Res Chem Intermed (2013) 39:139–146
DOI 10.1007/s11164-012-0638-2

Synthesis of 8-bromo-5,12-tetracenequinone and 2-bromotetracene derivatives

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Received: 26 September 2011 / Accepted: 30 November 2011 / Published online: 12 June 2012

Abstract A series of bromotetracenequinones **1** and bromotetracenes **2** were prepared from 4-bromophthalic anhydride. The parent tetracenequinone **1a** and tetracene **2a** were sparingly soluble in organic solvents. In contrast, dipropyl-substituted tetracenequinone **1b** and tetracene **2b** were a little more soluble. Preparation of dihexyl-substituted tetracene **2c** proved to be difficult. Sonogashira coupling of **1b** with trimethylsilylacetylene afforded the corresponding product.

Keywords Tetracene · Tetracenequinone · Bromo derivative · Alkyl side-chain · Solubility

Introduction

Oligoacenes have been intensively studied owing to their importance as organic semiconducting materials [1, 2]. Tetracene and pentacene are among the most promising molecular conductors for organic field-effect transistors, organic light-emitting diodes, and photovoltaic cells. However, they have limited solubility in organic solvents. Therefore, to improve the solubility of these molecules, many research groups have investigated acene functionalization and have reported the synthesis of various substituted oligoacenes [3–5]. Because oligomerization of anthracene has afforded some organic semiconductors with high field-effect mobilities [6], oligomerization of tetracene is also expected to improve its semiconducting properties. Very few studies of tetracene oligomers have been reported, with the exception of publications from the Merio et al. [7], Müller et al. [8], and Kimoto et al. [9] groups. This lack of extensive research can be attributed to

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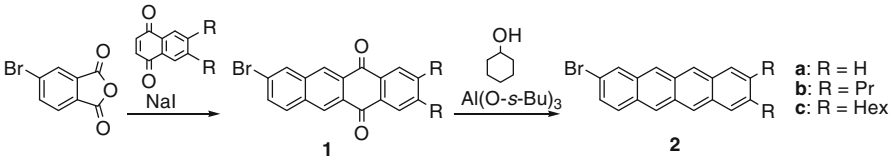
the limited availability of bromo-substituted tetracene as the building block required for various metal-catalyzed cross-coupling reactions [10]. Although 5-bromotetracene can be readily prepared via the bromination of tetracene with CuBr [11] or *N*-bromosuccinimide (NBS) [8], the preparation of 2-bromotetracene **2a** and its precursor 8-bromo-5,12-tetracenequinone **1a** has not been reported (Scheme 1). We became aware that commercially available 4-bromophthalic anhydride is a desirable starting material for preparing bromotetracenes **2** via bromotetracenequinones **1** (Scheme 1). Therefore, we decided to prepare **1** and **2**.

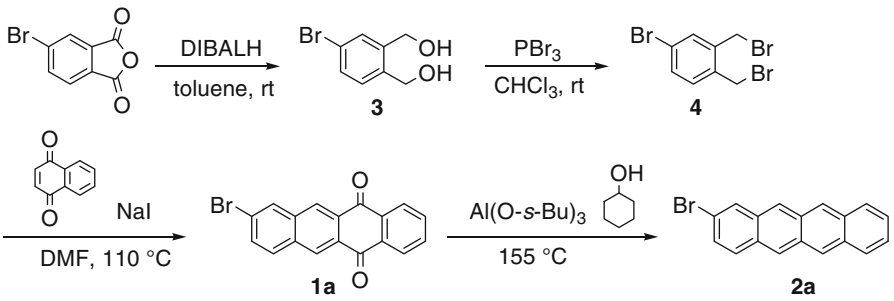
Results and discussion

As shown in Scheme 2, the parent **1a** and **2a** were synthesized. Reduction of 4-bromophthalic anhydride with diisobutylaluminum hydride (DIBAL-H) produced 1,2-bis(hydroxymethyl)benzene **3** in 79 % yield. Treatment of **3** with PBr_3 afforded 1,2-bis(bromomethyl)benzene **4** in 97 % yield. To obtain **1**, we adopted McOmie's protocol [12]. Reaction of **4** with 1,4-naphthoquinone in the presence of NaI at 110 °C in *N*,*N*-dimethylformamide (DMF), which generated the corresponding *o*-quinodimethane in situ, followed by subsequent Diels–Alder reaction with 1,4-naphthoquinone and oxidation afforded **1a** as a yellow solid in 44 % yield. Compound **1a** had poor solubility in common organic solvents. To transform **1a** into the corresponding tetracene **2a**, we utilized an improved Meerwein–Ponndorf reaction as described by Coffey and Boyd [13]. Heating **1a** with aluminum tri(cyclohexyl oxide), which was formed in situ by the reaction of aluminum tri(*s*-butoxide) and cyclohexanol, at 155 °C for 2 days, followed by vacuum sublimation using a gradient furnace gave **2a** as an orange solid in 32 % yield. We found that **2a** had lower solubility in organic solvents than **1a**. Thus, the ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra of **2a** could not be recorded. On the other hand, as for **1a**, the ^{13}C NMR spectrum could not be recorded. The structure of **2a** was supported by elemental analysis, infrared (IR) and mass spectrometry, while **1a** was characterized by elemental analysis, IR, ^{1}H NMR, and mass spectrometry. Therefore, **2a** may be an inadequate substrate for cross-coupling reactions.

To improve the solubility of **1a** and **2a** in organic solvents, we attempted to introduce alkyl side-chains onto the tetracene frameworks of **1a**. We anticipated that the use of alkyl-substituted 1,4-naphthoquinone would afford alkyl-substituted bromotetracenequinones **1** and bromotetracenes **2** (Scheme 2). Recently, we achieved the synthesis of 2,3-dialkyl-5,12-tetracenequinone via a Diels–Alder reaction between 1,4-anthraquinone and 3,4-dialkylthiophene-1,1-dioxides **5** [14].



Scheme 1 Transformation of 4-bromophthalic anhydride into **2** via **1**

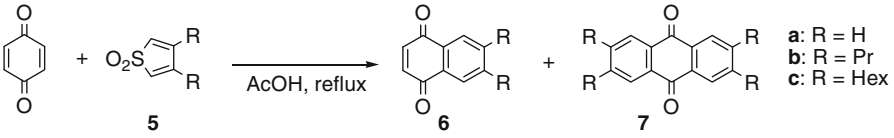


Scheme 2 Synthesis of **1a** and **2a**

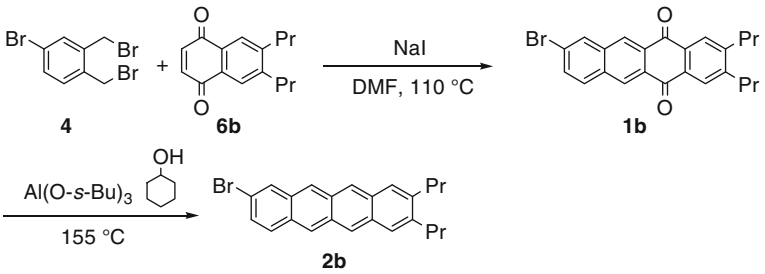
Therefore, instead of 1,4-anthraquinone, we used 1,4-benzoquinone to obtain 6,7-dialkyl-1,4-naphthoquinones **6** (Scheme 3). The Diels–Alder reactions of **5b** and **5c** with 1,4-benzoquinone, followed by the loss of sulfur dioxide and oxidation, afforded naphthoquinones **6b** and **6c** in 42 % and 37 % yields, respectively. As a by-product of the reaction, anthraquinones **7b** and **7c**, which were double Diels–Alder adducts, could also be isolated in 8 % and 10 % yields, respectively.

Next, we tried to prepare the dipropyl-substituted bromotetracenequinone **1** and bromotetracene **2** (Scheme 4). The iodine-induced reaction of **4** with **6b** afforded 8-bromo-2,3-dipropyl-5,12-tetracenequinone **1b** in 39 % yield. A Meerwein–Ponndorf reaction of **1b** gave 8-bromo-2,3-dipropyltetracene **2b** in 17 % yield. The dipropyl-substituted molecules **1b** and **2b** did not have high solubilities but were more soluble than the parent **1a** and **2a**. The tetracenequinone **1b** was relatively more soluble than the tetracene **2b**. The ^{13}C NMR spectrum of **2b** could not be recorded due to the low solubility. Thus, **1b** was fully characterized by elemental analysis, IR, ^{1}H and ^{13}C NMR, and mass spectroscopy, while **2b** was characterized by elemental analysis, IR, ^{1}H NMR, and mass spectroscopy.

To improve the solubility, we attempted to prepare the dihexyl-substituted bromotetracenequinone **1** and bromotetracene **2** (Scheme 5). The reaction of **4** and naphthoquinone **6b** in the presence of NaI gave 8-bromo-2,3-dihexyl-5,12-tetracenequinone **1c** in 8 % yield. The compound **1c** had good solubility and was completely characterized by elemental analysis, IR, ^{1}H and ^{13}C NMR, and mass spectroscopy. We were not able to perform the subsequent Meerwein–Ponndorf reaction of **1c** because the obtained amount of **1c** was small. Although introducing a longer alkyl chain length in bromotetracene is desirable for improving its solubility in organic solvents, it was found that the introduction of a longer alkyl chain



Scheme 3 Synthesis of **6**



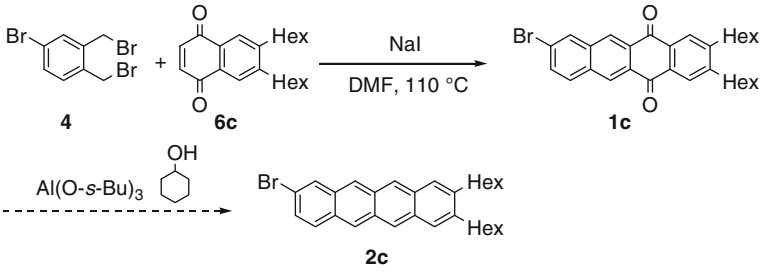
Scheme 4 Synthesis of **1b** and **2b**

inhibited the synthesis of bromotetracenequinones and bromotetracenes using our synthetic method.

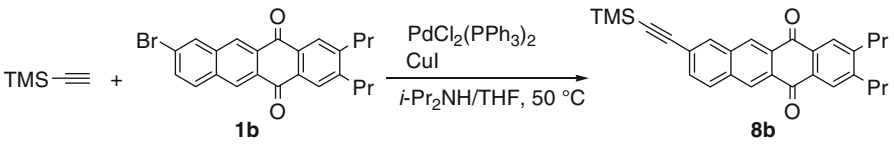
As we were able to prepare **1b** in reasonable quantities compared with the other isolated compounds such as **2b** and **1c**, we evaluated the Sonogashira coupling of **1b** with trimethylsilylacetylene. Reaction of **1b** with trimethylsilylacetylene in the presence of $PdCl_2(PPh_3)_2$, and CuI in *i*-Pr_2NH/tetrahydrofuran (THF) gave (trimethylsilylethynyl)tetracenequinone **8b** in 84 % yield (Scheme 6). Hence, oligomerization of **1b** and subsequent transformation into the corresponding soluble tetracene derivatives may prove to be an effective route to obtain tetracene-based oligomers.

Experimental

All reagents were commercially available and used without further purification. 3,4-Dialkylthiophene-1,1-dioxides were prepared according to the literature method [14]. All reactions were performed under a nitrogen atmosphere. Column chromatography was performed using a Wako C-300 silica-gel column (45–75 μm). Melting points were measured on a Yanaco melting-point apparatus. IR spectra of KBr pressed pellet samples were measured with a Shimadzu FTIR-8400 spectrometer. ^1H and ^{13}C NMR spectra were measured using a Bruker-Biospin DRX500 FT spectrometer. Electron impact (EI) mass spectra were measured on a Shimadzu GCMS-QP5050A mass spectrometer. Elemental analyses were performed using a Yanaco MT-5 CHN corder.



Scheme 5 Synthesis of **1c**



Scheme 6 Sonogashira coupling of **1b**

Synthesis of 4-bromo-1,2-bis(hydroxymethyl)benzene **3**

To an ice-cooled solution of 4-bromophthalic anhydride (459 mg, 2.02 mmol) in toluene (8 mL), 1 M DIBAL-H in toluene (10 mL, 10 mmol) was added dropwise. Then, the mixture was stirred at room temperature for 20 h. After cooling with ice, the reaction mixture was quenched with MeOH (2 mL) and conc. HCl (60 mL). After separation of the organic layer, the aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine and dried over Na_2SO_4. After evaporation of the solvents, column chromatography (CH_2Cl_2/AcOEt = 1:1) on silica gel afforded **3** (348 mg, 79 %) as a white solid: mp 65.5–66.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.33 (brs, 2H), 4.63 (s, 2H), 4.63 (s, 2H), 7.19 (d, J = 8.0 Hz, 1H), 7.43 (d, J = 8.0 Hz, 1H), 7.48 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 63.4, 63.4, 122.2, 131.2, 131.3, 132.4, 138.0, 141.4.

Synthesis of 4-bromo-1,2-bis(bromomethyl)benzene **4**

To an ice-cooled solution of **3** (872 mg, 4.02 mmol) in $CHCl_3$ (40 mL), a solution of PBr_3 (0.95 mL, 10.1 mmol) in $CHCl_3$ (5 mL) was added dropwise. The mixture was stirred at room temperature for 4 h and then poured into ice water. The organic layer was separated, washed with aqueous Na_2CO_3 and brine, and then dried over Na_2SO_4. After removal of the solvent and drying under vacuum, **4** was obtained as a pale yellow solid (1.33 g, 97 %) and used in the next reaction without further purification: mp 54–55 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.58 (s, 2H), 4.59 (s, 2H), 7.24 (d, J = 8.2 Hz, 1H), 7.43 (dd, J = 2.1, 8.2 Hz, 1H), 7.52 (d, J = 2.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 28.7, 29.0, 123.0, 132.4, 132.5, 133.9, 135.5, 138.5.

Synthesis of 8-bromo-5,12-tetracenequinone **1a**

A mixture of **4** (1.36 g, 3.96 mmol), 1,4-naphthoquinone (769 mg, 4.86 mmol), and NaI (2.97 g, 19.8 mmol) in DMF (18 mL) was stirred at 110 °C for 22 h. The reaction mixture was cooled to room temperature and then poured into aqueous Na_2SO_3. The resulting solid was filtered and washed with water and acetone. After drying under vacuum, **1a** was obtained as a yellow solid (591 mg, 44 %) and used in the next reaction without further purification: mp >300 °C; IR (cm^{-1}) 3,065, 1,672, 1,611, 1,593, 1,580, 1,449, 1,398, 1,325, 1,288, 1,063, 964, 939, 818, 708; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, J = 8.7 Hz, 1H), 7.84–7.86 (m, 2H), 7.98 (d, J = 8.7 Hz, 1H), 8.28 (s, 1H), 8.40–8.42 (m, 2H), 8.78 (s, 1H), 8.84 (s, 1H); MS m/z 100 (100), 336 (M^+, 88), 338 (M^+, 86); Anal. Calcd. for $C_{18}H_9BrO_2$: C, 64.12, H, 2.69; found: C, 64.22, H, 2.98.

Synthesis of 2-bromotetracene **2a**

A mixture of **1a** (999 mg, 2.96 mmol) and Al(O-*s*-Bu)$_3$ (7.83 g, 31.8 mmol) in cyclohexanol (20 mL) was stirred for 48 h at 155 °C. After cooling to room temperature, the reaction mixture was quenched with a solution of MeOH (18 mL), water (10 mL), and conc. HCl (5 mL). The resulting solid was filtered, washed with MeOH, and dried under vacuum. Vacuum sublimation using a gradient furnace provided **2a** as an orange solid (131 mg, 32 %): Decomp. 270 °C; IR (cm^{-1}) 3,048, 1,612, 1,601, 1,559, 1,458, 1,292, 1,047, 957, 934, 903, 793, 739; MS *m*/*z* 306 (M^+, 100), 308 (M^+, 80); Anal. Calcd. for $C_{18}H_{11}Br$: C, 70.38, H, 3.61; found: C, 70.74, H, 3.93.

Synthesis of 6,7-dipropyl-1,4-naphthoquinone **6b**

A mixture of 3,4-dipropylthiophene-1,1-dioxide **5b** (499 mg, 2.49 mmol) and 1,4-benzoquinone (1.35 g, 12.5 mmol) in AcOH (20 mL) was stirred at reflux for 16 h. After cooling to room temperature, the reaction mixture was poured into water, extracted with CH_2Cl_2, washed with brine, and dried over Na_2SO_4. After removal of the solvent, the residue was subjected to column chromatography (CH_2Cl_2/hexane = 1:1 → CH_2Cl_2) on silica gel, affording **6b** as a yellow solid (253 mg, 42 %) and anthraquinone by-product **7b** as a pale yellow solid (72 mg, 8 %). Data for **6b**: mp 53–54 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.02 (t, J = 7.4 Hz, 6H), 1.63–1.71 (m, 4H), 2.72 (t, J = 7.9 Hz, 4H), 6.91 (s, 2H), 7.85 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 23.8, 34.9, 127.1, 129.7, 138.6, 147.6, 185.4. Data for **7b**: mp 129–131 °C; δ 1.03 (t, J = 7.4 Hz, 12H), 1.66–1.74 (m, 8H), 2.74 (t, J = 7.9 Hz, 8H), 8.05 (s, 4H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.2, 23.9, 34.9, 127.7, 131.5, 147.5, 183.6.

Synthesis of 6,7-dihexyl-1,4-naphthoquinone **6c**

This compound was synthesized following the same procedure as for **6b** except that reagents 3,4-dihexylthiophene-1,1-dioxide **5c** (576 mg, 2.03 mmol) and 1,4-benzoquinone (1.09 g, 10.1 mmol) were used. Compound **6c** was obtained as a yellow oil (246 mg, 37 %), and the anthraquinone by-product **7c** was also obtained as a yellow solid (112 mg, 10 %). Data for **6c**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89–0.91 (m, 6H), 1.31–1.42 (m, 12H), 1.59–1.65 (m, 4H), 2.72 (t, J = 8.0 Hz, 4H), 6.91 (s, 2H), 7.84 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 29.3, 30.7, 31.6, 32.9, 127.1, 129.7, 138.6, 147.9, 185.4. Data for **7c**: mp 90–91.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90 (t, J = 6.9 Hz, 12H), 1.33–1.42 (m, 24H), 1.62–1.68 (m, 8H), 2.75 (t, J = 8.0 Hz, 8H), 8.04 (s, 4H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 29.4, 30.8, 31.7, 33.0, 127.7, 131.5, 147.7, 183.6.

Synthesis of 8-bromo-2,3-dipropyl-5,12-tetracenequinone **1b**

This compound was synthesized following the same procedure as for **1a** except that reagents **4** (363 mg, 1.06 mmol), **6b** (250 mg, 1.03 mmol), NaI (773 mg,

5.16 mmol), and DMF (5 mL) were used and the product was recrystallized from THF–hexane. Compound **1b** was obtained as a yellow solid (171 mg, 39 %): mp 256–258 °C; IR (cm^{-1}) 3,067, 2,957, 2,928, 2,870, 1,672, 1,597, 1,452, 1,398, 1,314, 1,296, 1,184, 1,063, 997, 930, 806, 739; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.04–1.07 (m, 6H), 1.71–1.75 (m, 4H), 2.76–2.79 (m, 4H), 7.76 (d, $J = 8.8$ Hz, 1H), 7.97 (d, $J = 8.8$ Hz, 1H), 8.16 (s, 2H), 8.27 (s, 1H), 8.74 (s, 1H), 8.81 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.2, 23.9, 35.0, 123.8, 128.1, 129.1, 130.3, 130.9, 131.4, 132.0, 132.1, 132.7, 133.4, 136.0, 148.2, 148.2, 182.8; MS *m/z* 420 (M^+, 99), 422 (M^+, 100); Anal. Calcd. for $C_{24}H_{21}BrO_2$: C, 68.42, H, 5.02; found: C, 68.57, H, 5.27.

Synthesis of 2-bromo-8,9-dipropyltetracene **2b**

This compound was synthesized following the same procedure as for **2a** except that reagents **1b** (190 mg, 0.45 mmol), Al(O-*s*-Bu)$_3$ (1.12 g, 4.54 mmol), and cyclohexanol (10 mL) were used and the product was recrystallized from THF–hexane. Compound **2b** was obtained as an orange solid (29 mg, 17 %). The bromotetracene **2b** in solution was slightly unstable when exposed to light and air, and therefore it required handling in the dark: Decomp. 270 °C; IR (cm^{-1}) 3,005, 2,955, 2,930, 2,870, 1,636, 1,605, 1,464, 1,458, 1,381, 1,296, 1,121, 1,044, 910, 895, 790; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.07–1.10 (m, 6H), 1.56–1.80 (m, 4H), 2.77–2.80 (m, 4H), 7.39 (d, $J = 9.1$ Hz, 1H), 7.75 (s, 2H), 7.85 (d, $J = 9.1$ Hz, 1H), 8.15 (s, 1H), 8.51 (s, 1H), 8.53 (s, 1H), 8.54 (s, 1H), 8.58 (s, 1H); MS *m/z* 390 (M^+, 100), 392 (M^+, 99); Anal. Calcd. for $C_{24}H_{23}Br$: C, 73.66, H, 5.92; found: C, 73.84, H, 6.09.

Synthesis of 8-bromo-2,3-dihexyl-5,12-tetracenequinone **1c**

This compound was synthesized following the same procedure as for **1a** except that reagents **4** (175 mg, 0.51 mmol), **6c** (166 mg, 0.51 mmol), NaI (386 mg, 2.57 mmol), and DMF (3 mL) were used and the product was recrystallized from THF–hexane. Compound **1c** was obtained as a yellow solid (20 mg, 8 %): mp 237–238 °C; IR (cm^{-1}) 3,067, 2,955, 2,924, 2,855, 1,672, 1,597, 1,455, 1,402, 1,312, 1,298, 1,184, 1,065, 943, 739; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90–0.93 (m, 6H), 1.34–1.44 (m, 12H), 1.65–1.71 (m, 4H), 2.76–2.80 (m, 4H), 7.75 (d, $J = 8.7$ Hz, 1H), 7.96 (d, $J = 8.8$ Hz, 1H), 8.15 (s, 2H), 8.26 (s, 1H), 8.73 (s, 1H), 8.80 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 29.4, 30.8, 31.7, 33.0, 123.8, 128.1, 129.2, 130.4, 130.9, 131.5, 132.0, 132.1, 132.7, 133.4, 136.0, 148.5, 148.5, 182.9; MS *m/z* 421 (100), 504 (M^+, 34), 506 (M^+, 42); Anal. Calcd. for $C_{30}H_{33}BrO_2$: C, 71.04, H, 6.69; found: C, 71.28, H, 6.58.

Synthesis of 2,3-dipropyl-8-trimethylsilylethynyl-5,12-tetracenequinone **8b**

To a degassed THF solution (15 mL) of **1b** (106 mg, 0.25 mmol), trimethylsilylacetylene (0.2 mL, 1.42 mmol), CuI (8 mg, 0.058 mmol), and *i*-Pr_2NH (2 mL), $PdCl_2(PPh_3)_2$ (9 mg, 0.026 mmol) was added. The reaction mixture was stirred at 50 °C for 16 h. After cooling to room temperature and then evaporating

the solvents, the residue was subjected to column chromatography (CH_2Cl_2/hexane = 1:1 → CH_2Cl_2) on silica gel to afford **8b** (92 mg, 84 %) as a yellow solid: mp 211–213 °C; IR (cm^{-1}) 3,056, 2,954, 2,928, 2,870, 2,151, 1,674, 1,601, 1,587, 1,460, 1,404, 1,317, 1,302, 1,248, 1,194, 995, 868, 839, 758, 739; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.31 (s, 9H), 1.04–1.07 (m, 6H), 1.69–1.77 (m, 4H), 2.76–2.79 (m, 4H), 7.68 (d, J = 8.5 Hz, 1H), 8.02 (d, J = 8.5 Hz, 1H), 8.16 (s, 2H), 8.21 (s, 1H), 8.77 (s, 1H), 8.79 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 0.0, 14.3, 23.9, 35.0, 97.8, 104.3, 124.1, 128.0, 129.0, 130.0, 130.5, 130.6, 131.9, 132.1, 132.2, 133.6, 134.4, 134.6, 148.1, 148.1, 182.8, 182.9; MS m/z 423 (100), 438 (M^+, 60); Anal. Calcd. for $C_{24}H_{30}O_2Si$: C, 79.24, H, 7.02; found: C, 79.41, H, 6.89.

Conclusions

We have developed a synthetic route to 8-bromo-5,12-tetracenequinone and 2-bromotetracene derivatives (**1** and **2**) from 4-bromophthalic anhydride. To improve the solubility of **1a** and **2a** in organic solvents, dialkyl-substituted bromotetracenequinones **1b–c** and bromotetracene **2b** were prepared. Bromotetracenequinones **2** were found to be slightly more soluble than bromotetracenes **1**. Sonogashira coupling of 8-bromo-2,3-dipropyl-5,12-tetracenequinone **1b** with trimethylsilylacetylene afforded the corresponding product **8b** in high yield.

Acknowledgments This work was supported by Grants-in-Aid for Scientific Research (nos. 23550161 and 23108720) from JSPS and MEXT.

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