Contents lists available at ScienceDirect

## Environmental Nanotechnology, Monitoring & Management

journal homepage: www.elsevier.com/locate/enmm

### Determination of vanadium in water samples from Brazilian mineral spring (Ibirá Spa) using ICP-MS



<sup>a</sup> Department of Chemistry and Environmental Sciences (DQCA), São Paulo State University (Unesp), Institute of Biosciences, Humanities and Exact Sciences (Ibilce),

Campus São José do Rio Preto, SP, 15054-000, Brazil

<sup>b</sup> Department of Chemistry, Federal University of Paraná, Curitiba, PR, 81531-980, Brazil

ARTICLE INFO	A B S T R A C T
<i>Keywords:</i> Vanadium determination Mineral water Drinking water ICP-UCT-MS KED	A methodology was developed for the determination of vanadium in water samples from a hydromineral spa located in Ibirá, Sao Paulo State, Brazil and in bottled drinking water, employing inductively coupled plasma mass spectrometry (ICP-MS). Evaluation of matrix isobaric interferences focused on the ${}^{35}Cl^{16}O^+$ polyatomic species at $m/z$ 51. The determinations were carried out in standard and kinetic energy discrimination (KED) modes. Optimization of the helium flow rate in the KED mode showed that the most effective interference correction was achieved at a flow rate of 5.0 mL min <sup>-1</sup> . The limits of detection (LOD) were 0.0097 and 0.0094 µg L <sup>-1</sup> for the standard and KED modes, respectively. The concentrations of vanadium quantified in the samples studied ranged from 65.2 $\pm$ 0.36 to 831 $\pm$ 8.7 µg L <sup>-1</sup> . The accuracy of the method was confirmed by

analysis of a certified reference material for water (NIST 1640A), achieving a recovery of 101%.

#### 1. Introduction

The freshwater present on the planet includes a small fraction classified as mineral water. In Brazil, this water is regulated by the Mineral Water Code (Decree Law number 7.841, of 08/08/1945), which covers medicinal mineral waters that possess physicochemical characteristics beneficial to health (Anon, 2017a). Interest in these waters arises from the fact that the human body requires a variety of chemical elements, some of which can be supplied in mineral water (Misund et al., 1999).

Existing Brazilian environmental legislation concerning water used for consumption and bathing includes Regulation 2914 (2011), of the Ministry of Health, and CONAMA Resolution No. 274 (2000), respectively. Regulation 2914 (formerly Regulation 518) of Ministry of Health describes the quality and portability of water using microbiological, physical, chemical, and radiological parameters (Anon, 2017b). CON-AMA Resolution No. 274 includes parameters indicating the suitability of surface waters for activities such as bathing and water sports (CONAMA, 2017).

In Brazil, one of the many sources of mineral waters is the hydrothermal spa of Ibirá, located in the north of São Paulo State, at an altitude of 480 m and 6 km from the town of Ibirá. The spa includes several mineral water fountains and a bathing facility (Pupo, 1974). The medicinal characteristics of this water include high levels of vanadium (in the form of vanadate,  $VO_3^{-}$ ), which can act to moderate the oxidation of organic compounds, accelerate cell renewal, and help in combating certain diseases (Pupo, 1974; Crans et al., 1990; Thompson et al., 1999). Vanadium concentration varies greatly in water ranging from 0.2 to 100 µg L<sup>-1</sup> in freshwater (National Academy of Sciences, 1974) and from 0.2 to 29 µg L<sup>-1</sup> in seawater (Waters, 1977), depending on geographical formation.

Contemporary medicinal interest in vanadium dates from 1977, when it was discovered that the element has the capacity to inhibit ATPase. Another important development was the discovery of its relationship to insulin levels and its use in drugs for diabetes, although some patients have shown problems associated with prolonged use, and various organic vanadium compounds are currently being studied (Cornelis et al., 2005).

Vanadium can be determined using several analytical techniques, including inductively coupled plasma mass spectrometry (ICP-MS), which provides multielemental analysis and offers low limits of detection and high sensitivity. Difficulties commonly encountered in ICP-MS include spectral interferences, especially isobaric overlaps caused by other isotopes with the same mass/charge ratio as the analyte or by polyatomic species that arise from the combination of isotopes present in the plasma, derived from the reagents or the sample matrix (Thomas, 2013). The determination of vanadium by ICP-MS can especially be compromised by spectral interference due to the <sup>35</sup>Cl<sup>16</sup>O<sup>+</sup>

\* Corresponding author. E-mail address: clariceamaral@yahoo.com.br (C.D.B. Amaral).

http://dx.doi.org/10.1016/j.enmm.2017.04.005





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Fig. 1. Satellite image showing the locations of the sampling sites. Adapted from Google Earth.

species. The formation of such interference is expected, because chloride is present in water samples and oxygen can originate from the atmosphere and the reagents (D'Ilio et al., 2011).

One strategy to eliminate this interference is to use a reaction/ collision cell in kinetic energy discrimination (KED) mode. The introduction of an inert gas, such as He, into the cell results in its collision with the polyatomic species, which has a larger diameter than the ion of interest, so collision with the latter is not favored. This enables determination of  ${}^{51}V^+$  free of interference from  ${}^{35}Cl^{16}O^+$ (Chrastný et al., 2006). The success of this correction depends on several factors, including the flow rate of the collision gas. Previous studies have shown that for water containing chloride at concentrations in the region of 500 mg L<sup>-1</sup>, helium flow rates of around 2.5 mL min<sup>-1</sup> can result in effective correction of the interference (Mazurova et al., 2015). However, the concentrations commonly found in potable water rarely reach this level, and Regulation 2914 (2011) sets a maximum chloride concentration of 250 mg L<sup>-1</sup> (Anon, 2017b).

When the instrument is operated in KED mode, the collision gas flow rate is critically important in the interference correction process. High flow rates can result in the collision of helium molecules with the analyte molecules, hence reducing the signal intensity. High flows can also adversely affect the region of the interface maintained under vacuum, disturbing the stability of the plasma (Salazar et al., 2011). Conversely, a low flow rate may inhibit effective collision with the interfering molecules, allowing them to reach the detector, where they are quantified together with the analyte molecules. Two simultaneous processes occur during the collision of helium with the interfering species. These are damping of the collision energy and defocusing, with a loss of axial energy of the ions and reduction of the breadth of the ion energy distribution, with thermal stabilization with the collision gas leading to migration of the ions towards the quadrupole (Tanner et al., 2002). This decreases the interference signal, as well as part of the analyte signal, and must be evaluated to ensure that the accuracy of the method is not adversely affected.

Most chemical elements have more than one isotope. In the case of vanadium, its most abundant isotope, <sup>51</sup>V, is selected for analytical purposes, since the <sup>50</sup>V isotope represents only 0.25% of the total abundance. However, in addition to overlap of the <sup>35</sup>Cl<sup>16</sup>O<sup>+</sup> ion (which is the most important interferent), the vanadium at the *m/z* 51 suffers interference from the ions <sup>40</sup>Ar<sup>11</sup>B<sup>+</sup> (79.78%), <sup>102</sup>Ru<sup>2+</sup> (31.34%), <sup>37</sup>Cl<sup>14</sup>N<sup>+</sup> (24.14%), and <sup>34</sup>S<sup>16</sup>O<sup>1</sup>H<sup>+</sup> (4.20%). The <sup>50</sup>V isotope suffers isobaric overlap interferences from <sup>50</sup>Ti (5.40%), <sup>50</sup>Cr (4.35%), the ions <sup>100</sup>Ru<sup>2+</sup> (12.70%) and <sup>100</sup>Mo<sup>2+</sup> (9.68%), and the polyatomic species, H<sup>14</sup>N<sup>35</sup>Cl<sup>+</sup>, <sup>38</sup>Ar<sup>12</sup>C<sup>+</sup> and SO<sup>+</sup> (D'llio et al., 2011).

for determination of  ${}^{51}V^+$  by inductively coupled plasma mass spectrometry, including the evaluation of spectral interferences at m/z 51 and the proposal of a correction method. This enabled determination of the contents of vanadium in samples of water from the Ibirá hydromineral spa and in commercial water samples.

#### 2. Materials and methods

#### 2.1. Samples collection

Five samples of fountain water (Saracura, Carlos Gomes, Seixas, Jorrante and Adhemar de Barros fountain), two samples of tap water (Tap at the main entrance and stream), one sample of lake water, and two bottled mineral water samples (purchased at a local supermarket of Ibirá city) were evaluated. The samples were collected at the Ibirá hydrothermal spa (São Paulo State, Brazil) using sterilized 50 mL plastic flasks appropriate for the collection and storage of water. The first set of samples was collected at the end of the rainy season and the second was collected after a long period of drought. Both collections were in the afternoon, between 13:30 and 15:30 h, and included the water fountains, the central lake, and the stream. Commercial water samples were analyzed, as well as a certified reference material of water (NIST 1640A).

The locations of the sampling points are indicated in the satellite image (adapted from Google Earth) shown in Fig. 1. Location 1 is the Saracura fountain  $(21^{\circ}6'28.37''S; 49^{\circ}10'55.77''W)$ ; location 2 is the Carlos Gomes fountain  $(21^{\circ}6'27.26''S; 49^{\circ}10'54.88''W)$ ; location 3 is the Seixas fountain  $(21^{\circ}6'26.79''S; 49^{\circ}10'50.21''W)$ ; location 4 is the Jorrante fountain  $(21^{\circ}6'31.36''S; 49^{\circ}11'8.62''W)$ ; location 5 is the Adhemar de Barros fountain  $(21^{\circ}6'28.25''S; 49^{\circ}10'54.31''W)$ ; location 6 is the tap at the main entrance  $(21^{\circ}6'16.82''S; 49^{\circ}10'47.16''W)$ ; location 7 is the central lake  $(21^{\circ}6'26.89''S; 49^{\circ}11'3.76''W)$ ; and location 8 is the stream  $(21^{\circ}6'25.62''S; 49^{\circ}10'42.28''W)$ .

#### 2.2. Analyses of the physicochemical parameters of the water samples

After collection of the samples, measurements of pH (PA 200 pH meter, Marconi, Piracicaba, São Paulo, Brazil), conductivity (CA 150 conductivity meter, Marconi, Piracicaba, São Paulo, Brazil), and turbidity (HI93703 portable turbidimeter with range of 0–1000 FTU, Hanna Instruments, Barueri, São Paulo, Brazil) were made.

#### 2.3. Determination of chloride using the Mohr method

On the above, the aim of this work was to develop a methodology

The chloride concentrations in the samples were determined using

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