

Analysis of anabolic steroids by partial filling micellar electrokinetic capillary chromatography and electrospray mass spectrometry

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“...17alpha-Hydroxy-17beta-methylandrosta-1,4-dien-3-one 300.44 16.00 The pK_a values of the functionalities were calculated with Pallas program. (Compudrug) Androstenedione (Fig. 1a) is the major immediate precursor of testosterone in the human...”

Abstract

A partial filling micellar electrokinetic capillary chromatography (PF-MEKC) separation of six anabolic androgenic steroids (androstenedione, metandienone, fluoxymesterone, methyltestosterone, 17-epimetandienone and testosterone) is introduced. The method utilises a mixed micellar solution consisting of sodium dodecyl sulphate (SDS) and sodium taurocholate. The analytes are detected with a photodiode array detector at 247 nm wavelength. Methyltestosterone is used as internal standard. The detection limits were 39 g/L for androstenedione, 40 g/L for testosterone, 45 g/L for fluoxymesterone, 45–90 g/L for 17-epimetandienone, 59 g/L for methyltestosterone and 90 g/L for metandienone. Linear correlation between concentration (0.1–5.0 mg/L) and detector response was obtained with r^2 of 0.994 for fluoxymesterone, 0.998 for 17-epimetandienone and 0.999 for androstenedione, metandienone and testosterone. In addition, ionisation of the investigated compounds in electrospray mass spectrometry (ESI-MS) was studied in positive ion mode. The most intense signal (100%) was the protonated molecular ion $[M+H]^+$, except for 17-epimetandienone, which gave its strongest signal at m/z corresponding to $[M-H_2O+H]^+$. Finally, separation and identification of fluoxymesterone, androstenedione and testosterone by PF-MEKC–ESI-MS is described. This is the first use of PF-MEKC and PF-MEKC–ESI-MS assays for anabolic androgenic steroids.

Author Keywords: Doping analysis; Partial filling micellar electrokinetic chromatography; Steroids; Hormones

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