Determination of Nifuroxazide in Capsules by Differential Pulse Polarography

A. RADI^{*†}, S. EL-LABAN^{*} and I. M. M. KENAWY^{**}

^{*}Chemistry Department, Faculty of Science, Mansoura University, 34517 Demiatta, Egypt

^{**}Chemistry Department, Faculty of Science, Mansoura University, Mansoura, Egypt

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Nifuroxazide, 4-hydroxybenzoic acid [(5-nitro-2-(furanyl)methylene]hydrazide (I) is used for the treatment of acute and chronic diarrhea, gastroenteritis, and colitis. Analytical methods used for the determination of nifuroxazide include titrimetric¹, complexometric², ultraviolet spectrophotometric³, and HPLC.³ At present, only a few studies dealing with nifuroxazide polarographic behavior have been reported and the authors seems to have encountered only two waves.⁴⁻⁶ In this work, the polarographic behavior of nifuroxazide and its analytical utility have been investigated.

(I)

Experimental

Reagents

Stock solutions (1×10^{-3} M) of nifuroxazide were prepared daily in DMSO and stored at 4°C in the dark. Britton-Robinson buffers were used as supporting electrolytes. All reagents were of AnalaR-reagent grade in doubly distilled water.

Apparatus

The differential pulse polarograms have been recorded using a polarographic analyzer (PAR Model 264A) coupled with a PAR 303A static mercury drop electrode. All the potentials are referred to Ag/AgCl/3 M KCl reference electrode and a platinum wire is used as counter electrode.

A SCHOTT GerÄte pH-meter with glass combination electrode was used for pH determination.

Procedure

The supporting electrolyte solution (9.5 ml) with an aliquot of nifuroxazide in DNSO, diluted to 10 ml with DMSO, was added to the cell. The cell was degassed by purging with purified nitrogen for 8 min. The voltammetric curves were recorded in the cathodic direction.

Capsule analysis

To eliminate the inconstancy of weight, the contents of 20 Antinal capsules of several packages were carefully weighed and then mixed. An average weight of one capsule was dissolved in ethanol, filtered and washed with ethanol. The residue was evaporated till dryness and dissolved in 10 ml DMSO and 0.5 ml of the solution was then transferred to the polarographic cell containing 0.5 ml DMSO and 9 ml Briton-Robinson buffer (pH 4.0). The concentration was calculated using a calibration graph or regression equation.

Fig. 1 Typical differential pulse polarogram of 1×10^{-6} M nifuroxazide in Britton-Robinson pH 4.0. Scan rate: 5 mV s⁻¹; pulse amplitude 25 mV.

[†] To whom correspondence should be addressed.

Fig. 2 $E_{1/2}$ -pH plots for the polarographic waves A, B, C and D. Other conditions as in Fig. 1.

Results and Discussion

Figure 1 shows a typical differential pulse polarogram of nifuroxazide in Britton-Robinson buffer of pH 4.0. In acid pH solutions nifuroxazide exhibited four reduction peaks. Since the nitro group is more easily reducible than the hydrazono group^{7,8}, these peaks can be attributed to the subsequent reduction of nitro group and the reductive fission of hydrazono group.

As illustrated in Fig. 2, nifuroxazide shows two or four voltammetric peaks according to the pH of solution. The peak potential (E_p) values shift linearly more negative with increasing the pH. At pH between 5.0 and 7.0, peak C grows in height at the expense of peak B and the two merge into a single peak at pH 7. Above pH 7.0, only two peaks (A and D) were observed for the reduction of nitro group.

Analytical application

The dependence of the first peak heights on the concentration of nifuroxazide was investigated by differen-

tial pulse polarography in Britton-Robinson (pH 4.0). There is a good linear relationship between the analytical signal i_p and c of nifuroxazide from the limit of detection (1×10^{-7}) to 7.5×10^{-5} M. For analytical determination the calibration curve method was used. This curve is described by the following regression equation:

$$i_p(\mu A) = 6.0805c \text{ (mM)} - 0.00866, \quad r = 0.99587$$

The reproducibility of the method was calculated from ten independent runs of 1×10^{-5} M nifuroxazide solution, obtaining a relative standard deviation of 1.5%. A recovery assay based on five separate determination performed on capsules containing 200 mg of nifuroxazide showed a mean recovery of nifuroxazide of 102% with a relative standard deviation of 1.8%.

In conclusion, the proposed polarographic method proved to have adequate precision and accuracy to enable reliable analysis of nifuroxazide and is therefore recommended as a useful tool for analysis of nifuroxazide in dosage form. It compared well with, complexometric², ultraviolet spectrophotometric³, and HPLC³ procedures or with the titrimetric¹ procedure of the official X-th French pharmacopoeia.

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