

QuECHERS as perspective extraction technique for isolation of steroid estrogens from sediments in environmentally relevant concentrations

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The presence of endocrine disrupting compounds including steroid estrogens in water environment is worldwide problem. These compounds are released from different sources such as wastewaters from household through ineffective wastewater treatment plant also as result of runoff from agricultural areas after animal manure applications. Steroid estrogens cause a negative effect on growth, development and reproduction of aquatic organisms at very low concentrations. Due to higher octanol-water partition coefficient they tend to sorption and accumulation to solid matrices such as sediments. For a study of estrogens behavior in water-sediment system the effective extraction technique is needed. For this reason the four extraction technique: Soxhlet warm extraction (SWE), Accelerated solvent extraction (ASE), Microwave assisted extraction (MAE) and Quick Easy Cheap Effective Rugged Safe (QuECHERS) were compared using different extraction conditions. Estron (E1), 17 α – estradiol (α E2), 17 β - estradiol (β E2), estriol (E3), 17 α -ethinilestradiol (EE2) and 17 α -ethinilestradiol 2,4,16,16-d4 (EE2d4) were extracted from sediment sample at very low initial spike concentrations (1ng/g). Wider range of extraction recoveries (40-120 %) was considered as acceptable in those cases. Simple, fast and inexpensive original QuECHERS with PSA clean up provided the best extraction recoveries for all tested estrogens (53-84 %). Also MAE achieved the satisfactory extraction efficiency using DCM:acetone 3:1 as extraction solvent (50-70 %) but only for less polar estrogens. For estriol the more polar extraction solvents (acetone or methanol) must be used. More time consuming extraction technique ASE provided lower extraction recovery or worse repeatability in almost all solvent-temperature combinations in comparison with previous extraction. The worst results were obtained by most time consuming technique SWE that is totally unsuitable for this purpose.

Deuterated 17 β -estradiol was firstly used in SWE and ASE as internal standard for all tested estrogens but this compound caused an overestimation of E3. For this reason deuterated 16 α -hydroxy-17 β -estradiol was selected as preferable internal standard for E3.

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