

## Tetradiazo(*o*-carboxy)phenylcalix[4]arene for Determination of Pb<sup>2+</sup> Ion

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A new azocalix[4]arene, 5,14,17,23-tetra[(2-benzoic acid)(azo)phenyl] calix[4]arene (**2**), has been prepared by hydrolysis of its ester derivative and characterized by <sup>1</sup>H NMR, IR, UV-Vis spectroscopy and elemental analysis. Based on UV spectral changes, we found that **2** exhibits Pb<sup>2+</sup> ion selectivity. In basic media, Pb<sup>2+</sup> forms a 1:1 complex with the ligand. Beer's law is obeyed in the range of  $2.0 \times 10^{-6}$ – $2.4 \times 10^{-5}$  mol L<sup>-1</sup> of Pb<sup>2+</sup>. The molar absorptivity ( $\epsilon$ ) of **2**-Pb<sup>2+</sup> complex is  $1.89 \times 10^4$  L mol<sup>-1</sup> cm<sup>-1</sup> at 440 nm, and the detection limit is  $1.6 \times 10^{-6}$  mol L<sup>-1</sup>.

**Key Words :** UV-Vis, Chromoionophore, Calixarene, Lead, Spectrophotometry

### Introduction

Among heavy metals, lead is the most commonly encountered toxic pollutant in the environment as a result of its current and previous use in batteries, gasoline, and paints.<sup>1,2</sup> Lead is known to cause health problems, such as digestive, neurological, cardiac, and mental troubles. In particular, it is dangerous for children, causing mental retardation. 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.<sup>1,2</sup>

Spectrophotometric methods are used more often because of their simplicity and low cost. The spectrophotometric determination of Pb<sup>2+</sup> is usually preceded by a reaction with reagents such as ammonium *N*-(dithiocarboxy)sarcosine, 1,1,1-trifluoro-4-mercapta-4-(2-thienyls)-but-3-one, porphyrin, Chrome azurol S.<sup>3</sup> Although a lot of chromogenic reagents for Pb<sup>2+</sup> detection have been reported,<sup>4</sup> none was prepared by attaching a chromogenic moiety to a platform of a macrocyclic compound, such as calixarene.

Calixarenes, which appeared after crown ethers and cyclodextrins as the third generation of inclusion compounds, have received much attention.<sup>5-15</sup> The chromoionophore is constructed from two functionally different parts: (a) an ionophore part, which recognizes ions and performs functions peculiar to a particular ionophore; and (b) a chromophore part, which transduces chemical information produced by ionophore-ion interaction into an optical signal. Many interesting aspects of complexation of alkali and alkaline earth metal ions and transition metal ions were disclosed by studying the absorption behavior of chromophoric groups.<sup>16-23</sup> However, very few calixarene derivatives were designed as chromogenic reagents for the determination of Pb<sup>2+</sup>. Therefore, the development of a simple, sensitive and reliable method for the determination of Pb<sup>2+</sup> is worthwhile.

Previous studies confirmed that *ortho*-ester diphenylazo calix[4]arenes exhibit excellent selectivity for transition metal ions.<sup>24</sup> In order to tune up the solubility of the corresponding ester, we have done the hydrolysis of it and obtain acid functionalized tetraazocalix[4]arene of which the water solubility is bit more increased. We now report the results of *ortho*-acid diphenylazocalix[4]arene complexation studies towards various metal ions.

### Experimental Section

**Apparatus.** All reagents used in the experiment were purchased from Merck. Absorption spectra were measured with a Varian-5000 UV-visible spectrophotometer (USA). <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on DPX 400 and Bruker AVANCE-600 spectrometers (Germany), respectively, using DMSO-d<sub>6</sub> as solvent and TMS as internal standard. IR spectrum was recorded on a Perkin Elmer-FT-IR 1000 spectrometer (USA) with KBr pellets. The elemental analyses were performed in the laboratory of Organic Department in T.U. Kaiserslautern (Germany).

**5,11,17,23-Tetra[(2-ethyl acetoethoxyphenyl)(azo)-phenyl]calix[4]arene (**1**).** A solution of 2-aminobenzoate (3.10 g, 18.7 mmol), NaNO<sub>2</sub> (1.46 g, 21.1 mmol) and conc. HCl (5 mL) in water (10 mL) was added dropwise over a period of about 15 minutes into a solution of calix[4]arene (2.00 g, 4.70 mmol) in THF:pyridine (5:2) (25 mL) at 0 °C. The solution was stirred for 15 mins and then additional 24 hrs at room temperature. After removal of the solvent *in vacuo*, the resulting solid was dissolved in EtOAc (100 mL) and the organic layer was washed three times with water. The organic layer was dried over anhydrous MgSO<sub>4</sub> and the solvent was removed *in vacuo* to give a reddish oil. Column chromatography using EtOAc:hexane (2:1) provided 2.4 g (45%) of **1** as a yellow orange solid. Mp: 158-162 °C. IR (KBr pellet, cm<sup>-1</sup>): 3220, 1735. <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  7.43-7.22 (m, 24H, Ar-H), 4.24 (d, 4H, Ar-CH<sub>2</sub>-Ar), 3.97-

3.86 (m, 8H, COCH<sub>2</sub>CH<sub>3</sub>, *J* = 6.9, 7.3 & 6.7 Hz), 3.42 (d, 4H, Ar-CH<sub>2</sub>-Ar), 0.82-0.75 (t, 12H, COCH<sub>2</sub>CH<sub>3</sub>, *J* = 6.9 Hz). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>): δ 168.1, 159.2, 151.5, 145.3, 132.3, 130.8, 129.7, 129.6, 129.0, 124.5, 119.7, 61.5, 32.3, 14.8. FAB MS *m/z* (M<sup>+</sup>): Calcd, 1129.18. Found, 1129.0. Anal. Calcd. for C<sub>64</sub>H<sub>56</sub>N<sub>8</sub>O<sub>12</sub>: C, 68.07; H, 5.00. Found: C, 68.05; H, 5.02.

**5,11,17,23-Tetra[(2-benzoic acid)(azo)phenyl]calix[4]-arene (2).** A solution of **1** (0.44 mmol) and NaOH (4.42 mmol) in ethanol (10 mL) and water (5 mL) was refluxed for 12 h and evaporated *in vacuo*. The residue was dissolved in ethyl acetate and the solution was washed twice with 20% HCl and then three times with water. The organic layer was dried over MgSO<sub>4</sub> and evaporated *in vacuo* to yield 0.35 g (78 %) of **2** as a red solid (Scheme 1). Mp: 164-168 °C, IR (KBr pellet, cm<sup>-1</sup>): 3220, 1735. <sup>1</sup>H NMR (200 MHz, DMSO-d<sub>6</sub>): δ 10.25 (4H, s, -OH), 7.33-7.02 (m, 24H, Ar-H), 4.24 (broad s, 4H, Ar-CH<sub>2</sub>-Ar), 3.42 (broad s, 4H, Ar-CH<sub>2</sub>-Ar). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>): δ 30.6, 116.8, 122.4, 130.1, 130.4, 130.9, 134.5, 146.1, 154.5, 155.3 and 169.7. FAB MS *m/z* (M<sup>+</sup>): Calcd, 1016.28. Found, 1016.96. Anal. Calcd. for C<sub>56</sub>H<sub>40</sub>N<sub>8</sub>O<sub>12</sub>: C, 66.14; H, 3.96; N, 11.02; O, 18.88. Found: C, 66.13; H, 3.94; N, 11.03; O, 18.89.

**Job's method of continuous variation:** A series of solutions with varying metal ion and hydroxyazobenzene concentrations were prepared by maintaining the total hydroxyazobenzene and metal ion concentration constant (*i.e.*,  $C = C_M + C_L$  where  $C$ ,  $C_M$  and  $C_L$  are the total concentration of metal ion and ligand, the concentration of the metal ion and the concentration of the ligand, respectively). The optical densities (OD) were measured at 530 nm and were plotted against the mole-fraction of the ligand,  $X_L$  ( $C_L/C$ ). Assuming that only one complex was formed with a composition  $ML_n$ , the value of '*n*' was calculated from the  $X_{max}$  (mole-fraction of the ligand ( $X_L$ ) at maximum absorption) from the relation:

$$n = \frac{X_{max}}{(1 - X_{max})}$$

The stability constants were estimated by monitoring the decrease in the intensity of the absorbance at the peak with the data reduction being effected using *Benesi-Hildebrand* plots.<sup>25</sup>

$$\frac{1}{(A - A_0)} = \frac{1}{(A_{sat.} - A_0)} + \frac{1}{(A_{sat.} - A_0) \cdot K \cdot [Guest]} \quad (1)$$

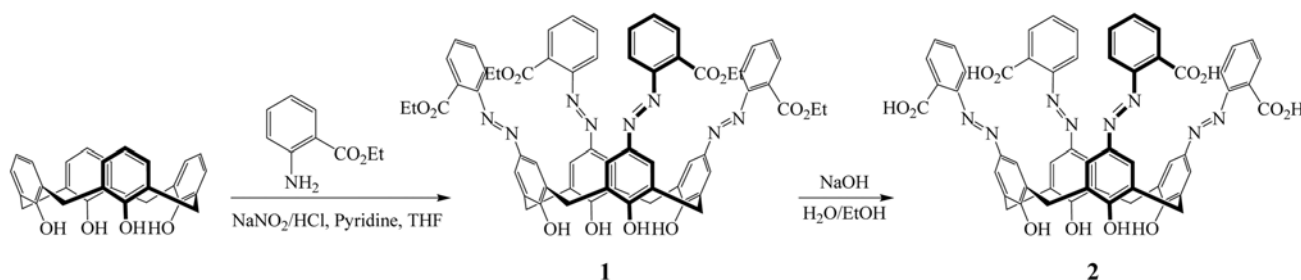
Where  $K$  is the stability constant,  $A$  the observed absorbance at each metal ion concentration tested,  $A_0$  is the absorbance of **2** in the absence of metal ion,  $A_{sat.}$  is the absorbance at the point of saturation and  $[Guest]$  is the concentration of metal ion.

**Chemicals and solutions.** A stock solution of **2** ( $1.0 \times 10^{-3}$  mol L<sup>-1</sup>) was prepared in MeCN. Lead perchlorate stock solution ( $2.0 \times 10^{-2}$  mol L<sup>-1</sup>) was prepared in water. The Pb<sup>2+</sup> solution was diluted 10 and 100 times to give  $2 \times 10^{-3}$  mol L<sup>-1</sup> and  $2 \times 10^{-4}$  mol L<sup>-1</sup> solutions. A working standard solution of Pb<sup>2+</sup> ( $2.0 \times 10^{-5}$  mol L<sup>-1</sup>) was prepared from this stock solution by dilution with distilled water. 0.1 mol L<sup>-1</sup> of Na<sub>2</sub>HPO<sub>4</sub>-NaOH buffer solution (pH = 12.5) was employed. All other chemicals were of analytical grade.

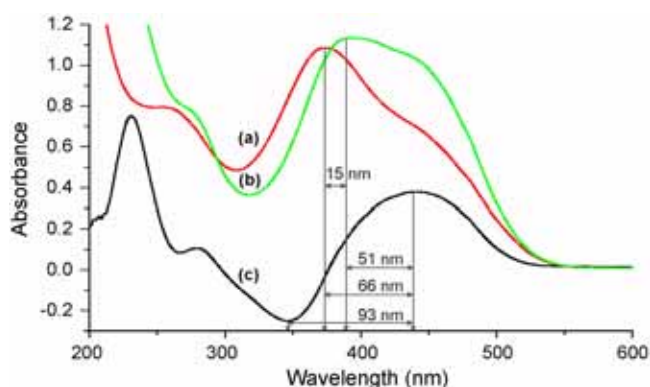
## Results and Discussion

The synthetic route of compound **1** and **2** is depicted in Scheme 1. Diazo coupling reaction of calix[4]arene with ethyl 2-aminobenzoate, sodium nitrite and concentrated HCl in pyridine with THF (5:2) gave desired product **1** in moderate yield. The cone conformation of **1** is confirmed by its NMR spectra: (a) two doublets at 4.24 and 3.42 ppm with an AB pattern for methylenic protons of ArCH<sub>2</sub>Ar in the <sup>1</sup>H NMR spectra and (b) singlet at 32.3 ppm for the ArCH<sub>2</sub>Ar bridge carbons in the <sup>13</sup>C NMR spectra. Hydrolysis of **1** provided **2** in quantitative yield. The cone-conformation of **2** is the same as that of **1** referring by similar <sup>1</sup>H and <sup>13</sup>C NMR patterns.

Absorption spectra of **2** and its Pb<sup>2+</sup> complex under the optimum conditions are shown in Figure 1. In this figure, curve 'a' and 'b' are the spectra of **2** and **2**-Pb<sup>2+</sup> complex against water blank, respectively. Whereas, curve 'c' is the spectrum of **2**-Pb<sup>2+</sup> complex against the corresponding reagent blank, *i.e.* it represents the spectrum of **2** in a solution containing Pb<sup>2+</sup> minus that of the same concentration of **2** in a similar, but Pb<sup>2+</sup>- free solution. The maximum absorption of **2** shows up at 374 nm, whereas the absorption peaks of the complex are located at 389 nm and 440 nm, hence wavelength changes ( $\Delta\lambda$ ) are 15 nm and 66 nm, respectively. The appearance of negative peak of the complex at 347 nm in curve c results from the complexation of **2** with Pb<sup>2+</sup> and the employment of the corresponding reagent blank as a reference. Similar observations can be found in literatures.<sup>22,23</sup> This result implies that there is a subtle balance between metal complexation-induced release of protons from the



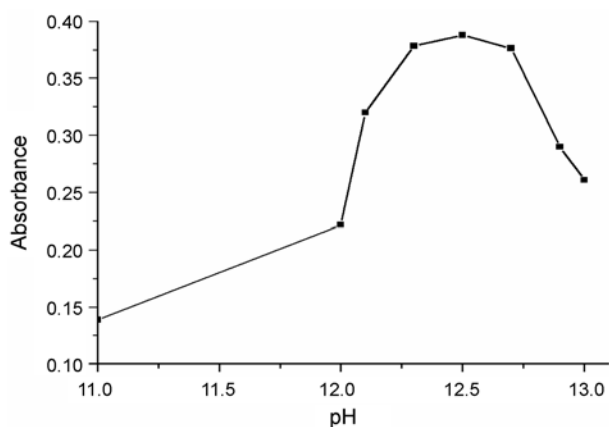
**Scheme 1.** Synthetic route to **1** and **2**.



**Figure 1.** Absorption spectra of **2** and its  $\text{Pb}^{2+}$  complex at pH 12.5. Conditions: (a) the solution containing  $2.0 \times 10^{-5} \text{ mol L}^{-1}$  of **2** against water blank; (b) the solution containing  $3.0 \times 10^{-5} \text{ mol L}^{-1}$  of **2** and  $2.0 \times 10^{-5} \text{ mol L}^{-1}$  of  $\text{Pb}^{2+}$  against water blank; (c) the solution containing  $3.0 \times 10^{-5} \text{ mol L}^{-1}$  of **2** and  $2.0 \times 10^{-5} \text{ mol L}^{-1}$  of  $\text{Pb}^{2+}$  against the corresponding reagent blank.

azophenols to the quinone-hydrazone tautomer,<sup>24</sup> and the *ortho*-acid groups of **2** can stabilize the quinone-hydrazone form in azocalix[4]arene after adding  $\text{Pb}^{2+}$  ion. Concerning complexation between **2** and  $\text{Pb}^{2+}$  ion,  $\text{Pb}^{2+}$  ion is able to interact with the phenol unit of the lower rim of **2** and the azo group in the medium cavity of **2**. However, due to the intramolecular hydrogen bonding of the lower rim,  $\text{Pb}^{2+}$  ion may be accommodated within the region of the medium cavity of the calixarene.<sup>21,22</sup> This result is in good agreement with those of Arnaud-Neu *et al.*<sup>8,21</sup>

In order to see the wavelength change upon metal ion encapsulation, we have first carried out the pH variable experiment. The effect of pH on the absorbance of **2**- $\text{Pb}^{2+}$  complex at 440 nm is shown in Figure 2. Different pH values were obtained by varying the relative amount of  $\text{Na}_2\text{HPO}_4$  and  $\text{NaOH}$ . The maximum and constant absorbance of the complex is obtained in the pH range of 12.2–12.8, and a pH of 12.5 may thus be chosen for the following experiments. At pH = 12.5, the effects of four kinds of buffers on the absorbance were also examined. The results indicated that  $0.1 \text{ mol L}^{-1} \text{ Na}_2\text{HPO}_4\text{-NaOH}$  is the best buffer,



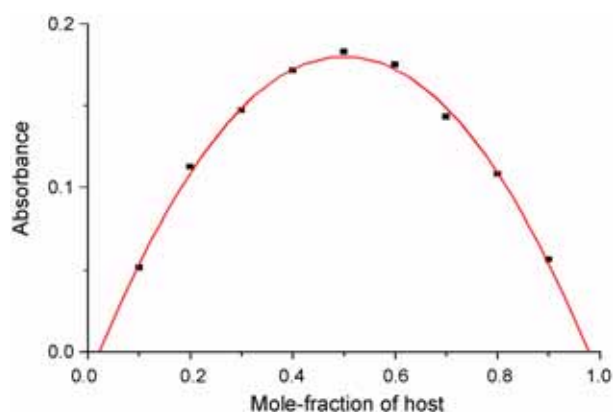
**Figure 2.** Effect of pH. Conditions:  $2.0 \times 10^{-5} \text{ mol L}^{-1}$  of **2**,  $2.0 \times 10^{-5} \text{ mol L}^{-1}$  of  $\text{Pb}^{2+}$ .

whose optimum volume is 1.5 mL for 5 mL of test solution.

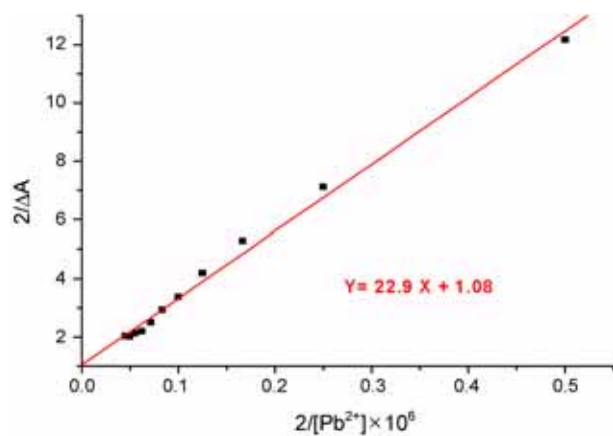
Concerning concentration of **2**, the absorbance at 440 nm reaches a maximum with the final concentration ranging from  $1.5 \times 10^{-5} \text{ mol L}^{-1}$  to  $3.5 \times 10^{-5} \text{ mol L}^{-1}$ . In this work,  $2.0 \times 10^{-5} \text{ mol L}^{-1}$  of **2** was chosen for the following experiments. Under the optimum conditions, the linear regression equation was determined to be: Absorbance ( $A$ ) =  $0.025 \times C$  ( $10^{-6} \text{ mol L}^{-1}$  of  $\text{Pb}^{2+}$ ) +  $0.021$  ( $n = 8, r = 0.99$ ). Beer's law is obeyed in the range of  $2.0 \times 10^{-6}$ – $2.4 \times 10^{-5} \text{ mol L}^{-1}$  of  $\text{Pb}^{2+}$ . The apparent molar absorptivity ( $\epsilon$ ) of **2**- $\text{Pb}^{2+}$  complex is  $1.89 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ , and the detection limit is  $1.6 \times 10^{-6} \text{ mol L}^{-1}$  ( $S/N = 3$ ).

We also take into account the stability of the complex. The absorbance of **2**- $\text{Pb}^{2+}$  complex can reach maximum in 10 min and remains unchanged for at least 3 h. Use of the continuous variation method revealed that the stoichiometric ratio of **2** to  $\text{Pb}^{2+}$  in the complex is 1:1 (Fig. 3). The stability constant ( $K$ ) was calculated to be  $6.2 \times 10^4 \text{ mol}^{-1} \text{ L}$  by using the continuous variation method (Fig. 4).<sup>25</sup>

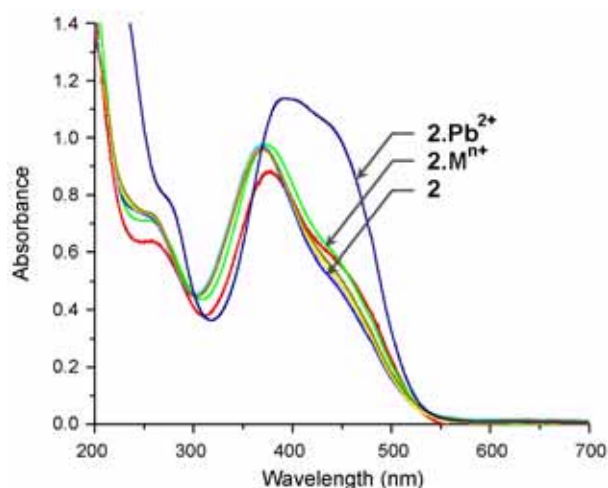
To study the competitive and anti-jamming effect, many metal ions were added to aqueous solution of **2** and their UV spectra were measured, the UV absorption of **2** and **2**- $\text{M}^{n+}$  ( $\text{M}^{n+}$ : alkali, alkaline earth metal ions,  $\text{Cu}(\text{ClO}_4)_2$ ,  $\text{Ni}(\text{ClO}_4)_2$ ,  $\text{Co}(\text{ClO}_4)_2$ ,  $\text{Fe}(\text{ClO}_4)_2$ ,  $\text{Cr}(\text{ClO}_4)_3$ ,  $\text{Zn}(\text{ClO}_4)_2$ ,  $\text{Mn}(\text{ClO}_4)_2$ ,  $\text{Pb}(\text{ClO}_4)_2$  and  $\text{Al}(\text{ClO}_4)_3$ ) complexes are showed in Figure



**Figure 3.** Job's plot for **2**- $\text{Pb}^{2+}$  complex.



**Figure 4.** Relative absorbance for mixture of  $\text{Pb}^{2+}$  and **2** vs.  $2/[\text{Pb}^{2+}]$ .



**Figure 5.** The absorption spectra of **2** and its complexes. Conditions:  $3 \times 10^{-5}$  mol L $^{-1}$  of **2** and  $2 \times 10^{-5}$  mol L $^{-1}$  of metal cations.

**Table 1.** The approximate selectivity coefficients and the color reaction contrast of other cations

M <sup>n+</sup>	Cu <sup>2+</sup>	Ca <sup>2+</sup>	Fe <sup>2+</sup>	Cr <sup>3+</sup> , Co <sup>2+</sup> , Ni <sup>2+</sup>	Other metal ions
K <sub>Pb<sup>2+</sup>/M<sup>n+</sup></sub>	280	300	320	500	> 5000
$\Delta\lambda$ (nm) <sup>a</sup>	97	125	165	170	— <sup>b</sup>

<sup>a</sup> $\Delta\lambda$  (nm) =  $\lambda_{\max}$  of **2**·M<sup>n+</sup> against reagent blank -  $\lambda_{\max}$  of **2** against water blank. <sup>b</sup>No color reaction occurs.

5. Upon interaction with aqueous solution of Pb<sup>2+</sup>, **2** exhibits a marked absorption peak at 440 nm, whereas addition of other metal ions to the solution of **2** does not cause any conspicuous change, although their absorption intensities at 374 nm change a little compared to free **2**.

To further verify the little interference of other metal ions on the **2**·Pb<sup>2+</sup> complex, the color reaction contrast  $\Delta\lambda$  ( $\lambda_{\max}$  (complex) -  $\lambda_{\max}$  (ligand)) was measured, and the approximate selectivity coefficients for Pb<sup>2+</sup> to other cations can be calculated as  $K_{Pb^{2+}/M^{n+}} = C_{M^{n+}}/C_{Pb^{2+}}$  using the salt concentrations required to give an absorbance value of 0.38 at 440 nm against the corresponding reagent blank.<sup>21,22,23</sup> The results are summarized in Table 1. It can be seen that **2** exhibits excellent selectivity for Pb<sup>2+</sup> over a wide range of transition, alkali and alkaline earth metal cations.

In conclusion, 5,14,17,23-*tetra*[(2-benzoic acid)(azo)phenyl]calix[4]arene has been newly synthesized and its chromogenic behaviors towards Pb<sup>2+</sup> has been also investigated. This new azocalix[4]arene of which the solubility is bit more increased can be used to effectively determine Pb<sup>2+</sup> ion in the concentration range of  $2.0 \times 10^{-6}$  -  $2.4 \times 10^{-5}$  mol L $^{-1}$ .

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