Croatian Society
for Materials
Protection

*Metalurgija/Metallurgy*

**Vol. 65 No. 3 / 2026**

# Preparation of Aluminum-Based Lithium Ion Adsorbent Supported on Activated Carbon and its Adsorption Lithium Experiment from Sodium Aluminate Solution

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https://doi.org/10.64486/m.65.3.6

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***Type of the Paper:*** Article
***Received:*** August 20, 2025
***Accepted:*** January 5, 2026

**Abstract:** Lithium-rich bauxite contains abundant associated lithium oxide resources and represents a largely untapped source of lithium. During the Bayer process, approximately 80 % of lithium enters the sodium aluminate solution. However, research on the efficient extraction of lithium from sodium aluminate solutions remains limited due to the lack of low-cost and high-efficiency technologies. In this study, a novel activated carbon–supported aluminum-based lithium-ion adsorbent (C-Li/Al-LDHs) was developed, and its lithium adsorption performance in sodium aluminate solution was systematically investigated. The results show that C-Li/Al-LDHs can effectively adsorb lithium from the solution. The adsorption efficiency increased with longer adsorption time, higher adsorbent dosage, elevated adsorption temperature, and higher lithium concentration. The key factors affecting the adsorption process were systematically examined, and a maximum lithium adsorption efficiency of 77.09 % was achieved under optimal conditions: an adsorbent dosage of 15 g/L, an adsorption time of 2 h, a water bath temperature of 70 °C, and a lithium concentration of 80 mg/L.

**Keywords:** lithium-rich bauxite; sodium aluminate solution; activated carbon–supported aluminum-based lithium-ion adsorbent; adsorption; lithium recovery

## 1. Introduction

Lithium, often referred to as the "green energy metal" and "white oil" of the 21st century, is a strategically critical metal globally. China currently holds proven lithium reserves of 6.8 million tons, ranking sixth worldwide [1]. However, due to the inherent characteristics of China's lithium resources and environmental factors associated with their locations, the country heavily relies on lithium imports [2]. While recovering lithium from waste lithium batteries is a feasible approach [3-5], it faces challenges such as high costs and secondary pollution [6]. Therefore, exploring new lithium resources is imperative. The bauxite resources in the Wuchuan, Zhengan, and Daozhen areas of northern Guizhou Province, China, are exceptionally rich in lithium [7,8]. Over 65 % of large bauxite samples exhibit lithium content meeting the comprehensive utilization index (Li > 260 × $10^{-6}$) [9], with 30 % of samples containing lithium levels exceeding 1000 × $10^{-6}$ and an average lithium content of 756 × $10^{-6}$ [10-12]. The lithium content is an approximate 2.5 times higher than the average lithium content in Chinese bauxite [13]. The associated lithium oxide ($Li_2O$) resource reserves in this region are estimated at approximately 1.82 million tons [14]. In the Bayer process, bauxite dissolution produces a pulp that is settled to obtain crude sodium aluminate, which is then processed through leaf filtration to yield purified sodium aluminate. This solution is subsequently cooled to produce aluminum hydroxide. Approximately 80 % bauxite-associated Li enters into the sodium aluminate solution during the Bayer process. This solution is subsequently cooled to produce aluminum hydroxide. Fresh $Al(OH)_3$ acts as an effective adsorbent, capable of purifying metal impurity elements, including lithium [15]. Studies indicate that under specific conditions, the adsorption rate of lithium in $NaAl(OH)_4$ solution by $Al(OH)_3$ exceeds 80 % [16]. Therefore, concentrating and extracting lithium from $NaAl(OH)_4$ solution should be prioritized before extensive aluminum hydroxide crystallization occurs during cooling and seeding. The narrow temperature control range increases the complexity of lithium extraction technology. Due to these challenges, research in this field is limited. Aluminum-based lithium adsorbents, especially for Li/Al layered double hydroxides, have been employed in industrial lithium extraction due to their high selectivity, rapid adsorption/desorption, and water elution for $Li^+$ in salt lake brine [17]. During the lithium adsorption process, Li+ ions preferentially occupy the octahedral void positions in layered Al(OH)3 due to size compatibility. In contrast, other ions are less likely to be adsorbed through the pores owing to significant steric hindrance [18,19]. However, the immobilization characteristics and cycle stability of these adsorbents require further optimization. In this work, activated carbon-supported aluminum-based lithium ion adsorbent (C-Li/Al-LDHs) was developed. Its adsorption performance for extracting lithium from sodium aluminate solution was systematically investigated.

## 2. Materials and Methods

First, hydrochloric acid (1 mol/L) was slowly added to the sodium aluminate solution to adjust its pH to neutral, producing aluminum hydroxide colloids. Granular activated carbon (GAC) was then immersed in hydrochloric acid (1 mol/L) at a liquid-solid ratio of 3:1, stirred, and soaked for 6 hours. After acid treatment, the GAC was washed with deionized water until neutral and dried at 120 °C to constant weight. The GAC particle size was controlled between 40 and 80 mesh to obtain the activated carbon carrier. The activated carbon carrier was added to the aluminum hydroxide sol at a molar ratio of 1:5, stirred for 2 hours, aged at 40 °C for 24 hours, washed with deionized water, and dried at 105 °C for 24 hours to yield the activated carbon-supported aluminum hydroxide adsorption material. Finally, a portion of this material was placed in high-purity water (solid content: 10-50 g/L), heated to 50 °C, and mixed with an equivalent weight of $LiCl \cdot H_2O$. After 24 hours of continuous stirring, the mixture was filtered, washed, dried at 105 °C for 24 hours, and crushed to a particle size of 0.5 mm – 2 mm to obtain the activated carbon-supported aluminum-based lithium ion adsorbent (C-Li/Al-LDHs). Its main components are activated carbon and $LiCl \cdot nAl(OH)_3 \cdot mH_2O$, where $m$ = 2–10 and $n$ = 0.5–10, representing the stoichiometric ratios. Particle sizes (0.5 mm – 2.5 mm) > 95 %, water content < 5 %. The surface morphology of the adsorbent was observed using a scanning electron microscope (SEM, model JSM-7610F, Nippon Electronics). The X-ray diffraction (XRD) pattern of the C-Li/Al-LDHs was recorded using an XRD-6100 diffractometer (Shimadzu, Japan).

A measured amount of lithium hydroxide was dissolved in sodium aluminate solution, heated, and diluted to a fixed volume. The solution was mixed with C-Li/Al-LDHs and stirred in a constant-temperature water bath. The adsorbed solution was filtered, and lithium content was determined using inductively coupled plasma mass spectrometry (ICP-MS, Agilent 7500a, USA).

## 3. Results

### *3.1. XRD and SEM analysis*

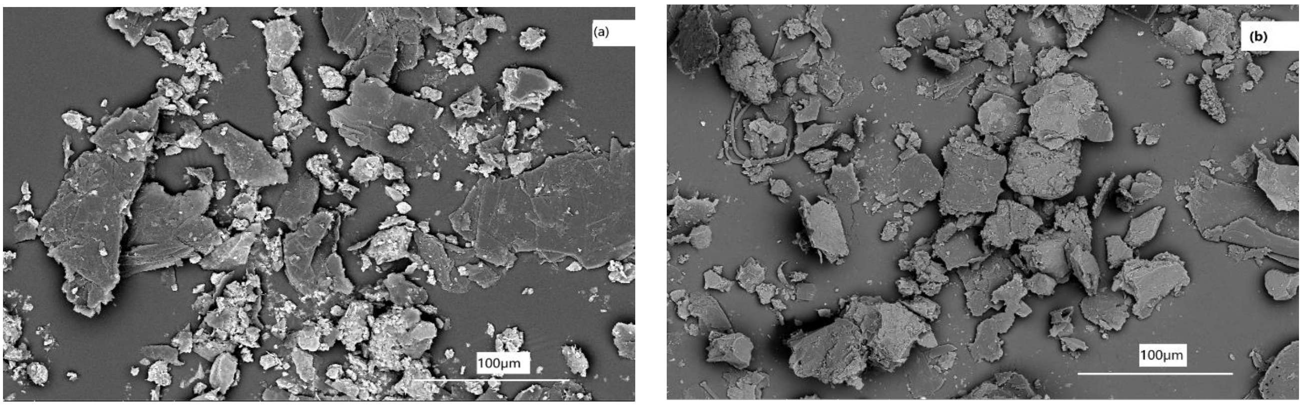

**Figure 1.** SEM images of C-Li/Al-LDHs: (a) before adsorption; (b) after adsorption.

Figure 1 shows the SEM image of the micro-morphology of C-Li/Al-LDHs. As can be seen from Figure 1. before adsorption the adsorbed materials were all irregular massive crystals with different sizes, with irregular micropore morphology and large specific surface area, which is conducive to promoting adsorption. After adsorption, the surface morphology of the adsorbent did not change significantly, and it was smooth and flat after adsorption. Indicating that the structure of the adsorbent was stable during the adsorption process.

XRD parameters: Starting Angle 10°, Stop Angle 80°, Increment 0.02°, tube voltage 40 kV, tube current 30 mA. Figure 2 shows the XRD pattern of C-Li/Al-LDHs before and after adsorption. It can be discerned from Figure 2 that C-Li/Al-LDHs exhibits a characteristic peak at 26.5°, which is the peak of C. It can thus be inferred that the main component of C-Li/Al-LDHs is C and LiCl·nAl$(OH)_3$·$mH_2O$. the position of the main characteristic peaks of the sample basically did not change before and after adsorption, indicating that its structure was relatively stable.

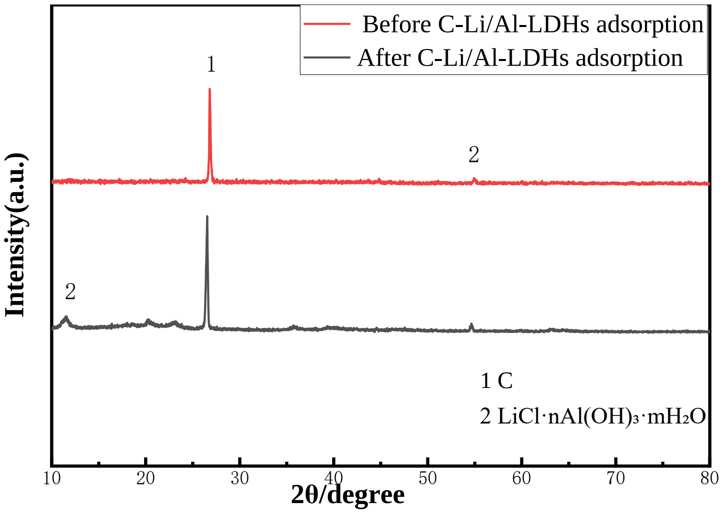

**Figure 2.** XRD of C-Li/Al-LDHs

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### *3.2. Selection of lithium adsorbent*

Amorphous aluminum hydroxide has an efficient selective precipitation for lithium from salt-lake brines. Therefore, some researchers used active $Al(OH)_3$ as the lithium adsorbent in $NaAl(OH)_4$ solution through several times adsorption, redissolution and concentration. Lithium solution prepared with pure water and $Li_2O{\cdot}H_2O$ were used for testing adsorption properties of alumina hydroxide, C-Li/Al-LDHs adsorbent. The study was investigated at a set temperature of 70 °C, lithium solution 80 mg/L and adsorbent of 10 g/L for 10 hours. Adsorption efficiency was calculated using equation (1):

$$\varepsilon_{AD}(\%) = \left(\frac{C_0 - C}{C_0}\right) \times 100 \tag{1}$$

Where, $\varepsilon_{AD}$ (%) is the adsorbent adsorption efficiency, $C_0$ and $C$ are the initial and final lithium concentrations (mg/L) in the adsorption test, respectively.

Adsorption capacity was calculated using equation (2):

$$Q_e = \frac{(C_0 - C) \times V}{m} \tag{2}$$

Where, $Q_e$ (mg/g) is the adsorption capacity, $V$ (L) represents the volume of the adsorption solution, $m$ is the mass of the adsorbent.

The experimental results show that after adsorption by dried aluminium hydroxide, the solubility of Li solution is 19.02 mg/L and the adsorption efficiency is 76.23 %, while after adsorption by C-Li/Al-LDHs, the solubility of Li solution is 14.97 mg/L and the adsorption efficiency is 81.29 %, which indicates that C-Li/Al-LDHs can adsorb lithium better than dried alumina hydroxide.

### *3.3. Effect of different solution systems on the adsorption efficiency of lithium adsorbent*

The effect of different solution systems on the adsorption efficiency of lithium adsorbent was investigated. The test conditions were as follows: adsorbent 30g/L, water bath temperature 70 °C, lithium concentration 80 mg/L and adsorption time of 10 h. Dried aluminum hydroxide and C-Li/Al-LDHs were used as adsorbents, and lithium solutions prepared with sodium hydroxide and sodium aluminate were employed to conduct multiple adsorption experiments. The experimental data are presented in Table 1, which shows that the adsorption efficiency of C-Li/Al-LDHs in different solution system is higher than that of aluminum hydroxide. In lithium solutions prepared with sodium hydroxide, the adsorption efficiencies of both adsorbents are higher than those observed in lithium solutions prepared with sodium aluminate, but lower than those obtained in single lithium solutions. This phenomenon can be attributed to the fact that sodium and lithium ions are both monovalent cations and therefore compete for adsorption sites. The adsorption efficiencies of both adsorbents decreased in lithium solutions containing sodium aluminate, which may be due to the complexity of the sodium aluminate system. The presence of sodium ions, as well as gallium, iron, potassium, calcium, and magnesium ions and organic matter, can interfere with the adsorption of lithium ions by the adsorbent. Based on these results C-Li/Al-LDHs was selected as the adsorbent for subsequent single factor experiments.

**Table 1 Adsorption efficiency of adsorbent**

| | SH / % | | | SAH / % | | |
|---|---|---|---|---|---|---|
| C-Li/Al-LDHs | 79.68 | 79.89 | 79.36 | 78.33 | 78.59 | 78.65 |
| Dried aluminum hydroxide | 76.65 | 75.98 | 76.40 | 75.03 | 75.59 | 75.35 |

*Note: SH represents the adsorption efficiency in lithium solutions prepared with sodium hydroxide; SAH represents the adsorption efficiency in lithium solutions prepared with sodium aluminate.*

### *3.4 Effect of adsorption time on adsorption efficiency*

https://doi.org/10.64486/m.65.3.6

The test conditions were as follows: adsorbent dosage of 30 g/L, water bath temperature of 70 °C, and lithium concentration of 80 mg/L. Figure 3 shows the effect of adsorption time on adsorption efficiency. The adsorption efficiency increased significantly during the initial stage from 0.5 h to 2 h. When the adsorption time was within this range, the lithium adsorption rate increased markedly with increasing adsorption time. At 0.5 h, the lithium concentration in solution after adsorption was 39.81 mg/L, corresponding to an adsorption efficiency of 50.24 %. When the adsorption time was extended to 2 h, the lithium concentration decreased to 18.02 mg/L, and the adsorption efficiency increased to 77.48 %, representing an increase of 27.24 % This behavior can be attributed to the gradual occupation of adsorption sites by $Li^+$ ions over time, leading to the establishment of a dynamic adsorption equilibrium between the solid and liquid phases [20]. When the adsorption time exceeded 2 h, further extension resulted in only minor changes in adsorption efficiency, indicating that equilibrium had been reached. According to the adsorption capacity equation, the adsorption capacity at equilibrium was 2.06 mg/g.

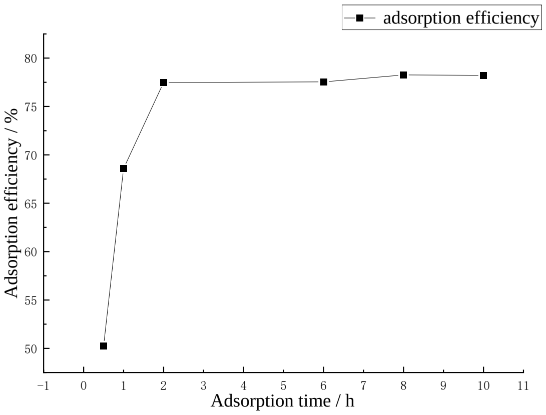

**Figure 3.** Influence of adsorption time on adsorption efficiency

*3.5 Effect of adsorbent concentration on adsorption efficiency*

The adsorption efficiency of adsorbent was shown in Figure 4 under different adsorbent concentration. Test conditions: 2-hour adsorption time, 70 ℃ water bath temperature and 80 mg/L lithium solution concentration. As observed from Figure 4 that the adsorption efficiency increases with the increase of the adsorbent concentration. When the amount of adsorbent from 5 g/L to 15 g/L, the lithium solubility changed from 45.43 mg/L to 18.33 mg/L after adsorption, the adsorption efficiency was from 43.21 % to 77.09 %, the adsorption efficiency increases rapidly. When the amount of adsorbent was increased to 30 g/L, the adsorption efficiency was only slightly increased. The reason for this phenomenon is that when the amount of adsorbent added is small, only part of the lithium in the solution can be adsorbed. When the amount of adsorbent added increases, the surface free hydroxyl group available for lithium ion adsorption increases, and the adsorption efficiency increases rapidly. When the amount of adsorbent added increases to a certain extent, the adsorption efficiency is basically stable, and the adsorption efficiency changes little when the amount of adsorbent added continues to increase.

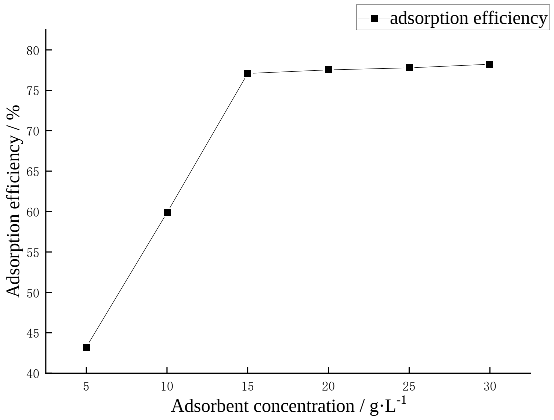

**Figure 4.** Influence of adsorbent concentration on adsorption efficiency

### *3.6 Effect of adsorption temperature on adsorption efficiency*

Test conditions: 15 g/L adsorbent dosage, 2 hour adsorption time and 80 mg/L lithium solution concentration. Figure 5 shows the effect of adsorption temperature on adsorption efficiency. As observed from Figure 5. When the temperature is increased from 50 °C to 70 °C, the lithium solubility changed from 40.93 mg/L to 18.33 mg/L, adsorption efficiency increases from 48.84 % to 77.09 %. This phenomenon is due to the slow movement of $Li^+$ towards the layered Al(OH)3 octahedral vacancy at low temperature, and the $Li^+$ concentration difference is the main driving force in the solution. Higher temperature can not only accelerate the diffusion rate of $Li^+$ inside the adsorbent, but also accelerate the utilization of the active site inside the adsorbent [21]. When the water bath temperature is 90 °C, the adsorption efficiency was 74.81 %, which is 2.28 % lower than that at 70 ℃. The phenomenon is due to lithium redissolving in solution at high temperatures.

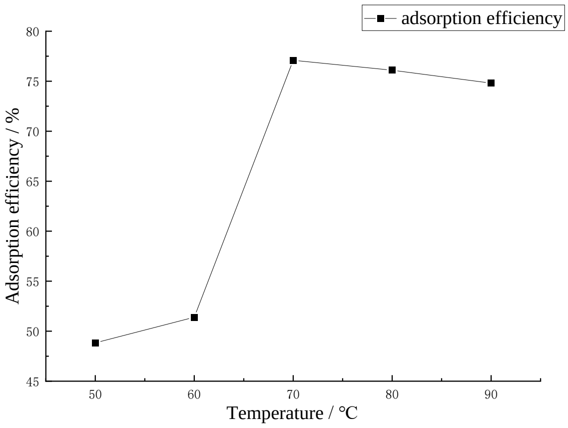

**Figure 5.** Influence of adsorption temperature on adsorption efficiency

### *3.7 Effect of lithium solution concentration on adsorption efficiency*

Test conditions: 15 g/L adsorbent dosage, 2-hour adsorption time and 70 ℃ water bath temperature. Figure 6 shows the effect of lithium solution concentration on adsorption efficiency. When the concentration of lithium solution is 10 mg/L, the adsorption efficiency amounts to 44.90 %. The adsorption efficiency is low, which is due to the low lithium content in the solution, the small concentration difference between the surface and layer of the adsorbent and the $Li^+$ in the solution, so the driving force in the adsorption process is small, and the lithium adsorption efficiency is low. When the concentration of lithium solution increases, the concentration difference between the surface and layer of the adsorbent and $Li^+$ is large. Driven by the concentra-

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tion difference of $Li^+$, the adsorbent can quickly occupy the adsorption sites in the adsorbent, and the adsorption efficiency increases [21]. When the concentration of lithium solution is 40 mg/L, the adsorption efficiency amounts to 76.45 %, which increases by 31.55 %. When the concentration of lithium solution continues to increase to 80 mg/L, the adsorption efficiency amounts to 77.09 %, which only increases by 0.65 %. This phenomenon indicates that with the adsorption process, a certain amount of aluminum-based lithium adsorbent has approached adsorption saturation, the concentration of $Li^+$ in the solution decreases, and the difference in the adsorption amount of $Li^+$ also decreases, and $Li^+$ reaches dynamic equilibrium in the two phases [21].

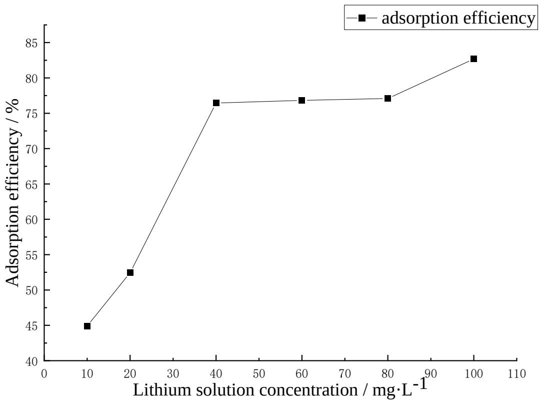

**Figure 6.** Influence of lithium solution concentration on adsorption efficiency

## 4. Discussion

This study systematically investigates the influence of various adsorption parameters on the performance of adsorbents. By comparing experimental results with those reported in the literature, it is demonstrated that C-Li/Al-LDHs exhibit significantly higher lithium adsorption capacity compared to dry aluminium hydroxide. Appropriately increasing the adsorption time can effectively enhance the adsorption efficiency and ensure that the adsorbent is fully adsorbed. With regard to adsorbent concentration, increasing the concentration can enhance the number of free hydroxyl groups in the solution, thereby improving the adsorption efficiency. Additionally, elevated temperatures accelerate the diffusion rate of lithium ions within the adsorbent, further enhancing adsorption efficiency. Characterization via XRD and SEM revealed that the surface of the adsorbent was in a layered state, which increased the specific surface area of the adsorbent and thereby improved the adsorption performance. However, the main framework structure of the adsorbent remains unchanged after adsorption, indicating that the properties of the adsorbent remain the same before and after use, and its structure was relatively stable.

The activated carbon-supported adsorbent used in this study was prepared with sodium aluminate solution, which effectively reduced its production cost and had high stability. Its adsorption efficiency was higher than that of dried aluminum hydroxide. This study lays the foundation for subsequent research on lithium adsorption. Future research can focus on exploring the influence of other adsorption parameters on adsorption efficiency, as well as its dissolution loss and the recycling property.

## 5. Conclusions

The adsorption experiments with different adsorbents show that the adsorption efficiency of C-Li/Al-LDHs for lithium ions in pure water was 81.29 %, in sodium aluminate solution amounts to 78.52 %. The analysis of C-Li/Al-LDHs adsorbent through single factor test shows that the adsorption efficiency of the matiral was facilitated by, longer reaction times, elevatede reaction temperatures, and more adsorbent addition amounts, and higher lithium solution concentration. The optimal adsorption conditions were: adsorbent 15 g/L, adsorption time 2 h, temperature 70 ℃, lithium solution concentration 80 mg/L, and the adsorption efficiency amounts to 77.09 % under these conditions.

**Acknowledgments:**This work was supported by the National Natural Science Foundation of China (51664010), the Guizhou Provincial Science and Technology Projects (Qiankehe support normal [2023] No.243，Science and Technology Foundation of Guizhou Province ([2018]0002), and High-level Talents Foundation of Guizhou Institute of Technology (XJGC20181010).

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