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Determination of Cephalosporines in Presence of Tungsten (VI) with the help of Polarographic Catalytic Waves

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A rapid and sensitive method for the polarographic determination of cephalosporins is described. In presence of tungsten (VI), Cefachlor exhibited a catalytic peak at -2.3 V vs SCE in 0.9 M H_2SO_4 medium. Optimum conditions are established for the method developed. This analytical method is useful for determining the drug as well as the metal. The method is applied to clinical samples containing these compounds.

SOME sulphur containing organic compounds produce characteristic polarographic catalytic waves when complexed with transition metal ions such as CO(II), Ni(II), Cr(VI) or W(VI)[1-9]. It has been observd that cephalosporins produce a catalytic hydrogen wave in 0.9 M H_2So_4 medium in presence of W(VI). Detailed investigations on the polarographic behaviour of three cephalosporin cefachlor, cephalexine and cefuroxime is presented in this communication.

EXPERIMENTAL

Polarograms are recorded with a dc recording Polarograph (M/s. ELICO Private Limited, Hyderabad, India) using 2 electrode cell. pH measurements are made on an ELICO pH meter. All reagents used in this investigation are of AnalaR grade. Millimolar solution of tungsten (VI) is prepared and used in these studies. 0.01 M Sodium tungstate solution is prepared by dissolving 359 mg of Na_2WO_4. $2H_2O$ in 100 ml distilled water; 6.5 g of KCl is dissolved in 100 ml water to get 0.81 M KCl solution. About 100 mg of cefachlor / cephalexine / cefuroxime powder was accurately weighed into a 100 ml volumetric flask containing 50 ml of 5 M HCl. The flask was heated on a water bath for half an hour, cooled and made up to the volume with distilled water. Ten ml of this solution was neutralised with 10 ml of 5 M NaOH and was made up to 100 ml with distilled water. Each ml of this solution contained 100 ug of cefachlor / cephalexine / cefuroxime. 0.5 ml of this solution was taken into a 10 ml volumetric flask and 3 ml of dilute H_2SO_4 was added so that the total concentration of the acid was 0.9 M in the solution. Two ml KCl (0.81 M) solution was added as supporting electrolyte and 1 ml of tungsten (VI) solution of required concentration was added. The solution was made up to the mark with distilled water and taken into the polarographic cell. The solution was deaerated with nitrogen gas for 10 min prior to recording.

RESULTS AND DISCUSSION

Typical polarograms of W(VI) (a), hydrolysed drug (b), (HC) and a mixture containing both (c) were recorded in 0.9 M H_2SO_4 medium (Fig. 1). An analysis of the figure revealed that either the drug or the metal did not produce any peak bahaviour. The mixture containing both produced a catalytic hydrogen wave at -2.3 V vs SCE.

Studies relating to the effect of different acids revealed that H_2SO_4 of 0.9 M concentration was most suitable and appropriate for these studies. A sharp and well defined peak could be noticed if the solution contained 0.81 M KCl.

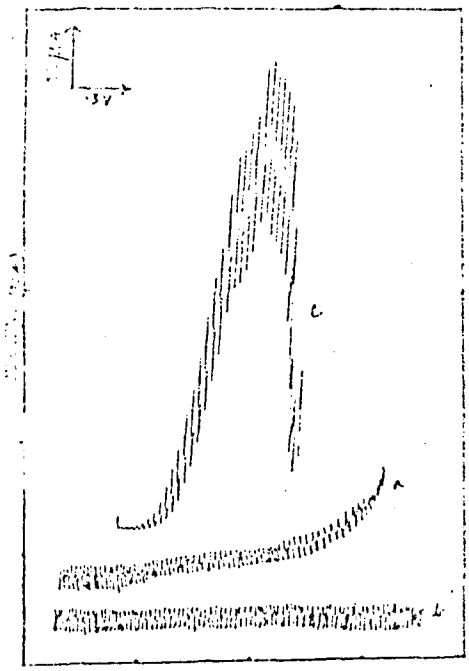
Experiments relating to the effect of surfactants, height of the mercury column suggested that the catalytic wave is controlled by a chemical reaction rather than diffusion. The Peak bahaviour is reproducible for at least two hours as indicated by the effect of time. Further presence of 50% non-aqueous solvents decreased the catalytic wave to an

Table 1

Drug	Concentration range of determination for Hydrolysed drug	W(VI)	E_p (V vs SCE)
Cefachlor	1.5-10.3 x 10^{-6} M	1.5-10.0 x 10^{-6} M	-2.3
Cephalexine	6.0-48.0 x 10^{-7} M	6.0-48.0 x 10^{-7} M	-1.6
Cefuroxine	7.1-71.0 x 10^{-7} M	5.0-50.0 x 10^{-8} M	-2.2

Table 2 : Assay of cephalosporins in pharmaceutical formulations (Average of five determinations)

Sample	Labelled amount mg/Tab or Cap	Amount found mg/Tab or Cap	% Recovery deviation	Standard
Cefachlor	250	249.5	99.8	0.007
Cephalexine	250	249.0	99.6	0.03
Cefuroxime	100	99	99.0	0.002



POTENTIAL (-V vs SCE)

Fig. 1 Typical Polarograms of

a) W (VI)
b) HC
c) W(VI) + HC
W (VI) = 1.0 x 10^{-6} M ; HC = 0.996 x 10^{-6} M
H_2SO_4 = 0.9 M; Starting Voltage = -1.5 V vs SCE

appreciable extent. This observation supports the authors' contention that the catalytic wave is controlled by a chemical reaction. Spectrophotometric studies carried out under the same conditions as in polarographic studies revealed a 1:1 complex formation between W(VI) and the drug.

For obtaining linear relationship between the metal ion/drug concentration and the peak current it was necessary to maintain 1:1 ratio of metal to drug. Drugs containing the three cephalosporines were analysed and the relevant data are presented in Tables 1 and 2.

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