Short Communication

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DETERMINATION OF TWO SPIRO NON-STEROIDAL OESTROGEN POTENTIATING AGENTS USING HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY WITH ELECTROCHEMICAL DETECTION

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These spiro non–steroidal agents which are not oestrogen in their activity potentiate oestrogen effects [1]. It has been assumed that the potentiation may be associated by having two keto groups in the adjacent spiro rings.

The present study deals with the quantitative determination of the compounds and to establish the reduction behaviour at the glassy carbon electrode. This study also explores the utility of liquid chromatography combined with electrochemical detector as a method for simultaneous analysis to demonstrate capability of quantitating as little as 0.1 μg/1.

The reduction potentials for the compounds were determined by use of current–voltage curves which showed markedly two one–electron reduction steps at glassy carbon electrode. The half–peak potentials were –0.73 V and –0.93 V for compound A whereas –0.65 V and –0.85 V for compound B (*vs.* $Ag^+/AgCl$ ref. electrode).

The reduction occurs by the following mechanism.

$$A + 2\,H^+ + 2\,e^- \rightleftharpoons AH_2$$

$$E_{1/2} = E_o - 0.03 \log \frac{(AH_2)}{(A)\,(H)^2}$$

The change of 0.03 V /pH in the reduction potential showed the pH dependence of these compounds.

Before selecting the proper packing material for column, several resins and mesh sizes were examined and it was found that Nucleosil SA 10μ gives more selective and reproducible performance than the others.

The variation of selectivity with working potential of the electro-chemical detector was also studied with various mobile phases. The present study is done by using 50:50 acetonitrile :0.1 M phosphate buffer (pH 8.20) at

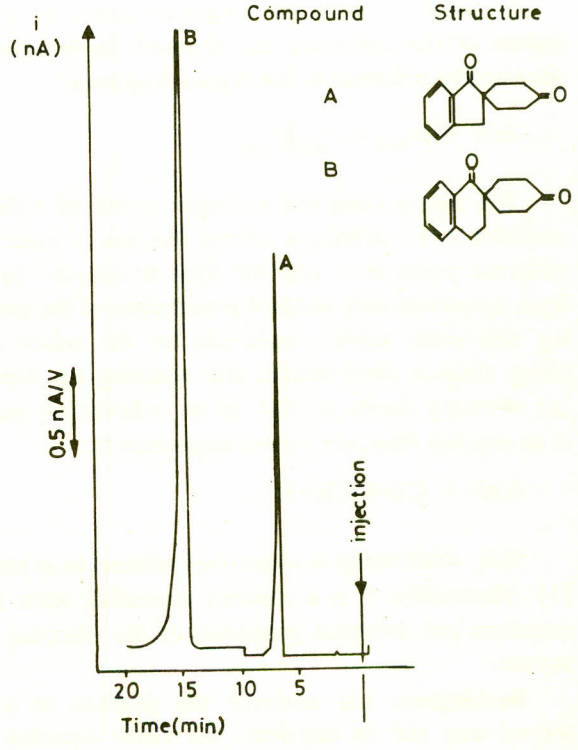

Fig 1.

–1.0 V. The increase of applied potential causes certain increases in peak–heights, but at the same time increases the back ground current. Fig. 1 illustrates the detector response to concentration of 1 μg/1 of both compounds. Linear standard curves with correlation coefficient of 0.999 were obtained from 1–5 μg/1 for both compounds.

REFERENCE

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