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Correlation between the Gas Temperature and the Atomization Behavior of Analyte Elements in Flame Atomic Absorption Spectrometry Estimated with a Continuum-light-source Spectrometer System



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ABSTRACT

In flame atomic absorption spectrometry (FAAS), the gas temperature for two types of the gas compositions, which was estimated based on a two-line method by using a simultaneous multi-wavelength spectrometer, on which a line pair of ruthenium, Ru I 372.692 nm and Ru I 372.803 nm having different excitation energies, was measured at the same time. Also using the spectrometer system, the absorption signals of both iron and ruthenium, whose oxides had different thermodynamic properties: the latter oxide was decomposed much more easily than the former one, were investigated with a nitrous oxide - acetylene flame, in comparison with an air - acetylene flame. The fuel/oxidant ratio of both the flames as well as the height of the optical path was varied as an experimental parameter. The atomization behavior of iron and ruthenium, which could be deduced from a variation in their absorption signals, was considered to be dependent not only on the gas temperature but on reducing atmosphere of the flame gas, which might be attributed to reducing radicals in a fuel-excess flame consisting of nitrous oxide - acetylene flame, a broader optical path having a constant and higher temperature was obtained, thus contributing to formation of analyte atoms with a stable atomization efficiency and eventually to better precision in the analytical result in FAAS.

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1. Introduction

Flame atomic absorption spectrometry (FAAS), which is suitable for quantification of elements in solution samples, has been extensively employed for in various application fields: for instance, metal, chemical and food industries, biological samples, and environmental assessment [1,2]. Also, FAAS has been employed as an analytical method for appropriate validation in Japanese Industrial Standards (JIS); for instance, general rules for atomic absorption spectrometry [3] and atomic absorption analysis of iron and steels [4]. The advantages of FAAS are that several metallic elements, such as zinc and cadmium, can be determined at a sub-ppm concentration level by using a relatively inexpensive apparatus and that it is easy to conduct the measurement and to maintain it with a low running-cost [2]. On the other hand, it has also several disadvantages: the dynamic range in acceptable concentration is narrow (1-2 orders of the magnitude), compared to ICP atomic emission spectrometry, and it may suffer from various types of interference against the accurate quantification, which always occur in a relatively low-temperature atomization source using a chemical flame. Furthermore, only an absorption line of a particular element can be measured and thus a multi-elemental analysis cannot be conducted, when a conventional spectrometer system equipped with a hollow cathode lamp as the primary light source is employed.

The atomization behavior of an analyte element should be understood so that the experimental conditions for FAAS, such as selection of the flame and a mixing ratio of the fuel/oxidant gases, can be optimized, which may be dependent of the thermal and chemical properties of the analyte element. For this purpose, gas temperature of the flame is a useful parameter, because it closely affects decomposition of salts and oxides, and then formation of the analyte atoms occurring in the flame. The gas temperature, which is defined by an average kinetic energy of flame gas species [5], can be measured by a spectrochemical method, as based on how probe atoms are distributed among their energy levels in an atomization/ excitation source.

A two-line method, where a characteristic temperature can be estimated from atomic absorption for two spectral lines having different lower energy levels, provides the excitation temperature, and it corresponds to a gas temperature (kinetic temperature) under local thermodynamic equilibrium (LTE) [5]. A chemical flame is a typical case in which the excitation/de-excitation well follows the LTE condition, based on frequent exchanges of the kinetic energy through collisions of gas particles. Therefore, we can consider that an excitation temperature determined by the two-line method represents a gas temperature of a chemical flame. Several papers have been published on the gas temperature of a chemical flame or a graphite furnace (GF) in atomic

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absorption spectrometry (AAS) obtained with the two-line method, providing basic information on how analyte atoms are produced and atomized in the graphite furnace [6–13]. We also reported on temporal variations in the gas temperature with the progress of an atomization stage in order to discuss the atomization process of cadmium and tellurium [14], and further discussed on a difference in the gas temperature when iron and nickel were employed for probe elements for the temperature determination in GF-AAS [15].

Recently, a new-type apparatus for AAS was commercialized and began to be employed [16], where a spectrometer system comprised a xenon-lamp continuum-light source and an Echelle-type spectrograph to conduct a sequential multi-wavelength detection, which thus enabled a simultaneous measurement of several absorption lines over a certain wavelength range. Due to its performance, a measurement for the twoline method can be easily conducted, where two absorption lines having close wavelength values are simultaneously measured and then the gas temperature can be calculated from their absorbances. This procedure is similar to an internal standard method for FAAS, by using an appropriate pair of an analytical line and an internal standard line to correct any drift of the absorption signals, as described in our previous papers [17,18]. Several papers reported on practical analysis of food [19] and environmental samples [20] when a multi-wavelength continuum-light-source spectrometer was employed; however, few fundamental studies of FAAS have been found in its application, while a strict single-beam measurement is required to estimate the gas temperature accurately.

This study focused on a different behavior of the absorption signals between iron and ruthenium in FAAS, when an air - acetylene and a nitrous oxide - acetylene flame were employed as the atomization source. These elements were selected from two groups of element having different chemical properties: 3d-transition metals and platinum-type metals. It should be concerned that the 3d-transition metals such as iron generally form more stable oxides, which may be harder to be reduced and decomposed, than the platinum-group elements in such chemical combustion flames. Variations in the gas temperature, which was estimated using a pair of ruthenium atomic lines, were compared between two different oxidants of the flame against the oxidant/fuel mixing ratio as well as the observation height, probably affecting the atomization efficiency of both iron and ruthenium.

2. Experimental

2.1. Apparatus and the operating conditions

A multi-wavelength atomic absorption measurement system (ContrAA-700, Analytik Jena Corp., Japan) was employed, equipped with a chemical flame atomizer [16]. This apparatus consisted of a high-intensity short-length xenon lamp as a continuum primary radiation source, covering the wavelength range of 190-900 nm, and a high-

resolution Echelle-type monochramator combined with a CCD detector having 200 pixels, as illustrated in Fig. 1. A wavelength window of 0.2 -0.5 nm, which is dependent on the wavelength range, can be simultaneously recorded at an effective spectral resolution of ca. 0.01 nm (full-width at the half maximum of an absorption peak). The chemical flame was generated with gas mixtures of air - acetylene or nitrous oxide - acetylene, where a length of the burner slot was 100 mm for the former and 50 mm for the latter. The mixing ratio of these gas mixtures was varied as an experimental parameter, as will be described later. A conventional pre-mixing-type spray chamber was employed to introduce sample solution into the burner. A background correction using the continuum light source was conducted in each spectrum through the measurement program. The beam from the primary radiation source passed above the burner slot at a height of 4 - 16 mm, which was also varied as an experimental parameter. The absorption values for each spectral line were recorded and analyzed statically on a personal computer through repeated measurements.

2.2. Reagents and sample preparation

The following stock solutions of analyte elements were made:

The following method to prepare test solution of ruthenium was selected to obtain large amounts of the test solution with a low cost. The stock solution was prepared by dissolving 1g of anhydrous ruthenium chloride (III) (Strem Chemicals Inc., USA) with 2.4 M-hydrochloric acid solution, heated on a hot plate. After cooling, the precipitates was removed with a 5C filter paper and the concentration of ruthenium was determined by ICP atomic emission spectrometry with a standard solution of ruthenium. A stock solution of 0.742 gdm⁻³ was obtained. The objective of this study is not quantitative determination of ruthenium, and thus the following result and discussions would not be affected by the purity and assay of ruthenium in the test solution.

A stock solution of 5.0-g/dm³ iron was prepared by dissolving 1 g of high-purity iron having a purity of 99.99 % (Toho Zinc Corp., Japan) with a 0.025-dm³ mixture of 2.4 M-hydrochloric acid and 5.6 M-nitric acid on a hot plate completely. After cooling, it was further diluted with 0.7 M-nitric acid to a volume of 0.2 dm³.

Working solutions for various concentrations of each element were prepared by diluting with de-ionized water or hydrochloric acid solution, just before the measurement. Hydrochloric acid and nitric acid were purchased from Nacalai Tesque Corp. (Special-grade reagent).

3. Results and Discussion

3.1. Gas temperature measurement using a pair of ruthenium atomic lines

The principle of the two-line method has been already described elsewhere [14]. This method is based on the difference in the number



Fig. 1. Schematic diagram of the measuring system.

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