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High-Triplet-Energy Poly(9,9′-bis(2-ethylhexyl)-3,6-fluorene) as Host for Blue and Green Phosphorescent Complexes**

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During the last two decades, organic and polymer light-emitting devices (OLEDs/PLEDs) have been the subject of intense academic and industrial research because of their potential applications in full-color flat-panel displays and solid-state lighting.[1–3] Recently, phosphorescent PLEDs, in which heavy metal complexes (such as Ir and Pt complexes) are doped into appropriate polymer host materials, have attracted increasing interest owing to the possible full utilization of both singlet and triplet excitons combined with solution processability.[4] Many studies[5] have shown that in order to realize high device efficiency, in phosphorescent PLEDs, the triplet energy state (E_T) of the polymer host should be located at higher energy than that of the guest complexes. Otherwise, triplet excitons on the guest could undergo back-transfer to the triplet state of the host, and as a result the host polymer would become a luminescence quencher of triplet emitters.[6] Since most conjugated polymers have a low-lying triplet state, so far only red-phosphorescent PLEDs with a conjugated polymer as the host have shown high device efficiency.[7] For green- and blue-light-emitting triplet emitters[8] with typically a higher triplet state than that of conjugated polymers,[9] the devices with the conjugated polymer host are poor. Therefore for green- and blue-light-emitting triplet devices, typically nonconjugated poly(vinylcarbazole) (PVK) has been used as the polymer host, since PVK has a relatively high triplet energy state (2.5 eV).[5c] Since PVK is a nonconjugated polymer with high resistivity, typically, the operating voltage of these devices is relatively high. Therefore in order to obtain phosphorescent PLEDs with high efficiency and low power consumption, it is crucially important to develop a wide-bandgap conjugated polymer host with high-lying triplet energy state.

The most widely used polymer hosts for fluorescent guest dyes are poly(2,7-fluorene)s and their derivatives, owing to their wide bandgap, high photoluminescence (PL) quantum efficiency, and excellent conductivity.[10] However, many authors have reported that poly(2,7-fluorene)s are not a good host for blue- and green-light-emitting triplet complexes because of their low-lying triplet energy state (2.15–2.3 eV).[5b,5d,6a] Surprisingly, despite poly(2,7-fluorene)s having been investigated for many years, there have been almost no reports on a fluorene homopolymer with fluorene units linked at the 3,6 position. As poly(3,6-carbazole)s[8,11] and poly(3,6-silafluorene)s[12] show higher triplet energy levels than their 2,7-counterparts, we thought it of interest to synthesize poly(3,6-fluorene)s and investigate their photophysical properties. The group of van Dijken and Brunner reported a 3,6-spirofluorene copolymer and oligomers with carbazole.[8a,13] Recently, Mo et al.[14] reported synthesis of poly(3,6-fluorene) by Ni-catalyzed coupling, but their polymer showed a strange and extremely broad PL spectra in the film and neither detailed photophysical properties nor device performance was reported for this polymer. We expected poly(3,6-fluorene) might have wider bandgap and much higher triplet energy level than poly(2,7-fluorene), and as a result it could be a good conjugated host material for the blue- and green-light-emitting triplet guests.

In this Communication, we report the synthesis of a novel conjugated homopolymer, poly(9,9′-bis(2-ethylhexyl)-3,6-fuorene) (P36EHF), with a wide bandgap of 3.65 eV showing PL and electroluminescence (EL) emission in the UV region peaked at around 344 and 400 nm, respectively. We have also shown that P36EHF has a triplet energy level at around 2.6 eV much higher than that of poly(2,7-fluorene)s. Therefore P36EHF can be a good host material for green- and even blue-light-emitting triplet emitters, such as iridium(III) *fac*-tris(2-phenylpyridine) (Ir(ppy)$_3$) and iridium(III) bis[2-(4′,6′-difluorophenyl)pyridinato-N,C$^{2'}$) picolate (FIrpic). To the best our knowledge, this is the first high-triplet-level conjugated polymer with polyfluorene structure.

The synthesis of 3,6-fluorene polymers is not as straightforward as that of 2,7-fluorene-based polymers. Indeed, both the 3 and 6 positions are in the *para* position of the bridged carbon at the 9 position of the fluorene unit. The 3 and 6 positions in fluorene cannot be directly halogenated by standard electrophilic aromatic substitution. The synthetic routes used to prepare the monomer and polymer in this study

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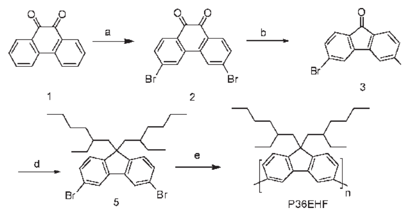

Scheme 1. Synthesis of P36EHF. Reagents and conditions: a) Br_2, $PhNO_2$, 130 °C; b) KOH, H_2O, 85 °C → 100 °C, $KMnO_4$; c) Zn-Hg, HCl, toluene, reflux; d) 1-bromo-2-ethylhexane, NaH, THF, reflux; e) $Ni(COD)_2$, COD, Bpy, DMF, toluene, 80 °C.

are outlined in Scheme 1. 3,6-Dibromo-9,10-phenanthrenequinone (**2**) and 3,6-dibromofluorenone (**3**) were prepared according to a modified version of a method published previously.[13] First, the bromination of 9,10-phenanthrenequinone (**1**) gave 3,6-dibromophenanthrenequinone (**2**). The following step, exploiting benzilic acid rearrangement and decarboxylation, afforded 3,6-dibromofluorenone (**3**). The key intermediate, 3,6-dibromofluorene (**4**), was obtained by the reduction of **3** using Zn-Hg as catalyst. Our initial attempts to use conventional Wolff–Kirshner reduction to obtain compound **4** were unsuccessful. The utilization of various other reduction agents[15] resulted either in only a trace of product or in no product at all. 3,6-Dibromo-9,9′-bis(2-ethylhexyl)fluorene) (**5**) was readily prepared by double alkylation of **4**. The structures of these compounds were confirmed by ^{1}H NMR and ^{13}C NMR spectroscopy as well as mass spectrometry (MS). The detailed procedure for synthesis of the monomer and intermediates and their characterization can be found in the Supporting Information (SI). The nickel-catalyzed Yamamato coupling polymerization was employed to obtain the desired homopolymer, poly(9,9′-bis(2-ethylhexyl)-3,6-fluorene, in good yield (80%). The polymer was readily soluble in common organic solvents such as chloroform, tetrahydrofuran (THF), and toluene. The number-average molecular weight (M_n) was around 7000 with a polydispersity index (PDI) of around 1.8 determined by gel permeation chromatography (GPC) against a polystyrene standard in THF.

The normalized UV-visible absorption and PL emission spectra of P36EHF in THF solutions (10^{-6} M) and in thin film are shown in Figure 1. The absorption spectrum in THF solution features a sharp absorption peak at 259 nm with a vibronic shoulder at 321 nm. The optical bandgap (E_g) of P36EHF was estimated as ca. 3.65 eV from the onset of the absorption peak (340 nm) in the film spectrum, which shows significantly hypsochromic shift compared to 380 nm (E_g = 2.95 eV) for the absorption onset for poly(9,9′-dioctyl-2,7-fluorene) (PFO).[16] We note that the absorption spectrum of the P36EHF thin film is almost identical to that in solution, without any red shift, which is typically observed for planar conjugated polymers. As reported by Liu et al.,[16] a 6 nm red shift was observed for poly(2,7-fluorene) in the solid state compared with its solution spectrum. This seems to indicate that the 3,6-fluorene linkage might have a much less planar conformation than poly(2,7-fluorene) due to the reduced interchain interaction between poly(3,6-fluorene) chains.

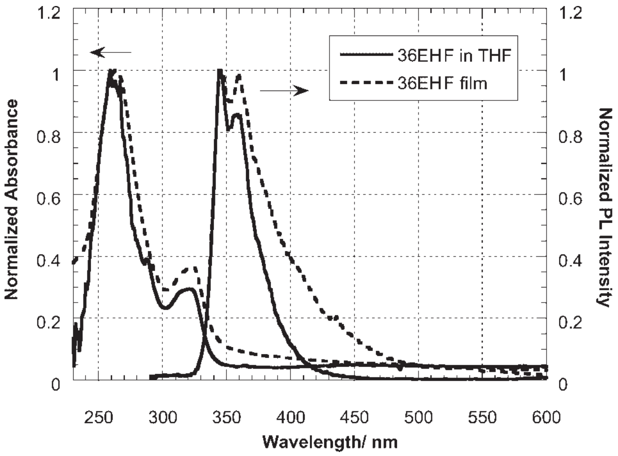

Figure 1. UV-vis and PL spectra of P36EHF in THF and films.

The PL spectra of P36EHF were recorded with 300 nm excitation (Fig. 1). P36EHF shows an emission maximum of 344 nm followed by a vibronic shoulder at 358 nm in THF solution. PL quantum efficiency of P36EHF in 1,2-dichloroethane solution is 34.6% with *p*-terphenyl (91% in 1,2-dichloroethane) used as reference.

Cyclic voltammetry (CV) curves of P36EHF showed only one irreversible oxidation, with onset at about +1.7 eV (Fig. S1, in the Supporting Information), which is much higher than that of poly(2,7-fluorene) (1.4 V vs. Ag/AgCl electrode).[17] The highest occupied molecular orbital (HOMO) energy level of P36EHF was estimated according to the empirical formula[18] $E_{HOMO} = -e(E_{ox} + 4.4)$ [eV] = −6.1 eV. We were unable to record the reduction process despite many attempts.

The thermal properties of P36EHF were evaluated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA reveals that the decomposition temperature (T_d) (5% weight loss) of P36EHF under nitrogen is 420 °C (Fig. S2), indicating excellent thermal stability. The DSC curve shows no crystallization or melting peak. The glass transition temperature (T_g) was observed at 115 °C (Fig. S3), which is much higher than that of poly(2,7-fluorene) (75 °C).[19]

To investigate the electroluminescent properties of blue-light-emitting devices made from pristine P36EHF, the polymer was used as the active layer and spin-coated on top of a PEDOT/PVK layer, where PEDOT is poly(3,4-ethylenedioxythiophene), following the thermal deposition of Ba/Al as cathode. The device configuration was ITO/PEDOT:PSS/PVK/polymer/Ba/Al, where ITO is indium tin oxide and PSS is poly(styrenesulfonate). As shown in Figure 2, EL emission in the UV region peaks at around 400 nm, which is red-shifted about 50 nm compared to the corresponding solid-state PL emission. At this stage of the experiment, it is not clear why the EL peak should show such a great red shift.

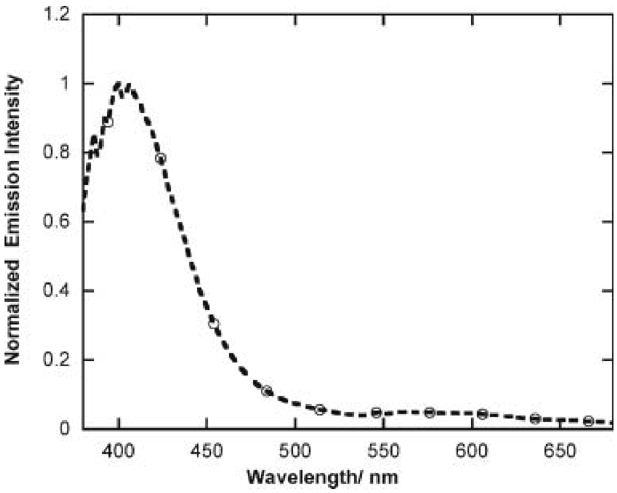

Figure 2. EL spectrum of P36EHF from device with configuration ITO/PEDOT/PVK/P36EHF/Ba/Al.

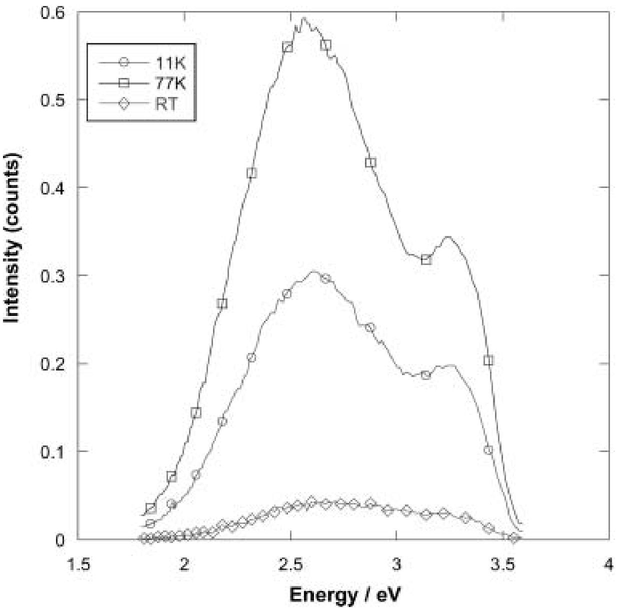

Figure 3. Phosphorescence spectra of P36EHF at 11 K, 77 K, and RT upon excitation with 325 nm He-Cd laser.

The EL device efficiency with P36EHF as emitting layer is summarized in Table 1. The best device with a TPBI/Ba/Al cathode, where TPBI is 1,3,5-tris(2-*N*-phenylbenzimidazolyl) benzene, reached an external quantum efficiency of 1.95% with emission maximum at around 400 nm.

The phosphorescent emission of P36EHF film at room temperature (RT), 77 K, and 11 K was measured in the phosphorescence mode of a PTI Fluorescence Master Series QM1 spectrophotometer with excitation at 325 nm by a He-Cd laser. The phosphorescent emission showed a broad peak with emission maximum at around 480 nm. Triplet energy level can be estimated from the peak position of phosphorescent spectra.[5b,6b] The E_T was estimated as 2.58 eV (Fig. 3), much higher than 2.15 eV for poly(2,7-fluorene)[20] and very close to that reported for PVK,[9] indicating that P36EHF could be a good host material for green- and even blue-light-emitting phosphorescent complexes.

In order to investigate the potential of P36EHF as a host for blue light emitters, a famous blue triplet material, FIrpic ($E_T = 2.62$ eV), was blended into P36EHF film by casting from a chlorobenzene solution (doping concentration: 10 wt%). The PL and EL spectra showed a maximum emission peak at 472 nm, typical for FIrpic in PVK and P36SiF, [12c,21] with no trace of host emission left at around 400 nm (characteristic of P36EHF emission), which indicated an efficient energy transfer from P36EHF to FIrpic. As reported previously by Chang et al.,[22] FIrpic doped into poly[9,9-bis(2-ethylhexyl)fluorene-2,7-diyl] (PF2/6) showed no signal of guest (FIrpic) emission up to high doping concentration while PF2/6 host emission was completely quenched, which indicates the back transfer of excitons to the low-lying triplet state of PF2/6. Figure 4 compares the PL spectra of FIrpic doped into P36EHF (Fig. 4a) and (PFO) (Fig. 4b) in different doping concentrations (FIrpic/P36EHF = 1 wt% and 10 wt%). As can be seen from Figure 4b, poly(2,7-fluorene) emission decreased with increasing FIrpic concentration while no FIrpic emission can be observed at all. This can be understood from Sudhakar

Table 1. EL data for the devices made from pristine P36EHF and FIrpic-doped films.

Active layer (EML)	Device configuration[a]	V_{on} [b] [V]	L_{max}[b] [cd m^{-2}]	Maximum QE$_{ext}$[b]			$J \sim 32$ mA cm^{-2}		
				Bias [V]	L[b] [cd m^{-2}]	QE$_{ext}$ [%]	Bias [V]	L [cd m^{-2}]	QE$_{ext}$ [%]
P36EHF	PEDOT/EML	19.0	82	24.0	37.7	0.07	25	14	0.06
P36EHF	PEDOT/PVK/EML	10.8	101	13.5	16.8	0.14	14.4	32	0.13
P36EHF	PEDOT/PVK/EML/TPBI	8.6	335	12.4	17.2	1.97	14.8	98	0.38
10 wt% FIrpic: P36EHF	PEDOT/EML	16.4	136	20.4	45	0.16	20.8	60	0.09
10 wt% FIrpic: P36EHF	PEDOT/PVK/EML	13.3	135	15.2	33.3	0.08	16.4	65	0.08

[a]ITO is used as the anode and Ba/Al as the cathode. [b]V_{on}: turn-on voltage defined as voltage at luminance of 1 cd cm^{-2}; L_{max}: maximum luminance; L: luminance; QE$_{ext}$: external quantum efficiency.

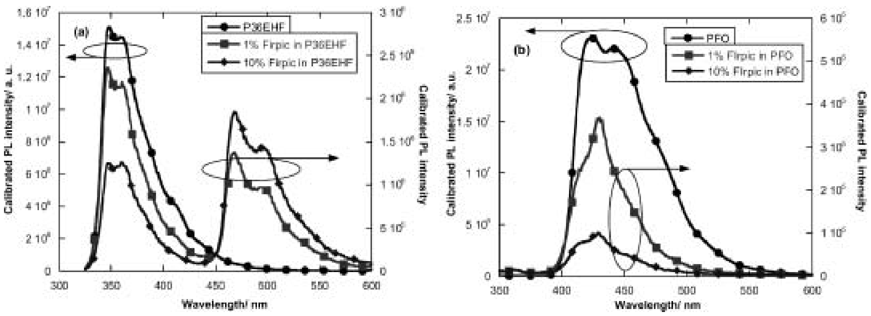

Figure 4. PL spectra (normalized by the maximal absorption coefficient of each film) of a) undoped P36EHF film and P36EHF films doped with 1 wt% and 10 wt% FIrpic, and b) undoped PFO film and PFO films doped with 1 wt% and 10 wt% FIrpic.

Table 2. Energy levels of the materials.

Guest or host	HOMO [eV]	LUMO [eV]	E_T[a] [eV]	Ref.
Ir(ppy)$_3$	5.1	2.6	2.41	[9]
FIrpic	5.8	2.9	2.62	[23]
Poly(2,7-fluorene)	5.8	2.1	2.15	[20]
P36EHF	6.1		2.58	This paper

[a]E_T: triplet energy level.

et al.'s work[6a] since the triplet level of poly(2,7-fluorene) (2.15 eV, Table 2) is much lower than the triplet level of FIrpic (2.6 eV, Table 2). Excitons on the FIrpic site underwent a back transfer to the low-lying triplet state of PFO, which is nonradiative by spin statistics. In contrast, as shown in Figure 4a, with increasing FIrpic content doped into the P36EHF film, the intensity of FIrpic emission (maximum peak at 468 nm) increases, along with decreasing P36EHF emission, which indicates that back transfer from FIrpic to P36EHF does not occur at all or not to a substantial degree. This fact implies that the triplet state energy of P36EHF should be much higher than that of poly(2,7-fluorene) and is close to 2.6 eV (that of FIrpic), which is consistent with the results of the phosphorescent spectra of P36EHF (Fig. 3). Further evidence that P36EHF has high triplet energy level can be provided by PL spectra of Ir(ppy)$_3$ and iridium(III) *fac*-tris(2-(4-*tert*-butylphenylpyridine) (Ir(Bu-ppy)$_3$) doped into P36EHF films (Fig. 5), where 510 nm emission, assigned to Ir(ppy)$_3$, increased with increasing guest content, indicating that efficient energy transfer from P36EHF to Ir(ppy)$_3$ and Ir(Bu-ppy)$_3$ (both have $E_T = 2.41$ eV)[9] occurred during photoexcitation. As reported by many authors,[5b,9,22] Ir(ppy)$_3$ emission was completely quenched by poly(2,7-fluorene) in the Ir(ppy)$_3$/PFO blend.

The normalized EL spectrum of the device made from a FIrpic/P36EHF blend (10 wt% doping concentration) is shown in Figure 6. The electroluminescent emission was dominated by a 470 nm emission feature from the FIrpic complex and no emission of the host P36EHF was detected. The preliminary device efficiency in the configuration ITO/PEDOT/FIrpic:P36EHF/Ba/Al reached 0.4 cd/A. Further optimization of the device structure is in progress.

In summary, a soluble homopolymer with high triplet energy level, poly(9,9′-bis(2-ethylhexyl)-3,6-fluorene), was successfully prepared by nickel-catalyzed coupling reaction. From the absorption onset, a bandgap of 3.65 eV has been deduced. The triplet energy level of P36EHF was determined to be around 2.6 eV from phosphorescent spectra taken at 77 K and 11 K. The resulting polymer showed a PL peak at 344 nm both in solution and in the solid film. The PLEDs based on the pristine P36EHF as active layer exhibited violet EL peaked at around 400 nm. The electrophosphorescent device showed typical blue light emission. Preliminary results indicate that poly(9,9′-bis(2-ethylhexyl)-3,6-fluorene) could be a good host material for green- and even blue-light-emitting triplet complexes.

Experimental

Materials and Device Characterization: ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker DRX-300 (300 MHz for ^{1}H and 75 MHz for ^{13}C) using CDCl$_3$ as the solvent. C, H elemental analyses were performed on a Vario EL elemental analysis instrument (Elementar Co.). Electron impact MS (EI-MS) data were obtained using a Trace GC-MS-2000 Series system (Finnigan). Atmospheric pressure chemical ionization MS (APCI-MS) data were obtained on an Esquire HCT-LC/MS (Bruker). UV-vis spectra were recorded using a HP 8453 UV-vis spectrometer. PL spectra were recorded on a Fluorolog JY luminescence spectrometer. M_n and M_w were determined by a Waters GPC 2410 in THF using a calibration curve of polystyrene standards. The phosphorescent spectra were measured on a PTI Fluorescence Master Series QM1 spectrophotometer using the 325 nm line (pulse width 4 ns, frequency 10 Hz) of a He-Cd laser as an

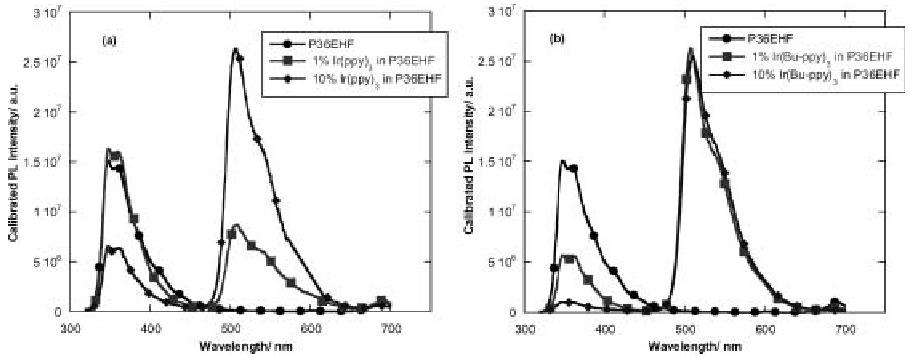

Figure 5. PL spectra (normalized by the maximal absorption coefficient of each film) of a) undoped P36EHF film and P36EHF films doped with 1 wt% and 10 wt% Ir(ppy)$_3$ and b) undoped PFO film and PFO films doped with 1 wt% and 10 wt% Ir(Bu-ppy)$_3$.

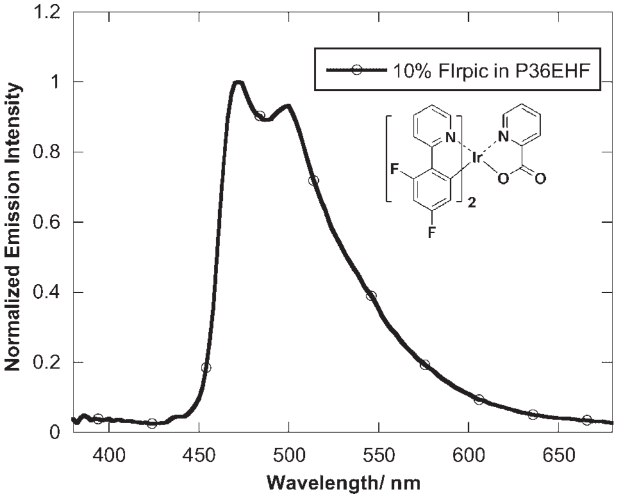

Figure 6. EL spectrum of a PLED made from P36EHF doped with FIrpic at a mass ratio of 10 wt%.

excitation source in phosphorescent mode with delay time 5 ms, gate time 100 ms, and the number of flashes set to 50. The spectra were analyzed by a 0.25 m focal length double monochromator with a Peltier cooled photomultiplier tube and processed with a lock-in amplifier. For the low-temperature measurements, samples were mounted in a closed-cycle cryostat (Oxford CC1104), in which the temperature can be adjusted from 10 to 330 K. The device fabrication procedure is described in the Supporting Information and in a previous publication [24]. Current density *I*–voltage *V*–luminance *L* characteristics of devices were collected using a Keithley 236 source meter and a calibrated silicon photodiode. External EL quantum efficiencies (QE$_{ext}$) were obtained by measuring the total light output in all directions in an integrating sphere (IS-080, Labsphere). The EL spectra and CIE coordinates were recorded using a PR-705 SpectraScan Spectrophotometer (Photo Research).

Synthetic Procedures: All starting materials were purchased from Aldrich, Acros Organics, or Alpha Aesar and used directly. FIrpic and Ir(ppy)$_3$ were purchased from American Dye Source, Inc. All the solvents were thoroughly purified under nitrogen before use. Monomer synthesis can be found in the Supporting Information.

Synthesis of P36EHF: In a reactor, bis(1,5-cyclooctadiene)nickel(0) (Ni(COD)$_2$) (0.165 g, 0.6 mmol), 1,5-cyclooctadiene (COD) (0.065 g, 0.6 mmol), and 2,2′-bipyridyl (Bpy) (0.094 g,0.6 mmol) were dissolved in 1 mL of anhydrous dimethylformamide (DMF) in a glove box with nitrogen. The solution was heated at 80 °C for 30 min to form active catalyst, and then the solution of compound **5** (274 mg, 0.5 mmol) in anhydrous toluene (5 mL) was added. The polymerization proceeded at 80 °C for 6 days in a glove box, and then dry bromobenzene (314 mg, 2 mmol) as end-capping agent was added to the reaction mixture and stirred for another 24 h. The resulting polymer was precipitated in methanol and then purified by silica chromatography to give a white solid. Yield: 188 mg (80%). ^{1}H NMR (300 MHz, CDCl$_3$, 25 °C, TMS): δ [ppm] = 8.12 (br, 2H), 7.66 (br, 2H), 7.49 (br, 2H), 2.06 (br, 4H), 0.94–0.58 (m, 30H). ^{13}C NMR (75 MHz, CDCl$_3$, 25 °C): δ [ppm] = 149.73, 141.86, 140.59, 126.44, 124.25, 118.74, 54.70, 44.77, 34.64, 33.82, 28.18, 26.99, 22.74, 13.99, 10.28. Anal. calcd for $(C_{29}H_{40})_n(388.6)_n$: C 89.63, H 10.37; found: C 89.80, H 10.47.

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