The separation and simultaneous determination of V(IV) and V(V) species complexed with EDTA by IC-ICP-OES

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Abstract

A method for the separation of V(IV) and V(V) in the form of the EDTA complexes using anion chromatography with a Dionex AG5 anion exchange guard column, and the simultaneous determination of V(IV) and V(V) by inductively coupled plasma optical emission spectrometery is described. The interference from other elements is negligible. The detection limits of V(IV) and V(V) were 0.02 mg/ℓ and 0.05 mg/ℓ, respectively, using a glass nebuliser and 2 µg/ℓ for both species by using an ultrasonic nebuliser. The linear range was two orders of magnitude. The method was applied to the analysis of spiked water and industrial samples containing V in different oxidation states.

Introduction

V(IV) and V(V) species play an important role in many industrial and environmental processes. V and its compounds are used extensively in the steel and petrochemical industries. The South African petrochemical industry, for example, uses the Sulfolin and Benfield processes, based on the V(IV)/V(V) redox couple to remove sulphur and CO_2, respectively, from process streams in the production of petrol and diesel from coal. Both species can exist in the environment but V(V) species are the most stable and also the most toxic (Browning, 1961; Cotton and Wilkinson, 1988). Other oxidation states such as V(II) and V(III) are not stable and will be oxidised to V(IV) and V(V) by atmospheric oxygen. Both V(IV) and V(V) species may find their way into the natural environment, in particular surface waters as toxic pollutants. V(IV) can be stabilised in natural waters by complexation with a variety of ligands, such as carboxylic acids.

In recent years V-speciation studies have focused on the determination of V in natural waters, (Bosque-Sendra et al., 1998; Dupont et al., 1991; Miura, 1990; Yamane et al., 1998) biological systems (Elvingson et al., 1997; Hirayama et al., 1992; Kawakubo et al., 1995) and in industrial processes (De Beer and Coetzee, 1994; Murthy et al., 1989). A literature survey shows that most published analytical methods focus on the determination of total V or the determination of one species at a time. These methods include high performance liquid chromatography (De Beer and Coetzee, 1994; Komarova et al., 1991; Miura, 1990; Miura et al., 1990), spectrophotometry (Balaji et al., 1998; Bosque-Sendra et al., 1998; Chauhan and Kakkar, 1992; Iranpoor et al., 1992; Kawakubo et al., 1995; Murthy et al., 1989; Shah et al., 1991; Zucchi et al., 1998), flow injection (Grudpan and Nacapricha, 1991; Taylor et al., 1996; Yamane et al., 1998), atomic absorption spectrometery (Chakraborty and Das, 1994; Frankenberger et al., 1991; Yaman and Gucer, 1994), atomic emission spectrometery (Dupont et al., 1991; Hirayama et al., 1992), colorimetric (Serrat and Morell, 1994), and electrochemical methods (Ensafi and Naderi, 1997; Sander and Henze, 1996; Vukomanovic and Van Loon, 1994). Bosque-Serdra developed a method for V speciaton (Bosque-Sendra et al., 1998) based on the pre-concentration of V(IV) in the first step and V(V) after reduction with ascorbic acid in the second step. Only a few methods are reported for the simultaneous measurement of V species (Murthy et al., 1989; De Beer and Coetzee, 1994; Komarova et al., 1991). Disadvantages of these methods include: low sensitivities, interference and lengthy procedures.

No general accepted or standard method for the simultaneous determination of vanadium species is currently available. This is particularly true for industrial samples with complex matrices and environmental samples with low concentrations of V.

The aim of this work was to develop a simple, fast, cost-effective, and interference-free method for the simultaneous determination of V(IV) and V(V) that would be useful in a routine industrial or water quality laboratory. This was achieved by using a hyphenated technique approach with ion chromatography inductively coupled plasma optical emission spectrometry (IC-ICP-OES). V(IV) and V(V) species were complexed with EDTA, separated on an anion exchange column using a modified carbonate and bicarbonate buffer spiked with EDTA as eluant, and quantitatively and element-specifically determined by ICP-OES.

Experimental

Instrumentation

Ion chromatography

A Dionex ion chromatography pump (2000i) and Waters injection system in conjunction with a Dionex guard column were used as the separation system. Three different guard columns, AG4, AG5 and AG14 were evaluated in this work.

Table1 lists the optimised chromatographic conditions for separation of V(IV) and V(V).

TABLE 1
Optimised chromatographic conditions

Column	AG5 Guard Column
Eluant	12 mmol/ℓ $NaHCO_3$, 4 mmol/ℓ Na_2CO_3 and 20 mmol/ℓ EDTA
Flow rate	1.8 mℓ/min
Sample size	50 µℓ

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Received 20 November 2000; accepted in revised form 16 October 2001.