

PD1) Online preconcentration and preelution for the ion chromatographic determination of trace anions in high-sulfate wastewater

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1. Introduction

Ion chromatography has been used for the quantitative analysis of dissolved anions in a wide range of water samples. However, it cannot be directly applied for the determination of trace anions in aqueous samples composed of concentrated electrolytes, such as high-salinity water or sulphur-rich wastewater. Drainage waters from active and derelict mines over our country are commonly acidic and contain sulfate at very high levels. The determination of trace anions such as fluoride or chloride in these samples is particularly important for treatment of contamination. Although the EPA recommends sequential pretreatment of such samples through Ba, Ag and H-form resins, this is cumbersome and difficult to automate; moreover, these disposable resins are expensive for our wastewater samples to obtain guarantee acceptable recoveries. We provide here a simple, automated analytical procedure that is acceptable to high-sulfate water from derelict mines. The sample is preconcentrated and more strongly held ions are preeluted to the principal separation system, prohibiting less strongly held sulfate ion from transferring to the main analytical column.

2. Experimental

2.1. Samples and reagents

About 50 samples of high-sulfate wastewater from mainly derelict mines in Korea were analyzed and high-purity reagents were used throughout together with Milli-Q purified deionized water. Inorganic metal solutions and anion solutions, used as the standard analytes, were obtained from Wako (Osaka).

2.2. Apparatus

A Dionex DX300 chromatograph equipped with an AS40 autosampler was used. An AG14 guard column was used in conjunction with an AS14 analytical column for

separation. The sample loop was located in front of a preconcentration column (AG14 guard column) with a low-pressure 6-port loop injector. The actuation of the low-pressure valve was controlled by software command using one of the relay outputs of the IC.

3. Results and discussion

Concentration and volume of preeluting eluent and injection duration had crucial relationship with detection limitation of trace anions and effectiveness in removing high concentrated sulfate. As analytical efficiency was enhanced by using a guard column as a concentration trap column, retention time and peak response could be more easily estimated according to injection duration and concentration of preeluting solution.

4. Summary

Trace anions in high-sulfate wastewater from mainly derelict mines were determined by ion chromatography with on-line preconcentration and preelution technique. As the sample was preconcentrated and more strongly held ions were preeluted to the principal separation system, this approach was highly effective in removing large concentration of sulfate in high-sulfate wastewater. With this practical on-line preelution treatment, the peaks of fluoride and chloride showed good resolution even when the sulfate concentration was as high as 2000 mg/L and the analyzed total metal concentration was above 500 mg/L.

Reference

- Hautman, D. P.; Munch, D. J.; Eaton, A. D.; Haghani, A. W. 1999, Determination of perchlorate in drinking water using ion chromatography, EPA method 314.0.
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