Supporting Information for

Photoswitching of Conductance of Diarylethene-Gold Nanoparticle Network Based on the Alteration of π -Conjugation

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Experimental Section

1. Preparation of Materials

General. All reactions were monitored by thin-layer chromatography carried out on 0.2 mm Merck silica gel plates (60F-254). Column chromatography was performed on silica gel (Nakalai, 70-230 mesh). Some compounds were purified by a JAI recycling preparative GPC LC-908 with JAIGEL-1H and 2H column. HPLC was carried out on a HITACHI LC System LaChrom with L-2130 controller and L-2420 UV detector. An analytical (Kanto Chemical, Mightysil Si60 250-4.6 (5 μm)) column was used to purify trimethylsilylethyl-protected precursors **18** and **20** and non-substituted diarylethene **50**. ¹H and ¹³C NMR spectra were recorded on a JNM-Alpha500 or JNM-ECA600 instrument. Mass spectra of EI and MALDI ionization were obtained by a JEOL JMS-SX102A and Thermo Scientific LTQ orbitrapXL mass spectrometers, respectively.

3-Bromo-2,4-dimethyl-5-trimethylsilylthiophene (9),^{S1} 2-iodo-5-phenylthiophene (11),^{S2} and 2,4-dibromo-3-methylthiophene (12),^{S3} are known compounds. The dibromide **19** was prepared according to the literature.^{S4} Synthesis was performed according to Scheme S1.



Scheme S1. Synthetic procedure for diarylethene 30, 40, and 50. (Same as Scheme 1 in the main text)

3-bromo-S-(2-trimethylsilylethyl)benzenethiol (7)

To a solution of 3-bromobenzenethiol (6) (5.0 g, 26 mmol) in dry DMF (30 mL) was added NaH (630 mg, 26 mmol) at room temperature under N₂. The mixture was stirred at room temperature for 30 min. After addition of (2-bromoethyl)trimethylsilane (4.2 mL, 26 mmol), the reaction mixture was stirred at room temperature for 3 h. The reaction mixture was quenched with water and extracted with Et_2O . The organic layer was washed with brine, dried over Na₂SO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (hexane) to give colorless oil 7 (4.3 g, 15 mmol, 56%).

¹H NMR (500 MHz, CDCl₃, δ): 0.05 (s, 9H), 0.91-0.95 (m, 2H), 2.94-2.97 (m, 2H), 7.12 (t, J = 8.0 Hz, 1H), 7.19 (ddd, J = 8.0, 1.9, 1.3 Hz, 1H), 7.27 (ddd, J = 8.0, 1.8, 1.3 Hz, 1H), 7.41 (t, J = 1.8 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃, δ): -1.8, 16.6, 29.2, 122.7, 126.8, 128.4, 130.0, 130.7, 139.9; EI HRMS m/z [M]⁺: calcd for C₁₁H₁₇BrSSi⁺, 287.9998; found, 288.0004.

1-(2,4-dimethyl-5-trimethylsilyl-3-thienyl)heptafluorocyclopentene (10)

To a solution of 3-bromo-2,4-dimethyl-5-trimethylsilylthiophene (9),^{S1} (3.0 g, 11 mmol) in dry THF (100 mL) was slowly added dropwise *n*-BuLi (1.6 M in hexanes, 7.1 mL, 11 mmol) at -78 °C under N₂ for 2 h. The resulting mixture was cooled to -95 °C. Then perfluorocyclopentene (4.59 mL, 34.2 mmol) was added by a cooled syringe in one portion and stirred for 30 min under N₂ with keeping below -50 °C. After stirring at -78 °C for 3 h, the reaction mixture was quenched with water, extracted with Et₂O. The organic layer was washed with brine, dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (hexane) to give a colorless oil **10** (3.5 g, 9.2 mmol, 81%).

¹H NMR (500 MHz, CDCl₃, δ): 0.33 (s, 9H), 2.14 (s, 3H), 2.36 (s, 3H); ¹³C NMR (125 MHz, CDCl₃, δ): -0.22, 14.0, 15.5, 108.1-117.8 (m), 120.5-121.1 (m), 122.3, 132.3, 143.2, 145.8, 153.1-156.0 (m); EI HRMS *m*/*z* [M]⁺: calcd for C₁₄H₁₅F₇SSi⁺, 376.0547; found, 376.0557.

3, 5-dibromo-4-methyl-5'-phenyl-2, 2'-bithiophene (13)

To a mixture of 2-iodo-5-phenylthiophene (11) (730 mg, 2.5 mmol),^{S2} 2,4-dibromo-3-methylthiophene 12 (780 mg, 3.1 mmol),^{S3} KF (350 mg, 6.1 mmol), $PdCl_2(PPh_3)_2$ (110 S3

mg, 0.15 mmol) in 15 mL of DMSO under N₂, AgNO₃ (520 mg, 3.1 mmol) was added in one portion. The resulting suspension was stirred at 100 °C for 6 h. The reaction mixture was allowed to cool to room temperature and extracted with CHCl₃. The organic layer was dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (hexane) to give yellow solid **13** (990 mg, 2.4 mmol, 94%).

¹H NMR (500 MHz, CDCl₃, δ): 2.25 (s, 3H), 7.26 (d, *J* = 4.0 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 4.0 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.62 (d, *J* = 7.5 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃, δ): 16.4, 107.4, 109.8, 123.2, 125.8, 127.6, 127.9, 129.0, 132.2, 133.3, 133.8, 138.0, 145.1; MALDI HRMS *m*/*z* [M]⁺: calcd for C₁₅H₁₀Br₂S₂⁺, 411.8586; found, 411.8603.

3'-bromo-4'-methyl-2-trimethylsilyl-2"-phenyl-5, 5':2', 5"-terthiophene (15)

To a solution of 2-trimethylsilylthiophene (14) (380 mg, 2.4 mmol) in dry THF (5 mL) was slowly added dropwise *n*-BuLi (1.6 M in hexanes, 1.6 mL, 2.6 mmol) at 0 °C under N₂. The mixture was stirred at room temperature for 1.5 h. After addition of B(OBu)₃ (0.68 mL, 2.5 mmol) at 0 °C, the reaction mixture was stirred at room temperature. The reaction mixture was quenched with water. The obtained dibutyl (5-trimethylsilyl-3-thienyl)boronate was used in next step without further purification.

To the reaction mixture, aqueous Na₂CO₃ (20 wt%, 5 mL), bromide **13** (9.3 g, 40 mmol), and Pd(PPh₃)₄ (1.2 g, 1.0 mmol) were added and the mixture was refluxed for 6 h. The reaction mixture was extracted with CH₂Cl₂, washed with aqueous NH₄Cl and water. The organic layer was dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (hexane) to give yellow solid **15** (59 mg, 1.2 mmol, 50%).

¹H NMR (500 MHz, CDCl₃, δ): 0.33 (s, 9H), 2.37 (s, 3H), 7.16 (m, 2H), 7.19 (d, *J* = 3.9 Hz, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 3.9 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃, δ): -0.13, 16.0, 112.2, 123.1, 125.6, 127.26, 127.31, 127.6, 128.8, 129.3, 130.3, 133.8, 133.9, 134.2, 134.4, 140.6, 141.3, 144.4. MALDI HRMS *m*/*z* [M]⁺: calcd for C₂₂H₂₁BrS₃Si⁺, 487.9753; found, 487.9778.

1-(2,4-dimethyl-5-trimethylsilyl-3-thienyl)-2-(3'-methyl-2-trimethylsilyl-2"-phenyl-5,2':5',5"-terth iophen-4'-vl)hexafluorocyclopentene (16)

To a solution of bromide **15** (630 mg, 1.3 mmol) in dry THF (10 mL) was slowly added dropwise *n*-BuLi (1.6 M in hexanes, 0.85 mL, 1.4 mmol) at -78 °C under N₂ for 1 h. After addition of heptafluorocyclopentene **10** (490 mg, 1.3 mmol) at -78 °C dropwise, the reaction mixture was stirred at -78 °C for 1 h and was allowed to warm up to room temperature. The reaction was quenched with water and extracted with CHCl₃. The organic layer was washed with brine, dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (gradient eluting hexane/CH₂Cl₂ = 100/0 - 98/2). Further purification was carried out using recycle GPC to obtain TMS-protected diarylethene **16** as a green oil (360 mg, 0.48 mmol, 37%).

¹H NMR (600 MHz, CDCl₃, δ): 0.16 (s, 5H), 0.24 (s,4H), 0.36 (s, 9H), 1.48 (d, 3.0 Hz, 1.7H), 1.71 (d, 3.6 Hz, 1.3H), 1.89 (s, 1.3H), 2.13 (s, 1.7H), 2.39 (d, 3.0 Hz, 1.3H), 2.42 (s, 1.7H), 6.91 (d, 4.2 Hz, 0.6H), 6.92 (d, 3.6Hz, 0.4H), 7.21-7.23 (m, 3H), 7.32 (t, 7.5 Hz, 1H), 7.40 (t, 7.5 Hz, 2H), 7.57 (d, 7.5 Hz, 2H); ¹³C NMR (150 MHz, CDCl₃, δ): -0.43 (3C), -0.22 (3C), -0.12 (6C), 13.3, 14.5, 14.7, 15.9 (2C), 16.0, 123.8, 123.9, 125.69, 125.73, 125.74 (2C), 125.78 (2C), 126.1, 126.8, 127.5, 127.6, 127.95, 127.97, 128.2, 128.6, 129.00 (2C), 129.06 (2C), 130.5, 131.2, 132.7, 132.9, 133.0, 133.2, 133.56, 133.67, 133.72, 133.9, 134.46, 134.48, 135.2, 135.7, 140.12, 140.13, 141.6, 141.7, 143.1, 144.0, 144.6, 146.2 (2C), 146.4. MALDI HRMS m/z [M]⁺: calcd for C₃₆H₃₇F₆S₄Si₂⁺, 767.1210; found, 767.1204.

1-(5-(3-((2-trimethylsilylethyl)thio)phenyl)-2,4-dimethyl-3-thienyl)-2-(2-(3-((2-trimethylsilylethyl) thio)phenyl)-3'-methyl-2"-phenyl-5,2':5',5"-terthiophen-4'-yl)hexafluorocyclopentene (18)

To a solution of trimethylsilylethyl-protected **16** (273 mg, 0.356 mmol) in acetic acid (5 mL) was added *N*-bromosuccinimide (140 mg, 0.78 mmol) by three portion at intervals of 30 min. The mixture was stirred at room temperature overnight. The reaction product was poured into aqueous NaOH (9 N) for neutralization. The mixture was extracted with CHCl₃, dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (hexane) to give dibromide **17** as a green oil (110 mg, 0.14 mmol, 38%). The compound **17** was used in the next reaction without further purification.

To a solution of 7 (3.0 g, 10 mmol) in dry THF (30 mL) was slowly added dropwise *n*-BuLi (1.6 M in hexanes, 6.5 mL, 10 mmol) at -78 °C under N₂. The mixture was stirred at -78 °C for 1 h. After addition of B(OMe)₃ (1.2 mL, 10 mmol) at -78 °C, the reaction mixture was allowed to warm up to room temperature. After 1h, the reaction mixture was quenched with water and extracted with 0.1 N NaOH. The aqueous layer was acidified by 0.1 N HCl and extracted with Et₂O. The organic layer was washed with brine, dried over Na₂SO₄, filtered, and evaporated. The boronic acid **8** was obtained as white solid. The compound **8** was used in the next reaction without further purification.

To a solution of dibromide **17** (110 mg, 0.14 mmol) and boronic acid **8** (137 mg, 0.54 mmol) in THF (5 mL) and aqueous Na₂CO₃ (20wt%, 5 mL) was added Pd(PPh₃)₄ (31 mg, 0.027 mmol) and refluxed overnight. The reaction mixture was extracted with CHCl₃ and organic layer was washed with brine, dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (gradient eluting from hexane/CH₂Cl₂= 95/5 to 85/15). Further purification was carried out using GPC and HPLC to give trimethylsilylethyl-protected **18** as a green oil (83 mg, 0.080 mmol, 59%).

¹H NMR (600 MHz, CDCl₃, δ): 0.010 (s, 4.5H), 0.016 (s, 4.5H), 0.064 (s, 4.5H), 0.068 (s, 4.5H), 0.89-0.93 (m, 2H), 0.96-0.99 (m, 2H), 1.44 (d, J = 3.6 Hz, 1.5H), 1.73 (d, J = 2.4 Hz, 1.5H), 1.90 (s, 1.5H), 2.15 (s, 1.5H), 2.45 (d, J = 3.6 Hz, 1.5H), 2.46 (d, J = 3.6 Hz, 1.5H), 2.91-2.96 (m, 2H), 3.00-3.04 (m, 2H), 6.95 (d, J = 3.6 Hz, 0.5H), 6.98 (d, J = 3.6 Hz, 0.5H), 7.02 (d, J = 7.8 Hz, 0.5H), 7.08 (d, J = 7.8 Hz, 0.5H), 7.08 (d, J = 7.8 Hz, 0.5H), 7.14-7.29 (m, 6H), 7.30-7.43 (m, 7H), 7.55 (m, 1H), 7.58 (d, J = 7.2 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃, δ): -1.78 (3C), -1.77 (3C), -1.74 (6C), 13.2, 13.3, 14.4 (2C), 16.0, 16.1, 16.77, 16.82, 16.88 (2C), 29.40, 29.43 (3C), 123.06, 123.08, 123.87, 123.89, 124.0 (2C), 124.9, 125.5, 125.69, 125.77 (2C), 125.79 (2C), 125.81 (2C), 125.9, 126.3, 126.4, 127.29, 127.31, 127.4, 127.5, 127.93, 127.94 (2C), 128.1, 128.5, 128.7 (2C), 128.8, 129.0 (2C), 129.1 (3C), 129.2, 129.39, 129.40, 132.1, 132.7, 132.8, 132.97, 132.98, 133.5, 133.6, 133.7 (2C), 133.9, 134.34, 134.35, 134.54, 134.57, 134.62, 134.7 (2C), 135.3, 135.4, 135.8, 137.6, 137.7, 138.27, 138.28, 138.6, 140.9, 144.3 (2C), 146.5, 146.8. MALDI HRMS m/z [M+H]⁺: calcd for C₅₂H₅₃F₆S₆Si₂⁺, 1039.1909; found, 1039.1903.

1-(5-(3-mercaptophenyl)-2,4-dimethyl-3-thienyl)-2-(2-(3-mercaptophenyl)-3'-methyl-2"-phenyl-5, 2':5',5"-terthiophen-4'-yl)hexafluorocyclopentene (30)

To a solution of trimethylsilylethyl-protected **18** (10 mg, 9.62 μ mol) in dry THF (1 mL) was slowly added TBAF (1 M in THF, 0.290 mL, 290 μ mol) at room temperature under N₂. The mixture was stirred at 35 °C for 15 min. The reaction mixture was quenched with HCl (1 N, 1 mL) and extracted with ether (1 mL). The solution of dithiol **30** in toluene was used in the fabrication of the network without further purification.

EI LRMS (m/z) [M]⁺: 838.

1-(2,4-dimethyl-5-phenyl-3-thienyl)-2-(2,2"-bis(3-((2-trimethylsilylethyl)thio)phenyl)-4'-methyl-5, 2':5',5"-terthiophen-3'-yl)hexafluorocyclopentene (20)

To a solution of 7 (410 mg, 1.4 mmol) in dry THF (15 mL) was slowly added dropwise *n*-BuLi (1.6 M in hexanes, 1.3 mL, 2.1 mmol) at -78 °C under N₂. The mixture was stirred at -78 °C for 1 h. After addition of B(OMe)₃ (1.0 mL, 8.3 mmol) at -78 °C, the reaction mixture was allowed to warm up to room temperature. After 1h, the reaction mixture was quenched with water and extracted with 0.1 N NaOH. The aqueous layer was acidified by 0.1 N HCl and extracted with Et₂O. The organic layer was washed with brine, dried over Na₂SO₄, filtered, and evaporated. The boronic acid **8** was obtained as white solid. The compound **8** was used in the next reaction without further purification.

To a solution of dibromide 19^{84} (90 mg, 0.12 mmol) and boronic acid **8** (249 mg, 0.98 mmol) in THF (2.3 mL) and aqueous Na₂CO₃ (20wt%, 2.3 mL) was added Pd(PPh₃)₄ (20 mg, 0.017 mmol) and refluxed 6 h. The reaction mixture was extracted with Et₂O and organic layer was washed with brine, dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (hexane/CHCl₃ = 4/1). Further purification was carried out using GPC and HPLC to give trimethylsilylethyl-protected **20** as a green oil (87 mg, 0.084 mmol, 59%).

¹H NMR (600 MHz, CDCl₃, δ): 0.05 (s, 4.5H), 0.065 (9H), 0.068 (4.5H), 0.94-0.99 (m, 4H), 1.45 (d, *J* = 3.0 Hz, 1.5H), 1.73 (d, *J* = 3.0 Hz, 1.5H), 1.91 (s, 1.5H), 2.15 (s, 1.5H), 2.45 (d, *J* = 3.0 Hz, 1.5H), 2.43 (d, *J* = 3.6 Hz, 1.5H), 2.97-3.04 (m, 4H), 6.96 (d, *J* = 3.6 Hz, 0.5H), 6.98 (d, *J* = 4.2 Hz, 0.5H), 7.16 (t, *J* = 4.2 Hz, 2H), 7.22-7.25 (m, 5.5H), 7.27-7.36 (m, 5.5H), 7.41-7.42 (m, 1.5H), 7.49 (s, 0.5H), 7.55 (s, S7

1H); ¹³C NMR (150 MHz, CDCl₃, δ): -1.78 (3C), -1.76 (3C), -1.74 (6C), 13.17, 13.26, 14.33, 14.35, 16.06, 16.12, 16.8 (2C), 16.9 (2C), 29.34 (2C), 29.43 (2C), 123.06 (2C), 123.08, 123.11, 123.87, 123.89, 124.3 (2C), 124.8, 125.4, 125.55, 125.56, 125.77 (2C), 125.84, 126.0, 127.25, 127.29, 127.30, 127.31, 127.9, 128.0 (3C), 128.4 (2C), 128.5 (3C), 128.6, 129.0 (2C), 129.1 (2C), 129.37, 129.40 (2C), 129.5, 131.8, 132.9, 133.0, 133.1, 133.26, 133.28, 133.8, 133.98 (2C), 134.04, 134.15, 134.23, 134.3 (2C), 134.5, 134.6, 135.20, 135.24, 135.6, 135.9, 138.28 (2C), 138.35, 138.43, 138.6, 140.6, 144.33, 144.35, 145.8, 146.0. MALDI HRMS m/z [M+H]⁺: calcd for C₅₂H₅₃F₆S₆Si₂⁺, 1039.1909; found, 1039.1903.

1-(2,4-dimethyl-5-phenyl-3-thienyl)-2-(2,2"-bis(3-mercaptophenyl)-4'-methyl-5,2':5',5"-terthioph en-3'-yl)hexafluorocyclopentene (40)

To a solution of trimethylsilylethyl-protected **20** (10 mg, 9.62 μ mol) in dry THF (1 mL) was slowly added TBAF (1 M in THF, 0.290 mL, 290 μ mol) at room temperature under N₂. The mixture was stirred at 35 °C for 15 min. The reaction mixture was quenched with HCl (1 N, 1 mL) and extracted with ether (1 mL). The solution of dithiol **40** in toluene was used in the fabrication of the network without further purification.

EI LRMS (*m*/*z*) [M]⁺: 838.

1-(2,4-dimethyl-5-phenyl-3-thienyl)-2-(2,2"-diphenyl-4'-methyl-5,2':5',5"-terthiophen-3'-yl)hexaf luorocyclopentene (50)

To a solution of dibromide 19^{S4} (28 mg, 36 µmol) and phenylboronic acid (160 mg, 0.63 mmol) in THF (6 mL) and aqueous Na₂CO₃ (20 wt%, 6 mL) was added Pd(PPh₃)₄ (41 mg, 36 µmol) and refluxed 3 h. The reaction mixture was extracted with CHCl₃ and organic layer was washed with brine, dried over MgSO₄. The crude product was purified by silica gel column chromatography (CH₂Cl₂). Further purification was carried out using GPC and HPLC to give non-substituted **50** as a green oil (18 mg, 0.023 mmol, 63%).

¹H NMR (600 MHz, CDCl₃, δ): 1.44 (d, 3.6 Hz, 1.5H), 1.73 (d, 3.0 Hz, 1.5H), 1.91 (s, 1.5H), 2.14 (s, 1.5H), 2.45 (d, 3.6 Hz, 1.5H), 2.46 (d, 4.2 Hz, 1.5H), 6.96 (d, 3.6 Hz, 0.5H), 6.98 (d, 4.2 Hz, 0.5H), 7.15 (d, 3.6 Hz, 0.5H), 7.16 (d, 1.8 Hz, 0.5H), 7.17 (d, 1.2 Hz, 0.5H), 7.23-7.42 (m, 13.5H), 7.58 (d, 7.2 S8

Hz, 1H), 7.63 (d, 7.2 Hz, 2H); ¹³C NMR (150 MHz, CDCl₃, δ): 13.19, 13.25, 14.4 (2C), 16.08, 16.14, 123.58, 123.59, 124.02, 124.05, 124.8, 125.5, 125.73 (4C), 125.75, 125.80 (4C), 126.0, 127.2, 127.30 (2C), 127.33, 127.89 (2C), 127.91, 128.1, 128.4 (2C), 128.45, 128.48 (2C), 128.6, 129.00 (2C), 129.03 (4C), 129.06 (2C), 129.10 (2C), 129.13 (2C), 131.8, 132.85, 132.92, 133.05, 133.09, 133.4, 133.6, 133.7 (2C), 133.8 (2C), 133.9, 134.08, 134.10, 134.3, 134.4, 135.2, 135.3, 135.7, 135.8, 138.4, 140.6, 144.97, 145.00, 146.5, 146.7. MALDI HRMS m/z [M]⁺: calcd for C₄₂H₂₈F₆S₄⁺, 774.0972; found, 774.0977.

The photochemical properties of the open-ring isomer (**50**): UV-vis (hexane:CH₂Cl₂ = 90:10) λ_{max} 368 nm (ϵ = 2.8 × 10⁴ M⁻¹ cm⁻¹), λ_{edge} = 431 nm (2.88 eV), CV (CH₂Cl₂, vs Ag/Ag⁺ in MeCN) +0.92 V; The closed-ring isomer (**5c**): UV-vis (hexane:CH₂Cl₂ = 90:10) λ_{max} 607 nm (ϵ = 1.5 × 10⁴ M⁻¹ cm⁻¹), λ_{edge} = 714 nm (1.74 eV), CV (MeCN, vs Ag/Ag⁺ in MeCN): +0.58 V.

2. Spectroscopy and Voltammetry

The photochromic reaction of trimethylsilylethyl-protected **18** and **20** were measured in CH_2Cl_2 . The non-substituted diarylethene **50** was measured in hexane: $CH_2Cl_2 = 90:10$. Photoirradiation was carried out using a USHIO 500 W super high-pressure mercury lamp by passing the light through a combination of some ATG band-pass filters and monochromator (Ritsu MC-20L) to induce the photoisomerization of the molecules in the solution. UV-vis spectra were measured with a JASCO V-670 spectrometer.

Quantum yield measurements were performed as follows. Photoirradiation was carried out by using a 300 W xenon lamp system (Asahi spectra, MAX-303). Wavelength of the light was selected by passing through band-pass filters (Asahi spectra, LX0313, 313 nm, FWHM 10 nm and Asahi spectra, MX0580, 580 nm, FWHM 10 nm). Irradiation photon number was measured by using a photoreaction quantum yield measurement system (Shimadzu, QYM-01) equipped with a corrected power meter. Absorption spectral changes upon photoirradiation were monitored at the peak top of closed-ring isomer (607 nm).

Cyclic voltammogram was measured by BAS ALS-600A electrochemical analyzer. The electronic properties of both isomers were estimated by cyclic voltammograms and electronic spectra.

 E_{HOMO} , which represents the HOMO level against the vacuum level, was estimated by the equation (S1),^{S5}

$$E_{\rm HOMO} = -5.10 - E_{\rm ox} \tag{S1}$$

where E_{ox} represents the oxidization potential versus ferrocene/ferrocenium. $E_{\text{ox},50}$ and $E_{\text{ox},5c}$ were obtained from the voltammograms as 0.92 and 0.58 V vs Ag/Ag⁺, respectively, and E_{redox} of the ferrocene/ferrocenium versus Ag/Ag⁺ in acetonitrile was measured as 0.088 V. Thus, $E_{\text{HOMO},50}$ and $E_{\text{HOMO},5c}$ were yielded as -5.93 and -5.59 eV, respectively.

The HOMO-LUMO gap can be obtained from the edge of the absorption band. $\Delta E_{\text{HOMO-LUMO,50}}$ and $\Delta E_{\text{HOMO-LUMO,5c}}$ were calculated as 2.88 and 1.74 eV, respectively. Thus, $E_{\text{LUMO,50}}$ and $E_{\text{LUMO,5c}}$ were yielded as -3.05 and -3.85 eV as illustrated in Figure 5.

3. Computational Methods

The ground state structures of both isomers were optimized by DFT with B3LYP/6-31G(d). For the analysis of molecular orbital energies, we adopted Kohn-Sham orbital energies as approximate molecular orbital energies. All the calculations were performed by Gaussian 09.^{S6}

The orbital energies of the HOMO and LUMO are found to be around -5 and -2 eV, respectively (Table S1). Figure S1 shows the orbital energies and DOS (density of states) spectra of both isomers **50** and **5c**.

compounds	30	<u>3c</u>	40	4c	50	5c	-
LUMO / eV	-1.90	-2.70	-1.94	-2.69	-1.84	-2.63	
HOMO / eV	-5.32	-4.94	-5.35	-4.94	-5.27	-4.88	

Table S1. HOMO and LUMO energy of compounds 3-5.



Figure S1. DOS spectra and orbital energies near HOMO and LUMO for 50 and 5c.

4. Preparation of Diarylethene-capped Au Nanoparticle Network

A. Electrode.

The gold electrode (gap span: 5 μ m) was purchased from NTT Advanced Technology. Thin film of 1.2 nm-thick SiO₂ on Si wafers (525 μ m) acts as electrical insulator. Gold electrode deposited onto SiO₂ was 78 nm-thickness and the width of gold was 20 μ m.

B. TOAB-capped Au nanoparticles.

To a solution of tetrachloroaurate(III) hydrate in ultrapure water (30 mM, 1.4 mL, 42 μ mol) was added tetraoctylammonium bromide (TOAB, 110 mg, 200 μ mol) in toluene (4 mL). The solution was vigorously stirred to generate suspension. Then freshly-prepared NaBH₄ (15 mg, 400 μ mol) in ultrapure water (1 mL) was added and the solution was stirred for 3 h. The organic phase was extracted with toluene and filtered with membrane filter (pore size: 200 nm) to give TOAB-capped gold nanoparticles.

C. Diarylethene-capped Au nanoparticle Network on the Electrode.

Deprotection of trimethylsilylethyl-protected **18** and **20** (10 mg) was carried out using the above procedure, Synthesis Section. The obtained solution of dithiols **30** and **40** in THF (1 mL) were extracted with ether (1 mL) and used in the fabrication of the network without further purification.

The electrode was soaked into the solution of dithiol for 30 min and rinsed by toluene several times to remove the excess thiol.

One drop (1 μ L) of dithiol in toluene and one drop (1 μ L) of TOAB-capped Au nanoparticle in toluene were dropped and mixed on the electrode. The electrode was dried on standing and washed with

toluene. This procedure was repeated 3–5 times. The fabricated electrode was moved to the vacuum chamber in the prober and used for conductance measurements.

5. Conductance Measurement

Conductance measurement was performed by a source meter (Agilent Technologies, B1500A) equipped with a preamplifier (Agilent Technologies, E5288A). The fabricated gold electrode was placed into the vacuum chamber in wafer prober system (Nagese Techno-Engineering, Grail10-205-4-LV-OP). Tungsten needles were used to probe the electrodes. An optical fiber was used to control the spotting position (ca. 300 µm in diameter). The cyclization reaction was performed using UV light (Ocean Optics, LLS-310, 310 nm, FWHM: 12 nm). The cycloreversion reaction was performed using visible light (Ocean Optics, LLS-530, 530 nm, FWHM : 35 nm).

6. SEM Observation

SEM observation of the electrode was performed on a JEOL SS-30 instrument. The measurement was performed at 20 kV and the WD was set as 10 mm. The observation was carried out after the conductance measurement and metal coating was not carried out.

7. TEM Observation

TEM observation of the network was performed on a JEOL JEM-1400. The measurement was performed at 120 kV. The sample was prepared by putting a drop of the mixture of diarylethene and Au-nanoparticle solution on Cu grid.

Additional Data



Figure S2. Absorption spectral change of trimethylsilylethyl-protected precursors of (a) **18** and (b) **20** in CH₂Cl₂ upon irradiation with 313 nm light: dashed line, open-ring isomer; solid line, in the photostationary state; dotted lines, spectra during the photoirradiation. λ_{max} : **180**, 371, 264 nm; **18c**, 617 nm; **20o**, 366, 260 nm; **20c**, 607 nm.



Figure S3. Histogram of the size of the nanoparticle in **4-Au** nanoparticle network. The diameter of each particle was obtained by measuring the diameter in the TEM image manually.

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S 7 TMS

Br

¹H NMR of comp. 7 (500 MHz, CDCl₃)





¹³C NMR of comp.7 (125 MHz, CDCl₃)





¹H NMR of comp. **10** (500 MHz, CDCl₃)





¹³C NMR of comp.**10** (125 MHz, CDCl₃)





¹H NMR of comp. **13** (500 MHz, CDCl₃)





¹³C NMR of comp. **13** (125 MHz, CDCl₃)





¹H NMR of comp. **15** (500 MHz, CDCl₃)





¹³C NMR of comp. **15** (125 MHz, CDCl₃)





¹H NMR of comp. **16** (600 MHz, CDCl₃)



¹³C NMR of comp. 16 (150 MHz, CDCl₃)



¹H NMR of comp. **18** (600 MHz, CDCl₃)



¹³C NMR of comp. **18** (150 MHz, CDCl₃)



¹H NMR of comp. **20** (600 MHz, CDCl₃)





¹H NMR of comp. **5** (600 MHz, CDCl₃)



¹³C NMR of comp. 5 (150 MHz, CDCl₃)

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C C C C S C C C C C H F F	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435
C C C C S C C C C C H F F F	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288
C C C C S C C C C C H F F F F	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.0252814	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809
C C C C S C C C C C H F F F F F	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989 2.76665720	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.02952814	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809
C C C C S C C C C C C H F F F F F F	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989 -3.76605670	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.02952814 1.63158517	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809 2.11845353
C C C C S C C C C C C H F F F F F F F	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989 -3.76605670 -3.35793023	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.02952814 1.63158517 0.20374110	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809 2.11845353 3.73713146
C C C C S C C C C C C H F F F F F F H	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989 -3.76605670 -3.35793023 -6.51326884	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.02952814 1.63158517 0.20374110 -3.43749352	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809 2.11845353 3.73713146 -4.08511214
C C C C S C C C C C C H F F F F F F H H	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989 -3.76605670 -3.35793023 -6.51326884 -5.71805630	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.02952814 1.63158517 0.20374110 -3.43749352 -3.77720628	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809 2.11845353 3.73713146 -4.08511214 0.80074382
С С С С S С С С С С С Н F F F F F F H H H	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989 -3.76605670 -3.35793023 -6.51326884 -5.71805630 -7.66203105	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.02952814 1.63158517 0.20374110 -3.43749352 -3.77720628 -4.92043530	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809 2.11845353 3.73713146 -4.08511214 0.80074382 -0.21352187
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CCCCSCCCCCHFFFFFFHHHHH	-0.65402225 -1.02395460 -1.31019108 -1.14675782 -0.61978386 -1.36014007 -1.72123817 -1.92355786 -1.78046473 -1.42445573 -1.20965269 -1.30081300 -1.96783789 0.61401557 -0.03841113 -1.72848989 -3.76605670 -3.35793023 -6.51326884 -5.71805630 -7.66203105 -8.07008926 -4.57899152 6.12202727	0.30883349 0.23440029 1.49385610 2.55538999 1.98578970 3.98608368 4.87071334 6.22458773 6.72414259 5.85617786 4.50459771 6.23500458 2.17856067 2.11842390 0.49832978 3.02952814 1.63158517 0.20374110 -3.43749352 -3.77720628 -4.92043530 -4.75684333 -2.27976519	-2.49581319 -3.08462100 -2.22267469 -0.65892469 -2.47690565 -1.44417211 -1.70407600 -2.99981546 -4.03456686 -3.77630950 -5.04579703 4.59814089 2.92166435 4.25926288 2.58573809 2.11845353 3.73713146 -4.08511214 0.80074382 -0.21352187 -2.66200100 -3.06674738
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