Determination of Four Residues of Fluoroquinolones with Similar Structure in Fish by Modified QuEChERS Protocol Coupled to Capillary Electrophoresis (CE) with End-Column Electrochemiluminescence (ECL)

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doi: 10.20964/2020.07.55

Received: 2 March 2020/ Accepted: 23 April 2020 / Published: 10 June 2020

A new method was established for simultaneous determining ciprofloxacin (CIP), enrofloxacin (ENR), norfloxacin (NOR) and pefloxacin (PEF) in fish by capillary electrophoresis (CE) coupled with end-column electrochemiluminescence (ECL) with modified QuEChERS. The structural differences of 4 FQs are too small to separate them by conventional CE. However, their separation efficiency can be improved by adjusting the composition of separation buffer. The parameters about ECL analysis and CE separation were investigated in detail. The use of methanol and sodium sulfate in QuEChERS can improve the pretreatment effect of the sample. The optimum experimental conditions include analysis conditions (detection potential 1.20 V vs. Ag/AgCl, Ru(bpy)₃²⁺ concentration 6 mmol/L, phosphate buffer solution concentration 40 mmol/L and pH 7.0 in ECL detection cell), separation conditions (separation voltage 13.0 kV, 17.5% cyclodextrin solution (v/v) as additive, phosphate buffer solution concentration 20 mmol/L and pH 5.0 in capillary), and sample conditions (injection time 7 s and injection voltage 12.0 kV). The limits of detection (3σ) of this method were 8.8×10⁻⁵ mg/mL for CIP, 3.5×10⁻⁶ mg/mL for ENR, 7.5×10⁻⁵ mg/mL for NOR and 2.7×10⁻⁶ mg/mL for PEF. The relative standard deviations (RSD) were less than 2.6% for ECL intensity and less than 2.3% for migration time. This method was successfully utilized to simultaneously determine CIP, ENR, NOR and PEF in fish.

Keywords: Fluoroquinolones; Residue; Fish; CE-ECL; QuEChERS

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