Study on the Extraction of Cerium(IV) from Nitric Acid Medium with tri-Isoamyl Phosphate

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This paper studied the extraction behavior of tri-isoamyl phosphate (TiAP)/n-dodecane as extractant for Ce(IV) in a nitric acid medium. In the study of extraction of pure Ce(IV) solution, the test results show that when the organic phase and the water phase ratio is 1:1, the organic phase and the water phase are mixed and contacted for 90 s, and the pH value is 0-2.0, the extraction tends to be balanced, and the complexes formed by the extraction are theoretically deduced and obtained the theoretical molecular formula of the complex is Ce(NO₃)₄·ₙTiAP, where n is between 2 and 3; under this condition, the extraction rate of pure Ce(IV) can reach 99.45% after 16-stage countercurrent extraction test; the relative standard deviation is less than 2%. In the extraction of Ce(IV) solution containing other metal ions, the test results show that the extraction rate of 0.05 mol·L⁻¹ TiAP/n-dodecane solution to the material liquid containing Ce(IV) 0.005 g·L⁻¹ is about 64%. However, the experiment found that the extraction rate tends to decrease with the increase of the material liquid’s standing time. After 24 hours, the extraction rate is close to 0. Then, the experiment researched the influence of each metal ion component on the extraction after standing and obtained the metal. In the presence of ionic components, the extraction rate tends to decrease with the increase of the liquid standing time. Considering the strong oxidizing property of Ce⁴⁺ under acidic conditions, the experimental study of TiAP on Ce³⁺ and Ce⁴⁺ mixtures with different percentages. The results show that as the percentage of Ce³⁺ in the feed liquid increases, the extraction rate is in a downward trend, and the trend is consistent with the increase in the standing time on the extraction rate. The original liquid contains impurity Ce⁴⁺ is slowly reduced to Ce³⁺, which affects the extraction results. Studies have shown that TiAP of 0.05 mol·L⁻¹ performs well for the extraction of Ce⁴⁺ in pure Ce(IV) solution; TiAP of 0.05 mol·L⁻¹ is effective for extraction of Ce(III) solution of 0~5 mg·L⁻¹. The effect of Ce³⁺ is not obvious; because Ce⁴⁺ is too oxidizing, when other metal ions studied are present at the concentration studied, it will accelerate the reduction of Ce⁴⁺ and affect the extraction results.

1. Introduction

Cerium (Ce) has been widely used in polishing materials [1], catalysts [2, 3], ceramic glazes [4, 5], and other chemical products. Ce(IV)/HNO₃ is used as detergent for radioactive decontamination on metal material surface, because of its high-efficient decontamination and low secondary waste production level [5–9]. The extraction and separation of cerium can be performed by amines [10, 11] and organic phosphorus [12–14] and ionic liquids [15, 16], but the cerium content studied is relatively high. So far, there are few studies on the extraction behavior of a trace amount of cerium. ¹⁴¹Ce, ¹⁴³Ce, and ¹⁴⁴Ce are three important fission product nuclides with short half-lives and small cumulative yield. Hence, it is in urgent need of new extractant and extraction process parameters to quickly separate and extract the abovementioned nuclides [17]. Therefore, in this paper, tri-isoamyl phosphate (TiAP), which has excellent
characteristics for the extraction of actinides, was used as the extractant to study its extraction behavior for trace Ce(IV) in nitric acid medium. Recycling cerium provides experimental reference.

2. Materials and Methods

2.1. Materials. All the chemicals and reagents used in this study were analytically pure: TiAP, provided by China Institute of Atomic Energy; n-dodecane, Guangdong Wengjiang Chemical Reagent Co., Ltd.; anhydrous sodium carbonate, Tianjin Tianli Chemical Reagent Co., Ltd.; nitric acid, Guangzhou Jinhua University Chemical Reagent Co., Ltd.; cerium ammonium nitrate, Shanghai Maclean Biochemical Technology Co., Ltd.; cerium nitrate hexahydrate, Tianjin Fuchen Chemical Reagent Factory; tribromoarsenazo, Guangdong Wengjiang Chemical Reagent Co., Ltd.; dihydrate oxalic acid, Guangdong Wengjiang Chemical Reagent Co., Ltd.; hydrochloric acid, Hengyang Kaixin Chemical Reagent Co., Ltd.; copper nitrate trihydrate, Tianjin Damao Chemical Reagent Factory; chromium nitrate nine water, Tianjin Damao Chemical Reagent Factory; ferric nitrate nine water, Guangdong Guanghua Chemical Factory Co., Ltd.; and nickel nitrate hexahydrate, Tianjin Fuchen Chemical Reagent Factory.

2.2. Mixer-Settler. Figure 1 is a schematic diagram of mixed clarifying tank for the experiment. There are 16 stages in total, and each stage is composed of mixing tank and clarifying tank.

2.3. TiAP Pretreatment. TiAP and n-dodecane were prepared into 0.05 mol·L⁻¹ TiAP/n-dodecane organic phase, and then, the organic phase and water phase were successively subjected to 5% sodium carbonate alkaline washing, 0.5 mol·L⁻¹ nitric acid pickling, and water washing in a 1:1 ratio to remove organic acid substances and reduce the influence of organic acid substances on their extraction performance.

2.4. Experiment. The extraction rate was calculated according to Equation (1).

\[
E = \frac{m_0 - m_1}{m_0} \times 100\%
\]

where \(E\) is the extraction rate, %; \(m_0\) is the amount of cerium in the feed liquid, g; \(m_1\) is through \(n (n = 1, 2 \cdots 16)\). The cerium content of the extraction liquid after the stage extraction is measured in grams.

Table 1: Contents of extract liquid containing other metal ions.

| Reagents name          | Specification | Ion name  | Contents        |
|------------------------|--------------|-----------|-----------------|
| \((\text{NH}_4)_2\text{Ce(NO}_3)_6\)   | AR           | Ce        | 0.005 g·L⁻¹     |
| \(\text{Cu(NO}_3)_2\cdot 3\text{H}_2\text{O}\) | AR           | Cu²⁺     | 4 g·L⁻¹         |
| \(\text{Fe(NO}_3)_3\cdot 9\text{H}_2\text{O}\) | AR           | Fe³⁺     | 0.01 g·L⁻¹      |
| \(\text{Ni(NO}_3)_2\cdot 6\text{H}_2\text{O}\) | AR           | Ni²⁺     | 0.008 g·L⁻¹     |
| \(\text{Cr(NO}_3)_3\cdot 9\text{H}_2\text{O}\) | AR           | Cr³⁺     | 0.005 g·L⁻¹     |
| HNO₃                   | AR           | HNO₃     | 2 Mol·L⁻¹       |

Figure 1: Extraction flow diagram of mixer-settler. a: Automatic collector; b: mixer-settler; c: constant current injection pump; d: feed liquid tank; e: extracted liquid tank; f: extraction solvent tank.
pretreatment. Organic phase and aqueous phase (O/A) of a certain volume were measured in proportion and placed in the separation funnel. After standing the separation solution for a certain time, the cerium content of the sample was analyzed using the National Standards of the People’s Republic of China GB/T13748.4-92 analysis method. There were more than three parallel tests.

By separating funnel extraction to obtain the best mixed contact time and O/A, 16-stage counter-current extraction experiment was carried out. Theoretically, it should be able to achieve equilibrium in 2 bed volumes (16-stage mixing tank plus clarification tank volume). Therefore, in this experiment, after 3 bed volumes are extracted, the machine stops and samples the lower clear liquid of each stage according to GB/T 13748.4-92 for cerium analysis; then, after starting up and running for 1 bed volume, the lower clear liquid of each stage was sampled and analyzed again, and the samples were sampled more than three times accordingly.

2.4.2. Extraction Test of Ce(IV) Solution Containing Other Metal Ions. Prepare 0.005 g·L\(^{-1}\) Ce(IV) solution containing other metal ions according to Table 1 and use the 0.05 mol·L\(^{-1}\) TiAP/n-dodecane extractant pretreated by TiAP pretreatment to carry out the extraction study. There are more than three parallel tests.

3. Results and Discussion

3.1. Extraction Test of Pure Ce(IV) Solution

3.1.1. Effect of O/A on Extraction Rate. O/A was injected into the parting funnel according to different conditions (O/A: 5:1, 3:1, 2:1, 1:1, 1:2, and 1:3). The mixed contact time of the organic phase and the aqueous phase was 90 s for the extraction test, and the results are shown in Figure 2.

As can be seen from Figure 2, the extraction rate of Ce(IV) by TiAP gradually increases with the gradual increase of the O/A. When the O/A is greater than 1:1, the extraction rate increases gently and gradually tends to balance. Therefore, the optimal O/A for this extraction is set as 1:1. Theoretical derivation of the extraction process is as follows:

In nitric acid medium, Ce(IV) and TiAP reaction equations may be as follows:

\[
\text{Ce}^{4+} + 4\text{NO}_2^- + n\text{TiAP} = \text{Ce(NO}_3)_4 \cdot n\text{TiAP}
\]  

(2)

\(K\) is the equilibrium constant of the chemical reaction, and the expression is given by Equation (3).

\[
K = \frac{[\text{Ce(NO}_3)_4 \cdot n\text{TiAP}]}{[\text{Ce}^{4+}]^4[n\text{TiAP}]^n}
\]  

(3)

\(D\) is for Ce(IV) distribution ratio, and the distribution of the expressions is given by type (4).

\[
D = \frac{[\text{Ce(NO}_3)_4 \cdot n\text{TiAP}]}{[\text{Ce}^{4+}]} = \frac{c_0 - [\text{Ce}^{4+}]}{R[\text{Ce}^{4+}]}
\]  

(4)

where \(c_0\) is the initial concentration of cerium in the aqueous phase, g·L\(^{-1}\); \(R\) for the O/A.

![Figure 2: Effect of the O/A on extraction rate.](image-url)
By changing Equation (4), the relationship between [Ce⁴⁺] and D in the aqueous phase in the extraction process can be obtained.

\[
[\text{Ce}^{4+}] = \frac{c_0}{1 + D \cdot R}
\]  

(5)

According to the conservation of nitrate ion material,

\[
[\text{NO}_3^-] = 6[\text{Ce}^{4+}] + 2[\text{Ce(NO)}_4 \cdot n\text{TiAP}] = 2c_0 + 4[\text{Ce}^{4+}] = \frac{6 + 2D \cdot R}{1 + DR}c_0
\]

(6)

After combining Equations (3) and (4), it can be concluded that

\[
K = \frac{D}{[\text{NO}_3^-]^4[\text{TiAP}]^n}
\]

(7)

\[
D = K[\text{NO}_3^-]^4[\text{TiAP}]^n
\]

(8)

\[
\ln D = \ln K + 4\ln[\text{NO}_3^-] + n\ln[\text{TiAP}]
\]

(9)

It can be seen from Equation (9) that the distribution ratio of Ce(IV) in the two phases is related to [NO₃⁻] and [TiAP]. According to Equation (6), [NO₃⁻] is related to [Ce⁴⁺]. Adding TiAP to the extraction system will reduce [Ce⁴⁺] in the aqueous phase, thus reducing [NO₃⁻]. However, the amount of TiAP is much larger than the amount of Ce(IV) in the aqueous phase, so the value of [TiAP] basically remains unchanged before and after extraction.

Data obtained from the test (shown in Table 2): when O/A is 1: 3, the extraction rate is 43.68%, that is, the distribution ratio D is 2.33; when O/A is 1: 2, the extraction rate is 63.16%, that is, the distribution ratio D is 3.43; when O/A is 1: 1, the extraction rate is 92.54%, that is, the distribution ratio D is 12.40. Substitute these experimental data into Equation (9), and it can be obtained that n is between 2 and 3.

3.1.2. Effect of Mixed Contact Time of Organic Phase and Water Phase on Extraction. O/A = 1 : 1 was injected into the parting funnel, and the extraction test was carried out according to different contact times (contact time: 30 s, 45 s, 60 s, 75 s, 90 s, and 120 s). The results are shown in Figure 3.

As can be seen from Figure 3, with the increase of the contact time between the organic phase and the water phase, the extraction rate gradually increased. When the contact time reached 90 s, the increase of the extraction rate was no longer significantly increased, and the extraction gradually tended to balance. Therefore, the optimal contact time between the organic phase and water was as 90 s.

3.1.3. Effect of pH on Extraction. The feed liquid with different pH (0, 0.5, 1, 1.5, and 2) was prepared with nitric acid, and then, each liquid was injected into different liquid sorting funnels 1: 1 compared with the organic phase, respectively. The mixed contact time between the organic phase and the water phase was 90 s for extraction test. The test results are shown in Figure 4.

It can be seen from Figure 4 that in the pH value range of 0-2.0, the extraction rate tends to rise with the increase of pH value. However, in general, when the pH value is between 0 and 2, the extraction rate is between 91.5% and 94%, and the variation range is not very great.

3.1.4. Extraction with 16-Stage Mixer-Settler. Test parameters were set according to the best O/A and the mixing contact time of organic phase and water phase: cerium concentration of the feed liquid: 0.005 g·L⁻¹; concentration of organic extractant: 0.05 mol·L⁻¹; O/A: 1: 1; mixed contact time: 90 s; temperature: 20°C, 16-stage of countercurrent extraction for the material liquid; and feed liquid and extractant precision injection pump: 0.8 ml⁻¹, 10 times-min
After the extraction process reaches the extraction equilibrium, the extraction rate corresponding to each stage is shown in Figure 5.

Figure 5 shows that the level of 16 countercurrent extraction 0.05 mol·L⁻¹ TiAP/n-dodecanoic extractant for 0.005 g·L⁻¹ that does not contain metal ion impurities of pure Ce(IV) solution extraction rate can reach 99.45%, and a few sample results are stable; the relative standard deviation is less than 2%.

3.2. Extraction Test of Ce(IV) Solution Containing Other Metal Ions

3.2.1. Extraction of the Feed Liquid. The feed liquid containing Ce⁴⁺ (0.005 g·L⁻¹), Fe³⁺ (0.01 g·L⁻¹), Cr³⁺ (0.005 g·L⁻¹), Ni²⁺ (0.008 g·L⁻¹), Cu²⁺ (4 g·L⁻¹), and nitric acid (2 mol·L⁻¹) was prepared and extracted with TiAP of 0.05 mol·L⁻¹. The extraction results are shown in Table 3.

As can be seen from Table 3, when the above metal ions were added into the cerium solution, the extraction rate of Ce(IV) was about 64% at 0.05 mol·L⁻¹ TiAP. The concentration of cerium in the solution is small, there are many impurities in the solution, and various metal ions are mixed in the solution. When the organic phase contacts with water, impurity ions interfere with TiAP and Ce⁴⁺ to form a complex, so that cerium cannot fully enter the organic phase to achieve the purpose of extraction. The relative standard deviation is less than 2%.

| Number | Extraction rate/% |
|--------|-------------------|
| 1      | 64.60             |
| 2      | 63.97             |
| 3      | 62.60             |

1. After the extraction process reaches the extraction equilibrium, the extraction rate corresponding to each stage is shown in Figure 5.

Figure 5 shows that the level of 16 countercurrent extraction 0.05 mol·L⁻¹ TiAP/n-dodecanoic extractant for 0.005 g·L⁻¹ that does not contain metal ion impurities of pure Ce(IV) solution extraction rate can reach 99.45%, and a few sample results are stable; the relative standard deviation is less than 2%.
deviation of the three parallel tests was 1.52%, and the test results were reliable.

The extraction effect disappeared when repeated tests were carried out on the above test feed liquid at intervals. It was verified by repeated tests for many times that the newly prepared feed liquid could repeat the above experimental results, but the extraction effect disappeared after standing for a period of time. It was determined that the standing time of the feed liquid had an effect on the extraction rate.

3.2.2. Effect of Standing Time on Extraction Rate. According to the unrepeatable test results of the raw material liquid in Section 3.2.1, the influence of the standing time on the extraction rate was studied. The newly prepared feed liquid in Section 3.2.1 was extracted with 0.05 mol·L⁻¹ TiAP at regular intervals, and the extraction results are shown in Figure 6.

It can be seen from Figure 6 that with the increase of the material liquid standing time, the extraction rate shows a downward trend. When the standing time exceeds 24 h, the
extraction rate is basically zero. It has been verified by repeated experiments that the extraction rate of the newly prepared liquid material shows a downward trend with the increase of the material liquid standing time.

3.2.3. The Effect of Single Metal Ion on Ce(IV) Extraction. In order to explain the result of the decrease in extraction rate with the increase of the material liquid standing time in Section 3.2.2, the experimental study of each single impurity component to 0.05 mol·L⁻¹ TiAP extraction 0.005 g·L⁻¹ Ce⁴⁺ solution. The effect is shown in Figure 7. Figure 7 shows that the extraction rate of ammonium cerium nitrate solution without other metal ions was stable with the increase of standing time and had no influence. With the addition of impurity ions with the above concentration, the extraction rate of each experiment tended to decrease with the increase of standing time, indicating that the impurity components studied had an effect on the extraction of 0.005 g·L⁻¹ Ce⁴⁺ solution by 0.05 mol·L⁻¹ TiAP.

In order to eliminate the influence of ammonium ion in ammonium cerium nitrate, a cerium-containing solution was prepared with cerium dioxide, and 0.005 g·L⁻¹ cerium solution was prepared according to the method of dissolving cerium dioxide in GB/T 13748.4-92. After 0.05 mol·L⁻¹ TiAP extraction, the extraction effect is 0, almost no extraction. In contrast to the dissolution process of ceria, the reaction formula of the dissolution process can be deduced as follows:

\[
2\text{CeO}_2 + \text{H}_2\text{O}_2 + 6\text{H}^+ \xrightarrow{\Delta} 2\text{Ce}^{3+} + 4\text{H}_2\text{O} + \text{O}_2\uparrow
\]  

(10)

It can be concluded from formula (10) that the dissolved cerium solution is a trivalent cerium solution, and the extraction rate of the material liquid with 0.05 mol·L⁻¹ TiAP is 0. It is judged that the material liquid prepared by cerium ammonium nitrate is added after the above impurity ions are allowed to stand still, and the tetravalent cerium inside is gradually reduced to trivalent cerium, which has an impact on the extraction.

3.2.4. Effect of \([\text{Ce}^{3+}]\) on Extraction Rate. Based on the judgment of Section 3.2.3, the experiment researched the effect of the proportion of \([\text{Ce}^{3+}]\) in \([\text{Ce}^{3+}, \text{Ce}^{4+}]\) on the extraction results. The total cerium concentration is the same when the cerium ammonium nitrate and cerium nitrate hexahydrate are used, but the proportion of \([\text{Ce}^{3+}]\) is different. The material liquid was extracted with 0.05 mol·L⁻¹ TiAP, and the extraction results are shown in Figure 8.

It can be seen from Figure 8 that as the content of trivalent cerium in the total cerium increases, the extraction rate tends to decrease until the extraction effect is zero. It is worth mentioning that, comparing Figures 6 and 8, the two graphs are in good agreement in trend, which can prove that the valence of cerium affects 0.05 mol·L⁻¹ TiAP to 0.005 g·L⁻¹ cerium solution of extraction.
3.2.5. Theoretical Analysis. Judging from the chemical properties of Ce⁴⁺, Ce⁵⁺ has strong oxidizing properties, especially in acidic environments. Figure 9 is the potential-pH diagram of cerium [14]. It can be seen from Figure 4 that the stable area of Ce⁴⁺ is small, and the stability conditions are more severe.

Referring to the standard electrode potential of cerium and various impurity ions in an acidic medium [18], cerium can theoretically react with various impurity ions as follows.

\[
2\text{Ce}^{4+} + \text{Ni}^{2+} + 2\text{H}_2\text{O} = 2\text{Ce}^{3+} + 4\text{H}^+ + \text{NiO}_2\downarrow E^\theta = 0.042\text{V} \\
\text{Ce}^{4+} + \text{Cr}^{3+} + 2\text{H}_2\text{O} = \text{Ce}^{3+} + 4\text{H}^+ + \text{CrO}_2\downarrow E^\theta = 0.24\text{V} \\
6\text{Ce}^{4+} + 2\text{Cr}^{3+} + 7\text{H}_2\text{O} = 6\text{Ce}^{3+} + 14\text{H}^+ + 2\text{CrO}_2\downarrow E^\theta = 0.36\text{V} \\
2\text{Ce}^{4+} + \text{NH}_4^+ + \text{H}_2\text{O} = 2\text{Ce}^{3+} + \text{NH}_3\text{OH}^+ + 2\text{H}^+ E^\theta = 0.37\text{V} \\
\text{2Ce}^{4+} + 2\text{NH}_4^+ = 2\text{Ce}^{3+} + \text{N}_2\text{H}_5^+ + 3\text{H}^+ E^\theta = 0.445\text{V} \\
4\text{Ce}^{4+} + 2\text{H}_2\text{O} = 4\text{Ce}^{3+} + \text{O}_2\uparrow + 4\text{H}^+ E^\theta = 0.491\text{V} \\
\]

(11)

It can be seen from the standard electromotive force of each reaction that all impurity ions and water can reduce tetravalent cerium to trivalent cerium, but it can be seen that the extraction rate of Ce⁴⁺ solution without impurities in Figure 7 is stable with the standing time. Although water can reduce tetravalent cerium to trivalent cerium, the reduction rate is very slow. When the studied impurity ions exist in the studied concentration, the impurity ions may participate in the reaction and catalyze the reaction between water and tetravalent cerium. The Ce⁴⁺ in the solution is reduced to Ce⁵⁺ in a relatively short time, which affects the extraction of 0.05 mol·L⁻¹ TiAP to 0.005 g·L⁻¹ cerium solution.

4. Conclusion

(1) The extraction rate of 0.005 g·L⁻¹ Ce(IV) solution with 0.05 mol·L⁻¹ TiAP/n-dodecane can reach over 92% under the condition of O/A 1:1 and contact time 90 s

(2) On the basis of single-stage extraction, the extraction system was extracted in a 16-stage mixer-settler. After 16-stage countercurrent extraction, the extraction rate of 0.005 g·L⁻¹ Ce(IV) solution with 0.05 mol·L⁻¹ TiAP/n-dodecane extractant reached more than 99.45%

(3) The extraction rate of 0.05 mol·L⁻¹ TiAP/n-dodecane with 0.005 g·L⁻¹ Ce(IV) solution which containing Fe³⁺ (0.01 g·L⁻¹), Cr³⁺ (0.005 g·L⁻¹), Ni²⁺ (0.008 g·L⁻¹), Cu²⁺ (4 g·L⁻¹), and nitric acid (2 mol·L⁻¹) is about 64%

(4) The extraction effect of 0.05 mol·L⁻¹ TiAP/n-dodecane on 0–0.005 g·L⁻¹ Ce⁴⁺ is not obvious

(5) Due to the strong oxidizing properties of Ce⁴⁺, the water and Ce⁴⁺ in the solution are undergoing a slow oxidation-reduction reaction. When the impurity ions under study exist in the solution at the concentration under study, Ce⁴⁺ is reduced to Ce⁵⁺ in a relatively short time, which affects the extraction of 0.005 g·L⁻¹ cerium solution by 0.05 mol·L⁻¹ TiAP/n-dodecane

In summary, 0.05 mol·L⁻¹ TiAP has a better extraction effect on 0–0.005 g·L⁻¹ Ce⁴⁺, but not obvious extraction effect on 0–0.005 g·L⁻¹ Ce⁵⁺; TiAP as an excellent extractant of lanthanum actinides, perhaps later studies can be carried out to use the valence change of cerium to affect the extraction effect of TiAP on Ce and to realize the separation of cerium from other lanthanum actinides at a trace level.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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