Abstract

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Title of Diploma Thesis: Determination of illegal compounds in sport supplements by modern chromatographic methods

This diploma thesis concerns on the development of method for the determination of steroid hormones in sport supplements using ultra-high performance supercritical fluid chromatography. The method development and optimization of chromatographic conditions include selection of a suitable stationary phase and a modifier together with a mobile phase additive. Furthermore, the influence of temperature, pressure, flow rates and mobile phase gradient on separation efficiency was examined. In conclusion, this method has been validated and applied on real samples.

Experiments were carried out on the device ACQUITY UPC^2 with PDA detector. Detection was at wavelength 232 nm. 11 compounds were analysed. The stationary phases screening included 5 different analytical columns ACQUITY UPC^2 BEH 1,7 μm, BEH 2-EP 1,7 μm, HSS C18 1,7 μm, CSH Fluoro-Phenyl 1,7 μm and 2-PIC 1,7 μm dimensions 3,0 x 100 mm. As the most suitable was selected column HSS C18 1,7 μm (3,0 x 100 mm). Mobile phase consisted of pure CO_2 with an organic modifier methanol and aditives ammonium acetate (10 mM) and water (2 %). Flow rate of mobile phase was set to 2 ml/min. The mobile phase gradient was from 3 % to 17 % of the modifier during 6 minutes. The temperature on a column thermostat was maintained at 20 °C. The back pressure was set at 2000 psi.

Key words: supercritical fluid chromatogramy, CO_2, anabolic androgenic steroids, doping, dietary supplement , BEH, BEH-2EP, HSS C18, 2-PIC, CSH Fluoro-Phenyl