The formation of bluish green coloured species in method C by Nimesulide with Ferric alum and MBTH is due to the reduction of ferric to ferrous by Nimesulide and subsequent chelation of ferrous with MBTH to form ferroin[3, 10-15]. The proposed methods are found to be simple, sensitive accurate and can be used for determination of Nimesulide in its pharmaceutical dosage form in a routine manner.

Table No.1: Precision and Accuracy

S.No	Optical Characters	A	B	C
1	λmax(nm)	402	392	432
2	Beers law limits(µg/ml)	2.5-12.5	2-20	1-15
3	Molecular absorptivity($lt \cdot mol^{-1}cm^{-1}$)	2.80×10^5	1.03×10^6	1.23×10^5
4	Sandellssensitivity($\mu g/cm^2$/0.001 abs. units)	0.109	0.00298	0.000004
5	Regression equation(y*)	Y=0.013+0.082x	Y=0.0015+0.0048x	Y=0.003+0.037x
6	Slope(b)	0.082	0.0048	0.037
7	Intercept(a)	0.013	-0.0015	0.003
8	Correlation coefficients	0.997	0.996	0.998
9	% RSD**	0.82	0.61	0.66
10	% Range of errors(0.05 level)**	0.0068	0.0051	0.0055

CONCLUSION

All the three methods are simple sensitive and reproducible and can be used for the routine estimation of nimesulide in bulk form and in pharmaceutical formulations.

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