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SPECTROPHOTOMETRIC DETERMINATION OF DEFERASIROX IN FORMULATIONS USING FOLIN-CIOCALTEU and FERRIC CHLORIDE REAGENTS

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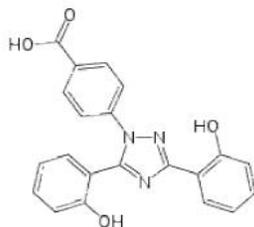
ABSTRACT

A simple, economical, accurate and precise spectrophotometric method has been developed for the determination of Deferasirox in formulations. The developed method is based on the formation of colored complex of drug with FC reagent and Ferric chloride, showed absorption maximums at 753 and 517nm subsequently. For both Beer's is obeyed in the concentration range of 8-40 $\mu\text{g/ml}$. the results of analysis have been validated statistically and also by recovery studies.

KEYWORDS: Spectrophotometry, Ferric Chloride, FC reagent, 20% Na_2CO_3 and Deferasirox

INTRODUCTION

Deferasirox Molecular formula $C_{21}H_{15}N_3O_4$ Molecular weight 373.36 g/mol. IUPAC name 4-[bis(2-hydroxyphenyl)-1H-1,2,4-triazol-1-yl]benzoic acid. Deferasirox is used as Antidote, Chelating Agent. The Literature review reveals HPLC Coupled With a MS/MS, LC, Terbium-sensitized fluorescence methods for the estimation of Deferasirox alone and Electro catalytic oxidation method for determination of Deferasirox in combination with Deferiprone in the formulations. The present investigation has been under taken to develop simple Visible Spectrophotometric methods for the estimation of Deferasirox in pure form and its formulation.



Structure of Dferasirox

2. MATERIALS AND METHODS

A Perkin Elmer model Lambda 25 UV/Visible spectrophotometer with 1 cm matched Quartz cells was used for present work. The chemicals used were of analytical grade. $FeCl_3$ solution (0.5% w/v), 20% Na_2CO_3 and FC reagent 1N was prepared in distilled water.

2.1 Preparation of Calibration curve:

2.1.1 $FeCl_3$ method

Standard drug solution (1000 $\mu\text{g/ml}$) was prepared in methanol. Aliquots of standard solution ranging from 0.2 to 1 ml (8-40 $\mu\text{g/ml}$) were transferred into a series of 25 ml standard flasks. To that 1 ml of 0.5% w/v $FeCl_3$ was added and the total volume was made up to 25 ml with distilled water. The content were shaken for few minutes and the absorbance of the solution was measured at 517 nm against the corresponding reagent blank. The amount of Deferasirox in the sample was computed from the calibration curve.

2.1.2 FC (Folin-Ciocalteu reagent) method

Standard drug solution (1000 $\mu\text{g/ml}$) was prepared in methanol. Aliquots of standard solution ranging from 0.2 to 1 ml (8-40 $\mu\text{g/ml}$) were transferred into a series of 25 ml standard flasks. To that 5 ml of 20% Na_2CO_3 , 10 ml of distilled water and 1 ml of 1N FC reagent was added and the total volume of the phase was made up to 25 ml with distilled water. The content were shaken for few minutes and the absorbance of the solution was measured at 753 nm against the corresponding reagent blank. The amount of Deferasirox in the sample was computed from the calibration curve.

2.2 Analysis of formulation:

Accurately weigh formulation powder equivalent to 100 mg of Deferasirox was transferred to a 100 ml volumetric flask. 50 ml of methanol is added and sonicated for 10 min. and diluted to the mark with methanol. The resulting solution was filtered through a Whatman filter paper No 0.45. The assay of formulation was carried out as above procedure.

3.RESULTS AND DISCUSSION

In the present work a Spectrophotometric methods has been developed for determination of Deferasirox from its formulations. The developed methods were based on the formation of yellow color complex of drug with $FeCl_3$ in distilled water and blue color complex of drug with FC reagent in 20% Na_2CO_3 and distilled water mediums. Wave length maximums was found to be at 517&753 nm for $FeCl_3$ and FC reagents. The linearity was observed in concentration range of 8-40 $\mu g/ml$. commercial formulations were successfully analyzed by the proposed method and the results are summarized in the table. 1

PARAMETERS	LIMIT	OBSERVATIONS	
		FC method	$FeCl_3$ method
Wave length measured (nm)	-----	753	517
Linearity range (mcg/ml)	-----	8-40	8-40
Correlation Coefficient	NLT 0.9990	0.9995	0.9990
Slope	-----	0.0124	0.0133
Intercept	-----	0.2282	0.1923
Assay	% RSD NMT 2	1.0414	0.6534
System precision	% RSD NMT 2	0.74	0.8391
Method Precision	% RSD NMT 2	0.8770	0.5052
% Recovery	98% – 102%	99.54	99.99

Table. 1

4. CONCLUSION

The proposed methods are simple, sensitive, accurate and precise for the determination of deferasirox in formulations and can be used for routine quality control of deferasirox formulations.

5. TABLES AND FIGURES

FC method

Linearity

THEORETICAL CONC.(µg/ML)	ABSORBANCE
8	0.3253
16	0.4295
24	0.5259
32	0.6313
40	0.7221

Table.2

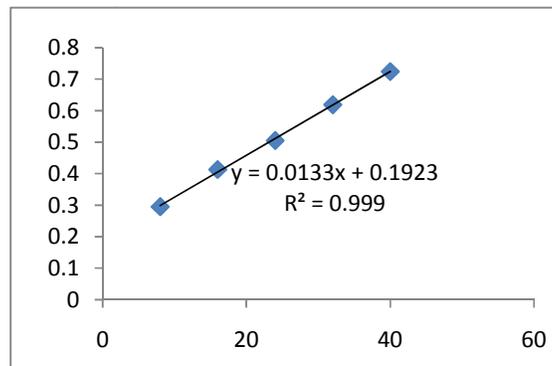


Figure.1

Linearity plot

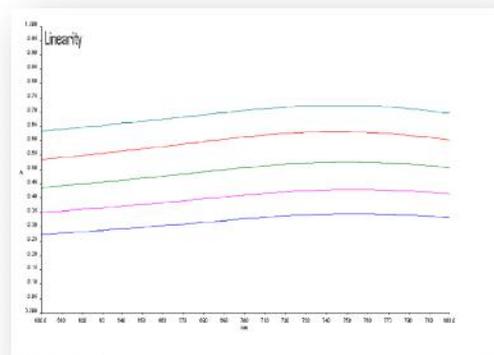


Figure.2

System Precision

S.NO	STD ABS
1	0.529
2	0.531
3	0.537
4	0.539
5	0.541
6	0.536
7	0.532
8	0.539
9	0.535
10	0.532
Average	0.5351
SD	0.003985
%RSD	0.74

Table.3

Method Precision

Set	Sample Absorbance	Standard Absorbance	Amount of Defersirox	% Assay	
1	0.5250	0.5212	99.31	99.31	
2	0.5250	0.5312	101.18	101.18	
3	0.5250	0.5221	99.45	99.45	
4	0.5250	0.5219	24	0.0619	
5	0.5250	0.5217	24	0.0619	
6	0.5250	0.5301	24	0.0619	
				Average	99.95
				SD	0.8765
				% RSD	0.8770

Table.4

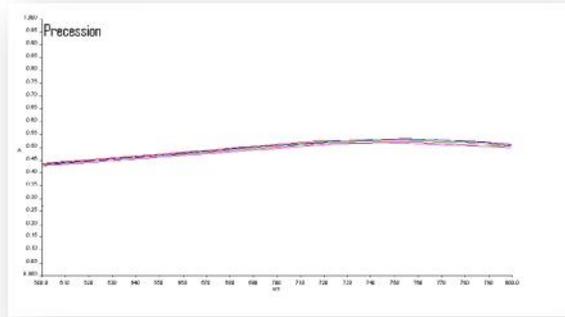


Figure.3

Accuracy

ACCURAY LEVEL %	AMOUNT PRESENT (µg/ml)	AMOUNT ADDED (µg/ml)	AMOUNT RECOVERED (µg/ml)	% RECOVERY	AVERAGE RECOVERY
80	10	9.2	19.21	100.14	99.54%
100	10	14	23.99	99.93	
120	10	18.8	28.53	98.33	

Table.5

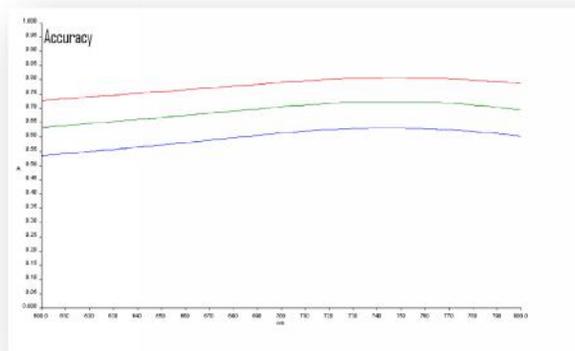


Figure 4

Assay

Set	Sample Absorbance	Standard Absorbance	Amount of Defersirox	% Assay
1	0.5250	0.5212	99.31	99.31
2	0.5250	0.5312	101.18	101.18
3	0.5250	0.5221	99.45	99.45
Average				99.98
SD				1.0412
% RSD				1.0414

Table.6

FeCl₃ method

Linearity

THEORETICAL CONC.(µg/ML)	ABSORBANCE
8	0.29527
16	0.41324
24	0.50564
32	0.61900
40	0.72450

Table 7

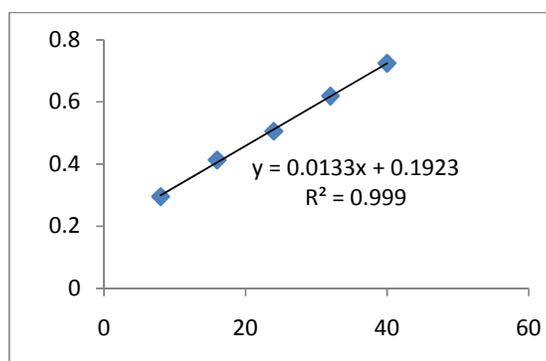
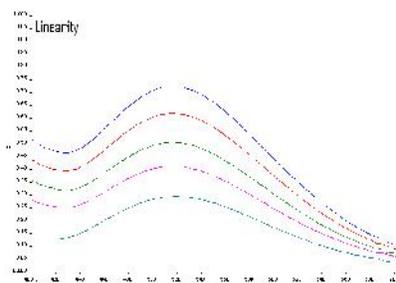


Figure.5

System Precision



S.NO	STD ABS
1	0.5055
2	0.5015
3	0.4995
4	0.4985
5	0.5005
6	0.4980
7	0.4983
8	0.4889
9	0.5005
10	0.4983
Average	0.4985
SD	0.004183
%RSD	0.8391

Table.8

Method Precision

Set	Sample Absorbance	Standard Absorbance	Amount of Defersirox	% Assay
1	0.5056	0.5025	99.43	99.43
2	0.5056	0.5012	99.13	99.13
3	0.5056	0.5075	100.38	100.38
4	0.5056	0.5059	100.06	100.06
5	0.5056	0.5017	99.23	99.23
6	0.5056	0.5023	99.35	99.35
			Average	99.59
			SD	0.5032
			% RSD	0.5052

Table.9

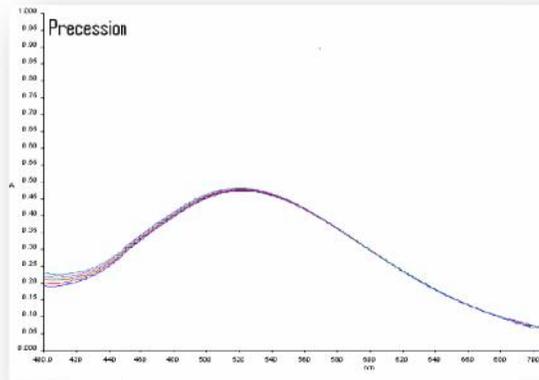


Figure 7- Precession

Accuracy

ACCURAY LEVEL %	AMOUNT PRESENT (µg/ml)	AMOUNT ADDED (µg/ml)	AMOUNT RECOVERED (µg/ml)	% RECOVERY	AVERAGE RECOVERY
80	10	9.2	19.10	98.93	99.99 %
100	10	14	23.95	99.97	
120	10	18.8	99.66	101.08	

Table 10

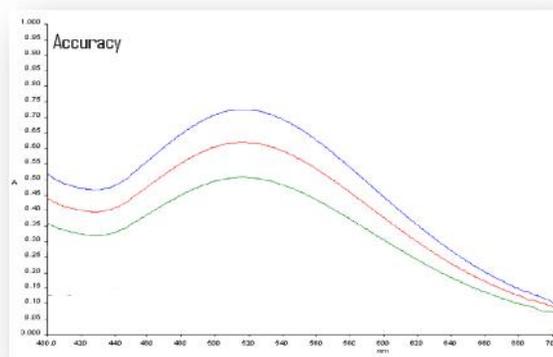


Figure 8

Assay

Set	Sample Absorbance	Standard Absorbance	Amount of Deferasirox	% Assay	
1	0.5056	0.5025	6	0.0155	
2	0.5056	0.5012	6	0.0155	
3	0.5056	0.5075	6	0.0155	
Table.10				Average	99.64
				SD	0.6511
				% RSD	0.6534

6.ACKNOWLEDGMENTS

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7. REFERENCES

1. Chauzit, Emmanuelle PharmD*; Bouchet, Stéphane PharmD*; A Method to Measure Deferasirox in Plasma Using HPLC Coupled With MS/MS Detection and its Potential Application, *The Drug Monit.* 32(4),476-81
2. Jamshid L. Manzoori, Abolghasem Jouyban, Mohammad Amjadi, Vahid Panahi-Azar, Elnaz Tamizi, Jalil Vaez-Gharamaleki Terbium-sensitized fluorescence method for the determination of deferasirox in biological fluids and tablet formulation *The journal of biological and chemical sciences*, 63(3),236-240.
3. Lough WJ, Wainer IW. *High Performance Liquid Chromatography: fundamental principles & practice*. Glasgow (UK): Blackie Academic & Professional; 1995. p. 2-28.
4. M. Hajjizadeh, A. Jabbari, H. Heli, A.A. Moosavi-Movahedi, A. Shafiee and K. Karimian Electrochemical oxidation and determination of deferasirox and deferiprone on a nickel oxyhydroxide-modified electrode *Anal Biochem.* 373(2):337-48.
5. Ravi Kiran Kaja, K. V. Surendranath, P. Radhakrishnanand, J. Satish, P. V. V. Satyanarayana A Stability Indicating LC Method for Deferasirox in Bulk Drugs and Pharmaceutical Dosage Forms *Chromatographia*, 72(5-6), 441-446.
6. Ronald C. "Visible and ultra violet spectroscopy", 3rd Edn, John Wiley and sons, Russia, 1999, 56-132.
7. Day RA, Underwood AL. *Quantitative Analysis*. 4th ed. New Delhi: Prentice Hall; 1986. p. 14-19.
8. Michael E, Schartz IS, Krull. *Analytical method development and Validation*. 3rd ed. London: John Wiley & sons; 2004: p. 25-46.