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Nota de aplicación

Analysis of Natural and Synthetic Estrogens at Sub-PPT Levels in Surface Water and Crude Influent Water Utilizing the ACQUITY UPLC System with 2D LC Technology and Xevo TQ-S

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

In this technology brief we describe the analysis of 17β-estradiol, estrone and 17α-ethinylestradiol in surface water, crude influent, and final effluent from a waster water treatment plant. Technologies used were Oasis off-line solid phase extraction, followed by analysis on the ACQUITY UPLC System with 2D LC Technology, coupled to the Xevo TQ-S Mass Spectrometer. This method has undergone a full validation and was found to meet the required performance criteria for this challenging analysis.

Benefits

- This method has undergone a full validation and was found to meet the required performance criteria for this challenging analysis.
- Ability to confirm and quantify the presence of natural and synthetic estrogens in surface and final effluent waters at sub ppt levels.

Introduction

Estrogens are routinely used either as contraceptive medicines or in hormone replacement therapy and can enter aquatic environments via the discharge of final effluent waters. Estrogens are believed to have a negative effect on aquatic environments by disrupting the hormonal systems of fish. In the EU directive 2013/39/EU, 15 additional priority substances were added to the water framework directive (WFD, 2000/60/EC). In this latest update, 17α-ethinylestradiol and 17β-estradiol were not included in this list but instead added to a watch list in order to gather further data regarding the presence of these compounds in aquatic environments and the risks they pose.

In this technology brief we describe the analysis of 17β-estradiol, estrone and 17α-ethinylestradiol in surface water, crude influent, and final effluent from a waster water treatment plant utilizing off-line solid phase extraction followed by analysis on Waters ACQUITY UPLC System with 2D LC Technology, coupled to the Xevo TQ-S Mass Spectrometer.

Results and Discussion

Surface water samples were initially extracted utilizing an optimized method on an off-line Oasis HLB Solid Phase Extraction (SPE) Cartridge. Crude influent and final effluent samples were first filtered, and then underwent the same Oasis HLB offline extraction step. This was followed by a second SPE step utilizing Sep-Pak Silica Cartridges. These off-line SPE steps are critical to achieving lower limits of detection by providing the initial concentration step and cleaner extracts; thus reducing ion suppression on the tandem quadrupole mass spectrometer.

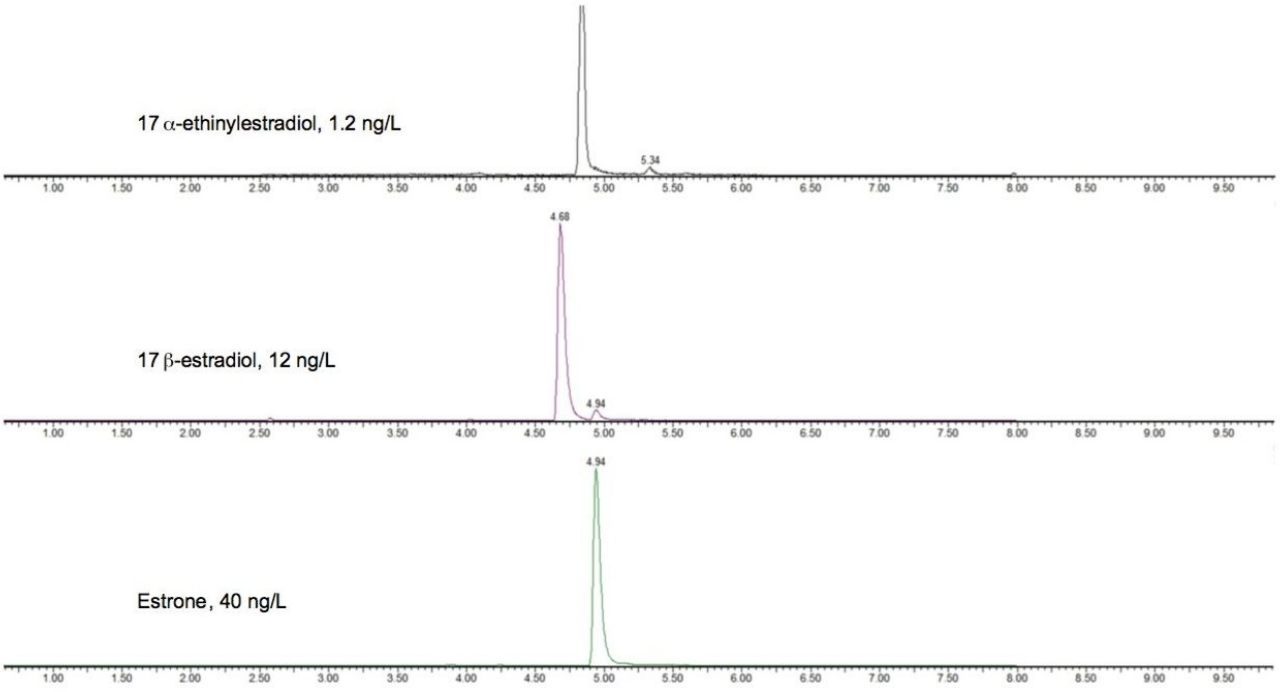

Figure 1. Example of chromatography on the ACQUITY UPLC BEH C_{18} Column in Elga water.

Extracted samples were injected onto the ACQUITY UPLC System with 2D LC Technology, coupled to the Xevo TQ-S. The use of the UPLC System with 2D LC Technology allows for a further concentration of the sample on-line by utilizing a Direct Connect Oasis HLB Column. Once concentrated on the on-line column the compounds were then eluted onto an ACQUITY UPLC BEH C_{18} Column to provide a reversed-phase separation. Figure 1 provides an example of the chromatography achieved. The samples were determined using electrospray in negative ion mode allowing two MRM transitions per compound. The unique RADAR function of the Xevo TQ-S, which allows simultaneous acquisition of both MRM and full scan MS data, was also employed to aid with method development.

This method has undergone a full validation* and was found to meet the required performance criteria, providing excellent linearity, as shown in Figure 2, and precision for this challenging analysis. A chromatogram showing detection of 17β-estradiol in low level final effluent spiked sample (0.6 ng/L) is shown in Figure 3.

** The performance test data was comprised of a NS30-style set (NS30, 1989) of tests of 11 batches of duplicate analyses of blanks, low and high standards, and low and high spiked samples of effluent. Spiked recovery data was also produced for river and influent matrices.*

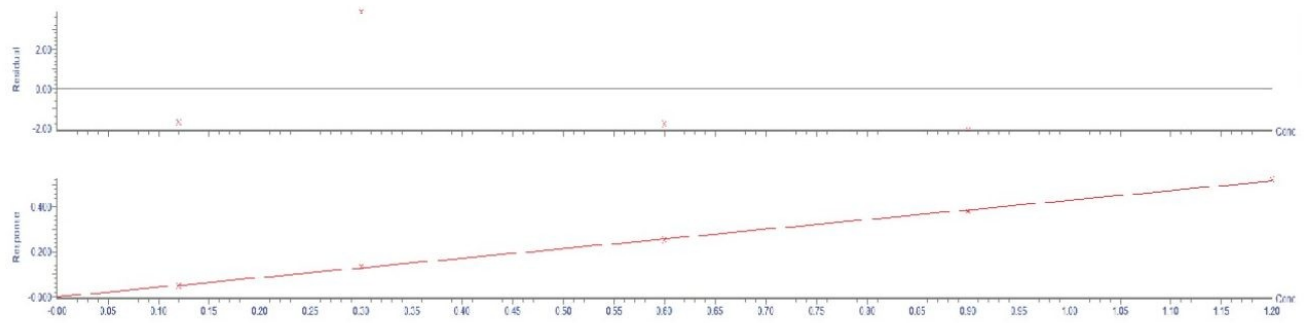

Figure 2. Example of calibration curve for 17α ethinyl estradiol over the range 0.120 to 1.20 ng/L, where excellent linearity (R2 >0.999) and %RSD (<±3 %) were achieved.

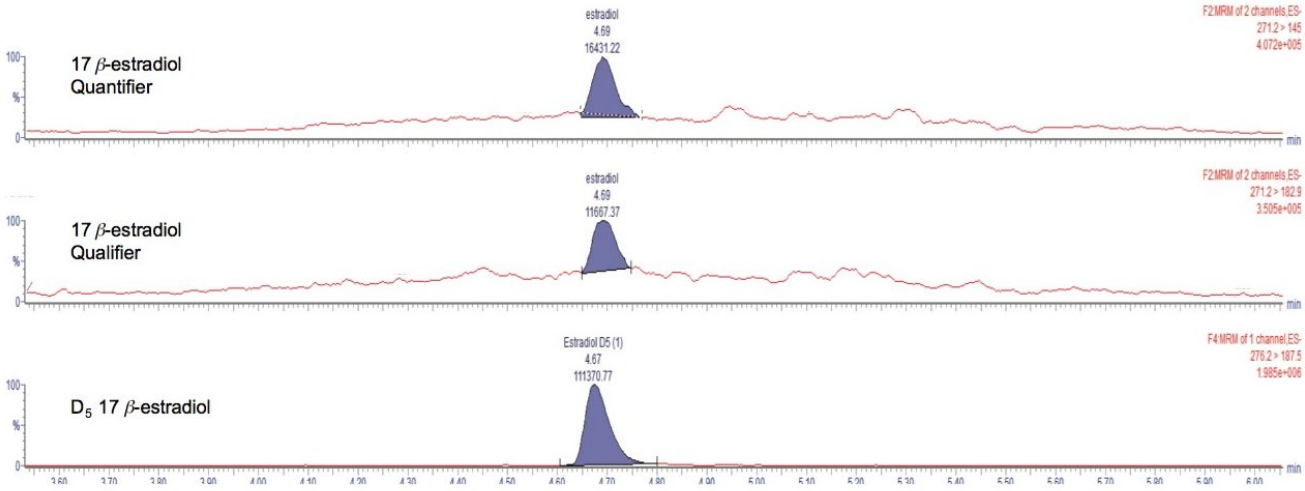

Figure 3. Example of the detection of 17β-estradiol in a low level final effluent sample spiked at 0.6 ng/L.

Conclusion

The combination of off-line SPE followed by analysis on the ACQUITY UPLC System with 2D LC Technology and Xevo TQ-S allows for ultra-sensitive detection of natural and synthetic estrogens in surface water, crude influent, and final effluent waters.

Featured Products

- ACQUITY UPLC System <https://www.waters.com/514207>
- Xevo TQ-S Mass Spectrometer <https://www.waters.com/10160596>
- RADAR <https://www.waters.com/134798882>

Available for purchase online:

- Oasis HLB Direct Connect Column <https://www.waters.com/186001792>

720005626, March 2016