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Development and validation of uv spectrophotometric method for estimation of dexibuprofen in bulk and dosage form

Pritesh G. Dhartarkar*, Rajan V. Kalamkar, Suprit D. Saoji, Shailesh G. Ingle, Sandeep C. Atram, Madhuri D. Game.

Vidyabharti College of Pharmacy, Camp, Amravati, Maharashtra, India

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ABSTRACT

The aim of present work is to develop and validate simple, sensitive and specific spectrophotometric method for the determination of Dexibuprofen, a non-steroidal anti-inflammatory drug (NSAID) in pure form and in pharmaceutical formulations. UV spectrophotometric method, which is based on measurement of absorption at maximum wavelength in phosphate buffer pH 6.8, was found to be at 221.8 nm by using 5% methanol. The developed method was validated with respect to linearity, accuracy (recovery), precision and specificity. The optimum conditions for analysis of the drug were established. The drug obeyed the Beer's law and showed good correlation. Beer's law was obeyed in concentration range 0-60 µg/ml having line equation y = 0.046x +0.017 with correlation coefficient of 0.999. The results of analysis were validated by recovery studies. The method was found to be simple, accurate, precise, economical and robust.

Keywords: - Accuracy, Dexibuprofen**,** Recovery, UV spectrophotometric method.

INTRODUCTION

Dexibuprofen is known as chemically (2S)-2-[4-(2-methylpropyl) phenyl] propanoic acid [1,2,3]. The structural formula is $C_{13}H_{18}O_2$, and molecular weight is 206.28. It is non-steroidal anti inflammatory drug (NSAID). It is used for relief of symptoms of osteoarthritis, primary dysmenorrheal, muscular-skeletal pain or dental pain. It reduces gastric damage and improves analgesic and anti-inflammatory effect than racemic ibuprofen. The chemical structure of Dexibuprofen is as shown in figure I [4-7].

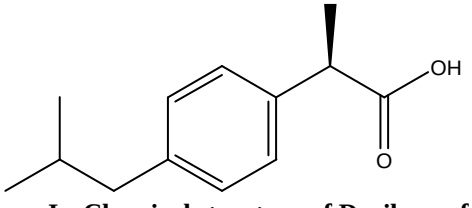

Figure I:- Chemical structure of Dexibuprofen.

A suitable and validated method is to be available for the analysis of drug in the bulk, dosage forms and in biological samples. If a suitable method, for specific need, is not available then it becomes essential to develop a simple, sensitive, accurate, precise, reproducible method for the estimation of drug samples.

The methods for estimation of Dexibuprofen given in literature are high performance thin layer chromatography (HPTLC)[8], RP-HPLC method[9], densitometric analysis of 2- aryl propionate derivatives in pharmaceutical preparations[10].

Although simultaneous UV estimation of Dexibuprofen and paracetamol has been reported, but single estimation of this drug has not been reported in bulk and in pharmaceutical formulation. Thus the present study was undertaken to develop and validate a simple, sensitive, accurate, precise, and reproducible UV spectrophotometric method for determination of Dexibuprofen.

MATERIALS AND METHODS

Apparatus:
Shimadzu UV 1800 spectrophotometer with 1 cm matched quartz cells were used for the absorbance measurements connected to computer and loaded with UV Probe software. Ohaus weighing balance and bath sonicator, borosil glass apparatus were used for experimental purpose.

Chemicals and reagents:
Dexibuprofen pure drug was obtained as a gift sample from Noven Lifesciences (P) Ltd. Hyderabad. Methanol, Potassium dihydrogen orthophosphate, Sodium hydroxides were purchased from S.D. Fine (P) Ltd. Mumbai. All chemicals and reagents used were of analytical grade.

Preparation of standard and test solutions
Dexibuprofen standard stock solution:
10 mg Dexibuprofen was accurately weighed and dissolved in 5 ml methanol then transferred to a 100 ml volumetric flask sonicate it for 5 min, finally volume was made up to the mark with phosphate buffer pH 6.8 to make 100µg/ml stock solution.

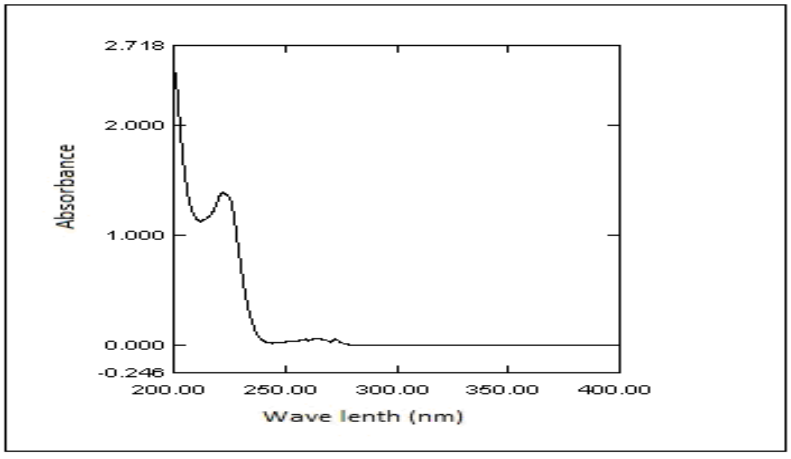

Figure II: Absorption spectrum of Dexibuprofen showing maximum absorbance at 221.8nm

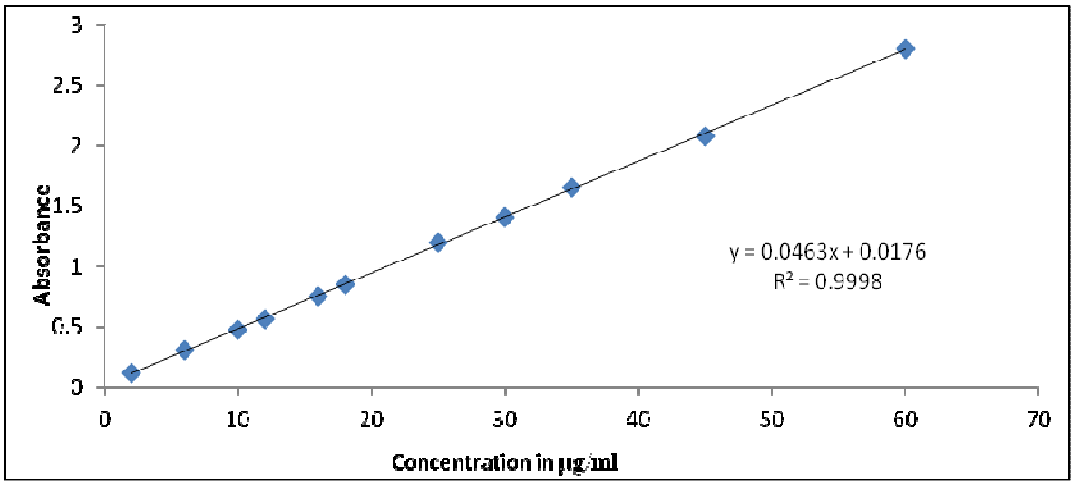

Figure III: Standard calibration curve for analysis of Dexibuprofen at 221.80 nm

Procedure for calibration curve:-

The standard solutions were prepared by the proper dilution of the primary stock solution with phosphate buffer pH 6.8 to obtain working standard. All the measurements were performed at room temperature. The absorbance of the solutions containing Dexibuprofen was determined in the UV range 200-800nm using an appropriate blank. The λ_{max} was found to be 221.8nm. The spectrum of Dexibuprofen was as shown in figure II. For linearity study, dilutions were made for Dexibuprofen in the range of 2 to 70 µg/ml concentrations were prepared by diluting the stock solution with phosphate buffer pH 6.8. The calibration curve was established at this wavelength by plotting graph between absorbance and concentration. The standard calibration was as shown in figure III.

Preparation of sample solution:

The proposed method was successfully applied for the determination of Dexibuprofen in tablet dosage form.

Ten tablets were weighed and powdered. The amounts of tablet powder equivalent to 30 mg of Dexibuprofen was weighed accurately and transferred to 5 ml methanol and kept for 5 min in sonicator and volume was made up to mark with phosphate buffer pH 6.8 in 100ml Volumetric flask. The solution was then filtered through Whatmann filter paper # 41. This filtrate was diluted suitably with phosphate buffer pH 6.8 to get the solution of 30µg/ml concentration. The absorbance was measured against blank. The drug content of the preparation was calculated using standard calibration curve. Amount of drug estimated by this method is given in Table I.

Table I: Determinations of Active Ingredients in Tablets

Sample	Label claimed (mg)	Amount found(mg) per tablet	% label claim *
Dexibuprofen	300	298.75±0.871	99.33±0.129

(Average of Three Determinations)*

Validation of method parameters

Precision:

Assay of method precision (intra-day precision) was evaluated by carrying out three independent assays of test samples of Dexibuprofen. The intermediate precision (inter-day precision) of the method was also evaluated using two different analysts, systems and different days in the same laboratory.

Linearity:-
The aliquots of concentration ranging 2-70 µg/ml were prepared in triplicate, but linearity was found to be between 2-60µg/ml concentrations. The calibration graphs were obtained by plotting the absorbance versus the concentration data and were treated by linear regression analysis.

Accuracy (recovery test) [11]:
The accuracy of the method is the closeness of the measured value to the true value for the sample. Accuracy of the method was studied by recovery experiments. The recovery experiments were performed by adding known amounts to tablet. The recovery was performed by preparing of concentration 30 µg/ml of Dexibuprofen standard solution. Three samples were prepared for each recovery level. The solutions were then analyzed, and the percentage recoveries were calculated from the calibration curve. The % recovery of the added pure drug was calculated as % recovery = [(*Ct–Cs*)/*Ca*] x 100, where *Ct* is the total drug concentration measured after standard addition; *Cs*, drug concentration in the formulation sample; *Ca*, drug concentration added to formulation. The results were as shown in Table II.

Table II - Results of Recovery

Sample	Amount added % µg/ml	Amount recorved	% Recovery ± SD
Brutek	24	23.92 ± 0.36	99.66 ± 0.21
Brutek	30	30.09 ± 0.21	100.3 ± 0.11
Brutek	36	36.15 ± 0.19	100.4 ± 0.24

Limit of detection (LOD) and limit of quantification (LOQ):
The LOD and LOQ of Dexibuprofen were determined by using standard deviation of the response and slope approach as defined in International Conference on Harmonization (ICH) guidelines. LOD and LOQ values were calculated using the relation,

$$LOD=3.3\delta /S$$
$$LOQ=10\ \delta /S$$

Where, δ= standard deviation of residuals from the curve; S=slope of the curve

Table III - Regression and validation parameters of Dexibuprofen

Sr. No.	**Parameter**	**Result**
Regression parameters		
1	Slope	0.046
2	Intercept	0.017
3	Standard Regression Equation	y = 0.046x +0.017
4	Correlation Coefficient (R^2)	0.999
5	Chi square	0.00857
6	Standard error of estimate	0.02950
7	Residual standard deviation	0.02812
Validation parameters		
1	Absorption maxima(nm)	221.8
2	Molar absorptivity	9495.68
3	A(1%, 1 cm)	460.33
3	LOD (µg/ml)	2.15
4	LOQ (µg/ml)	6.52
5	Linearity range (µg/ml)	2-60
6	Accuracy(% Recovery ±SD)	99.74±1.21

RESULTS AND DISCUSSION

The development of a simple, rapid, sensitive, and accurate analytical method for the routine quantitative determination of samples will reduce unnecessary tedious sample preparations, the cost of materials and labor. Dexibuprofen is a UV-absorbing molecule with specific chromospheres in the structure that absorb at a particular wavelength and this fact was successfully employed for their quantitative determinations using the UV spectrophotometric method. The absorption spectrum of Dexibuprofen in phosphate buffer pH 6.8 contain 5 % methanol was shown in Figure II. Calibration curve data was constructed in the range of the concentrations of 2-70µg/ml, but Beer's law obeyed in concentration range of 2-60 µg/ml. The regression equation was found to be y = 0.046x +0.017. The correlation coefficient (r^2) of the standard curve was found to be greater than 0.999. The stock solutions and working standards were made in phosphate buffer pH 6.8 contains 5% methanol. The λ_{max} of the drug for analysis was determined by taking scans of the drug sample solutions in the entire UV region.

Performing replicate analyses of the standard solutions was used to assess the accuracy, precision, and reproducibility of the proposed method. The selected concentration within the calibration range was prepared in phosphate buffer pH 6.8 contains 5 % methanol and analyzed with the relevant calibration curve to determine the intra and inter day variability. The proposed method can be successfully applied for assay in tablet dosage forms without any interference. The assay showed that the drug content of this product to be in accordance with the labeled claim (Table I). The recovery of the analyte of interest from a given matrix can be used as a measure of the accuracy of the method (Table II). The obtained results demonstrate the validity and accuracy of the proposed method for the determination of drug in tablet (Table III). In order to check the accuracy and precision of the developed method and to prove the absence of interference by excipients, recovery studies were carried out after the addition of known amounts of the pure drug to various pre-analyzed formulations of all drugs. It was found that the sample solution was stable up to 20 hrs in which no decomposition was observed. These results reveal that the developed method have an adequate precision and accuracy, and consequently, can be applied to the determination of Dexibuprofen tablet in pharmaceuticals without any interference from the excipients.

CONCLUSION

A spectrophotometric method for quantifying Dexibuprofen in formulation samples has been developed and validated. The assay is selective, precise, accurate and linear over the concentration range studied. LOD was approximately 2.15µg/ml in formulation and the LOQ was found to be 6.52µg/ml. The sample solution was stable for 20 hr. In summary, the proposed method can be used for the drug analysis in routine quality control.

Acknowledgement
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