



Improved determination of iodine by sequential (photo)chemical vapor generation and pneumatic nebulization in the programmable temperature spray chamber and inductively coupled plasma optical emission spectrometry



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ARTICLE INFO

Article history:

Received 16 September 2013
Received in revised form 23 October 2013
Accepted 1 November 2013
Available online 15 November 2013

Keywords:

Iodine
CVG–ICP–OES
Programmable temperature spray chamber
Water

ABSTRACT

Continuous generation of volatile iodine species based on chemical or photochemical oxidation may be further improved using a sequential (photo)chemical vapor generation/pneumatic nebulization/programmable-temperature spray chamber (P)CVG–PN–PTSC system providing the benefits of a temperature-controlled ICP sample introduction technique. Two experimental setups were designed and optimized to obtain the higher sensitivity. In the CVG mode, the sample and the oxidizing solution are continuously pumped and mixed directly before entering a concentric nebulizer placed in the PTSC, while in PCVG mode the sample solution containing 3%_{v/v} acetic acid is on-line exposed to UV radiation before nebulization.

A study was conducted to evaluate the performance of a new CVG–PN–PTSC technique for determination of iodine element by ICP–OES. The ICP–OES intensity is enhanced by a factor of up to 12 by prior oxidation of the iodide to volatile species combined with subsequent pneumatic nebulization, presumably because of increased analyte transport efficiency into the plasma. Next, by heating the spray chamber at 40 °C, signal intensities were enhanced by a factor of 3 as compared to the results found at room temperature. Furthermore, using an elevated temperature of the spray chamber slightly increased plasma robustness (MgII to Mgl line intensity ratio exceeds 10), likely as a result of increased water vapor that improves plasma stability as well as sensitivity. Both systems were optimized in a wide range of chemical and operating parameters.

With both CVG and PCVG–PN–PTSC, the slopes of analytical working graphs of ICP–OES are improved by over an order of magnitude for iodine compared to those with a conventional pneumatic nebulization. Generally, for determination of iodine, the technique exhibits a linear dynamic range of 3 orders of magnitude. For iodine, LODs of 11 ng mL^{−1} were achieved in CVG mode and 17 ng mL^{−1} in PCVG mode at 206.15 nm. The technique also gave good short-term stabilities below 2% RSD for both modes. Finally, a continuous CVG technique using sodium nitrite exhibits a better analytical performance than the PCVG mode. Quantitative determination of iodide in mineral water is preferentially carried out by the standard addition procedure. The proposed methodologies were validated with determining of trace iodide in simulated seawater in 2% nitric acid standard reference material CRM–SW, giving rise to satisfactory results.

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1. Introduction

Among analytical methodologies applicable for the determination of trace iodine, those involving molecular and atomic spectrometry are widely represented [1–3]. Inductively coupled plasma optical emission spectrometry (ICP–OES) is a suitable technique for iodine determination, however is not without its limitations. The most sensitive analytical lines of iodine lie in the vacuum-ultraviolet region of the spectrum providing the detection limits at level of tens of ng mL^{−1}. Moreover, the lines at 178.3 nm and 183.0 nm suffer from both spectral

interferences from common elements and relatively narrow linear dynamic ranges of 3 orders of magnitude that complicate routine iodine determination in food and environmental samples [1,4,5]. The moderate power microwave induced plasma (MIP–OES) operating with helium offers similar analytical performance [6,7]. ICPMS provides excellent detection power and isotopic selectivity however in the case of iodine it suffers from low ionization efficiency and matrix effects. As a result, these methods are not readily applicable for routine analysis, especially when ultra-trace iodine concentrations need to be analyzed. The analytical performance characteristics needed for determination of iodine require the use of well adapted sample introduction techniques to enhance detection power [1,8,9].

Chemical vapor generation (CVG) has frequently been applied for iodine determination by ICP/MIP atomic spectrometry [10–13]. This

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technique exhibits several advantages: efficient matrix separation, which leads to reduction of interferences, and high transport efficiency of the analyte to the detector for enhanced sensitivity and relatively low detection limits at the low ng mL^{-1} . The major drawback with the conventional CVG method is that it takes several minutes to reach a steady-state signal of iodine. Also, the technique is disadvantaged, by use of high concentrations of acids and hence poor stability of pump tubings, and also by memory effects caused by adhesion of iodine vapors on the walls of elements of the sample introduction system. Recently, it has been shown that vapor generation can also be achieved with the use of UV radiation and several volatile iodine species including I_2 , CH_3I and $\text{C}_2\text{H}_5\text{I}$ may be generated in radical induced reactions with low molecular weight organic acids [9].

Recent work [9,14] in CVG of iodine focused on using the solution nebulization systems in order to introduce in-situ generated volatile iodine species into ICP. With iodine, only some of their species are readily converted into volatile compounds upon CVG. Matusiewicz and Ślachciński employ a ultrasonic nebulizer equipped with a triple sample introduction system, taking advantage of direct CVG by processing the reaction solutions as an aerosol in the spray chamber just before entering the plasma. Previously, the conventional pneumatic nebulizer-spray chamber system has been used for direct CVG-PN-ICP-OES of iodine by Nakahara and Wasa [15]. A similar system was used by Anderson and Markowski for developing the analytical procedure for determination and speciation of iodine in environmental matrices [16].

On the other hand, the advantageous effect of spray chamber heating to vaporize the aerosol without removing water has been claimed for some analytical applications. Several heated nebulization systems were developed to improve the analytical performance of plasma spectrometric techniques using a thermospray nebulizer [17], pre-evaporation tube [18] or heated spray chamber [19].

The temperature of the sample introduction system, and more specifically the spray chamber, can have dramatic effects on sensitivity and plasma stability. In order to achieve this, a pneumatic nebulization in the programmable temperature spray chamber (PN/PTSC) system was modified to allow for the addition of a photochemical (PCVG) or chemical (CVG) line into the nebulization setup. The systems were evaluated in terms of aerosol characteristics, sensitivity, limits of detection. The analytical potential of the direct iodine (P)CVG-ICP-OES based on the PN/PTSC is demonstrated in the analysis of mineral water samples and simulated seawater standard reference material CRM-SW.

2. Experimental

2.1. Reagents and samples

High grade analytical reagent chemicals obtained from (POCh, Poland) were employed in the preparation of all the solutions. A standard stock solution of iodide (1000 mg L^{-1}) was prepared from potassium iodide. Working standard solutions were freshly prepared daily by serial dilution of the stock solution in water (for CVG mode) or with addition of appropriate volume of 99.5% v/v acetic acid to obtain iodide standard solutions in 3% v/v acetic acid for PCVG mode.

25 mmol L^{-1} solution of sodium nitrite was freshly prepared daily by diluting appropriate amount of NaNO_2 in 0.5 mol L^{-1} nitric acid. Mineral water samples were purchased in the local market and simulated seawater standard reference material CRM-SW was supplied from High Purity Standards (USA).

2.2. Instrumentation

All the measurements were made with an inductively coupled plasma optical emission spectrometer ICP-OES Integra XL (GBC Scientific Equipment, Australia). Iodine generation was accomplished in a continuous flow mode by modification of liquid sample introduction system. In first system, the sample and acidified oxidant solutions were

simultaneously pumped into the CVG-PN-PTSC system and mixed in a T-shaped glass tube where a reaction occurred to produce the iodine vapor. The length of PTFE tubing connecting the T-shaped mixing chamber and pneumatic nebulizer was 0.15 m. The waste solution was rapidly removed from the chamber to drains with the use of additional peristaltic pump.

The second system accomplished photochemical generation prior to nebulization in the PTSC by pumping an analyte solution through PTFE tubing irradiated by a UV lamp. The 1.1 mm i.d. wide and 1.5 m long PTFE tubing was wrapped around a low-pressure 11 W, UV-C lamp (Philips). The length of PTFE tubing between the UV lamp and pneumatic nebulizer was 0.5 m. This PCVG mode is a simpler system than the CVG mode, and almost does not need to be changed to work in typical ICP arrangement of sample introduction system. A sample nebulization setup, consisting of a conical nebulizer AR40-07 and an Isomist Module LOL equipped with standard cyclonic spray chamber V2 (Glass Expansion Pty. Ltd., Australia) for both controlling and monitoring spray chamber temperatures between $-10 \text{ }^\circ\text{C}$ and $+60 \text{ }^\circ\text{C}$ to provide the optimum conditions for the analyte transport efficiency was used. The system requires similar plasma operating conditions to conventional nebulizer-spray-chamber arrangements. A schematic diagram of both CVG- and PCVG-PN systems with the PTSC is shown in Fig. 1.

Additionally, a standard setup with the same conical nebulizer for conventional solution nebulization was used as the reference system. Typical operating conditions for (P)CVG-PN-PTSC-ICP-OES are given in Table 1.

2.2.1. Wavelength selection

Conventional spectrometric systems present several difficulties in the spectral region below 200 nm due to absorption of emitted radiation. Thus, purging the optical system with nitrogen is necessary. Moreover, spectral interferences are observed for the most sensitive iodine lines at 178.3 nm and 183.0 as well as shorter linear dynamic ranges in the case of lines in the VUV region. The iodine line at 206.15 nm can be potentially interfered by spectral lines of Bi, Cr and Zn, however, it was not the case in this study. Only the Zn line was observed, but I and Zn lines were resolved. In this study, emission intensities of iodine were measured at 206.15 nm with an ambient air operating monochromator.

2.3. Procedure

2.3.1. Chemical vapor generation mode

The aqueous sample and the acidic oxidant solution (25 mmol L^{-1} NaNO_2 in 0.5 mol L^{-1} nitric acid) are delivered separately by the peristaltic pump and mixed just before entering the conical nebulizer. Both aerosol and iodine vapors are generated in the PTSC at $40 \text{ }^\circ\text{C}$ and then iodine is determined by ICP-OES.

2.3.2. Photochemical generation mode

The iodide solution containing 3% v/v acetic acid is pumped through the PTFE tubing of the 1.5 m-long reaction coil and subjected to UV

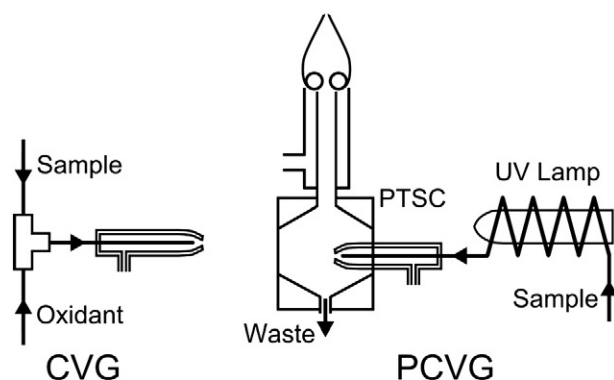


Fig. 1. The schematic diagram of CVG- and PCVG-PN-PTSC-ICP-OES experimental setup.

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