

# A New Supramolecular Hole Injection/Transport Material on Conducting Polymer for Application in Light-Emitting Diodes

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Organic and polymer light-emitting diodes (LEDs) have drawn considerable attention because of their low power consumption, light weight, fast response and wide viewing angle.<sup>[1]</sup> Charge transport is an important factor with regard to the performance of these devices. For high-performance LEDs devices, charge injection and transport from both anode and cathode must be balanced off by excitons formed in light emission layer.<sup>[2]</sup> General LEDs devices possess three layers sealed between two electrodes, including the hole injection/transport layer (HITL), the electron-emitting layer (EML) and the electron-transporting layer (ETL). Package configurations allow each layer to be optimized individually for charge injection, transport and emission. Studies on organic materials possessing high hole-injection have been focused on poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) (PEDOT-PSS) due to its reasonable ionization potential ( $I_p = -5.2$  to  $-5.3$  eV), high conductivity ( $\sim 1-10$  S/cm) and good hole-injection ability.<sup>[3]</sup> However, PEDOT-PSS is fabricated from water dispersion and water is relatively more destructive than oxygen for organic and polymer LEDs.<sup>[4]</sup> Therefore, PEDOT-PSS is not very stable in the LEDs architecture.<sup>[5]</sup> Efforts to develop novel hole injection/transport materials (HITMs) containing neither ion nor hydrophilic functionality to replace PEDOT-PSS have been actively pursued.

Covalently cross-linked HITMs leading to the formation of solvent-resistant hole-injection layers have been extensively studied. Varieties of thermally,<sup>[6]</sup> photochemically<sup>[7]</sup> and electrochemically<sup>[8]</sup> cross-linked materials can overcome the interfacial mixing caused by solution processing, however, additional processes are required. Highly complementary noncovalent bonds (e.g., hydrogen bonds, electrostatic interactions or chain entanglements) leading to physically cross-linked structure may act as well as the covalently cross-linked materials without additional processes.

Our previous studies reported that the hydrogen bonding interactions of nucleobase-functionalized polymers resulted in highly cross-linked structures through biocomplementary hydrogen bonding with substantially increased glass transition temperature ( $T_g$ ).<sup>[9]</sup> Recently, Weder et al.<sup>[10]</sup> found that the network structure of cross-linked conjugated polymers has significant benefits for the

electronic communication between chains and thus has higher hole mobility. Li et al. also used the cross-linked polythiophenes with vinylene-terthiophene-vinylene as conjugated bridge to enhance the hole mobility.<sup>[11,12]</sup> We speculated that the nucleobase interactions may have chance to generate an effective supramolecule-based polymeric HITM. The charge transfer through nucleobase interactions is expected to be improved through the donor-acceptor conjugate from the complex of nucleobases.<sup>[13,14]</sup>

In this study, a new DNA-mimetic  $\pi$ -conjugated polymer PTC-U containing pendant uracil (U) groups has been successfully synthesized to function as a hole injection/transport polymer possessing a suitable glass transition temperature and are soluble in polar solvents (Scheme 1). We chose triphenylamine (TPA) and carbazole (CBZ) as the conjugated main chain because of their high hole mobility<sup>[15-18]</sup> with alkyl chain spacers between the conjugated polymer backbone with the uracil units can modulate electrical response through intermolecular U-U interaction.<sup>[19]</sup> Excellent hole injection/transport and electron-blocking properties in combination with good solubility only in polar solvents make the PTC-U a promising candidate for HITL in the fabrication of high-efficiency organic or multilayer polymer light-emitting devices. We achieved a luminous efficiency of 1.5 cd/A and a maximum brightness of 8828 cd/m<sup>2</sup> from the Alq3-based bilayer device, which is comparable to the reported data (efficiency: 1.70 cd/A or 1.08 cd/A, maximum brightness: 7500 cd/m<sup>2</sup>) using a conventional PEDOT-PSS layer as a HITL.<sup>[20-22]</sup> Moreover, a trilayer device using PTC-U as HITL achieved a maximum brightness levels as high as 48233 cd/m<sup>2</sup>, external quantum efficiency and luminance efficiency of 2.40% and 8.0 cd/A, respectively, which is approximately 1.6 times higher than that of PEDOT-PSS-based devices.

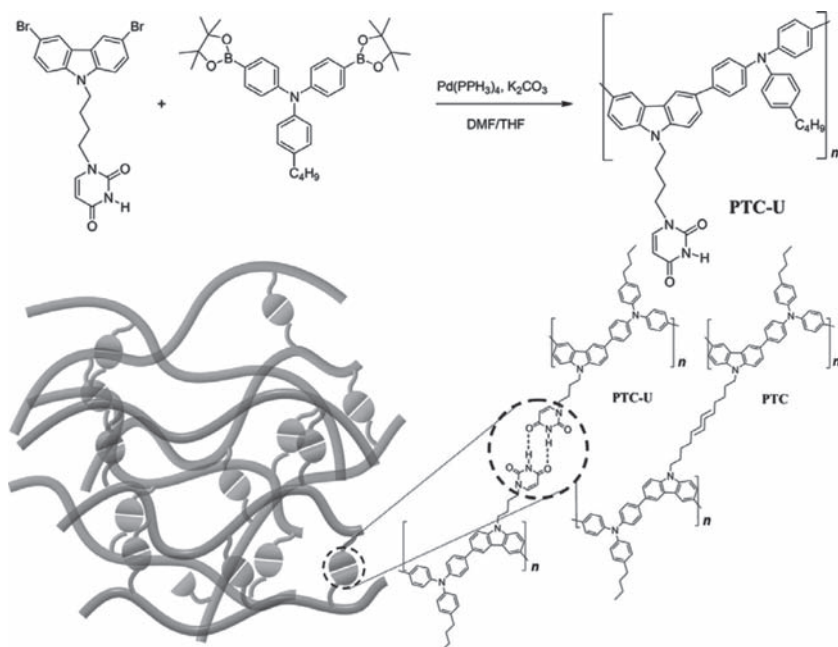
To synthesize the new HITM, PTC-U, a five-step reaction was carried out as shown in Scheme 1 while a control polymer (PTC) was also synthesized (Detailed synthetic schemes of monomers, PTC-U and PTC are listed in supporting information, Scheme S1 and S2.). The properties of PTC-U and PTC are summarized in Table 1.

The self-assembly behavior of PTC-U was confirmed via <sup>1</sup>H NMR titration experiment. The association constants ( $K_a$ ) for hydrogen-bonded complexes characterized at 25 °C in tetrachloroethane-d<sub>2</sub> and the PTC-U concentration systematically increased from 0.625 mM to 5 mM, the uracil N-H proton peak is dramatically shift downfield (9.07 to 9.65 ppm) consistent with increasing the concentration of the PTC-U. In addition, the chemical shift of the amide proton of uracil was monitored to give a value of  $K_a$  of 26.3 M<sup>-1</sup> from Benesi-Hildebrand plots (Figure S1), which is consistent with  $K_a$  values reported previously for U-U base pair recognition.<sup>[23]</sup>

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**Scheme 1.** Physically cross-linked network formed by uracil self-complementary bonding.

**Figure 1** presents the PL spectra of PTC and PTC-U in tetrachloroethane and solid film, the thin film were fabricated on glass slides through the spin coating method using tetrachloroethane as solvent. The PL spectra of PTC-U in tetrachloroethane and solid film show a red shift relative to PTC, most likely due to the closer packing of the chromophores and increased film density through hydrogen bonding.<sup>[24]</sup> These observations indicate that the U-U hydrogen-bonding interactions result in physically cross-linked structures<sup>[25]</sup> and promote the interactions of both inter-chain backbones and chromophoric segments in solution and solid state. The U-U hydrogen-bonding interaction between PTC-U chains are extremely stable in the bulk state as observed by the FTIR spectra obtained from 30 to 200 °C (Figure S2),<sup>[9]</sup> which is consistent with its relatively higher  $T_g$  (Figure S3).

PTC-U is able to self-assemble to form a physically crosslinked polymer-like structure which is potentially applicable in the fabrication of multilayer PLED device. However, high solvent resistant is prerequisite for multilayer PLED fabricated by solution processes. Solvent resistance of PTC-U film was investigated by monitoring the UV-vis spectra of hole injection/transport materials (HITMs) before and after rising

with toluene (a good solvent for emissive polymer such as polyfluorene). As shown in Figure S4, most of the absorption of the PTC film disappears after rising with toluene, indicating that the film is dissolved out and removes during the rising process. On the contrary, the absorption intensity of the PTC-U film remains almost unchanged after rising with toluene. The highly solvent resistance is restrained due to the intermolecular fixing effect (Scheme 1) from U-U hydrogen bonding interactions. Therefore, the solution-processable and physically cross-linkable characteristics of PTC-U are both beneficial to the fabrication of multi-layer PLED device.

To assess the charge injecting of the polymer, the electrochemical characteristics of PTC and PTC-U thin films were investigated through cyclic voltammetry (CV) using tetrabutylammonium hexafluorophosphate (TBAPF6) as the supporting electrolyte and ferrocene as the internal standard and the results are summarized in Figure S5 and Table 1. PTC has the highest occupied molecular orbital (HOMO) energy level at

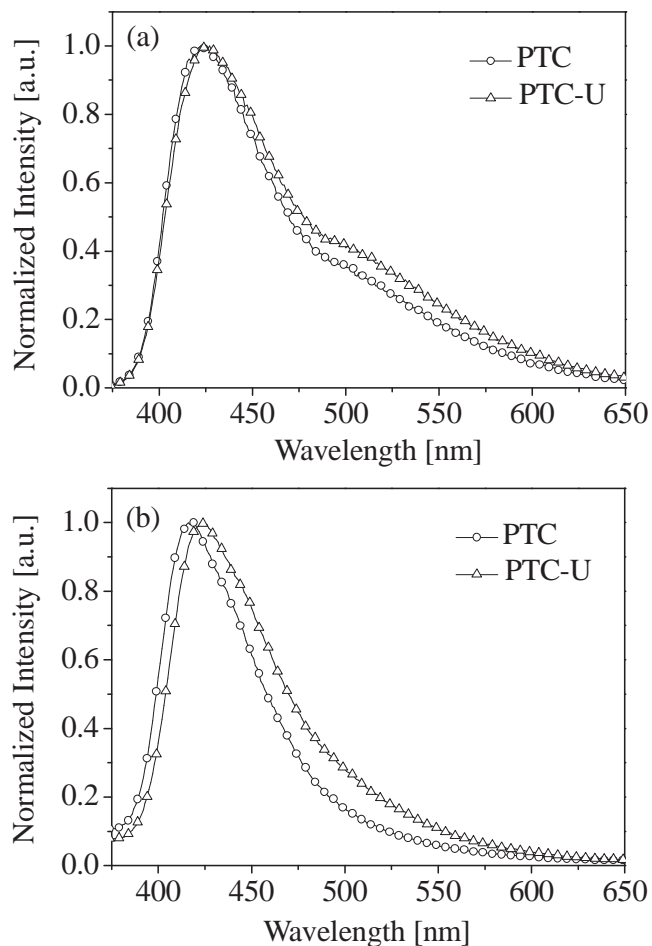
approximately  $-5.25$  eV while the lowest unoccupied molecular orbital (LUMO) energy level is at  $-2.20$  eV. On the other hand, the HOMO of PTC-U is at  $-5.15$  eV. The energy barrier for hole injection from ITO (HOMO =  $-4.9$  eV) to PTC-U is lower as compared with PTC. In addition, the LUMO of PTC-U is at  $-2.12$  eV which is higher than that of PTC, suggesting that PTC-U possessed better electron-blocking property. Our results are consistent with previous report<sup>[19]</sup> suggesting that specific hydrogen-bonding interaction strongly modulates the electrical and optical properties of the conjugated polymers. As a result, improved device performance using PTC-U as the HITM can be expected.

To further compare the hole-transporting properties, PTC and PTC-U were investigated by fabricating hole-only devices with structures of ITO/HITL (50 nm)/Al. The HITL were coated on an ITO substrate using tetrachloroethane as solvent. The results are shown in Figure S6, where the current density (hole injecting/transporting ability)<sup>[26]</sup> of the hole-only device from the PTC-U increases as compared to PTC. We attribute the increase in current density to lower injection barrier and higher inter-chain hole-diffusing mobility of the PTC-U, indicating that the physically cross-linked network structure

**Table 1.** Physical Properties of HITMs.

HITMs	Mn	Mw	PDI	Td [°C]	Tg [°C]	Eg <sup>a)</sup> [eV]	E <sub>ox,onset</sub> <sup>b)</sup> [eV]	HOMO <sup>c)</sup> [eV]	LUMO <sup>d)</sup> [eV]
PTC	6420	12064	1.88	420	150	3.05	0.45	-5.25	-2.20
PTC-U	7999	10758	1.34	395	197	3.03	0.35	-5.15	-2.12

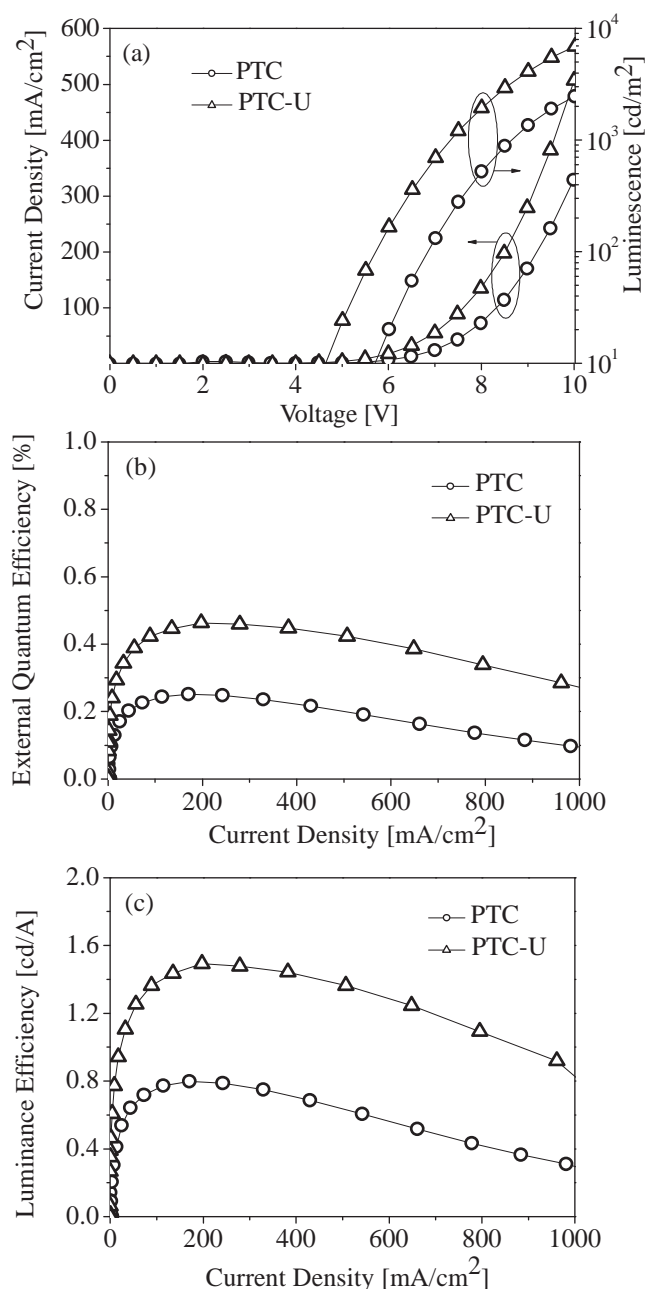
<sup>a)</sup>Eg was obtained from the UV-vis absorption spectra (Film); <sup>b)</sup>E<sub>ox,onset</sub> were measured by cyclic voltammetry using ferrocene as the internal standard; <sup>c)</sup>HOMO = E<sub>ox,onset</sub> + 4.8 eV; <sup>d)</sup>LUMO = HOMO - Eg.



**Figure 1.** Normalized emission spectra in tetrachloroethane (a) and solid film (b).

through hydrogen bonds promotes the hole injecting/transporting ability.<sup>[10–14]</sup>

The OLED device has been assembled to improve the hole injection/transport through sequential coating of PTC-U and PTC (as HITL), *Alq3* (as an EML as well as ETL), LiF (electron injection layer, EIL) and Al (electrode) layers onto ITO (transparent anode) [i.e., ITO/HITL (15 nm)/*Alq3* (60 nm)/LiF (1 nm)/Al (100 nm)]. The analysis of HOMO-LUMO energy levels of the materials for this research is shown in Figure S7. When the PTC-U or PTC was used as the HITL, the EL spectrum of the *Alq3* dual-layer device peaked at 513 nm (Figure S8), indicating that these HITMs only serve as hole-transporting materials without causing exciplex formation at interface with *Alq3*. This can be attributed to their proper HOMO energy levels for hole transporting. **Figure 2(a)** displays the current density–voltage–luminance (I–V–L) characteristics of these green-light-emitting devices. The PTC-U-based device exhibits significantly lower operating voltage than the control PTC device at the same current density, and also exhibited a lower turn-on voltage (3.7 V, corresponding to Figure 2 at 1 cd m<sup>-2</sup>) than did the PTC-based device. The L-V properties of the devices shown in Figure 2(a) indicate that the L values of the devices prepared from the



**Figure 2.** Plot of (a) I–V–L, (b) EQE–I and (c) LE–I characteristics of ITO/HITLs/*Alq3*/LiF/Al devices.

PTC-U are substantially higher than the PTC over the entire V range. **Figure 2(b)** and **2(c)** displays the luminescence efficiency (LE) and external quantum efficiency (EQE) plotted against the current density. The LE of the PTC-U-based device is 1.50 cd/A, nearly twice as the PTC (0.80 cd/A). The performance improvement of the PTC-U-based device over the PTC-based device is due to the difference in HITL, because the anode, cathode, ETL, EML, EIL and the thickness from both devices were the same. Since the HOMO level of PTC-U (5.15 eV) is lower than that of PTC (5.25 eV), the energy barrier for hole injection from ITO to the PTC-U layer in the PTC-U-based device is smaller. In

**Table 2.** Electroluminescence of Device Based on Structure ITO/HITLs/Alq3/LiF/Al.

HITLs	V <sub>on</sub> [V]	Q <sub>max</sub> [%]	LE <sub>max</sub> [cd/A]	ηE <sub>max</sub> [lm/W]	B <sub>max</sub> [cd/m <sup>2</sup> ]	V <sub>max</sub> [V]
PTC	4.8	0.25	0.80	0.29	3375	11.99
PTC-U	3.7	0.46	1.50	0.57	8828	11.50

**Table 3.** Electroluminescence of Device Based on Structure ITO/HITLs/NPB/Alq3/LiF/Al.

HITLs	V <sub>on</sub> [V]	Q <sub>max</sub> [%]	LE <sub>max</sub> [cd/A]	ηE <sub>max</sub> [lm/W]	B <sub>max</sub> [cd/m <sup>2</sup> ]	V <sub>max</sub> [V]
PTC-U <sup>a)</sup>	2.8	2.40	8.0	4.56	48233	12
PEDOT <sup>b)</sup>	2.4	1.46	4.7	3.57	41948	12
PEDOT <sup>c)</sup>	2.3	1.44	4.8	3.62	37934	11
PEDOT <sup>d)</sup>	2.3	1.37	4.5	3.51	34955	10

<sup>a)</sup>15 nm; <sup>b)</sup>15 nm; <sup>c)</sup>30 nm; <sup>d)</sup>50 nm.

In addition, the hole injecting and transporting ability of PTC-U is better than PTC as revealed previously for the hole-only devices. Thus, more holes moves to the emitting layer to balance the charge recombination at the emitting interface. The enhanced brightness and efficiency of PTC-U-based device relative to the PTC device can be attributed to balanced charge fluxes within the EML (better hole injection, hole transporting and electron blocking properties) and better thermal stability due to the physically cross-linked structure. **Table 2** lists the characteristics of the electroluminescence obtained using PTC-U and PTC as HITMs. Although the PTC-U possesses fairly high uracil content, its presence does not interfere the hole-transporting property of the PTC-U. It's notable that the performance of the ITO/PTC-U/Alq3/LiF/Al device in terms of maximum brightness (8828 cd/m<sup>2</sup>) and luminance efficiency (1.5 cd/A) is higher than multilayer device of ITO/PEDOT-PSS/Alq3/LiF/Al reported elsewhere,<sup>[20–22]</sup> indicating improved hole injection, transport and electron-blocking from the U-U interactions of PTC-U.

In order to gain further improved performance, we introduced 1,4-bis-[(1-naphthyl)-phenylamino]-diamine (NPB) into a HTL for better charge injection from HITL to EML<sup>[27]</sup> and compared the performance with device using conventional PEDOT-PSS [i.e., ITO/PTC-U (15 nm) or PEDOT-PSS (15, 30, 50 nm)/NPB (15 nm)/Alq3 (60 nm)/LiF (1 nm)/Al (100 nm)]. A summary of the display characteristics is shown in **Table 3** and Figure S9, the maximum external quantum efficiency, and power efficiency of the PTC-U-based device is almost 1.6 times higher than that form the device using PEDOT-PSS. In addition, the consequences of different PEDOT-PSS film thickness on device performance also show similar trend (Table 3), these results suggest that this PTC-U-based HITM provides cross-linking capability to enhance a hole-electron recombination in the emitting layer. In other word, the high efficiency achieved with PTC-U was very encouraging and supported its potential as a viable alternative to PEDOT-PSS. Therefore, our current

research efforts are focused on further improving the device lifetime and enhancing the hole injection by optimizing the device characteristics.

In summary, a new DNA-mimetic HITM (PTC-U) containing neither ion nor hydrophilic functionality has been developed. The physically cross-linked PTC-U results in remarkable enhancement on thermal stability, hole injection and electron blocking ability. The performance of the PTC-U-based device is twice higher than that of PTC. In addition, the trilateral LED fabricated using PTC-U exhibit superior performance relative to devices from commercialized PEDOT-PSS. PTC-U is the first example of supramolecular HITM possessing high hole-transporting capacity, providing a potential route towards next-generation high efficiency LED device.

## Experimental Section

**Device Fabrication and Measurement:** The EL devices were fabricated through vacuum deposition of the materials at 10<sup>-6</sup> torr onto ITO glass having a sheet resistance of 25 Ω square<sup>-1</sup>. All of the organic layers were deposited at a rate of 1.0 Å s<sup>-1</sup>. The cathode was completed through thermal deposition of LiF at a deposition rate of 0.1 Å s<sup>-1</sup>, then capped with Al metal through thermal evaporation at a rate of 4.0 Å s<sup>-1</sup>. The relationships of the current density and brightness of the devices with respect to voltage were measured using a Keithley 2400 source meter and a Newport 1835C optical meter equipped with an 818ST silicon photodiode. The EL spectrum was obtained using a Hitachi F4500 luminescence spectrometer.

**Synthesis of PTC-U:** To a mixture of 4-Uracilbutyl-9(3,6-dibromocarbazole) (0.75 g, 1.5 mmol), 4-Butyl-N,N-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane-4-phenyl)-aniline (0.82 g, 1.5 mmol), and freshly prepared Pd(0)(PPh<sub>3</sub>)<sub>4</sub> (0.18 g) was added a deoxygenated mixture of THF (6 mL), DMF (6 mL) and aqueous 2 M K<sub>2</sub>CO<sub>3</sub> (8 mL). The mixture was vigorously stirred at 85–90 °C for 48–72 h. After the solution was cooled, the whole mixture was poured slowly into a cold mixture of methanol/deionized water (10/1 in volume). The polymer was collected by filtration and washed with methanol. The solid was then washed for 24 h in a Soxhlet apparatus using acetone to remove oligomers and catalyst residues. The polymer was further purified by redissolving in DMF and then precipitated from cold methanol prior to drying at room temperature under high vacuum. <sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>, δ): 11.2 (br, NH), 8.54 (br, ArCH), 7.67 (br, ArCH and CH), 7.06 (br, ArCH), 5.48 (br, CH), 4.40 (br, CH<sub>2</sub>), 3.63 (br, CH<sub>2</sub>), 2.52 (br, CH<sub>2</sub>), 1.73 (br, CH<sub>2</sub>), 1.63 (br, CH<sub>2</sub>), 1.51 (br, CH<sub>2</sub>), 1.26 (br, CH<sub>2</sub>), 0.86 (br, CH<sub>2</sub>).

## Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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