



Development of a miniature dielectric barrier discharge–optical emission spectrometric system for bromide and bromate screening in environmental water samples



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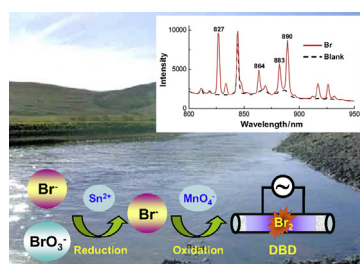
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HIGHLIGHTS

- Dielectric barrier discharge (DBD) is for the first time used as a radiation source for the excitation of bromine and its emission.
- A DBD–optical emission spectrometric (OES) system was developed for the detection of bromide and bromate.
- The DBD–OES system has been demonstrated by screening trace bromide and bromate in a series of environmental water samples.

GRAPHICAL ABSTRACT



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ABSTRACT

Dielectric barrier discharge (DBD) at atmospheric pressure provides an efficient radiation source for the excitation of bromine and it is used for the first time for optical emission spectrometric (OES) detection of bromide and bromate. A portable DBD–OES system is developed for screening potential pollution from bromide and bromate in environmental waters. Bromide is on-line oxidized to bromine for in-situ generation of volatile bromine. Meanwhile, a helium stream carries bromine into the DBD micro-plasma for its excitation at a discharging voltage of 3.7 kV and optical emission spectrometric detection with a QE65000 charge-coupled device (CCD) spectrometer in the near-infrared spectral region. Similarly, the quantification of bromate is performed by its pre-reduction into bromide and then oxidized to bromine. The spectral characteristics and configuration of the DBD micro-plasma excitation source in addition to the oxidation vapor generation of bromine have been thoroughly investigated. With a sampling volume of 1 mL, a linear range of 0.05–10.0 mg L⁻¹ is obtained with a detection limit of 0.014 mg L⁻¹ by measuring the emission at 827 nm. A precision of 2.3% is achieved at 3 mg L⁻¹ bromide. The system is validated by bromine detection in certified reference material of laver (GBW10023) at mg L⁻¹ level, giving rise to satisfactory agreement. In addition, it is further demonstrated by screening trace bromide and bromate as well as spiking recoveries in a series of environmental water samples.

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1. Introduction

Naturally, bromide presents in many water sources and some are used as drinking waters via disinfection processes [1]. It is reported that the content of bromide in source waters is closely related to the public health impact, because bromine-containing

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disinfection by-products in drinking water [2], e.g., brominated organic compounds and bromate, are prone to be formed during chlorination or ozonation disinfection processes. Among these bromine-containing disinfection by-products, bromate is classified as a potential human carcinogen by the International Agency for Research on Cancer [3,4], whose maximum allowed concentration is limited at $10 \mu\text{g L}^{-1}$ in drinking water by China National Standard. It is demonstrated that as the precursor of bromate, the concentration level of bromide has a significant impact on the quality of drinking water [5,6]. Nowadays, bromine-containing compounds, e.g., flame retardants [7] and pesticides [8], have been widely used, and the quality of environmental water is seriously threatened with the development of industrial and agricultural productions, which are gradually released into environmental water. Seawater intrusion and mineralization during mining, processing and usage also contribute to the pollution of bromide in environmental water [9,10]. Therefore, it is highly desired to provide a timely warning of possible pollution from bromide and bromate in environmental water in view of protecting public health.

In order to evaluate the bromide contamination in environmental samples, a series of analytical techniques have been developed for bromide detection in a variety of real samples. Spectrophotometry [10] and chemiluminescence [11] are alternatives for fast determination of bromide with simple instruments, but they are actually rarely used restricted by the poor detection limit and matrix interferences. Neutron activation analysis (NAA) [12], X-ray fluorescence spectroscopy (XRF) [13] and laser-induced plasma spectroscopy (LIPS) [14] possess a major advantage in direct solid analysis without sample dissolution. Nevertheless, their performance depends on the quality of the solid matrix-matched standards used for instrument calibration. Inductively coupled plasma–optical emission spectrometry (ICP–OES) [15] and inductively coupled plasma–mass spectrometry (ICP–MS) [16–18] are most attractive for the determination of trace bromide attributed to their high sensitivity and selectivity. Especially, their combination with chromatographic separation, e.g., gas chromatography (GC) [19], liquid chromatography (LC) [20] and capillary electrophoresis (CE) [21], facilitates bromine speciation at trace levels. However, one important drawback for ICP–OES and ICP–MS is the use of expensive bulky instruments, which restricts their application in some specific cases, especially for field analysis. From this point of view, simple and portable instruments for the determination of bromide with favorable sensitivity are highly demanded.

The miniaturization of atomic spectrometric systems opens a new avenue for portable instrument and has been paid extensive attentions, where the employment of micro-plasma as excitation source is among the most attractive approach for elemental analysis [22,23]. In order to improve the detection sensitivity, micro-plasma is frequently hyphenated with an appropriate sample introduction approach, e.g., vapor generation, instead of direct solution sampling [24,25]. The vaporized analytes are injected into the micro-plasma to increase their excitation, which are favorable for the preconcentration of analyte and at the same time for the elimination of interfering matrix components. Among the various micro-plasma generation approaches, dielectric barrier discharge (DBD) at atmospheric pressure exhibits powerful capability in atomic spectrometric instrument miniaturization, which can offer a non-thermal micro-plasma with respect to conventional thermal excitation sources [26,27]. Its prominent feature of small size, ease of fabrication, low temperature and power consumption as well as high dissociation/excitation/ionization ability are favorable for developing a series of analytical devices [28]. As far as OES is concerned, DBD micro-plasma instead of ICP as an excitation source has been demonstrated for the elemental determination of mercury [29,30], thimerosal [31], ammonia [32], nitrogen [33], iodine [34] and halohydrocarbons [35].

In the present study, DBD is used for the excitation of bromine. A simple and sensitive emission spectrometric system is for the first time developed for trace bromide and bromate screening in environmental waters. Sequential injection is employed for sample introduction for on-line oxidation vapor generation [36,37] to produce vaporized bromine, which is afterwards carried into the DBD excitation source by a helium flow and is excited therein. The capability of DBD micro-plasma for bromine excitation is demonstrated, and the emission spectra obtained in the near-infrared spectral region facilitate its quantification. This provides a promising portable analytical system for fast in field screening of bromide and bromate.

2. Experimental section

2.1. Apparatus and the experimental set-up

The schematic diagram of the miniature DBD–OES system used for the determination of bromine is shown in Fig. 1. The sample introduction is achieved with oxidation vapor generation by using a sequential injection system (FIALab Instruments, USA), which consists of two syringe pumps of 2.5 mL and 1.0 mL, a 6-port selection valve and an auxiliary peristaltic pump. All connecting tubes are made of 0.8 mm i.d. PTFE (polytetrafluoroethylene) with PEEK nuts/ferrules. The capacities of the holding coil and the reaction coil are both ca. 2 mL. A piece of ceramic tube (1.5 mm i.d., 2.9 mm o.d., and 100 mm length) serving as a DBD excitation chamber provides a small gas path for the introduction of a helium stream. There are two copper ring electrodes of 1 mm length surrounding the outside of the ceramic tube at a distance of 30 mm from each other. The distance from the nearer electrode to the end of the ceramic tube is set at 10 mm. When an appropriate high-frequency and high-voltage electric field is applied to the two ring electrodes by using a neon power supply (ENT-106B, Xinxing Neon Light Supply Company Ltd., Guangzhou, China) controlled by a touch regulator (TDGC2-0.5kVA, Hongbao Electric Co., Ltd., Wenzhou, China), the DBD micro-plasma is ignited and served as an excitation source. The neon power supply provides a sinusoidal discharging voltage of 2–4 kV with a frequency of ca. 38 kHz. A QE65000 CCD spectrometer (Ocean Optics, USA) combined with a fiber-optic probe (20 cm length) with a core diameter of 950 μm is placed at 5 mm distance to the exit of the DBD excitation chamber for recording the optical emission spectra from 200 to 980 nm, which is furnished with a 50 μm slit and a 300 lines/mm grating. An integration time of 100 ms and an average of 3 scans for the CCD spectrometer are employed.

2.2. Reagents

All the reagents used in this work are at least of analytical reagent grade and purchased from Sinopharm Chemical Reagent Co. (China–SCRC). De-ionized water (18 $\text{M}\Omega \text{ cm}$) is used throughout the experiments. Stock solutions (1000 mg L^{-1} bromine) of bromide and bromate are prepared by dissolving appropriate amount of potassium bromide and potassium bromate in de-ionized water, respectively. Working standard solutions of bromide are prepared daily by step-wise dilution of the stock solution of bromide with 1 mol L^{-1} sulfuric acid. 0.08 mol L^{-1} potassium permanganate is used as an oxidant for bromine vapor generation. SnCl_2 is used for the pre-reduction of bromate to bromide which is afterwards oxidized to bromine vapor. De-ionized water is used as carrier solution.

2.3. Sample pretreatment

The validation of the miniature DBD–OES system for bromide and bromate determination is performed by using a certified

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