

# Determination of inorganic anions in ethyl acetate by in-line hollow fiber membrane extraction with ion chromatography

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## Abstract

In this work, a novel hollow fiber membrane extractor was set up to extract inorganic anions from ethyl acetate using deionized water. Inorganic anions in slightly soluble organic solvents can be determined by the in-line hollow fiber membrane extractor coupled with ion chromatography at first time. Different aspects of the extraction procedure such as magnetic stirring speed, extraction flow rate and extraction time were optimized to achieve high extraction efficiency and good separation results. Satisfactory linear range, limits of detection and good repeatability were obtained. The procedure was applied to analyze inorganic anions in two commercial ethyl acetate samples.

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*Keywords:* Inorganic anions; Ethyl acetate; In-line hollow fiber membrane extraction; Ion chromatography

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Ion chromatography is a common method to analyze inorganic anions. In the last years, with the rapid advances in ion chromatography, it is no longer a difficult problem to determine inorganic anions even in some water-miscible solvents such as methanol, iso-propanol, acetone and N-methylpyrrolidone [1–3]. However, it is still a conundrum to analyze inorganic anions in organic solvents which are slightly soluble in water even with traditional sample pretreatment methods such as evaporation-enrichment method, electrodialysis and column-switching technology [1,2].

Hollow fiber membrane extraction (HFME) is a new-developed sample pretreatment model based on liquid–liquid extraction. In traditional methods, organic solvents are often used as extraction solvents to extract organic compounds from aqueous solutions [4–13]. In the present work, a simple hollow fiber membrane extractor was set up to extract inorganic anions from ethyl acetate with deionized water. Inorganic anions in slightly soluble organic solvents can be determined by the in-line hollow fiber membrane extractor coupled with ion chromatography at first time. Different aspects of the extraction procedure such as magnetic stirring speed, extraction flow rate and extraction time were investigated. In addition, the proposed method was validated for quantitative purposes and applied to two commercial ethyl acetate samples.

## 1. Experimental

The analysis of inorganic anions in ethyl acetate consisted of three steps: (1) using the in-line hollow fiber membrane extractor to extract anions with deionized water from ethyl acetate meanwhile eliminate the matrix; (2)

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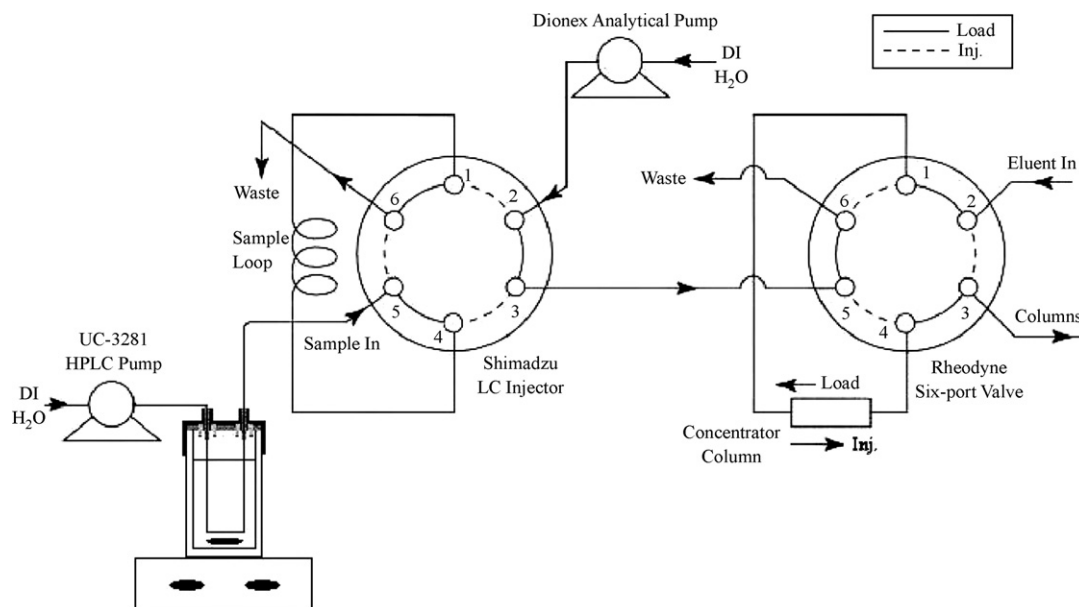


Fig. 1. Schematic diagram of in-line hollow fiber extractor coupled with ion chromatography.

concentrating the target analytes using a concentration column; (3) eluting out the concentrated anions for analysis by ion chromatography. Fig. 1 illustrates how these tasks were performed by the system. This initial configuration shows Shimadzu LC injector in the “Load” position and Rheodyne six-port valve in the “Inject” position.

Firstly, 8 mL ethyl acetate was placed into a sample vial. Deionized water was delivered by a UC-3281 HPLC pump (Union Opto-Electronic Technology Co., Ltd., Beijing, China) as acceptor phase through hollow fiber membrane to extract inorganic anions from ethyl acetate. The extract solution was loaded into 1.5 mL sample loop of Shimadzu LC injector. After the extraction was completed, Shimadzu LC injector was turned into the “Inject” position and Rheodyne six-port valve was turned into the “Load” position. The extract solution was transited from the sample loop to the concentration column with the deionized water by a Dionex analytical pump. Anions were retained on the concentration column and a little ethyl acetate which permeated into deionized water through hollow fiber membrane because of its slightly soluble in water was eliminated by washing the concentration column with deionized water at 2.0 mL/min. This step required 5 min. Meanwhile, the hollow fiber membrane was flushed by deionized water at 0.50 mL/min for 10 min to clean up anion residues before a new extraction cycle. Finally, by switching Rheodyne six-port valve into the “Inject” position, the anions were eluted out from the concentration column into the analytical column for separation.

All columns used in this work were purchased from Dionex (Sunnyvale, CA, USA). The separations were performed on an IonPac AS12A analytical column (250 mm × 4 mm I.D.) and an IonPac AG12A guard column (50 mm × 4 mm I.D.). An IonPac AG12A concentration column (50 mm × 4 mm) was used to concentrate the anions and eliminate the sample matrix. An eluent of 2.7 mmol/L sodium carbonate–0.3 mmol/L sodium bicarbonate was used to elute the four anions from the AS12A column at a constant flow rate of 1.00 mL/min. An anions self-regenerating suppressor (ASRS) with a 50 mA suppressor current from Dionex (Sunnyvale, CA, USA) was used to reduce the conductivity of the eluent.

## 2. Results and discussion

HFME is based on liquid–liquid extraction. Polypropylene hollow fiber membrane used in this work is a kind of hydrophobic microporous membrane. Ethyl acetate filled the pores of hollow fiber membrane because of its hydrophobic characteristic and contacted with deionized water in the inner wall of the hollow fiber membrane. Owing to the low solubility of the inorganic anions in the ethyl acetate and because of the correspondingly high solubility of

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