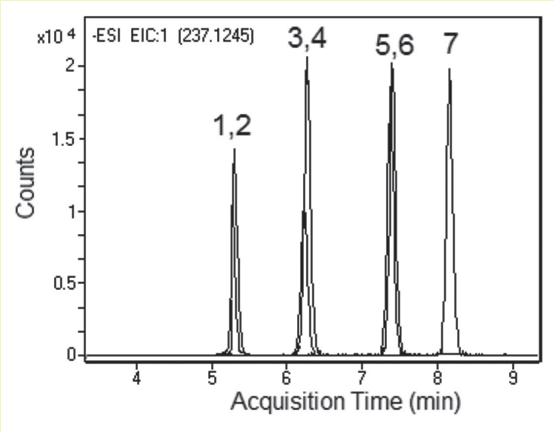
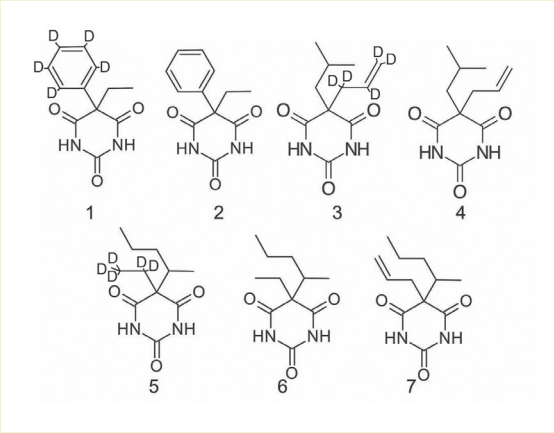
Barbiturates in Urine

Easy LC-MS method





Method Conditions

Column: **Cogent Bidentate C18™**, 4µm, 100Å

Catalog No.: 40018-05P-2

Dimensions: 2.1 x 50 mm

Solvents: A: DI H_2O / 10 mM ammonium formate
B: 95% acetonitrile / 5% DI H_2O / 10 mM ammonium formate (v/v)

Gradient:

time (min.)	%B
0	10
1	10
10	45
10.1	90
12	90
12.1	10

Post Time: 3 min

Injection vol.: 1 microL

Flow rate: 0.4mL/min

Detection: ESI – NEG - Agilent 6210 MSD TOF mass spectrometer

Samples: Stock solutions of barbiturates were prepared at a concentration of 1 mg/mL in methanol. Then 2 mL of a urine sample was spiked with the stock solutions diluted, (dilution 1:100) and filtered through a 0.45µm nylon syringe filter (MicroSolv Tech Corp.) into autosampler vials.

Peaks: 1. Phenobarbital-D5 (m/z = 236.1089)
2. Phenobarbital (m/z = 231.0775)
3. Butalbital-D5 (m/z = 228.1402)
4. Butalbital (m/z = 223.1088)
5. Pentobarbital-D5 (m/z = 230.1558)
6. Pentobarbital (m/z = 225.1245)
7. Secobarbital (m/z = 237.1245)

t_0: 0.3 min

Discussion

After minimal sample preparation (dilute-and-shot approach), spiked urine samples were analyzed using Cogent Bidentate C-18 column. The analysis was based on a separation of standards[1]. The obtained peaks were symmetrical (As<1.05) and efficient (> 10^6 pl/m). No shift in retention times was observed after the samples were diluted ten-fold (data not shown). Matrix effects that would diminish the signal intensity were less than 5%. This method shows a possible methodology for analysis of these compounds in forensic samples.

1. J.J.Pesek, M.T. Matyska, A.M. Kim, J. Sep. Sci. 2013, 36, 2760–2766.

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