Asian Journal of Chemistry Vol. 20, No. 4 (2008), 3295-3297

NOTE

Derivative Spectrophotometric Determination of Moclobemide in Pharmaceutical Formulations

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A first derivative UV spectroscopic method was developed for determination of moclobemide in the tablet dosage form. The first derivative spectrum recorded between 200 and 300 nm and a zero-crossing technique for first derivative measurement at 225 nm was selected. The linear concentration ranges were 2-16 μg/mL, r = 0.9994, n = 5). Between days CV % = 2.8, within day CV % = 2.1, analytical recovery close to 98.37-101.08 % shows the suitability of the method for determination in quality control.

Key Words: Moclobemide, First-derivative spectrophoto-metry.

Moclobemide is a short-acting, reversible inhibitor of monoamine oxidase[1,2]. It is a benzamide derivative which inhibits the deamination of serotonin, norepinephrine and dopamine. This action leads to increased concentrations of these neurotransmitters, which may account for the antidepressant activity of moclobemide. This drug was developed as tablet dosage form for the symptomatic relief of depressive illness. There is not any official method for analysis of moclobemide. Various analytical techniques have been described for analysis of moclobemide by using HPLC with different directors like photodiode array[3], electrospray-ionization mass spectroscopy[4], UV detector[5], gas chromatography[6], moclobemide selective membrane electrode[7], spectrophotometric determination by charge transfer complexation with chloranilic acid[8] and HPTLC method[9].

All reagents used were of analytical reagent grade. Pharmaceutical grade moclobemide was obtained from Intas Pharmaceuticals Ltd. Vatva, Gujarat, India. The dosage form of moclobemide was procured from the local pharmacy. Methanol (analytical grade, Merck).

Spectrophotometric analyses were performed on a Shimadzu, 1601 double beam UV-Visible spectrophotometer with a pair of 10 mm matched quartz cells was used to measure absorbance of the resulting solutions. Measurements were carried out using the first derivative of the absorbance spectra, measuring the amplitude at 225 nm.

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Preparation of moclobemide standard solutions and calibration: A stock solution containing 100 µg/mL of moclobemide was prepared by dissolving 10 mg of moclobemide in methanol and diluted to the mark with same solvent. All measurements were made at room temperature. The standard solutions were prepared by the proper dilutions of the stock standard solution with methanol 2-16 µg/mL.

Estimation of moclobemide from tablets: 20 Tablets were accurately weighed and powdered and the quantity of powder equivalent to 100 mg of moclobemide was transferred to a 100 mL volumetric flask and mixed with methanol (50 mL) and sonicated for 20 min. The solution was filtered through Whatmann filter paper no. 41 and the residue was washed thoroughly with methanol. The filterate and washings were combined in a 100 mL volumetric flask and diluted to the mark with methanol. 1 mL of this solution was transferred to 10 mL volumetric flask and diluted up to mark with methanol. The resulting solution (1.0 mL) was analyzed by UV. The analysis was replicated in triplicate.

Precision assays: Moclobemide standard solutions were prepared and analyzed eight times within the same day to obtain the repeatability and eight times over different days to obtain the reproducibility. Each assay was carried out on a different sample of moclobemide. The percentage relative standard deviation (RSD %) of the data obtained was calculated.

Accuracy: Accuracy was determined in terms of percentage recovery. The proposed method was applied to determine moclobemide in tablet dosage form. The recovery experiments were carried out in triplicate by spiking previously analyzed samples (4 µg/mL) of the tablets with four different concentration of standard and the amount of moclobemide was calculated at each level.

Linearity of the method: Intraday and interday precision was determined by analyzing moclobemide (2-16 µg/mL) for 3 times in the same day and daily for three days, respectively and % CV was calculated. LOD determined by measuring the D1 absorbance's at 225 nm of at least 25 separate base placebo tablet samples.

The zero order and first derivative spectra for all investigated ingredients of the moclobemide placebo tablets were recorded in the wavelength range from 200-300 nm. The zero and D1 spectra of moclobemide in the wavelength range of 200-300 nm are shown in Figs. 1 and 2. Zeroth order spectrum of moclobemide is shown in Fig. 1, it can be seen that maximum absorbance was observed at 236 nm wavelength. First derivative spectrum is shown in Fig. 2, it can be seen that maximum absorbance was observed at 225 nm. The D1 spectra (Fig. 2) have a through at 225 nm with a good sensitivity and linearity, because of this, the first order derivative spectra were selected for quantitative analysis.

The excipients cornstarch, ethylcellulose, lactose, magnesium stearate, ethylhydroxy-propyl cellulose, povidone, red iron oxide, sodium starch glycolate, talc, titanium dioxide and yellow iron oxide. The data shown in Table-1 indicate good accuracy and precision of the proposed procedure.

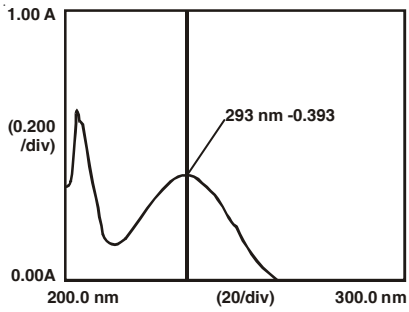

Fig. 1. Zero order spectra of moclobemide

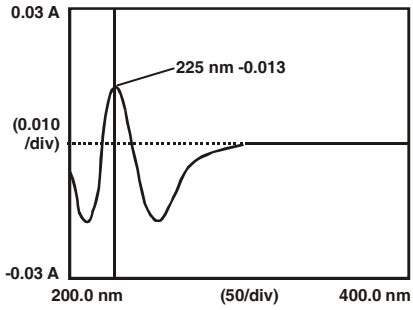

Fig. 2 First derivative spectra of moclobemide

The detection limit (LOD) and quantification limit (LOQ) were 0.684 and 1.5 μg/mL, respectively. Furthermore, the proposed method does not require the elaboration of treatment and procedures, which are usually associated with chromatographic methods.

TABLE-1
FIRST-DERIVATIVE SPECTROPHOTOMETRIC DETERMINATION OF MOCLOBEMIDE TABLET

Concentration range (μg/mL)	2-16
Y = aX + b	Y = 0.0018X - 0.0005
Correlation coefficient	0.9994
Relative standard deviation (% CV)	0.0-4.38
Within day CV %	2.1
Between day CV %	2.8
Limit of detection (μg/mL)	0.684
Limit of quantitation (μg/mL)	1.5
Losartan tablet (taken) (mg)	150
Losartan tablet (found) (%)	100.925 ± 3.207
Recovery (%)	98.379-101.080 ± 2.672-5.345

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(*Received*: 13 February 2007; *Accepted*: 21 January 2008) AJC-6248