

Investigation of Spectral Interference between Uranium and Iron in the Optical Emission Spectrometric Technique

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1. Introduction

Uranium is a naturally occurring element, which is abundant in environmental medium (e.g., approximately 3 mg/kg in soil and sediment). In addition, anthropogenic uranium also can be released into the environment by accident or human activities. It is well known that uranium is chemically toxic and can cause severe internal radiation exposure owing to alpha decay. Therefore, it is important to evaluate and determine the uranium levels in the environment. Among the various methods for uranium determination (e.g. alpha spectrometry, inductively coupled mass spectrometry, gamma spectrometry), the inductively coupled plasma optical emission spectrometry (ICP-OES) comes out due to its high sensitivity and multi-element detection ability. [1] However, due to complex emission lines from elements in the spectral field, the presence of other metallic ions can cause occasional severe spectral interferences.

Meanwhile, iron is one of the most abundant elements in the earth's crust. Concentrations of iron in soil and sediment have been estimated at an average of 2.6%. [2] As the concentration of iron present in the environment is much higher than that of uranium, it could make the spectral or chemical interferences in the analytical process. Thus, in this study, the degree of spectral interferences from the various iron levels in the sample aliquot were evaluated when ICP-OES was applied to determine the uranium concentration. Finally, for the determination of uranium in environmental samples using ICP-OES, an appropriate analytical line was suggested.

2. Methods and Results

High purity deionized water (>18 MΩ) from MilliQ Element (Millipore) and analytical grade nitric acid (Merck, Germany) were used for making 0.5 M nitric acid. Uranium (Inorganic Ventures, USA) and iron (AccuStandard, USA) standard solutions were appropriately diluted with 0.5 M nitric acid. Determination of U and Fe concentrations were performed by ICP-OES (SPECTRO BLUE, SPECTRO Analytical Instruments GmbH, Germany). The instrumental specifications and experimental parameters were summarized in Table 1.

Table 1. The instrumental specifications and experimental parameters

Wavelength range	165 -770 nm		
Detector	15 linear CCD array detectors		
Plasma torch	Demountable glass torch		
Spray Chamber	Scott Spray Chamber, Double Pass		
Nebulizer	V-Groove nebulizer		
Plasma power	1200 V	Plasma view	Axial
Coolant flow	13.00 L/min	Auxiliary flow	0.80 L/min
Nebulizer flow	0.65 L/min	Total time	17 s

In order to evaluate the degree of spectral interference, three different emission lines (385.958 nm, 409.014 nm, and 279.394 nm) for uranium were identically investigated. To determine the spectral interference of uranium intensity affected by the iron concentration level, sample aliquots with 0.5, 2, 4, and 8 mg·L⁻¹ of uranium concentration were prepared and then pure iron was spiked in the sample aliquot until Fe concentration was 0.5, 1, 15, 20, 150 and 500 mg·L⁻¹, respectively. The prepared samples were quantified by measuring 10 times each using ICP-OES.

3. Results and discussion

Figure 1 shows the calibration results for each emission line of uranium. At the 385.958 nm, the sensitivity (counts/concentration) for uranium was the highest, followed by 409.014 nm and 279.394 nm. The detection limit of 385.958 nm was the lowest at 0.0808 mg·L⁻¹ and 0.106 mg·L⁻¹ at 409.014 nm and 0.155 mg·L⁻¹ at 279.394 nm.

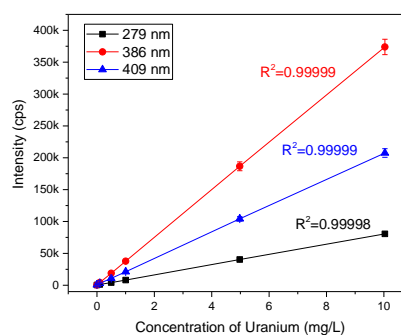


Fig. 1. Calibration results for three different uranium emission lines.

Spectral overlapping with uranium and iron for the respect emission lines in ICP-OES were shown in Fig. 2. In terms of sensitivity for the uranium, use of the 385.958 nm could be a good choice. However, as shown in Figure 2(a), the uranium signal at the 385.958 nm line clearly overlaps the two signals of iron (e.g., 385.991 nm and 385.921 nm). The intensities of Fe at those lines were 160,000 and 500, respectively. Thus, because of its high emission intensity of Fe, if the iron is present in sample aliquot about two times higher than uranium, the determination of uranium is not possible at 385.958 nm peak. In this experiment, determination at the 385.958 nm line could not be achieved for the samples, which the Fe/U ratio was more than 2.

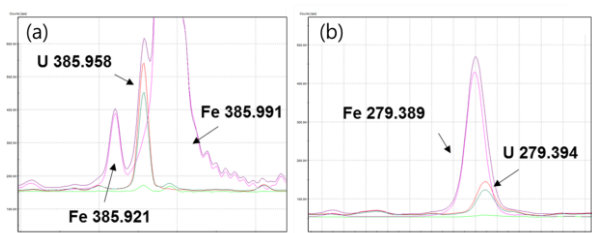


Fig. 2. Spectral overlapping with uranium and iron. (a) Uranium line of 279.394 nm, (b) line of 385.958 nm

As shown in Fig. 2(b), the signal at 279.389 nm of uranium was completely superimposed on the signal with 279.394 nm of iron. Although the emission intensity of Fe at the 279.394 nm is 40, the difference between the two emission lines was too small ($\Delta\lambda=0.005$ nm). Due to this spectral characteristics, the peaks could not be resolved. Therefore, 279.389 nm of iron was regarded as a signal of uranium, and it brought out higher relative error depending on Fe/U ratio (Fig. 3(a)).

In case of 409.014 nm spectral emission line of uranium, there was no obvious tendency for the relative error of analytical results for the all of sample aliquot with iron (fig. 3(b)). There are some emission lines (409.007 nm, 409.095 nm, 409.155 nm, and 409.246 nm) of iron molecule, which have similar wavelength with 409.014 nm line of uranium, but it was judged that it does not affect to 409.014 nm line due to its extremely low emission intensity (≈ 0). As shown in Fig. 3(b), the relative errors of analytical results for all cases up to 1000 of Fe/U ratio fell within 5 %, except for 0.5 $\text{mg}\cdot\text{L}^{-1}$ of uranium samples. For the low concentration level of U, some measurement error could be considered due to the detection limit ($0.106 \text{ mg}\cdot\text{L}^{-1}$).

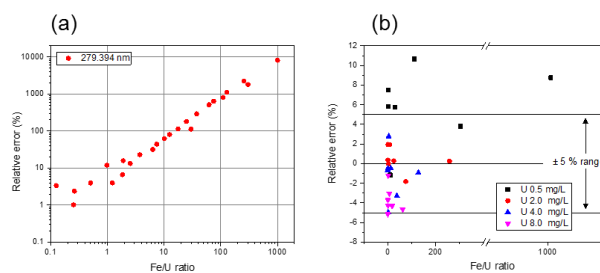


Fig. 3. Distribution of relative error (%) with Fe / U ratio in the line of 279.394 nm (a) and 409.014 nm (b).

4. Conclusions

To understand the spectral interference of uranium with iron using ICP-OES, comprehensive experiment on the various concentration levels was carried out. It was confirmed that, 407.014 nm of uranium was not influenced by the coexistence of iron among the effective emission lines (e.g., 385.958 nm, 409.014 nm, and 279.394 nm). Therefore, when analyzing samples containing iron, especially environmental samples, the use of 407.014 nm emission line will give accurate results even if the detection limit is slightly higher. The analytical results using 385.958 nm and 279.394 nm could be inaccurate depending on the iron content. Contrary, for the determination of U in purified or Fe-free samples, these two lines could be good choice with high sensitivity.

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