生物與毒性分析

以極致液相層析/串聯質譜術偵測人體檢體中15種內分泌干擾物

質

Determination of 15 Endocrine-Disrupting Chemicals in Biospecimens with Ultra-performance Liquid Chromatography/Tandem Mass Spectrometry

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Concentrations of contaminants in biospecimens could represent internal doses despite exposure routes. This study determined 15 endocrine-disrupting chemicals in human serum and human urine, including 10 perfluoroalkyl substances (PFASs), and 5 phthalate metabolites. After spiking with 20-µL (500 ng/mL) isotope labeled standards in methanol, 100 µL of serum samples were transferred to a Waters Ostro plate, then mixed with 300 µL of 1% formic acid/acetonitrile (ACN); the filtrate was concentrated to barely dry with a SpeedVac concentrator, then was reconstituted with 100 µL of methanol for instrumental analysis. 100 µL of urine samples were spiked with 20-µL (500 ng/mL) isotope labeled standards in methanol and were diluted with 100-µL 10-mM ammonium acetate(aq). After mixing with ACN, urine samples were loaded to Waters Sirocco plate; the filtrate was concentrated to nearly dry with a SpeedVac concentrator, and then was reconstituted with 100 μ L of methanol for instrumental analysis. Extracted samples were analyzed on a Waters UPLC I-Class PLUS coupled with a Waters Xevo TQ-XS triple-quadrupole mass spectrometer at negative Unispray ionization. Analytes were separated on a Waters CORTECS C18 column (30×2.1 mm, 1.6μ m). The mobile phase consisted of (A) 0.1% acetic acid_(aq) and (B) ACN. The gradient started at 3% (B) for 0.5 min, then increased to 95% in 4 min. After holding for 0.5 min, the gradient turned back to the initial condition in 0.2 min, then was held for 2.3 min for column re-equilibrium. The column oven temperature, flow rate, and injection volume were 35°C, 0.5 mL/min and 1 µL, respectively.

The positive rates of 10 PFASs in serum were almost 100%, and the average concentrations of PFASs ranged from 0.35 ng/mL to 15.1 ng/mL. Regarding urine samples, only four PFASs were detectable, and their positive rates were 47.5% to 100%, with average concentrations ranged from 1.39 ng/mL to 14.5 ng/mL. In serum samples, only two phthalate metabolites were observed at positive rates of 92.5% and 97.5%, respectively; their average concentrations were 0.76 ng/mL and 1.24 ng/mL, respectively. In urine samples, the positive rates of the five phthalate metabolites were 25% to 100% with average concentrations ranged from 2.14 ng/mL to 47.2 ng/mL. The study demonstrated the prevalence of endocrine-disrupting chemicals in human bodies. Biomonitoring of these chemicals is crucial for evaluating their potential health effects.

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