Supporting Information

Noncovalently Netted, Photoconductive Sheets with Extremely High Carrier Mobility and Conduction Anisotropy from Triphenylene-Fused Metal Trigon Conjugates

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Section 1. Materials and Methods

2,3,6,7,10,11-hexabromo-triphenylene **2**,^[S1] and 4-(dodecyloxy)-2- hydroxy-benzaldehyde ^[S2] were prepared according to reported methods.

Crystals were obtained with vapor diffusion method from DMSO-diethyl ether mixed solvent and the data crystal was a red prism of approximate dimensions $0.20 \times 0.20 \times 0.20$ mm $(C_{84}H_{102}N_6O_{17}Zn_3S_{11})$. Data were collected on a Rigaku Mercury CCD diffractometer in the scan range $\theta \le 27.5^\circ$. Of the 38907 reflections collected, 7360 were unique ($R_{int} = 0.078$). The

structures were solved by direct methods and refined by full-matrix least square procedures. Solution and structure refinement calculations for the structure were performed using the Crystal Structure 3.8 software of Molecular Structure Corporation.

¹H and ¹³C NMR spectra were recorded on a JEOL model JNM-LA400 NMR spectrometer, where chemical shifts (δ in ppm) were determined with a residual proton of the solvent as standard. Matrix-assisted laser desorption ionization time-of-flight mass (MALDI-TOF MS) spectroscopy was performed on an Applied Biosystems BioSpectrometry model Voyager-DE-STR spectrometer in reflector or linear mode using 9-nitroanthracene or dithranol as matrix. Electronic absorption spectra were recorded using a quartz cell of 1-cm path length on a JASCO model V-570 spectrophotometer equipped with a temperature controller. Fluorescence spectra were recorded using a quartz cell of 1-cm path length on a JASCO model FP-6600 spectrofluorometer equipped with a temperature controller. The samples (dispersed in PEG (Mw = 400)) for fluorescence spectroscopy and fluorescence depolarization measurement are prepared with an absorbance of less than 0.1 at the excitation wavelength. Fluorescence quantum yields were measured by using quinine sulfate $(1N H_2SO_4)$ as an external standard. Infrared (IR) spectra were recorded on a JASCO model FT IR-6100 Fourier transform infrared spectrometer. Field emission scanning electron microscopy (FE SEM) was performed on a JEOL model JSM-6700 FE-SEM operating at an accelerating voltage of 5.0 kV. Transmission electron microscope (TEM) images were obtained on a JEOL model JEM-3200 microscope. Powder x-ray diffraction (PXRD) data were recorded on a Rigaku model RINT Ultima III diffractometer by depositing the xerogel on glass substrate. Semi-empirical calculations at the AM1 level were performed on Spartan'04 (Wavefunction Inc.). Electrochemistry measurements were performed with CV on an ALS Model 610B electrochemical analyzer with a three electrodes configuration using a platinum wire as the counter electrode, a platinum disc as the working electrode (2 mm diameter), and a non aqueous Ag/Ag⁺ electrode (Ag wire in 10 mM AgNO₃ in anhydrous acetonitrile solution) as the reference electrode.

Electrical measurements were carried out at ambient temperature in air between a 10– μ m width Pt electrodes by a two-probe method using a Keithley model 2635 sourcemeter. Typically, 3 μ L of **TSZn**₃gel in decalin (10 mg/mL) was homogenously drop casted onto the electrode to give a film and dried under vacuum. For iodine doping, the same **TSZn**₃ gel electrode was put into an iodine atmosphere for 2h before measurement. For the sandwich structure device fabrication, 3 μ L of **TSZn**₃gel in decalin (10 mg/mL) was homogenously spin coated onto an Al substrate to give a film with thickness around 10 μ m and then over coated with 20-nm thick Au. Light irradiation was performed using an Asahi Spectra model MAX-301

xenon light source. I–V curves were recorded at bias voltages from -2.0 to 2.0 V, on irradiation from the Au electrode with visible light (400 – 700 nm). Film thickness was measured by FE SEM.

Laser flash photolysis time-resolved microwave conductivity (FP TRMC) measurements were carried out at 25 °C in air, where the resonant frequency and microwave power were properly adjusted at 9.1 GHz and 3 mW, respectively. Charge carrier were photochemically generated using the nanosecond laser pulses from a Nd:YAG laser (a third harmonic generation ($\lambda = 355$ nm) of Spectra-Physics INDY-HG (FWHM 5 – 8 ns)). The power density of the laser was set at 3.4 – 14.7 mJcm⁻²pulse⁻¹. Transient conductivities ($\Phi\Sigma\mu$) were evaluated according to an equation $\Phi\Sigma\mu = (1/A)(\Delta P_r/P_r)$, where A and P_r are sensitivity factor and reflected microwave power, respectively. Time-of-flight transient current was measured at 25 °C by photoirradiation from with the 355-nm pulse laser. The laser intensity was attenuated at 9.6 mJcm⁻² pulse⁻¹, while bias voltage was 2 – 16 V. For the fabrication, **TSZn₃** was dissolved in toluene (0.2 wt%) and drop-casted onto an Au-interdigitated electrode with 5 μ m gap to form a thin film with thickness of about 10 μ m.

Section 2. Synthesis

(1) Synthesis of Triphenylene-Cored Hexamine



3: A toluene solution (50 mL) of tris(dibenzylideneacetone)dipalladium(0) (0.448 g, 0.49 mmol) and rac-BINAP (0.607 g, 0.98 mmol) was degassed by four freeze-pump-thaw cycles, purged with Ar, and stirred at 110 °C for 30 min. After cooling at r.t., the mixture was added with benzophenone imine (2.65 mL, 15.77 mmol), 2,3,6,7,10,11-hexabromo-triphenylene 2 (1.419 g, 2.02 mmol), and sodium tert-butoxide (1.516 g, 15.77 mmol), and the mixture was stirred at 110 °C overnight. The mixture was cooled at r.t., diluted with CH₂Cl₂, filtered through a pad of Celite, and evaporated to dryness. The residue was subjected to column chromatography on silica gel with AcOEt/hexane (1:4) as eluent. The last fraction was collected and evaporated to dryness, to give 3 (2.001 g, 1.53 mmol) as yellow solid in 76% yield. ¹H NMR (CDCl₃): δ (ppm) 7.62-7.64 (m, 12H, *o*-Ph CH), 7.34-7.64 (m, 6H, *o*-Ph CH), 7.27-7.30 (m, 12H, m-Ph CH), 7.19-7.22 (m, 6H, o-Ph CH), 7.12-7.15 (m, 12H, m-Ph CH), 7.04 (s, 6H, triphenylene CH), 6.93-6.94 (m, 12H, p-Ph CH). ¹³C NMR (CD₂Cl₂): δ (ppm) 168.2, 142.0, 140.2, 137.2, 130.8, 129.6, 129.1, 128.4, 128.2, 125.8, 114.6. MALDI-TOF MS for C₉₆H₆₆N₆ (calcd. 1302.53): m/z = 1303.76 ([M + H]⁺, 100%). UV/Vis (THF): λ_{max} (nm) 287, 387. IR (KBr pellet): v (cm⁻¹) 3056, 3024, 1621, 1597, 1445, 1288, 956, 694. Anal. Calcd. for C₉₆H₆₆N₆: C, 88.45; H, 5.10; N, 6.45. Found: C, 87.97; H, 5.40; N, 6.23%.

4: To a THF solution (10 mL) of **3** (0.219 g, 0.17 mmol) was added a 2.0 M aqueous HCl solution (0.5 mL, 1.0 mmol) and the mixture was stirred at r.t. for 0.5 h. The precipitate was isolated by centrifugation, washed with hexane (5.0 mL × 3), and dried under vacuum, to give **4** (0.076 g, 0.14 mmol) as light yellow solid in 84% yield. ¹H NMR (DMSO-*d*₆): δ (ppm) 7.91 (s, 6H, triphenylene C*H*), 3.94 (brs, 12H, N*H*₂). ¹³C NMR (DMSO-*d*₆): δ (ppm) 132.66, 129.55, 125.55. MALDI-TOF MS for C₁₈H₁₈N₆ (calcd. 318.16): m/z = 318.23 ([M + H]⁺, 100%). UV/Vis (MeOH): λ_{max} (nm) 286, 331. IR (KBr pellet): v (cm⁻¹) 3388, 3332, 3231, 2926, 2610, 1635, 1517, 1438, 1281, 872. Anal. Calcd. for C₁₈H₂₄N₆Cl₆: C, 40.25; H, 4.50; N, 15.65. Found: C, 40.66; H, 4.48; N, 15.78%.

(2) Synthesis of TSH₆ and Metal Trigon TSZn₃



TSH₆: To a suspension of **4** (0.067 g, 0.12 mmol) in EtOH (3.0 mL) was added Et₃N (0.109 g, 1.08 mmol). The mixture was stirred at r.t. for 10 min, added with an EtOH (3.0 mL) solution of 4-dodecyloxy-2-hydroxybenzaldehyde (0.345 g, 1.12 mmol), and the mixture was stirred at reflux temperature for 12 h. The resulting suspension was cooled at –10 °C and stirred for 1 h. The precipitate was collected by filtration, washed with cold EtOH, and dried under vacuum, to give **TSH**₆ (0.234 g, 0.11 mmol) as red solid in 91% yield. ¹H NMR (THF-*d*₈): δ (ppm) 10.73 (brs, 6H, O*H*), 8.73 (brs, 6H, N=C*H*), 8.41 (brs, 6H, triphenylene C*H*), 7.25 (d, *J* = 8.4 Hz, 6H, Ar-*H*), 6.36 (s, 6H, Ar-*H*), 6.33 (d, *J* = 8.4 Hz, 6H, Ar-*H*), 3.89 (brs, 12H, ArOC*H*₂), 1.21-1.39 (m, 108H, C*H*₂), 0.79 (t, *J* = 6.8 Hz, 18H, C*H*₃). MALDI-TOF MS for C₁₃₂H₁₈₆N₆O₁₂ (cacld. 2048.92): *m*/*z* = 2049.08 ([M]⁺, 100%). UV/Vis (CH₂Cl₂): λ_{max} (nm) (ε / M⁻¹cm⁻¹) 297 (5.21 × 10⁴), 379 (1.12 × 10⁵). IR (KBr): ν (cm⁻¹) 2923 (ν_{CH2}), 2852 (ν_{CH2}), 1614 (ν_{HC=N}), 1514, 1296, 1186, 1115. Anal. Calcd. for C₁₃₂H₁₈₆N₆O₁₂: C, 77.38; H, 9.15; N, 4.10. Found: C, 77.11; H, 9.32; N, 4.16%.

TSZn₃: To a suspension of **TSH**₆ (0.053 g, 0.026 mmol) in MeOH (20 mL) was added a MeOH solution (2 mL) of Zn(II) acetate dihydrate (0.034 g, 0.156 mmol), and the mixture was stirred at r.t. for 3 days. The precipitate was collected by filtration, washed with MeOH (10 × 3 mL), and dried under vacuum, to give **TSZn₃** (0.049 g, 0.022 mmol) as red orange solid in 86% yield. ¹H NMR (CD₂Cl₂/pyridine- d_5 , 95:5 in vol): δ (ppm) 8.66 (brs, 6H, N=CH), 8.28 (brs, 6H, arene-H), 7.19 (brs, 6H, Ar-H), 6.24-6.37 (m, 12H, Ar-H), 3.95 (brs, 12H, ArOCH₂), 1.32-1.79 (m, 108H, CH₂), 0.91 (brs, 18H, CH₃). MALDI-TOF MS for C₁₃₂H₁₈₀Zn₃N₆O₁₂ (cacld. 2239.04): m/z = 2238.46 ([M]⁺, 100%). UV/Vis (CH₂Cl₂): λ_{max} (nm) (ε / M^{-1} cm⁻¹) 334 (6.78 × 10⁴), 419 (9.95 × 10⁴), 488 (4.95 × 10⁴). IR (KBr): v (cm⁻¹) 2924 (v_{CH2}), 2852 (v_{CH2}), 1611 ($v_{HC=N}$), 1585, 1521, 1435. Anal. Calcd. for C₁₃₂H₁₈₀Zn₃N₆O₁₂•6H₂O: C, 67.55; H, 8.25; N, 3.58. Found: C, 67.19; H, 8.00; N, 3.66%.

(3) Synthesis of Non-Alkyl-Tethered Zn(II) Trigon



Nonalkoxylated TSH₆: To a suspension of **4** (52.3 mg, 0.097 mmol) in EtOH (3.0 mL) was added Et₃N (89 mg, 0.88 mmol) at r.t. under Ar. The mixture was stirred for 10 min, added with an EtOH (2.0 mL) solution of 2-hydroxybenzaldehyde (0.107 g, 0.88 mmol), and the mixture was stirred at reflux temperature for 12 h. The suspension was cooled at -10 °C and stirred for another 1 h. The precipitate was collected by filtration, washed with cold EtOH, and dried under vacuum, to give a **Nonalkoxylated TSH₆** (0.041g, 0.044 mmol) as orange solid in 45% yield. ¹H NMR (CD₂Cl₂) δ (ppm) 12.91 (brs, 6H, OH), 8.10 (s, 6H, CH=N) 7.39 (s, 6H, triphenylene CH), 7.13 (t, J = 7.2 Hz, 6H, aromatic CH), 7.01 (d, J = 7.2 Hz, 6H, aromatic CH), 6.72 (d, J = 8.0 Hz, 6H, aromatic CH), 6.64 (d, J = 6.8 Hz, 6H, aromatic CH). MALDI-TOF MS for C₆₀H₄₂N₆O₆ (Cacld. 942.32), m/z = 942.87 ([M]⁺, 100%). Anal. Calcd. for C₆₀H₄₂N₆O₆: C, 76.42; H, 4.49; N, 8.91. Found: C, 77.01; H, 4.30; N, 8.69%.

Nonalkoxylated TSZn₃: To a suspension of **Nonalkoxylated TSH**₆ (12.8 mg, 0.014mmol) in MeOH (6.0 mL) was added a MeOH solution (1.0 mL) of Zn(II) acetate dihydrate (0.018 g, 0.08 mmol), and the mixture was stirred at r.t. for 3 days. A red–orange precipitate was collected by filtration, washed with MeOH (5.0×3 mL), and dried under vacuum, to give a **Nonalkoxylated TSZn**₃ (0.0148 g, 0.013 mmol) as red-orange solid in 93% yield. ¹H NMR (DMSO-*d*₆) δ (ppm) 9.50 (s, 6H, *CH*=N), 9.25 (S, 6H, triphenylene *CH*), 7.64 (d, *J* = 7.2 Hz, 6H, aromatic *CH*), 6.66 (t, *J* = 7.2 Hz, 6H, aromatic *CH*). MALDI-TOF MS for C₆₀H₃₆N₆O₆Zn₃ (Cacld. 1132.05), *m*/*z* = 1131.25 ([M]⁺, 100%). Anal. Calcd. for C₆₀H₃₆Zn₃N₆O₆•6H₂O: C, 58.06; H, 3.90; N, 6.77. Found: C, 58.19; H, 3.92; N, 6.44%.

(4) Synthesis of Control Compounds SH₂ and SZn



SH₂: To a hot EtOH (1.0 mL) solution of *o*-phenylenediamine (110.8 mg, 1.03 mmol) was added dropwise an EtOH (5.0 mL) solution of 4-dodecyloxy-2-hydroxylbenzaldehyde (691 mg, 2.25 mmol), and the mixture was stirred at reflux for 12 h. The mixture was cooled at –10 °C, and the resulted yellow precipitate was filtered, washed with cold EtOH, and dried under vacuum, to give **SH**₂ (0.525 g, 0.77 mmol) as bright yellow solid in 75% yield. ¹H NMR (CDCl₃) δ (ppm) 13.58 (s, 2H, OH), 8.53 (s, 4H, CH=N), 7.30-7.25 (m, 4H, aromatic CH), 7.23-7.20 (m, 2H, core aromatic CH), 6.52(d, *J* = 2.0 Hz, 2H, aromatic CH), 6.46 (dd, *J* = 8.8, 2.0 Hz, 2H, aromatic CH), 3.98 (t, *J* = 6.4 Hz, 4H, OCH₂), 1.78 (m, 4H, OCH₂CH₂), 1.27-1.44 (m, 36H, alkyl CH), 0.88 (t, *J* = 6.8 Hz, 6H, CH₃). MALDI-TOF MS for C₄₄H₆₄N₂O₄ (Cacld. 684.49), *m*/*z* = 685.32 ([M + H]⁺, 100%). UV/Vis (CH₂Cl₂): λ_{max} (nm) (ε/M⁻¹cm⁻¹) 295 (2.47 × 10⁴), 331 (3.06 × 10⁴). IR (KBr): *v* (cm⁻¹) 2922 (*v*_{CH2}), 2852 (*v*_{CH2}), 1610 (*v*_{HC=N}), 1583, 1516, 1472. Anal. Calcd. for C₄₄H₆₄N₂O₄: C, 77.15; H, 9.42; N, 4.09. Found: C, 76.88; H, 9.43; N, 3.96%.

SZn: To the suspension of **SH**₂ (69.8 mg, 0.101mmol) in MeOH (10.0 mL) was added a MeOH solution (1.0 mL) of Zn(II) acetate dihydrate (0.044 g, 0.202 mmol), and the mixture was stirred at r.t. for 3 days. A bright yellow precipitate was collected by filtration, washed with MeOH (5.0 × 3 mL), and dried under vacuum, to give **SZn** (0.065 g, 0.087 mmol) as bright yellow solid in 86% yield. ¹H NMR (CD₂Cl₂) δ (ppm) 8.32 (s, 4H, CH=N), 7.32-7.29 (m, 2H, aromatic CH), 7.25-7.22 (m, 2H, aromatic CH), 6.90 (d, J = 8.8 Hz, 2H, aromatic CH), 6.11 (d, J = 8.8 Hz, 4H, aromatic CH), 3.80 (t, J = 6.4 Hz, 4H, OCH₂), 1.72 (m, 4H, OCH₂CH₂), 1.42-1.28 (m, 36H, alkyl CH), 0.87 (t, J = 6.8 Hz, 6H, CH₃). MALDI-TOF MS for C₄₄H₆₂N₂O₄Zn (Cacld. 746.40), m/z = 747.47 ([M + H]⁺, 60%), 769.47 ([M + Na]⁺, 100%). UV/Vis (CH₂Cl₂): λ_{max} (nm) (ε / M⁻¹cm⁻¹) 261 (1.98 × 10⁴), 313 (2.78 × 10⁴), 375 (2.50 × 10⁴). IR (KBr): ν (cm⁻¹) 2921 (ν_{CH2}), 2851 (ν_{CH2}), 1612 ($\nu_{HC=N}$), 1584, 1527, 1467, 1432. Anal. Calcd. for C₄₄H₆₂ZnN₂O₄·H₂O: C, 68.96; H, 8.42; N, 3.66. Found: C, 68.88; H, 8.35; N, 3.59%.

Section 3. MALDI-TOF MS Spectral Profiles



Figure S1. MALDI-TOF MS spectral profiles of a) TSH₆, b) TSZn₃, c) SH₂ and d) SZn.

Section 4. Absorption Coefficient Measurements



Figure S2. Plot of absorbance of $TSZn_3$ at 419 nm *vs* concentration in CH_2Cl_2 . No aggregation was observed in this concentration region.

Section 5. Gelation Experiment

Typical procedure: \mathbf{TSZn}_3 (10 mg, 4.48 μ mol) was added to decalin (1 mL) in a vial or quartz cell. Upon heating at 80 °C, \mathbf{TSZn}_3 was dissolved to give a clear solution, which was allowed to stand at r.t. for 2 h. The formation of the gels was evaluated by the "stable to inversion of a test tube" method.

The gel-to-sol transition temperature was measured by the drop ball method.^[9a]

Organic Solvents	State	
	TSH ₆	TSZn ₃
Benzene	S	S
Toluene	S	S
THF	S	S
Chloroform	S	S
Dichoromethane	S	S
Hexane	S	PG
Dodecane	S	PG
Cyclohexane	S	G (opaque)
Decalin	S	G (transparent)
Acetone	Ι	S
Diethylether	Ι	Ι
Methanol	Ι	Ι

Table S1. Gelation test of \mathbf{TSH}_6 and \mathbf{TSZn}_3 in organic solvents (10 mg/mL).

S: soluble, I: insoluble, G: gel, PG: partial gel.

Section 6. Thermally Driven Sol-Gel Transition



Figure S3. Photos of the TSZn₃ gel in decalin (4.5 mM) upon heating from 25 to 70 °C.



Figure S4. Photos of the $TSZn_3$ gel in decalin (9.0 mM) upon heating from 25 to 60 °C. It showed that the steel ball laid on the top of the gel at 30 °C, dropped to the gel between 47-49 °C, and finally sank to the bottom at 55°C. The temperature for gel-to-sol transition was estimated to be 48 °C.

Section 7. Simulation and Calculation of Sheet Structure

The experimental PXRD pattern shows three different kinds of peaks corresponding to characteristic interatomic or interlayer distances resulted from the specific molecular arrangement of **TSZn₃** (Figure *S5*). The first sharp peak at 0.189 Å⁻¹ corresponds to the *d*-spacing of 33.19 Å, which is well matched with the interlayer distance between aliphatic chain along *b*-direction. Two broad peaks at 0.557 Å⁻¹ and 1.394 Å⁻¹ are comprised of several peaks, as shown in Fig. *S*6. However, their calculated *d*-spacing values of 11.28 Å and 4.51 Å are also well matched with interatomic distance between Zn---Zn in single **TSZn₃** molecule and interlayer distance between molecular plane along *c*-direction respectively.

Figure *S*7 shows this molecular arrangement viewed along in *c*- (up) and *b*-direction (down) in the *P*32 lattice. Simulated PXRD pattern (blue curve) is calculated from *P*32 unit cell and shows a good agreement with the experimental PXRD pattern (red curve). All molecular modeling and simulation of PXRD pattern were carried out using Reflex, a software package for crystal structure determination from PXRD pattern, implemented in MS modeling ver. 4.3 (Accelrys Inc.).



Figure S5. Experimental PXRD pattern and molecular arrangement. Three peaks in PXRD pattern show characteristic distances related to molecular arrangement of **TSZn**₃.



Figure S6. Comparison of simulated PXRD pattern (black and blue curves) from the *P*32 lattice with experimental one (red curve).



Figure *S*7. Construction of *P*32 lattice with optimized $TSZn_3$ molecule in view along the *c*-direction (up) and along the *b*-direction (down). When the unit cell is elongated in *c*-direction, all atomic coordinates are exactly superposed at their positions.

Section 8. Electric Conductivity of Control Samples



Figure S8. *I*–*V* profiles of a) the \mathbf{TSZn}_3 solid sample before assembly and b) the \mathbf{SZn} solid sample (black curve: the gap itself; red curve: the sample; blue curve: the sample doped with iodine).

Section 9. Spectral Change Profiles of TSZn₃ Sheet upon Doping with Iodine



Figure S9. a) Time-dependent FT IR spectral change of the $TSZn_3$ sheet upon doping with iodine (a typical new band appeared at 1023 cm⁻¹ upon doping). b) Time-dependent electronic absorption spectral change of the $TSZn_3$ sheet upon doping with iodine (the absorption band blue-shifted from 428 to 382 nm).

Section 10. Cyclic Voltammetric Profile of TSZn₃



Figure S10. Cyclic voltammetric profile of \mathbf{TSZn}_3 in dichloromethane solution (0.5 mM) containing 0.1 M TBAPF₆. Potentials are reported versus the Fc/Fc⁺ redox couple as an internal standard.

Section 11. Photoconductivity of TSZn₃ Sheet on 10-µm-wide Pt Gap Electrodes



Figure S11. Photocurrent of the $TSZn_3$ Sheet on 10- μ m-wide Pt Gap Electrodes when light is turned on or off.

Section 12. Time-of-Flight Transient Current Integration



Figure S12. Time-of-flight transient current profile. The excitation was carried out by 355-nm laser at 1.7×10^{16} photons/cm². The current was obtained under 8 V bias voltage applied between 5 μ m interdigitated electrodes.



Figure S13. Number of charge carriers for various bias voltages.

Section 13. Supporting References

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