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Atomic Absorption Spectrometer, AA-7000

Application News

The Optimization of Conditions to Analyze Neodymium (Nd) on C_2H_2-N_2O Flame

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User Benefits

- Dynamic range and analytical conditions for analysis of neodymium using AAS
- Identification of factors affecting sensitivity on high temperature burner.
- Effects of ionization suppressant on flame of AAS.

■ Introduction

Neodymium (Nd) that is one of the rare earth elements belonging to the lanthanoid is used as a raw material for strong permanent magnets, lasers, optical glass, and special rubber in various industries. There is not much known information about Nd analysis using atomic absorption spectroscopy (AAS), because recently inductively coupled plasma atomic emission spectroscopy (ICP-AES) is mainly used to analyze trace rare earth elements. According to known references, Nd can be analyzed at hundreds of ppm levels using AAS. In 1973, Hiroshi Sunahara (Japan) conducted a study using AAS to analyze Nd, Eu, Gd, and Er under different conditions of inorganic acids, measurement methods, and interfering substances, which also showed a similar level of analytical sensitivity.[1] However, since conventional instrument such as AAS are also improving their performance little by little, it is necessary to reconfirm the dynamic range and analytical conditions in the device of the recent specification. This application news confirm the sensitivity and dynamic range to analyze Nd using AAS, and introduce the optimized conditions for quantitative analysis of trace Nd. The optimized analytical conditions using the AA-7000 model (Shimadzu Co., Ltd., Japan) in Fig. 1 were determined while changing several factors affecting absorbance of Nd.

Fig. 1 AA-7000 System

■ Preparation for Analysis & Variables

It was confirmed from several papers and application data that a high-temperature burner using C_2H_2-N_2O is commonly used for the analysis of Nd and the sensitivity was the best at 492.5 nm. Therefore, the same burner type and wavelength were used in this test. The slit width was based on 0.2 nm than 0.7 nm in consideration of noise rather than sensitivity.

The other factors and ranges changed for optimization of the analysis conditions are as follows.

1. Fuel gas (C_2H_2) flow rate : (6.5 - 8.0) L/min
   & Burner height : (5 - 11) mm
2. The kind of solvent : (0 - 5) % HCl or HNO_3 solution
3. Ionization suppressant added : (0 - 0.4) % potassium

The Nd standard solution was prepared by diluting to the desired concentration using a commercial product of 10 000 mg/L (AccuStandard®). For nitric acid and hydrochloric acid, Chemitop's electronic grade (EP-S) reagent was used, and Sigma-Aldrich's potassium chloride (KCl, ACS grade) reagent was used as an ionization suppressant. All water used in the preparation of the solution was deionized water of ASTM type I.

■ Analysis Result by Conditions

1. Fuel gas (C_2H_2) flow rate and burner height

The temperature of the flame depends on its height and the flow rate of acetylene gas, which consequently determines the atomization efficiency of the element to be analyzed.. While changing the flow rate of fuel gas to 6.5, 7.0, 7.5 and 8.0 L/min, measurements were conducted at heights of 5, 7, 9 and 11 mm from the burner head. The difference in absorbance was confirmed by preparing a high concentration test solution of Nd 500 mg/L, and the results are shown in Fig. 2. Based on this, the optimal analytical conditions were determined as the flow rate of acetylene was 7.0 L/min and the observation height of the flame was 11 mm.

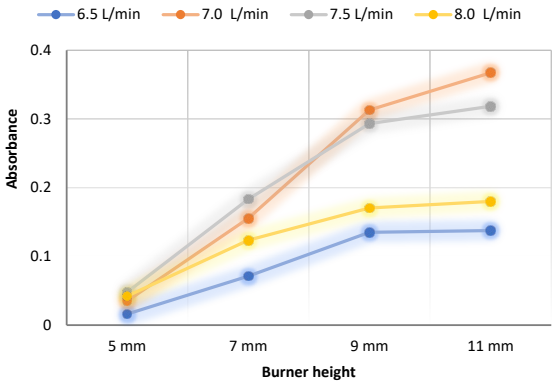

Fig. 2 Effect of acetylene flow rate and burner height on absorbance of Nd (500 mg/L Nd solution)

2. The kind of solvent (added Inorganic acids)

Inorganic acids such as hydrochloric acid and nitric acid are added in small amounts so that metal ions can stably exist in the solution, and various inorganic acids are also used for dissolution of main metals or organic decomposition. So, it was confirmed how the absorbance of Nd changes depending on the kind and concentration of the inorganic acid.

Fig. 3 shows the change in the absorbance of the 500 mg/L Nd solution when nitric acid and hydrochloric acid, which are representative inorganic acids, were added at concentrations of 0, 1, 3, and 5 %, respectively. At this time, other analytical conditions such as gas flow rate and burner height were based on the previously determined. The pattern of changes with acid concentrations were significantly different in hydrochloric acid and nitric acid. The absorbance of Nd increased as the concentration of nitric acid increased, but the absorbance did not significantly increase when the concentration of nitric acid was 3 % or more, so the 3 % nitric acid solution was determined as the optimal solvent.

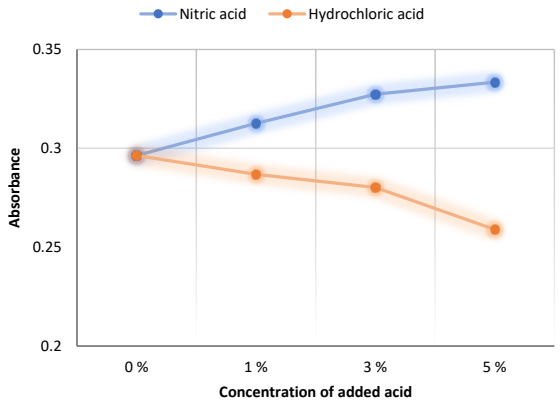

Fig. 3 Effect of inorganic acids on absorbance of Nd

3. Ionization suppressant added

The atomized elements on the acetylene flame of AAS can be ionized at different rates depending on their ionization energy. Ionization can occur relatively easily in high temperature burners using C_2H_2-N_2O, which can lead to decreased sensitivity in AAS, which mainly uses atomic line.

Therefore, the addition of alkali elements such as Na, K, Rb, and Cs with low ionization energy as ionization suppressant from 0.1 % to 1 % can reduce ionization of the analytical elements and increase absorbance of atomic lines. In addition, precise results can be produced by reducing the fluctuation of the ionization rate affected by the matrix.

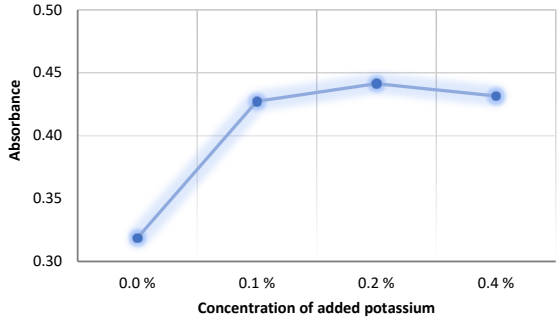

Fig. 4 Effect of ionization suppressant on absorbance of Nd

Accordingly, under the previous determined analytical conditions, KCl solution was added to the 500 mg/L Nd solution to confirm whether the absorbance increased. As shown in Fig. 4, it can be seen that the absorbance increased after the addition of potassium, but there was little change above 0.1%. In general, ionization suppressant are not necessarily required, so this result was used only as useful information for optimizing conditions.

■ Linearity under optimized condition

Based on the above test results, the optimized conditions for analyzing Nd on the C_2H_2-N_2O flame were determined as shown in Table 1.

Table 1 Optimized analytical conditions for Nd (AA-7000)

Wavelength	: 492.5 nm
Gas & burner type	: C_2H_2-N_2O (High-temp burner)
C2H2 gas flow	: 7.0 L/min
N2O gas flow	: 11.0 L/min
Slit width	: 0.2 nm
Burner height	: 11 mm
Solvent (matrix)	: 3 %(w/v) Nitric acid solution
Ionization suppressant	: 0.1 %(w/v) Potassium solution

Nd standard solutions for calibration curve were prepared in a concentration range of (20 - 200) mg/L to confirm linearity in the range of (0.0044 - 0.4) abs. The standard solutions were prepared with a 2 % nitric acid solution, and one more set was prepared with 0.1 % potassium added to the same solution.

Table 2 Absorbance of Nd standard solution (3 % nitric acid) for calibration curve

Conc.(mg/L)	20	50	100	200
Absorbance	0.0055	0.0157	0.0390	0.1018

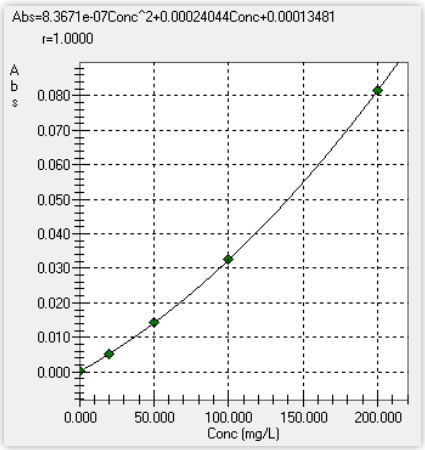

Fig. 5 Linearity of Nd in 3 % nitric acid solution

The measurement results of the solution without potassium were shown in the shape of a secondary curve as the change in absorbance depending on the concentration decreased in the low concentration range as shown in Fig. 5 and Table 2. The calibration curve of solution prepared by adding 0.1 % potassium shows a good linearity with a correlation coefficient (*R*) of 1.0000 in the shape of a straight line as shown in Fig. 6 and Table 3. Calculating the concentration expected to have an absorbance of 0.0044 (1 % absorption) by the calibration curve equation of the above result is about 4 mg/L. Therefore, it may be confirmed that Nd of several ppm levels may be measured.

Table 3 Absorbance of Nd standard solution (3 % nitric acid + 0.1 % potassium) for calibration curve

Conc.(mg/L)	20	50	100	200
Absorbance	0.0220	0.0538	0.1102	0.2223

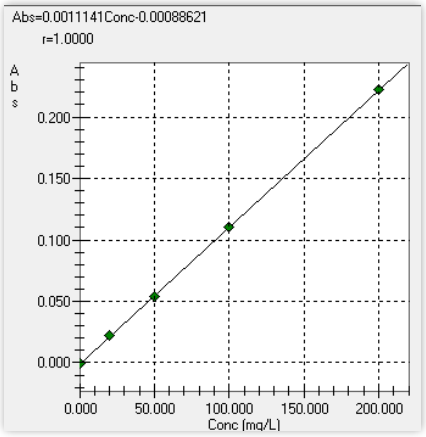

Fig.6 Linearity of Nd in 3 % nitric acid and 0.1 % potassium solution

■ Conclusion

This newsletter provides the guidance for analyzing the content of neodymium, a lanthanoid rare earth metal, using AAS.

Sensitivity and linearity were verified under several different conditions to determine the optimal conditions for analyzing Nd on a high-temperature burner using C_2H_2-N_2O gas. As a result, the analysis conditions in Table 1 were optimally determined, and it was confirmed that the sensitivity and linearity were good in the range of (20 - 200) mg/L. Based on these results, it was confirmed that the content of Nd of several ppm or more in the test solution can be analyzed using AAS.

■ Reference

1) Toshio ISHIZUKA, Yoshinori UWAMINO and Hiroshi SUNAHARA. (1973). Determination of neodymium, europium, gadolinium and erbium by flame emission and atomic absorption spectrometry. JAPAN ANALYST Vol. 22: 1450 – 1455.

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