



RESEARCH ARTICLE

# UV SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION FOR ENTACAPONE IN BULK AND FORMULATION

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**A simple, rapid, accurate, precise and economic method has been developed and validated for the estimation of entacapone in bulk and tablet dosage form using UV spectrophotometry. Methanol was used as the solvent for entacapone. The UV spectrum of entacapone in water showed  $\lambda_{\max}$  at 308 nm and Beer-Lambert law was obeyed in the concentration range of 2-15  $\mu\text{g/ml}$ . The result of analysis has been validated statistically. The recovery studies ranged from  $99.29 \pm 1.11\%$ , confirmed the accuracy of the proposed method. The method was found to be precise with % relative standard deviation 0.80% for inter-day precision and for 0.85% intra-day.**

**Key words:** UV Spectrophotometry, Entacapone, Validation, Antiparkinson agent .

## INTRODUCTION

Entacapone is chemically known as 2-cyano-3-(5-dihydroxyamino-3,4-dioxo-1-cyclohexa-1,5-dienyl)-N,N-diethyl-prop-2-enamide and belongs to the class of antiparkinson agents. Entacapone is a selective and reversible inhibitor of catechol-O-methyltransferase (COMT), with mainly peripheral actions. It is used in the treatment of Parkinson's disease as an adjunct to Levodopa/Carbidopa therapy (O'Neil, 2001; Sweetman, 2002). Recently, a few analytical methods for the determination of entacapone were reported and most of them reported high-performance liquid chromatography (HPLC) assay of entacapone in dosage forms, in addition to derivative spectroscopic method (Ramakrishna *et al* 2005; Paim *et al* 2007; Doshi *et al* 2009; Sivasubramanian *et al* 2009; Mohamed and Mohamed, 2010; Soukhova *et al* 2011; Tekale *et al* 2011). But literature survey has not revealed any simple UV-spectrophotometric method for estimation of entacapone. Thus, in continuation of

our previous work on the UV spectrophotometric method development (Patil *et al* 2011), present study is directed toward the development of a simple, precise, accurate and economical spectrophotometric method for estimation of entacapone in bulk and formulation.

## MATERIALS AND METHODS

### Instruments

Perkin-Elmer UV-Visible spectrophotometer was used for spectral bandwidth 1 nm, wavelength accuracy 0.5 nm and 1 cm matched quartz cells. All the reagents used in this assay were of analytical grade. Pure entacapone was obtained as a gift sample from Ajanta Pharma Ltd., Mumbai. Tablets of entacapone, Entacom (Intas Pharma Ltd.), were purchased from local market.

### Analytical procedure

Standard stock solution (100  $\mu\text{g/ml}$ ) of the entacapone was prepared by dissolving 10 mg

of pure entacapone in 100 ml methanol and aliquot of this solution was further diluted to get a concentration of 10  $\mu\text{g/ml}$ . This solution was then subjected to scanning in the wavelength range of 200-400 nm. The aliquots of the standard working solution (100  $\mu\text{g/ml}$ ) were diluted serially with sufficient methanol to obtain the concentration range of 2-15  $\mu\text{g/ml}$ . The calibration curve was obtained by plotting absorbance vs concentration data (**Figure 2**) and linearity was evaluated by linear regression analysis. Coefficient of correlation was found to be 0.997 (**Table 1**).

#### Analysis of marketed formulation

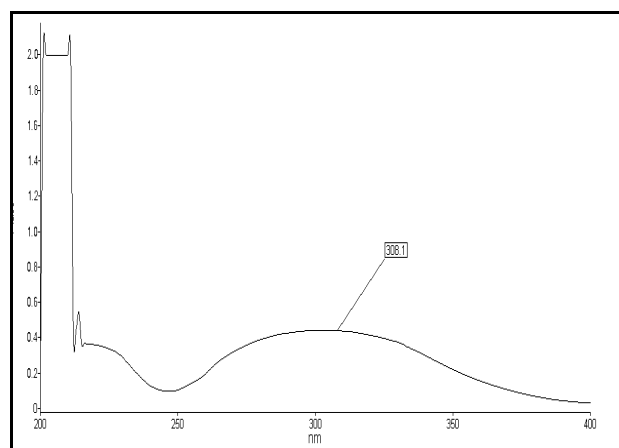
Twenty tablets were weighed, the average weight was determined and crushed into fine powder. A quantity of tablet powder equivalent to 10 mg of entacapone was transferred into 100 ml volumetric flask containing 60 ml methanol, shaken for 5 min, volume was adjusted to mark with methanol and filtered through Whatman filter paper no. 41. The appropriate aliquots were transferred to 10 ml glass volumetric flask and volume was adjusted to the mark with methanol to obtain concentration of 10  $\mu\text{g/ml}$ .

#### Validation

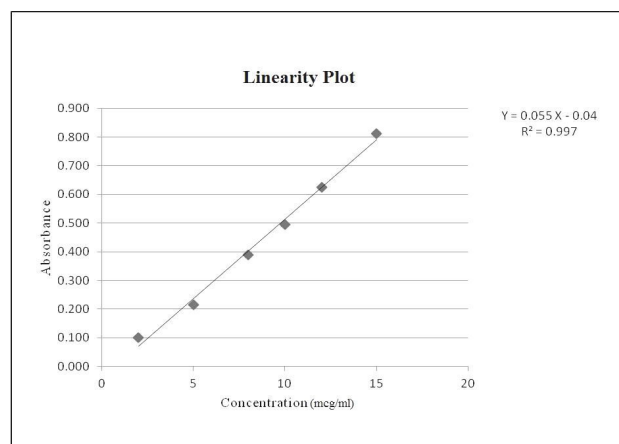
As a part of determining accuracy of the proposed method, standard addition method was done. Different concentrations of standard solution in three levels were added to a known fixed concentration of drug in the formulation solution previously analyzed to get 80, 100 and 120%, and the total concentration of the drug was determined. Repeatability was determined by using different levels of pure drug concentrations (same concentrations levels taken in accuracy study) prepared from independent stock solution in triplicates three different times in a day for intra-day variation ( $n=3$  and analyzed (ICH-Q2B, 2006). To study the inter-day variations, the same protocol was followed as that of repeatability for three different days and the results were documented as % relative standard deviation (RSD).

#### RESULTS AND DISCUSSION

The UV scan of standard solution between 200-400 nm showed the absorption maxima at 308 nm (**Figure 1**). The Beer's law was verified from the calibration curve. Regression analysis showed very good correlation of 0.997. The plot clearly showed a straight line passing through origin (**Figure 2**).



**Fig. 1.** UV spectrum of entacapone



**Fig. 2.** Linearity plot of entacapone

The parameters of regression analysis are summarized in **Table 1**. The concentration of the entacapone marketed tablets was determined and % label claim of tablet dosage form was found to be 100.08%. The results for analysis of marketed formulation were found satisfactory. The percentage recoveries were found in the range of  $99.29 \pm 1.11\%$  (**Table 2**). The method was found to be precise with %RSD of 0.80% for inter-day precision and 0.85% for intra-day precision (**Table 3**).

#### CONCLUSION

The proposed method was found to be simple, precise, accurate and rapid for the determination of entacapone in bulk and tablet dosage forms. Analysis of authentic samples containing the drug entacapone showed no interference from common additives and excipients. Hence, proposed procedure is well suited for assay and evaluation of drugs in the pharmaceutical preparations and can be easily and conveniently adopted for the routine quality control analysis.

**Table 1.** Regression analysis of entacapone

Sr. No.	Parameter	Result
1.	Regression equation	$Y = 0.055X - 0.04$
2.	Correlation coefficient	0.997
3.	Slope	0.055
4.	Y intercept	-0.04

**Table 2.** Accuracy studies

Sr. No.	Accuracy level	Mean % recovery	Overall % recovery
1.	80%	99.6±1.46	99.29±1.11%
2.	100%	99.1±1.06	
3.	120%	99.2±1.26	

**Table 3.** Precision studies

Sr. No.	Level	Intra-day variations		Inter-day variations	
		%RSD	Overall %RSD	%RSD	Overall %RSD
1.	80%	0.89	0.85	0.97	0.80
2.	100%	0.79		0.75	
3.	120%	0.95		0.75	

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