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Synthesis and structural properties of novel polycyclic aromatic compounds using photo-induced cyclisation of 2,7-di-*tert*-butyl-4-(phenylethenyl)pyrenes

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Wittig reaction of 2,7-di-*tert*-butylpyrene-4-carbaldehyde with benzyltriphenylphosphonium salts in the presense of *n*-BuLi gave 2,7-di-*tert*-butyl-4-(phenylethenyl)pyrenes, from which naphthalene condensed aromatic compounds, 2,7-di-*tert*-butyl-dibenzo[*ij*,*no*]tetraphenes, were obtained by photo-induced cyclisation in the presense of iodine.

Keywords: pyrenes, Wittig reaction, (phenylethenyl)pyrene, photo-induced cyclisation, dibenzo[*ij*,*no*]tetraphene

The phenanthrene nucleus is present in a large number of natural substances, some of them having valuable chemotherapeutic properties, which has resulted in considerable efforts being made to synthesise them.[1–4] Despite the various approaches now avalable, there exist no efficient method for the preparation of phenanthrene annulated pyrenes at positions 4 and 5 as in **1**.

On the other hands, since electrophilic substitution of pyrene occurred at 1, 3, 6, and 8 positions, but not at the other positions (2, 4, 5, 7, 9, and 10).[5–12] Therefore, pyrenes substituted at the latter positions must be prepared in ways other than by direct electrophilic substitution of pyrene itself.[13] For example, Moyle *et al.*[10] prepared 4,9-diethylpyrene in a low total yield from ethylbenzene in 14 steps using Friedel–Crafts tramolecular acylation to construct a pyrene ring. Thus there is substantial interest in investigating the selective introduction of substituents at positions 4, 5, 9 and 10 in ways other than by direct electrophilic substitution of pyrene itself.

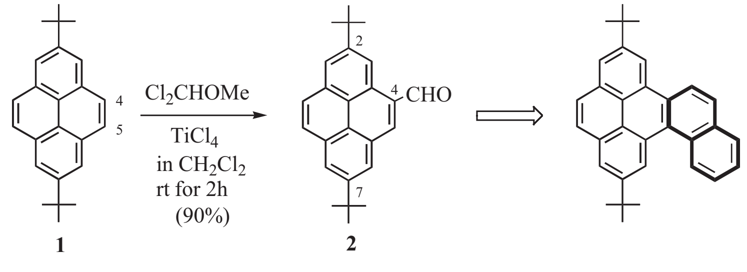
We previously reported the $TiCl_4$-catalysed formylation of 2,7-di-*tert*-butylpyrene (**1**) with dichloromethyl methyl ether using the *tert*-butyl group as a positional protective group to afford only 4-monoformylated product, 2,7-di-*tert*-butylpyrene-4-carbaldehyde **2** in 90% yield.[14,15] This compound **2** was a convenient starting material for the preparation of 4,5-naphthalene annulated pyrenes by using photo-induced cyclisation of 2,7-di-*tert*-butyl-4-(phenylethenyl)pyrenes. We report here synthesis and structural properties of novel 4,5-naphthalene condensed pyrenes, in which the [4]helicene[16,17] structures are contained.

Results and discussion

The preparation of 2,7-di-*tert*-butylpyrene-4-carbaldehyde **2** was carried out according to the reported procedure.[14,15] Thus, formylation of **1** with dichloromethyl methyl ether in the presence of titanium tetrachloride occurred selectively at 4-position to afford the corresponding 4-formyl derivative **2** in 90% yield. The reaction of **2** and the (4-methoxybenzyl)triphenyl-phosphonium chloride **3b** with *n*-butyllithium in THF gave the desired 2,7-di-*tert*-butyl-4-(4-meth oxyphenylethenyl)pyrene **4b** in 85% yield, whose *E*/*Z* ratio was 80 : 20. The *E*/*Z* ratio of the product was determined by its NMR spectrum. The *E* isomer was isolated pure in 65% yield by silica gel column chromatography and recrystallisation from hexane. Attempted isolation of the pure *Z*-isomer failed. Similarly, (*E*)-2,7-di-*tert*-butyl-4-(phenylethenyl)pyrene **4a** was prepared in 85% yield.

The structures of products *(E)*-**4b** and *(Z)*-**4b** were determined on the basis of their elemental analyses and spectral data. ^1H NMR signals of the olefinic protons for *E*-olefins should be observed at lower magnetic field ($\delta > 7.4$ ppm) than that of *Z*-olefins ($\delta < 6.9$ ppm).[18] ^1H NMR spectrum of *(E)*-**4b** in $CDCl_3$ shows a singlet at δ 3.88 for methoxy protons, a pair of doublets (J = 15.9 Hz) at δ 7.35, 7.91 ppm for olefinic protons, and a pair of doublets (J = 8.4 Hz) at δ 6.99, 7.63 ppm for aromatic protons. The structure of the *(Z)*-isomer *(Z)*-**4b** is also readily assigned from the smaller coupling constant (J = 8.8 Hz) than that of (*E*)-**4b** (J = 15.9 Hz) at δ 7.14, 7.43 ppm for olefinic protons. Furthermore, the methoxy protons and the aromatic protons are observed much higher field (δ 3.62 ppm and δ 6.52, 6.85 ppm) than those of (*E*)-**4b** (δ 3.88 ppm and δ 6.99, 7.63 ppm) due to the ring current of the pyrene ring. Also one of the *tert*-butyl protons of *(Z)*-**4b** was observed at higher field, δ 1.46 ppm due to the shielding effect of the aromatic ring. These data strongly support that the structure of (*E*)-**4b** is the *(E)*-configuration, and the structure of *(Z)*-**4b** is the *(Z)*-configuration.

When a solution of (*E*)-**4a** and a stoichiometric amount of iodine in benzene was irradiated with a high-pressure murcury lamp (400W) at room temperature for 6 h, the photocyclisation product, 2,7-di-*tert*-butyldibenzo[*ij*,*no*]tetraphene (**5a**) was obtained only 20% along with the recovery of the starting compound. Prolonging the reaction time to 24 h led to an increase of the yield to 30%. Interestingly, when the irradiation



Scheme 1

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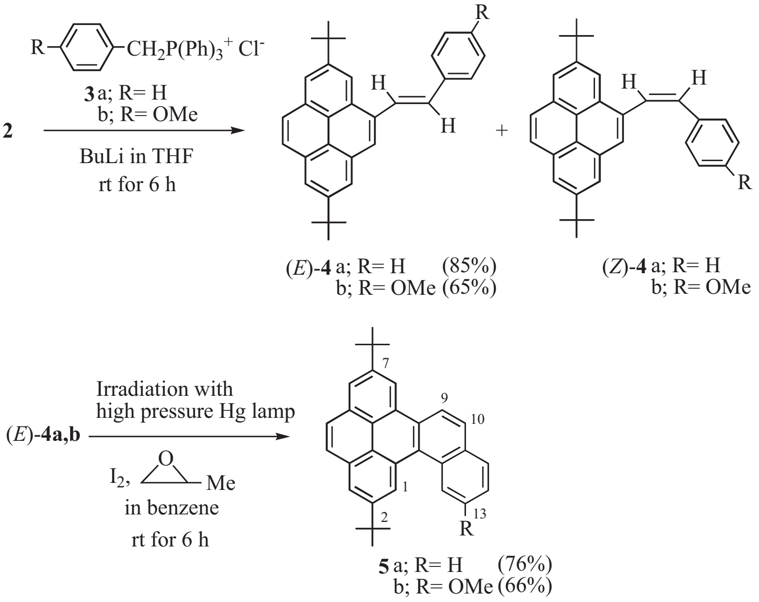


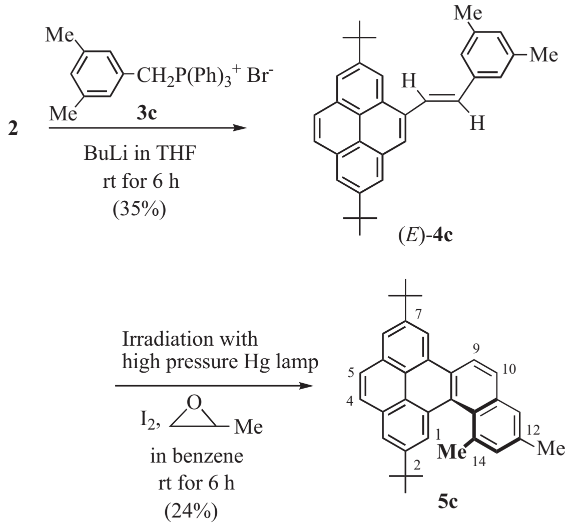
Scheme 2

was carried out at room temperature for 6 h with a stoichiometric amount of iodine plus a large amount of propylene oxide[19,20] in the absence of air led to an increase of the yield of the desired cyclisation product **5a** to 76%. The propylene oxide prevents HI from photoreducing double bond. Also absence of air prevents photooxidative side reactions, such as causing by a photogenerated oxidant, possibly hydrogen peroxide.

A similar result was obtained in the case of (*E*)-**4b** and 2,7-di-*tert*-butyl-13-methoxydibenzo[*ij,no*]tetraphene **5b** was obtained in 66% yield as light-yellow prisms.

The structures of products **5a** and **5b** were determined on the basis of their elemental analyses and spectral data. Thus, the structures of **5a** and **5b** were established on the basis of the base peak molecular ion at *m/z* 414 and 444 in their mass spectrum, respectively. ^1H NMR spectrum of **5b** in $CDCl_3$ shows a singlet at δ 4.02 ppm for methoxy proton, a pair of doublets ($J = 9.0$ Hz) at δ 8.72, 7.98 ppm for H_9, H_{10} protons, and a pair of doublets ($J = 8.3$ Hz) at δ 8.03, 8.06 ppm for H_4, H_5 protons. Further the 1- and 14-aryl hydrogens can clearly be seen to be deshielded at δ 9.33 ppm ($J = 1.5$ Hz) and 8.57 ppm ($J = 2.4$ Hz), respectively by the adjacent ring, a common consequence of the expanded benzene ring.[17]

To investigate this finding in more detail, we have introduced methyl group at C_{14} position for dibenzo[*ij,no*] tetraphenes which could lead the increased skeletal distortion like benzo[*c*]phenanthrenes.[17] Thus, the reaction of **2** and the (3,5-dimethylbenzyl)triphenylphosphonium bromide **3c** with *n*-butyllithium in THF gave the desired (*E*)-2,7-di-*tert*-butyl-4-(3,5-dimethylphenylethynyl)pyrene (*E*)-**4c** in 35% yield. When a solution of (*E*)-**4c** and a stoichiometric amount of iodine in the presence of propylene oxide in benzene was irradiated with a high pressure murcury lamp (400W) under the same conditions as in (*E*)-**4a**, the photocyclisation product, 2,7-di-*tert*-butyl-12,14-dimethyldibenzo[*ij,no*]tetraphene (**5c**) was obtained in 24% yield as light yellow prisms. ^1H NMR spectrum of **5c** in $CDCl_3$ shows a singlet at δ 2.30, 2.63 ppm for methyl protons, a pair of doublets ($J = 9.0$ Hz) at δ 8.67, 7.96 ppm for H_9, H_{10} protons, and a pair of doublets ($J = 8.3$ Hz) at δ 8.03 ppm for H_4, H_5 protons. Further, as expected, the 1-hydrogen H_1 and one of the methyl groups at the 14-position can clearly be seen to be deshielded at δ 8.88 ppm ($J = 1.5$ Hz) and δ 2.63 ppm, respectively by the adjacent ring, a common consequence of the expanded benzene ring.[17]



Scheme 3

Consequently, we have succeeded in preparing a series of substituted (phenylethenyl)pyrene derivatives **4** and dibenzo[*ij,no*]tetraphene derivatives **5**. The UV spectra of (phenylethenyl)pyrene derivatives **4** in CH_2Cl_2 along with that of 2,7-di-*tert*-butylpyrene (**1**) are shown in Fig. 1. The spectra were recorded in CH_2Cl_2 in the range of 10^{-5}–10^{-6} M concentration. For these (phenylethenyl)pyrene derivatives **4**, the spectra are almost identical and three absorption bands were observed in the range of 285–360 nm. The longest wavelength π–π* bands of these (phenylethenyl)pyrene derivatives **4** are bathochromically shifted by 10–20 nm in comparison with that of 2,7-di-*tert*-butylpyrene (**1**) due to the introduction of the phenylethenyl group. On the other hand, the increased bathochromic shift of (*E*)-**4b** (*e.g.* 8 nm) in comparison with that of (*E*)-**4a** were observed which are ascribed to the increased π-electron density on the benzene ring arising from methoxy group introduced.

On the other hand, the UV spectra of dibenzo[*ij,no*]tetraphene derivatives **5** in CH_2Cl_2 are almost identical and four absorption bands were observed in the range of 280–380 nm.

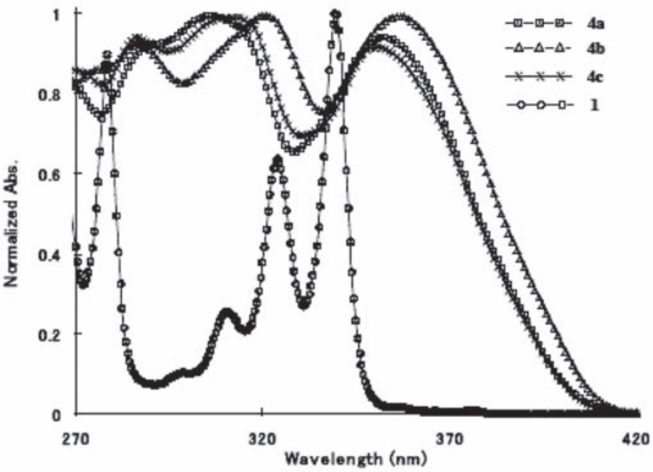


Fig. 1 Electronic absorption spectra of compounds **4a–c** and **1** in dichloromethane at room temperature (before cyclisation).

The quite different shape in the UV spectrum of **5** in comparison with those of **1** and **4** are ascribed to the expanded conjugation of π-electron system by the cyclisation reaction. Interestingly, the slightly different shape in the UV spectrum of **5c** confirms the increased non-planarity of the aromatic ring to avoid the sterically crowding by the overlapping at the C$_1$ proton and the methyl group at the C$_{14}$ position leading to an increase of the strain of these systems.[16,17]

Conclusions

We conclude that the Wittig reaction of 2,7-di-*tert*-butylpyrene-4-carbaldehyde with benzyltriphenylphosphonium salts in the presense of *n*-BuLi should be useful for the preparation of 2,7-di-*tert*-butyl-4-(phenylethenyl)pyrenes **4**. Photo-induced cyclisation in the presense of iodine and propylene oxide led to the novel expanded π-conjugated systems, dibenzo[*ij,no*]tetraphene derivatives **5**, in which the first number of the [*n*]helicene structures are contained. Further chemical and structural properties of the present novel dibenzo[*ij,no*]tetraphene derivatives **5** are currently under study in our laboratory.

Experimental

All melting points are uncorrected. ^{1}H NMR spectra were recorded at 300 MHz on a Nippon Denshi JEOL FT-300 NMR spectrometer in deuteriochloroform with Me$_4$Si as an internal reference. UV-vis spectra were recorded on a Perkin Elmer Lambda 19 UV/VIS/NIR spectrometer. Mass spectra were obtained on a Nippon Denshi JMS-HX110A Ultrahigh Performance Mass Spectrometer at 75 eV using a direct-inlet system. Elemental analyses were performed by Yanaco MT-5.

Materials: Preparation of 2,7-di-*tert*-butylpyrene-4-carbaldehyde (**2**) was previously described.[14,15]

*Typical procedure for Wittig reactions of 2,7-di-tert-butylpyrene-***4***-carbaldehyde* **2**

To a solution of benzyltriphenylphophonium chloride **3a** (1.15 g, 3.0 mmol) in THF (15 cm^3) was added *n*-BuLi (1.6 M solution in hexane) (3.5 cm^3, 3.0 mmol) at room temperature. After the solution was stirred for 10 min, the solution of 2,7-di-*tert*-butylpyrene-4-carbaldehyde **2** (342 mg, 1.0 mmol) in THF (15 cm^3) was added. The reaction mixture was stirred at room temperature for 6 h under argon, and then it was poured into a large amount of ice-water and extracted with ethyl acetate (100 cm^3 × 2). The extract was washed with water and brine, dried over anhydrous MgSO$_4$, and concentrated under reduced pressure. The residue was chromatographed over silica gel (Wako C-300, 200 g) with hexane-ethyl acetate (5:1) as eluent to give (*E*)-**4a** as light-yellow solids. Recrystallisation from hexane afforded (*E*)-2,7-di-*tert*-butyl-4-(phenylethenyl)pyrene (*E*)-**4a** (354 mg, 85%) as light-yellow prisms, m.p. 188–190 °C (dec.); δ_H (CDCl$_3$): 1.59 (9H, s, *t*Bu), 1.60 (9H, s, *t*Bu), 7.34 (1H, t, *J* = 7.5 Hz, Ar*H*), 7.40 (1H, d, *J* = 15.9 Hz, pyrene–CH$_b$ =CH_a–Ar), 7.46 (2H, t, *J* = 7.5 Hz, Ar*H*), 7.70 (2H, d, *J* = 7.5 Hz, Ar*H*), 8.03 (2H, s, pyrene–$H_{9,10}$), 8.06 (1H, d, *J* = 15.9 Hz, pyrene–CH_b =CH$_a$–Ar), 8.16 (1H, d, *J* = 1.8 Hz, pyrene–H_1), 8.22, 8.25 (2H, each d, *J* = 1.8 Hz, pyrene–$H_{6,8}$), 8.29

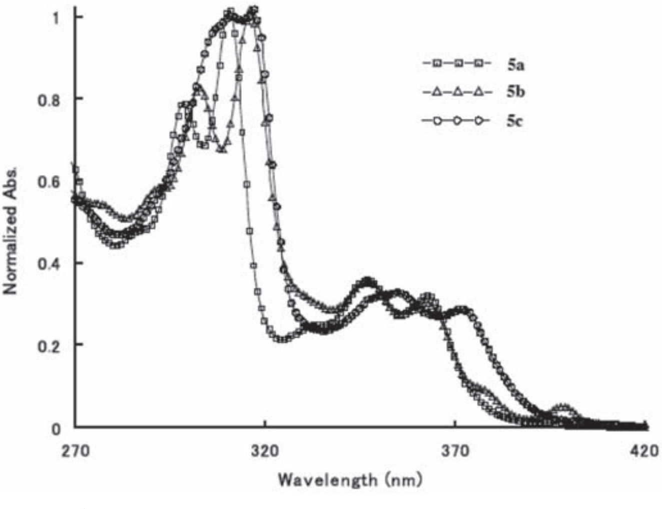


Fig. 2 Electronic absorption spectra of compounds **5a–c** in dichloromethane at room temperature (after cyclisation).

(1H, s, pyrene–H_5), 8.49 (1H, d, J = 1.8 Hz, pyrene–H_3); *m*/*z*: 416 (M^+). Anal. calcd. for $C_{32}H_{32}$ (416.60): C, 92.26; H, 7.74. Found: C, 91.78; H, 7.94.

Similarly, (*E*)-**4b** and (*E*)-**4c** were obtained in 65% and 35% yields, respectively. In the case of **4b**, a mixture of (*E*)-**4b** and (*Z*)-**4b** was obtained in the ratio of 80:20 (determined by ^1H NMR spectrum) in 85% yield. (*E*)-**4b** isomer was obtained pure by recrystallisation from hexane in 65% yield as light-yellow prisms, but several attempts to obtain (*Z*)-isomer in pure failed.

(E)-2,7-Di-tert-butyl-4-(4-methoxyphenylethenyl)pyrene (*E*)-(**4b**): (290 mg, 65%) Light-yellow prisms, m.p. 224–226 °C; δ_H ($CDCl_3$): 1.59 (18H, s, *t*Bu), 3.88 (3H, s, O*Me*), 6.99 (2H, d, J = 8.4 Hz, Ar*H*), 7.35 (1H, d, J = 15.9 Hz, pyrene–CH_b =CH_a–Ar), 7.63 (2H, d, J = 8.4 Hz, Ar*H*), 7.91 (1H, d J = 15.9 Hz, pyrene–CH_b =CH_a–Ar), 8.02 (2H, s, pyrene–$H_{9,10}$), 8.15 (1H, d, J = 1.8 Hz, pyrene–H_1), 8.21 (2H, d, J = 1.8 Hz, pyrene–$H_{6,8}$), 8.26 (1H, s, pyrene–H_5), 8.49 (1H, d, J = 1.5 Hz, pyrene–H_3); *m*/*z*: 446 (M^+). Anal. calcd. for $C_{33}H_{34}O$ (446.26): C, 88.74; H, 7.67. Found: C, 88.36; H, 7.61.

(Z)-2,7-Di-tert-butyl-4-(4-methoxyphenylethenyl)pyrene (*Z*)-(**4b**): δ_H ($CDCl_3$): 1.46 (9H, s, *t*Bu), 1.55 (9H, s, *t*Bu), 3.62 (3H, s, O*Me*), 6.52 (2H, d, J = 7.5 Hz, Ar*H*), 6.85 (2H, d, J = 7.5 Hz, Ar*H*), 7.14 (1H, d, J = 8.8 Hz, pyrene–CH_b =CH_a–Ar), 7.43 (1H, d J = 8.8 Hz, pyrene–CH_b =CH_a–Ar), 7.71 (2H, s, pyrene–H_5), 7.82 (1H, d, J = 1.5 Hz, pyrene–H_6), 7.98, 8.12 (1H, each d, J = 1.5 Hz, pyrene–$H_{9,10}$), 8.15 (1H, d, J = 1.5 Hz, pyrene–H_1), 8.23 (1H, d, J = 1.5 Hz, pyrene–H_8), 8.36 (1H, d, J = 1.5 Hz, pyrene–H_3).

(E)-2,7-Di-tert-butyl-4-(3,5-dimethylphenylethenyl)pyrene (*E*)-(**4c**): (128 mg, 35%) Light-yellow prisms, m.p. 141–142 °C; δ_H ($CDCl_3$): 1.58 (9H, s, *t*Bu), 1.59 (9H, s, *t*Bu), 2.41 (6H, s, *Me*), 7.00 (1H, s, Ar*H*), 7.31 (2H, s, Ar*H*), 7.32 (1H, d, J = 15.9 Hz, pyrene–CH_b =CH_a–Ar), 8.00 (1H, d J = 15.9 Hz, pyrene–CH_b =CH_a–Ar), 8.028, 8.031 (2H, each s, pyrene–$H_{9,10}$), 8.16 (1H, d, J = 1.8 Hz, pyrene–H_1), 8.21 (2H, d, J = 1.8 Hz, pyrene–$H_{6,8}$), 8.25 (1H, s, pyrene–H_5), 8.48 (1H, d, J = 1.5 Hz, pyrene–H_3); *m*/*z*: 444 (M^+). Anal. calcd. for $C_{34}H_{36}$ (444.66): C, 91.84; H, 8.16. Found: C, 91.63; H, 8.25.

Photo-irradiation of **4a**: *typical procedure*

2,7-Di-*tert*-butyl-4-(phenylethenyl)pyrene **4a** (50 mg, 0.119 mmol), I_2 (28.3 mg, 0.12 mmol) and propylene oxide (1.53 cm^3, 21.1 mmol) was dissolved in benzene (260 cm^3), and the solution was irradiated with a high-pressure Hg lamp (400 W) for 6 h. The reaction mixture was poured into ice-water (300 cm^3) and extracted with ethyl acetate (50 $cm^3 \times 3$). After the ethyl acetate solution had been washed successively with 10% sodium thiosulfate, water and brine the extract was dried over anhydrous sodium sulfate and concentrated. The residue was washed with methanol (10 cm^3) to afford 2,7-di-*tert*-butyl-dibenzo[*ij,no*]tetraphene **5a** (37.6 mg, 76%) as light-yellow prisms, m.p. 163–165 °C; δ_H ($CDCl_3$): 1.62 (9H, s, *t*Bu), 1.64 (9H, s, *t*Bu), 7.65–7.68 (2H, m, $H_{12,13}$), 8.05, 8.06 (2H, each d, J = 8.3 Hz, $H_{4,5}$), 8.08 (1H, d, J = 5.7 Hz, H_{11}), 8.09 (1H, d, J = 9.0 Hz, H_{10}), 8.21, 8.23 (2H, each d, J = 1.5 Hz, $H_{3,6}$), 8.86 (1H, d, J = 9.0 Hz, H_9), 8.95 (1H, d, J = 1.5 Hz, H_8), 9.16 (1H, dd, J = 1.2, 7.8 Hz, H_{14}), 9.27 (1H, d, J = 1.2 Hz, H_1); *m*/*z*: 414 (M^+). Anal. calcd. for $C_{32}H_{30}$ (414.58): C, 92.71; H, 7.29. Found: C, 92.78; H, 7.49.

Similarly, **5b** and **5c** were obtained in 66 and 24% yields, respectively.

2,7-Di-tert-butyl-13-methoxydibenzo[ij,no]tetraphene (**5b**): Light-yellow prisms, m.p. 173–174 °C; δ_H ($CDCl_3$): 1.63 (9H, s, *t*Bu), 1.62 (9H, s, *t*Bu), 4.02 (3H, s, O*Me*), 7.30(1H, dd, J = 2.4, 9.0 Hz, H_{12}), 7.98 (1H, d, J = 9.0 Hz, H_{10}), 8.02 (1H, d, J = 9.0 Hz, H_{11}), 8.03, 8.06 (2H, each d, J = 8.3 Hz, $H_{4,5}$), 8.18, 8,20 (2H, each d, J = 1.5 Hz, $H_{3,6}$), 8.57(1H, d, J = 2.4 Hz, H_{14}), 8.72 (1H, d, J = 9.0 Hz, H_9), 8.93 (1H, d, J = 1.5 Hz, H_8), 9.33 (1H, d, J = 1.5 Hz, H_1); *m*/*z*: 444 (M^+). Anal. calcd. for $C_{33}H_{32}O$ (444.61): C, 89.15; H, 7.25. Found: C, 89.09; H, 7.26.

2,7-Di-tert-butyl-12,14-dimethyldibenzo[ij,no]tetraphene (**5c**): Light-yellow prisms, m.p. 110–111 °C; δ_H ($CDCl_3$): 1.54 (9H, s, *t*Bu), 1.63 (9H, s, *t*Bu), 2.30 (3H, s, Me_{12}), 2.63 (3H, s, Me_{14}), 7.35 (1H, s, H_{13}), 7.69 (1H, s, H_{11}), 7.96 (1H, d, J = 8.7 Hz, H_{10}), 8.03 (2H, each d, J = 8.3 Hz, $H_{4,5}$), 8.15, 8.21 (2H, each d, J = 1.5 Hz, $H_{3,6}$), 8.26 (1H, each d, J = 1.5 Hz, H_8), 8.67 (1H, d, J = 8.7 Hz, H_9), 8.88 (1H, d, J = 1.5 Hz, H_1); *m*/*z*: 442 (M^+). Anal. calcd. for $C_{34}H_{34}$ (442.65): C, 92.26; H, 7.77. Found: C, 92.37; H, 7.64.

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