

Simultaneous Estimation of Febuxostat and Ketorolac in Pharmaceutical Formulations by spectroscopic Method

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Abstract: A simple, Accurate and precise, selective spectroscopic method was developed and validated for the simultaneous determination of Febuxostat and Ketorolac from bulk and formulations. FBT and KTC were determined at 321nm and 215 nm respectively with the concentration ranges of 2–12µg/ml of FBT and 4-24µg/ml for KTC respectively. The correlation coefficient was found to be 0.9997 & 0.9993 for FBT & KTC drugs at selected wavelengths. The method was validated as per standard analytical procedures, and the results were found to be within limit. Percentage recoveries obtained for both the drugs were 99.65-100.16% and 98.78-99.90%, FBT and KTC respectively. The method was validated according to the ICH guidelines with respect to specificity, linearity, accuracy, precision and robustness. The method developed can be used for the routine analysis of Febuxostat and Ketorolac from their tablet dosage form.

Key words: Simultaneous Estimation, Febuxostat and Ketorolac, Pharmaceutical Formulations ,Spectroscopic Method.

INTRODUCTION

TORADOL (Ketorolac tromethamine) is a member of the pyrrolo-pyrrole group of nonsteroidal anti-inflammatory drugs (NSAIDs). The chemical name for ketorolac tromethamine is (±)-5-benzoyl-2,3-dihydro-1H-pyrrolizine-1-carboxylic acid, compound with 2-amino-2-(hydroxymethyl)-1,3-propanediol (1:1), and ULORIC (febuxostat) is a xanthine oxidase inhibitor. The active ingredient in ULORIC (febuxostat) is 2-[3-cyano-4-(2-methylpropoxy) phenyl]-4-methylthiazole-5-carboxylic acid, with a molecular weight of 316.38. The empirical formula is C₁₆H₁₆N₂O₃S. The chemical structures of FBT &KTC shown.Fig.1&2.

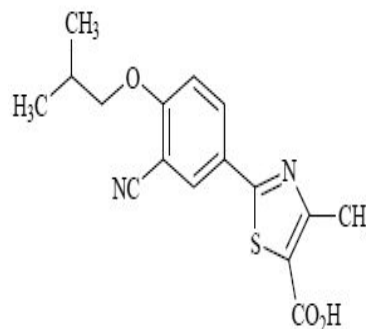


Fig.01 Chemical structure of Febuxostat.

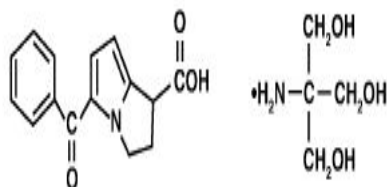


Fig.02. Chemical structure of Ketorolac

OBJECTIVE

To develop a simple, selective, precise and accurate Validated Spectroscopic Method for Simultaneous Estimation of Febuxostat and Ketorolac in bulk material and in pharmaceutical formulation. The method was validated according to ICH guidelines. The method was cost effective and can be applicable for the routine analysis of Febuxostat and Ketorolac in bulk and in tablet formulation.

EXPERIMENTAL METHODS

The validated method utilized a shimadzu 1800 with UV PROBE software and Methanol as solvent for both drugs estimation was detected

METHOD

The spectroscopic Method of Febuxostat and Ketorolac were determined at 321nm Fig.03 respectively with the concentration range of 2-12 μ g/ml for FBT and 4-24 μ g/ml for KTC respectively fig.04. For analysis of tablet formulation, the tablet powder equivalent to 25 mg was taken, dissolved in 25 ml volumetric flask and made up to 25ml with Methanol. The solution was sonicated for 15min, centrifuged at 100 rpm for 15 min and filtered through Whatmann filter paper No.41. From clear solution, further dilutions were made to get 10 μ g/ml of FBT and KTC theoretically.

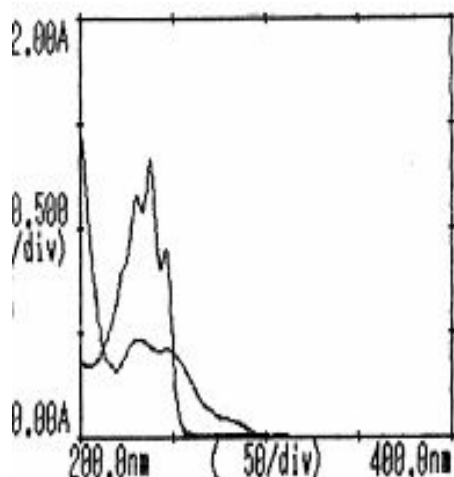


Fig .03.Overlain spectra of febuxostat and keterolac

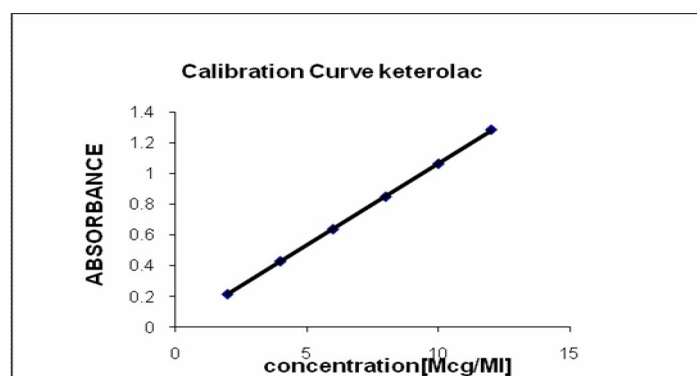
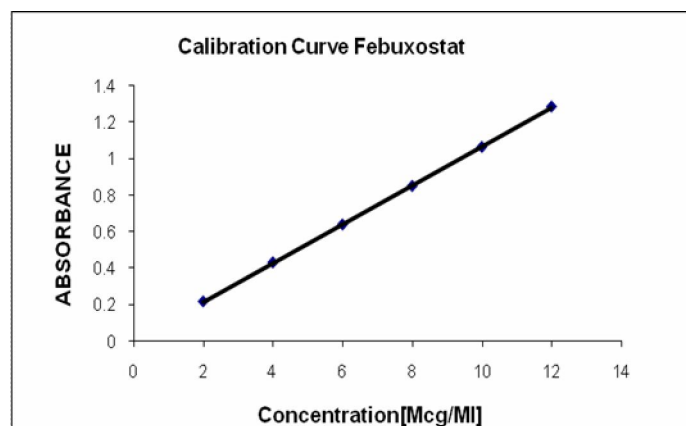


Fig.04. Calibration graphs of FBT and KTC

For recovery studies, to the reanalyzed formulation, solutions of raw material containing different concentrations were added and the amount of drug recovered was calculated. The procedure was repeated as per the analysis of formulation. The amount of drug recovered was calculated by using slope and intercept values from the calibration graph. Finally the method was validated as per ICH guide lines for precision, accuracy, specificity, linearity, reproducibility, limit of detection and limit of quantification.

Table02. Recovery Studies

Drug	Sample No.	Amount present ($\mu\text{g/ml}$)	Amount added ($\mu\text{g/ml}$)	% Recovery*	S.D	% R.S.D
FBT	1	6.0235	2.00	97.94	0.6433	0.6533
	2	6.0235	4.00	98.71		
	3	6.0235	6.00	99.22		
KTC	1	12.0431	2.5.	100.66	0.8438	0.8406
	2	12.0431	5.0	99.44		
	3	12.0431	7.5	101.06		

RESULTS AND DISCUSSION

A simple, selective, rapid and precise validated spectroscopic Method for Simultaneous Estimation of Febuxostat and Ketorolac in bulk material and in pharmaceutical formulation has been developed and validated. The correlation coefficient was found to be 0.9997&0.9998 for FBT&KTC respectively. In this method the % purity of Febuxostat and Ketorolac were found to be 101.25 ± 1.074 and 100.19 ± 1.031 respectively. The recovery studies range is 97.10 – 100.91 % and 97.22 – 103.04% for FBT and KTC, respectively. The Intraday and Inter day analysis carried out for precision. The ruggedness study was performed. In First order Derivative method the %

purity were found to be 100.25 ± 1.0054 and 101.49 ± 1.9305 for FBT and KTC, respectively. The recovery studies range is 97.94 – 99.22%, 99.16 – 100.16%. The Intraday and Inter day analysis carried out for precision. The ruggedness study was performed. The method was validated for statistical analysis.

CONCLUSION

The developed validated spectroscopic method was validated and the statistical validation was performed with the simplicity and ease of operation ensures that the validated method can successfully used for routine Analysis of Febuxostat and Ketorolac in bulk and tablet dosage formulation.

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