



SPECTROPHOTOMETRIC ESTIMATION OF NARATRIPTAN HYDROCHLORIDE WITH IRON REAGENTS

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ABSTRACT

Three simple spectrophotometric methods (method A, B and C) were developed based on the oxidation of Naratriptan. Hcl in presence of ferric chloride and subsequent complexation of ferrous with reagents such as 1, 10-phenanthroline (method A), 2, 2'-bipyridyl (method B) and Batho-phenanthroline (method C). These complex formation results in yielding orange-red coloured chromogens with λ_{max} of 510nm, 520nm and blue coloured species with λ_{max} of 620nm.

Key words: Spectrophotometric method, Naratriptan. Iron reagents

INTRODUCTION:

Naratriptan (trade name Amerge) is a triptan drug marketed by GlaxoSmithKline and is used for the treatment of migraine headaches. The drug is official in U.S.P 2007. Several methods have been reported for the estimation of Naratriptan.Hcl including chromatographic, electrochemical, capillary electrophoresis, e.t.c

In the present study, the reaction of Naratriptan with iron reagents was utilized for the colour development. The proposed methods were sensitive, fast, simple and economical for the determination of Naratriptan in pure form as well as in formulations without the need of extraction.

MATERIALS:

INSTRUMENTATION:

A systronics 2201 UV-Visible double beam spectrophotometer with 1cm matched quartz cells were used for all spectral and absorbance measurements.

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REAGENTS PREPARATION:

1, 10-Phenanthroline solution (Qualigens, 0.198% w/v, 1.0×10^{-2} M)

198mg of 1, 10-Phenanthroline was accurately weighed and dissolved in 100ml of 0.1N hydrochloric acid.

2, 2' Bipyridyl solution (Qualigens, 0.156% w/v, 1.0×10^{-2} M)

Prepared by dissolving 156mg of 2,2'-bipyridyl in 100ml of 0.1N hydrochloric acid.

Batho phenanthroline (CDH, 0.332% w/v, 1.0×10^{-2} M)

About 332 mg of batho phenanthroline was accurately weighed and dissolved in 100 ml of 0.1N Hcl.

FeCl₃ stock solution (CDH, 0.162 % w/v, 1M)

162 mg of anhydrous ferric chloride was accurately weighed and dissolved in 100 ml of distilled water

33.3 ml of above stock solution was further diluted to 100 ml with distilled water and used for recommended procedures

O-Phosphoric acid (CDH, 2.0×10^{-1} M)

1.3ml of orthophosphoric acid is diluted to 100ml with distilled water

STANDARD PREPARATION:

About 100 mg of NRT was accurately weighed and dissolved in 100 ml of water. This stock solution was used as such for Method B. The stock solution was further diluted with water to get the working standard solution of concentration 100µg/ml for methods Method A and Method C.

Procedure for Estimation:

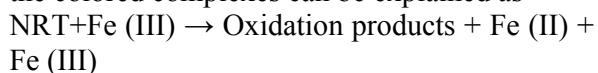
Aliquots of standard NRT solution (100µg/ml) containing 1.0 to 6.0 µg for Method A, 20.0 to 80.0 µg (100µg/ml) for Method B and 1.0 to 5.0 µg (1000µg/ml) for Method C were transferred into a series of 10 ml volumetric flasks and 1.0 ml of 0.003 M ferric chloride was added to each flask. Then 1.0 ml of PTL solution for Method A, 1.0 ml of BPN solution for Method B, 1.0 ml of BPTL solution for Method C were added to all flasks and the volume in all volumetric flasks were equalized with water. The contents were gently boiled for 30 min. for Method A, 50 min. for Method B and 10 min. for Method C. The flasks were cooled to room temperature and 2.0 ml of OPA was added to all and final volume of all volumetric flasks was brought to 10 ml with water. The absorbance was measured at 510 nm, 520nm and 620nm (Method A, B and C) respectively against corresponding reagent blanks. The amount of NRT in sample was estimated from corresponding calibration graphs

Analysis of pharmaceutical preparations:

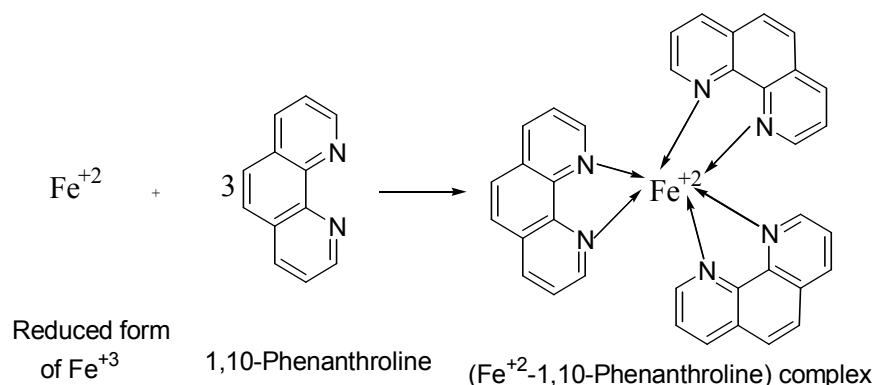
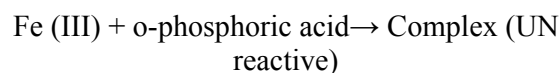
Application of the proposed methods to the determination of drug in its dosage forms was successfully made. The results were presented in table-2. The excellent recoveries obtained indicated the absence of any interference from the excipients.

Mechanism involved:

Naratriptan.Hcl has reducing property due to the presence of functional moieties (one or more) vulnerable to oxidation selectively with oxidizing agents such as Fe (III) under controlled experimental conditions. When treated with known excess of oxidant, NRT undergoes oxidation, giving products of oxidation (inclusive of reduced form of oxidant, Fe (II) from Fe (III), besides unreacted oxidant. It is possible to estimate the drug content colorimetrically, which is equivalent to either the reacted oxidant or reduced form of oxidant formed. The reduced form of Fe III (Fe II) has a tendency to give colored complex on treatment with PTL (Method A) or BPN (Method B) or BPTL (Method C). The chemistry involved in the colored complexes can be explained as



(Excess) (Reduced form of Oxidant)
(Unreacted)



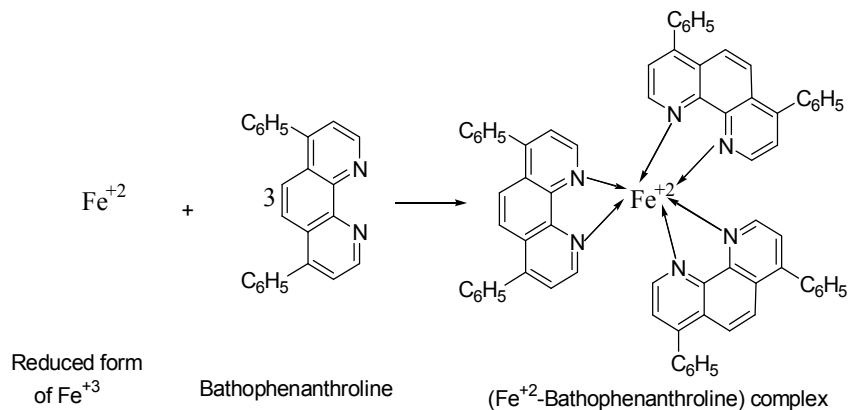
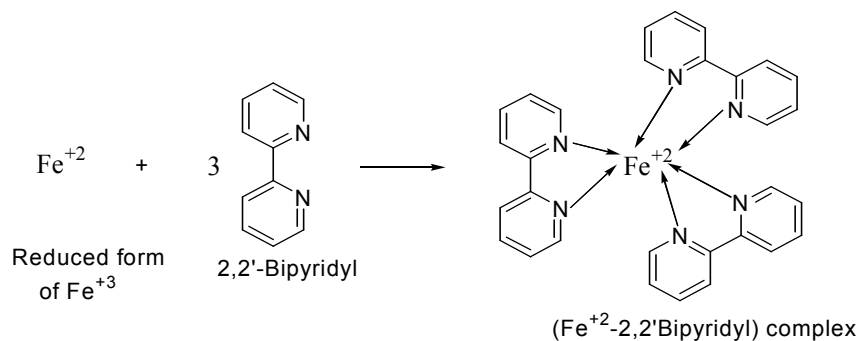


Table 1: Optical characteristics, regression data, Precision and accuracy of the proposed Methods for NRT

Parameter	A	B	C
λ_{max} (nm)	510	520	620
Beer's law limits ($\mu\text{g} / \text{ml}$)	1-6	20-80	1-5
Molar absorptivity ($\text{L. mole}^{-1} \text{ cm}^{-1}$)	4.946×10^4	3.589×10^3	7.085×10^4
Sandell's sensitivity ($\mu\text{g} / \text{cm}^2 / 0.001$ absorbance unit)	0.00752	0.1036	0.00525
Correlation coefficient (r)	0.9997	0.9999	0.9992
% Relative standard deviation*	0.335	0.363	0.306
% Range of Error * (Confidence limits) 0.05 level	0.352	0.382	0.321
0.01 level	0.552	0.596	0.503
% Error in bulk samples**	0.05	0.92	0.45

* Average of six determinations

** Average of three determinations.

Table 2: Assay and recovery of NRT in dosage forms

Method	Pharmaceutical Formulation	Labelled Amount (mg)	Proposed Method			% Recovery by proposed methods** \pm S.D
			Amount found* (mg) \pm S.D	t (value)	F (Value)	
M _A	Brand-I	2.5	2.53 \pm 0.015	0.617	1.874	100.2 \pm 0.54
		1.0	0.95 \pm 0.010	0.821	2.206	99.81 \pm 1.01
M _B	Brand-II	2.5	2.41 \pm 0.008	0.401	2.638	99.92 \pm 1.04
		1.0	1.03 \pm 0.011	0.527	1.526	100.3 \pm 0.69
M _C	Brand-I	2.5	2.55 \pm 0.012	0.396	2.540	100.2 \pm 1.01
		1.0	0.99 \pm 0.017	0.262	2.175	99.82 \pm 0.75

* Average \pm standard deviation of six determinations,

The t and F- values refer to comparison of the proposed method with reference method.

Theoretical values at 95 % confidence limits t = 2.571 and F = 5.05.

** Average of five determinations

CONCLUSION:

The applicability of the proposed method for the assay of pharmaceutical preparations was examined. Conclusively, it can be inferred that the proposed method was simple, rapid, precise, fairly sensitive and selective and hence it can be used for routine quantitation of Naratriptan hydrochloride.

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