

Abstract

Endocrine disrupting compounds (EDCs) have brought attention because their potential ecological and health effects. We analyzed Estrone, estriol, 17 β -estradiol, and 17 α -ethinyl estradiol, which belong to this group of chemicals.

We used solid-phase extraction and LC/MS/MS techniques to analyze the four steroids. Without adjusting pH of water sample, we found J.T. Baker PolarPlus C₁₈ disks provided better retention and faster flow rates than traditional C₁₈ adsorbents. After washing with 40% methanol/60% water (in order to remove polar co-extracts), analytes were eluted by 50% methanol/50% dichloromethane. The eluate was concentrated to almost dryness using Speedvac and was reconstituted by 20 μ L of methanol. Four μ L were injected onto LC/MS/MS-ESI in negative ion mode and the mobile phase was 10 mM *N*-methylmorpholine aqueous solution (pH = 9.6)/acetonitrile with gradient elution. Quantification was done by isotope-dilution technique using deuterium- or carbon 13-labeled compounds.

At 50 ng/L level, the retention of four analytes were almost 100% in one liter of water sample. But at 200 ng/L level, breakthrough was found during last 300 mL for estriol. For the other three analytes, no any breakthrough was observed up to one liter. The wash step during the sample preparation only lose 1-3% of the analytes. The recoveries of elution and concentration were between 88-95% and 89-112%, respectively.

We estimate the instrument detection limits (mean \pm SD) of estrone, 17 β -estradiol, 17 α -ethinyl estradiol and estriol were between 49 \pm 31 pg, 70 \pm

33 pg, 95 ± 83 pg and 82 ± 39 pg, respectively. The limits of quantification were 200 pg, which is the lowest level at the calibration curve.

Key words : steroid estrogen, LC/MS/MS, solid-phase extraction