# Spectrophotometric Determination of Cerium Using Azocalixarene Derivative in Geological Samples

Le Van Tan and Nguyen Thi Ngoc Le

***Abstract*—A simple, apid and sensitive spectrophotometric method has been proposed for determination of Cerium(IV) base on Azocalixarene drivertive, 5,11,17,23-tetrakis[(o-carboxyphenyl)azo]-25,26,27,28-tetrahydroxycalix[4]arene (1). This methode base on the formation of a orange color radical cation upon a reaction of azocalixarene with Cerium(IV) in the acidic solution having maximum absorbance at 515 nm (bathochromic shift of 150 nm). The Beer law in the range of $8.0\times10^{-6}$ to $3.0\times10^{-5}$ $mol.L^{-1}$ of cerium(IV) and the linear regression equation was determined to be: Absorbance (A) = $0.701\times C(10^{-5}$ $mol\ L^{-1}$ cerium(IV)) + 0.021, r = 0.997, n = 5. The proposed method has been successfully applied to the analysis Cerium in soil samples collected in the Kontum**

***Index Terms*—Cerium; azocalix[4]arene; spectrophotometrys.**

## I. INTRODUCTION

Cerium (Ce) is a rare earth metal, used as a getter in the metal industry, is used in nuclear reactor, in alloy with nickel and chromium; and in microwave devices, lasers, masers and in television sets, besides agriculture, forestry, animal husbandry and in environmental. Optimum concentration of some light rare earth elements including Ce are useful in agronomy as well as for environmental pollution assessment or in studies of biochemical processes [1], [2]; blurring and polished in glass industry, in the Welsbach gas, in carbon arc and as agents extracted liquid - liquid to separation of fission products from uranium fuel has consumed all. Cerium is one of the rare chemicals that can be found in houses in equipment such as colour televisions, fluorescent lamps, energy-saving lamps and glasses. The metal is used as a core for the carbon electrodes of arc lamps, for incandescent mantles for gas lighting. Cerium is used in aluminium and iron alloys, in stainless steel as a precipitation hardening agent, to make permanent magnets. Cerium oxide is part of the catalyst of catalytic converters used to clean up exhaust vehicles, it also catalyzes the reduction of nitrogen oxides (NOx) to nitrogen gas. All new cars are now equipped with catalytic conveter which consist in a ceramic or metal substrate, a coating of aluminium and cerium oxides and a layer of finely dispersed metal such as platinum or rhodium, which is the active surface. However, Cerium is dumped in the environment in many different places, mainly by petrol-producing industries. It can also enter the environment when household equipment is thrown away. Cerium will gradually accumulate in soils and water soils and this will eventually lead to increasing concentrations in humans, animals and soil particles. With water animals cerium causes damage to cell membranes, which has several negative influences on reproduction and on the functions of the nervous system. Cerium is known to cause health problems, such as mostly dangerous in the working environment, due to the fact that damps and gasses can be inhaled with air. This can cause lung embolisms, especially during long-term exposure. Cerium can be a threat to the liver when it accumulates in the human body. Thanks to its use in catalytic converters cerium is slowly improving the atmosphere of cities, or wherever diesel engines operate. Diesel engines emits particulates, carbon particles only a few micrometers in diameter. One way to reduce particulates emissions is trap them in a ceramic filter and then burn them off. If a little cerium oxide is added to the fuel itself, it will catalyse the burning of the particulates and eliminate them [1].

Cerium levels in the crust are 66 ppm ($6.10^{-3}\%$), usually in the form Xerit. So development of a simple, sensitive and reliable method for the determination of cerium(IV) is essential, it is even more interesting and significant to develop a new type of calixarene-based chromogenic reagent for selective detection of cerium(IV).

There has been much recent interest in the design of new stimuli responsive host systems. Of these, the developments of chromogenic or fluorogenic ionophores have been a very active research area in supramolecular chemistry, since these can be applied to many useful and important chemical sensor technologies.[3] Calixarenes, which are accessible by the base-catalyzed condensation of para-substitued phenols with formaldehyde, are ideal frameworks for the development of chromogenic ionophores in the molecular recognition of ionic species of chemical and biological interest since the incorporation of a suitable sensory group into the calixarene result in a tailored chromogenic receptor [4]. A variety of compounds based upon calixarene having nitrophenylazophenol, nitrophenol, indoaniline, indophenol, and azophenol functional groups have been successfully designed and exhibit a pronounced chromogenic behavior towards $Na^+$, $K^+$, $Cs^+$, $Ca^{2+}$, $UO_2^{2+}$, and even chiral amines. Shinkai et al. had synthesized calix[4]arene with a 4-(4-nitrophenyl)azophenol unit and three ethyl ester residues on the lower rim and observed the lithium sensitivity of the ionophore [5], [6]. Toth et al. also reported the sodium selectivity of related azophenol derivatives [7]. Some recent authors have also reported selectivity of reagent ions with $Th^{4+}$, $Pb^{2+}$ [8] - [11]. However, there has been no reports on

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Manuscript received June 22, 2011; revised August 14,2011

This work was supported in part by the Ho Chi Minh City University of Industry and Kontum of Pedagogical College.

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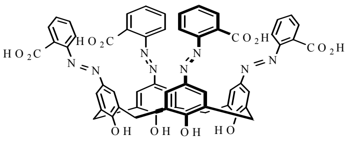
calixarene-based selective chromoionophores for detecting cerium(IV) ion up to now.

This necessitated the development of convenient and reliable analytical methods for the determination of cerium. Analytical techniques, such as *ICP-AES*, or electrothermal vaporization ICP-AES after HPLC separations, spectrofluorometry, stripping voltammetry reported for the determination of cerium are of high cost and may not be available at most laboratories.[12]

Therefore, the development of methods for measuring the level of this detrimental ion in the environment with high sensitivity and selectivity is highly desirable. Currently, amounts of lead are mainly determined using atomic absorption or emission spectrophotometry.[8]

Generally, the most chromogenic reagents based on the supramolecular calixarene were prepared by attaching a chromogenic moiety for the optical reading-out to the platform of calixarene, their ability to recognize and discriminate metal ions is one of the most remarkable features in previous reports, which makes them suitable as specific receptors. Similarly the target reagents 5,11,17,23-tetrakis[(o-carboxyphenyl)azo]-25,26,27,28-tetra hydroxycalix[4]arene (**1**) (Scheme 1) in this paper were synthesized through a diazocoupling reaction between the calix[4]arene and diazonium salts, and it displayed excellent chromogenic behavior towards cerium(IV). This motivated us to study it deeply, the following experiment did validate that the reagent exhibit good selectivity for cerium(IV).



**1**

Scheme 1

## II. EXPERIMENTAL

### A. Reagents and Materials

All chemicals and solvents were of analytical grade and used without further purification unless otherwise mentioned. Doubly distilled and degasified water was used throughout, *$HNO_3$ 65%, MeOH, EtOH, MeCN, $Pb(NO_3)_2$, $Cr(NO_3)_3$, $Ni(NO_3)_2$, $CH_3COOH$, NaOH, $Ce(NO_3)_4$, $KNO_3$, $Th(NO_3)_4.5H_2O$; $UO_2(NO_3)_2$, $La(NO_3)_3$, $Sm(NO_3)_3.6H_2O$ ....* were purchased from Merck.

Synthesis of (**1**) was prepared by Le Van Tan and colleagues [6].

### B. Apparatus

Uv/Vis Lamda 25 (Perkin Elmer), FT-IR: Bomen DA 8 spectrometer as KBr pellets (Canada) and $^1$HNMR spectroscopy: AVANCE- 600FT-MNR 600 MHz. (Germany). Fourier-transform Raman spectroscopy was carried out in the macroscopic mode with a specimen footprint of about 100 microns using a Bruker IFS 66 instrument with a FRA 106 Raman module.

The pH values were determined at room temperature with *211* Microprocessor *pH* Meter (Portugal) calibrated to standard buffer solutions of pH 6.96 and 4.00.

### C. UV Absorption Spectra Research General Procedure:

A stock solution of reagent **1** was prepared in double water. $Ce^{4+}$ stock solution were prepared by dissolving $Ce(NO_3)_4$ with double water. In various experiments, a diverse amount of $Ce(NO_3)_4$ crystal was added to a stock solution of reagent **1**, respectively, the final volume of the solution was adjusted to same volume with HAc–NaAc buffer solution after crystal being dissolved. Two minutes later the absorbance was measured at 515 nm in 1 cm quartz cell against water blank and the reagent blank, respectively, at room temperature.

## III. RESULTS AND DISCUSSION

### A. Absorption Spectra

Preparatory experiments for determining the optimum testing conditions have been done, the absorption spectra of the reagent **1** and its **1**–cerium(IV) complex under the optimum conditions are shown in Fig. 1. In the figure, curve 'a' and 'b' are the spectra of **1** and **1**–cerium(IV) complex against water blank, respectively. In the UV spectra of reagent **1**, the only absorption peak at 365 nm arised from $\pi\rightarrow\pi$ transition of the N - N bond (Fig. 1a), but in the spectra of **1**–cerium, the change was marked by shift to 370 nm and a decrease in intensity, with the concurrent appearance of a new conspicuous absorption peak at 515 nm (Fig. 1b), along with the incremental addition of cerium in a certain range the peak at 370 nm trailed off and the peak at 515 nm boosted up (not shown). As can be seen, the maximum absorption peak of the reagent **1** lies at 365 nm, whereas the absorption peak of the **1**–cerium(IV) complex is dissimilar to it.

The remarkable thing to note is the fact that the addition of cerium(IV) induced a weak red shift and a new absorption peak at 515 nm, exhibited an isosbestic point at 470 nm. This feature was expected to be utilized to detect the important ion cerium(IV), and our experiment in this work approved this point well. Similar observation can be found in literatures [9], [12] – [14]. The UV absorption spectra of the reagent **1** and cerium mixture was measured periodically, we found that the absorption peak at 515 nm of **1**–cerium(IV) appeared only 10 s after addition of cerium(IV) to the stock solution of the reagent **1**, the equilibrium was attained in ca. 2 min. From these characteristics, it is proposed that the topic water-soluble reagent **1** could be a significant chromogenic ionophores for the recognition of ion cerium(IV), this is very advantageous in the practical application of it.

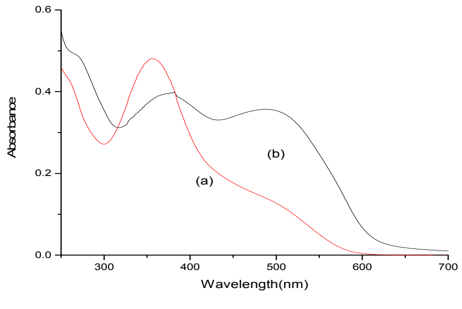


Fig. 1. Absorption spectra of **1** and its cerium complex at pH 6.0 conditions: (a) $2.0\times10^{-5}$ mol $L^{-1}$ of **1** solution against water blank;

(b) The solution containing $2.0\times10^{-5}$ mol $L^{-1}$ of **1** and $2.0\times10^{-5}$ mol $L^{-1}$ of $Ce^{4+}$ against water blank;

We have measured the IR spectrum of reagent and complex. FT-IR spectrum of the compound BAPC assume only weak in the range 3339-3200 $cm^{-1}$ respectively *vOH.* Low values indicate the -OH group related to internal links H molecules. *N* = *N* group located in the region 1600-1500 $cm^{-1}$, the infrared images of long BAPC compounds at 1145-1087 $cm^{-1}$. We can see these compounds do not exist in the form of keto-hydrazo in the solid state. FT-IR spectrum obtained new sheet in the region 649-594 $cm^{-1}$ demonstrated binding of Ce and oxygen. Spectrum appears only in the azo group reagents that do not appear in the complex. This shows that azo groups involved with metal complexes.

### *B. Effect of pH*

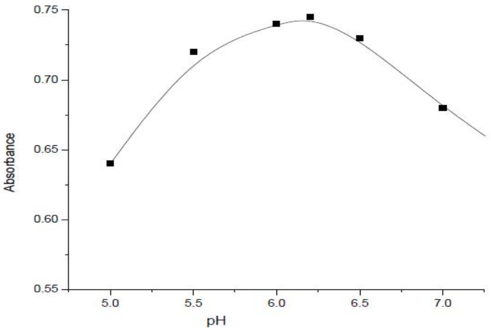


Fig. 2. Effect of pH on absorbance of complex at 515 nm ($3.0\times10^{-5}$ mol $L^{-1}$ of **1** and $2.5\times10^{-5}$ mol $L^{-1}$ of $ce^{4+}$).

Since cerium(IV) tends to form precipitate in weak acidic environments, the absorption spectra of 1-cerium(IV) complex was obtained in the pH range 5.0–7.0. To keep the other conditions optimum, appropriate concentration ($3.0\times10^{-5}$ $molL^{-1}$) of reagent 1 and mezzo cerium(IV) ion concentration ($2.5\times10^{-3}$ mol $L^{-1}$) were chosen. The effect of pH on the absorbance of 1–cerium(IV) complex at 515 nm is shown in Fig. 2. Different pH values were obtained by varying the relative amounts of *NaAc* and *HAc*, and confirmed by a digital pH-meter. As can be seen from Fig. 2, in the pH range 5.0–7.0, different acidities display prominent affect on the absorption peak intensity, the absorbance at 515 nm increase with acidity increment first, then reach the peak point and decrease last. The maximum absorbance of the complex is obtained in the pH range 6.0–6.5, so pH condition of 6.0 was assumed as the best and chosen for the following experiments.

### *C. Effect of Cerium (Iv) Concentration On Absorbance of Complex*

Under the optimum acidic condition (pH 6.0, the concentration of **1** is $2.0\times10^{-5}$ mol $L^{-1}$), the effect of the concentration of cerium(IV) on absorbance at 515 nm was checked. As can be seen from Fig. 3, the absorption of **1**–cerium(IV) complex at 515 nm show a continuous increase in intensity along with the augment of the cerium(IV) concentration in the range of $8.\times10^{-6}$ to $3.0\times10^{-5}$ mol $L^{-1}$, a plateau is reached until the cerium(IV) ion concentration increase to $2.5\times10^{-5}$ mol $L^{-1}$, and linear regression equation was determined to be: absorbance (A) = $0.701\times C(10^{-5}$ mol $L^{-1}$ cerium(IV)) + 0.021, r = 0.997, n = 5, the insert in the Fig. 3 shows the linearity of them.

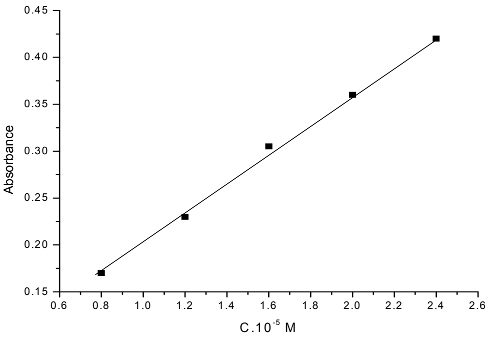


Fig. 3. Effect of $ce^{4+}$ concentration (pH 6.0, $3.0\times10^{-5}$ mol $L^{-1}$ of **1**).

### *D. Selectivity Towards Cerium(iv) Against other Metal Ions*

For the sake of studying the anti-jamming ability of reagent **1** selectivity towards cerium(IV), many metal ion were added to the reagent **1** aqueous solution and their UV spectra were measured. Upon interaction with aqueous $Ce(NO_3)_4$ solution, the reagent **1** experienced a marked absorption peak at 515 nm and the absorption peak at 365 nm experienced minor red shift, whereas addition of other metal ions to the solution of reagent **1** did not cause any conspicuous change, although their absorption intensities at 365 nm increased or decreased a little compared to the metal ion free reagent **1**. This phenomenon is very important, and showed that reagent **1** possess good selectivity towards cerium(IV) even $Mn^{2+}$, $Al^{3+}$, $K^+$, $Sm^{2+}$, $La^{3+}$, $Eu^{3+}$,… are present.

TABLE 1. EFFECT OF FOREIGN SPECIES [15.0 μG OF CE(IV) PER 25 ML]

| Ion | Tolerance limit (μg/25ml) | Ion | Tolerance limit (μg/25ml) |
|---|---|---|---|
| $Cu^{2+}$ | 1000 | $Al^{3+}$ | 1500 |
| $Cr^{3+}$ | 1700 | $Mg^{2+}$ | 5000 |
| $Zn^{2+}$ | 1300 | $Ca^{2+}$ | 6000 |
| $Mn^{2+}$ | 1450 | $La^{3+}$ | 1600 |
| $Cd^{2+}$ | 1750 | $MoO_4^{2-}$ | 1300 |
| $Ni^{2+}$ | 1100 | $Ti^{4+}$ | 1300 |
| $Pb^{2+}$ | 1000 | $Fe^{3+}$ | 1200 |
| $Zr^{4+}$ | 800 | $UO_2^{2+}$ | 800 |
| $Hf^{4+}$ | 1000 | $Sm^{3+}$ | 1600 |

### *E. Stoichiometry and Stability Constant of Complex*

The stoichiometry of the complex was determined by the Job's method of continuous variation. The result was described in Figure 4. From the experimental curve, we find that absorbance reaches the maximum value when the concentration ratio [BAPC]/[Ce]+ [BAPC] at 0.5. Thus, the stoichiometric ratio of complex is 1:1.

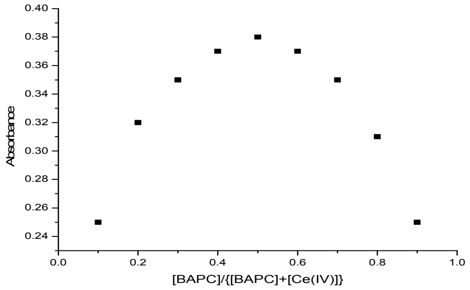


Fig. 4. Determination of stoichiometry of complex by Job's method

### F. Determination of Cerium in soil Samples

Samples were collected from some areas of KonTum provinces such as Dak Ha (DH), Sa Thay (ST) ... Before sampling, all the equipment was cleaned according to standard cleaning procedures. After that the samples were dried at $105^0$C until the weight reached a constant value, they were ground in blender and kept in the clean polyethylene containers for elemental analysis.

Weigh accurately 0.500 g of soil samples into a Platinum crucible and add 15- 20 ml of conc. $HF+H_2SO_4$, evaporating the resulting solution to almost dryness and then dissolving the residue with dilute 0.1N $HNO_3$. Add 0,2mL $La^{3+}$ solution 1mg/ml as a carrier, and use $NH_4OH$ dilute to adjust pH =2, and add 2mL 10 %, oxalic acid, filtrate the precipitate and wash by 1% $H_2C_2O_4$. After that, this precipitate is dissolved by conc. $HNO_3$. Transfer the solution into a 25 ml volumetric flask, wash the beaker and dilute with water to mark.[14]

Transfer 2ml of solution sample into a 25 ml volumetric flask and add 5 ml buffer solution and then add 1 ml of $10^{-3}$M BAPC solution. Dilute to the mark with mixture MeOH: $H_2O$ and mix well. Measure the absorbance at 515nm against water as blank.

Absorption spectra were measured with Lamda 25 UV–visible spectrophotometer (USA). Stock solution of BAPC ($10^{-3}$ M, in pure $CH_3OH$) and 1000 mg/mL metal nitrate solutions were prepared. The metal ion solutions were diluted 10 and 100 times to give 100 and 10 mg/L solutions. The working solutions were prepared daily by adding an appropriate volume of the stock solutions and $CH_3OH/H_2O$ mixed solvent solution (1:1 v/v).

Calibration curve were constructed by plotting the absorbance measured at 515 nm against the amount of Ce(IV). Equation of calibration curve is

$$\Delta A = 0.8455C + 0.1067 \ (R^2 = 0.9951)$$

where: A is the absorbance of the complex. C is the concentration of Cerium (μg/L).

The results of the amounts of Cerium in some ore samples in Kontum province is given in Table 2.

TABLE 2. ANALYTICAL RESULTS OF CERIUM IN SOME SAMPLES (N = 5, P = 0.95).

| Sample | I | II | III | IV | V |
|---|---|---|---|---|---|
| m(mg/kg) | 58.2 | 159.1 | 70.5 | 163.6 | 217.4 |

TABLE 3. COMPARISON OF ANALYTICAL METHODS CERIUM

| Reagents | λ | $\varepsilon_\lambda$ (l.mol$^{-1}$cm$^{-1}$) | pH |
|---|---|---|---|
| | | $9.3.10^{-3}$ | |
| Cupferron – Ce(IV) | 400 | | 4,5 |
| β-Diketone – Ce(IV) | 440 | | |
| Oxine–5–sulfonicCe(IV) | 480 | | 1,5 |
| Ferroin-Ce(IV) | 520 | | |
| 3,3Diaminobenzidine-Ce | 355 | $2.10^4$ | 2.0-3.0 |
| 2,4 Dihydroxy benzophenone benzoic hydrazone | 400 | | 10 |
| N-p-Chlorophenyl p-methoxy benzohydroxamic acid | 460 | $7.2.10^{-3}$ | 8-10 |
| BAPC-Ce(IV) | 515 | $7.8.10^{-4}$ | 5.0-7.0 |

The results show that, the amounts of cerium in samples of Kontum are at high level. The results of our analysis are quite consistent with the analytical results of Center of Analytical Techniques (CATech), Da Lat Nuclear Research Institute.

### G. Compared with other Methods

The figures compare reagents Cerium determined by traditional optical methods with reagents BAPC is given in Table 3. These data indicate that highly selective BAPC reagents for ion Cerium and BAPC reagents can be used for analysis.

## IV. CONCLUSION

On the basis of complexation of $Ce^{4+}$with BAPC, we have investigated the optimum conditions of the complex. The results show that complex formed in acidic solution with a pH of 5.0-7.0, and 1:1 ratio. Beer's law is obeyed in the range of $5.0x10^{-6}$-$2.5x10^{-5}$ $molL^{-1}$ of $Ce^{4+}$, molar absorptivity of complex (ε) is $7,81x10^4$ $Lmol^{-1}cm^{-1}$ at a wavelength of 515 nm and detection limit is $2.5x10^{-6}$ $molL^{-1}$. The proposed method based on the complexation of Ce(IV) and BAPC can be applied successfully for determination of Cerium in the real samples such as soil samples in Kontum

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