Simultaneous Spectrophotometric Estimation of Valdecoxib and Paracetamol in Tablet Formulations

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A simple, accurate, economical, and reproducible method for simultaneous estimation of valdecoxib and paracetamol in two-component tablet formulation has been developed. The method of analysis is derivative spectroscopy to eliminate spectral interference by measuring absorbances at two wavelengths 284 nm and 301 nm for valdecoxib and paracetamol, respectively. The results obtained in triplicate were validated statistically and by recovery studies.

Valdecoxib¹ (VAL), 4-(5-methyl-3-phenyl-4-isooxazoyl) benzene sulphonamide) has analgesic and anti-inflammatory activity. Extensive literature survey revealed its estimation by high performance liquid chromatography method²-³ only. Paracetamol⁴ (PCM), 4-hydroxy acetanilide, has analgesic and antipyretic activity. Reported methods of analysis involve spectrophotometric methods⁵-⁷, chromatographic methods⁸-¹⁰, titrimetric¹¹ and electrochemical methods¹². The analysis of this
combination is of prime importance since valdecoxib is present in a small quantity relative to paracetamol. Therefore, development of simultaneous spectrophotometric method for estimation of both drugs in tablet formulation is a worthwhile objective. The drug combination of PCM and VAL is indicated for symptomatic relief of rheumatoid arthritis and in the treatment of dysmenorrhoea. The present work is a simple, rapid, and economical method for the simultaneous estimation of VAL and PCM in two-component tablet formulation.

A Shimadzu UV spectrophotometer 160A model with spectral bandwidth of 3 nm and wavelength accuracy of ±0.5 nm was used. Ten mm matched quartz cells were employed for this work. Methanol (AR grade) was used as solvent. VAL and PCM were obtained as gift samples from Torrent Research Center, Gandhinagar; and Ranbaxy Laboratories, Dewas, respectively. The commercial formulations of VAL and PCM are available in ratio of 1:25 {Valeron Plus (20/500 mg) [Formulation A]} and 1:32.5 {Validay Plus (10/325 mg) [Formulation B]}, respectively as tablets.

Standard stock solutions of VAL and PCM of 100 µg/ml each were prepared in methanol. Each was suitably diluted to different concentrations and linearity was studied. Linear relationships were observed in the range of 0-25 µg/ml for both VAL and PCM. Standard solutions of 10 mg/ml from pure samples of VAL and PCM were prepared separately. These solutions were scanned over the range of 400-200 nm. The overlain zero-order spectra of VAL and PCM are shown in fig. 1. Spectra indicate the absorption maxima of VAL and PCM at 239 nm and 248 nm, respectively.

Tablet powder equivalent to 25/32.5 mg of PCM was weighed in case of Formulation A/B, respectively and was dissolved in about 25 ml of methanol, stirred for 30 min, and final volume was made up to 50 ml with methanol. The solution was filtered through Whatman filter paper No. 41, and the first few ml were rejected. 19.2/25.2 ml of standard solution of VAL (25 µg/ml) was added to 1 ml of filtered solution in case of Formulation A/B, respectively. To make the solution of equal concentrations for both the drugs, final volume was made up to the mark (50 ml) with methanol.

The zero-order absorption spectra were derivatized, and derivative spectra from first to fourth order were recorded. Considering all these derivative spectra of VAL and PCM, the second-order derivative spectrum for VAL and first-order derivative spectrum for PCM were selected (fig. 2 and 3, respectively).

Five mixed standards of 2.5, 5.0, 7.5, 10.0, and 12.5 µg/ml concentrations of each drug were prepared from stock solutions of the drugs. All the mixed standards were scanned over the range of 400-200 nm and derivatized to measure substantial absorbance at 284 nm for VAL and

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**Fig. 1:** Overlaid spectra of valdecoxib (val) and paracetamol (pcm) in methanol.

**Fig. 2:** Overlaid first-order derivative spectrum of valdecoxib (val) and paracetamol (pcm) in methanol.

**Fig. 3:** Overlaid second-order derivative spectrum of valdecoxib (val) and paracetamol (pcm) in methanol.
Valdecoxib and paracetamol obeyed Beer’s law in the concentration range of 0-25 µg/ml. In formulation A, the percentage composition of valdecoxib was found to be 100.2% (Standard deviation ±0.2115) and that of paracetamol was found to be 98.8% (Standard deviation ±0.1281). In formulation B, the percentage composition of valdecoxib was found to be 101.0% (Standard deviation ±0.1221) and that of paracetamol was found to be 99.0% (Standard deviation ±0.0739). Simultaneous estimation of valdecoxib and paracetamol by derivative spectrophotometry was found to be simple and accurate. The value of standard deviation and coefficient of variance was significantly low and recovery was close to 100%, indicating the reproducibility of the method.

The method is used to eliminate the spectral interference from one of the two drugs while estimating the other drug by selecting the zero-order crossing point on the derivative spectra of each drug at the selected wavelengths.

It is concluded that the proposed method is simple, accurate, and can be successfully employed for simultaneous estimation of valdecoxib and paracetamol in two-component tablet formulations.

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