

INTERNATIONAL JOURNAL OF SCIENCE AND TECHNOLOGY

SIMPLE SPECTROPHOTOMETRIC METHOD DEVELOPMENT OF MOXIFLOXACIN IN PURE FORM AND IN DOSAGE FORM

ABSTRACT

Three simple spectrophotometric methods (A,B & C) have described for the assay of moxifloxacin(MXF) in bulk and in pharmaceutical formulations all these three methods are based on the oxidation of MXF with ferric ions to form an yellow colored complex with a λ_{max} of 425 nm (ferric chloride), 445nm(ferric alum), 410nm(ferric nitrate). These methods have been statistically evaluated and were found to be precise accurate.

KEY WORDS: Moxifloxacin, visible spectroscopy.

INTRODUCTION:

Moxifloxacin is a synthetic broad spectrum 8-methoxy fluoro quinolone anti bacterial agent. Chemically it is 1-cyclopropyl-6-fluro-1, 4-di hydro-8 methoxy-7-((4, 7) octa hydro –pyridine-6-yl)-4-oxo-3-quinoline carboxylic acid MXF is not official in any pharmacopoeia. Literature survey revealed the presence of some HPLC and spectrophotometric methods for the estimation of specific compounds. But the problem of assay of MXF for the pharmaceutical formulations has been limited number of investigations. Very few analytical methods have been reported for the estimation of MXF using visible spectrophotometry. The analytically important functional group of MXF have not been exploited for designing sensitive, accurate and flexible visible spectrophotometric methods for the determination of MXF in the pharmaceutical formulations.

This paper describes three UV, visible spectrophotometer methods for the determination of MXF by making use of the reported procedures. Ferric ions are commonly used for the oxidation of many functional groups to form colored complexes.

MATERIALS AND METHODS:

INSTRUMENTS:

A Systronics UV visible single beam spectrophotometer model no 117 with 1cm matched quartz cells were used for all spectral methods.

REAGENTS:

All the chemicals used were of analytical grade and all the solutions were prepared with distilled water. Aqueous solutions of ferric chloride (0.1% w/v) for method –A, and aqueous solutions of ferric alum (0.1% w/v) for method-B, and aqueous solutions of ferric nitrate (0.1% w/v) for method-C, were used to produce colored complex.

STANDARD DRUG SOLUTION:

Stock solution of MXF (1mg /ml) was prepared by dissolving 100mg of MXF in 100ml of 0.1N $\,$ Hcl. The working standards were prepared by dilution to 100ml with distilled water (for methods A, B and C 100 μ g /ml).



REREARCH ARTICLE Swathi.K et al, The Experiment, April, 2013 .Vol. 9(3), 560-565

SAMPLE SOLUTION:

Different formulations (eye drops, tablets, i.v infusion) were used for purpose of analysis for tablet. 20 tablets were powdered and powder equivalent to 100mg of MXF was weighed and solution was prepared as under standard solution preparation and filtered, if insoluble portion is present. In the same manner eye drops and i.v infusion were diluted.

ASSAY PROCEDURE:

METHOD A:

Aliquots of standard drug solution representing $200-800\mu$ g/ml of MXF ,ferric chloride were added successfully to a series of 10ml volumetric flask the contents of each tube was mixed well and diluted to 10ml with distilled water. The absorbance was measured against reagent blank at 425nm. The amount of drug present in sample solution was estimated from the calibration curve.(Fig:A)

METHOD B:

Aliquots of standard drug solution representing $100-500 \mu g/ml$ of MXF ,ferric alum were added successfully to a series of 10ml volumetric flask the contents of each tube was mixed well and diluted to 10ml with distilled water. The absorbance was measured against reagent blank at 445nm. The amount of drug present in sample solution was estimated from the calibration curve.(Fig:B)

METHOD C:

Aliquots of standard drug solution representing 100-500µg/ml of MXF ,ferric nitrate were added successfully to a series of 10ml volumetric flask the contents of each tube was mixed well and diluted to 10ml with distilled water. The absorbance was measured against reagent blank at 410nm. The amount of drug present in sample solution was estimated from the calibration curve. (Fig:C)

RESULTS AND DISCUSSION:

The optimum conditions for each methods were establish by varying one parameter at a time and keeping the others fixed and observing the effect produced and incorporated in the procedure the optical characteristics and the figures of results given in table-1, together the regression equation for the calibration products. The accuracy and precision were found by analyzing 6 replicate samples containing known amount of drug and the results were summarized in table-1.

Commercial formulations (tablets, eye drops, IV infusion) containing MXF were successfully analyzed by the proposed methods. The values obtained by proposed and reference (UV method) for formulations were compared statistically by t-test and f-test and found not to differ significantly as an additional check of accuracy recovery experiments were performed by adding a fixed amount of drug to the pre analyzed formulations. These results were summarized in table-2. The ingredients present in the formulations for MXF did not interfere with the proposed analytical methods.

CHEMISTRY OF COLORED SPECIES:

The formation yellow colored species in method A,B,C is due to the complex formed by drug and ferric ion .the proposed methods were found to be simple, sensitive ,accurate and can be used for the determine of MXF in the pharmaceutical dosage form in routine manner.



REREARCH ARTICLE Swathi.K et al, The Experiment, April, 2013.Vol. 9(3), 560-565



Fig: A



Fig B







OPTICAL AND REGRESSION CHARACTERISTICS OF MXF USING PROPOSED METHODS

Onticel character	Methods				
Optical character	Α	В	C		
λmax (nm)	425	445	410		
Beer's law limits (µg/ml)	20-80	10-50	10-50		
Molar absorptivity(Litre.mole ⁻¹ .cm ⁻¹)	1.72X 10 ⁻³	1.48X 10 ⁻³	2.40X 10 ⁻³		
Sandell's sensitivity (µg/cm ² /0.001 abs.unit)	0.0606	0.0926	0.0925		
Regression equation $(Y)^*$					
Slope (b)	1.07X10 ⁻³	1.07X10 ⁻³	8.24X10 ⁻⁴		
Intercept (a)	5X10 ⁻³	6X10 ⁻³	2.0X10 ⁻³		
% RSD**	0.29	0.38	0.41		
% Range of error ^{**} (0.05 level)	0.05-±0.32	0.05-±0.40	0.05-±0.43		

Table-1

- * Y= a+bX, where X is the concentration of ESZ in μ g/ml and Y is the absorbance at respective λ max.
- ** For six replicate samples.



ASSAY AND RECOVERY OF MXF WITH DOSAGE FORMS

Pharmaceutical Labeled		Amount found (mg)		Found by	%recovery ^{***}			
formulations a (1	amount (mg/tablet)	Method A	Method B	Method C	method [*] *	Method A	Method B	Method C
Tablet	400	394	394	400	399	98.5	98.5	100
Eye drops	25	23	25	24	24	92	100	96
IV infusion	400	23	432	394	399	92	108	98.5

Table-2

*Average \pm standard deviation of six determinations the t -& F values refers to comparison of the proposed method with the reference method .theoretical values at 95% confidence limits t = 2.57, F=5.05

** UV method (λmax: 288nm in methanol)

*** Recovery of 10mg added to the pharmaceutical formulations (average of 3 determinations)

CONCLUSION:

Since Moxifloxacin is a relatively new drug and the analytical methods available for its assay are very limited, it is worthwhile to develop some methods for its assay. As part of the present investigations 3 methods have been developed for the purpose of assay of Moxifloxacin. It can be seen from the results presented above that the proposed methods have reasonable sensitivity. Statistical analysis of the results shows that the proposed procedures have good precision and accuracy. Results of the analysis of pharmaceutical formulations reveal that the proposed methods are suitable for their analysis with virtually no interference of the usual additives present in them.

Beer's law limits (mg/ml) of the proposed methods are better than many of the reported spectrophotometric methods. All the proposed methods are simple simple, sensitive and reliable with good precision and accuracy. These methods can be used for the routine determination of Moxifloxacin in bilk samples.

AKNOWLDGEMENTS:

The authors are thankful for management of Sir C.R.Reddy college pharmaceutical sciences, Eluru for providing necessary facilities.



REREARCH ARTICLE Swathi.K et al, The Experiment, April, 2013 .Vol. 9(3), 560-565

REFERENCES:

- 1. Atherden L.M., Edr, Organic reactions used in visible spectroscopy Bently and Drivers Test Book of pharmaceutical Chemistry, 8th Edn., Oxford University Press, 1966, 4th Impression
- 2. Higuchi, T., Brochman-Hansen, E., Organic reagents used in visible spectroscopy 4th Edt., Pharmaceutical Analysis, Interscience, London, 1961.
- 3. Mitsuhashi, s., Nakanisha, A., Seibutsgaku spectroscopy of drugs (med boil), 1953, 27.
- 4. Folin, O., Ciocalteu, D., Spectrophotometric determination of purines and pyramidines j.boil.chem, 1927, 73, 627.
- 5. Ramana Rao, G., Kagilal, G., Rammohan, K., Visible spectrophotometric method for the determination of Omeprazole in dosage form Ind. J pharm., 1977, 37,140.
- 6. Besthorn, E., Reaction of MBTH Ber.Dtsch, chem.ges, 1904,43,1519
- 7. Hunig, s. and Fritsch, K.H., Oxidation Reduction reaction with MBTH Ann. Chi., 1957, 609: 143
- 8. Hunig, s. and Fritsch, K.H., Justus Liebigs., Ann. Chem., 1957, 609, 143 & 172.
- 9. Hunig, S.and Balli, H., Justus Liebigs., Ann. Chem., 1957, 609, 160
- 10. Sawicki, E.Hauser, T.R., Stantly, T.W. and Elbert, W., Anal. Chem, 1961, 3, 93
- 11. Altshuller, a.p. and leng, L.J., anal. chem., 1963, 35, 1541
- 12. Kamata ,e.,bull.chem.soc,japan,1965,58,2005
- 13. George, j.n., reaction of aldehydes and ketones with MBTH anal.chem., 1981, 53, 1708



Ganga ratnam.P, Swathi.K*, Ushabhagyarekha.S, Jhansi.D, Srija reddy.K, Sudheerbabu.I

Sir C.R.Reddy college pharmaceutical sciences, Eluru