



## N,N'-Bis(2-Aminobenzoyl)ethylenediamine as a Reagent for the Spectrophotometric Determination of Neodymium(III)

K. GIRISH KUMAR<sup>1</sup> and R. MUTHUSELVI<sup>2,\*</sup>

<sup>1</sup>Department of Applied Chemistry, Cochin University of Science & Technology, Cochin-682 022, India

<sup>2</sup>Department of Chemistry, Sri Meenakshi Government College for Women (Autonomous), Madurai-625 002, India

\*Corresponding author: E-mail: muthuselvi03@yahoo.co.in

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A simple, direct, rapid and cost effective spectrophotometric method is described for the determination of neodymium(III) in pure form, natural samples and industrial effluents. When neodymium(III) complexes with N,N'-bis(2-aminobenzoyl)ethylenediamine in the pH range of 6.0-7.5, it shows an absorption maxima at 350 nm and obeys Beer's law in the concentration range of 3.04-13.17  $\mu\text{g mL}^{-1}$ . The absorbance was found to increase linearly with increasing concentration of neodymium(III); the apparent molar absorptivity of the species is  $0.214 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ . The validity of the described procedure was assessed. Statistical analysis of the result has been carried out revealing high accuracy and good precision. The proposed method has been successfully applied to the determination of Nd(III) in natural samples and industrial effluents without much interference from foreign ions.

**Key Words:** Neodymium(III) determination, N,N'-Bis(2-aminobenzoyl)ethylenediamine, Spectrophotometry, Industrial effluents.

### INTRODUCTION

Neodymium plays an important role in agriculture, medicine, paints, textiles, leather industry and in nuclear applications. In view of the importance of neodymium, its analysis in micro amounts becomes highly essential. A few methods are available for determination of rare earths. In this context, development of a sensitive method to determine rare earth ion is of prime importance. In continuation of the development of suitable methods for the determination of metals, especially in natural samples<sup>1-3</sup>, a sensitive and selective method has been developed for the determination of neodymium(III) in its pure form, natural samples and industrial effluents. Though a number of spectrophotometric methods have been reported for the determination of neodymium<sup>4-6</sup>, however, no reagent exhibits outstanding performance for direct spectrophotometric determination of neodymium(III) in aqueous medium. The use of N,N'-bis(2-aminobenzoyl) ethylenediamine (BABEN) as a reagent for the sensitive and selective determination of neodymium(III) is investigated in the present work.

### EXPERIMENTAL

A Shimadzu UV-visible spectrophotometer with 1.0 cm quartz cells has been used for absorbance studies and a Systronics pH meter with combined electrode has been used for the pH studies.

**N,N'-Bis(2-aminobenzoyl)ethylenediamine (BABEN):** Isatoic anhydride (0.1 g) was dissolved in boiling methanol (100 mL) and mixed with ethylenediamine (2 mL) to get the reagent as reported<sup>7</sup>.

**Standard neodymium(III) solution:** Prepared by dissolving neodymium chloride (0.22 g) (obtained from Indian Rare Earths Ltd., Kerala) in distilled water and diluting it to 250 mL. This solution was standardized by conventional method<sup>8</sup>. Working solutions were prepared by appropriate dilution of the stock solution.

Buffer solution (pH 6.0-7.5) was prepared by mixing disodium hydrogen orthophosphate (0.01 M) and sodium hydrogen orthophosphate (0.01 M).

**General procedure:** To an aliquot of the sample solution (76-329  $\mu\text{g/mL}$ ), 2 mL of buffer solution (pH 6.0-7.5), 5 mL of ethanol, 2 mL of (2 % w/v) hydrochloric acid, 5 mL of ( $1 \times 10^{-2}$ ) reagent (BABEN) are added. The solutions were diluted to 25 mL to get a final concentration of 3.04-13.17  $\mu\text{g/mL}$ . The absorbance of the resulting violet coloured solution was measured at 350 nm against the reagent blank. A calibration curve has been prepared.

### RESULTS AND DISCUSSION

**Absorption spectra:** The absorption spectra of BABEN and the neodymium(III)-BABEN complex in disodium hydrogen orthophosphate and sodium hydrogen orthophosphate buffer (pH 6-7.5) solution were recorded against water and

TABLE-1  
DETERMINATION OF NEODYMIUM(III)

BABEN method				Perchlorate method		
Nd(III) taken ( $\mu\text{g mL}^{-1}$ )	Nd(III) found ( $\mu\text{g mL}^{-1}$ )	Recovery (%)	Error (%)	Nd(III) found ( $\mu\text{g/mL}^{-1}$ )	Recovery (%)	Error (%)
3.04	3.05	100.3	0.3	3.01	99.0	1.0
5.06	5.05	99.8	0.2	5.01	99.0	1.0
7.09	7.10	100.1	0.7	7.05	99.4	0.6
9.12	9.13	100.1	0.8	9.03	99.0	1.0
11.14	11.16	100.2	0.7	11.09	99.5	0.5
13.17	13.20	100.2	0.2	13.09	99.4	0.6

reagent blank, respectively. The complex shows an absorption maximum at 350 nm. Hence, all measurements were made at 350 nm.

**Effect of pH:** Keeping the concentration of neodymium(III) constant, the complexation reaction between neodymium(III)-BABEN was studied over the pH range 1.0-10.0. A constant and maximum value of absorbance was obtained in the pH range of 6-7.5. Hence, the pH was fixed at 6.0-7.5 for further analysis, by use of disodium hydrogen orthophosphate buffer solution.

**Analytical characteristics:** The system obeys Beer's law over the concentration range  $1.01\text{-}16.24 \mu\text{g mL}^{-1}$  of neodymium(III). The determinations of neodymium(III) were carried out in the concentration range of  $3.04\text{-}13.17 \mu\text{g/mL}$  and the results of the determinations are presented in Tables 1 and 2. The apparent molar absorptivity was found to be  $0.214 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ . The method was compared with established per chlorate method<sup>8</sup> and the results reveal that the developed method is highly accurate and precise. The standard deviation and co-efficient of variation calculated for six replicate measurements are in good agreement with the standard method. It was found that the order of addition of reactants has no effect on the formation of the complex.

TABLE-2  
STATISTICAL COMPARISON

Method	Av. error* (%)	SD*	CV* (%)
N,N'-Bis(2-aminobenzoyl) ethylenediamine (BABEN)	0.55	0.17	0.17
Perchlorate	0.78	0.24	0.24

\*6 replicates.

**Stability of the complex:** The formation of neodymium-BABEN complex is instantaneous and the absorbance of the violet coloured complex remained constant for not less than 24 h.

**Effect of diverse ions:** In order to establish the applicability of the method, the effect of foreign ions on the determination of neodymium(III) was systematically studied. In this study, various amounts of ionic species were added to  $4.05 \mu\text{g/mL}$  of neodymium(III) in buffer solution taken in a 25 mL standard flask and colour was developed as outlined for general procedure. The tolerance limits are shown in Table-3. The tolerance was taken as the amount of foreign ions required to cause a  $\pm 2\%$  error in absorbance. The main interference was shown by manganese(II) which was effectively masked with potassium iodide solution (5 mL, 1% w/v). The results on closer examination show that all the studied anions and most of the cations do not interfere with the determination of neodymium(III).

TABLE-3  
EFFECT OF FOREIGN IONS: AMOUNT OF NEODYMIUM(III) TAKEN- $4.05 \mu\text{g mL}^{-1}$

Foreign ions	Tolerance limit ( $\mu\text{g mL}^{-1}$ )
Acetate, carbonate, bicarbonate, sulphate, sodium(I), calcium(II), ammonium(I) and lithium(I)	500
Citrate, oxalate, hydroxide, thiosulphate, mercury(II) and zirconium(II)	375
Iodide, fluoride, nitrate, cadmium(II), barium(II), magnesium(II) and potassium(I)	150
Chloride, cyanate, borate, molybdate, lead(II), zinc(II), iron(III), nickel(II) and chromium(III)	50
Phosphate, bromate and manganese(II)*	20

\*Masked with KI (5 mL, 1% w/v).

**Application:** The method was applied to the determination of neodymium(III) in natural water sample and industrial effluents.

**Water and effluent samples:** Water samples were collected from a borewell and effluent samples from a tannery and dyeing unit. All the samples were filtered before analysis and known amounts of neodymium(III) were added, as none of the samples contained any amount of neodymium(III). The samples were analyzed for neodymium(III) by the proposed method and the recoveries are shown in Tables 4 and 5. A close examination of the table reveals that the presently developed method can be applied for determination of neodymium(III) in these samples with a high degree of accuracy and precision.

TABLE-4  
DETERMINATION OF NEODYMIUM(III) IN ENVIRONMENTAL SAMPLE

Nd(III) added ( $\mu\text{g mL}^{-1}$ )	Nd(III) found ( $\mu\text{g mL}^{-1}$ )	Recovery (%)
3.04	3.05	100.3
5.06	5.05	99.8
7.09	7.14	100.7
9.12	9.19	100.8
11.14	11.22	100.7
13.17	13.15	99.8

## Conclusion

From the above discussions, it can be concluded that N,N'-bis(2-aminobenzoyl)ethylenediamine (BABEN) is a potential reagent for the spectrophotometric determination of neodymium(III). An exhaustive search through the literature revealed that only limited methods are available for the quantitative determination of neodymium. In view of this the developed method attains high importance as it is direct, sensitive, accurate, selective, specific, non-extractive, reproducible and simple to

TABLE-5  
DETERMINATION OF NEODYMIUM(III) IN EFFLUENT  
SAMPLE (DYEING UNIT AND TANNERY)

Tannery		Dyeing unit		
Nd(III) added ( $\mu\text{g mL}^{-1}$ )	Nd(III) found ( $\mu\text{g mL}^{-1}$ )	Recovery (%)	Nd(III) found ( $\mu\text{g mL}^{-1}$ )	Recovery (%)
3.04	3.03	99.7	3.02	99.3
5.06	5.05	99.8	5.04	99.6
7.09	7.10	100.1	7.11	100.3
9.12	9.11	99.9	9.09	99.7
11.14	11.15	100.1	11.17	100.3
13.17	13.16	99.9	13.18	100.1

analyze in a quantitative manner. The most important advantage of the method is that it is free of interference from iron(III). The results of the investigations were highly fruitful with a

considerably high value of molar absorptivity, accuracy and precision and it is hoped that this method can be suggested for the routine analysis of neodymium in free state, environmental sample and effluent samples.

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