Supporting Information

Directional Hydrogen Bonding Controlled 2D Self-Organization of Phenyleneethynylene: From Linear Assembly to Rectangular Network

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Sl. NO.		Page
1	General experimental materials and methods	2
2	Fig. S1: Packing diagram of the **p-acid** molecules viewed along a-axis.	3
3	Table S1: Crystal data and structure refinement for **p-acid**.	4
4	Scheme S1: Schematic representation of the 2D organization of **p-acid** through ortho and meta interaction.	5
5	Synthesis of **p-acid** and **m-acid**.	6
6	Table S2: Atomic coordinates and equivalent isotropic displacement parameters for **p-acid**.	10
7	Table S3: Bond lengths [Å] and angles [deg] for **p-acid**.	11
8	Table S4: Anisotropic displacement parameters for **p-acid**.	14
9	Table S5: Hydrogen coordinates and isotropic displacement parameters for **p-acid**.	15
10	Table S6. Torsion angles for **p-acid**.	16

1. General experimental materials and methods

Samples used for the studies were purified by passing through recycling HPLC manufactured by Japan Analytical Industry Co., Ltd. All melting points are uncorrected and were determined with a Mel-Temp-II melting point apparatus. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker DPX-300 MHz or 500 MHz spectrometers. FAB mass spectra were recorded on a JEOL JM AX 5505 mass spectrometer. Matrix-assisted laser desorption ionization time-of-flight (MALDI-TOF) mass spectra were obtained on a Perseptive Biosystems Voyager DE-Pro MALDI-TOF mass spectrometer. Samples for STM investigations were prepared by drop casting 0.1 mM solution of molecules in a 9:1 mixture of 1,2,4-trichlorobenzene and THF on to a freshly cleaved HOPG surface, followed by drying in air for ~12 h. The STM images were acquired in the constant-height mode under ambient conditions on a multimode scanning probe microscope (Nano-scope IV controller, Digital Instruments). Electrochemically etched Pt/Ir wire (80:20) was used as the STM tip.

Figure S1: Packing diagram of the **p-acid** molecules viewed along a-axis. The minor disordered component is not shown in the images for clarity.

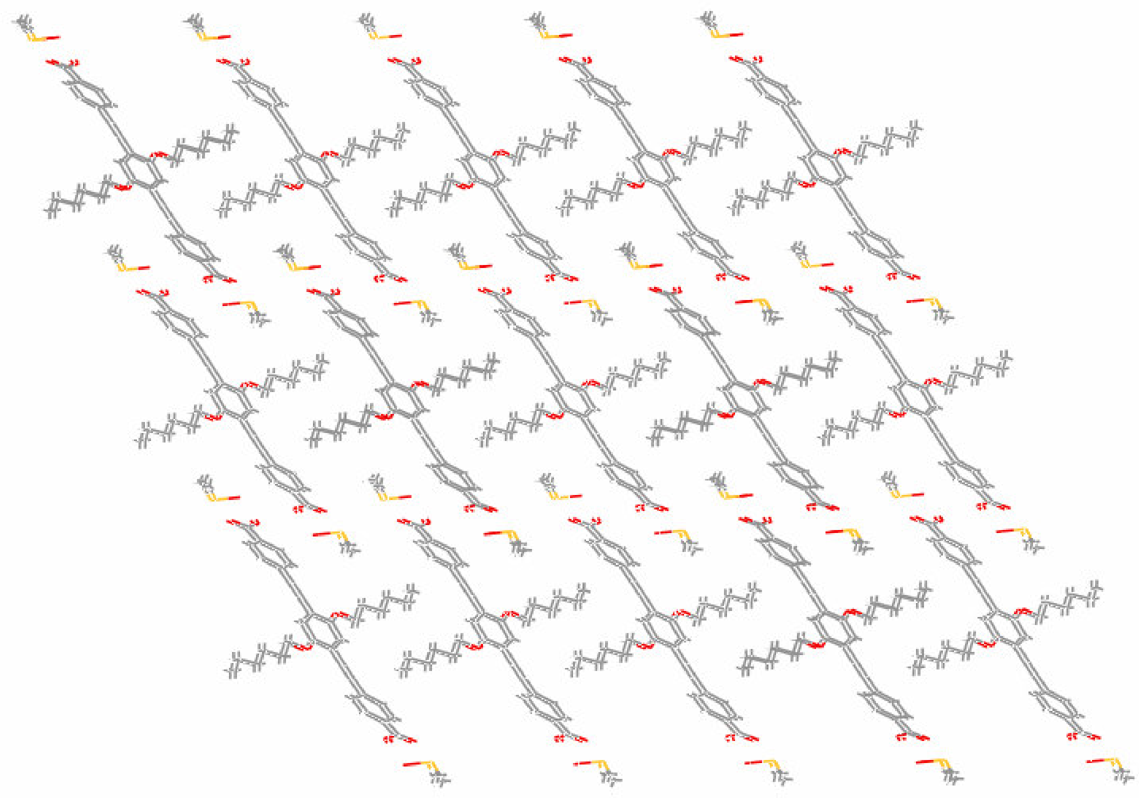
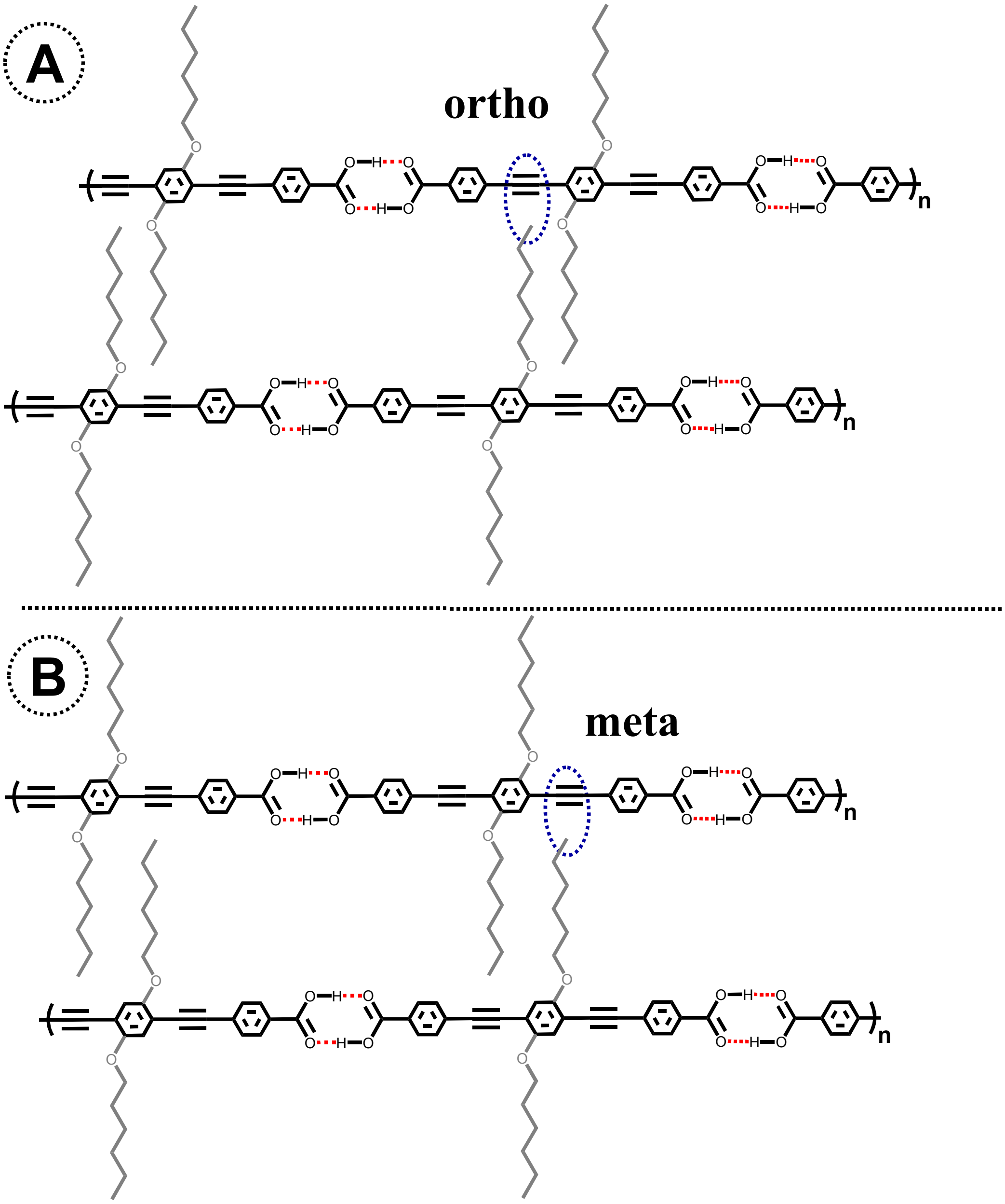


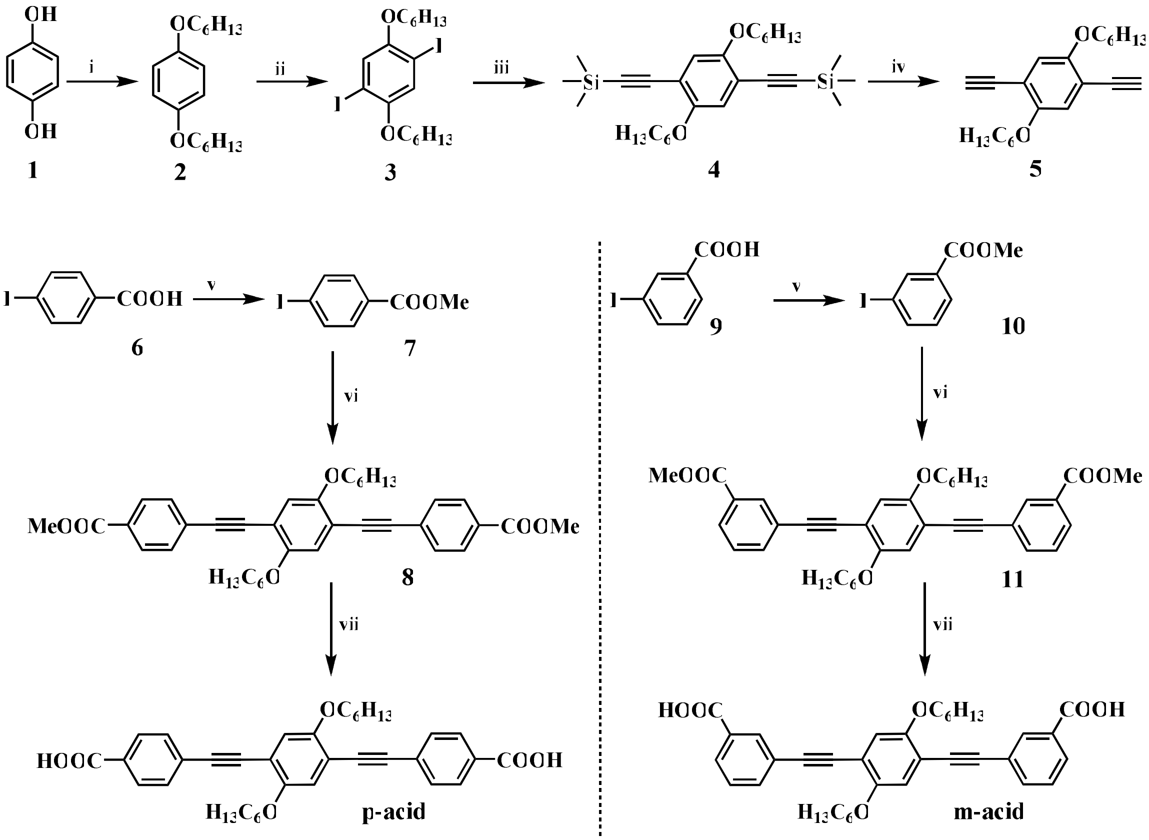
Table S1. Crystal data and structure refinement for **p-acid**.

Empirical formula	$C_{40}H_{50}O_8S_2$
Formula weight	722.92
Temperature (K)	293(2)
Wave length (Å)	0.71073
Crystal system,	Triclinic
space group	p-1
a (Å)	6.9271(3)
b (Å)	10.3631(4)
c (Å)	15.0103(7)
alpha (deg)	101.984(2)
beta (deg)	102.617(2)
gamma (deg)	101.418(2)
Volume (Å^3)	994.62(7)
Z	1
Calculated density (mg/m^3)	1.207
Absorption coefficient (mm^{-1})	0.183
F(000)	386
Crystal size (mm)	0.30 x 0.20 x 0.20
θ range for data collection (deg)	1.44 to 28.58
Limiting indices	$-9 \leq h \leq 9$, $-13 \leq k \leq 13$, $-20 \leq l \leq 19$
Reflections collected / unique	23175 / 5061 [R(int) = 0.0250]
Completeness to theta= 28.58	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9644 and 0.8573
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5061 / 1 / 254
Goodness-of-fit on F^2	1.048
Final R indices [I>2sigma(I)]	R1 = 0.0517, wR2 = 0.1522
R indices (all data)	R1 = 0.0767, wR2 = 0.1770
Largest diff. peak and hole	0.422 and -0.361 e. Å^{-3}

Scheme S1. Schematic representation of the 2D organization of **p-acid** through (A) ortho and (B) meta interaction.



5. Synthesis of p-acid and m-acid.



Scheme S2. Synthetic scheme for the preparation of **p-acid** and **m-acid**. (i) $C_6H_{13}Br$, KOH, C_2H_5OH, reflux (ii) I_2, KIO_3, H_2SO_4, H_2O, reflux (iii) TMSA, $Pd(PPh_3)_2Cl_2$, CuI, Diisoprpylamine, reflux (iv) NaOH, MeOH, THF, rt. (v) H_2SO_4, MeOH, reflux (vi) **5**, $Pd(PPh_3)_2Cl_2$, CuI, Diisoprpylamine, rt. (vii) NaOH, MeOH, reflux.

Compounds **p-acid** and **m-acid** were prepared following the procedure reported in the literature.[1] Solvents were deoxygenated by purging them with argon for 15 min, before using them in the reaction. The pre-final ester derivatives were purified by passing through recycling HPLC equipped with UV and RI detectors and which on further hydrolysis yielded final compounds.

Preparation of 2: A suspension of KOH (11.19 g, 0.19 mol) in ethanol (50 mL) was degassed and stirred at room temperature. Hydroquinone (10 g, 90 mmol) in ethanol (20 mL) was added drop wise. 1-bromohexane (32.98 g, 190 mmol) in ethanol (13 mL) was added to the stirred mixture. After stirring for 24 hours with heating at reflux, the ethanol was evaporated at reduced pressure. The residue was then extracted with chloroform. The combined chloroform layer was washed with water and dried over anhydrous sodium sulphate. The white solid product **2** (20.75 g, 83%) was obtained after column chromatography over neutral alumina using hexane as eluent; mp 43-44 °C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 6.81 (s, 4H, aromatic), 3.91-3.87 (t, 4H, OCH_2), 1.79-0.87 (m, 22H, aliphatic). ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 153.20, 115.39, 68.66, 31.61, 29.37, 25.74, 22.61, 14.03. *m/z* (FAB): 278.09 (M^+. Calcd for $C_{18}H_{30}O_2$: 278.22)

Preparation of 3: To a solution of 1,4-dihexyloxy benzene (**2**) (10 g, 35 mmol) in 81 mL of acetic acid, 6.3 mL of water and 2.7 mL of concentrated H_2SO_4, KIO_3 (9.22 g, 0.043 mol) and I_2 (10.22 g, 0.040 mol) were added. The reaction mixture was stirred at 120 °C for 24 hours and then cooled to room temperature. The excess iodine was removed by the addition of sodium thiosulphate solution until the brown color of iodine disappeared. The excess H_2SO_4 was neutralized with Na_2CO_3 solution until the brisk effervescence stopped. The residue was extracted with chloroform. The white solid product **3** (8.68 g, 46%) was separated using a silica column with hexane as eluent. mp 60-61 °C; ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 7.19 (s, 2H, aromatic), 3.94-3.90 (t, 4H, OCH_2), 1.84-0.88 (m, 22H, aliphatic). ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 149.35, 123.76, 86.33, 70.30, 31.36, 29.07, 25.64, 22.51, 13.20. *m/z* (FAB) 530.21 (M^+. Calcd for $C_{18}H_{28}I_2O_2$: 530.02)

Preparation of 4: To an ice cold solution of 1,4-dihexyloxy-2,5-diiodobenzene (**3**) (3 g, 5.66 mmol), CuI (53.77 mg, 0.283 mmol) and $Pd(PPh_3)_2Cl_2$ (198.38 g, 0.283 mmol) in 37.72 mL of diisopropylamine, trimethylsilylacetylene (TMSA) (1.75 mL, 12.452 mmol) was slowly added. The mixture was stirred at reflux using ice-cold condensing circulation for about 2 hours. The reaction mixture passed through a short celite column and further purified by column chromatography using silica column with hexane as eluent to obtain white crystals of **4** (1.4 g, 52%). mp 91-92 °C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 6.88 (s, 2H, aromatic), 3.96-3.92(t, 4H, OCH_2), 1.80-0.88(m, 22H, aliphatic), 0.25(s, 18H, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 154.40, 117.60, 114.30, 101.50, 100.40, 69.90, 31.99, 29.80, 26.00, 23.01, 14.53, 0.33. *m/z* (FAB) 470.00 (M^+. Calcd for $C_{28}H_{46}O_2Si_2$: 470.30)

Preparation of 5: To a solution of **4** (0.40 g) in THF (2.83 mL), methanol (4.25 mL) and NaOH (5 N, 0.28 mL) were added and the mixture was stirred for 2 hours. The solvent was evaporated and the residue was poured into water and extracted with chloroform. The organic layer was washed with water and dried over anhydrous sodium sulphate. A yellow solid product (0.27 g, 97%) was obtained after the solvent was removed. mp 67-68 °C. ^{1}H NMR

(300 MHz, $CDCl_3$, TMS): δ 6.95(s, 2H, aromatic), 3.97(t, 4H, OCH_2), 3.33(s, 2H, CH), 1.84-0.88(m, 22H, aliphatic). ^{13}C NMR (75 MHz, $CDCl_3$): δ 154.30, 116.60, 84.09, 83.96, 69.90, 31.97, 29.82, 26.03, 23.01, 14.51. *m/z* (FAB) 326.27 (M^+. Calcd for $C_{22}H_{30}O_2$: 326.22).

Preparation of 7: To an ice cold solution of conc. H_2SO_4 (1 mL) in methanol (10 mL), 4-iodobenzoic acid (**6)** (1.0 g) was added and refluxed for 3 hours. After cooling, the reaction mixture was added to cold water and the precipitate obtained was filtered and dried to get white crystals (720 mg, 68%). mp 112-113 °C. ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 7.81-7.75 (m, 4H, aromatic), 3.91 (s, 3H, OCH_3). ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 166.59, 139.27, 131.01, 129.60, 100.88, 52.28. *m/z* (FAB) 261.73 (M^+. Calcd for $C_{22}H_{30}O_2$: 261.95).

Preparation of 8: To a solution of 4-iodomethylbenzoate (**7**) (160 mg, .62 mmol), CuI (5.82 mg, 0.03 mmol), triphenylphosphine (16.07 mg, 0.061 mmol) and $Pd(PPh_3)_2Cl_2$ (21.49 mg, 0.03 mmol) in 10 mL diisopropylamine, **5** (100.0 mg, 0.306 mmol) was added and refluxed for 24 hours. The residue was concentrated and the yellow solid product (140 mg, 77%) purified by column chromatography over silica using ethyl acetate/hexane (4:96) as eluent. The product further purified by recycling HPLC to ensure high purity of the sample. mp 113-114 °C. ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 8.03-8.01 (d, 4H, aromatic), 7.60-7.57 (d, 4H, aromatic), 7.02 (s, 2H, aromatic), 4.06-4.02 (t, 4H, OCH_2), 3.93 (s, 6H, OCH_3), 1.88-0.87 (m, 22H, aliphatic). ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 184.89, 153.78, 131.44, 129.50, 127.41, 116.84, 114.68, 95.65, 83.21, 70.52, 31.56, 29.25, 25.73, 22.62, 14.02. *m/z* (FAB) 594.12 (M^+. Calcd for $C_{38}H_{42}O_6$: 594.30).

Preparation of p-acid: To a solution of HPLC purified **8** (100.0 mg) in THF (15 mL), KOH (1.0 g) and methanol (8 mL) were added and refluxed at a temperature of 70 °C for about 5 hours. The reaction mixture neutralized with dilute HCl to remove excess alkali and 50 mL water was added to precipitate the product. The mixture extracted with chloroform and the combined chloroform layer was evaporated under reduced pressure to yield the yellow solid product **p-acid** (90 mg, 95%). mp 278-279 °C. ^1H NMR (500 MHz, THF-d_8): δ 7.99-7.97 (d, 4H, aromatic), 7.54-7.52 (d, 4H, aromatic), 7.08 (s, 2H, aromatic), 4.04-4.02 (t, 4H, OCH_2), 1.85-0.88 (m, 22H, aliphatic). ^{13}C NMR (75 MHz, THF-d_4): δ 167.24, 155.01, 132.14, 130.79, 128.88, 118.38, 115.00, 95.00, 89.80, 70.24, 32.71, 30.39, 26.72, 23.72, 14.66. *m/z* (FAB): 566.35, (MALDI-TOF MS): 566.14 (M^+. Calcd for $C_{36}H_{38}O_6$: 566.27).

Preparation of 10: To a solution of conc. H_2SO_4 (1 mL) in methanol (10 mL), 3-iodobenzoic acid **9** (1.0 g) was added and refluxed for 3 hours. After cooling, the reaction mixture was added to cold water and the precipitate obtained was filtered and dried to get brown powder product **10** (0.6 g, 57%). mp 43-45 °C. ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 8.37 (s, 1H, aromatic), 8.01-7.98 (d, 1H, aromatic) 7.89-7.86 (d, 1H, aromatic) 7.20-7.15(m, 1H, aromatic) 3.91 (s, 3H, OCH_3). ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 165.67, 141.81, 138.54, 132.04, 130.13, 128.80, 93.86, 52.48. *m/z* (FAB) 262.07 (M^+. Calcd for $C_8H_7IO_2$: 261.95).

Preparation of 11: To a solution of 3-iodomethylbenzoate (**10**)(530.31 mg, 2.02 mmol), CuI (17.4 mg, 0.09 mmol), triphenylphosphine (48.2 mg, 0.18 mmol) and $Pd(PPh_3)_2Cl_2$ (64.4 mg, 0.09 mmol) in 25 mL diisopropylamine, **5** (300.0 mg, 0.91 mmol) was added and the reaction mixture stirred for 24 hours at room temperature. The residue was concentrated and the yellow solid product yield **11** (395 mg, 72%) purified by column chromatography over silica using ethyl acetate/hexane (4:96) as eluent. The product further purified by recycling HPLC to ensure high purity of the sample. mp 125-126 °C. ^1H NMR (300 MHz, $CDCl_3$, TMS): δ 8.21 (s, 2H, aromatic), 7.98-7.97 (d, 2H, aromatic), 7.71-7.69 (d, 2H, aromatic), 7.46-7.40 (m, 2H, aromatic) 7.03 (s, 2H, aromatic) 4.06-4.02 (t, 4H, OCH_2), 3.93 (s, 6H, OCH_3), 1.91-0.86 (m, 22H, aliphatic). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.41, 153.60, 136.17, 133.34, 130.39, 128.96, 128.42, 123.81, 113.74, 93.79, 86.79, 69.57, 51.7, 31.55, 29.22, 25.72, 22.60, 13.99. *m/z* (FAB) 594.25 (M^+. Calcd for $C_{38}H_{42}O_6$: 594.30).

Preparation of m-acid : To a solution of HPLC purified **11** (170.0 mg) in THF (10 mL), KOH (1.0 g) and methanol (5 mL) were added and refluxed at a temperature of 70 °C for about 5 hours. The reaction mixture neutralized with dilute HCl to remove excess alkali and 50 mL water was added to precipitate the product. The mixture extracted with chloroform and the combined chloroform layer was evaporated under reduced pressure to yield yellow solid product **m-acid** (155 mg, 95%). mp 216-218 °C. ^1H NMR (500 MHz, THF-d_8): δ 8.14 (s, 2H, aromatic), 7.98-7.95 (d, 2H, aromatic), 7.67-7.65 (d, 2H, aromatic), 7.45-7.42 (m, 2H, aromatic) 7.09 (s, 2H, aromatic) 4.05-4.03 (t, 4H, OCH_2) 1.84-0.86 (m, 22H, aliphatic). ^{13}C NMR (125.75 MHz, THF-d_4): δ 167.08, 155.02, 135.98, 133.54, 132.65, 130.28, 129.44, 125.06, 117.81, 115.07, 94.59, 87.89, 70.37, 32.70, 30.44, 26.91, 23.68, 14.52. *m/z* (FAB): 566.47, (MALDI-TOF MS): 566.19 (M^+. Calcd for $C_{36}H_{38}O_6$: 566.27).

Reference: P. V. James, P. K. Sudeep, C. H. Suresh, and K. George Thomas, *J. Phys. Chem. A 2006*, **110***,* 4329.

Table S2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$) for **p-acid.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	-1453(4)	14372(3)	8280(2)	85(1)
C(2)	689(4)	14421(2)	8799(2)	76(1)
C(3)	1271(3)	13097(2)	8561(1)	59(1)
C(4)	3330(3)	13107(2)	9166(1)	58(1)
C(5)	3933(3)	11782(2)	8898(1)	57(1)
C(6)	5904(3)	11776(2)	9550(1)	54(1)
C(7)	8181(2)	10317(2)	9621(1)	48(1)
C(8)	8516(2)	9054(1)	9243(1)	46(1)
C(9)	10341(2)	8750(2)	9624(1)	48(1)
C(10)	6935(2)	8075(2)	8500(1)	49(1)
C(11)	5521(3)	7284(2)	7913(1)	51(1)
C(12)	3729(2)	6350(2)	7251(1)	48(1)
C(13)	1904(3)	6735(2)	7078(1)	56(1)
C(14)	137(3)	5827(2)	6489(1)	56(1)
C(15)	164(2)	4509(2)	6069(1)	48(1)
C(16)	1986(3)	4135(2)	6223(1)	58(1)
C(17)	3762(3)	5042(2)	6804(1)	59(1)
C(18)	-1748(3)	3472(2)	5482(1)	56(1)
C(19)	9763(4)	9818(3)	6290(2)	100(1)
C(20)	6124(4)	10065(3)	6520(2)	89(1)
O(1)	6374(2)	10524(1)	9193(1)	62(1)
O(2)	-3339(2)	3979(2)	5343(1)	86(1)
O(3)	-1804(2)	2292(1)	5174(1)	85(1)
O(4)	6657(3)	7693(2)	5811(2)	71(1)
S(1)	7129(1)	9135(1)	5705(1)	64(1)
S(1')	7541(6)	8794(3)	6203(3)	92(1)
O(4')	6522(10)	8274(9)	5109(6)	110(3)

Table S3. Bond lengths [Å] and angles [deg] for **p-acid.**

C(1)-C(2)	1.503(3)
C(1)-H(1A)	0.9600
C(1)-H(1B)	0.9600
C(1)-H(1C)	0.9600
C(2)-C(3)	1.499(3)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(4)	1.511(3)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-C(5)	1.515(2)
C(4)-H(4A)	0.9700
C(4)-H(4B)	0.9700
C(5)-C(6)	1.498(2)
C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700
C(6)-O(1)	1.4261(19)
C(6)-H(6A)	0.9700
C(6)-H(6B)	0.9700
C(7)-O(1)	1.3560(19)
C(7)-C(9)#1	1.384(2)
C(7)-C(8)	1.399(2)
C(8)-C(9)	1.393(2)
C(8)-C(10)	1.424(2)
C(9)-C(7)#1	1.384(2)
C(9)-H(9)	0.9300
C(10)-C(11)	1.189(2)
C(11)-C(12)	1.432(2)
C(12)-C(13)	1.387(2)
C(12)-C(17)	1.389(2)
C(13)-C(14)	1.376(2)
C(13)-H(13)	0.9300
C(14)-C(15)	1.386(2)
C(14)-H(14)	0.9300
C(15)-C(16)	1.379(2)
C(15)-C(18)	1.489(2)
C(16)-C(17)	1.375(2)
C(16)-H(16)	0.9300
C(17)-H(17)	0.9300
C(18)-O(3)	1.204(2)
C(18)-O(2)	1.307(2)
C(19)-S(1')	1.649(4)
C(19)-S(1)	1.770(2)
C(19)-H(19A)	0.9600
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600
C(20)-S(1)	1.749(2)
C(20)-S(1')	1.838(4)
C(20)-H(20A)	0.9600
C(20)-H(20B)	0.9600
C(20)-H(20C)	0.9600
O(2)-H(2)	0.8200
O(4)-S(1)	1.514(2)
S(1')-O(4')	1.569(10)
C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5

C(3)-C(2)-C(1)	114.2(2)
C(3)-C(2)-H(2A)	108.7
C(1)-C(2)-H(2A)	108.7
C(3)-C(2)-H(2B)	108.7
C(1)-C(2)-H(2B)	108.7
H(2A)-C(2)-H(2B)	107.6
C(2)-C(3)-C(4)	113.81(17)
C(2)-C(3)-H(3A)	108.8
C(4)-C(3)-H(3A)	108.8
C(2)-C(3)-H(3B)	108.8
C(4)-C(3)-H(3B)	108.8
H(3A)-C(3)-H(3B)	107.7
C(3)-C(4)-C(5)	113.32(15)
C(3)-C(4)-H(4A)	108.9
C(5)-C(4)-H(4A)	108.9
C(3)-C(4)-H(4B)	108.9
C(5)-C(4)-H(4B)	108.9
H(4A)-C(4)-H(4B)	107.7
C(6)-C(5)-C(4)	112.39(14)
C(6)-C(5)-H(5A)	109.1
C(4)-C(5)-H(5A)	109.1
C(6)-C(5)-H(5B)	109.1
C(4)-C(5)-H(5B)	109.1
H(5A)-C(5)-H(5B)	107.9
O(1)-C(6)-C(5)	107.18(13)
O(1)-C(6)-H(6A)	110.3
C(5)-C(6)-H(6A)	110.3
O(1)-C(6)-H(6B)	110.3
C(5)-C(6)-H(6B)	110.3
H(6A)-C(6)-H(6B)	108.5
O(1)-C(7)-C(9)#1	124.96(14)
O(1)-C(7)-C(8)	115.34(13)
C(9)#1-C(7)-C(8)	119.71(14)
C(9)-C(8)-C(7)	119.78(14)
C(9)-C(8)-C(10)	121.11(14)
C(7)-C(8)-C(10)	119.05(14)
C(7)#1-C(9)-C(8)	120.51(14)
C(7)#1-C(9)-H(9)	119.7
C(8)-C(9)-H(9)	119.7
C(11)-C(10)-C(8)	175.43(19)
C(10)-C(11)-C(12)	175.84(19)
C(13)-C(12)-C(17)	119.15(15)
C(13)-C(12)-C(11)	119.65(14)
C(17)-C(12)-C(11)	121.16(15)
C(14)-C(13)-C(12)	120.62(15)
C(14)-C(13)-H(13)	119.7
C(12)-C(13)-H(13)	119.7
C(13)-C(14)-C(15)	120.01(16)
C(13)-C(14)-H(14)	120.0
C(15)-C(14)-H(14)	120.0
C(16)-C(15)-C(14)	119.40(15)
C(16)-C(15)-C(18)	119.20(15)
C(14)-C(15)-C(18)	121.37(16)
C(17)-C(16)-C(15)	120.83(15)
C(17)-C(16)-H(16)	119.6
C(15)-C(16)-H(16)	119.6
C(16)-C(17)-C(12)	119.94(16)
C(16)-C(17)-H(17)	120.0
C(12)-C(17)-H(17)	120.0
O(3)-C(18)-O(2)	124.05(17)
O(3)-C(18)-C(15)	122.97(18)
O(2)-C(18)-C(15)	112.98(15)
S(1')-C(19)-S(1)	30.49(15)
S(1')-C(19)-H(19A)	106.0
S(1)-C(19)-H(19A)	109.5
S(1')-C(19)-H(19B)	83.0

S(1)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
S(1')-C(19)-H(19C)	135.0
S(1)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
S(1)-C(20)-S(1')	29.13(13)
S(1)-C(20)-H(20A)	109.5
S(1')-C(20)-H(20A)	124.4
S(1)-C(20)-H(20B)	109.5
S(1')-C(20)-H(20B)	80.5
H(20A)-C(20)-H(20B)	109.5
S(1)-C(20)-H(20C)	109.5
S(1')-C(20)-H(20C)	118.2
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
C(7)-O(1)-C(6)	118.64(12)
C(18)-O(2)-H(2)	109.5
O(4)-S(1)-C(20)	105.38(15)
O(4)-S(1)-C(19)	104.72(14)
C(20)-S(1)-C(19)	98.82(14)
O(4')-S(1')-C(19)	103.6(4)
O(4')-S(1')-C(20)	99.9(4)
C(19)-S(1')-C(20)	99.87(19)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y+2,-z+2

Table S4. Anisotropic displacement parameters ($Å^2 x 10^3$) for **p-acid.** The anisotropic displacement factor exponent takes the form: $-2\pi^2$.[h^2.a^{*2}.U11+...+ 2h.k.a*.b*.U12]

	U11	U22	U33	U23	U13	U12
C(1)	77(2)	112(2)	93(2)	48(1)	31(1)	54(1)
C(2)	72(1)	84(1)	81(1)	22(1)	21(1)	41(1)
C(3)	54(1)	66(1)	61(1)	21(1)	14(1)	22(1)
C(4)	49(1)	58(1)	67(1)	14(1)	11(1)	19(1)
C(5)	46(1)	56(1)	64(1)	13(1)	4(1)	16(1)
C(6)	48(1)	48(1)	60(1)	9(1)	5(1)	15(1)
C(7)	41(1)	46(1)	48(1)	10(1)	0(1)	10(1)
C(8)	43(1)	43(1)	43(1)	7(1)	2(1)	6(1)
C(9)	46(1)	41(1)	49(1)	4(1)	2(1)	11(1)
C(10)	46(1)	44(1)	48(1)	6(1)	1(1)	10(1)
C(11)	47(1)	48(1)	47(1)	6(1)	2(1)	9(1)
C(12)	45(1)	47(1)	41(1)	6(1)	1(1)	4(1)
C(13)	53(1)	40(1)	62(1)	2(1)	-1(1)	9(1)
C(14)	46(1)	50(1)	60(1)	6(1)	1(1)	11(1)
C(15)	46(1)	46(1)	41(1)	6(1)	5(1)	2(1)
C(16)	56(1)	47(1)	57(1)	-6(1)	6(1)	11(1)
C(17)	47(1)	58(1)	59(1)	-3(1)	2(1)	15(1)
C(18)	50(1)	53(1)	50(1)	3(1)	9(1)	-1(1)
C(19)	58(1)	79(2)	128(3)	-10(1)	3(1)	5(1)
C(20)	83(2)	80(2)	99(2)	7(1)	24(1)	29(1)
O(1)	48(1)	51(1)	68(1)	-2(1)	-12(1)	18(1)
O(2)	46(1)	74(1)	104(1)	-19(1)	-2(1)	2(1)
O(3)	70(1)	51(1)	101(1)	-7(1)	-1(1)	-1(1)
O(4)	54(1)	49(1)	88(2)	4(1)	-6(1)	3(1)
S(1)	57(1)	62(1)	60(1)	6(1)	2(1)	10(1)
S(1')	104(2)	72(2)	121(3)	38(2)	48(2)	38(2)
O(4')	59(4)	117(6)	98(6)	-53(5)	16(4)	-12(4)

Table S5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2x10^3$) for **p-acid.**

	x	y	z	U(eq)
H(1A)	-1593	14154	7611	127
H(1B)	-1726	15244	8477	127
H(1C)	-2408	13687	8419	127
H(2A)	1638	15118	8655	91
H(2B)	832	14687	9473	91
H(3A)	1287	12885	7903	71
H(3B)	233	12378	8635	71
H(4A)	3300	13286	9823	70
H(4B)	4363	13845	9110	70
H(5A)	2856	11033	8909	68
H(5B)	4067	11636	8257	68
H(6A)	6987	12544	9569	65
H(6B)	5762	11844	10186	65
H(9)	10567	7914	9369	58
H(13)	1875	7615	7363	68
H(14)	-1076	6097	6371	67
H(16)	2015	3257	5930	70
H(17)	4985	4781	6897	71
H(19A)	10535	9246	6020	123(10)
H(19B)	9986	9858	6950	172(17)
H(19C)	10197	10720	6216	160(14)
H(20A)	6589	11021	6572	104(9)
H(20B)	6574	9895	7126	153(14)
H(20C)	4657	9791	6309	162(14)
H(2)	-4315	3400	4961	145(13)

Table S6. Torsion angles [deg] for **p-acid.**

C(1)-C(2)-C(3)-C(4)	-173.33(18)
C(2)-C(3)-C(4)-C(5)	-178.02(17)
C(3)-C(4)-C(5)-C(6)	-175.81(16)
C(4)-C(5)-C(6)-O(1)	-176.34(15)
O(1)-C(7)-C(8)-C(9)	179.05(15)
C(9)#1-C(7)-C(8)-C(9)	-0.6(3)
O(1)-C(7)-C(8)-C(10)	-3.8(2)
C(9)#1-C(7)-C(8)-C(10)	176.58(16)
C(7)-C(8)-C(9)-C(7)#1	0.6(3)
C(10)-C(8)-C(9)-C(7)#1	-176.52(16)
C(9)-C(8)-C(10)-C(11)	136(2)
C(7)-C(8)-C(10)-C(11)	-41(2)
C(8)-C(10)-C(11)-C(12)	-11(4)
C(10)-C(11)-C(12)-C(13)	46(2)
C(10)-C(11)-C(12)-C(17)	-131(2)
C(17)-C(12)-C(13)-C(14)	1.5(3)
C(11)-C(12)-C(13)-C(14)	-176.13(17)
C(12)-C(13)-C(14)-C(15)	0.7(3)
C(13)-C(14)-C(15)-C(16)	-2.2(3)
C(13)-C(14)-C(15)-C(18)	175.68(16)
C(14)-C(15)-C(16)-C(17)	1.6(3)
C(18)-C(15)-C(16)-C(17)	-176.38(17)
C(15)-C(16)-C(17)-C(12)	0.6(3)
C(13)-C(12)-C(17)-C(16)	-2.2(3)
C(11)-C(12)-C(17)-C(16)	175.45(17)
C(16)-C(15)-C(18)-O(3)	4.2(3)
C(14)-C(15)-C(18)-O(3)	-173.65(19)
C(16)-C(15)-C(18)-O(2)	-175.93(17)
C(14)-C(15)-C(18)-O(2)	6.2(2)
C(9)#1-C(7)-O(1)-C(6)	-1.6(3)
C(8)-C(7)-O(1)-C(6)	178.75(15)
C(5)-C(6)-O(1)-C(7)	174.94(15)
S(1')-C(20)-S(1)-O(4)	42.8(3)
S(1')-C(20)-S(1)-C(19)	-65.2(2)
S(1')-C(19)-S(1)-O(4)	-32.3(2)
S(1')-C(19)-S(1)-C(20)	76.3(2)
S(1)-C(19)-S(1')-O(4')	34.8(4)
S(1)-C(19)-S(1')-C(20)	-67.9(2)
S(1)-C(20)-S(1')-O(4')	-28.1(4)
S(1)-C(20)-S(1')-C(19)	77.8(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y+2,-z+2