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Validated Colorimetric methods for the determination of Tapentadol in bulk and its tablet dosage form

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Abstract: Tapentadol (TAP) is a novel opioid pain reliever drug that is unusual in its possession of dual mechanism of action (mu opioid-receptor agonist and nor adrenaline reuptake inhibitor); this feature makes the active ingredient an attractive potential progenitor of a new pharmacological class. Two simple, rapid, sensitive and reproducible visible spectrophotometric methods were developed for the determination of Tapentadol HCl (TAP) in bulk and in its tablet dosage form. First method (M_1) employs formation of orange red colour complex by the reaction with bypyridyl in the presence of ferric chloride, which shows absorption maxima at 500 nm. The second method (M_2) was based on the reaction of TAP with ferric ammonium sulphate-1, 10 phenanthroline that produces Red coloured complex, exhibits an absorption maximum at 617 nm. Regression analysis of Beer-Lambert plots showed good correlation in the concentration ranges 10-60 µg/ml for method M_1, and 10 - 80 µg/ml µg/ml for method M_2 respectively. Commercially available tablets were analyzed; the results obtained by the proposed methods were in good agreement with the labeled amounts. These methods offer the advantages of rapidity, simplicity, sensitivity and normal cost and can be easily applied to resource-poor settings without the need for expensive instrumentation and reagents.

Key words: Tapentadol, bypyridyl, phenanthroline, visible spectrophotometric.

Contents

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1. Introduction

Tapentadol Hydrochloride (TAP) a new drug, which is chemically (E3-[(1R, 2R)-3- dimethylamino)-1-ethyl-2-methylpropyl] phenol monohydrochloride (Figure 1). It is not official in any pharmacopoeia. TAP is a centrally acting narcotic (opioid) analgesic with a dual mechanism of action. Like classic narcotics such as morphine and hydrocodone, tapentadol activates μ-opioid receptors. In addition, similar to tricyclic antidepressants, tapentadol blocks the neuronal reuptake of norepinephrine, which, in turn, increases synaptic concentrations of this neurotransmitter [1-3]. Up till now only few methods are reported for estimation of TAP which includes, estimation of tapentadol and its metabolite N-desmethyl tapentadol in urine and oral fluids by using ultra pressure liquid chromatography with tandem mass detection (LC-MS/MS) [4] and HPLC [5-9]. A method has been reported on symultanious estimation of tapentadol with paracetamol by RP-HPLC [10-12]. Furthermore one study is reported which discusses about determination of four stereoisomers of TAP by X-ray crystal structure analysis[13]. Two simple, rapid, sensitive and reproducible visible spectrophotometric methods were developed for the determination of Tapentadol HCl (TAP) in bulk and in its tablet dosage form, which can be implemented easily for routine use in quality control laboratories. The statistical analysis proved that these methods were reproducible and selective for the analysis of TAP in bulk drug and tablet formulation. Confirmation of the applicability of the developed method was validated according to the International Conference on Harmonization (ICH) [14] for the determination of Tapentadol HCl in bulk and in tablet dosage form.

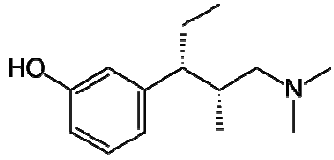

Figure 1: Structure of tapentadol

2. Materials and Methods

Instruments:

Shimadzu UV-1800, UV-Visible double beam Spectrophotometer with matching pair of 1 cm quartz cuvettes (Shimadzu Corporation, Kyoto, Japan). The spectral bandwidth is 0.5 nm.

Reagents and Chemicals

Pure drug powder (> 99.9% purity) of TAP was supplied by MSN Laboratories Pvt. Ltd., Hydrabad. The tablet Dosage form of TAP was purchased from Anukar Pharmacy, Hyderabad. The chromogenic reagents 2, 2'-bipyridyl and ferric ammonium sulphate -1, 10-phenanthroline, were purchased from Merck, Mumbai. Water was obtained by distilling deionised water produced by a Milli-Q millipore water system (Milford, MA, USA). All the other reagents and materials were of analytical grade and supplied from commercial sources.

Preparation of acetate buffer pH 4.6

5.4 gm of sodium acetate was dissolved in 50 ml of distilled water. 2.4 ml of glacial acetic acid was added and diluted with distilled water to 100ml.

Preparation of 2, 2'-bipyridyl solution

100mg of 2.2'-bipyridyl was dissolved in sufficient amount of distilled water and the volume was made upto100ml with the same.

Preparation of Ferric ammonium sulphate-1, 10–phenanthroline solution

2 ml of 1 M HCl was added to 0.19 g of 1, 10-Phenanthroline and 0.16 g of ferric ammonium sulphate was added to it and dissolved in distilled water with vigorous shaking and finally the volume was made upto 100 ml with distilled water.

Preparation of standard stock solution: Tapentadol hydrochloride, equivalent to 100 mg of Tapentadol was accurately weighed and transferred to 100 ml volumetric flask. 20 ml of Water was added and sonicated for 30 min. The volume was made up to the mark with water to give 1000 μg/ml solution. Working standard (100μg/ml) of TAP was prepared from this stock solution.

Determination of λ max

Method-1:

10 ml of working standard of the drug solution was taken in a beaker; 10 ml of ferric chloride solution was added and heated the mixture for 5-10 min at 60 °c. The reaction mixture was allowed to cool at room temperature, and finally added 10 ml of 2, 2'-bipyridyl, which produces orange red colour complex. The solution was scanned in the visible region (400-800 nm).

Method-2:

1ml of working standard of the drug solution was taken in a 10ml volumetric flask to this 1ml of ferric ammonium sulphate -1,10-phenanthroline and 4 ml of acetate buffer pH 4.5 was added, the reaction mixture was heated on a

water bath at 80°c for 10 mins. Then the mixture was allowed to cool at room temperature and the final volume was made upto the mark with distilled water. A red coloured complex was formed.

Preparation of sample solution

Method- 1:

Commercially available tablets of TAP 50mg were selected for the estimation of total content by the proposed method. An amount equivalent to 100 mg of Tapentadol hydrochloride was transferred into 100 ml volumetric flask, containing 20 ml of distilled water, mix thoroughly and the final volume was made up to the mark with the same. From the above solution 10 ml of the drug solution was taken in a beaker and 10 ml of ferric chloride solution was added and heated the mixture for 5-10 min at 60°c. The reaction mixture was allowed to cool at room temperature, and finally added 10 ml of 2, 2'-bipyridyl. The solution was filtered and first few ml of the filtrate was discarded and the resulting solution was determined for the absorbance at the proposed λ_{max}.

Method-2:

An amount equivalent to 100 mg of Tapentadol hydrochloride was transferred into 100 ml volumetric flask, containing 20 ml of distilled water, mix thoroughly and the final volume was made up to the mark with the same. From the above solution 10 ml of the drug solution was taken in a 100ml volumetric flask to this 10 ml of ferric ammonium sulphate -1, 10-phenanthroline and 40 ml of acetate buffer pH 4.5 was added, the reaction mixture was heated on a water bath at 80°C for 10 min. Then the mixture was allowed to cool at room temperature and the final volume was made upto the mark with distilled water. The solution was filtered and first few ml of the filtrate was discarded and the resulting solution was determined for the absorbance at the proposed λ max.

$$Percentage\ purity\ = \frac{(Sample\ absorbace\ XStandard\ Dilution\ XAverage\ Weight\ X\ Potency)}{standard\ absorbanceXSample\ DilutionX\ label\ Claim} X100$$

Method validation: The optimized spectrophotometric methods were validated according to the procedures described in ICH guidelines Q2 (R1) for the validation of analytical methods [12].

Accuracy: Accuracy of proposed method, were performed by recovery studies, standard addition method was employed at three different levels (50%, 100% and 150% of final concentration). A known amount of TAP pure drug was added to pre-analyzed tablet powder and the sample was then analyzed by proposed method. Results of recovery studies were found to be satisfactory and reported in table 1.

Precision: The precision of the method was determined by repeatability and intermediate precision (intraday and inter-day).

Intra-day precision: Intra–day precision was determined by analyzing the optimized concentration of drug (100µg/ml) for three times in the same day. %RSD was calculated and the results were shown in table 1.

Inter-day precision: Inter-day precision was determined by t analyzing the optimized concentration of drug (100µg/ml) for three days in a week. %RSD was calculated and the results were shown in table 1.

Limit of detection and Limit of quantification: Limit of detection (LOD) and Limit of Quantification (LOQ) were determined by using the formula based on the standard deviation of the response and the slope. The results were given in table 1.

3. Results and Discussion

Two simple, precise, accurate visible spectrophotometric methods have been developed for the estimation of TAP in pure and in formulations. The present study describes highly sensitive, economic, accurate, precise and reproducible methods for the determination of TAP. The λ_{max} of TAP by Method-1 was found to be 500nm and by Method-2 was found to be 617nm (Figure 2 & 3).

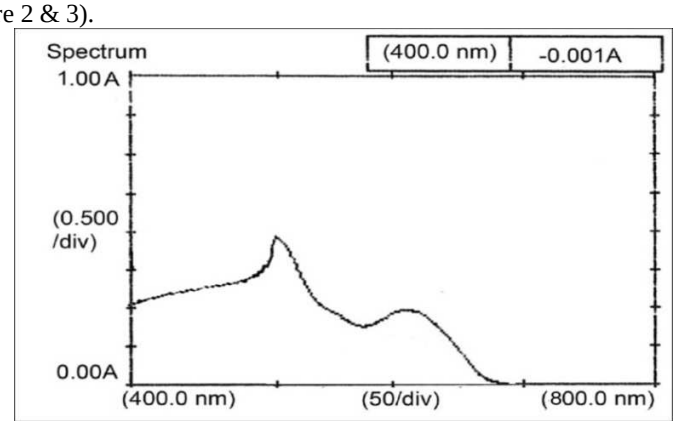

Figure 2: λ_{max} of Tapentadol (Method 1)

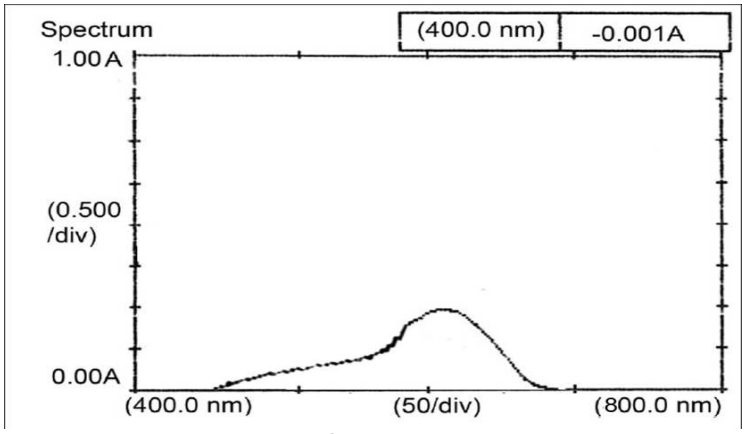

Figure 3: λ_{max} of Tapentadol (Method 2)

The Regression analysis of Beer-Lambert plots showed good correlation in the concentration ranges 10-60 µg/ml for method M_1, and 10 - 80 µg/mL µg/ml for method M_2 respectively (table 1). The correlation (r^2) value was found to be 0.999 and 0.997for Method 1 & 2 respectively (Figure 4 and 5) The percentage recovery values of pure drug from the analyzed formulation were in between 99.75% - 100.09% and 99.00-100.03% for Method 1 & 2 respectively (Figure 2 and 3). The precision of the proposed method was checked in terms of the repeatability, inter-day and intra-day time periods and %RSD was found to be less than 2%. LOD and LOQ were found to be 1.69 µg/ml & 2.40 µg/ml respectively for Method 1. And LOD and LOQ for Method 1was 5.14 µg/ml & 7.30 µg/ml respectively. The assay values for marketed formulation were found to be within limit. Hence the results of the analysis were validated and recovery studies were carried out as per ICH guidelines. Therefore the newly developed methods were successfully applied in tablet dosage form.

Table 1: Summary of Optical characteristics and Validation parameters

Parameters	Results	
	Method -1	**Method -2**
Lamda max (λ_{max})*	500nm	617nm
Beer's law limits (µg/ml)	10-60	10-80
Molar absorptivity(L/mol/cm)	9.02x 10-6	1.33 x 10-5
Sandell's sensitivity (µg/cm2/0.001absorbance unit)	0.109	0.0775
Regression equation	Y= 0.0095x-0.008	Y=0.010x+0.097
Slope(m)	0.0095	0.010
Intercept(c)	-0.008	0.094
Correlation coefficient (r2)	0.9998	0.9978
Precision (%RSD)**	0.03	1.28
LOD	1.69	2.409
LOQ	5.147	7.3
Assay (% Purity)**	100.00	99.98

* Average of 6 replicate samples; ** Average of 3 replicate samples.

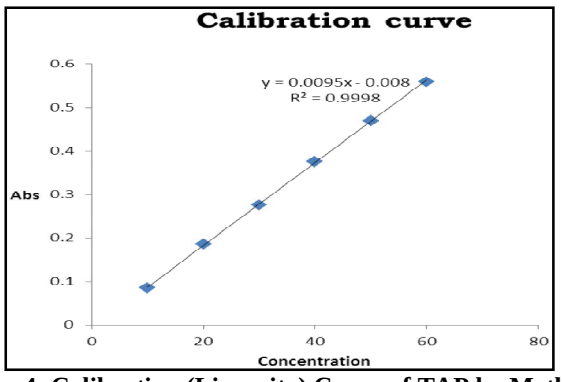

Figure 4: Calibration (Linearity) Curve of TAP by Method 1

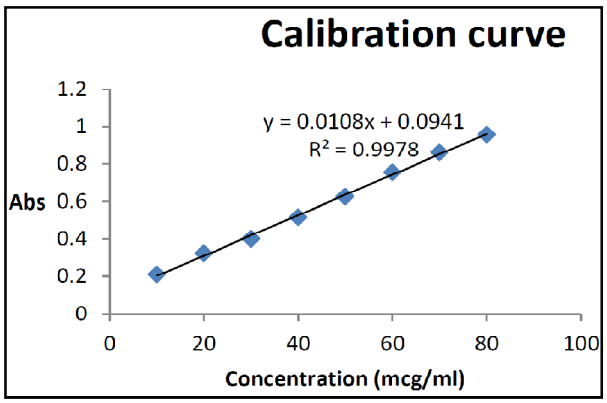

Figure 5: Calibration (Linearity) Curve of TAP by Method 2

4. Conclusion

The proposed analytical methods were rapid, accurate, precise and reproducible and hence can be used for the routine analysis of TAP in bulk, tablet dosage forms. High percentage recovery showed that the methods were free from interference of excipients used in the formulation. The most striking features of the methods were their simplicity and rapidity, not requiring tedious sample preparations such as extraction of solvents, degassing which are many needed for HPLC procedure. Values of LOD and LOQ showed that the proposed methods were too sensitive enough to analyze the drug in bulk and as well as in its pharmaceutical formulation. All the above result indicates that, the methods employed here were very simple, accurate, economic and rapid for routine analysis of the Tapentadol in quality control laboratories.

5. Acknowledgements

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