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Study on physicochemical properties and basicity of carbanion-functionalized ionic liquids

Xian-Lu Wu, Xiao-Yan Sang, Zhang-Min Li *, Duan-Jian Tao *

College of Chemistry and Chemical Engineering, Jiangxi Normal University, Nanchang 330022, China

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ABSTRACT

Densities, viscosities, refractive indices and conductivities of four carbanion-functionalized ionic liquids (ILs) were measured in the temperature range of 298.2–348.2 K. The effect of temperature on these physicochemical properties were also studied. The results showed that the values of density and refractive index decreased linearly but the conductivity increased exponentially with the increase of temperature, while the viscosity decreased exponentially with the temperature increased. Moreover, the conductivities of phosphonium carbanion-functionalized ILs were much lower than common phosphonium amino acid ILs because of high ionic association and poor ion mobility of carbanion. In addition, the characterization of FT-IR and ^{1}H NMR by using pyrrole as a probe molecule showed that [P_{4448}][Dib] exhibited the strongest basicity among these four carbanion-functionalized ILs.

1. Introduction

Ionic liquids (ILs), which possess many specific properties such as designable ability, negligible vapor pressure, high thermal and chemical stability, and excellent solubility, are studied and used in many fields such as gas absorption, organic catalysis, electrolytes, and biomass conversion in recent years [1–5]. Carbon anion, as being an important organic intermediate and nucleophile, is usually involved in many organic reactions such as Michael addition and Knoevenagel condensation [6,7]. Because of the active unstable nature of carbanion anion, it is very attractive and challenging to prepare ionic liquids composed of carbanions. In 2017, we had reported a pioneering work that four new carbanion-functionalized ILs were successfully synthesized in one step from neutralization of tributyl(octyl)phosphonium hydroxide with various β-diketones. These carbanion-based ILs exhibited ultrahigh capacity for absorption of carbon monoxide [8]. Therefore, to study the potential of such carbanion-functionalized ILs to be applied in various fields of chemical industry, the physicochemical properties of carbanion-based ILs are very important for analysis. It is highly worth to study their physicochemical properties including density, viscosity, refractive index, and conductivity, etc.

On the other hand, the basicity and the acidity of ILs are important property, in which affect their catalytic or gas absorption performance. Methods for characterization of basicity and acidity are usually including potentiometric titration [9], FT-IR using pyridine (pKa = 3.4 in DSMO) as a probe molecule [10], and ^{1}H NMR using pyrrole (pKa = 23.0 in DMSO) as a probe molecule [11]. For example, Nachtigall [12] studied basic sites in ZSM-5 through Fourier transform infrared spectroscopy (FT-IR) using pyrrole as a probe molecule, and Blasco [13] characterized the basicity of zeolite basicity by NMR using pyrrole as a probe molecule. Therefore, it is worthwhile using these two techniques to investigate the basicity of carbanion-functionalized ILs.

In this work, four carbanion-functionalized tributyl(octyl)phosphonium ILs ([P_{4448}][Pen], [P_{4448}][Mho], [P_{4448}][Ido], [P_{4448}][Dib]) were designed and prepared by neutralization of tributyl(octyl)phosphonium hydroxide with four β-diketone compounds (Fig. 1), and their physicochemical properties (density, viscosity, refractive index, and conductivity) were determined in the temperature range of 298.2–348.2 K. These properties were further correlated with thermodynamic and empirical equations. In addition, the basicity of these four carbanion-functionalized ILs were characterized by FT-IR and ^{1}H NMR using pyrrole as probe molecule.

2. Experimental

2.1. Materials

Tributyl(octyl)phosphonium bromide ([P_{4448}][Br], ≥98%) was obtained from the Centre of Green Chemistry and Catalysis, LICP, CAS. An anion-exchange resin (DowexMonosphere 550A (OH)) was purchased from Dow Chemical Company. Acetylacetone (Pen, ≥99%), dimedone (Mho, ≥99%), 1,3-indanedione (Ido, ≥98%), and dibenzoylmethane (Dib, ≥98%) were purchased from Macklin Biochemical Co. Ltd. (Shanghai, China). Pyrrole and other reagents were all analytical grade and used directly without any further purification.

* Corresponding authors.
E-mail addresses: zmli@jxnu.edu.cn (Z.-M. Li), djtao@jxnu.edu.cn (D.-J. Tao).

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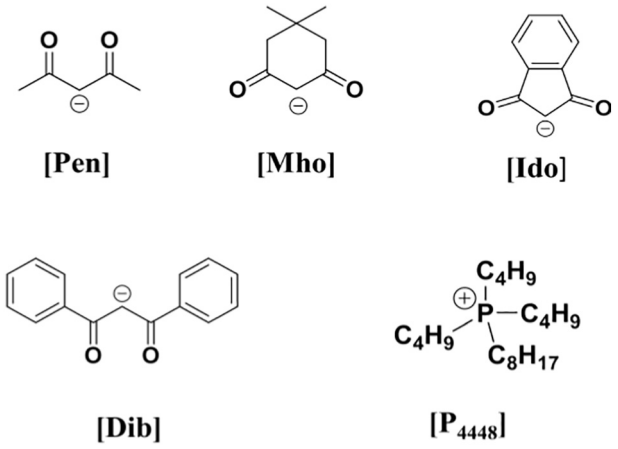


Fig. 1. Structures of the anions and cation in four carbanion-functionalized ILs.

2.2. Preparation and characterization of carbanion-functionalized ILs

The synthesis of carbanion-functionalized ILs had been described in our previous work [8]. Shortly, tributyl(octyl)phosphonium hydroxide ([P_{4448}][OH]) in ethanol was obtained from [P_{4448}][Br] using the anion-exchange resin DowexMonosphere 550A (OH). Then four β-diketones were neutralized with a solution of [P_{4448}][OH] in ethanol for 24 h in a molar ratio of 1:1, respectively. After the completion of reaction, the as-prepared carbanion-functionalized ILs were dried in high vacuum for 48 h at 333.2 K to remove possible traces of ethanol and water. Then the purities of these four carbanion-functionalized ILs were found to be >99.2 wt % confirmed by ^{1}H and ^{13}C NMR, and the results are shown in Figs. S1–S8 in the Supporting Information. The water content of these carbanion-functionalized ILs was measured to be less than 0.15 wt% by the Karl Fisher titration. The concentration of Br^- measured by Mohr titration was less than 0.02 wt%.

2.3. Determination of physicochemical properties and basicity

The densities and viscosities of these four carbanion-functionalized ILs were measured by an Anton Paar densimeter (model DMA4500) and a cone-plate viscometer (Brookfield DV II + Pro) over the temperature range from 298.2 to 353.2 K with a temperature control of ±0.1 K. Refractive indices were recorded by using an Abbe refractometer (WYA-2S, Shanghai Shenguang Instrument Co., Ltd.) at temperatures from 298.2 to 343.2 K with a temperature precision of ±0.1 K. The equipment is calibrated before each series of measurements and checked using distilled water with a known refractive index. Conductivity was carried out by a conductivity meter (DDJS-308A, Shanghai Leici Company) with a DJS-1C electrode over temperatures range from 298.2 K to 343.2 K. Take care to prevent evaporation, the electrode and the solution were sealed in a typical glassware. In order to obtain the basicity of carbanion-functionalized ILs, the mixture of a carbanion IL with equimolar probe molecular pyrrole was characterized by nuclear magnetic resonance (Bruker Ascend 400) and Fourier transform infrared (NexuS870). Furthermore, the standard uncertainty of data repeatability, IL purity, testing temperature, and instrument precision were evaluated to estimate the combined expanded uncertainty of each measured properties at a 0.95 level of confidence ($k \approx 2$).

3. Results and discussion

3.1. Density

Fig. 2 shows the effect of temperature on the densities of four carbanion-functionalized ILs. It was demonstrated that the density values of these ILs decreased linearly with the increase of temperature, and the order was as follows: [P_{4448}][Ido] > [P_{4448}][Dib] > [P_{4448}][Mho] > [P_{4448}][Pen]. It was showed that the carbanion-functionalized ILs with aromatic rings in the anions may have a relatively high density [14], high molecular weight anion probably enhanced the density values for the present carbanion-functionalized ILs [15]. However, [P_{4448}][Dib] did not perform the biggest density, which is due to its largest molecular volume.

3.2. Viscosity

Fig. 3 shows the effect of temperature on the viscosities of carbanion-functionalized ILs. The results indicated that the viscosity values of these carbanion-functionalized ILs decreased with the temperature increased, and the viscosity order was as follows: [P_{4448}][Ido] > [P_{4448}][Mho] > [P_{4448}][Pen] > [P_{4448}][Dib]. Interestingly, [P_{4448}][Dib] performed the lowest viscosity, although it has the highest molecular weight. It is known from the previous reported litertures [16] that a relatively large volume of anion in the IL can weaken the interaction between the cation and anion, and thus leads to lower viscosity.

3.3. Refractive index

The influence of temperature on refractive index was shown in Fig. 4. It was observed that the improvement of temperature had an obvious decrease in the value of refractive index and the refractive index was linearly decreasing with the increase of temperature. [P_{4448}][Dib] was found to have the highest refractive indexes, while [P_{4448}][Mho] exhibited the smallest refractive indexes. This finding demonstrated that the order of refractive indexes was not only related to the anionic molecular weight, but also relied on the molecular polarizability of ion pairs per volume on the basis of the Lorentz–Lorenz equation [17].

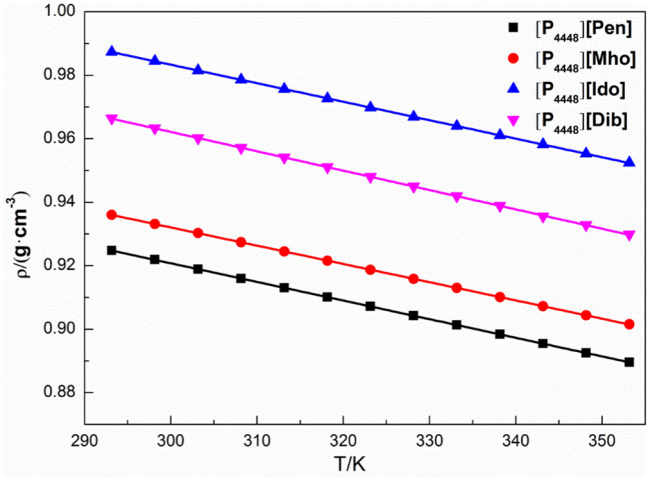


Fig. 2. Density ρ as a function of temperature. The relative standard uncertainties u are $u(T) = 0.1$ K, and the combined expanded uncertainty U_c is $U_c(\rho) = 0.012$ g·cm^{-3} with a 0.95 level of confidence ($k \approx 2$).

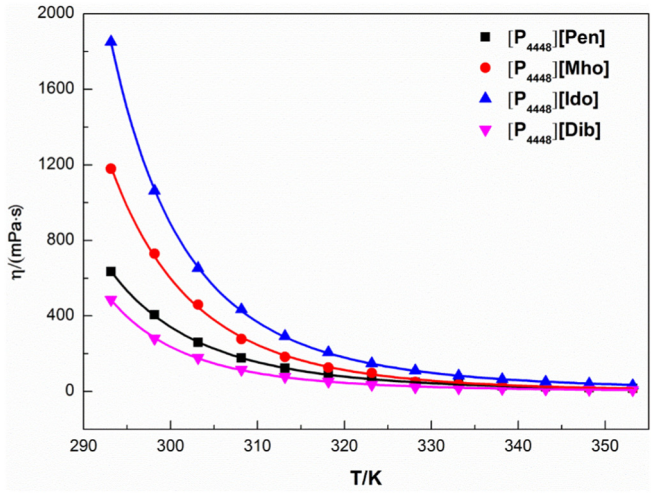


Fig. 3. Viscosity η as a function of temperature. The relative standard uncertainties u are $u(T) = 0.1$ K, and the combined expanded uncertainty U_c is $U_c(\eta) = 1.1$ mPa·s with a 0.95 level of confidence ($k \approx 2$).

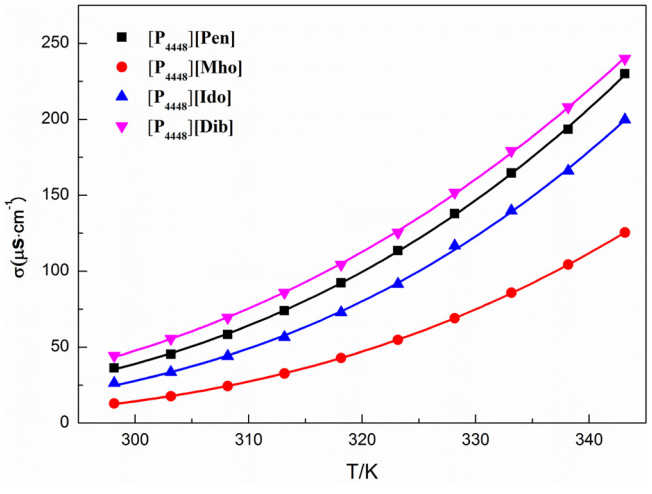


Fig. 5. Conductivity σ as a function of temperature. The relative standard uncertainties u are $u(T) = 0.1$ K, and the combined expanded uncertainty U_c is $U_c(\sigma) = 0.3$ with a 0.95 level of confidence ($k \approx 2$).

3.4. Conductivity

Fig. 5 shows the effect of temperature on conductivity. It was demonstrated that the temperature had a positive influence on the value of conductivity. It is validated from the literatures [18] that a lower viscosity often leads to higher ionic mobility of ILs. Thus, [P_{4448}][Dib] showed the highest conductivity owing to its lowest viscosity among these four carbanion-functionalized ILs. [P_{4448}][Mho] possessed less charge density in the anion [Mho], contributing to a relatively low electrical conductivity.

3.5. Basicity

It is well known that the N—H group in pyrrole has a very weak acidity (pKa = 23.0 in DMSO). Thus, pyrrole was employed as a probe molecular to test the basicity of these carbanion-functionalized ILs. Fig. 6 shows the FT-IR spectra of carbanion-functionalized ILs, pyrrole, and the mixture of pyrrole with equimolar carbanion-functionalized ILs, respectively. It was found that a characteristic peak was observed at 3400 cm^{-1}, which ascribes to the stretching vibration of N—H group in pyrrole. Moreover, the characteristic peak of 3400 cm^{-1} disappeared in the sample of carbanion ILs + pyrrole. This finding indicates that these four carbanion-functionalized ILs can efficiently interact with the N—H group in pyrrole, showing the nature of super base.

Furthermore, ^{1}H NMR spectra of pyrrole and carbanion-functionalized ILs + pyrrole was performed and shown in Fig. S9. Table 1 also lists the chemical shift of ^{1}H NMR for the N—H group in pyrrole. The results demonstrated that all the ^{1}H NMR signal of N—H shifted to lower magnetic fields, when pyrrole was added to each carbanion-functionalized IL. The more ^{1}H NMR shift of N—H group is, the stronger alkalinity the IL has. The basicity order of carbanion-functionalized ILs is as follows: [P_{4448}][Dib] > [P_{4448}][Pen] > [P_{4448}][Mho] > [P_{4448}][Ido], and [P_{4448}][Dib] was demonstrated to have the strongest basicity among these carbanion-functionalized ionic liquids.

3.6. Empirical correlation

Based on the above results, the density (ρ), viscosity (η), refractive index(n_D), and conductivity (σ) for the present carbanion-

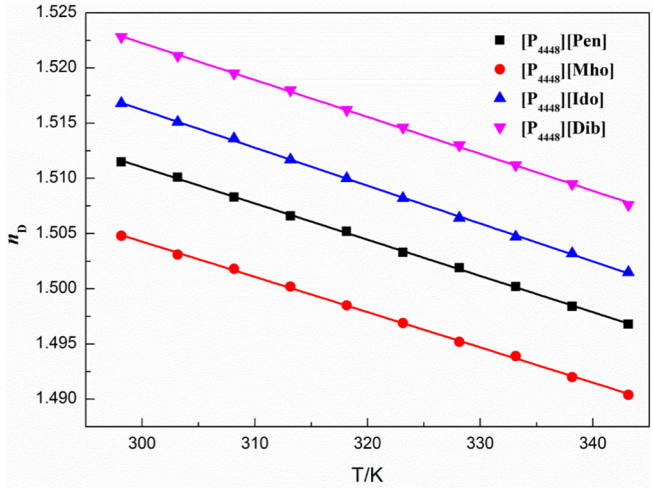


Fig. 4. Refractive index n_D as a function of temperature. The relative standard uncertainties u are $u(T) = 0.1$ K, and the combined expanded uncertainty U_c is $U_c(n_D) = 0.0119$ with a 0.95 level of confidence ($k \approx 2$).

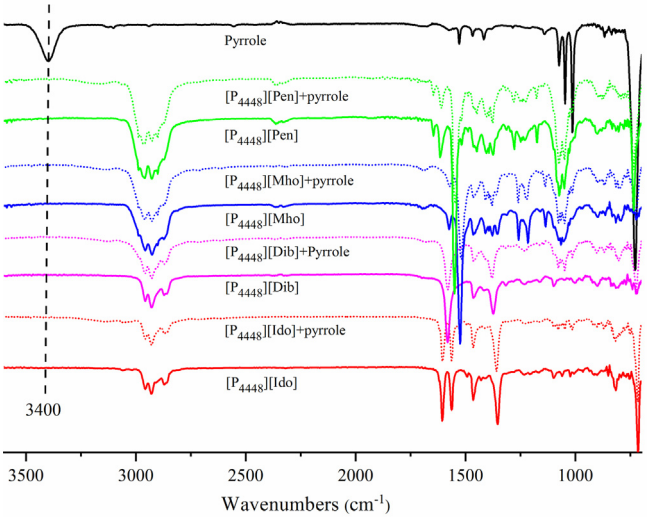


Fig. 6. FT-IR spectra of carbanion-functionalized ILs, pyrrole, and carbanion-functionalized ILs + pyrrole.

Table 1
^{1}H NMR chemical shifts of pyrrole as a probe molecular in carbanion-functionalized ILs.

Samples	Pyrrole	[P_{4448}][Dib] + pyrrole	[P_{4448}][Pen] + pyrrole	[P_{4448}][Mho] + pyrrole	[P_{4448}][Ido] + pyrrole
N-H/ppm	10.72	12.58	12.27	11.70	11.23
Δδ/ppm	–	1.86	1.55	0.98	0.51

functionalized ILs on temperature can be described by the empirical equations [21–23].

$$\rho = A_1 + A_2 T \tag{1}$$

$$\eta = A_3 \cdot \exp\left(\frac{A_4}{T - A_5}\right) \tag{2}$$

$$n_D = A_6 + A_7 T \tag{3}$$

$$\sigma = A_8 \cdot \exp\left(\frac{A_9}{T - A_{10}}\right) \tag{4}$$

where T is the Kelvin temperature and A_1, A_2, A_3, A_4, A_5, A_6, A_7, A_8, A_9 and A_{10} are fitting parameters. The correlation coefficients were estimated using linear regression analysis. The standard deviations (SD) are expressed as

$$SD = \sqrt{\sum_{i}^{N} (Z_{exp} - Z_{cal})^2 / N} \tag{5}$$

where Z_{exp} and Z_{cal} represent the experiment and calculated values and N are the number of the experimental dates. As seen from Tables S1 to S4, it was showed that all of the correlation coefficients were over 0.99. The relationship between temperature with viscosity and conductivity could be well fitted by the Arrhenius and Vogel-Tamman-Fulcher (VTF) equations. Furthermore, since the temperature–density relationship for these carbanion-functionalized ILs was linear, density values as a function of temperature were devoted to calculate the thermal expansion coefficient (α_p) (Table 2). The thermal expansion coefficients (α_p), also known as the volume expansion rate, are a function of temperature at atmospheric pressure, and its determination were as following Eq. (6):

$$\alpha = -\frac{1}{\rho}\left(\frac{\partial \rho}{\partial T}\right)_p - \frac{A_2}{A_1 + A_2 T} \tag{6}$$

where α_p, ρ, and T are the thermal expansion coefficient, density, and absolute temperature, respectively, and A_1 and A_2 are the fitting parameters of Eq. (1). As seen from Table 2, it was showed that the values of thermal expansion coefficient did not obviously changed as the temperature range from 298.2 to 353.2 K, and the averaged relative deviation of α_p was found to be less than 2%. Thus, the results showed that the thermal expansion coefficient of carbanion-functionalized ILs was almost independent of temperature, a similar phenomenon was observed for reported imidazolium, pyridinium, and ammonium-based ILs [24,25].

Table 2
Thermal expansion coefficient values of carbanion-functionalized ILs.

T/K	$\alpha_p \cdot 10^4/(K^{-1})$			
	[P_{4448}][Pen]	[P_{4448}][Mho]	[P_{4448}][Ido]	[P_{4448}][Dib]
293.2	6.359	6.134	5.960	6.205
298.2	6.380	6.153	5.978	6.224
303.2	6.400	6.172	5.995	6.243
308.2	6.421	6.191	6.013	6.263
313.2	6.441	6.210	6.032	6.283
318.2	6.462	6.230	6.050	6.302
323.2	6.483	6.250	6.068	6.322
328.2	6.504	6.269	6.087	6.342
333.2	6.525	6.289	6.105	6.363
338.2	6.547	6.308	6.124	6.383
343.2	6.568	6.328	6.143	6.403
348.2	6.590	6.348	6.162	6.423
353.15	6.611	6.369	6.181	6.444

3.7. Comparison with other phosphonium ILs

For comparison, we have further compared the physicochemical properties of carbanion-functionalized ILs with previous reported ILs, where all these ILs contain the phosphonium cation [8,14,19,20]. The results are listed in Table 3. It is found that compared with phosphonium amino acid ILs, the viscosity of phosphonium carbanion ILs had changed dramatically through the variation in anions. Moreover, the densities for these two kinds of phosphonium ILs were of similar values, but the conductivities of phosphonium carbanion ILs were much lower than those of phosphonium amino acid ILs. It may be because that phosphonium carbanion ILs has high ionic association and less charge density, resulting in poor ion mobility and thus low conductivity.

4. Conclusions

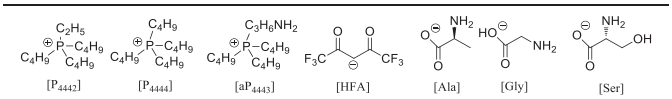
Four carbanion-functionalized ILs were prepared, and their densities, viscosities, refractive index, and conductivities were determined in the temperature range of 298.2–348.2 K. The results showed that the conductivities of phosphonium carbanion-functionalized ILs were much lower than common phosphonium amino acid ILs. The value of density and refractive index decreased linearly with increasing the temperature, while the conductivity improved exponentially but the viscosity reduced exponentially with the increase of temperature. Empirical equations could provide a good description of the experimental data of their physicochemical properties. The thermal expansion coefficients were found to be independent of temperature. In addition, [P_{4448}][Dib] exhibited the strongest basicity than the other three carbanion-functionalized ILs.

CRediT authorship contribution statement

Xian-Lu Wu: Data curation, Investigation, Writing - original draft. **Xiao-Yan Sang:** Methodology, Software. **Zhang-Min Li:** Writing -

Table 3
The comparison results of carbanion-functionalized ILs with the previous reported ILs.

ILs	T/°C	ρ ($cm^3 \cdot g$)	η (mPa·s)	n_D	σ ($\mu S \cdot cm^{-1}$)	Ref.
[P_{4448}][Pen]	25	0.9219	406	1.5115	36.2	This work
[P_{4448}][Mho]	25	0.9332	730	1.5048	13.0	This work
[P_{4448}][Ido]	25	0.9845	1062	1.5168	26.3	This work
[P_{4448}][Dib]	25	0.9633	280	1.5228	44.3	This work
[P_{4442}][HFA]	30	1.1812	92	–	–	[19]
[P_{4442}][Pen]	25	0.9589	142	–	–	[8]
[aP_{4443}][Ala]	25	0.9858	758	–	106	[14]
[P_{4444}][Ala]	25	0.9500	227	–	418	[20]
[P_{4444}][Gly]	25	0.9630	233	–	485	[20]
[P_{4444}][Ser]	25	0.9910	734	–	168	[20]



review & editing, Supervision. **Duan-Jian Tao:** Conceptualization, Writing - review & editing, Supervision.

Declaration of competing interest

There are no conflicts to declare.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.molliq.2020.113405.

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