Separation of Seven Residues of Fluoroquinolones from Fish Samples using Solid Phase Microextraction and Electrophoresis and their detection by Electrochemiluminescence method

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A new method for the simultaneous determination of 7 fluoroquinolones (FQs) in fish was established based on purifying and enriching by solid phase microextraction, separating by capillary electrophoresis (CE) and detecting by column end electrochemiluminescence. The effects of pH value, extraction time, extraction temperature, ionic strength and eluate type on the extraction efficiency of 7 FQs were investigated. The effects of buffer additives and separation voltage on the separation efficiency of 7 FQs were investigated. The best purification and enrichment parameters of 10.0 ml extraction solution made from 2.0 g fish sample are: pH value of extraction system is 7, extraction time is 40 min, extraction temperature is 50 °C, addition of NaCl is 2.0 g, and eluate is 2.0 ml methanol (containing 5% formic acid). The separation solution of CE is 0.02 mol/L phosphate buffer solution (pH=6, containing 18% cyclodextrin), and the separation voltage is 20 kV. All the 7 FQs have good linear relationship. Their detection limits were 0.4-0.8 µg/L. The recovery of fish samples was 89.2%-110.4%. 4 FQs were detected in two fish samples. This method has the advantages of small matrix effect, high sensitivity, simplicity and accuracy. It can be used for the detection of FQs in other fish or meat.

Keywords: Fluoroquinolones, Fish, Solid phase microextraction, Capillary electrophoresis, Electrochemiluminescence

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