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ORIGINAL PAPER

Synthesis and characterization of multipyrene-modified platinum acetylide oligomers

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Abstract A series of new multipyrene-modified platinum acetylide oligomers were successfully synthesized utilizing the coupling reaction of *trans*-Pt complexes with C–H bonds in alkynes as the key step. These new conjugated oligomers were characterized by multinuclear NMR (^{1}H, ^{13}C, and ^{31}P) and mass spectrometry (MALDI-TOF–MS). Preliminary spectroscopic studies of these oligomers were also carried out.

Keywords Pyrene · Sonogashira coupling reaction · Organometallic compounds · Absorption spectrum · Fluorescence spectroscopy

Introduction

During the past few decades, conjugated oligomers have attracted increasing attention in chemistry and materials science owing to their interesting electrical, optical, and optoelectronic properties [1–7]. These excellent properties make them suitable for a range of promising applications, such as organic light-emitting diodes (OLEDs) [8–11], solar cells [12–15], field-effect transistors (FETs) [16–19], light detectors [20, 21], solid-state lasers [22–24], and sensors [25, 26].

Recently, the incorporation of some identical chromophores into a conjugated oligomer to construct multichromophoric conjugated oligomers has evolved to be one of the most attractive fields in photochemistry and materials science [27–31]. The arraying of multiple chromophores at close proximity with one another leads to interesting and novel properties, fueling the design of novel materials for many applications. In addition to the aforementioned properties of conjugated oligomers, multichromophoric conjugated oligomers offer many advantages compared to the single chromophoric conjugated analogues. For example, the collective absorption of multichromophoric conjugated oligomers is usually higher, which is particularly useful in light-harvesting applications in solar cell research [32–34]. Moreover, some new features, such as physical, optical, electrochemical, and photochemical properties, have been observed in multichromophoric conjugated oligomers [35, 36]. For instance, interactions between components in the ground state and excited state usually result in the formation of new complexes such as excimers and exciplexes [37, 38]. These complexes typically exhibit broadened absorptions and red-shifted emission spectra. Finally, the properties of multichromophoric conjugated oligomers can readily be adjusted to suit some application needs through geometric and directional control of the complexes because the relative orientation and arrangement of chromophores play a crucial role in their photophysical properties [27, 39]. Thus, numerous efforts have been taken to construct novel multichromophoric conjugated oligomers.

As mentioned above, the wide range of applications of multichromophoric conjugated oligomers has prompted the need to develop efficient methods to synthesize them. However, it is still very challenging to realize the

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well-controlled synthesis of desired multichromophoric conjugated oligomers. Owing to its ease of synthesis and favorable optical properties, the platinum acetylide skeleton has been widely used as a connecting bridge to construct conjugated oligomers [40–43]. In our previous research, we synthesized a series of platinum acetylide-containing building blocks, which have been employed to prepare supramolecular metallacycles and organometallic gels [44–49]. Moreover, platinum has a large spin–orbit coupling constant and consequently π-conjugated materials that contain this element typically exhibit high triplet yields and efficient phosphorescence [50–55]. Therefore, platinum acetylide-containing conjugated oligomers have been widely studied because they represent a class of materials with a high quantum efficiency of phosphorescence. Platinum acetylide-containing conjugated oligomers have also been used in various optoelectronic fields [51]. However, there have been very few successful examples of the construction of multichromophoric conjugated oligomers by using π-conjugated platinum acetylide moieties as the main skeleton, perhaps because of difficulties with their synthesis.

Pyrene is a polycyclic aromatic hydrocarbon consisting of four fused benzene rings and it has received significant attention owing to its interesting optical properties such as relatively long excited-state lifetimes and exceptional distinction of the fluorescence bands for monomer and excimer [56, 57]. In particular, the tendency of pyrene to form excimers has been widely employed for probing the structural properties of macromolecular systems [58]. In addition to the excimer sensing, there has been a recent increased interest in the use of pyrenes as optoelectronic materials [59–62]. Recently, multipyrene-modified oligomers have evolved to be one of the most attractive targets within materials science as it is expected to possess better fluorescent and electroluminescent properties than those of the pyrene monomer, because of its longer delocalized π-electron chain sequence. Thus, multipyrene-modified oligomers have been used as active components in molecular electronic devices such as OLEDs, OFETs, and solar cells [63–66].

Inspired in part by our successful results on the synthesis of platinum acetylide building blocks, we envisioned that the synthesis of a series of platinum acetylide-containing multipyrene conjugated oligomers with a varying number of pyrene units could be realized via a convergent strategy. It should be noted that these novel multipyrene conjugated oligomers are of special interest owing to their potential applications in the optoelectronics field. Herein, we present the synthesis of a series of novel platinum acetylide-containing multipyrene conjugated oligomers with a varying number of pyrene units (Scheme 1). The structures of all complexes were characterized by multinuclear NMR (^{1}H, ^{13}C, and ^{31}P) and mass spectrometry (MALDI-TOF–MS). A preliminary study of their spectroscopic behavior was also carried out.

Results and discussion

Synthesis of multipyrene platinum acetylide conjugated oligomers

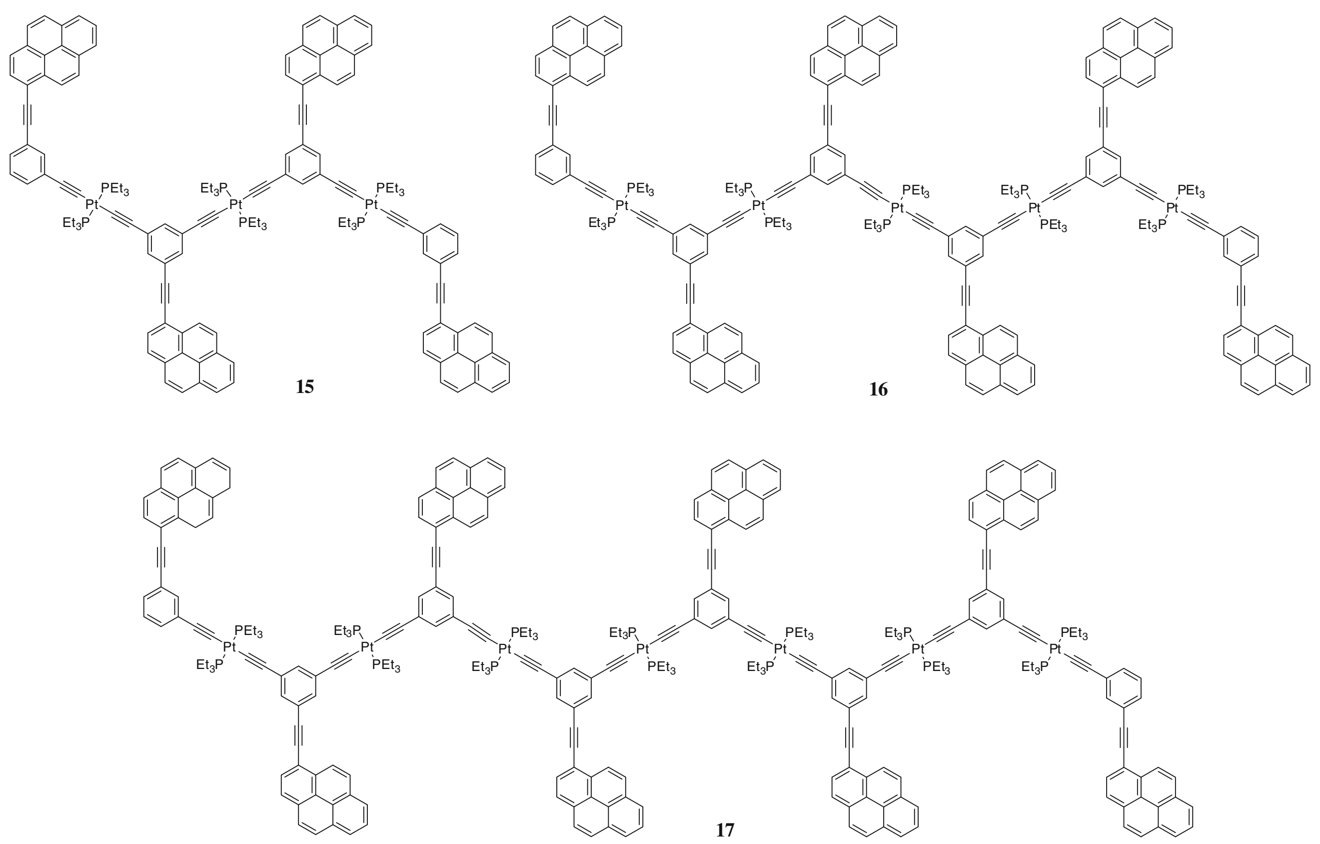
The acidity of C–H bonds in alkynes induced by transition metals has received considerable attention in organometallic chemistry. Herein, we utilized the coupling reaction of *trans*-Pt complexes with C–H bonds in alkynes as the key step in the synthesis of all multipyrene-modified platinum acetylide conjugated oligomers. The synthesis of tetrakis-pyrene conjugated oligomer **15** involved a series of Pd-catalyzed cross-coupling reactions starting from 1,3,5-tribromobenzene as shown in Scheme 2. Pyrene motifs were introduced by a Sonogashira coupling reaction of **1** with 1-ethynylpyrene to afford **2**, followed by deprotection with NaOH in toluene to afford **3**. Compound **3** was then reacted with 2 equiv of *trans*-$PtI_2(PEt_3)_2$ to give iodo-metal complex **4**. Subsequent coupling reactions of **4** with **5** resulted in the formation of trimethylsilyl protected **6** with two pyrenes in 72 % yield. Then further deprotection and coupling reactions were performed to get target complex **15**.

Two other oligomers **16** and **17** with geometric structural similarity to **15** were obtained through similar procedures in satisfactory yields as outlined in Schemes 3 and 4, respectively.

In all cases, the desired complexes were purified by column chromatography on silica gel using dichloromethane as eluent and obtained as yellow solids with high purity. They exhibited good solubility in common organic solvents such as CH_2Cl_2 and $CHCl_3$. Moreover, these newly designed oligomers are air- and humidity-stable at room temperature.

Structure characterization

Multinuclear NMR (^{1}H, ^{13}C, and ^{31}P) analysis of all multipyrene-modified platinum acetylide oligomers **15**–**17** revealed the formation of discrete and symmetric species. The room-temperature ^{31}P NMR spectra of oligomers **15**–**17** displayed a sharp singlet ($\delta = 11.6$ ppm for **15**, 11.3 ppm for **16**, and 11.6 ppm for **17**) accompanied by platinum satellites ($J_{Pt–P} = 2{,}362.77$ Hz for **15**, 2,358.07 Hz for **16**, and 2,369.73 Hz for **17**), which is consistent with the *trans* configuration around the Pt(II) atom. It should be noted that the singlet in each ^{31}P NMR spectra of **15**–**17** exhibited a small downfield shift from the iodo-metal



Scheme 1

precursors by 2.7, 2.4, and 2.5 ppm, respectively (Fig. 1). This change, as well as the increase in coupling of the flanking ^{195}Pt satellites (ca. $\Delta J = +42$ Hz for **15**, +36 Hz for **16**, and +48 Hz for **17**), might be caused by the electron-withdrawing effect of the large π-conjugate bridges.

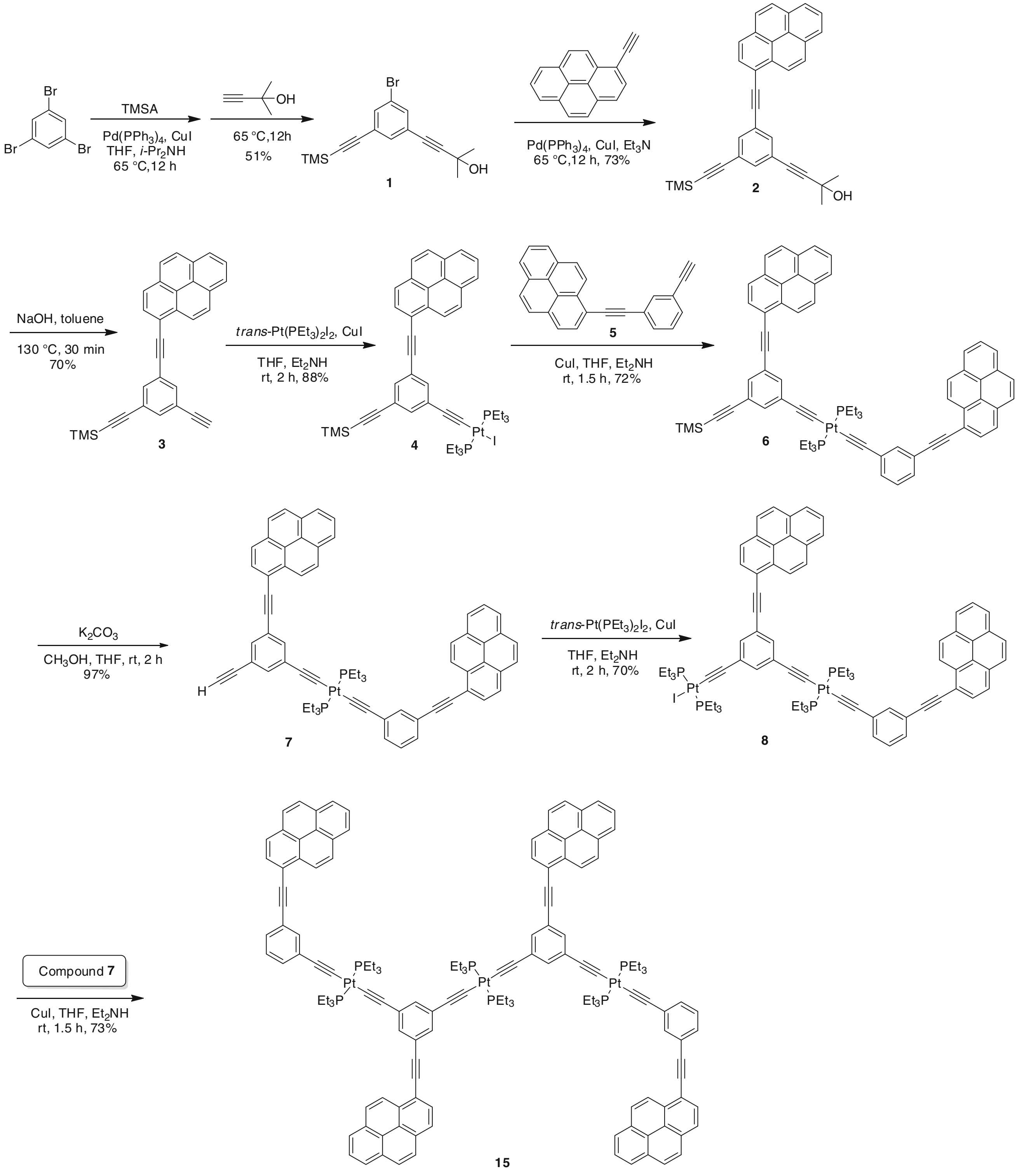
Usually matrix-assisted laser desorption/ionization time of flight mass spectrometry (MALDI-TOF–MS) studies allow the complexes to remain intact during the analysis process and provide the high resolution required for the unambiguous determination of their absolute molecular weight and molecularity. MALDI-TOF–MS investigation of multipyrene-modified oligomers **15–17** was therefore carried out. Because of the high molecular weight and complex isotope splitting of such multipyrene-modified oligomers, it is difficult to get strong mass signals even under MALDI-TOF–MS conditions. However, with considerable effort, satisfactory high resolution MALDI-TOF–MS results of **15–17** were obtained, which unambiguously support the formation of multipyrene-modified platinum acetylide oligomers with different length (Fig. 2). For example, the MALDI-TOF–MS spectra of **15–17** exhibited a strong signal of 2,640.50, 4,203.09, and 5,767.65, respectively, corresponding to the $[M+H]^+$ species. Thus, MALDI-TOF–MS results provided strong support for the existence of target multipyrene-modified platinum acetylide complexes.

Simulated molecular models

All attempts to grow X-ray-quality single crystals of multipyrene conjugated oligomers **15–17** have proven to be unsuccessful to date. Thus, PM6 semi-empirical molecular orbital methods were employed to optimize the geometry of all oligomers. In the optimized structure of each platinum acetylide oligomer, two phenyl acetylides are σ-bonded to platinum in a *trans* configuration. Coordination around the platinum atoms is square planar with slight tetrahedral distortions. Moreover, upon increasing the number of pyrene units, extended molecular length (4.5 nm for **15**, 6.5 nm for **16**, and 8.1 nm for **17**) was observed (Fig. 3).

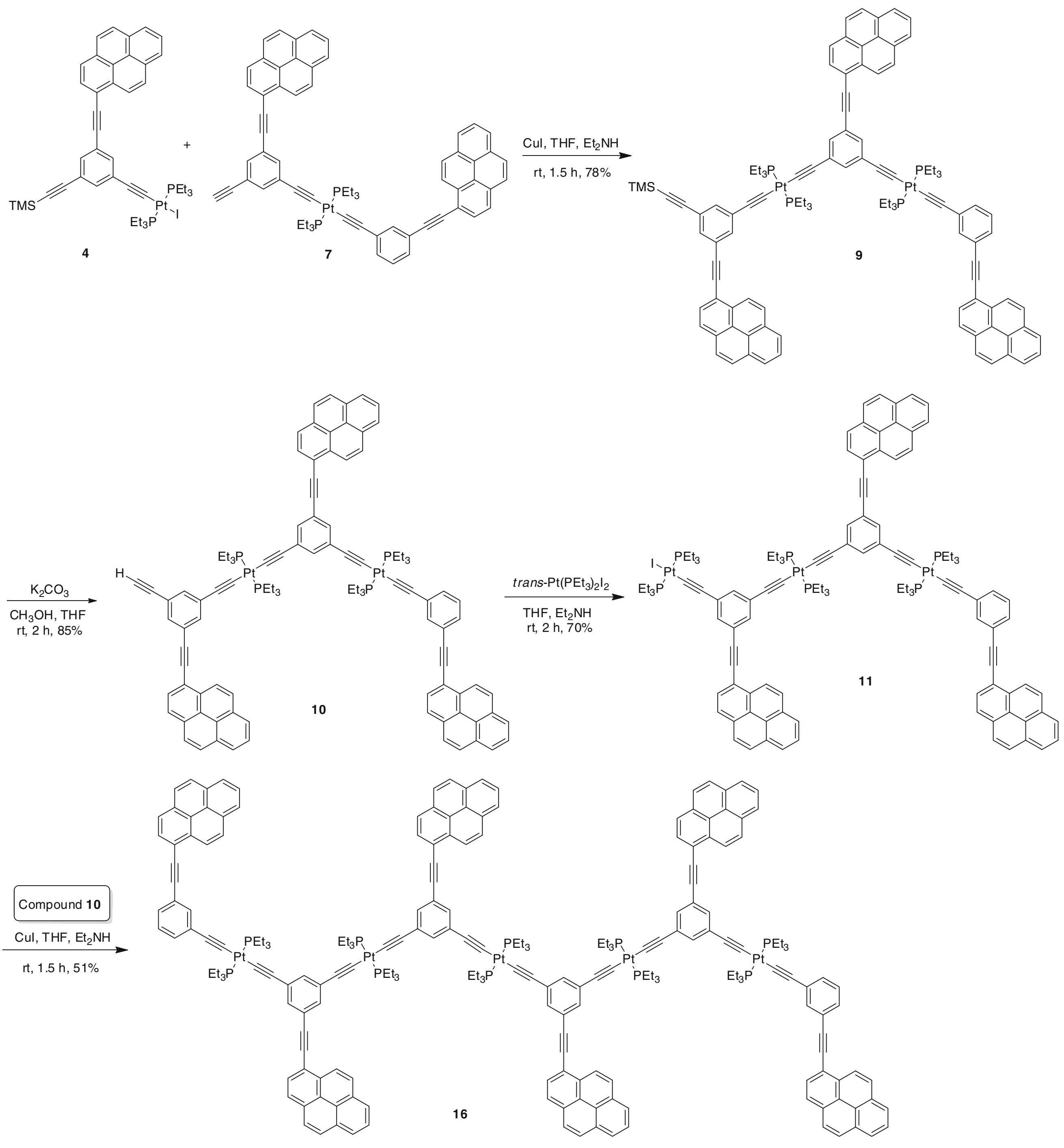
Absorption and emission properties

The absorption and emission properties of this series of multipyrene-modified platinum acetylide were investigated in dilute dichloromethane solution (ca. 10^{-6} M) and the data are collected in Table 1 and Fig. 4. The absorption spectra of **15–17** are structurally similar to each other and



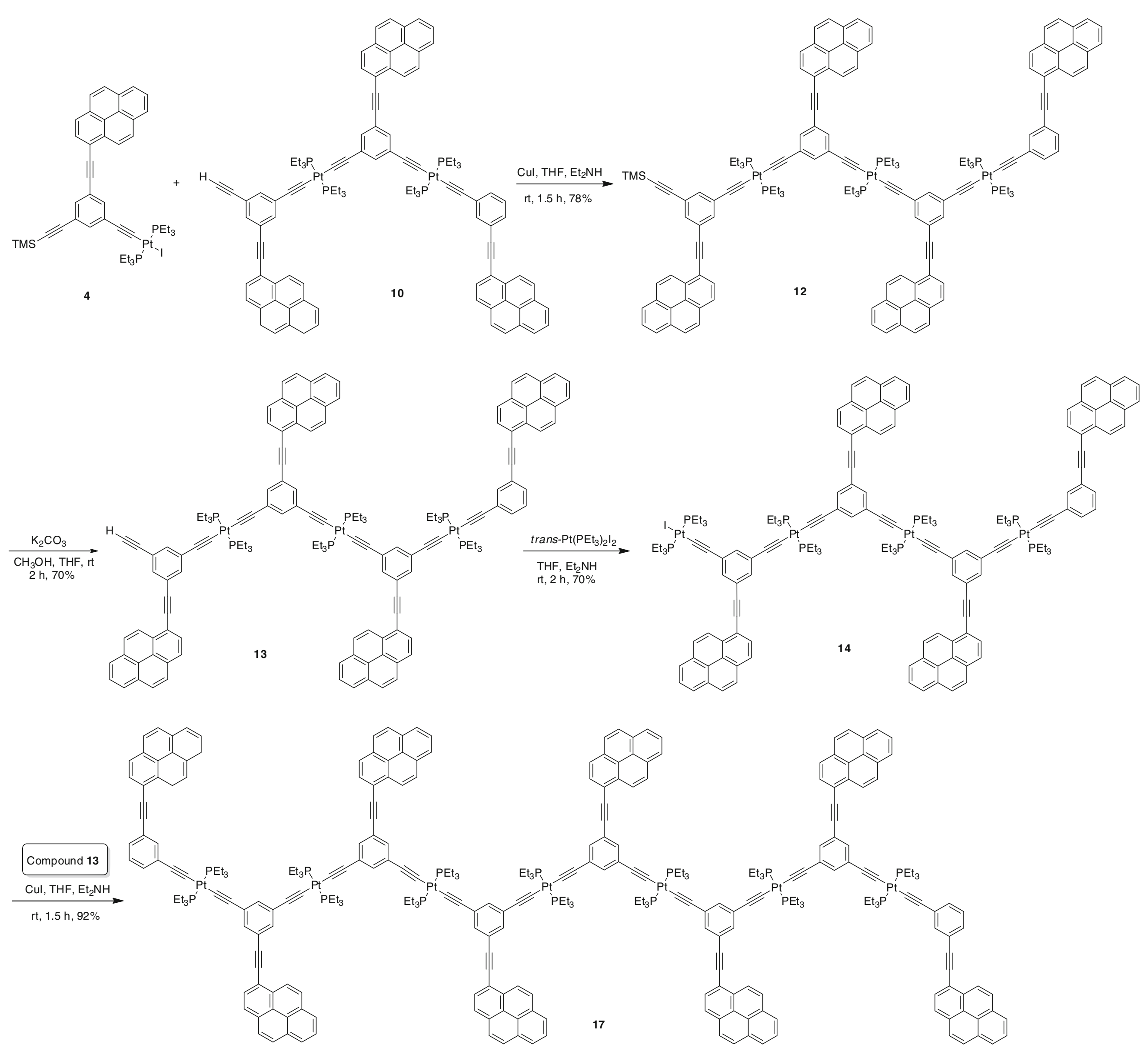
Scheme 2

show a broad and structureless band around 220–290 and 300–400 nm. Within previous spectroscopic work on *trans*-[Pt(PEt$_3$)$_2$(C$\equiv$CR)$_2$] the absorption bands at approximately 300–360 nm were assigned as metal-to-ligand charge transfer (MLCT) transitions [55, 67, 68]. Moreover, the low energy absorption bands (300–400 nm)



Scheme 3

in oligomers **15–17** may involve an admixture of intraligand (IL) and MLCT transitions. The higher energy absorption bands (ca. 220–290 nm) in oligomers **15–17** were dominated by intraligand π–π* transitions of aromatic pyrene rings [69]. Their absorption coefficients (ε) were enhanced with the increase of oligomer length because the effective concentration of pyrene was increasing [70]. Likewise, their fluorescence intensity increased with the increase of oligomer length, which might be also due to the higher effective concentration of pyrene. Interestingly, in the case of **17**, a shoulder band at 500 nm was found in the emission spectrum. This finding indicated that it was much



Scheme 4

easier for **17** to form excimers when compared to **15** and **16**. It is well known that the formation of pyrene excimers requires the complexation of an excited-state molecule with a ground-state molecule. Increasing the density of pyrene molecules assembled in conjugated oligomers enhances the probability of forming excited-state excimers. Surprisingly, it was found that the fluorescence quantum yields of these conjugated oligomers decreased with the increase of oligomer length. Usually the molecular skeleton becomes more flexible when the length of oligomer increases. Thus, in the longer oligomers, intersystem crossing and nonradiative decay processes might to some extent compete with the fluorescence radiation process. All of the above results indicated that the length of multipyrene-modified oligomers has a different effect on the absorption coefficient, fluorescence quantum yield, and the formation of excimers. Thus, these multipyrene conjugated oligomers are promising models to understand the fundamental properties of their analogous polydisperse polymers.

Conclusions

By employing the coupling reaction of *trans*-Pt complexes with C–H bonds in alkynes as the key step, we have

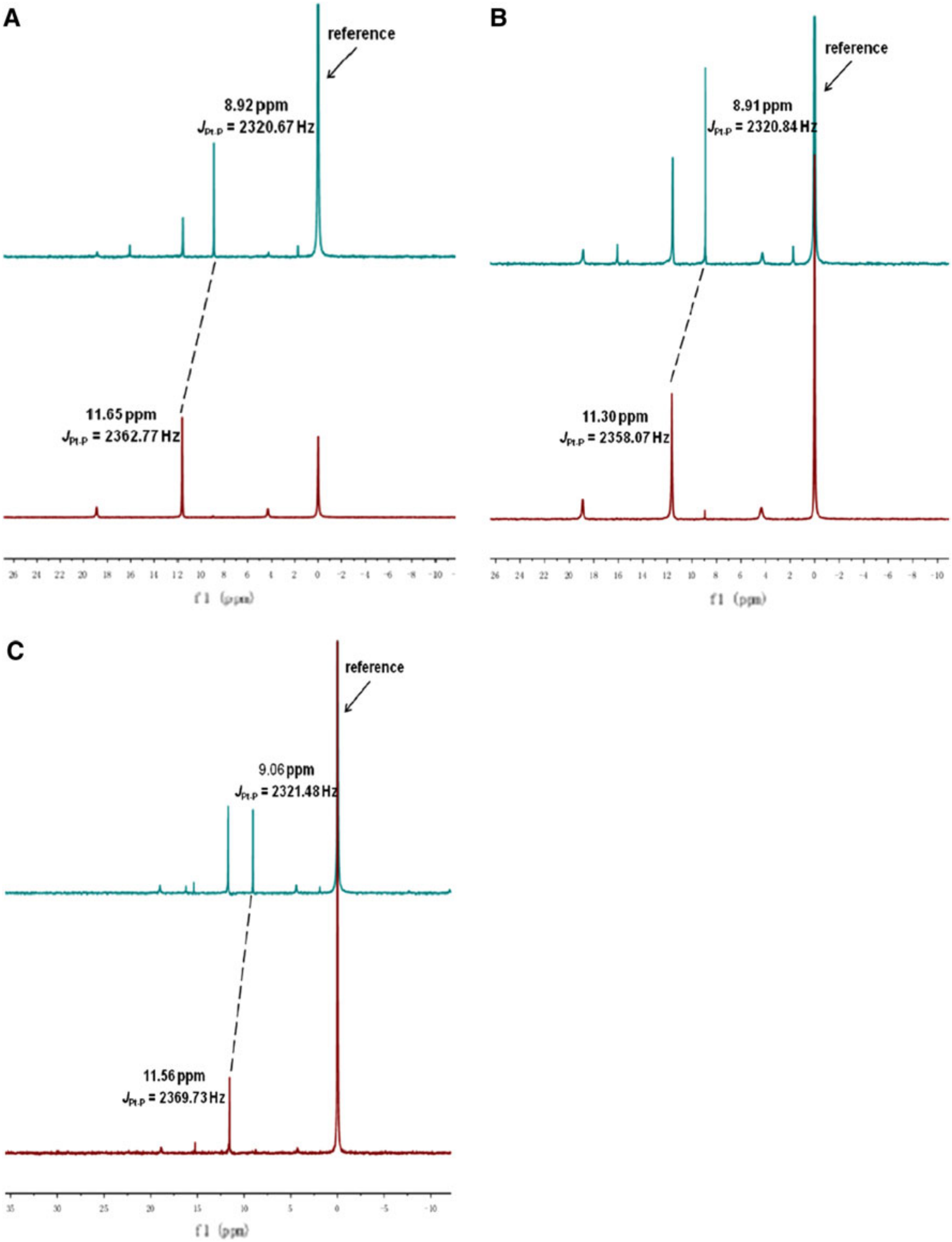


Fig. 1 Partial ^{31}P NMR spectra (400 MHz, $CDCl_3$, 298 K) of **15** (**a**), **16** (**b**), and **17** (**c**)

successfully prepared a new family of multipyrene-modified platinum acetylide oligomers from simple starting materials in satisfactory yields via a convergent strategy. Multinuclear NMR (^{1}H, ^{13}C, and ^{31}P) analysis of all conjugated oligomers was carried out. The sharp NMR signals in both $^{31}P\{^1H\}$ and ^{1}H NMR spectra along with the solubility of these species confirm the formation of such oligomers. Moreover, mass spectrometric (MALDI-TOF–MS) studies provided further evidence for the unambiguous determination of their absolute molecular weight and

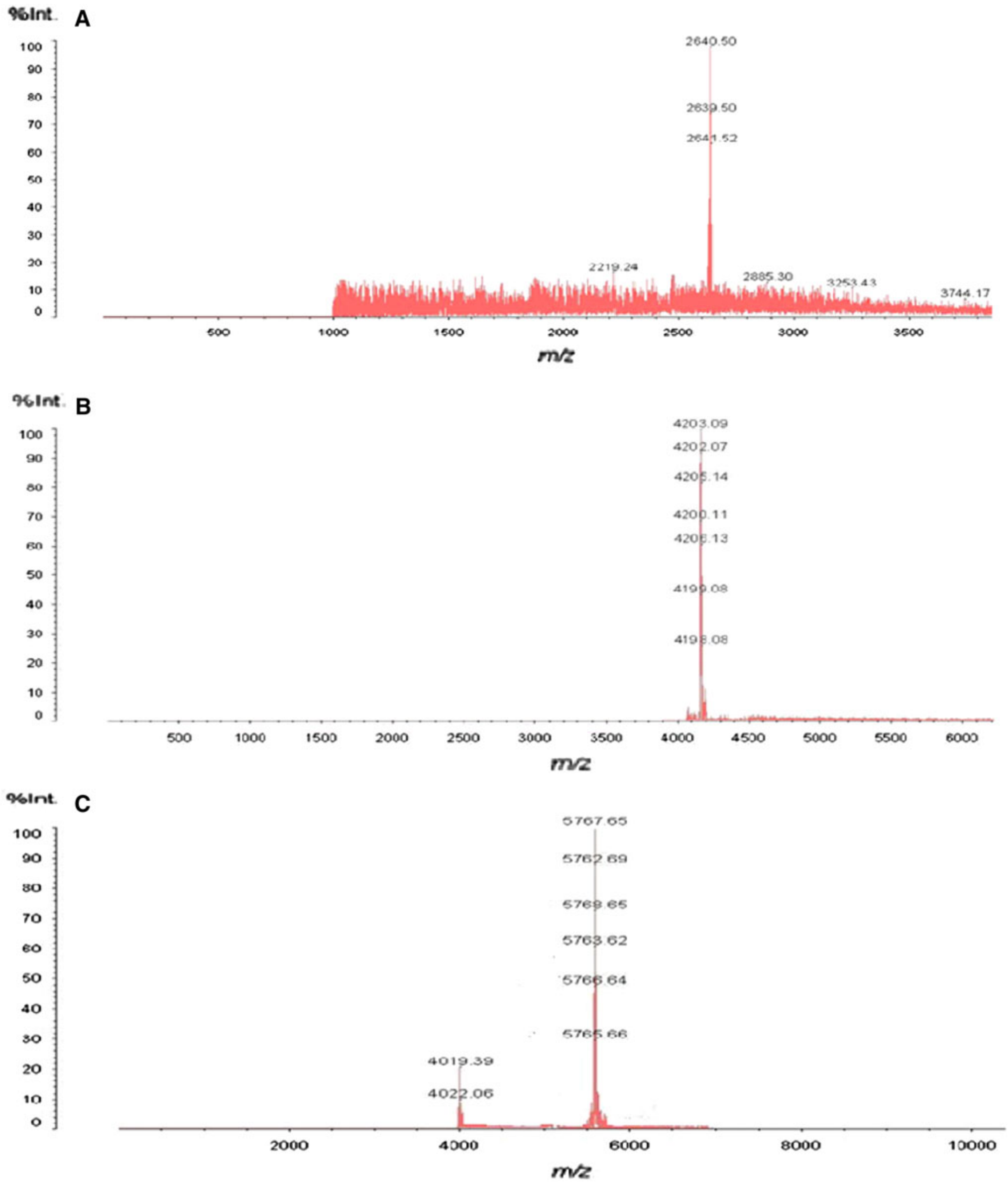


Fig. 2 MALDI-TOF–MS spectra of **15** (**a**), **16** (**b**), and **17** (**c**)

molecularity. All conjugated oligomers exhibit remarkable solubility in common organic solvents, such as dichloromethane and chloroform. Furthermore, these newly designed conjugated oligomers are air- and humidity-stable at room temperature. The preliminary spectroscopic studies indicated that the length of multipyrene conjugated oligomers has a different effect on the absorption coefficient, fluorescence quantum yield, and the formation of excimers. All obtained

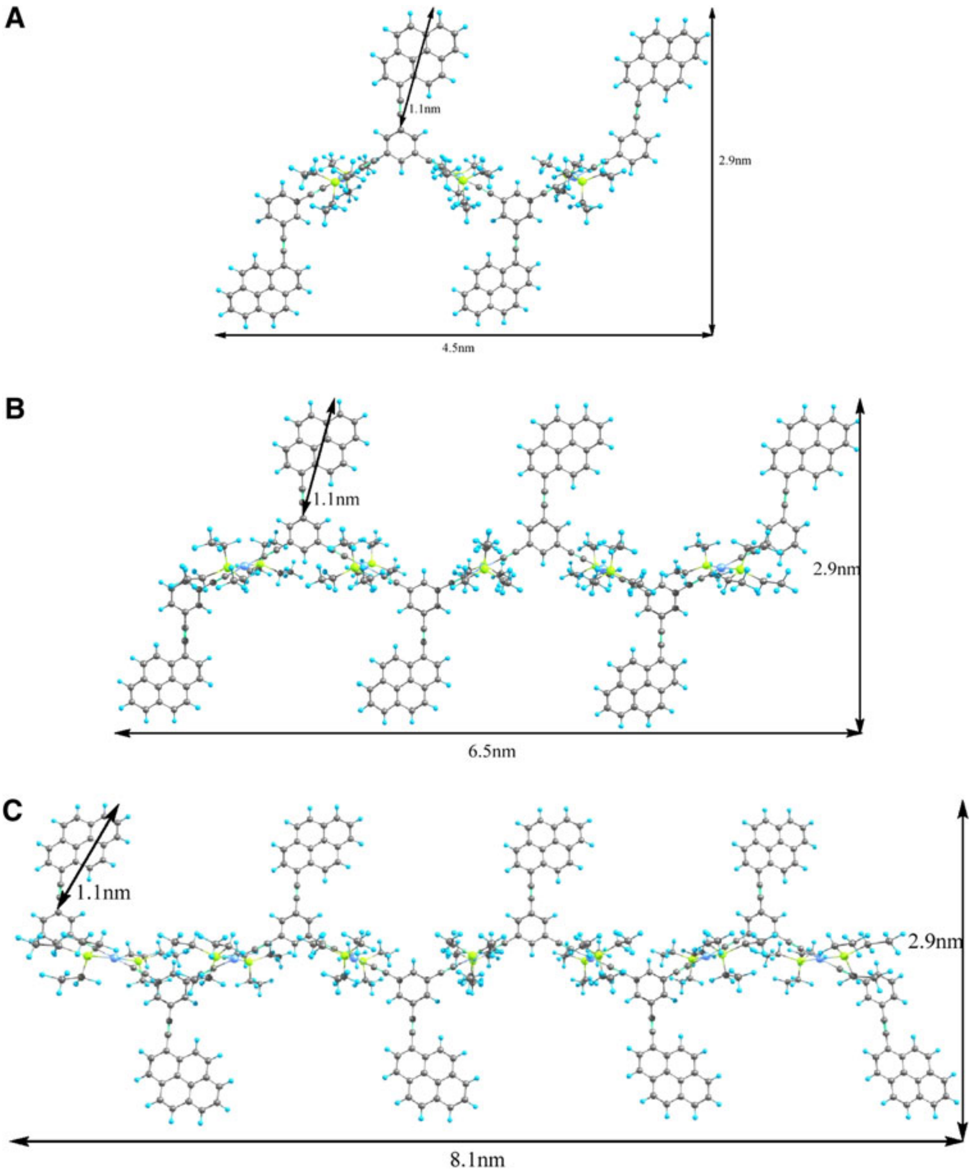


Fig. 3 Simulated molecular model of **15** (**a**), **16** (**b**), and **17** (**c**) optimized with PM6 semi-empirical molecular orbital methods

Table 1 UV–vis absorption and emission data for complexes **15**–**17**

Compound (concentration/M)	λ_{abs}/nm	ε/M^{-1} cm^{-1}	λ_F/nm	ΦF
15 (1.0 × 10^{-6})	391	72,165	398	0.023
	366	92,415	418	
	286	48,355		
16 (1.0 × 10^{-6})	391	171,095	398	0.014
	366	218,055	419	
	286	162,760		
17 (1.0 × 10^{-6})	392	284,410	398	0.011
	366	357,870	419	
	286	287,875	491	

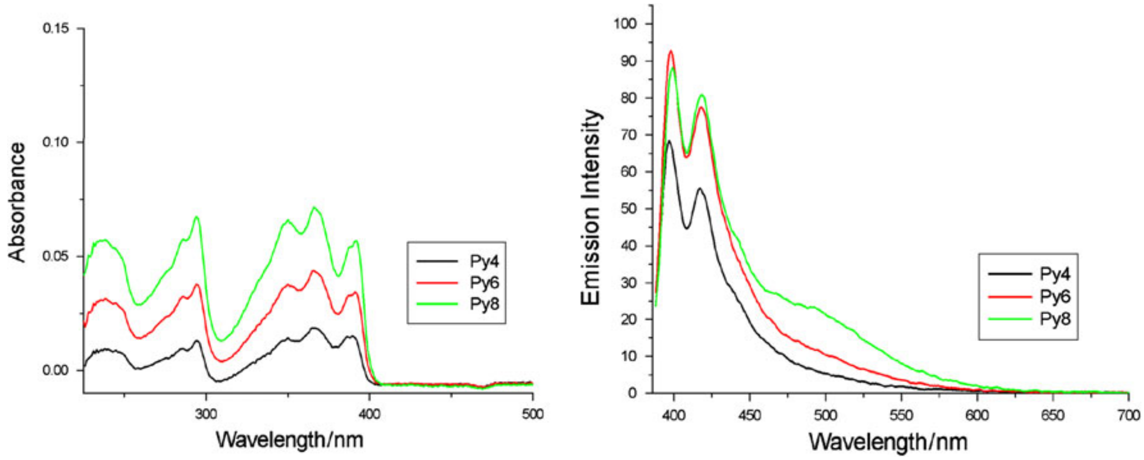


Fig. 4 UV–vis absorption (*left*) and fluorescence spectra (*right*) of **15**–**17** (all of them 1 μM) in CH_2Cl_2

results obviously enrich the library of functionalized conjugated oligomers and provide a new platform to design potential functional organometallic complexes and molecular devices.

Experimental

Toluene and THF were distilled from sodium; diethylamine was dried over potassium hydroxide; all of them were degassed under N_2 for 30 min before use. NMR solvents ($CDCl_3$) and all other reagents were of commercial quality and used without further purification.

^{1}H NMR, ^{13}C NMR, and ^{31}P NMR spectra were recorded on a Bruker 400 MHz spectrometer (^{1}H: 400 MHz, ^{13}C: 100 MHz, ^{31}P: 162 MHz) at 298 K. The ^{1}H and ^{13}C NMR chemical shifts are reported relative to residual solvent signals, and ^{31}P NMR resonances are referenced to an internal standard sample of 85 % H_3PO_4 ($\delta = 0.0$). Coupling constants (J) are denoted in Hz and chemical shifts (δ) in ppm. Multiplicities are denoted as follows: s = singlet, d = doublet, t = triplet, m = multiplet. Melting points were determined with an X-4 micromelting point apparatus.

UV–vis spectra were recorded on a Cary 50Bio UV–vis spectrophotometer. Emission spectra were measured on a Cary eclipse luminescence spectrometer. Samples for absorption and emission measurements were contained in 1-cm quartz cuvettes.

4-[3-Bromo-5-[(trimethylsilyl)ethynyl]phenyl]-2-methylbut-3-yn-2-ol (**1**, $C_{16}H_{19}BrOSi$)

Under an atmosphere of nitrogen, 3.00 g 1,3,5-tribromobenzene (9.53 mmol), 275 mg $Pd(PPh_3)_4$ (0.24 mmol), 45 mg CuI (0.24 mmol), and 1.38 cm^3 trimethylsilylacetylene (9.53 mmol) were dissolved in 30 cm^3 THF and 30 cm^3 *i*-Pr_2NH. The reaction mixture was stirred for 12 h at 65 °C, then 0.93 cm^3 2-methyl-3-butyn-2-ol (9.53 mmol) was added through a syringe and the mixture was allowed to continue stirring for 12 h. Then insoluble materials were filtrated through filter paper and the solvent was removed by evaporation on a rotary evaporator. The residue was purified by column chromatography on silica gel with petroleum ether (PE)/acetone (15:1, v/v) as eluent to afford colorless liquid **1** with a yield of 51 %. $R_f = 0.58$ (PE/acetone, 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz): $\delta = 7.53$ (s, 1H), 7.49 (s, 1H), 7.44 (s, 1H), 2.02 (br, 1H), 1.59 (s, 6H), 0.22 (s, 9H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): $\delta = 134.28$, 134.21, 133.60, 125.10, 124.67, 121.68, 103.15, 95.63, 94.84, 79.98, 31.33, −0.24 ppm; HRMS: *m*/*z* calcd for $C_{16}H_{19}BrOSi$ 334.0389, found 334.0388.

[[3-Ethynyl-5-(pyren-1-ylethynyl)phenyl]ethynyl]-trimethylsilane (**3**, $C_{31}H_{22}Si$)

A 100-cm^3 Schlenk flask was charged with 1.64 g **1** (4.88 mmol), 282 mg $Pd(PPh_3)_4$ (0.24 mmol), 46 mg CuI (0.24 mmol), 1.10 g 1-ethynylpyrene (4.86 mmol), and 30 cm^3 Et_3N under nitrogen atmosphere. The reaction mixture was stirred for 12 h at 65 °C, then insoluble materials were filtrated through filter paper and the solvent was then removed by evaporation on a rotary evaporator. The residue was purified by column chromatography on silica gel with PE/acetone (20:1, v/v) as eluent to afford **2** with a yield of 73 %. A mixture of 400 mg **2** (0.83 mmol) and 166 mg NaOH (4.15 mmol) in 10 cm^3 dry toluene was refluxed for 0.5 h. The filtrate was evaporated to give a dark yellow residue. The residue was purified by column chromatography on silica gel with PE/DCM (5:1, v/v) as eluent to afford yellow solid **3** with a yield of 70 %. $R_f = 0.54$ (PE/DCM, 5;1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz): $\delta = 8.59$ (d, $J = 9.2$ Hz, 1H), 8.23–8.01 (m, 8H), 7.80 (s, 1H), 7.77 (s, 1H), 7.62 (s, 1H), 3.16 (s, 1H), 0.31 (s, 9H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz):

δ = 134.93, 134.73, 131.90, 131.41, 131.08, 130.89, 129.53, 128.39, 128.26, 127.08, 126.16, 125.67, 125.60, 125.19, 124.39, 124.28, 124.22, 124.11, 123.99, 122.85, 116.90, 103.18, 96.03, 93.08, 90.01, 82.03, 78.45, −0.10 ppm; HRMS: *m/z* calcd for $C_{31}H_{22}Si$ 422.1491, found 422.1493.

[[3-(Pyren-1-ylethynyl)-5-[(trimethylsilyl)-ethynyl]phenyl]ethynyl]bis(triethylphosphine)-platinum(IV) iodide (**4**, $C_{43}H_{51}IP_2PtSi$)

CuI (9.5 mg, 0.03 mmol), 1.36 g *trans*-Pt(PEt_3)$_2$$I_2$ (1.98 mmol), and 420 mg **3** (0.99 mmol) were added to a mixed solvent of 30 cm^3 THF and 30 cm^3 Et_2NH under an atmosphere of nitrogen. The mixture was then stirred at room temperature for 2 h. The solvent was removed in vacuo and the residue was purified via column chromatography with PE/DCM (8:1, v/v) as eluent to afford yellow solid **4** with a yield of 88 %. R_f = 0.43 (PE/DCM, 2:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.65 (d, *J* = 8.8 Hz, 1H), 8.25–8.02 (m, 8H), 7.64 (s, 1H), 7.53 (s, 1H), 7.36 (s, 1H), 2.24 (br, 12H), 0.29 (s, 9H) ppm; ^{31}P NMR ($CDCl_3$, 161.9 MHz): δ = 8.94 ($J_{Pt–P}$ = 2,313.55 Hz) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 133.63, 133.53, 131.91, 131.81, 131.32, 131.17, 130.97, 129.54, 129.10, 128.33, 128.21, 127.15, 126.22, 125.62, 125.36, 124.46, 124.40, 124.22, 123.63, 123.44, 117.38, 104.19, 98.85, 94.82, 94.13, 92.46, 89.00, 16.65, 8.28, −0.04 ppm; MALDI HRMS: *m/z* calcd for $C_{43}H_{51}IP_2PtSi$ ($[M+H]^+$) 980.19, found 980.14.

1-[(3-Ethynylphenyl)ethynyl]pyrene (**5**, $C_{26}H_{14}$)

A 50-cm^3 Schlenk flask was charged with 336 mg [2-(3-bromophenyl)ethynyl]trimethylsilane (1.33 mmol), 77 mg Pd(PPh_3)$_4$ (0.07 mmol), 13 mg CuI (0.07 mmol), 300 mg 1-ethynylpyrene (1.33 mmol), and 10 cm^3 Et_3N under nitrogen atmosphere. The reaction mixture was stirred for 12 h at 70 °C, then insoluble materials were filtrated through filter paper and the solvent was removed by evaporation on a rotary evaporator. The residue was purified by column chromatography on silica gel with PE/DCM (20:1, v/v) as eluent to afford yellow solid trimethyl[[3-(pyren-1-ylethynyl)phenyl]ethynyl]silane with a yield of 74 %. A mixture of 391 mg trimethyl[[3-(pyren-1-ylethynyl)phenyl]ethynyl]silane (0.98 mmol) and 542 mg K_2CO_3 (3.93 mmol) in 10 cm^3 THF and 10 cm^3 MeOH was stirred at room temperature for 2.0 h. The filtrate was evaporated to give a dark yellow residue. The residue was purified by column chromatography on silica gel with PE/DCM (10:1, v/v) as eluent to afford yellow solid **5** with a yield of 92 %. R_f = 0.41 (PE/DCM, 10:1, v/v); m.p.: 134 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.61 (d, *J* = 9.2 Hz, 1H), 8.20–7.98 (m, 8H), 7.89 (s, 1H), 7.71 (d, *J* = 7.6 Hz, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 1H), 3.19 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 135.11, 131.85, 131.81, 131.31, 131.11, 130.92, 129.51, 128.51, 128.31, 128.15, 127.09, 126.13, 125.60, 125.54, 125.27, 124.39, 124.32, 124.15, 123.93, 122.56, 117.23, 93.94, 89.45, 82.88, 77.92 ppm; HRMS: *m/z* calcd for $C_{26}H_{14}$ 326.1096, found 326.1094.

[[3-Ethynyl-5-(pyren-1-ylethynyl)phenyl]ethynyl]-[[3-(pyren-1-ylethynyl)phenyl]ethynyl]bis-(triethylphosphine)platinum (**7**, $C_{66}H_{56}P_2Pt$)

A 50-cm^3 Schlenk flask was charged with 6.0 mg CuI (0.03 mmol), 617 mg **4** (0.63 mmol), and 308 mg **5** (0.94 mmol), then a mixed solvent of 10 cm^3 THF and 10 cm^3 Et_2NH was added under an atmosphere of nitrogen. The mixture was then stirred at room temperature for 1.5 h. The solvent was removed in vacuo and the residue was purified via column chromatography with PE/DCM (3:1, v/v) as eluent to afford yellow solid **6** with a yield of 72 %. Then 627 mg **6** (0.53 mmol) was dissolved in a mixture of 10 cm^3 THF and 10 cm^3 methanol, 294 mg K_2CO_3 (2.13 mmol) was added, and the solution was stirred at room temperature for 2.0 h. The filtrate was evaporated to give a dark yellow residue. The residue was purified by column chromatography on silica gel with PE/DCM (1:1, v/v) as eluent to afford yellow solid **7** with a yield of 97 %. R_f = 0.50 (PE/DCM, 1:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.66–8.64 (m, 2H), 8.25–8.02 (m, 16H), 7.62 (s, 2H), 7.58 (s, 1H), 7.50 (d, *J* = 7.2 Hz, 1H), 7.41 (s, 1H), 7.33–7.29 (m, 2H), 3.11 (s, 1H), 2.23 (br, 12H), 1.33–1.25 (m, 18H) ppm; ^{31}P NMR ($CDCl_3$, 161.9 MHz): δ = 11.88 ($J_{Pt–P}$ = 2,358.88 Hz), 8.57 ($J_{Pt–P}$ = 2,320.03 Hz) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 133.78, 133.33, 131.86, 131.83, 131.18, 131.14, 130.99, 131.33, 129.49, 129.15, 128.99, 128.55, 128.25, 128.17, 128.04, 127.16, 126.15, 125.51, 125.48, 124.45, 124.24, 123.17, 123.03, 117.81, 108.84, 108.70, 99.51, 95.26, 95.21, 90.34, 88.23, 88.09, 31.87, 29.64, 29.30, 22.64, 16.81, 16.63, 16.45, 16.41, 16.24, 8.39, 8.29 ppm.

[[[3-[[Bis(triethylphosphine)][3-(pyren-1-ylethynyl)-phenylethynyl]platinumethynyl]][5-(pyren-1-ylethynyl)]-phenyl]ethynyl]bis(triethylphosphine)platinum iodide (**8**, $C_{78}H_{85}IP_4Pt_2$)

Under an atmosphere of nitrogen, 2.7 mg CuI (0.01 mmol), 97 mg *trans*-Pt(PEt_3)$_2$$I_2$ (0.14 mmol), and 79 mg compound **7** (0.07 mmol) were dissolved in 20 cm^3 THF and 20 cm^3 *i*-Pr_2NH. The reaction mixture was stirred at room temperature for 2 h. The solvent was removed in vacuo and the residue was purified via column chromatography with PE/DCM (1:1, v/v) as eluent to afford a yellow solid of **8** with a yield of 70 %. R_f = 0.40 (PE/DCM, 1:1, v/v); m.p.: 105 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.69–8.67 (d, *J* = 8.8 Hz, 2H), 8.25–8.02 (m, 16H), 7.63 (s, 1H), 7.51 (d, *J* = 8.8 Hz, 1H), 7.45 (s, 1H), 7.39 (s, 1H), 7.34–7.29 (m, 2H), 2.25 (br, 24H), 1.33-1.17 (m, 36H) ppm; ^{31}P NMR ($CDCl_3$, 161.9 MHz): $\delta = 11.59$ ($J_{\text{Pt–P}} = 2367.14$ Hz), 8.92 ($J_{\text{Pt–P}} = 2320.67$ Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta =$ 133.78, 133.34, 131.87, 131.83, 131.18, 131.14, 130.99, 130.33, 129.50, 129.16, 128.99, 128.55, 128.26, 128.18, 128.04, 127.17, 126.16, 125.51, 125.49, 124.45, 124.24, 123.17, 123.03, 117.81, 108.84, 108.70, 99.51, 95.26, 95.21, 90.35, 88.28, 88.09, 16.64, 16.42, 8.39, 8.29 ppm.

[[[[3-Ethynyl-5-(pyren-1-ylethynyl)]phenyl]ethynyl]]-[[[3-[[bis(triethylphosphine)][3-(pyren-1-ylethynyl)-phenylethynyl]platinumethynyl]][5-(pyren-1-ylethynyl)]phenyl]ethynyl]bis(triethylphosphine)platinum (**10**, $C_{106}H_{98}P_4Pt_2$)

Compound **10** was prepared from **4** and **7** by using the similar procedure described for preparation of **7**. Yellow solid; yield 85 %; $R_f = 0.41$ (PE/DCM, 1:1, v/v); ^{1}H NMR (CDCl$_3$, 400 MHz): $\delta =$ 8.70–8.64 (m, 3H), 8.23–8.02 (m, 24H), 7.62 (s, 2H), 7.58 (s, 1H), 7.50 (d, $J = 7.2$ Hz, 1H), 7.41 (br, 3H), 7.32 (m, 3H), 3.11 (s, 1H), 2.24 (br, 24H), 1.33–1.27 (m, 36H) ppm; ^{31}P NMR (CDCl$_3$, 161.9 MHz): $\delta = 11.60$ ($J_{\text{Pt–P}} = 2{,}368.60$ Hz), 11.51 ($J_{\text{Pt–P}} = 2{,}368.60$ Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta =$ 134.24, 134.11, 133.81, 133.68, 131.92, 131.88, 131.85, 131.53, 131.31, 131.20, 131.11, 131.02, 130.98, 129.51, 129.21, 128.84, 128.33, 128.19, 128.04, 127.18, 126.20, 126.16, 125.62, 125.58, 125.51, 125.38, 124.45, 124.27, 124.22, 123.62, 123.18, 122.87, 122.30, 117.95, 117.85, 117.41, 109.00, 108.80, 107.93, 95.45, 95.29, 94.21, 88.95, 88.28, 87.93, 83.03, 77.45, 31.89, 29.66, 29.32, 22.66, 16.42, 8.40 ppm; MALDI HRMS: *m/z* calcd for $C_{106}H_{98}P_4Pt_2$ ([M+H]$^+$) 1,885.59, found 1,885.53.

[[3-[Iodide-[bis(triethylphosphine)]platinumethynyl]]-[5-(pyren-1-ylethynyl)phenyl]ethynyl][[[3-[(triethylphosphine)][3-(pyren-1-ylethynyl)phenylethynyl] platinumethynyl]][5-(pyren-1-ylethynyl)]phenyl]ethynyl]bis(triethylphosphine)platinum (**11**, $C_{118}H_{127}IP_6Pt_3$)

Compound **11** was prepared from **10** by using the similar procedure described for preparation of **8**. Yellow solid; yield 70 %; $R_f = 0.43$ (PE/DCM, 1:1, v/v); m.p.: 138 °C; ^{1}H NMR (CDCl$_3$, 400 MHz): $\delta =$ 8.70 (d, $J = 9.2$ Hz, 3H), 8.25–8.02 (m, 24H), 7.64 (s, 1H), 7.51–7.40 (m, 5H), 7.32 (br, 4H), 2.25 (br, 36H), 1.33–1.19 (m, 54H) ppm; ^{31}P NMR (CDCl$_3$, 161.9 MHz): $\delta = 11.56$ ($J_{\text{Pt–P}} = 2{,}365.84$ Hz), 8.91 ($J_{\text{Pt–P}} = 2{,}320.84$ Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta =$ 134.24, 134.13, 133.82, 133.69, 131.92, 131.88, 131.85, 131.54, 131.31, 131.20, 131.17, 131.14, 131.11, 131.02, 130.98, 130.75, 129.53, 129.51, 129.22, 128.87, 128.79, 128.32, 128.19, 128.04, 127.18, 127.15, 126.20, 126.16, 125.62, 125.57, 125.51, 125.37, 124.45, 124.40, 124.27, 124.22, 123.63, 123.19, 122.89, 122.31, 117.95, 117.85, 117.41, 110.96, 109.02, 108.88, 108.81, 108.42, 108.09, 107.94, 95.46, 95.30, 94.21, 88.96, 88.30, 87.94, 83.04, 77.46, 16.44, 8.41 ppm.

[[[3-[Bis(triethylphosphine)][[3-ethynyl-5-(pyren-1-ylethynyl)phenyl]ethynyl]platinumethynyl][5-(pyren-1-ylethynyl)]phenyl]ethynyl][[[3-[[bis(triethylphosphine)][3-(pyren-1-ylethynyl)phenylethynyl]platinumethynyl]][5-(pyren-1-ylethynyl)]phenyl]ethynyl]bis(triethylphosphine)platinum (**13**, $C_{146}H_{140}P_6Pt_3$)

Compound **13** was prepared from **4** and **10** by using the similar procedure described for preparation of **7**. Yellow solid; yield 70 %; $R_f = 0.40$ (PE/DCM, 1:2, v/v); m.p.: 147 °C; ^{1}H NMR (CDCl$_3$, 400 MHz): $\delta =$ 8.71–8.64 (m, 4H), 8.21–8.02 (m, 32H), 7.65–7.31 (m, 13H), 3.15 (s, 1H), 2.26 (br, 36H), 1.31 (br, 48H) ppm; ^{31}P NMR (CDCl$_3$, 161.9 MHz): $\delta = 11.90$ ($J_{\text{Pt–P}} = 2{,}374.91$ Hz), 11.80 ($J_{\text{Pt–P}} = 2374.91$ Hz), 11.77 ($J_{\text{Pt–P}} = 2{,}374.91$ Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta =$ 134.23, 134.11, 133.80, 133.69, 131.88, 131.52, 131.31, 131.19, 131.11, 131.02, 130.69, 129.51, 129.20, 128.84, 128.32, 128.16, 128.03, 127.18, 126.16, 125.57, 125.50, 125.37, 124.45, 124.26, 123.62, 123.18, 122.86, 122.30, 117.95, 117.84, 117.41, 109.00, 108.86, 107.91, 95.46, 95.28, 94.21, 88.95, 88.29, 87.91, 83.02, 77.46, 16.41, 8.40 ppm; MALDI HRMS: *m/z* calcd for $C_{146}H_{140}P_6Pt_3$ ([M+H]$^+$) 2,664.83, found 2,664.49.

[[[3-[Bis(triethylphosphine)][[3-[iodide-[bis(triethylphosphine)]platinum]ethynyl]-[5-(pyren-1-ylethynyl)phenyl]ethynyl]platinumethynyl]-[5-(pyren-1-ylethynyl)]phenyl]ethynyl][[[3-[[bis(triethylphosphine)][3-(pyren-1-ylethynyl)phenylethynyl]-platinumethynyl]][5-(pyren-1-ylethynyl)]phenyl]ethynyl]bis(triethylphosphine)platinum (**14**, $C_{158}H_{169}IP_8Pt_4$)

Compound **14** was prepared from **13** by using the similar procedure described for preparation of **8**. Yellow solid; yield 70 %; $R_f = 0.45$ (PE/DCM, 1:2, v/v); m.p.: 136 °C; ^{1}H NMR (CDCl$_3$, 400 MHz): $\delta =$ 8.70 (d, $J = 8.8$ Hz, 4H), 8.25–8.02 (m, 32H), 7.63 (s, 1H), 7.50–7.39 (m, 9H), 7.32 (br, 3H), 2.25 (br, 48H), 1.33–1.19 (m, 72H) ppm; ^{31}P NMR (CDCl$_3$, 161.9 MHz): $\delta = 11.72$ ($J_{\text{Pt–P}} = 2{,}363.90$ Hz), 11.70 ($J_{\text{Pt–P}} = 2{,}363.90$ Hz), 9.06 ($J_{\text{Pt–P}} = 2{,}321.48$ Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta =$ 133.82, 133.68, 133.35, 131.93, 131.26, 131.20, 131.16, 131.08, 130.71, 129.56, 128.84, 128.24, 128.19, 128.09, 127.24, 126.21, 125.64, 125.57, 125.54, 124.50, 124.33, 124.00, 123.47, 123.18, 123.03, 122.85, 118.03, 117.91, 115.92, 108.88, 95.31, 88.08, 16.63, 8.64, 8.32 ppm.

[Bis[[[3-[[bis(triethylphosphine)][3-(pyren-1-ylethynyl)phenylethynyl]platinumethynyl]][5-(pyren-1-ylethynyl)]phenyl]ethynyl]]bis(triethylphosphine)platinum (**15**, $C_{144}H_{140}P_6Pt_3$)

Under an atmosphere of nitrogen, 2.0 mg CuI (0.01 mmol), 55 mg compound **7** (0.05 mmol), and 83 mg **8** (0.05 mmol) were dissolved in 10 cm^3 THF and 10 cm^3 Et$_2$NH. The reaction mixture was stirred at room temperature for

1.5 h. The solvent was removed in vacuo and the residue was purified via column chromatography with PE/DCM (1:1, v/v) as eluent to afford a yellow solid of **15** in 73 % yield. $R_f = 0.40$ (PE/DCM, 1:2, v/v); m.p.: 151 °C; ^{1}H NMR (CDCl$_3$, 400 MHz): $\delta = 8.70$ (d, $J = 9.2$ Hz, 4H), 8.25–8.02 (m, 32H), 7.63 (s, 2H), 7.50 (d, $J = 7.2$ Hz, 3H), 7.41 (s, 4H), 7.32 (m, 6H), 2.25 (br, 36H), 1.30 (m, 54H) ppm; ^{31}P NMR (CDCl$_3$, 161.9 MHz): $\delta = 11.65$ ($J_{\text{Pt–P}} =$ 2,362.77 Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta =$ 133.82, 133.69, 131.91, 131.89, 131.23, 131.18, 131.14, 131.06, 130.70, 129.55, 128.80, 128.23, 128.19, 128.08, 127.22, 126.19, 125.63, 125.55, 124.49, 124.31, 123.18, 122.85, 118.01, 117.89, 108.87, 95.30, 88.27, 16.44, 8.42 ppm; MALDI HRMS: *m/z* calcd for $C_{144}H_{140}P_6Pt_3$ ([M+H]$^+$) 2,640.83, found 2,640.50.

[Bis[[3-[[[3-[[bis(triethylphosphine)][3-(pyren-1-ylethynyl)phenylethynyl]platinumethynyl]]-[5-(pyren-1-ylethynyl)]phenyl]ethynyl][bis(triethylphosphine)]platinumethynyl][5-(pyren-1-ylethynyl)]-phenylethynyl]]bis(triethylphosphine)platinum (**16**, $C_{224}H_{224}P_{10}Pt_5$)

Compound **16** was prepared from **11** and **10** by using the similar procedure described for preparation of **15**. Yellow solid; yield 51 %; $R_f = 0.48$ (PE/DCM, 1:1, v/v); m.p.: 155 °C; ^{1}H NMR (CDCl$_3$, 400 MHz): $\delta = 8.70$ (d, $J = 9.2$ Hz, 6H), 8.25–8.02 (m, 48H), 7.63 (s, 2H), 7.49 (br, 3H), 7.42 (s, 7H), 7.32 (br, 6H), 2.24 (br, 60H), 1.33–1.26 (m, 90H) ppm; ^{31}P NMR (CDCl$_3$, 161.9 MHz): $\delta = 11.30$ ($J_{\text{Pt–P}} = 2{,}358.07$ Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta = 133.80$, 133.61, 131.93, 131.91, 131.25, 131.18, 131.08, 131.73, 129.57, 128.82, 128.22, 128.10, 127.24, 126.21, 125.56, 124.50, 124.33, 123.22, 117.96, 108.90, 95.25, 88.31, 16.48, 8.45 ppm; MALDI HRMS: *m/z* calcd for $C_{224}H_{224}P_{10}Pt_5$ ([M+H]$^+$) 4,202.30, found 4,203.09.

[Bis[[[3-[[bis(triethylphosphine)[3-[[[3-[[bis(triethylphosphine)][3-(pyren-1-ylethynyl)phenylethynyl]-platinumethynyl]]][5-(pyren-1-ylethynyl)]phenyl]ethynyl]-[bis(triethylphosphine)]platinumethynyl][5-(pyren-1-ylethynyl)]phenylethynyl]platinumethynyl]]][5-(pyren-1-ylethynyl)]phenyl]ethynyl]]bis(triethylphosphine) platinum (**17**, $C_{304}H_{308}P_{14}Pt_7$)

Compound **17** was prepared from **14** and **13** by using the similar procedure described for preparation of **15**. Yellow solid; yield 92 %; $R_f = 0.41$ (PE/DCM, 1:4, v/v); m.p.: 158 °C; ^{1}H NMR (CDCl$_3$, 400 MHz): $\delta = 8.70$ (d, $J = 8.8$ Hz, 8H), 8.22–8.04 (m, 64H), 7.70 (s, 4H), 7.62 (s, 3H), 7.48 (m, 5H), 7.40 (s, 8H), 2.24 (br, 84H), 1.31–1.26 (m, 126H) ppm; ^{31}P NMR (CDCl$_3$, 161.9 MHz): $\delta = 11.56$ ($J_{\text{Pt–P}} = 2{,}369.73$ Hz) ppm; ^{13}C NMR (CDCl$_3$, 100 MHz): $\delta = 133.82$, 131.93, 131.27, 130.72, 129.57, 128.83, 128.20, 127.25, 126.21, 125.55, 124.51, 122.84, 118.04, 108.90, 95.51, 87.88, 29.67, 16.45, 8.43 ppm; MALDI HRMS: *m/z* calcd for $C_{304}H_{308}P_{14}Pt_7$ ([M+H]$^+$) 5,760.88, found 5,767.65.

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