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A NEW METHOD DEVELOPMENT AND VALIDATION FOR ESTIMATION OF ELETRIPTAN BY USING RP-HPLC METHOD

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ABSTRACT

A simple, rapid, specific, accurate and precise reverse phase high performance liquid chromatographic method was developed for estimation of eletriptan in tablet dosage form. Chromatographic separation was carried out by using mobile phase 0.02M potassium dehydrogenate Phosphate buffer (ph 3.0) : Acetonitrile (50:50 v/v, PH-3.0 adjusted with Orthophosphoric acid) on Hypersil, Symmetry C_8 150×4.6mm, 5µm at flow rate 0.8ml/min in isocratic mode and effluents are monitored at 221 nm. The retention time for eletriptan was found to be 2.45 min respectively and calibration curves were linear over concentration from 10-50µg/ml. The method was validated for accuracy, precision, specificity, linearity, system suitability. LOD and LOQ were 0.0258µg/ml and 0.0897µg/ml for eletriptan. The percentage recovery of eletriptan was found to be 98-102%.The developed method was fast, accurate, precise, and successfully applied to estimate the amount of eletriptan in bulk sample and tablet dosage form so it can be used for quality control department.

Keywords: Eletriptan, RP-HPLC, Hypersil column.

INTRODUCTION

Eletriptan hydro bromide is a second generation triptan drug and it intended for treatment of migraine headaches [1-3]. It is used as an abortive medication, and blocks migraine attack which is already in progress. Eletriptan chemically known to be 3-[(-1-methylpyrrolidin-2-yl) methyl]-5-(2-phenylsulfonylethyl)- 1H-indole and brand name is Replax.Eletriptan reduce swelling of the blood vessels surrounding the brain. This swelling is associated with the head pain of a migraine attack and blocks the release of substances from nerve endings that cause more pain and other symptoms like nausea, and sensitivity to light and sound. Mechanism action of eletriptan is selective at 5-$HT_{1B/1D}$ receptor agonist; thought to be due to the agonist effects at the 5-$HT_{1B/1D}$ receptors located on intracranial blood vessels (including arteriovenous anastomoses) and sensory nerves of the trigeminal system that result in cranial vessel constriction and inhibition of proinflammatory neuropeptide release [4-6].

MATERIALS AND METHODS

Equipment

High performance liquid chromatography of WATERS-n2000 chromatographic system software, waters 515 pump, 2487with photo diode array (PDA) detector, UV-Spectrophotometer UV- 2310 of TECHCOMP company.

Chemicals and Reagents

Methanol, Acetonitrile (HPLC grade) was used. Buffers used was Potassium dehydrogenate phosphate, Orthophosphate. Reference standards Eletriptan were obtained from pharma-tech labs pvt ltd.

Chromatographic conditions for RP-HPLC method

Chromatographic separation was carried out by using mobile phase 0.02m potassium dehydrogenate phosphate buffer (pH 3.0): Acetonitrile (50:50 v/v, ph-3.0 adjusted with Orthophosphoric acid) on Hypersil, symmetry C_8 150×4.6 mm, 5µm at flow rate 0.8ml/min in isocratic mode and effluents are monitored at 221 nm. The

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retention time for eletriptan was found to be 2.45 min respectively and calibration curves were linear over concentration from 10-50µg/ml in (figure no 1).

Preparation of standard solutions preparation

Accurately weighed and transferred 10 mg of Eletriptan working standard into a 10 ml clean dry volumetric flask added about 7 ml of solvent and sonicated to dissolve it completely and made the volume up to the mark with the same solvent in (figure no 2).

Preparation of Sample Solutions Preparations

Accurately weighed and transferred 55 mg of Eletriptan Tablet powder into a 10 ml clean dry volumetric flask added about 7 ml of solvent and sonicated to dissolve it completely and made the volume up to the mark with the same solvent in (figure no 3).

Preparation of buffer solutions (buffer 0.02M KH_2PO_4)

Added 2.72gms of KH2PO4 to HPLC water in 1000 ml beaker, diluted to 1000 ml with HPLC water. PH of the solutions was adjusted to 3.0 with Orthophosphoric acid. 500 mL buffer (50%) and 500 mL of Acetonitrile (50%) were mixed, degassed in ultrasonic water bath for 5 minutes and filtered through 0.45 µ filter under vacuum filtration.

Optimized chromatographic conditions

Flow rate	**:** 0.8(ml/min)
Column	**:** Symmetry C_8 (150×4.6 mm, 5µm, Make: X-terra)
Detector wave length	**:** 221
Column temperature	**:** Ambient
Injection volume	**:** 20µL
Run time	**:** 6 min
Solvents used	**:** Phosphate buffer (pH 3.0): Acetonitrile 50: 50 %

RESULTS AND DISSCUSSION

The developed method for determination of Eletriptan was evaluated by validation parameters:

Accuracy

The accuracy studies were performed by standard addition method and % recovery for Eletriptanat 50 %, 100 % and 150 % the limits of % recovered should be in range of 98-102 % the results obtained for Eletriptanwere found to be within the limits and results are shown in (table no 1).

Linearity

It is an analytical procedure is its ability with in a given range to obtain test results which are directly proportional to the concentration of analyte in the sample observed in figure no 4. Accurately weighed and transferred 10 mg of Eletriptan working standard into a 10 ml clean dry volumetric flask and added about 7 ml of diluents and sonicated to dissolve it completely and made volume up to the mark with the same solvent. (Stock solution).from the stock solution prepare concentrations of 10µg/ml, 20µg/ml, 30µ/ml, 40µg/ml, 50µg/ml and injected, the linearity (Correlation coefficient) should be not less than 0.999 seen in (table no 2).

Precision

The solution was injected for five times and measured the area for all five injections in HPLC. The % RSD for the area of five replicate injections was found to be within the specified limits and it was less than 1.0%.

Robustness

As part of the Robustness, deliberate change in the Flow rate, Mobile Phase composition, Temperature Variation were made to evaluate the impact on the method. so the evaluation done for flow rate ± 10%, Organic composition in the Mobile phase was varied from 40% to 60%, temperature($\pm 5^0$c).

Limit of detection (LOD) and limit of quantification (LOQ)

The limit of detection and limit of quantification was calculated from Slope and Standard deviation of response and LOD limit of Eletriptan is 0.02µg/ml, LOQ limit is 0.08µg/ml respectively. The results indicates minimum level at which the Analyte was Quantified and detected with acceptable accuracy and precision.

System Suitability Parameters

System suitability is the evaluation of the components of an analytical system to show that the performance of a system meets the standards required by a method. A system suitability evaluation usually contains its own set of parameters. For chromatographic assays, these may include tailing factor, resolution, precision, capacity factor, retention time and theoretical plates. System suitability parameter Results were reported in (table 3).

Table 1. Accuracy at 3 levels

% Concentration (at specification Level)	Area	Amount Added (mcg)	Amount Found (mcg)	% Recovery
50 %	1982240	15.0	15.09	100.6
100 %	2594119	30.0	30.09	1000.3
150 %	3269320	45.0	45.39	100.3

Table 2. Linearity

S.No	Linearity Level	Concentration	Area
1	I	10 ppm	634405
2	II	20 ppm	1192275
3	III	30 ppm	1832861
4	IV	40 ppm	2374397
5	V	50 ppm	2956772
Correlation Coefficient			0.9997

Table 3. Validation parameters

S.No	Parameter	Acceptance criteria	Results Obtained
1	System suitability	Theoretical Plates-NLT2000	2754
		Tailing factor-NMT 2	1.6
		Resolution- NLT 2	0.0
2	Precision	% RSD	0.34
3	ID Precision	% RSD	0.33
6	Linearity	Correlation coefficient NLT 0.999	0.9997
7	Accuracy	Percentage Recovery98-102%	100.3
8	LOD	0.02ug/ml	
9	LOQ	0.08ug/ml	

Figure 1. UV-spectrum of Eletriptan

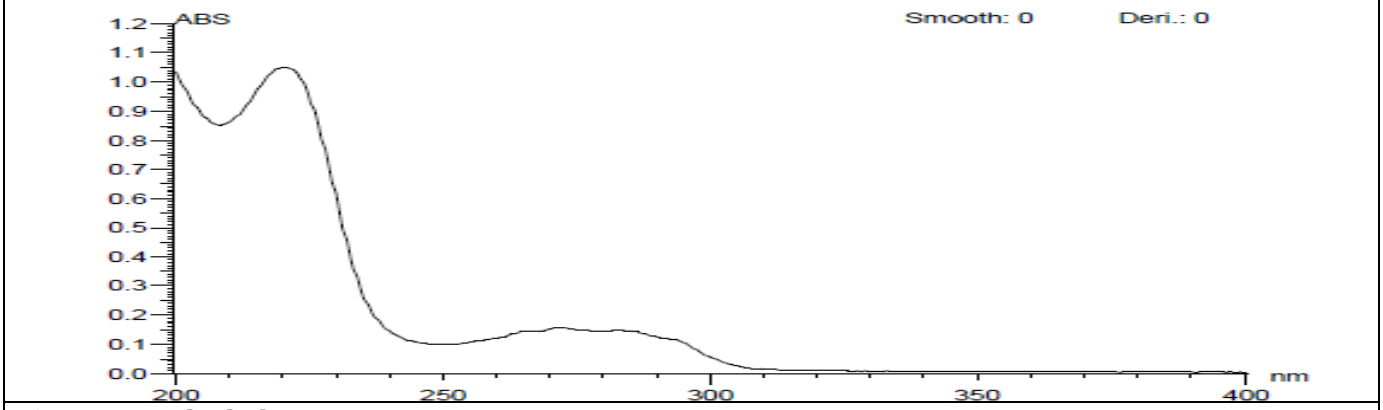


Figure 2. Standard Chromatogram – 1

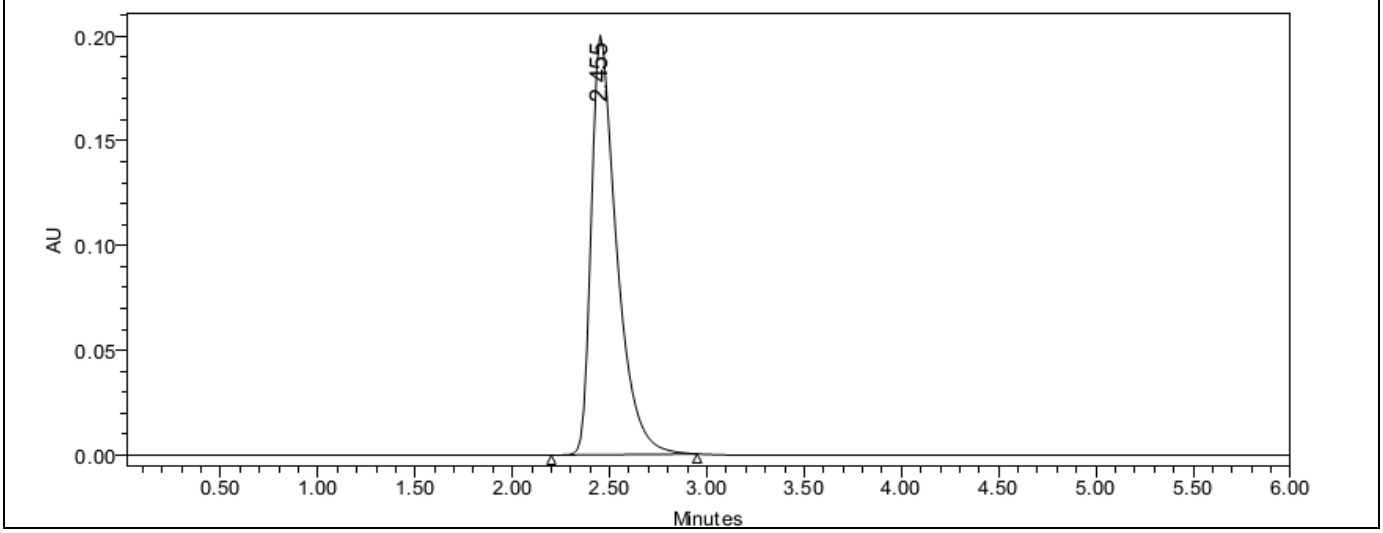


Figure 3. Sample chromatogram -1

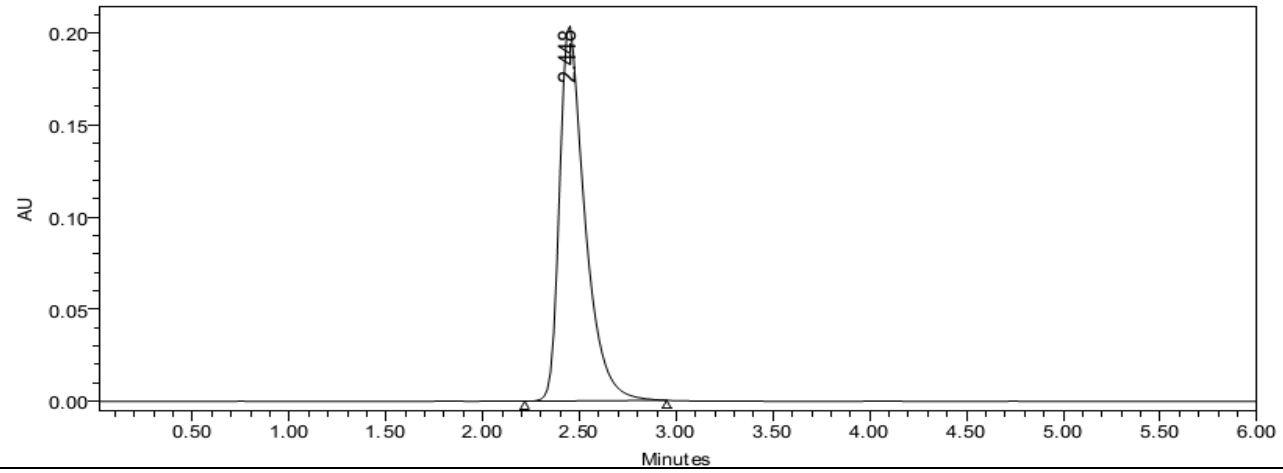
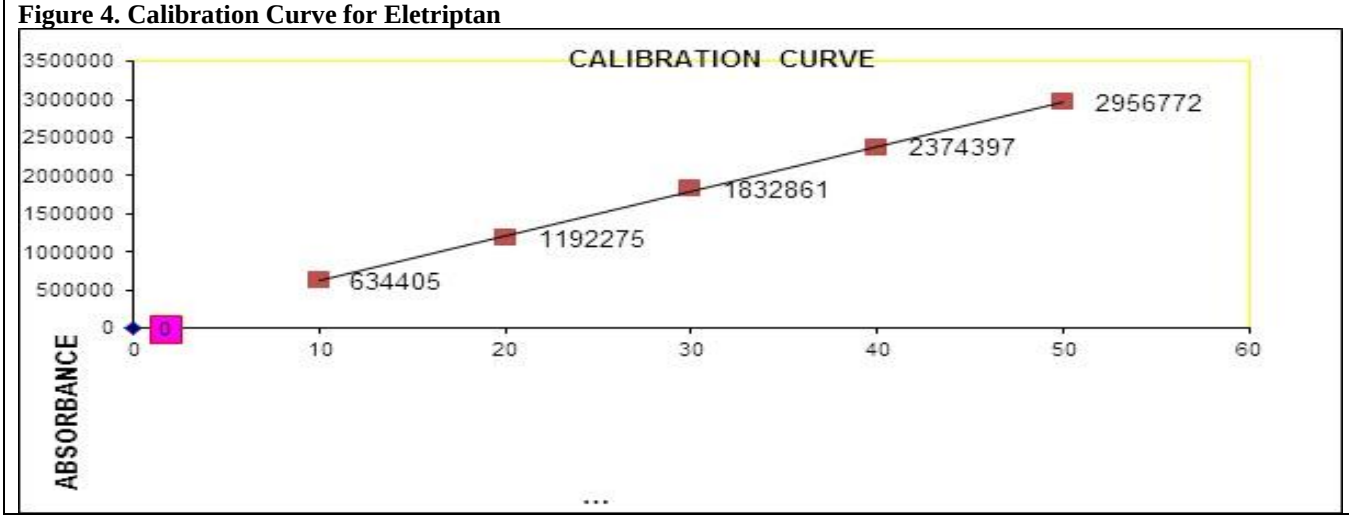


Figure 4. Calibration Curve for Eletriptan



CONCLUSION

The new RP-HPLC method was developed and validated for pharmaceutical dosage form of eletriptan according to international conference on harmonization (ich) guidelines and it is applicable for routine analysis of quality control department of raw materials, formulations and dissolutions studies. The developed method was found to be simple, accurate, rapid, economical and reproducible with high sensitivity.

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