Supporting Information:

Synthesis of Monodisperse Platinum-Acetylide Oligomers

End-Capped with Naphthalene Diimide Units

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Synthesis of Intermediates

cis-bis(Tri-n-butylphosphine)dichloroplatinum(II) was prepared by literature methods. Yield = 1.45 g (89.5%). M.p. 138-139°C.¹

 $3-\{4-[(Triisopropylsilyl)-ethynyl]-phenyl\}-prop-2-yn-1-ol$ (1). This compound was synthesized by literature methods. ²⁻⁵ Yield 3.1 g (32.7%). M.p. 61-63°C. ¹H NMR (300 MHz, CDCl₃) δ 1.20 (s, 21H), 3.82 (br s, 1H), 4.58 (s, 2H), 7.40 (dd, 4H).

1-Ethynyl-4-(tri-iso-propylsilylethynyl)-benzene (**2***a*) was prepared by literature methodology.⁵ Yield = 1.31 g (71.2%). M.p. 47-48°C. ¹H NMR (75 MHz, CDCl₃) δ 1.18 (s, 21H), 3.15 (s, 1H), 7.45 (s, 4H). Mass spec. (DIP-CI-HRMS) calc'd for C₁₉H₂₆Si [M+H]⁺ 283.1877, found [M+H]⁺ 283.1867.

3-(4-Ethynyl-phenyl)-prop-2-yn-1-ol (**2b**). To a 25 mL round bottom flask with a stir bar was added **1** (1.5 g, 4.80 mmol) and THF (5 mL). The solution was degassed 5 min under argon, followed by the addition of TBAF, 1 M in THF (11.9 mL, 11.9 mmol). The reaction was let stir at room temperature for 1 h. After this time, the solution was diluted with Et₂O and washed with both deionized water and brine. The organic layer was then dried over NaSO_{4(s)}. The solvent was evaporated to give a crude brown solid that was purified by flash chromatography (silica gel, 1:1 to 3:1 hexane/CH₂Cl₂, then addition of 1% MeOH to afford fraction 2 as product). The solvent of the second fraction was evaporated to afford a dark brown oil as the product. Yield 0.70 g (93%). ¹H NMR (300 MHz, CDCl₃) δ 1.25 (br, 1H), 3.15 (s, 1H), 4.45 (s, 2H), 7.38 (dd, 4H). M.p. 70-71°C.⁶

trans-Bis(tri-n-butylphosphine)chloro{(4-Ethynyl-phenylethynyl)-triisopropyl-

silane}platinum(II) (3). 2a (0.36 g, 1.28 mmol) and HNEt₂ (30 mL) were added to a round bottom flask with a stir bar. The mixture was stirred and degassed with argon for 10 min, follow

by the addition of *cis*-Pt(PBu₃)₂ Cl₂ (0.95 g, 1.41 mmol). The reaction was stirred at reflux under argon overnight, then cooled and diluted with CH₂Cl₂. The organic solution was washed with both deionized water and brine then dried over NaSO_{4(s)}. TLC in 5:1 hexane/CH₂Cl₂ gave 4 spots (spot 3 was product). Solvent was evaporated to give a crude solid that was purified by flash chromatography (silica gel, 9:1 to 3:1 hexane/CH₂Cl₂). Fraction 3 solvent was evaporated to afford a yellow crystalline solid that did not phosphoresce under a long wave UV lamp. Yield 0.97 g (82.6%). M.p. 65-67°C. ¹H NMR (300 MHz, CDCl₃) δ 0.89 (t, 18H), 1.18 (s, 21H), 1.3-1.6 (br m, 24H), 1.98 (br m, 12H), 7.14 (d, 2H), 7.31 (d, 2H). ³¹P NMR (CDCl₃) δ 8.0 (J = 2356 Hz). Mass Spec. (+TOF-HRMS) Calc'd for C₄₃H₇₉ClP₂PtSiNa [M+Na]⁺ 939.4658; Found [M+Na]⁺ 939.4685. Anal. Calcd. for C₄₃H₇₉ClP₂PtSi : C, 56.34; H, 8.69; N, 0.0. Found C, 56.62; H, 9.05; N, 0.01.

trans-Bis(tributylphosphine)bis{(4-Ethynyl-phenylethynyl)-triisopropyl-

silane]platinum(II) (*4*). To a 50 mL round bottom flask with a stir bar was added 2a (0.30 g, 1.06 mmol), *cis*-Pt(PBu₃)₂Cl₂ (0.33 g, 0.48 mmol) and HNEt₂ (10 mL). The solution was degassed with argon for 10 min, followed by the addition of CuI (~3 mg). Within minutes, the reaction was a bright yellow color with salt formation. After 1 h, the solvent was evaporated and the solid diluted with CH₂Cl₂. The organic solution was washed with deionized water and brine, then dried over NaSO_{4(s)}. Evaporation of solvent afforded a crude yellow oil that was purified by flash chromatography (silica gel, 100% hexane to 4:1 hexane/CH₂Cl₂). Fractions 1 and 2 were impurities; fraction 3 was eluted as a large yellow band. Solvent was removed to yield a yellow crystalline solid as product. Yield 0.32 g (57%). M.p. 65-68°C. ¹H NMR (300 MHz, CDCl₃) δ 0.88 (t, 18H), 1.08 (s, 39H), 1.19 (sextet, 12H), 1.55 (br m, 12H), 2.05 (br m, 12H), 7.13 (d, 4H), 7.29 (d, 4H); ³¹P NMR (CDCl₃) δ 4.2 (J_{PLP} = 2346.94 Hz).²

trans-Bis(tri-n-butylphosphine)-bis[2-{4-ethynylphenyl}ethyne]-platinum(II) (5). To a 25 mL round bottom flask with a stir bar was added **4** (300 mg, 0.26 mmol) and THF (5 mL). The solution was degassed 5 min with argon, followed by the addition of TBAF, 1 M in THF (0.77 mL, 0.77 mmol). The reaction was stirred at room temperature for 1 h. TLC (silica, 10:1 hexane/acetone) revealed complete transformation of the starting material. The solution was diluted with Et₂O and washed with both deionized water and brine. The organic solution was dried over MgSO_{4(s)}, and the solvent was removed to give a crude yellow solid that was purified by a short flash chromatography column (silica gel, 10:1 hexane/acetone). The eluted yellow fraction solvent was evaporated to afford a yellow crystalline solid (starburst-like crystals). Yield 218 mg (99%). M.p. 85-88°C. ¹H NMR (300 MHz, CDCl₃) δ 0.91 (t, 18H), 1.45 (m, 12H), 1.55 (m, 12H), 2.10 (br m, 12H), 3.09 (s, 2H), 7.19 (d, 4H), 7.32 (d, 4H).²

trans-Bis(tributylphosphine)-[2-{4-(prop-1-yn-3-ol)phenyl}ethyne]-[2-{4-(2-

triisopropylsilyl-1-phenylethynyl]ethyne]-platinum(II) (6a). To a round bottom flask with a stir bar was added **3** (0.55 g, 0.60 mmol) and **2b** (94 mg, 0.60 mmol). HNEt₂ (15 mL) was added and the solution was degassed for 10 min under argon. CuI (2 mg) was then added under argon flow, and the reaction was let stir at room temperature overnight. The solution was then diluted with CH₂Cl₂ and washed with deionized water and brine. The bright yellow organic solution was dried over MgSO_{4(s)}, then solvent was evaporated to give a dark yellow oil. The crude product was purified by flash chromatography (silica gel, CH₂Cl₂). Fraction 2, a bright yellow band on the column, was evaporated to afford a yellow solid. Yield 0.37 g (60%). ¹H NMR (300 MHz, CDCl₃) δ 1.0 (br t, 19 H *hexanes), 1.2 (s, 21H), 1.4-1.8 (br m, 24H), 2.05 (br, 1H), 2.21 (br m, 12H), 4.60 (br s, 2H), 7.28 (dd, 4H), 7.41 (dd, 4H); ³¹P NMR (CDCl₃) δ 4.1(J_{PLP} = 2346.33 Hz). *Residual solvent proved difficult to remove; the compound was used as is for further synthesis.

Complex (6b). To a 100 mL round bottom flask with a stir bar was added **6a** (0.40 g, 0.39 mmol) and Et₂O (20 mL). The solution was degassed with argon, followed by the addition of powdered KOH (0.22 g, 8.86 mmol) and activated MnO₂ (0.67 g, 7.72 mmol). The reaction was let stir at room temperature for 12 h, and then it was quenched with deionized water. The solution was diluted with Et₂O and washed with 5% HCl_(aq), deionized water, and brine. The organic layer was dried over NaSO_{4(s)}, and the solvent was removed to give a crude orange oil that was purified by flash chromatography (silica gel, 9:1 CH₂Cl₂/hexane). Fraction 1 eluted as a yellow band off of the column, and this was evaporated to give the product as a yellow oil. Yield = 0.38 g (96%). ¹H NMR (300 MHz, CDCl₃) δ 0.91 (t, 18H), 1.13 (s, 20H*), 1.35-1.70 (br m, 24H), 2.10 (br m, 12H), 3.1 (s, 1H), 7.19 (dd, 4H), 7.35 (dd, 4H); ³¹P NMR (CDCl₃) δ 4.1(J_{Pt}. p = 2346.34 Hz). *The integration of the TIPS hydrogens was slightly less than expected, but the deprotected compound was used as is for further synthesis.

Complex (7). To a 100 mL round bottom flask with a stir bar was added **6b** (250 mg, 0.25 mmol) and HNEt₂ (30 mL). The mixture was stirred and degassed at room temperature under argon for 10 min, followed by the addition of *cis*-PtCl₂(PBu₃)₂ (200 mg, 0.30 mmol). The reaction was heated to reflux and let stir under argon overnight. After 15 h, the reaction was cooled and the solvent evaporated to give a crude yellow oil. The sample was diluted with CH_2Cl_2 and washed with deionized water and brine. The organic layer was dried over $NaSO_{4(s)}$, and then the solvent was removed to afford a yellow oil. The crude oil was purified by flash chromatography (silica gel, 4:1 then 2:1 hexane/CH₂Cl₂). Fraction 2 moved as a large yellow band through the column. Solvent was evaporated then put under vacuum 2 h to give a yellow

solid product, although residual hexane was difficult to completely remove. Yield = 380 mg (93%). M.p. 73-74°C. ¹H NMR (300 MHz, CDCl₃) δ 0.8-1.0 (m, 45H *residual hexanes), 1.12 (s, 21H), 1.26 (m, *hexanes), 1.37-1.70 (br m, 50H), 1.92-2.22 (br m, 24H), 7.07 (dd, 4H), 7.18 (d, 2H), 7.30 (d, 2H); ³¹P NMR (CDCl₃) δ 4.2 (J_{Pt-P} = 2346.34 Hz). Mass Spec. (+TOF-HRMS) Calc'd for C₁₅₄H₂₇₄Cl₂P₈Pt₄ Si₂Na₂ [2M+2Na]⁺² 1663.3327; Found [2M+2Na]⁺² 1663.8327. Anal. Calcd. for C₇₇H₁₃₇ClP₄Pt₂Si : C, 56.37; H, 8.42; N, 0.0. Found C, 56.65; H, 8.75; N, 0.04.

N-(4'-Ethynylphenyl)-N'-(octyl)naphthalene-1,8:4,5-tetracarboxydiimide (8). This compound was prepared via routes previously decribed in the literature. All intermediate data given below.

N-(*n*-octyl)naphthalene-1,8-dicarboxyanhydride-4,5-dicarboximide⁷: Yield = 2.87 g (20.4%). 1H NMR (300 MHz, CDCl₃) δ 0.88 (t, 3H), 1.30 (br m, 12H), 4.20 (t, 2H), 8.80 (s, 4H). M.p. 173-175°C. Mass spec. (DIP-CI-HRMS) calc'd for C₂₂H₂₁NO₅, [M+H]⁺ 380.1492; found, [M+H]⁺ 380.1510.

 $N-(4'-iodophenyl)-N'-(octyl)naphthalene-1,8:4,5-tetracarboxydiimide^8$: Yield 1.31 g (85.6%). ¹H NMR (300 MHz, CDCl₃) δ 0.90 (t, 3H), 1.30 (br m, 10H), 1.70 (m, 2H), 4.20 (t, 2H), 7.2 (d, 2H), 7.6 (d, 2H), 8.80 (s, 4H). M.p. 252-253°C. Mass spec. (DIP-CI-HRMS) calc'd for C₂₈H₂₅N₂O₄I [M+H]⁺ 581.0932, found [M+H]⁺ 581.0884. Anal. Calcd. for C₂₈H₂₅N₂O₄I: C, 57.94; H, 4.34; N, 4.83. Found C, 58.23; H, 4.27; N, 4.77.

N-(4'-(2-Triis opropyl silyl-ethynyl phenyl)-N'-(octyl) naphthalene-1, 8:4, 5-tetra carboxy diimide.

Yield = 1.97g (95.6%). M.p. 253-255°C. ¹H NMR (300 MHz, CDCl₃) δ 0.88 (t, 3H), 1.15 (s, 21H), 1.30 (br m, 10H), 1.70 (m, 2H), 4.20 (t, 2H), 7.28 (d, 2H), 7.68 (d, 2H), 8.80 (s, 4H). Mass Spec. (+TOF-HRMS) Calc'd for C₃₉H₄₆N ₂O₄SiNa [M+Na]⁺ 657.3119; Found [M+Na]⁺

657.3119. Anal. Calcd. for C₃₉H₄₆N₂O₄Si: C, 73.78; H, 7.30; N, 4.41. Found C, 73.51; H, 7.51; N, 4.28.

N-(4'-*Ethynylphenyl*)-*N*'-(*octyl*)*naphthalene-1,8:4,5-tetracarboxydiimide* (8): To a round bottom flask with a stir bar was added *N*-(4'-(2-TIPS-ethynylphenyl)-*N*'-(octyl)naphthalene-1,8:4,5-tetracarboxydiimide (1.97 g, 3.10 mmol) and THF (45 mL). The solution was stirred and degassed under argon for 15 min, followed by addition of TBAF, 1M in THF (6.83 mL, 6.83 mmol) to the reaction. This was stirred at room temperature for 4 h, followed by dilution with CH₂Cl₂. The organic layer was washed with deionized water and brine, then it was dried over NaSO_{4(s)}. The solvent was evaporated to give a dark yellow solid. Flash chromatography (silica gel, 10:1 CHCl₃/Hexane, 4:1 CHCl₃/THF) was used to purify the solid. The product was recovered as a yellow solid in the second eluted fraction. Yield = 1.07 g (72.3%). M.p. 257-259°C. ¹H NMR (300 MHz, CDCl₃) δ 0.88 (t, 3 H), 1.30 (br m, 10H), 1.75 (m, 2H), 3.10 (s, 1H), 4.20 (t, 2H), 7.28 (d, 2H), 7.68 (d, 2 H), 8.80 (s, 4H). Mass spec. (DIP-CI-HRMS) for C₃₀H₂₆N₂O₄ [M+H]⁺ 479.1965, found [M+H]⁺ 479.1972.⁸

Complex (9). To a 200 mL round bottom flask equipped with a stir bar was added **8** (0.82, 1.71 mmol), *i*Pr₂NH (30 mL) and MeCN (60 mL). The mixture was stirred and degassed with argon for 10 min, followed by addition of *cis*-Pt(PBu₃)₂ Cl₂ (1.49 g, 2.23 mmol). The reactions was refluxed overnight, then cooled to room temperature and diluted with CH₂Cl₂. The organic solution was washed with deionized water and brine, then it was dried over NaSO_{4(s)}. The solvent was removed to give a dark red solid. The crude product was purified by flash chromatography (silica gel, CH₂Cl₂). The first fraction eluted was a yellow band of Pt (II) starting material; the second fraction was the pure product **9** as a bright orange band. All other color remained at the top of the column. The solvent was evaporated from fraction 2 to yield an

orange powder. This product was recrystalized in hexane to afford a very small, shiny needlelike powder. Yield 0.91g (48%). M.p. 145-146°C. ¹H NMR (300 MHz, CDCl₃) δ 0.8-1.0 (br m, 21H), 1.2-1.9 (br m, 36H), 2.0 (br m, 12H), 4.20 (t, 2H), 7.15 (d, 2H), 7.40 (d, 2H), 8.80 (s, 4H); ³¹P NMR (CDCl₃) δ 8.1 (J_{Pt-P} = 2365.26). Mass Spec. (+TOF-MS) Calc'd for C₅₄H₇₉ClN₂O₄P₂PtNa [M+Na]⁺ 1135.4751; Found [M+Na]⁺ 1135.4768. Anal. Calcd. for C₅₄H₇₉ClN₂O₄P₂Pt: C, 58.29; H, 7.16; N, 2.52. Found C, 58.43; H, 7.36; N, 2.42.

Complex (10). This compound was prepared by literature procedure.⁵ Yield = 0.58 g (90%). M.p. 103-104°C. ¹H NMR (75 MHz, CDCl₃) δ 0.91 (t, 36H), 1.45 (m, 24H), 1.55 (m, 24H), 2.0 (br m, 24H), 7.05 (s, 4H); ¹³C NMR (300 MHz, CDCl₃) δ 14.0, 22.1, 24.5, 26.3, 101.6, 215.3, 130.5; ³¹P NMR (121 MHz, CDCl₃) δ +7.89 (J_{Pt-P} = 2390.8 Hz). Mass Spec. (+TOF-HRMS) Calc'd for C₅₈H₁₁₂Cl₂P₄Pt₂Na [M+Na]⁺ 1416.6276; Found [M+Na]⁺ 1416.6275.

1,4-Diethynylbenzene (13) was prepared by previous literature methods.^{2,3} Yield = 0.92 g (88.5%). M.p. 91-93°C (90-94°C). ¹H NMR (300 MHz, CDCl₃) δ 3.18 (s, 2H), 7.42 (s, 4H).

Complex (14a). To a 50 mL round bottom flask with stir bar was added 1,4diethynylbenzene 13 (15 mg, 0.12 mmol), 7 (425 mg, 0.26 mmol), THF (5 mL) and HNEt₂ (15 mL). The solution was degassed for 10 min under argon, followed by the addition of CuI (3 mg). The reaction was stirred under argon for 3 h at room temperature, followed by quenching it with deionized water. The solution was diluted with CH₂Cl₂, washed with deionized water and brine, and dried over NaSO_{4(s)}. The solvent was evaporated to afford a crude yellow solid that was purified by flash chromatography (silica gel, 100:1 hexane/ acetone). Fraction 1 traveled as a yellow band down the column, and solvent was evaporated to give a yellow solid as product. Yield = 0.38 g (95%). M.p. 161-162°C. ¹H NMR (300 MHz, CDCl₃) δ 0.91 (t, 72H), 1.13 (s, 42H), 1.38-1.48 (br m, 99H *water in solvent), 2.15 (br m, 49H), 7.10 (s, 12H), 7.18 (d, 4H), 7.32 (d, 4H); ³¹P NMR (CDCl₃) δ 4.0 (J_{Pt-P} = 2365.87 Hz), 4.2 (J_{Pt-P} = 2355.49 Hz). Mass Spec. (MALDI-TOF-HRMS with HABA as matrix) Calc'd for C₁₆₅₄H₂₇₈P₈Pt₄ Si₂ [M]⁺ 3333.7813; Found [M]⁺ 3333.7568.

Complex (14b). To a 25 mL round bottom flask with a stir bar was added 14a (230 mg, 0.07 mmol) and THF (5 mL). The mixture was degassed for 10 min, followed by the addition of TBAF, 1 M in THF (0.69 mL, 0.69 mmol) via syringe. The reaction was let stir at room temperature with the flask covered in aluminum foil for 1 h, and then it was quenched with deionized water and diluted with CH₂Cl₂. The organic layer was washed with deionized water and brine, and it was dried over NaSO_{4(s)}. Once the organic solvent was evaporated, the crude yellow solid was purified by flash chromatography (silica gel, 20:1 hexane/acetone). Fraction 1 was collected as a yellow solution. The solvent was evaporated to afford a light yellow solid as product. Yield = 210 mg (96%). ¹H NMR (CDCl₃) δ 0.91 (t, 71H), 1.05 (s, 5H *TIPS-F salt contaminate in NMR sample), 1.38-1.48 (br m, 99H *water in solvent), 2.15 (br, 48H), 3.09 (s, 2H), 7.10 (s, 12H), 7.18 (d, 4H), 7.32 (d, 4H) ; ³¹P NMR (CDCl₃) δ 3.95 (J_{Pt-P} = 2352.43 Hz), 4.2 (J_{Pt-P} = 2356 Hz).

Complex (*16a*). To a 50 mL round bottom flask with a stir bar was added **14b** (218 mg, 0.07 mmol), **7** (249 mg, 0.15 mmol), *i*Pr₂NH (8 mL) and THF (3 mL). The solution was degassed with argon for 10 min, followed by addition of CuI (~1 mg). The reaction was stirred under argon at room temperature overnight. After 15 h, the solvent was evaporated and the crude solid diluted in CH₂Cl₂. The organic solution was washed with deionized water and brine, and then dried with NaSO_{4(s)}. The solvent was evaporated to give a crude yellow solid that was purified by flash chromatography (silica gel, 1:1 to 2:1 CH₂Cl/hexane) as fraction 2 (fraction 1 was a very small band on the column; fraction 2 was a very large yellow band). Solvent was

evaporated from this fraction to afford a light yellow solid as product. Yield 395 mg (90%). ¹H NMR (300 MHz, CDCl₃) δ 0.90 (t, 145H), 1.12 (s, 40H), 1.3-1.7 (br m, 201H), 2.15 (br m, 97H), 7.05-7.20 (m, 30H), 7.32 (d, 4H); ³¹P NMR (CDCl₃) δ 4.0 (J_{Pt-P} = 2349.38 Hz), 4.2 (J_{Pt-P} = 2369.53 Hz).

Complex (*16b*). To a 25 mL round bottom flask with a stir bar was added *16a* (120 mg, 19 µmol) and THF (5 mL). The solution was degassed with argon for 5 min, followed by the addition of TBAF, 1 M in THF (56.4 µL, 56.5 µmol) via syringe. The reaction was stirred at room temperature under argon with the flask covered in aluminum foil (to prevent light exposure) for 1 h. After this time, the reaction was diluted with CH₂Cl₂ and washed with deionized water and brine. The organic layer was dried over NaSO_{4(s)} and solvent then evaporated to afford a crude yellow solid. The product was purified by flash chromatography (silica gel, 3:1 CH₂Cl₂/hexane). Fraction 1, a yellow band on the column, was collected and the solvent evaporated to give a yellow solid product, however some residual hexane proved difficult to remove. Yield 100 mg (96%). ¹H NMR (300 MHz, CDCl₃) δ 0.90 (br m, 180H *residual hexanes), 1.26 (s, 42H *TIPS-F salt contaminate), 1.3-1.7 (br m, 203H *residual solvents), 2.15 (br m, 96H), 3.10 (s, 2H), 7.05-7.20 (m, 31H), 7.32 (d, 4H); ³¹P NMR (300 MHz, CDCl₃) δ 4.0 (J_{Pt-P} = 2366.87 Hz), 4.2 (J_{Pt-P} = 2348.24 Hz). *The crude deprotected product was used as is for further synthesis without further analysis.

Cyclic Voltammetry

Cyclic voltammetry experiments were performed in dry methylene chloride (CH_2Cl_2) solution containing 0.1 M tetra-*n*-butylammonimum hexafluorophosphate (TBAHFP) as the supporting electrolyte. The three-electrode setup consisted of a platinum microdisk (2 mm²) working electrode, a platinum wire auxiliary electrode and a silver wire reference electrode.

Solutions were degassed with argon flow prior to measurements, maintaining positive argon pressure during the measurements. The concentrations of oligomers in the solutions were 1 mM. A scan rate of 100 mV/s was used. All potentials were internally calibrated against the ferrocene/ferricinium couple (E = 0.43 V vs. SCE in CH₂Cl₂). Below is the voltammagram of Pt₂NDI₂ with ferrocene; the potentials plotted are versus the Ag wire reference electrode.

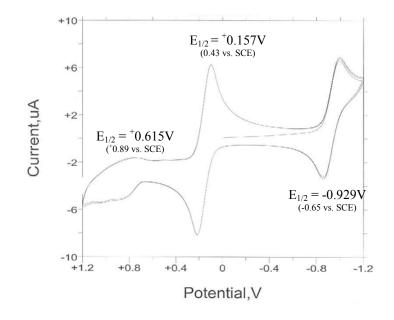


Figure S-1. Cyclic voltammagram of the Pt₂NDI₂/methylene chloride/0.1 M TBAHFP sample solution with ferrocene added as an internal standard. A silver wire was used as the reference electrode.

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