

Environmental

Analysis of Synthetic Steroids by UHPLC/SQ MS



Synthetic steroids and naturally occurring hormones are potent endocrine disruptors that may cause an adverse effect on aquatic species once they enter the environment through water systems. We present a UHPLC/SQ MS method for separating and detecting these compounds.

Experimental Conditions

Target Analytes: Norethindrone, d_6 -norethindrone, tiblone, drospirenone, norgestrel, medroxyprogesterone, dehydrogesterone, cyproterone-acetate, progesterone, d_9 -progesterone and medroxyprogesterone-acetate

Liquid Chromatography Conditions

Pump Type:	PerkinElmer® Flexar™ FX-15		
Column:	PerkinElmer Brownlee™ HRes C18 column (2.1 mm x 50 mm, 1.9 μ m)		
Mobile Phase:	A: water with 0.1% acetic acid B: acetonitrile with 0.1% acetic acid		
Flow Rate:	0.5 mL/min		
Injection Volume:	2 μ L in partial fill mode		
Gradient:	Time (min)	%A	%B
	0	60	40
	2.9	54	46
	2	54	46
	1	40	60

Mass Spectrometer Conditions

Ionization: Ultraspray™ ESI – Negative mode

The $[M-H]^-$ ions of each of the steroids were measured in three different time periods:

Time Period 1: (0.0-2.0 min) SIM ions 299.2, 305.4, 313.2 for norethindrone, d_6 -norethindrone and tiblone respectively; dwell time of 200 ms each

Time Period 2: (2.0-3.5 min) SIM ions 313.2 for norgestrel and dehydrogesterone, 345.26 and 367.2 for medroxyprogesterone and drospirenone respectively; dwell time of 200 ms each

Time Period 3: (3.5-5.9 min) SIM ions 315.21, 324.3, 387.28, 417.22 for progesterone, d_9 -progesterone, medroxyprogesterone-acetate and cyproterone-acetate respectively; dwell time of 150 ms each

Capillary Exit Voltage: 100 V

Results

To obtain optimal sensitivity the $[M-H]^-$ ions were monitored in SIM mode in three different time periods (Figure 1). The detection limits were in the range of 2.5 ng/mL for majority of the analytes ($S/N > 3$).

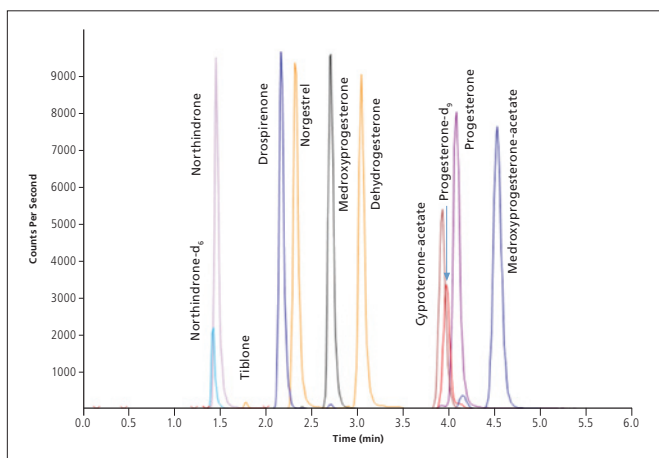


Figure 1. Overlaid chromatograms of $[M-H]^-$ ions for each of the steroids in SIM mode measured in three different time periods.