

Supplementary Information of the manuscript:

Two-dimensional Self-Assembly of Linear Molecular Rods at Liquid/Solid Interface

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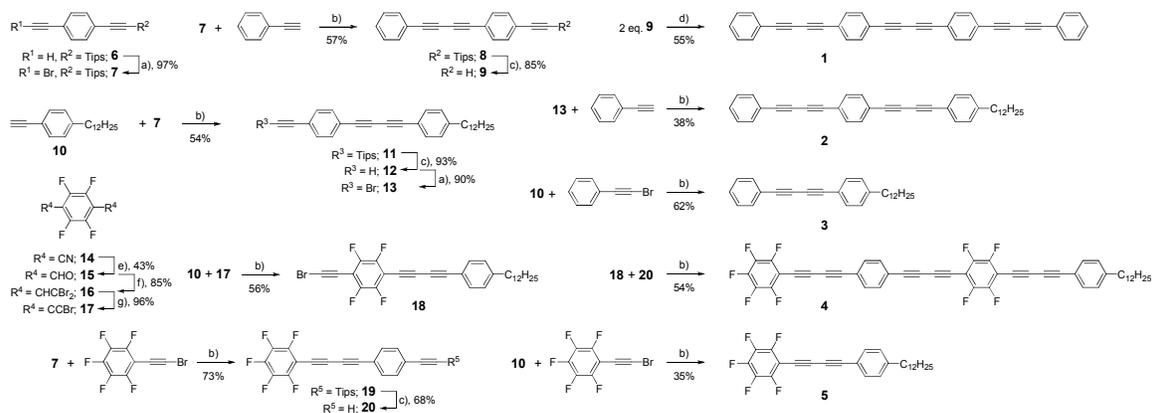
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Synthesis and characterization of the molecular rods 1-5: The synthesis of the molecular rods **1-5**

is displayed in scheme 1.



Scheme 1: Synthesis of the molecular rods **1-5**. a) NBS, AgNO₃, CH₃COCH₃, r.t.; b) Pd₂(dba)₃·CHCl₃, CuI, C₆H₅CH₃, EtN(*i*-prop.)₂, r.t.; c) TBAF, wet THF, r.t.; d) PdCl₂(PPh₃)₂, CuI, THF, EtN(*i*-prop.)₂, r.t.; e) DIBAL-H, C₆H₅CH₃, r.t.; f) CBr₄, Zn, PPh₃, CH₂Cl₂, r.t.; g) aq. KOH, BTEAC, THF, r.t.

Experimental Protocols

General Remarks: All reagents were purchased from Aldrich and used without further purification. Dry toluene was obtained by distillation from a Na/benzophenone slurry. Bromoethynylpentafluorobenzene,^[1] mono TIPS-protected 1,4-diethynylbenzene **6**,^[2] bromoethynylbenzene,^[3] the molecular rod **5**^[1] and its precursor **10**^[1] were prepared according to reported procedures. Unless otherwise noted, all reactions were carried out under a N₂ protection gas atmosphere. ¹H-, ¹³C- and ¹⁹F-NMR spectra were recorded on a Bruker Ultra Shield 400 MHz. MALDI-TOF MS spectrometry was performed with a PerSeptive Biosystems Voyager –DE PRO time-off-flight mass spectrometer. Thin layer chromatography (TLC) was carried out on 20 Merck silica gel 60 F254 plates and Merck silica gel 60 (0.040-0.063 mm) was used for column chromatography (CC). Elemental analyses (EA) were recorded using a ThermoQuest FlashEA 1112 N/Protein Analyzer.

((4-(bromoethynyl)phenyl)ethynyl)triisopropylsilane 7: A suspension of **6** (0.80 g, 2.84 mmol), N-bromosuccinimide (0.60 g, 3.4 mmol) and silver nitrate (0.01 g, 0.06 mmol) in acetone (60 mL) was stirred for 2 h at RT. Removal of the solvent and CC (silica, hexane) gave **7** (1.0 g, 2.77 mmol, 97%) as slightly yellow oil. ¹H NMR (300 MHz, CDCl₃) δ 1.15 (s, 21H), 7.41 (q, 4H). ¹³C-NMR (75 MHz, CDCl₃) δ 11.30, 18.64, 51.72, 79.76, 93.14, 106.40, 122.49, 123.86, 131.76, 131.91. EI-MS 70 eV, m/z: 363.1 (25) [M]⁺, 362.1 (100) [M]⁺, 361.1 (24) [M]⁺, 360.1 (98) [M]⁺. C₁₉H₂₅BrSi (361.39): calcd. C 63.15, H 6.97; found C 62.96, H 6.87.

triisopropyl((4-(phenylbuta-1,3-diyndyl)phenyl)ethynyl)silane 8: A suspension of **7** (0.33 g, 0.91 mmol), ethynylbenzene (0.15 g, 1.47 mmol), Pd₂(dba)₃·CHCl₃ (0.04 g, 0.04 mmol), CuI (0.03 g, 0.16 mmol), N-ethyldiisopropylamine (1 ml) in toluene (20 ml) was degassed with nitrogen and stirred at room temperature for 2 hours. After removal of solvents, CC over silica gel with hexane as eluent provided **8** (0.20 g, 0.52 mmol, 57%) as slightly yellow oil. ¹H-NMR (300 MHz, CDCl₃) δ 1.13 (s, 21 H), 7.40 (m, 3H), 7.51 (br, 4H), 7.60 (m, 2H). ¹³C-NMR (75 MHz, CDCl₃) δ 11.30, 18.63, 73.82, 75.75, 81.05, 82.50, 93.69, 106.43, 121.64, 122.23, 124.25, 128.38, 129.45 132.10, 132.35, 132.58.

MALDI-ToF MS: m/z : 382.15 $[M]^+$. $C_{27}H_{30}Si$ (382.61): calcd. C 84.76, H 7.90; found C 84.91, H 7.82.

1-ethynyl-4-(phenylbuta-1,3-diynyl)benzene 9: TBAF (0.20 g, 0.61 mmol) in THF (15 mL) was added to the stirred solution of **8** (0.18 g, 0.47 mmol) in wet THF (15 mL). After 2 h stirring at RT, the solvent was removed and CC (silica, hexane) provided **9** (90 mg, 0.40 mmol, 85%) as white solid. 1H -NMR (300 MHz, $CDCl_3$) δ 3.17 (s, 1H), 7.36 (m, 3H), 7.47 (br, 4H), 7.53 (m, 2H). ^{13}C -NMR (75 MHz, $CDCl_3$) δ 73.79, 75.91, 79.63, 80.84, 82.64, 83.06, 121.71, 122.31, 122.96, 128.45, 129.32, 132.12, 132.32, 132.53. MALDI-ToF MS: m/z : 226.35 $[M]^+$. $C_{18}H_{10}$ (226.27): calcd. C 95.55, H 4.45; found C 95.13, H 4.35.

1,4-bis(4-(phenylbuta-1,3-diynyl)phenyl)buta-1,3-diyne 1: **9** (0.15 g, 0.66 mmol), $Pd(PPh_3)_2Cl_2$ (0.05 g, 0.07 mmol), CuI (0.015 g, 0.08 mmol), $EtN(i-prop)_2$ (1 mL) in THF (50 mL) was stirred for 20 h under air. CC (hexane) and SEC (toluene as eluent) yielded **1** (0.08 g, 0.18 mmol, 55%) as a white solid. 1H -NMR (300 MHz, $CDCl_3$) δ 7.37 (m, 6H), 7.47 (br, 4H), 7.54 (br, 8H). MALDI-ToF MS: m/z : 450.04 $[M]^+$. $C_{36}H_{18}$ (450.53): calcd. C 95.97, H 4.03; found C 95.79, H 4.01.

((4-((4-dodecylphenyl)buta-1,3-diynyl)phenyl)ethynyl)triisopropylsilane 11: **10** (0.135 g, 0.5 mmol), **7** (0.18 g, 0.5 mmol), $Pd_2(dba)_3 \cdot CHCl_3$ (0.02 g, 0.02 mmol) and CuI (0.015 g, 0.08 mmol), in *N*-ethyl-diisopropylamine (1.5 ml) and toluene (35 ml) was degassed with nitrogen and stirred at room temperature for 24 hours. After removal of solvents, CC (silica gel, hexane) provided **11** (0.15 g, 0.27 mmol, 54%) as a slightly yellow liquid. 1H -NMR (300 MHz, $CDCl_3$) δ 0.90 (t, 3H), 1.14 (br, 21H), 1.27 (br, 16H), 1.32 (q, 2H), 1.61 (q, 2H), 2.64 (t, 2H), 7.15 (d, 2H), 7.26 (t, 2H), 7.45 (m, 6H). ^{13}C -NMR (75 MHz, $CDCl_3$) δ 11.28, 14.13, 18.64, 22.70, 29.25, 29.37, 29.47, 29.57, 29.65, 29.68, 31.16, 31.93, 36.01, 73.19, 75.90, 80.75, 82.92, 93.69, 106.42, 118.71, 121.72, 124.18, 128.58, 131.98, 132.19, 132.45, 144.75. MALDI-ToF MS: m/z : 549.25 $[M-H]^+$. $C_{39}H_{54}Si$ (550.93): calcd. C 85.02, H 9.88; found C 85.38, H 10.04.

1-dodecyl-4-((4-ethynylphenyl)buta-1,3-diyne)benzene 12: TBAF (0.1 g, 0.3 mmol) in THF (30 mL) was added to the stirred solution of **11** (0.15 g, 0.27 mmol) in wet THF (15 mL) over 2 h at RT. The solvent was removed and CC (silica, hexane) yielded **12** (98 mg, 0.25 mmol, 93%) as a white solid. $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 0.88 (t, 3H), 1.26 (br, 16H), 1.30 (q, 2H), 1.60 (q, 2H), 2.61 (t, 2H), 3.20 (s, 1H), 7.15 (d, 2H), 7.43 (d, 2H), 7.45 (q, 4H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 14.10, 22.68, 29.24, 29.35, 29.46, 29.56, 29.63, 29.66, 31.13, 31.92, 35.99, 73.14, 76.08, 79.58, 80.46, 83.00, 83.04, 118.63, 122.38, 122.74, 128.57, 132.07, 132.27, 132.46, 144.79. MALDI-ToF MS: m/z : 393.85 $[\text{M-H}]^+$. $\text{C}_{30}\text{H}_{34}$ (394.59): calcd. C 91.32, H 8.68; found C 91.36, H 8.22.

1-(bromoethynyl)-4-((4-dodecylphenyl)buta-1,3-diyne)benzene 13: A suspension of **12** (0.20 g, 0.51 mmol), N-bromosuccinimide (0.10 g, 0.56 mmol) and silver nitrate (0.01 g, 0.06 mmol) in acetone (100 mL) was stirred for 1.5 h at RT. Removal of the solvent and CC (silica, hexane) gave **13** (0.22 g, 0.46 mmol, 90%) as slightly yellow solid. $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 0.90 (t, 3H), 1.27 (br, 16H), 1.31 (q, 2H), 1.61 (q, 2H), 2.61 (t, 2H), 7.15 (d, 2H), 7.43 (d, 2H), 7.45 (q, 4H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 14.11, 22.68, 29.23, 29.35, 29.45, 29.55, 29.63, 29.66, 31.14, 31.91, 36.01, 52.66, 73.13, 76.19, 79.61, 80.49, 83.09, 118.64, 122.25, 123.32, 128.60, 131.96, 132.32, 132.47, 144.85. EI MS: 70 eV, m/z : 475.20 (32) $[\text{M}]^+$, 474.20 (97) $[\text{M}]^+$, 473.20 (33) $[\text{M}]^+$, 472.20 (100) $[\text{M}]^+$. $\text{C}_{30}\text{H}_{33}\text{Br}$ (473.49): calcd. C 76.10, H 7.02; found C 75.96, H 6.77.

1-dodecyl-4-((4-(phenylbuta-1,3-diyne)phenyl)buta-1,3-diyne)benzene 2: 13 (0.20 g, 0.42 mmol), ethynylbenzene (0.05 g, 0.49 mmol), $\text{Pd}_2(\text{dba})_3\cdot\text{CHCl}_3$ (0.02 g, 0.02 mmol), CuI (0.015 g, 0.08 mmol), N-ethyl-diisopropylamine (1 ml) in toluene (25 ml) was degassed with nitrogen and stirred at room temperature for 24 hours. After removal of solvent, CC (silica gel, hexane) followed by SEC (biobeads, toluene) gave **2** (78 mg, 0.16 mmol, 38%) as a slightly yellow solid. $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 0.90 (t, 3H), 1.28 (br, 16H), 1.31 (q, 2H), 1.62 (q, 2H), 2.62 (t, 2H), 7.16 (d, 2H), 7.36 (br, 3H), 7.45 (d, 2H), 7.48 (s, 4H), 7.54 (m, 2H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 14.04, 22.68, 29.26,

29.34, 29.46, 29.56, 29.64, 29.66, 31.08, 31.94, 36.05, 73.27, 73.88, 76.54, 76.86, 80.58, 80.95, 83.00, 83.50, 118.82, 121.81, 122.59, 122.91, 128.49, 128.63, 129.36, 132.43, 132.45, 132.54, 132.58, 144.91. MALDI-ToF MS: m/z: 493.59 [M-H]⁺. C₃₈H₃₈ (494.71): C 92.26, H 7.74; found C 91.29, H 8.11.

1-dodecyl-4-(phenylbuta-1,3-diynyl)benzene 3: A mixture of **10** (0.22 g, 0.81 mmol), Bromoethynyl benzene (0.20 g, 1.1 mmol), Pd₂(dba)₃·CHCl₃ (0.04 g, 0.04 mmol), CuI (0.03 g, 0.16 mmol), N-ethyl-diisopropylamine (1 ml) in toluene (25 ml) was degassed with nitrogen and stirred at room temperature for 24 hours. After removal of solvent, CC (silica gel, hexane) and SEC (biobeads, toluene) yielded **3** (0.18 g, 0.50 mmol, 62%) as white solid. ¹H-NMR (300 MHz, CDCl₃) δ 0.91 (t, 3H), 1.28 (br, 16H), 1.31 (q, 2H), 1.62 (q, 2H), 2.62 (t, 2H), 7.16 (d, 2H), 7.34 (m, 1H), 7.37 (m, 2H), 7.46 (d, 2H), 7.55 (m, 2H). ¹³C-NMR (75 MHz, CDCl₃) δ 14.13, 22.70, 29.24, 29.36, 29.47, 29.57, 29.64, 29.67, 31.17, 31.91, 35.98, 73.26, 74.08, 81.15, 81.90, 118.75, 121.86, 128.37, 128.53, 129.04, 132.39, 132.41, 144.60. MALDI-ToF MS: m/z: 370.05 [M]⁺. C₂₈H₃₄ (370.57): calcd. C 90.75, H 9.25; found C 90.70, H 9.26.

2,3,5,6-tetrafluoroterephthalaldehyde 15: To a solution of **14** (5.4 g, 27.0 mmol) in dry toluene (250 mL) at 0 °C, a 1.5 M diisopropylaluminum-hydride toluene solution (50 mL) was added dropwise in 2 h. After 3 h stirring at RT, the reaction mixture was cooled to 0 °C and EtOAc (10 mL) and 2N HCl (150 mL) was added. The organic layer was separated and the aqueous one was extracted with CH₂Cl₂. Removal of the solvents followed by CC (silica, hexane/ethylacetate: 2/1) provided **15** (2.4 g, 11.7 mmol, 43%) as white solid. ¹H-NMR (300 MHz, CDCl₃) δ 10.36 (s, 2H). ¹³C-NMR (75 MHz, CDCl₃) δ 119.21 (m), 146.85 (d, m, J_d 270.1 Hz), 182.02. ¹⁹F-NMR (376.5 MHz, CDCl₃) δ -144.81. EI-MS: 70 eV, m/z: 206.0 (100) [M]⁺, 205.0 (41) [M]⁺, 177.0 (29) [M-CHO]⁺, 149.0 (40) [M-2CHO]⁺. C₈H₂F₄O₂ (206.09): calcd. C 46.62, H 0.98; found 46.47, H 0.94.

1,4-bis(2,2-dibromovinyl)-2,3,5,6-tetrafluorobenzene 16: To a suspension of CBr₄ (6.67 g, 20 mmol)

and zinc powder (1.3 g, 20 mmol) in dry CH₂Cl₂ (15 mL), a solution of PPh₃ (5.3 g, 20 mmol) in dry CH₂Cl₂ (50 mL) was added dropwise over 3 h at RT. After completed addition, the mixture was allowed to stir for 18 h. Subsequently, a solution of **15** (1.0 g, 4.8 mmol) in dry CH₂Cl₂ (20 mL) was added to the above suspension. After 2 h stirring, the suspension was filtered through a short silica plug with CH₂Cl₂. CC (silica, hexane) provided **16** (2.1 g, 4.06 mmol, 85%) as white solid. ¹H-NMR (300 MHz, CDCl₃) δ 7.24 (s, 2H). ¹³C-NMR (75 MHz, CDCl₃) δ 99.67, 115.23 (m), 123.62, 143.09 (d, m, *J*_d 251.2 Hz). ¹⁹F-NMR (376.5 MHz, CDCl₃) δ -138.70. EI-MS: 70 eV, *m/z* 519.6 (63) [M]⁺, 517.6 (100) [M]⁺, 515.6 (67) [M]⁺, 359.8 (23) [M-2Br]⁺, 357.8 (47) [M-2Br]⁺, 355.8 (24) [M-2Br]⁺ 198.0 (79) [M-4Br]⁺. C₁₀H₂F₄Br₄ (517.73): calcd. C 23.20, H 0.39; found C 23.28, H 0.39.

1,4-bis(bromoethynyl)-2,3,5,6-tetrafluorobenzene 17: A solution of KOH (7 g, 0.125 mol) in water (9 mL) was dropped to the stirred mixture of **16** (8.5 g, 16.4 mmol) and benzyltriethylammonium-chloride (0.1 g, 0.44 mmol) in THF (20 mL) at RT over 1 h. After being stirred for 2 h, the organic layer was separated and washed with saturated aqueous NaHCO₃. CC (silica, hexane) afforded **17** (5.6 g, 15.7 mmol, 96%) as a white solid. ¹³C-NMR (75 MHz, CDCl₃) δ 65.32, 65.62 (m), 104.70 (m), 147.54 (d, m, *J*_d 254.3 Hz). ¹⁹F-NMR (376.5 MHz, CDCl₃) δ -137.82. EI-MS: 70 eV (100 °C) *m/z* 357.8 (49) [M]⁺, 355.8 (100) [M]⁺, 353.8 (50) [M]⁺ 196.0 (64) [M-2Br]⁺. C₁₀Br₂F₄ (355.91): calcd. C 33.75; found C 33.61.

1-(bromoethynyl)-4-((4-dodecylphenyl)buta-1,3-dienyl)-2,3,5,6-tetrafluorobenzene 18: A suspension of **17** (3.7 g, 10.4 mmol), **10** (0.7 g, 2.6 mmol), Pd₂(dba)₃·CHCl₃ (0.10 g, 0.10 mmol) and CuI (0.06 g, 0.32 mmol) in N-ethyl-diisopropylamine (3 ml) and toluene (50 mL) was degassed with N₂ and stirred for 4 hours at RT. Removal of solvent and CC (silica gel, hexane) gave **18** (0.80 g, 1.47 mmol, 56%) as yellow solid. ¹H-NMR (300 MHz, CDCl₃) δ 0.89 (t, 3H), 1.26 (br, 16H), 1.29 (m, 2H), 1.61 (q, 2H), 2.62 (t, 2H), 7.17 (d, 2H), 7.46 (d, 2H). ¹³C-NMR (75 MHz, CDCl₃) δ 14.10, 22.69, 29.25, 29.36, 29.46, 29.56, 29.64, 29.67, 31.09, 31.92, 36.07, 65.55 (q, 4.13 Hz), 65.83 (m), 65.89, 72.43, 86.69, 87.39 (t, *J* 3.75 Hz), 104.70 (m), 117.71, 128.70, 132.70, 145.71, 147.51 (d, m, *J*_d

151.25). ^{19}F -NMR (376.5 MHz, CDCl_3) δ -136.9 (m), -137.8 (m), -138.9 (m), -139.2 (m). EI-MS: m/z : 547.1 (30) $[\text{M}]^+$, 546.1 (96) $[\text{M}]^+$, 545.1 (31) $[\text{M}]^+$, 544.1 (95) $[\text{M}]^+$, 392.0 (25) $[\text{M}-\text{C}_{11}\text{H}_{23}]^+$, 390.9 (100) $[\text{M}-\text{C}_{11}\text{H}_{23}]^+$, 389.7 (27) $[\text{M}-\text{C}_{11}\text{H}_{23}]^+$, 388.7 (89) $[\text{M}-\text{C}_{11}\text{H}_{23}]^+$. $\text{C}_{30}\text{H}_{29}\text{F}_4\text{Br}$ (545.45): calcd. C 66.06, H 5.36, found C 65.75, H 5.28.

triisopropyl((4-((perfluorophenyl)buta-1,3-diynyl)phenyl)ethynyl)silane 19: A mixture of bromoethynylpentafluorobenzene (0.29 g, 1.07 mmol), **7** (0.30 g, 1.07 mmol), $\text{Pd}_2(\text{dba})_3\cdot\text{CHCl}_3$ (0.04 g, 0.04 mmol), CuI (0.015 g, 0.08 mmol), *N*-ethyl-diisopropylamine (3 ml) and toluene (40 ml) was degassed with N_2 and stirred for 4 hours at RT. Removal of solvents under reduced pressure and CC (silica gel, hexane) gave **19** (0.37 g, 0.78 mmol, 73%) as a colourless liquid. ^1H -NMR (300 MHz, CDCl_3) δ 1.15 (s, 21H), 7.48 (q, 4H). ^{13}C -NMR (75 MHz, CDCl_3) δ 11.35, 18.61, 65.45 (q, 3.97 Hz), 74.39, 84.67, 85.29 (q, 3.6 Hz), 94.58, 99.28 (m), 106.19, 120.42, 125.29, 132.08, 132.46, 137.76 (d, m, J_d 248.2 Hz), 142.24 (d, m, J_d 257.63 Hz), 148.34 (d, m, J_d 253.5 Hz). ^{19}F -NMR (376.5 MHz, CDCl_3) δ -135.90, -151.51, -162.09. EI-MS: 70 eV (150 °C), m/z 472.1 (15) $[\text{M}]^+$, 430.0 (30) $[\text{M}-\text{C}_3\text{H}_6]^+$, 429.0 (100) $[\text{M}-\text{C}_3\text{H}_7]^+$, 386.8 (34) $[\text{M}-\text{C}_6\text{H}_{14}]^+$, 373.0 (69) $[\text{M}-\text{C}_3\text{H}_6-3\text{F}]^+$. $\text{C}_{27}\text{H}_{25}\text{F}_5\text{Si}$ (472.56): calcd. C 68.62, H 5.33; found C 68.59, H, 5.24.

1-((4-ethynylphenyl)buta-1,3-diynyl)-2,3,4,5,6-pentafluorobenzene 20: **19** (0.90 g, 1.9 mmol) and TBAF (0.7 g, 2.1 mmol) in wet THF (30 ml) was stirred at RT for 2 hours. Subsequent removal of solvent and CC (silic gel, hexane) yielded **20** (0.41 g, 1.3 mmol, 68%) as a brown solid. ^1H -NMR (300 MHz, CDCl_3) δ 3.22 (s, 1H), 7.49 (q, 4H). ^{13}C -NMR (75 MHz, CDCl_3) δ 65.59 (q), 74.50, 80.13, 82.78, 84.35, 85.15 (q), 99.26 (m), 121.10, 123.87, 132.19, 132.53, 137.80 (d, m, J_d 250.1 Hz), 142.30 (d, m, J_d 258.2 Hz), 148.43 (d, m, J_d 254.3 Hz). ^{19}F -NMR (376.5 MHz, CDCl_3) δ -135.8, -151.3, -162.0). EI-MS: 70 eV (100 °C), m/z : 317.0 (19) $[\text{M}]^+$, 316.0 (100) $[\text{M}]^+$. $\text{C}_{18}\text{H}_5\text{F}_5$ (316.22): calcd. C 68.37, H 1.59; found C 68.27, H 1.62.

1-((4-dodecylphenyl)buta-1,3-diynyl)-2,3,5,6-tetrafluoro-4-((4-((perfluorophenyl)buta-1,3diynyl)

phenyl)buta-1,3-diyne]benzene 4: 18 (0.12 g, 0.22 mmol), **20** (0.07 g, 0.22 mmol), Pd₂(dba)₃·CHCl₃ (0.04 g, 0.04 mmol) and CuI (0.015 g, 0.08 mmol) in N-ethyldiisopropylamine (1 ml) and toluene (25 ml) was degassed with N₂ and stirred at RT for 24 hours. After removal of solvent, CC (silica gel, hexane) and SEC (biobeads, toluene) provided **4** (94 mg, 0.12 mmol, 54%) as a slightly yellow solid. ¹H-NMR (300 MHz, CDCl₃) δ 0.87 (t, 3H), 1.27 (br, 16H), 1.32 (m, 2H), 1.62 (m, 2H), 2.63 (t, 2H), 7.18 (d, 2H), 7.47 (d, 2H), 7.55 (s, 4H). ¹³C-NMR (125 MHz, CDCl₃) δ 14.00, 22.63, 29.20, 29.29, 29.40, 29.50, 29.59, 29.61, 30.99, 31.88, 36.06, 65.65, 66.24, 67.60, 72.48, 75.63, 75.78, 84.04, 85.05, 85.15, 86.83, 87.20, 88.10, 99.10 (m), 104.22 (m), 105.32 (m), 117.79, 122.36, 122.42, 128.72, 132.74, 137.70 (d, m, J_d 251.4 Hz), 142.31 (d, m, J_d 256.3 Hz), 145.79, 147.70 (d, m, J_d 256.4 Hz), 148.30 (d, m, J_d 255.2 Hz). ¹⁹F-NMR (376.5 MHz, CDCl₃) -137.0 (m), -137.5 (m), -152.2 (m), -163.2 (m) MALDI-ToF MS: m/z: 779.45 [M-H]⁺. C₄₈H₃₃F₉ (780.76): calcd. C 73.84, H 4.26, found C 73.45, H 4.35.

References:

- [1] Shu, L.; Mu, Z.; Fuchs, H.; Chi, L.; Mayor, M. *Chem. Comm.* **2006**, 1862-1863.
- [2] Weil, T.; Reuther, E.; Beer, C.; Müllen, K. *Chem. Eur. J.* **2004**, *10*, 1398-1414.
- [3] Mu Z.; Shu L.; Fuchs H.; Mayor M. Chi L. *J. Am. Chem. Soc.* **2008**, *130*. 10840-10841.