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New spectrophotometric methods for the determination of Entacapone in bulk and pharmaceutical dosage forms

Neeha Tadipaneni and Devala Rao Garikapati

K.V.S.R Siddhartha College of Pharmaceutical Sciences, Vijayawada-520010, A.P, India.

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ABSTRACT

Two simple and sensitive spectrophotometric methods (Method A& Method B) in the visible region have been developed for determination of Entacapone in bulk and pharmaceutical formulations. Method A is based on Oxidation of Entacapone with Fe(III) under controlled experimental conditions followed by complex formation between Fe(II) and bathophenanthroline to give red colored complex which can be measured at 535 nm. Method B (p-nitroaniline) is based on electrophilic aromatic substitution to form brown colored complex which can be measured at 470 nm. The color obeyed Beer's law in the concentration range of 2-10 µg/ml for Method A and 10-50 µg/ml for Method B respectively. When Pharmaceutical formulations (Tablets) containing Entacapone were analyzed, the results obtained by proposed methods are in good agreement with labeled amounts. Recovery in both methods was 98%-102%.

Introduction

Entacapone is a second generation COMT (catechol O-methyl transferase) inhibitors which have been established as effective agents for the treatment of Parkinson's disease. Chemically, Entacapone is (E)-2-cyano- 3-(3,4-dihydroxy-5-nitrophenyl)-N,N-diethyl-2-propenamide.Only few HPLC1,2 and spectrophotometric methods3,4 are reported earlier for determination of Entacapone in biological fluids and dosage forms. Therefore, an attempt had been made in developing two visible spectrophotometric methods by utilizing bathophenanthroline5,6 and P-nitroaniline7 as Chromogenic reagents.

Materials and Methods

Instrument:

A Systronics double beam UV-Visible spectrophotometer 2201 with 1 cm matched Quartz cells were used for absorbance measurement.

Preparation of reagents:

All the chemicals used were of Analytical grade and prepared with double distilled water.

Method A:

Bathophenanthroline solution (0.3%w/v): Prepared by dissolving 300mg of bathophenanthroline in 100ml of methanol.

Ferric chloride solution (0.162%w/v): About 162mg of anhydrous ferric chloride was accurately weighed and dissolved in 100ml of distilled water.

Othophosphoric acid solution (CDH, 2.0×10-1 M): 1.3ml of orthophosphoric acid was diluted to 100ml with distilled water.

Method B:

P-nitroaniline (0.5%w/v): Prepared by dissolving 500mg of p-nitroaniline in 100ml of MeOH.

HCL solution (10%v/v): About 10ml of HCL was made up to 100ml of distilled water.

Sodium nitrite (3%w/v): About 3g of sodium nitrite was accurately weighed and dissolved in 100ml of distilled water.

NaOH solution: Prepared by dissolving 5g of NaOH in 100ml of distilled water.

Standard drug solution:

Method A:

A standard solution containing Entacapone at a concentration of 1 mg/ml in methanol is prepared and suitably diluted to get a Working standard solution of 10 µg/ml with the same solvent.

Method B:

A standard solution containing Entacapone at a concentration of 1 mg/ml in methanol is prepared and suitably diluted to get a Working standard solution of 100 µg/ml with the same solvent.

Standard curve:

Method A:

Aliquots of standard solution (10 µg/ml) ranging from 0.2-1.0 ml were transferred into a series of 10 ml volumetric flasks. To each flask 1ml of ferric chloride was added. Then 1ml of bathophenanthroline was added. The contents of flask were boiled for 20 min. Then the flask contents were cooled to room temperature and add 2ml of OPA was added to all flasks. The volume was made up to 10 ml with water. The absorbance was measured at 535 nm against reagent blank. The amount of ECP was deduced from its Beer-Lambert's plot.

Method B:

Into a series of 10ml volumetric flasks, 1ml of p-nitroaniline was added and 1ml of $NaNO_2$ was added. Then, aliquots of standard solution (100 µg/ml) ranging from 0.1-0.5 ml were transferred to set of flasks. Then 2 ml of NaOH solution was added to all flasks. The volume was made up to 10ml with water. The absorbance was measured at 470 nm against reagent blank. The amount of ECP was deduced from its Beer-Lambert's plot.

For Pharmaceutical Formulations

The contents of twenty tablets were transferred to a mortar. The tablet powder equivalent to 200 mg of ECP was taken and a standard stock solution of 1mg/ml was prepared. This was appropriately diluted and the amount of ECP was found out as described in the procedure.

Tele:

E-mail addresses: drgdr1964@gmail.com

Recovery studies

To study the accuracy, reproducibility and precision of the proposed methods, recovery experiments were carried out. The recovery of the added standard was studied at 3 different levels. Each level was repeated 6 times. A plot of drug found by proposed method (Y-axis) against standard added (X-axis) was drawn. The intercept on Y-axis indicates the amount of drug present per formulation.

Results and Discussion

The optimum conditions for each method were established by varying one parameter at a time and keeping the other fixed and observing the effect of product on the absorbance of colored species and incorporated in the procedure. The optical characteristics are given in Table 1, together with regression equation for calibration plots. The precision and accuracy were found by analyzing six replicate samples containing known amount of drugs and the results were summarized in Table1. Table2 shows that the values of % recovery are between 98 to 101 % and the values of coefficient of variation are sufficiently low indicating that the proposed methods are free of interferences from any excipients like starch; talc etc and the results are reproducible. Thus these developed methods can be employed for the routine determination of ECP in pure and in pharmaceutical preparations.

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Table 1. Optical characteristics, precision and accuracy of the proposed method

Parameters	Methods	
	A	B
λmax (nm)	535	470
Beer's law limit (μg/ml)	2-10	10-50
Sand ell's sensitivity ($\mu g/cm^2$/0.001abs.unit)	0.0019	0.0093
Molar absorptivity ($L\ mol^{-1}\ cm^{-1}$)	1.58756×10^5	3.2667×10^4
Regression equation(Y*)		
Slope (b)	0.0512	-0.0035
Intercept (a)	0.0034	0.4227
Correlation coefficient (r)	0.9996	0.9996
%Relative standard deviation**	0.32	0.628
%Range of error		
0.05 significance level	0.267	0.525
0.01 significance level	0.395	0.776

*Y= a + bX, where 'Y' is the absorbance and X is the concentration of Entacapone

**For six replicates

Table 2. Results of Assay and Recovery Experiments

Pharmaceutical Formulation	Labeled Amount(mg/tablet)	Method A	Method B	Reference method (mg)	%Recovery
Tablet-1	200	199.6	200.21	200.19	99.92
Tablet-2	200	199.48	200.72	200.64	100.41
Tablet-3	200	200.54	200.16	200.81	99.76