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Development and validation of analytical methods for estimation of rizatriptan benzoate in bulk and tablet dosage forms by UV spectroscopy

Ishan K. Chinnapurkar*, Manjusha N. Dole and Sanjay D. Sawant

Department Quality Assurance Technique, STES's, Smt. Kashibai Navale College of Pharmacy Kondhwa, Pune, Maharashtra, India

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ABSTRACT

The present work deals with to development three rapid, precise and accurate spectrophotometric methods for the estimation of Rizatriptan Benzoatein bulk and dosage form. Method A is Absorption maxima where UV Spectra of Rizatriptan Benzoate showed that maximum absorbance at 225 nm. Method B First order derivative where derivative amplitudes were calculated by considering minima and maxima of the curve, Method C is Area under curve in which wavelength range 210-231 nm was selected for estimation of Rizatriptan Benzoate. Linearity was observed in the concentration range 2-10µg/ml for all the methods ($r^{2=}$ 0.996 for method A, r^2=0.9993 for method B and r^2=0.9992 for method C). The results of an analysis have been validated statistically, which confirm the accuracy and reproducibility of the methods. All the methods were found to be simple, precise and accurate and can be employed for routine quality control analysis of Rizatriptan Benzoate in bulk as well as in its tablet dosage form.

Keywords: Rizatriptan Benzoatein, UV-spectrophometry, Absorption maxima, first order derivative spectroscopy, area under the curve.

INTRODUCTION

Rizatriptan Benzoate is described chemically as the *N*,*N*-Dimethyl-2-[5-(1*H*-1,2,4-triazol-1-ylmethyl)-1*H*-indol-3-yl]ethanamine benzoate.. Rizatriptan Benzoate is official in European Pharmacopoeia 7.3[(1)].Literature survey reveals that some methods have been developed for their determination by HPLC[(2-4)] or spectrophotometry [(5-6)]either alone or in combination. The purpose of this work was to develop a simple, accurate, precise, reproducible and economical UV spectrophotometric first order derivative spectroscopy and Area Under Curve method and Absorption maxima for estimation of Rizatriptan Benzoate.

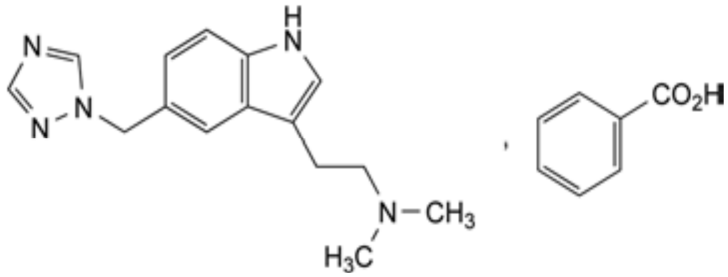

Figure 1: Chemical Structure of Rizatriptan Benzoate

MATERIALS AND METHODS

Apparatus and Instrumentation

Jasco V630 with Spectra manager software, was employed for this work. Single pan electronic balance (Mettler Toledo ME204) was used for weighing purpose. Sonication of the solutions was carried out using an Ultrasonicator (Spectra lab UCB 40, India). Calibrated volumetric glasswares (Borosil) were used in this study.

Materials

Active pharmaceutical ingredient (API) of Rizatriptan Benzoate was procured from **Cipla pharmaceuticals,Solan.** Commercially available tablets manufactured by Cipla RIZACT-10 containing 10mg of Rizatriptan Benzoate was procured from local pharmacy.

METHOD DEVELOPMENT

Preparation of standard solution

The standard stock solution of Rizatriptan Benzoate was prepared by transferring, accurately weighed 100mg of API to100ml of volumetric flask. The drug was dissolved with sonication in 50ml of distilled water and volume was made up to the mark by using distilled water. The standard stock solution (1000µg/ml) was further diluted with distilled water to get the concentration of 10µg/ml.

METHOD A–ABSORPTION MAXIMA METHOD

1.**Calibration Curve for Rizatriptan Benzoate**

For the selection of analytical wavelength standard solutions of Rizatriptan Benzoate were prepared and scanned from 200-400nm.Furthe dilutions of standard solutions of Rizatriptan Benzoate were prepared by using distilled water and were scanned from 200 to 400nm.

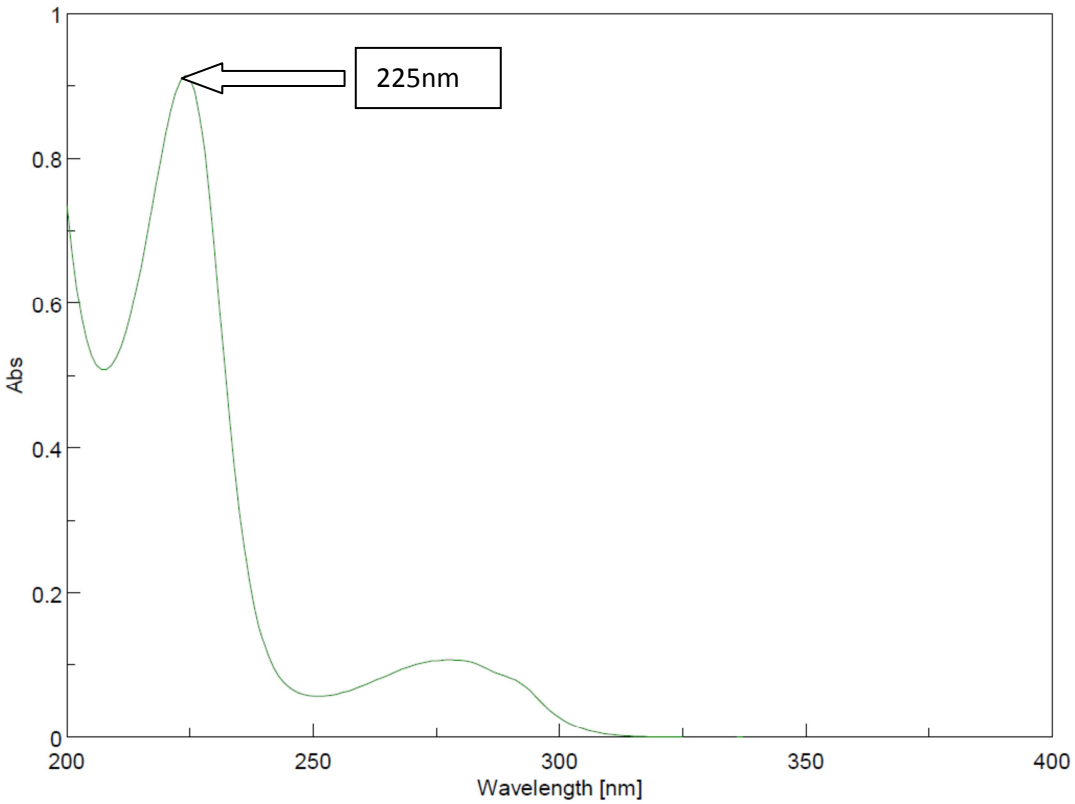

Figure 2: UV Spectra of 10µg/ml Rizatriptan Benzoate solution

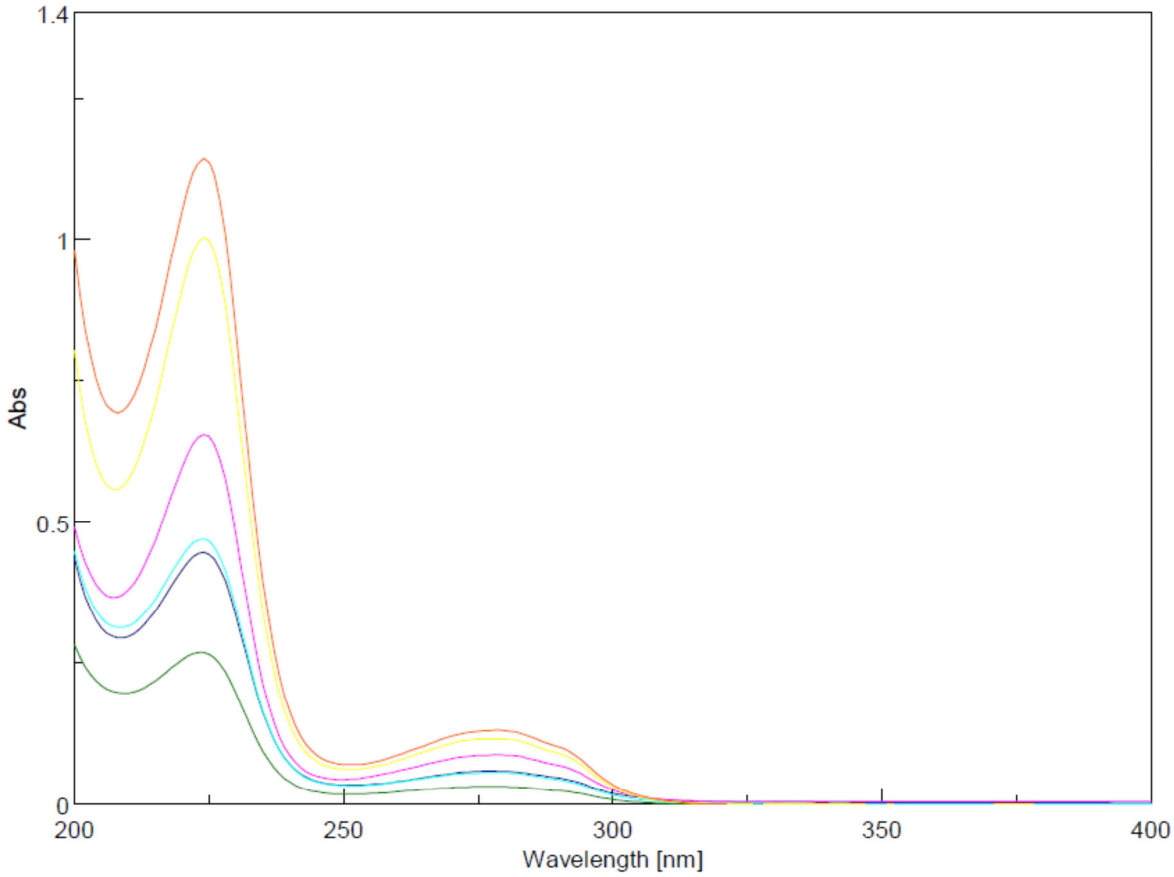

Figure 3: Overlay spectra of Rizatriptan Benzoate for Absorption Maxima method

2. Analysis of marketed formulation

Twenty tablets were weighed and average weight was calculated. These tablets were crushed and powdered in a glass mortar. The tablet powder equivalent to 10 mg of Rizatriptan Benzoate was accurately weighed, transferred to a 100 ml of volumetric flask and diluted up to mark with water. The solution was filtered with Whatmann filter paper No. 41 and the first 5 ml of filtrate was discarded. This solution was further diluted to obtain 10µg/ml solution with same solvent and the absorbance was measured at 225 nm and data obtained were shown in Table No1.

METHOD B -FIRST ORDER DERIVATIVE SPECTROSCOPY

1.Calibration curve for Rizatriptan Benzoate

From the standard stock solution, appropriate dilutions were made to obtain concentration in range of 2-10µg/ml. Absorbance measured at first order derivative spectrum is characterized by a maximum and minimum and a cross over point at λ max of absorption band. Further the absorbance difference at n=1 (dA/dλ) was calculated by the inbuilt software of the instrument which was directly proportional to the concentration of the standard solution. The calibration curve of dA/dλ versus concentration was plotted. (Figure 3).

2.Analysis of marketed formulation

Twenty tablets were weighed and average weight was calculated. These tablets were crushed and powdered in a glass mortar. The tablet powder equivalent to 10 mg of Rizatriptan Benzoate was accurately weighed, transferred to a 100 ml of volumetric flask and diluted up to mark with water. The solution was filtered with Whatmann filter paper No. 41 and the first 5 ml of filtrate was discarded. This solution was further diluted to obtain 10µg/ml solution with same solvent and subjected for UV analysis. In first order derivative spectroscopy, derivative amplitude was determined directly from calibration plot by measuring difference between absorbance maxima at 231 nm and minima at 217 nm (i.e. dA/dλ) (table no. 1).

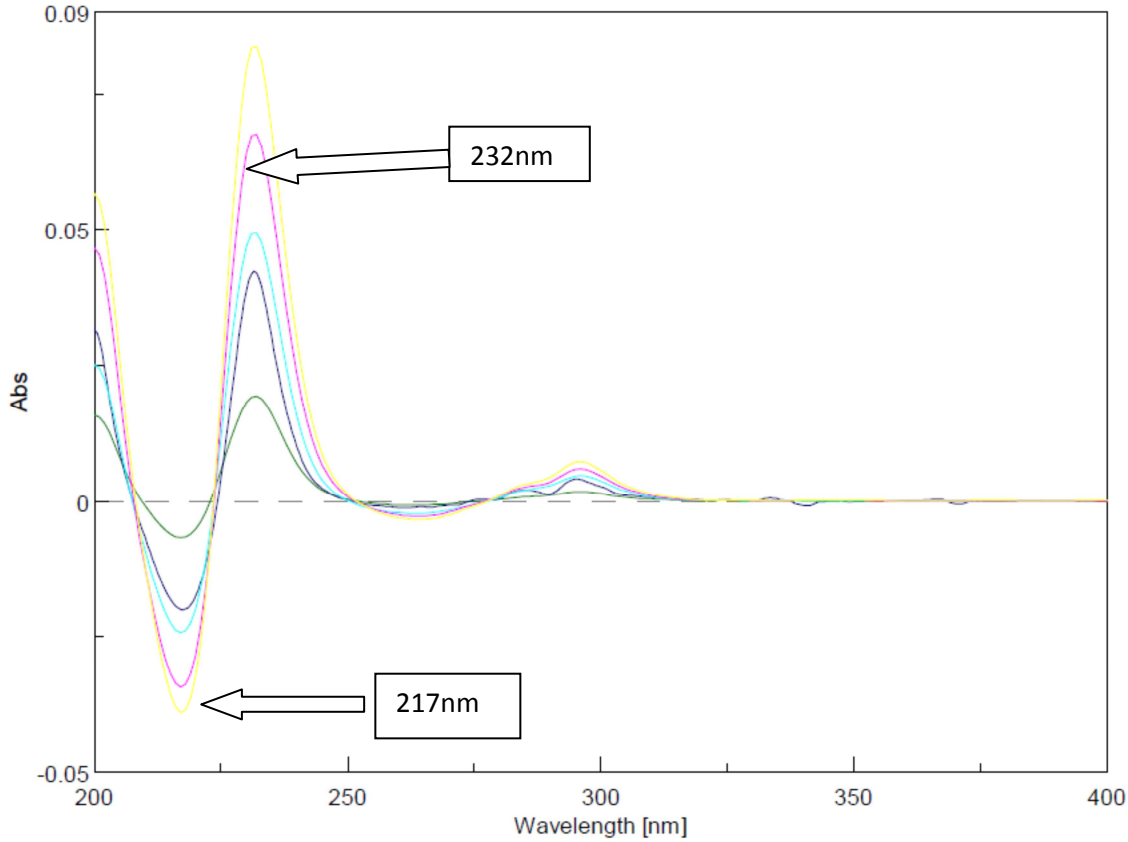

Figure 3: Overlay spectra of Rizatriptan Benzoate for first order derivative method

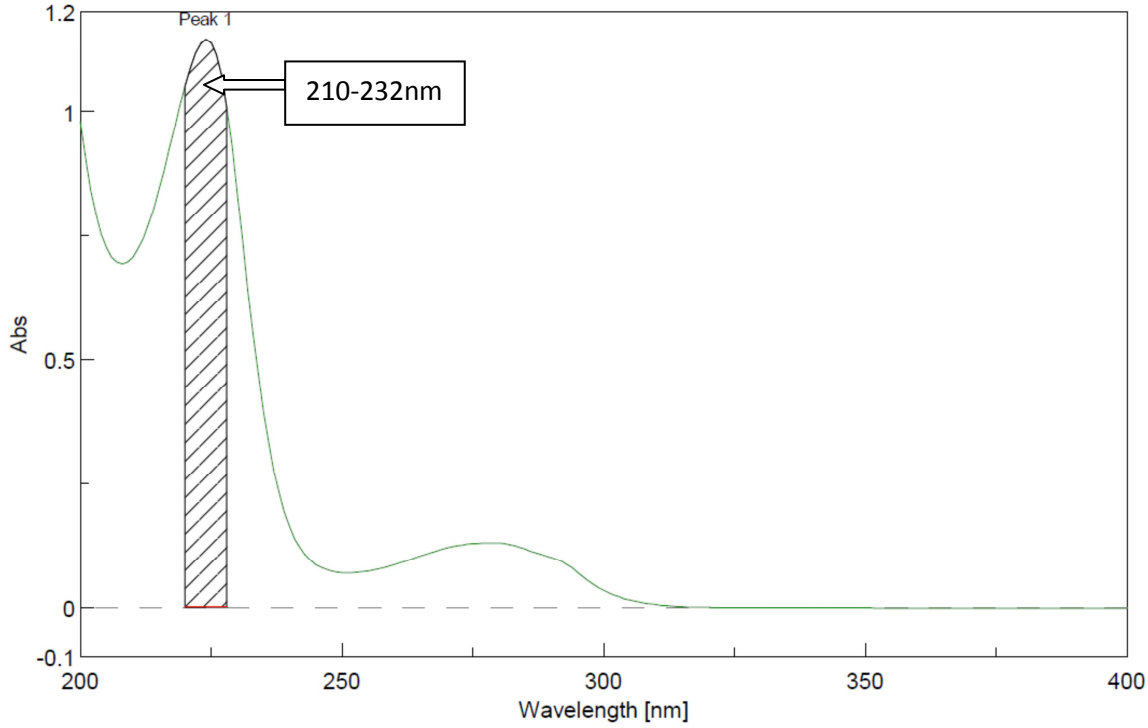

Figure 3: Area Under Curve graph of 10µg/ml Rizatriptan Benzoate solution

METHOD C -AREA UNDER CURVE

1.Calibration curve for Rizatriptan Benzoate

For the selection of analytical wavelength standard solutions of Rizatriptan Benzoate were prepared and series of dilutions of standard solutions of Rizatriptan Benzoate were prepared by using distilled water and were scanned from 200 to 400nm. From the spectra of drug obtained after scanning of standard solution of Rizatriptan benzoate area under the curve in the range of 210 nm-232 nm was selected for the analysis, the calibration curve was plotted with concentration versus area under the curve and regression equation was calculated. (Figure.2)

2. Analysis of marketed formulation

Tablets were procured from local market and average weight was determined. For the analysis of drugs, quantity of powder equivalent to 100mg of Rizatriptan Benzoate was transferred to 100ml volumetric flasks and dissolved in sufficient quantity of distilled water. It was sonicated for 45 minutes and then filtered through whatmann filter paper No 41. The filtrate was appropriately diluted with distilled water to give standard stock solution of 1000 µg/ml. Further dilutions were made using distilled water to get required concentration. Area in the range of 210 nm-231 nm was measured and content was calculated (table no. 1)

Table No. 1: Result of marketed formulation analysis

Method	Label claim(mg)	%Label Claim*(Mean±SD)	%RSD*
Absorbance maxima	**10**	**99.98±0.1835**	**0.19**
First order derivative	**10**	**101.30±0.0049**	**0.8087**
Area under curve	**10**	**100.20± 0.0012**	**0.0124**

Accuracy studies:

Accuracy studies were carried out by standard addition method. Pure Rizatriptan Benzoate was added at different levels i.e. 80%, 100% and 120% to pre analyzed sample solution. 100mg of Rizatriptan Benzoate was accurately weighed and transferred into a 100ml volumetric flask. This was dissolved in distilled water and volume was made up to 100ml to give standard stock solution of 1000µg/ml. To 1ml tablet stock solution in 3 different volumetric flask, aliquots of 0.8 ml, 1ml and 1.2ml of standard stock solution were added, volume was made up to 100ml with distilled water to give concentration of 18µg/ml (80%), 20µg/ml (100%) and 22µg/ml (120%).These were analyzed by absorbance maxima, first order derivative and area under the curve method. Procedure was repeated 3 times for 80%, 100% and 120% of level for recovery studies. The results are discussed in (table no. 2).

Results of Accuracy studies

Table No.2: Results of Accuracy studies

Level Recovery	Conc. Of Drug (µg/ml)		Total Conc. (µg/ml)	Method A *		Method B*		Method C*	
	Drug taken	Std drug added		% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
80	10	8	18	101.43	0.0283	98.98	0.450	101.2	0.373
100	10	10	20	102.33	0.0056	101.49	0.325	100.9	0.0302
120	10	12	22	101.62	0.0056	101.52	0.109	100.6	0.0099

Validation

The developed method was validated as per ICH (ICH Q2 R1) guidelines.

Linearity:

The linearity of measurement was evaluated by analyzing different concentrations of the standard solution of Rizatriptan benzoate. The Beer's law was obeyed in the concentration range 2-10 µg/ml. The correlation coefficient was found to be 0.9996,0.9993 and 0.9992 for method A,method Band method C respectively.

Table 3. Linearity and range of Rizatriptan benzoate

Concentration	Method A	Method B	Method C
2	0.2677	0.0125	5.0699
4	0.4744	0.021	8.4393
6	0.7078	0.029	11.9984
8	0.9138	0.037	15.9897
10	1.1437	0.044	19.5565

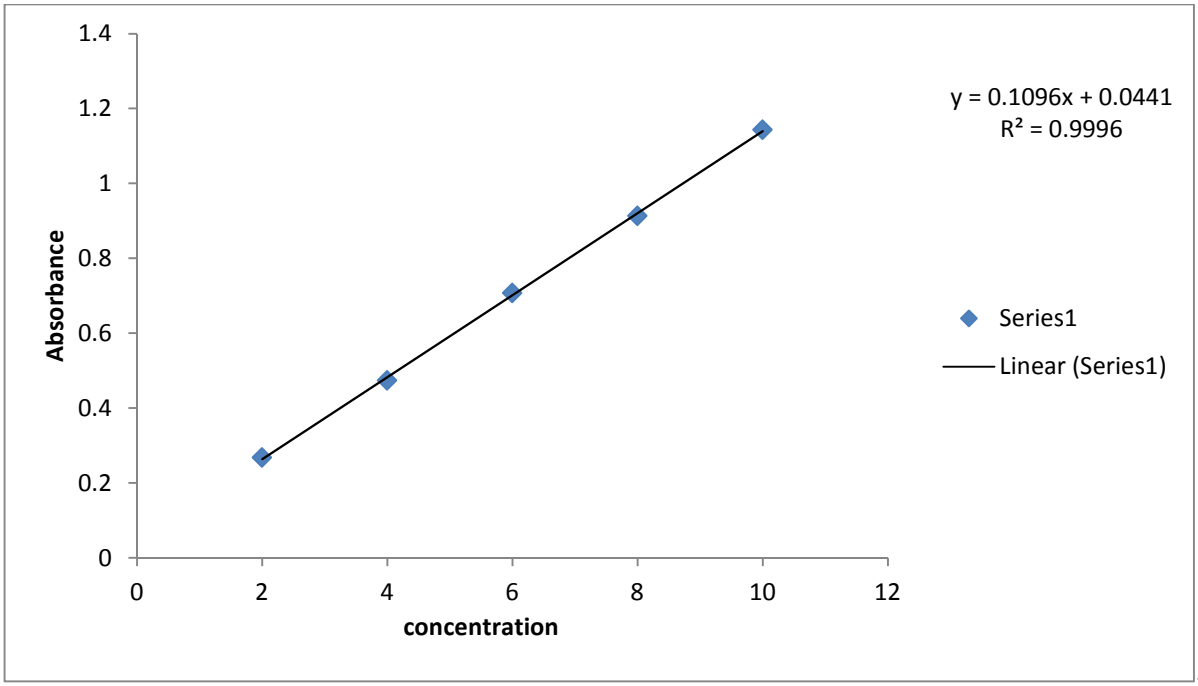

Figure 5: Calibration curve for Absorbance MAxima of Rizatriptan benzoate

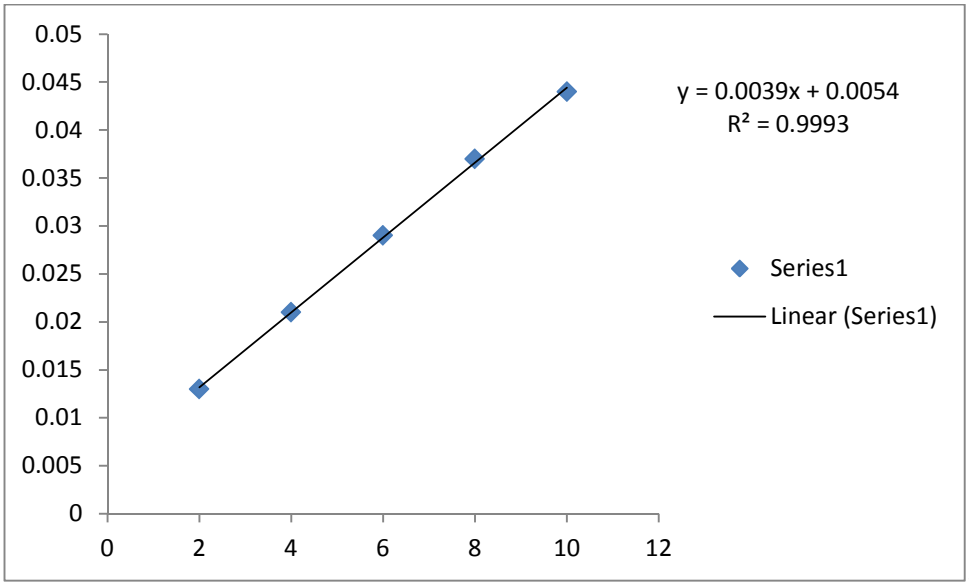

Figure 5: Calibration curve for Frist order derivative of Rizatriptan benzoate

Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Five sets of known concentrations (2-10µg/ml) were prepared. Calibration curves were plotted for each set. LOD and LOQ were calculated using the regression equation and following formulae as:

LOD = 3.3 * SD/S
LOQ = 10 * SD/S

Where,
SD is standard deviation of y-intercept of the calibration curves
S is mean slope of six calibration curves.

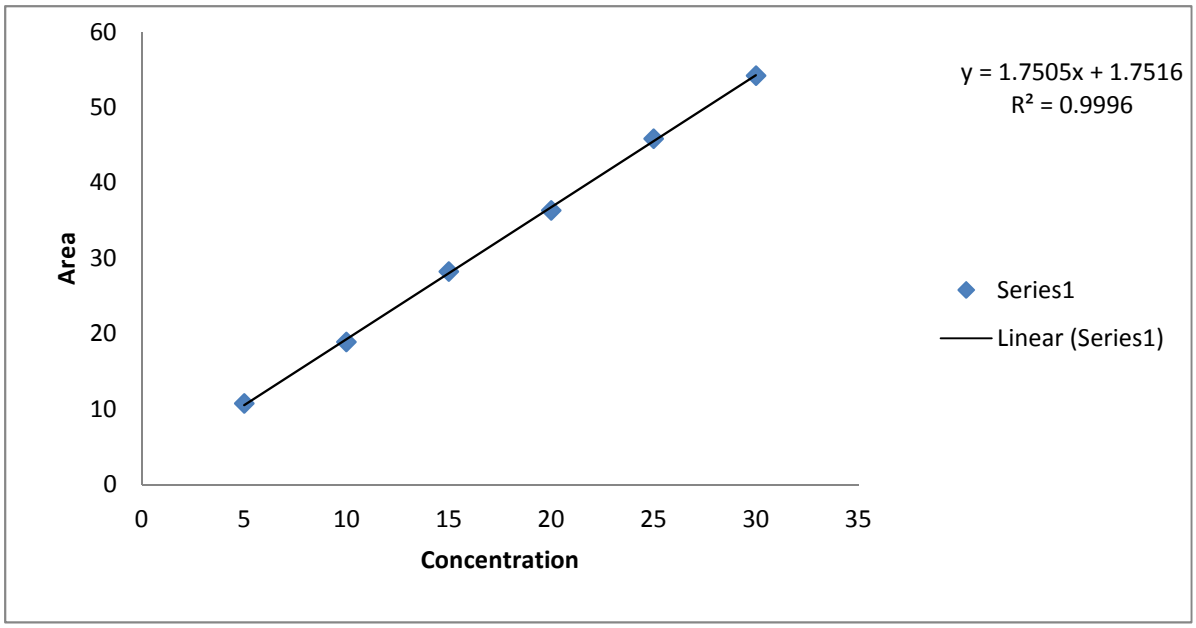

Figure 6: Calibration curve for Area under derivative of Rizatriptan benzoate

Table 4. LOD and LOQ of Rizatriptan benzoate.=

Paramters	Method A	Method B	Method C
LOD	0.46	0.46	0.75
LOQ	1.55	2.07	2.27

Precision:

100mg of Rizatriptan Benzoate was weighed accurately and dissolved in 100ml of distilled water to give concentration of 1000µg/ml. From the standard stock solution appropriate quantity of solution was taken further dilutions were made with distilled water to give 4µg/ml. This procedure was repeated six times for each method individually. (Table No.3 & 4)

Table 5. Intraday and Interday precision study of Rizatriptan Benzoate Method A

Drug	Concentration (µg/ml)	% RSD*	
		Intraday	Interday
Rizatriptan benzoate	4	0.9809	1.04
	4	0.041	0.005
	4	0.002	0.08

Table 5. Intraday and Interday precision study of Rizatriptan BenzoateMethod B

Drug	Concentration (µg/ml)	% RSD*	
		Intraday	Interday
Rizatriptan benzoate	4	0.7779	1.1926
	4	0.77	2.73
	4	0.0699	0.061

**n=3*

Table 6. Intraday and Interday precision study of Rizatriptan benzoate. Method C

Drug	Concentration (µg/ml)	% RSD*	
		Intraday	Interday
Rizatriptan benzoate	4	3.77	3.84
	4	0.88	0.234
	4	0.39	0.11

**n=3*

RESULTS AND DISCUSSION

The present work describes the absorbance maxima, first order derivative method and area under curve method for the estimation of Rizatriptan Benzoate in bulk and dosage form. For all the methods linearity was observed in the concentration range of 2-10 µg/ml for Rizatriptan Benzoate and correlation coefficient was found to be greater than

0.9996. Accuracy of proposed method was assessed by recovery studies. The results of marketed formulation analysis are found in range of 99-101%. Percent recovery for Rizatriptan Benzoate by all the three the methods was found in the range of 98.20% to 102 % (Table No. 2). The % RSD for six determinations of tablet sample by all the three the methods was found to be less than 2.0 indicating the good precision of both the methods. Hence, it can be concluded that the developed spectrophotometric methods are accurate, precise and can be employed successfully for the estimation of Rizatriptan Benzoate in bulk and formulation

CONCLUSION

The three spectrophotometric methods were developed for Rizatriptan Benzoate and validated as per ICH guidelines. The standard deviation and % RSD calculated for the proposed methods are within limits, indicating high degree of precision of the methods. The results of the recovery studies performed indicate the methods to be accurate. Hence, it can be concluded that the developed spectrophotometric methods are accurate, precise and can be employed successfully for the estimation of Rizatriptan Benzoate in bulk and dosage form.

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