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NOTE

Spectrophotometric Estimation of Celecoxib from Capsule Formulation

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Two simple and sensitive spectrophotometric methods have been developed for the quantitative estimation of celecoxib from its capsule formulation. The first developed method is a UV spectrophotometric method using methanol as solvent; the drug showed absorption maxima at 253.2 nm in methanol and linearity was observed in the concentration range of 5–15 μg/mL. The second developed method is a visible spectrophotometric method, based on formation of red coloured complex of drug with 1,10-phenanthroline and ferric chloride, the complex showed absorbance maxima at 509.2 nm and linearity was observed in the concentration range of 50–400 μg/mL. Results of analysis for both the developed methods were validated statistically and by recovery studies.

Key Words: Celecoxib, Spectrophotometric method, Capsule formulation.

Celecoxib, chemically 4-[[5-(*p*-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl]] benzene sulfonamide is recently developed cyclooxygenase-2 specific inhibitor[1]. Few analytical methods for the estimation of celecoxib from biological fluid including HPLC[2–5], LC[6–7] and RPLC[8] are reported. In the present work, simple and sensitive spectrophotometric methods have been developed for the estimation of celecoxib from capsule formulation.

A Systronics UV-Visible double beam spectrophotometer (model 2101) with 1 cm matched quartz cells was used for spectral measurement. All the chemicals used were of analytical grade. Methanol was used for the preparation of 0.5% solution of 1,10-phenanthroline and stock solution of drug. Ferric chloride (0.5%) and orthophosphoric acid (0.5 M) solutions were freshly prepared in double distilled water. The capsule samples for analysis were procured from the local market.

For method I, in a series of 10 mL volumetric flasks, aliquots of standard drug solution (100 μg/mL) in methanol were transferred and diluted with same so as to give several dilutions in the concentration range of 5–15 μg/mL of celecoxib. Absorbance of this dilution was measured at 253.2 nm against blank (methanol) and a calibration curve was prepared by plotting concentration *vs.* absorbance.

For method II, in a series of 10 mL volumetric flasks, 0.5–4 mL aliquots of standard drug solution (1000 µg/mL) of celecoxib in methanol were transferred. To each flask 1 mL of ferric chloride and 1 mL of 1,10-phenanthroline was added. The flasks were then heated on a boiling water bath for 15 min, cooled to room temperature and 2 mL of orthophosphoric acid was added to each flask and the total volume was brought up to the mark with methanol so as to give several dilutions in the concentration range of 50–400 µg/mL of celecoxib. The absorbance of coloured complex was measured at 509.2 nm against a reagent blank and a calibration curve was preparaed by plotting concentration *vs.* absorbance.

For analysis of formulation contents of twenty capsules of celecoxib were accurately weighed and average weight of powder per capsule was determined. The contents were powdered and powder equivalent to 50 mg of celecoxib was accurately weighed and extracted four times with 20 mL portions of methanol. The combined extract was filtered through Whatmann filter paper No. 41 into 100 mL volumetric flask. The residue was washed with methanol and the washings were added to the filtrate, final volume of filtrate was made up to the mark with methanol.

For method I, 5 mL of filtrate was diluted to 50 mL with methanol, 1 mL of this was further diluted to 10 mL with methanol and the absorbance of this final dilution was measured at 253.2 nm against blank. The concentration of drug was computed from the respective calibration curve.

For method II, 2 mL of the filtrate was taken in 10 mL volumetric flask, this was treated as per the respective procedure of the calibrated curve for the visible spectrophotometry and amount of drug present in the sample was computed from the respective calibration curve.

Both the developed methods were repeated five times for two different strenghts of capsule formulations. Results of analysis are reported in Table-1.

Recovery studies were carried out for both the developed methods by addition of pure drug samples to pre-analyzed capsule sample solution at three different concentration levels. The results of recovery studies are reported in Table-1.

TABLE-1
ANALYSIS OF CAPSULE FORMULATION

Method	Formulation	Label claim (mg/capsule)	Label claim estimated* (%)	Recovery† (%)	Standard deviation
I	Capsule 1	100	98.93	101.30	0.600
	Capsule 2	200	99.70	100.40	0.588
II	Capsule 1	100	99.35	99.15	0.582
	Capsule 2	200	99.42	99.22	0.546

* Average of five determinations.

† Average of determination at three different concentration levels.

The proposed spectrophotometric methods for determination of celecoxib from capsule formulations are based on UV determination and formation of coloured complex of drug with 1,10-phenanthroline and ferric chloride. The heating of the reaction mixture was optimized at 15 min and the concentration of 1,10-phenanthroline and ferric chloride was optimized at 0.5%. The results of analysis for both the developed methods were close to 100% and standard deviation was satisfactorily low indicating accuracy and reproducibility of the methods. Results of recovery studies were satisfactory which shows that there is no interference of excipients. The developed methods were found to be simple, rapid, sensitive and economical. These developed methods may be used for routine analysis of drug from capsule formulation.

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