Optimization of separation of a complex mixture of natural and synthetic corticoids by micellar liquid chromatography using sodium dodecyl sulphate. Application to urine samples

A. Santos Montes; R. Izquierdo Hornillos

Abstract-

A systematic optimization of the separation of a mixture of corticoids by micellar liquid chromatography, using sodium dodecyl sulphate as surfactant, a Hypersil (250 mm x 3.2 mm I.D.) C18 column, a flow-rate of 0.5 ml min(-1), and UV absorbance detection at 245 nm has been carried out. Several mobile phases consisting of sodium dodecyl sulphate and different organic modifiers were tested of which tetrahydrofuran, PrOH and BuOH were finally selected. On the basis of analysis time, resolution and number of compounds separated, a mobile phase containing 36 mM sodium dodecyl sulphate and 1.91\% butanol allowed the separation of thirteen corticoids out of sixteen in about 27 min. Under these conditions the optimal concentration of sodium dodecyl sulphate was found to be 36 mM. A bivariant optimization method for the mobile phase BuOH-sodium dodecyl sulphate corrobored these results. The effects of temperature, ionic strength and flow-rate effect have also been studied. The most important analytical figures of merit were assessed and compared with those obtained using conventional mobile phases. The optimized method was applied to human urine samples of subjects administered with Dezacor (tablets containing 30 mg of the active ingredient deflazacort) with and without sample preparation.

Index Terms- Corticoids

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