



A new automated system for the determination of Al(III) species in dialysis concentrates by electrothermal atomic absorption spectrometry using a combination of chelating resin



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ABSTRACT

A new and rapid on-line flow injection (FI) procedure for the preconcentration and separation of aluminum(III) species in dialysis concentrates is described based on the combining of solid phase extraction with Lewatit TP-207 resin and electrothermal atomic absorption spectrometry (ETAAS). Lewatit TP-207 resin being a weakly acidic, macroporous cation exchanger with chelating iminodiacetate groups was firstly used in the determination of Al(III) species. The influences of the analytical parameters such as pH, type of eluent, flow rates of the eluent and sample, volume of the eluent, and matrix ions were investigated. A preconcentration factor of 40 and a detection limit (LOD, defined as 3 s/b) of 7.4 ng L^{-1} were achieved along with the sampling frequency of 21 h^{-1} , a sample loading time of 170 s, and a sample consumption of 2.8 mL. The relative standard deviation (RSD) was 2.1% for $1 \text{ } \mu\text{g L}^{-1}$ Al(III) level. The developed method was performed for the determination of Al(III) in dialysis concentrates. The results of the analysis of the certified reference material (CRM 403) are in good agreement with the certified value.

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1. Introduction

Aluminum (Al) is the third most abundant element in the lithosphere, very insoluble, and in most neutral natural waters its concentration is very low [1]. Aluminum is a common, nonessential element to which humans are frequently exposed by the wide use of aluminum containing foods, drinking waters, pharmaceuticals and cooking utensils [2–4]. Because of the wide occurrence of Al(III) and its potential negative roles to human beings there is growing interest in determining of ultra-trace Al(III) [5–7]. Dissolved Al is significant to environmental science because of its bio-availability and toxicity [8].

Biological effect of aluminum has not received much attention in the past. In recent years, however, a large amount of Al has been released into the environment and its solubility is significantly increasing because human activities have resulted in a serious problem of acidification [1]. Al was indicated to be involved as a causative factor in several clinical and neuropathological diseases, such as Alzheimer's disease, Parkinson's disease, Parkinson–Guam disease, amyotrophic lateral sclerosis, diabetes and cancer. Several epidemiological studies investigated the correlation between the over-taken of Al from drinking waters and the Alzheimer's disease [9]. Because Al is still used as a flocculating agent in potable water treatment, it would be present in drinking

waters. The WHO guideline for the permissible level of Al in drinking water is only 0.2 mg L^{-1} [10].

The serious toxic effect of aluminum was observed also on patients with renal failure subjected to dialysis, such dialysis dementia [11–13]. It was confirmed that decrease of aluminum content under critical level has a preventive effect. The dialysis fluids are prepared from dialysis concentrates that are mixed with pure water. If aluminum is present as a contaminant in these fluids, it is able to diffuse through the dialysis membranes and penetrate into the blood stream of the patient. The contamination levels in these cases depend strongly on the quality of the water and the dialysis concentrates used in the dialysis fluid preparation. The Official Pharmacopoeias require an accurate control of the trace levels of aluminum in commercial dialysis solutions, which must be lower than $10 \text{ } \mu\text{g L}^{-1}$. Hence, a simple and sensitive method is of primary importance for the effective monitoring of aluminum present as contaminant in dialysis concentrates [14].

The complex matrix with high levels of $2\text{--}5 \text{ mol L}^{-1}$ sodium, $0.03\text{--}0.05 \text{ mol L}^{-1}$ calcium, $0.01\text{--}0.02 \text{ mol L}^{-1}$ magnesium, and $0\text{--}0.07 \text{ mol L}^{-1}$ potassium as chlorides, sodium acetate or bicarbonate, sodium lactate and glucose, makes dialysis concentrates difficult to analyze [14,15]. Therefore, there is a strong need for Al monitoring in treated and raw waters.

Atomic absorption spectrometry with electrothermal atomization (ET-AAS) suffers from serious matrix interferences and contamination problems. Analysis of dialysis concentrates cannot be directly carried out by ET-AAS due to the high salt content. About 400 g L^{-1} of

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Table 1
Graphite furnace temperature/time program for the determination of Al(III).

Step	Temperature (°C)	Ramp time (sec)	Hold time (sec)	Internal flow (mL min ⁻¹)	Gas type
Drying	110	1	30	250	Ar
Drying	130	15	30	250	Ar
Ashing	1200	10	20	250	Ar
Atomization	2300	0	3	0	Ar
Cleaning steps	2550	1	3	250	Ar

essentially chlorides causes matrix interferences and additionally, insufficient precision since the aluminum content in these fluids is normally close to the limits of detection [16]. To reduce matrix effects in ET-AAS determinations, dialysis concentrates have been analyzed after up to 35-fold dilution with water [17]. The usual practice of diluting the sample aggravates the problem and leads to poor results. Addition of nitric acid [18] or orthophosphoric acid [19] as a matrix modifier was also used to minimize matrix effects. The results obtained were not reliable since the salt content in the solutions was still high [20]. Thus, the determination of aluminum in environmental, biological systems and dialysis concentrates are of current interest and the development of a simple, reliable, and accurate method to determine trace aluminum is in great demand.

Recently, the determination of trace levels of Al has been reported by various methods, such as electrothermal atomic absorption spectrometry (ET-AAS) [21], spectrophotometry [22,23], spectrophotofluorometry

[24], inductively coupled plasma mass spectrometry (ICP-MS) [25], inductively coupled plasma atomic emission spectrometry (ICP-AES) [26] and high-resolution continuum source atomic absorption spectrometry (HR-CS AAS) [27] have been used for element analysis in dialysis fluids. However, for dialysis fluids there is a lack of simultaneous analysis methods since the determinations cannot be directly performed even by ET-AAS because of the high saline content of the samples, which causes serious matrix interferences [28].

In this study, Lewatit TP-207 is a weakly acidic, macroporous cation exchange resin with chelating iminodiacetate groups for the selective extraction of heavy metal cations from weakly acidic to weakly basic solutions. It was firstly used for the preconcentration/separation of Al(III) in samples with high salt content such as dialysis concentrates using the flow injection (FI) system which combined a solid phase extraction procedure and ET-AAS technique. The accuracy of the method was verified by analysis of a certified reference material (CRM 403) and recovery measurements made with spiked natural water samples. The results showed good agreement with the certified value and the recoveries were sufficiently high.

2. Experimental

2.1. Instrument

A PerkinElmer model AAnalyst 800 atomic absorption spectrometer (Norwalk, CT, USA) with a transversely heated graphite tube atomizer

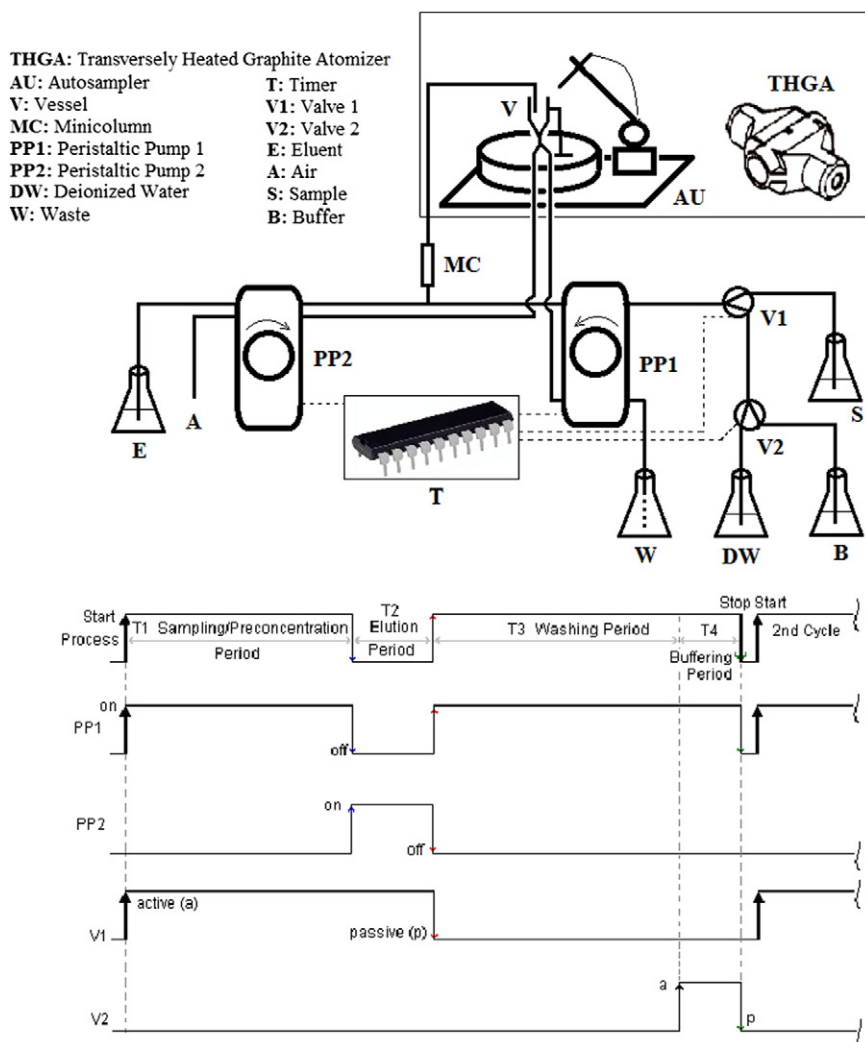


Fig. 1. FI-ETAAS manifold and its operation steps.

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