Supporting Information

Oxidized forms of tripyrrane: α-tripyrrinone, β-tripyrrinone and C2 symmetric hexapyrrole

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Contents.

1. Experimental

1-1. General

^{1}H and ^{13}C NMR spectra were recorded with Bruker Avance 300, 400, 600 MHz NMR spectrometers in CD_2Cl_2 using tetramethylsilane (TMS) as internal standard. Chemical shifts are expressed in ppm downfield from TMS using the residual non-deuterated solvent as internal standard (CD_2Cl_2: ^{1}H 5.32 ppm, ^{13}C 54.0 ppm). Mass spectra were determined on a time-of-flight (TOF) mass spectrometers equipped with electrospray (ESI) or MALDI ion source, and a high resolution double focusing hybrid mass spectrometer (EI) having EBQQ configuration. Optical spectra were recorded with a Cary 5000 UV-vis spectrophotometer using a 1 cm cell. The crystals were grown by diffusion of hexane into CH_2Cl_2 solutions. Data were collected and integrated using the Bruker SAINT software package and corrected for absorption effects using the multi-scan technique (SADABS). The structures were solved by direct methods and all refinements were performed using the SHELXTL crystallographic software package of Bruker-AXS. All chemicals were purchased from commercial suppliers and used without further purification.

1-2. Experimental procedures and spectroscopic data

Polypyrranes. Pentafluorobenzaldehyde (2.0 g, 10.2 mmol) or 2,6-dichlorobenzaldehyde, pyrrole (15 mL), and penta-fluorophenyl dipyrromethane (2.0 g, 6.4 mmol) or 2,6-dichlorobenzaldehyde were dissolved in CH_2Cl_2 (25 mL) at -5 °C under Ar. TFA (200 μL) was added and the reaction mixture was stirred for 45 min. After quenching with a saturated NaOH aqueous solution, the organic layer was dried over anhydrous Na_2SO_4. The solvent and excess pyrrole were removed

by rotary evaporation and vacuum distillation. Column chromatography on silica gel with CH_2Cl_2/hexane eluent followed by Bio-Bead gel permeation chromatography with toluene gave tripyrrane in 21% yield (1.2 g).
*Spectral data for **1***: ^{1}H NMR (300 MHz, CD_2Cl_2) δ = 8.25 (bs, 2H, NH), 8.17 (bs, 1H, NH), 6.75 (m, 2H, *β*H), 6.16 (m, 2H, *β*H), 6.04 (s, 2H, *α*H), 6.02 (d, *J* = 2.4 Hz, 2H, *β*H), 5.90 (s, 2H, *meso*-H); ^{19}F NMR (282.4 MHz, CD_2Cl_2) δ = -142.75 (dd, *J* = 14.1 Hz, ^{dd}J = 8.5 Hz, 4F, *o*-F), -157.16 (dt, *J* = 19.8 Hz, ^{dt}J = 5.6 Hz, 2F, *p*-F), -162.65 (m, 4F, *m*-F); ^{13}C NMR (75.48 MHz, CD_2Cl_2) δ = 135~150 (the peaks are broadened by the multiple ^{13}C-^{19}F couplings of the perfluorophenyl rings), 129.16 (*α*C, 2C), 128.42(*α*C, 2C), 118.60 (terminal *α*CH, 2C), 109.17 (*β*CH, 2C), 108.40 (*β*CH, 2C), 108.08 (*β*CH, 2C), 33.73 (meso-C, 2C); *m/z* EIMS found 557.2, calcd. 557.1 for $C_{26}H_{13}F_{10}N_3$ ([M]$^+$, 100%).
*Spectral data for **2***: ^{1}H NMR (400 MHz, CD_2Cl_2) δ = 8.43 (bs, 1H, NH), 8.25 (bs, 2H, NH), 7.34 (dd, *J* = 8.0 Hz, ^{dd}J = 3.5 Hz, 4H, *m*-H), 7.16 (dt, *J* = 8.0 Hz, ^{dd}J = 2.6 Hz, 2H, *p*-H), 6.68 (m, 2H, *α*H), 6.41 (s, 2H, *meso*-H), 6.11 (m, 2H, *β*H), 5.99 (d, *J* = 8.0, 1H, *β*H), 5.94 (m, 4H, *β*H)

Compounds 3-6 and 7 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ, 1.1 g, 4.8 mmol) was added to a solution of *meso*-pentafluorophenyl tripyrrane (400 mg, 0.72 mmol) in CH_2Cl_2 (100 mL) and stirring was continued for 2 h. After the solvent was removed by rotary evaporation, the isomeric target products **3** and **5** were isolated as purple and red fractions by column chromatography on silica gel using mixed eluents of CH_2Cl_2 and MeOH with gradually increasing polarity (23 and 10% yields, respectively). Similarly, compounds **4** and **6** were prepared from *meso*-2,6-dichlorophenyl tripyrrane in less than 5% yield for **4** and an even smaller yield (< 0.5%) for **6**. Additionally, compound **7** was obtained from this reaction in ca. 30% yield.
*Spectral data for **3***: ^{1}H NMR (600 MHz, CD_2Cl_2) δ = 11.58 (bs, 1H, NH), 10.01 (bs, 1H, NH), 7.51 (s, 1H, *α*H), 6.85 (d, *J* = 5.7, 1H, *β*H), 6.75 (d, *J* = 4.7, 1H, *α*H), 6.43 (s, 1H, *β*H), 6.42 (d, *J* = 3.8,1H, *β*H), 6.35 (d, *J* = 5.7, 1H, *β*H), 6.28 (d, *J* = 4.8, 1H, *β*H); ^{19}F NMR (282.4 MHz, CD_2Cl_2) δ = -139.13 (dd, *J* = 8.6, ^{dd}J = 23.6, 2F, *o*-F), -139.61 (dd, *J* = 6.5, ^{dd}J = 19.3, 2F, *o*-F), -153.07 (t, *J* = 21.5, 1F, *p*-F), -153.25 (t, *J* = 21.5, 1F, *p*-F), -161.88 (m, 4F, *m*-F); ^{13}C NMR (150.96 MHz, CD_2Cl_2) δ = 171.77, 167.13, 151.14, 144.53, 147 ~137 (the peaks are broadened by the multiple ^{13}C-^{19}F couplings of the perfluorophenyl rings), 136.76, 135.23, 132.15, 131.95, 129.13, 127.73, 126.62, 124.41, 122.96, 113.98, 112.5 ~ 110.5 (the peaks are broadened by the multiple ^{13}C-^{19}F couplings of the perfluorophenyl rings), 101.71; *m/z* EIMS found 570.0674, calcd. 570.0664 for $C_{26}H_{10}N_3OF_{10}$ ([M+H]$^+$).
*Spectral data for **4***: ^{1}H NMR (400 MHz, CD_2Cl_2) δ = 11.43 (bs, 1H, NH), 9.91 (br.1H, NH), 7.45 (m, 5H, Ary-H and αH), 7.39 (dt, *J* = 7.0, ^{dt}J = 9.0, Ary-H), 6.71 (d, *J* = 5.5, 1H, βH), 6.57 (d, *J* = 4.3, 1H, βH), 6.37 (dd, *J* = 3.9, ^{dd}J = 1.6, βH), 6.28 (dd, *J* = 4.5, ^{dd}J = 1.0, βH), 6.26 (d, *J* = 5.9, βH), 6.08 (d, *J* = 4.7, βH); m/z ESIMS found 526.0045, calcd. 526.0047 for $C_{26}H_{16}N_3OCl_4$ ([M+H]$^+$).
*Spectral data for **5***: ^{1}H NMR (300 MHz, CD_2Cl_2) δ = 12.03 (bs, 1H, NH), 8.06 (dd, *J* = 5.8, ^{dd}J = 1.4, 1H, *α*H), 8.00 (bs, 1H, NH), 7.35 (s, 1H, *α*H), 6.74 (d, *J* = 4.7, 1H, *β*H), 6.50 (d, *J* = 4.7, 1H, *β*H), 6.39 (dd, *J* = 6.0, ^{dd}J = 1.7, 1H, *β*H), 6.37 (m, 1H, *β*H), 6.36 (s, 1H, *β*H); ^{19}F NMR (282.4 MHz, CD_2Cl_2) δ = -138.40 (d, *J* = 17.2, 2F, *o*-F), -139.67 (dd, *J* = 4.1, ^{dd}J = 8.9, 2F, *o*-F), -153.24 (t, *J* = 20.6, 1F, *p*-F), -153.25 (t, *J* = 21.5, 1F, *p*-F), -161.51 (m, 2F, *m*-F), -161.98 (m, 2F, *m*-F); ^{13}C NMR (75.48 MHz, CD_2Cl_2) δ = 171.64, 164.64, 150.01, 144.02, 147 ~137 (the peaks are broadened by the multiple ^{13}C-^{19}F couplings of the perfluorophenyl rings), 137.27, 134.54, 132.88, 132.27, 127.96, 126.14, 122.68, 119.57, 113.96; *m/z* ESIMS (+ev) found 570.0, calcd. 570.1 for $C_{26}H_{10}N_3OF_{10}$ ([M+H]$^+$), (-ev) found 567.9, calcd. 568.1 for $C_{26}H_8N_3OF_{10}$ ([M-H]$^-$).
*Spectral data for **6***: *m/z* ESIMS found 526.0052, calcd. 526.0047 for $C_{26}H_{16}N_3OCl_4$ ([M+H]$^+$).
*Selected data for **7***: ^{1}H NMR (300 MHz, CD_2Cl_2) δ = 12.84 (bs, 2H, NH), 12.35 (bs, 2H, NH), 7.88 (s, 2H, αH), 749-7.36 (m, 12H, Ary-H), 6.95 (dd, *J* = 5.5, ^{dd}J = 1.7, 2H, βH), 6.60 (d, *J* = 5.5, ^{dd}J = 1.7, 2H, βH), 6.47 (d, *J* = 4.4, 2H, βH), 6.63 (m, 2H, βH), 6.29 (m, 2H, βH), 6.05 (d, *J* = 4.4, 2H, βH); ^{13}C NMR (100 MHz, CD_2Cl_2) δ = 183.02, 163.12, 162.38, 151.55, 143.38, 138.86, 138.57, 136.80, 136.09, 135.94, 134.87, 134.73, 134.16, 134.07, 132.71, 131.53, 131.11, 128.86, 128.57, 127.72, 127.03, 126.09, 124.47, 117.68, 114.82, 83.03; m/z found 1248.1, calcd. 1247.9 for $C_{60}H_{32}Cl_{10}N_8O_2$ (M^+).

Spectra of compounds 3-7.

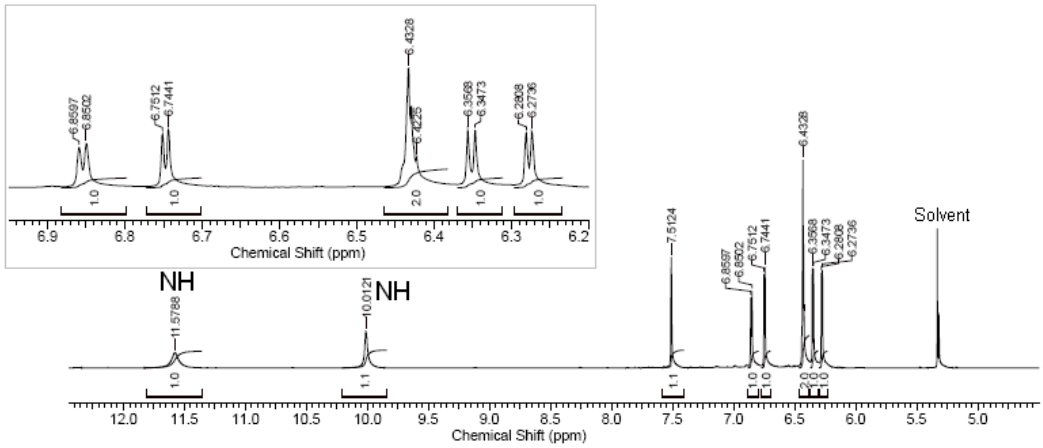

Fig. S1 ^{1}H NMR (600 MHz) spectrum of **3** in CD_2Cl_2.

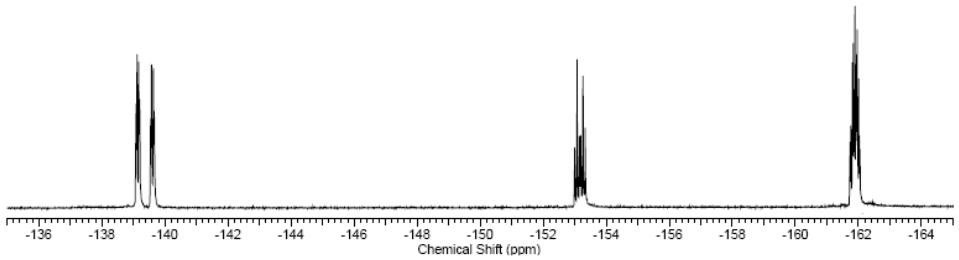

Fig. S2 ^{19}F NMR (282.4 MHz) spectrum of **3** in CD_2Cl_2.

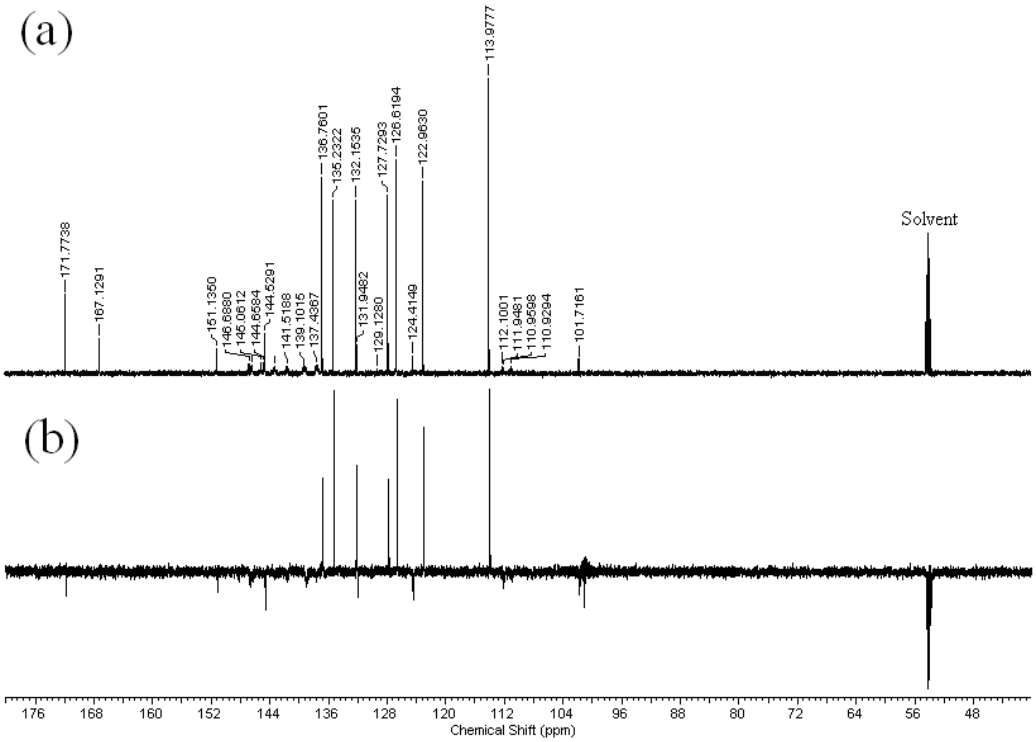

Fig. S3 ^{13}C NMR (151 MHz) spectra of **3** in CD_2Cl_2; (a) H-decoupled ^{13}C NMR and (b) ^{13}C APT spectra.

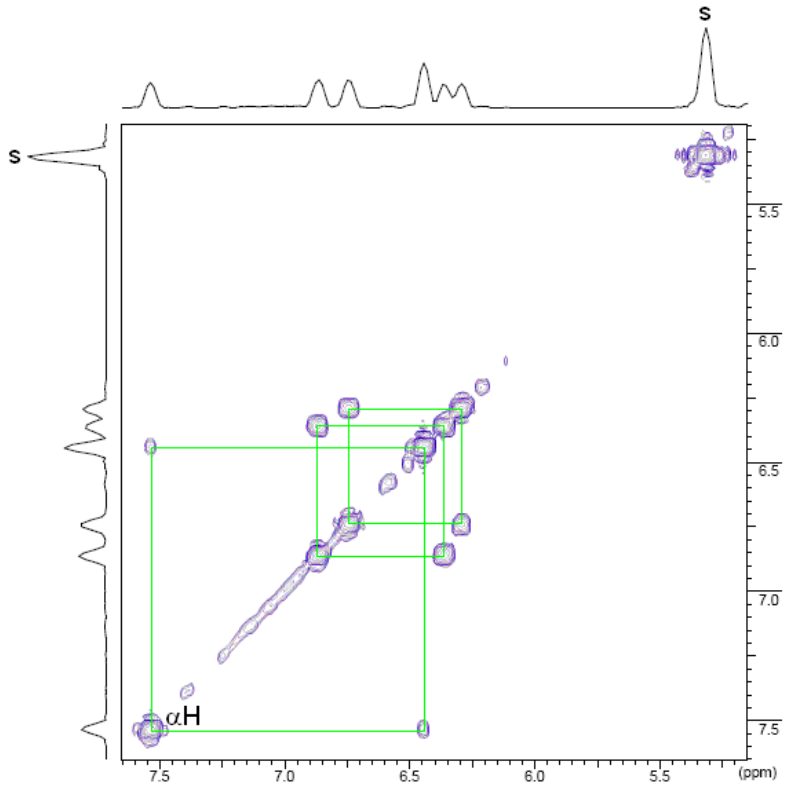

Fig. S4 HH COSY (F1 and F2: 600 MHz) spectrum of **3** in CD_2Cl_2.

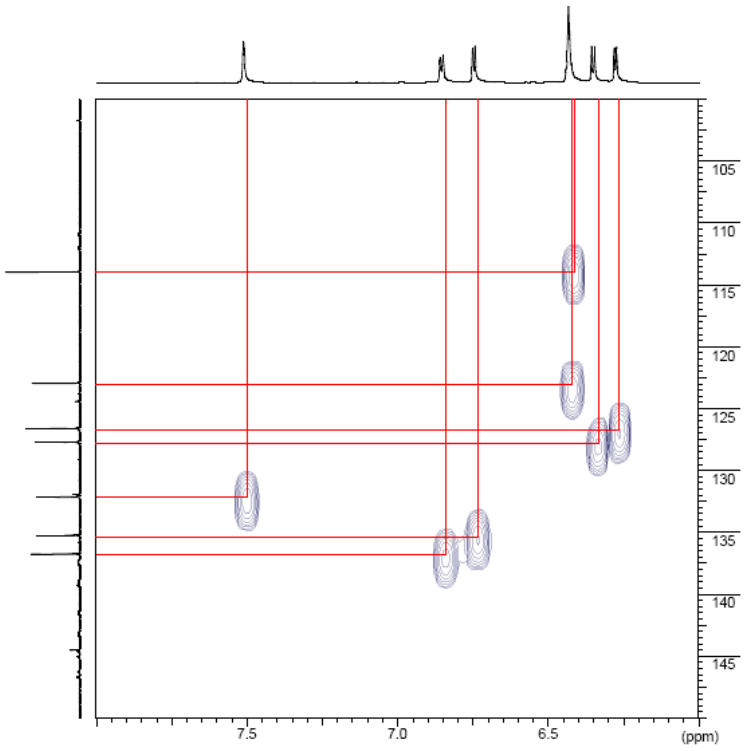

Fig. S5 HMQC NMR (F1: 600 MHz and F2: 151 MHz) spectrum of **3.**

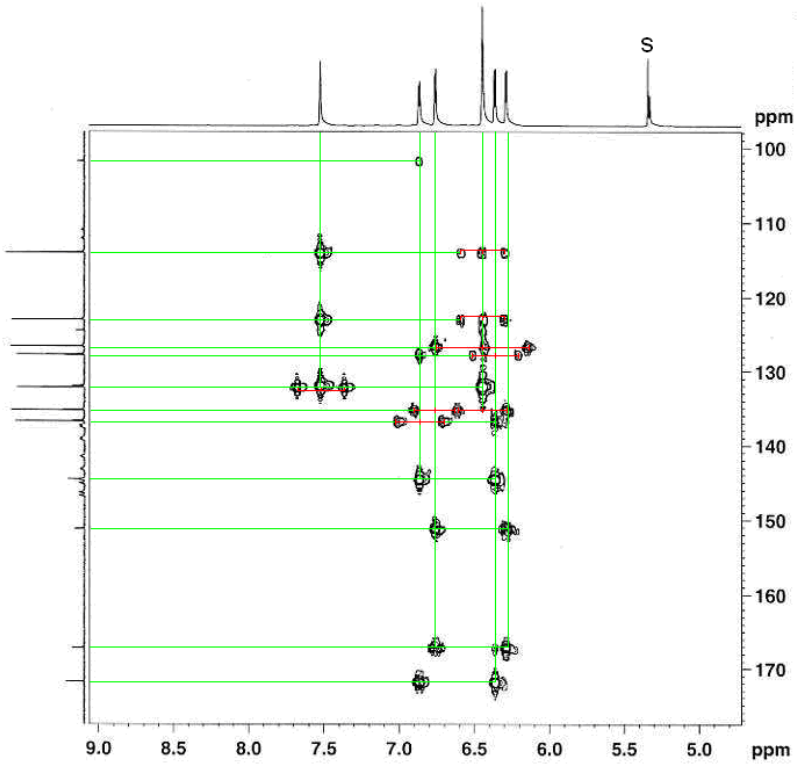

Fig. S6 HMBC NMR (F1: 600 MHz and F2: 151 MHz) spectrum of **3.**

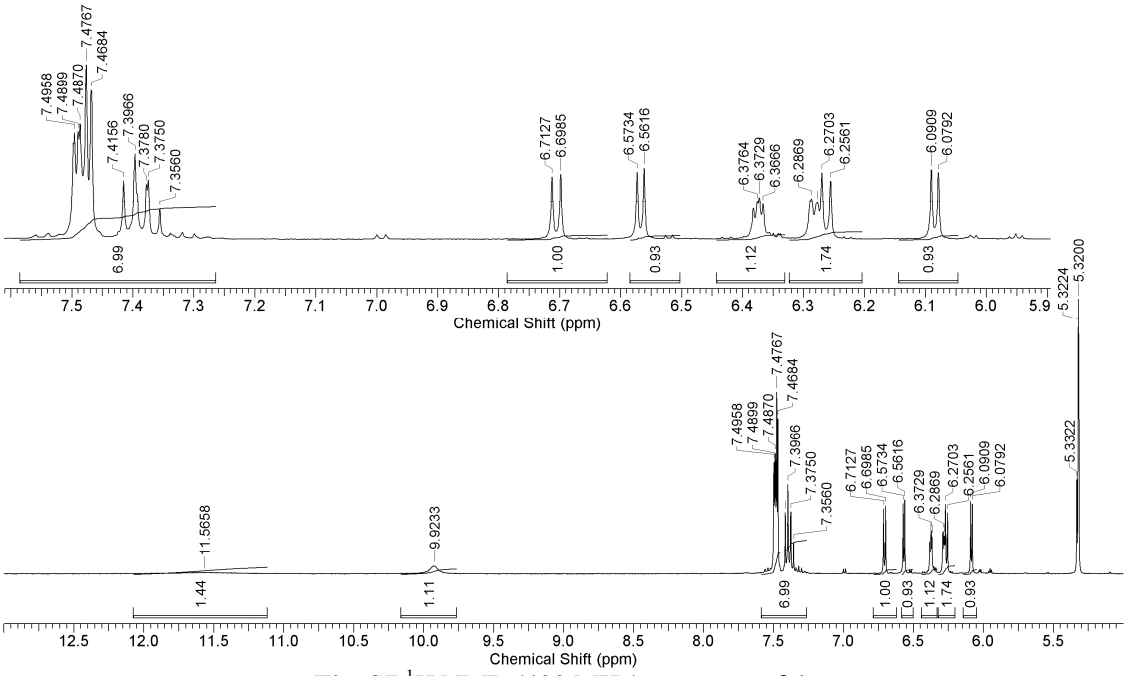

Fig. S7 ^{1}H NMR (400 MHz) spectrum of **4.**

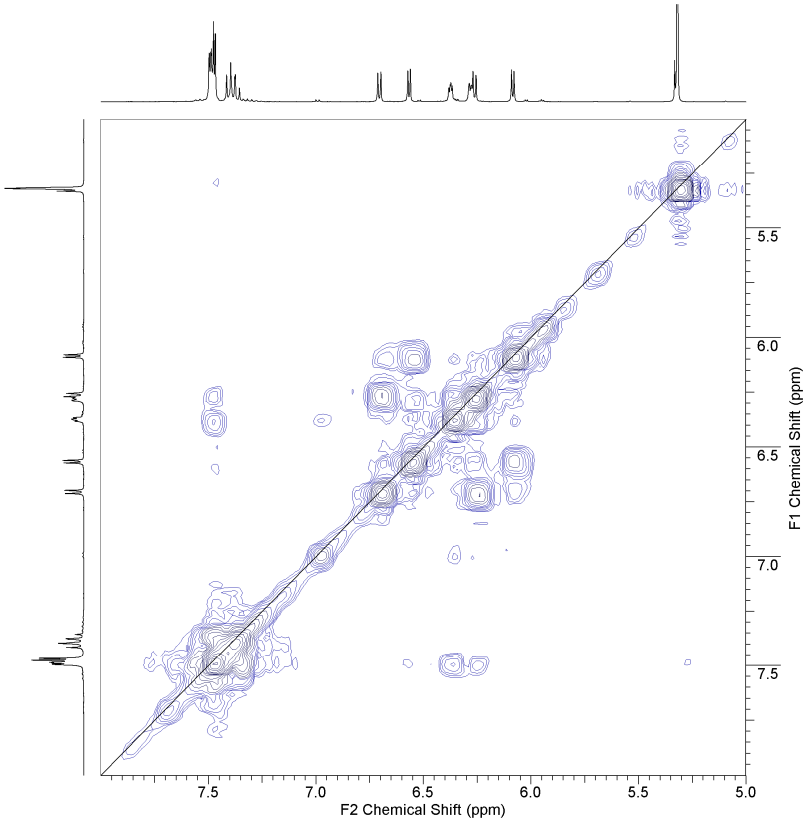

Fig. S8 HH COSY NMR (F1 and F2: 400 MHz) spectrum of **4**.

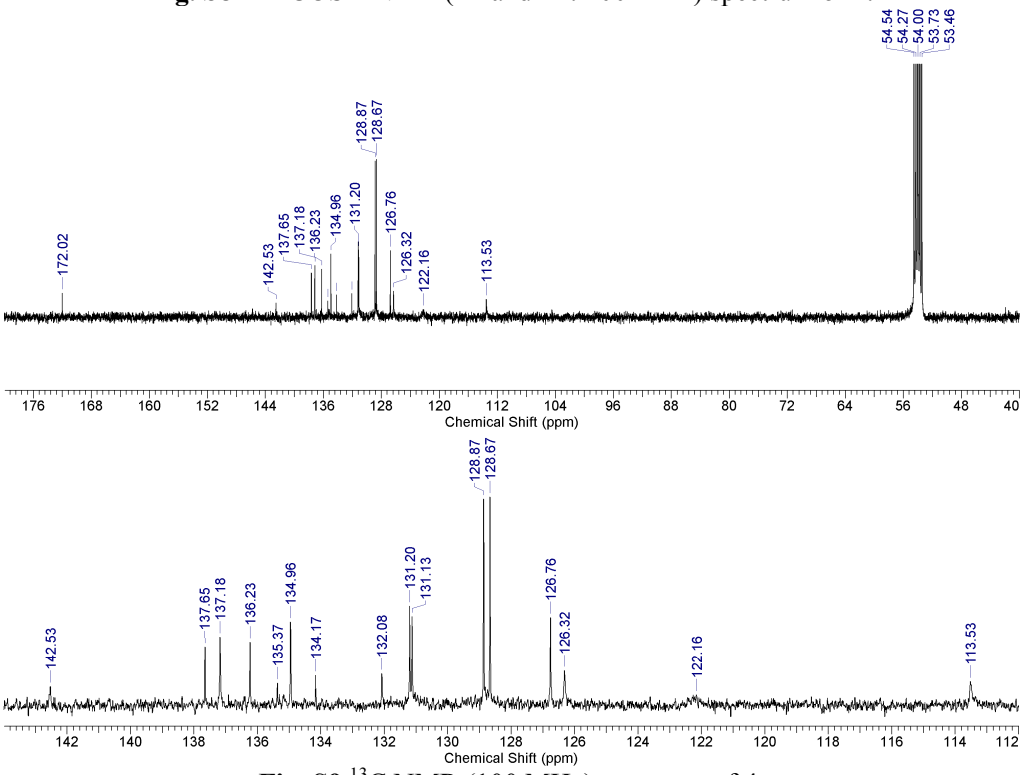

Fig. S9 ^{13}C NMR (100 MHz) spectrum of **4.**

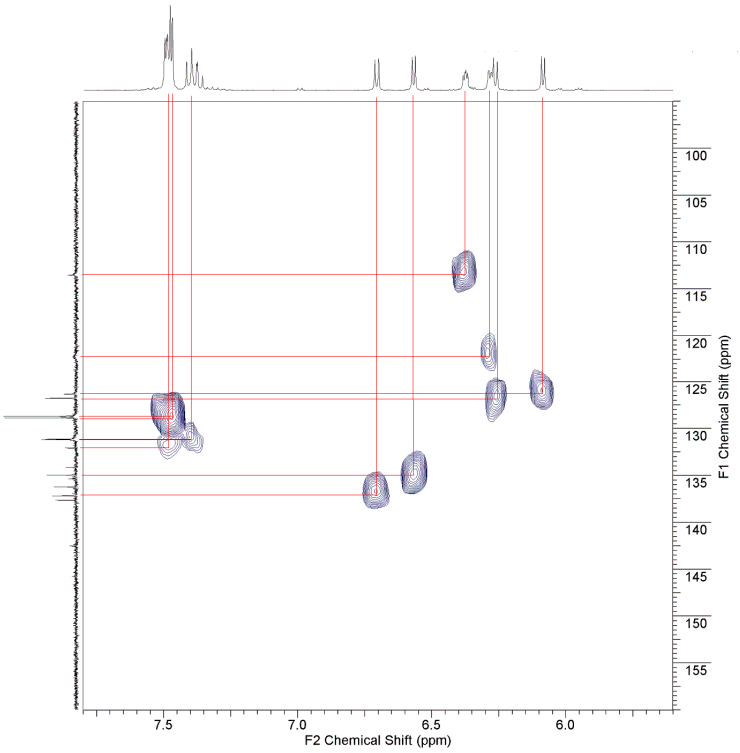

Fig. S10 HMQC NMR (F1: 100 MHz and F2: 400 MHz) spectrum of **4.**

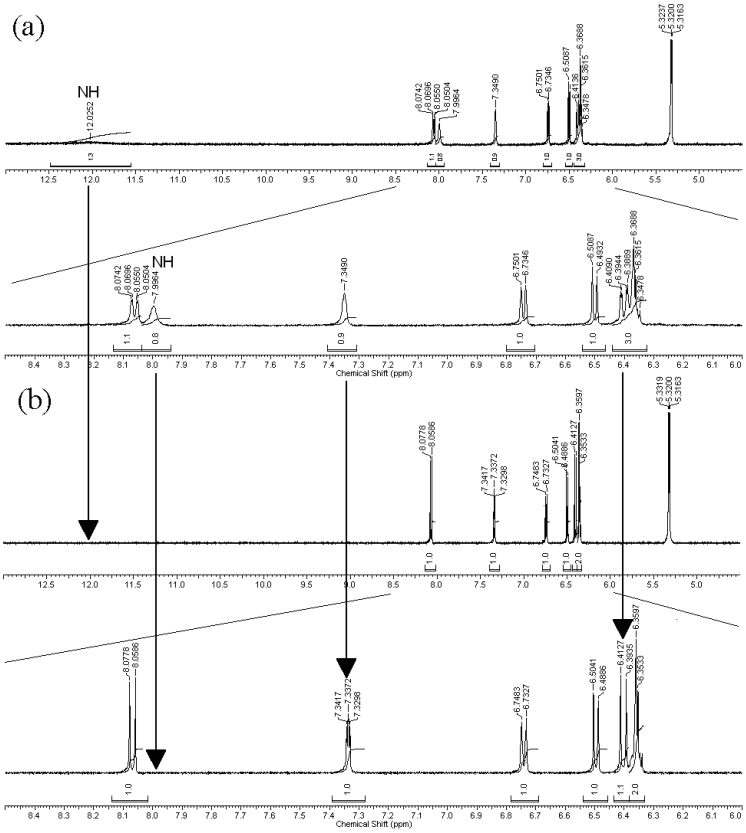

Fig. S11 ^{1}H NMR (300 MHz) spectra change for **5** in CD_2Cl_2; (a) before and (b) after adding D_2O.

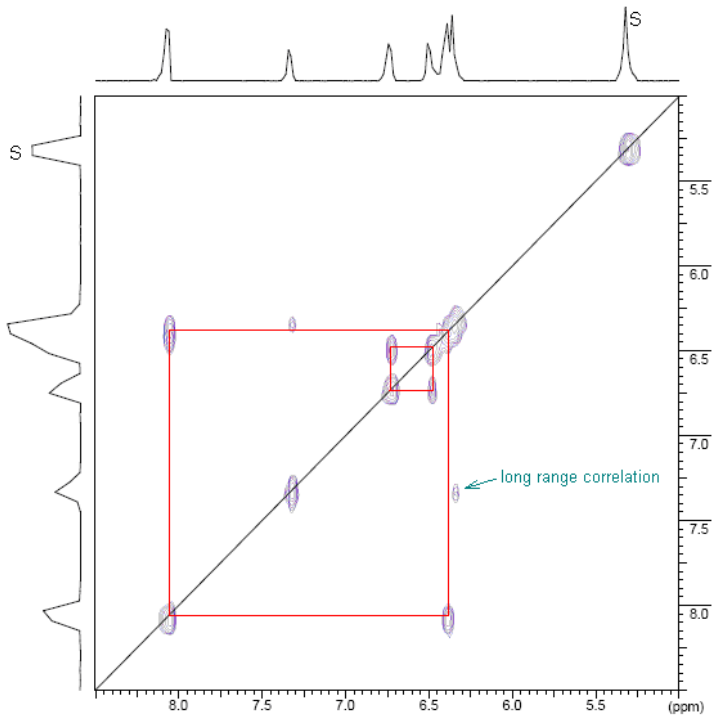

Fig. S12 HH COSY NMR (F1 and F2: 300 MHz) spectrum of **5.**

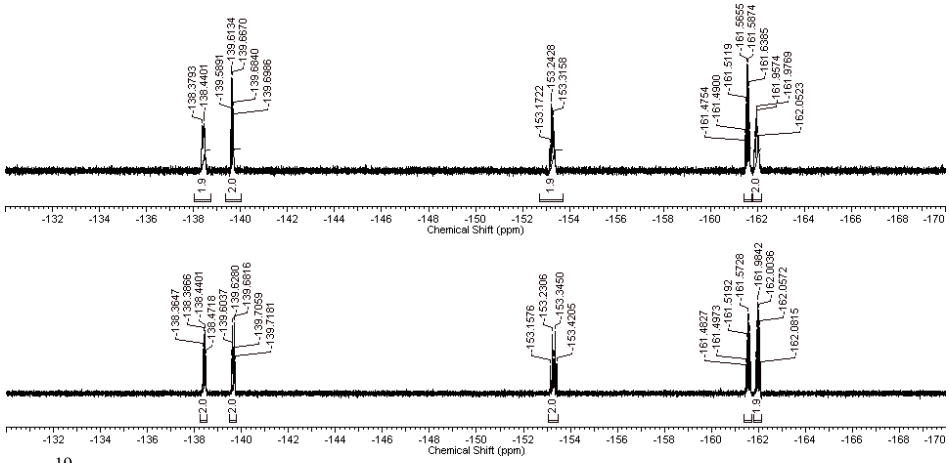

Fig. S13 ^{19}F NMR (282.4 MHz) spectra of **5** in CD_2Cl_2; before (a) and after (b) adding D_2O.

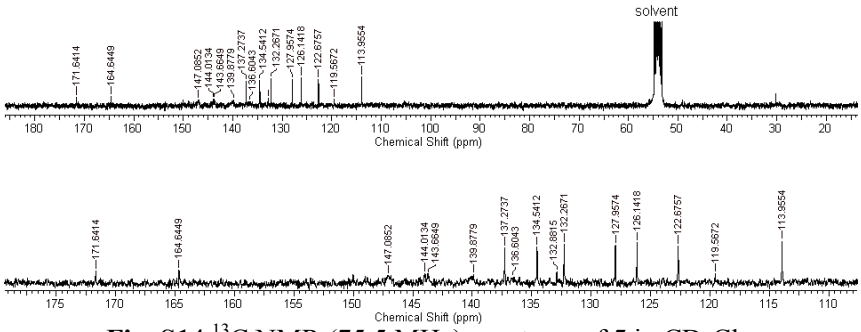

Fig. S14 ^{13}C NMR (75.5 MHz) spectrum of **5** in CD_2Cl_2.

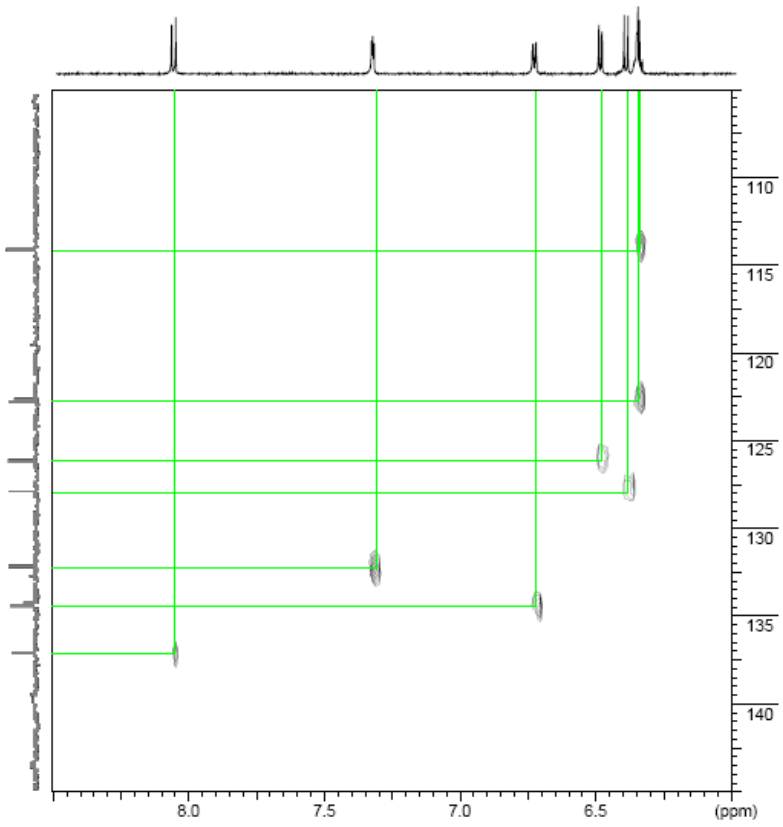

Fig. S15 HMQC NMR (F1: 300 MHz and F2: 75.5 MHz) spectrum of **5.**

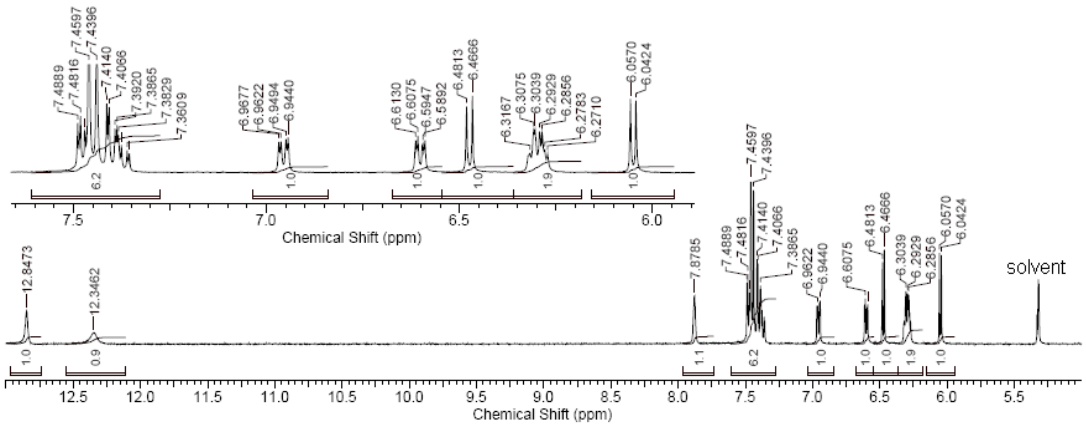

Fig. S16 ^{1}H NMR (300 MHz) spectrum of **7** in CD_2Cl_2.

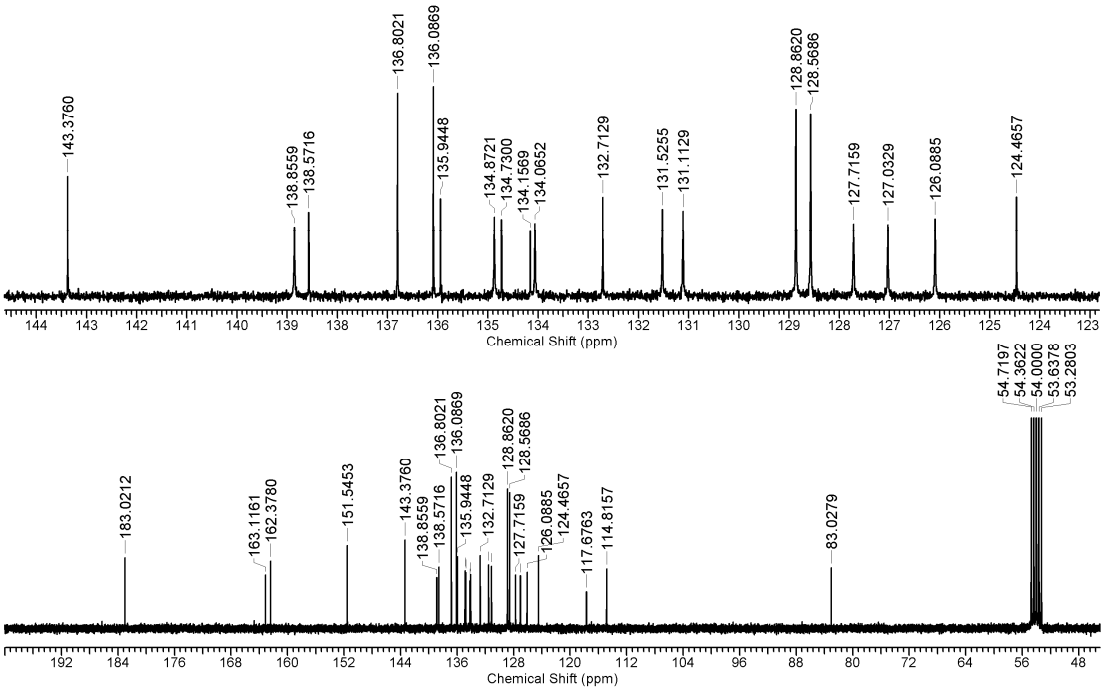

Fig. S17 ^{13}C NMR (100 MHz) of 7 in CD_2Cl_2.

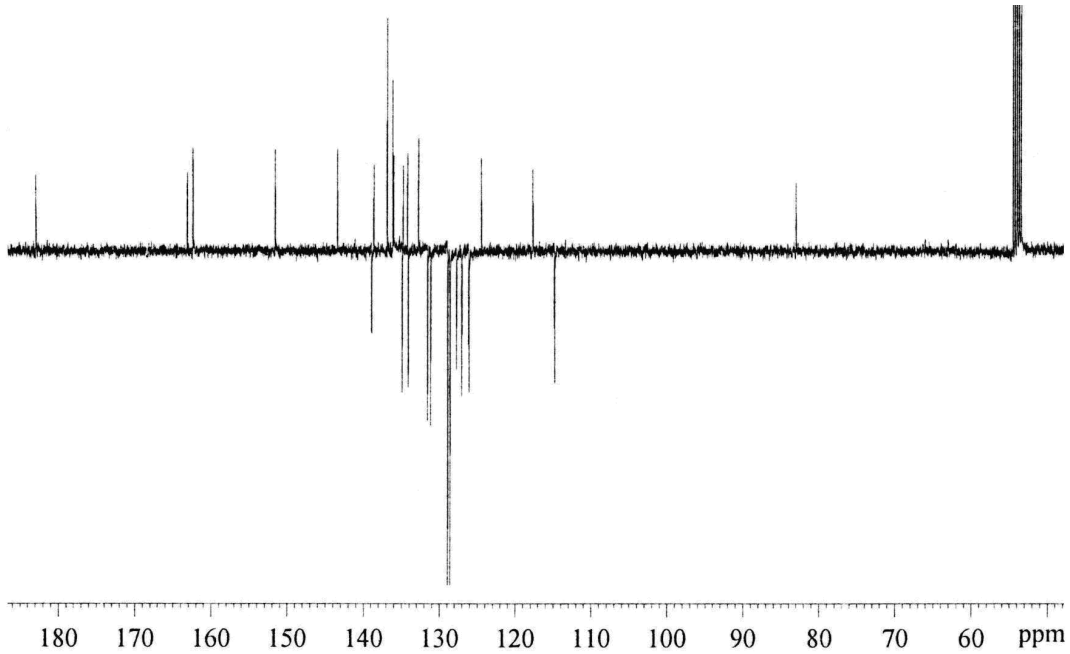

Fig. S18 ^{13}C APT (45 deg, 100 MHz) spectrum of **7** in CD_2Cl_2.

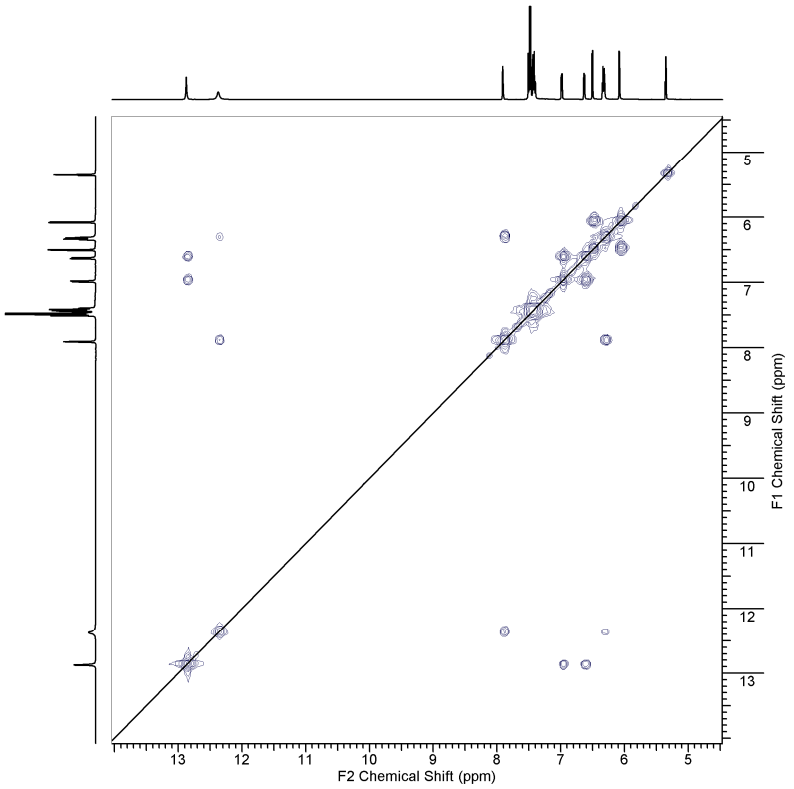

Fig. S19 HH COSY (F1 and F2: 400 MHz) spectrum of **7** in CD_2Cl_2.

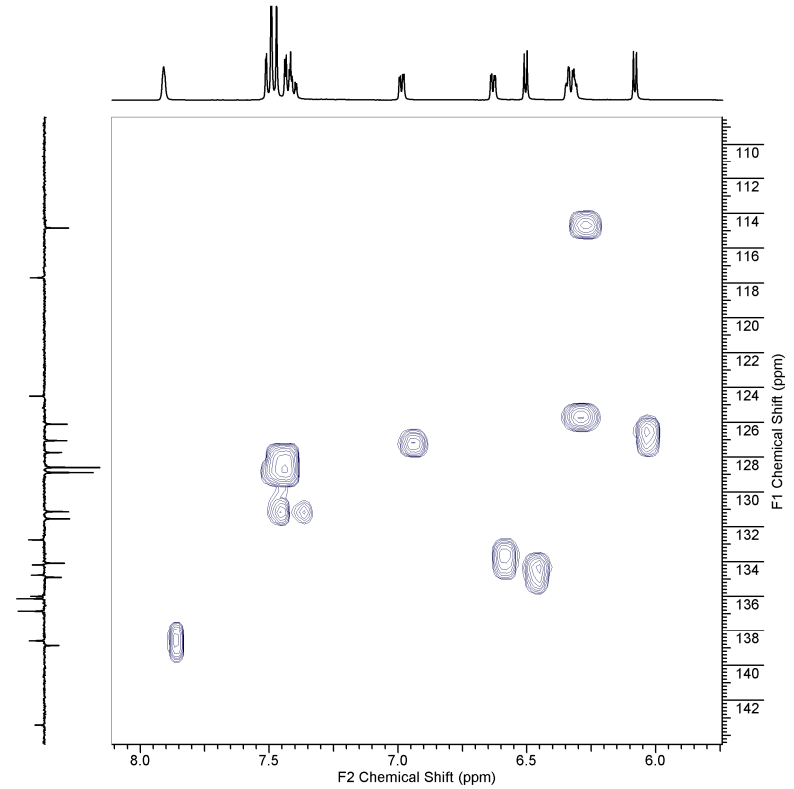

Fig. S20 HMQC (F1: 400 MHz and F2: 100 MHz) spectrum of **7** in CD_2Cl_2.

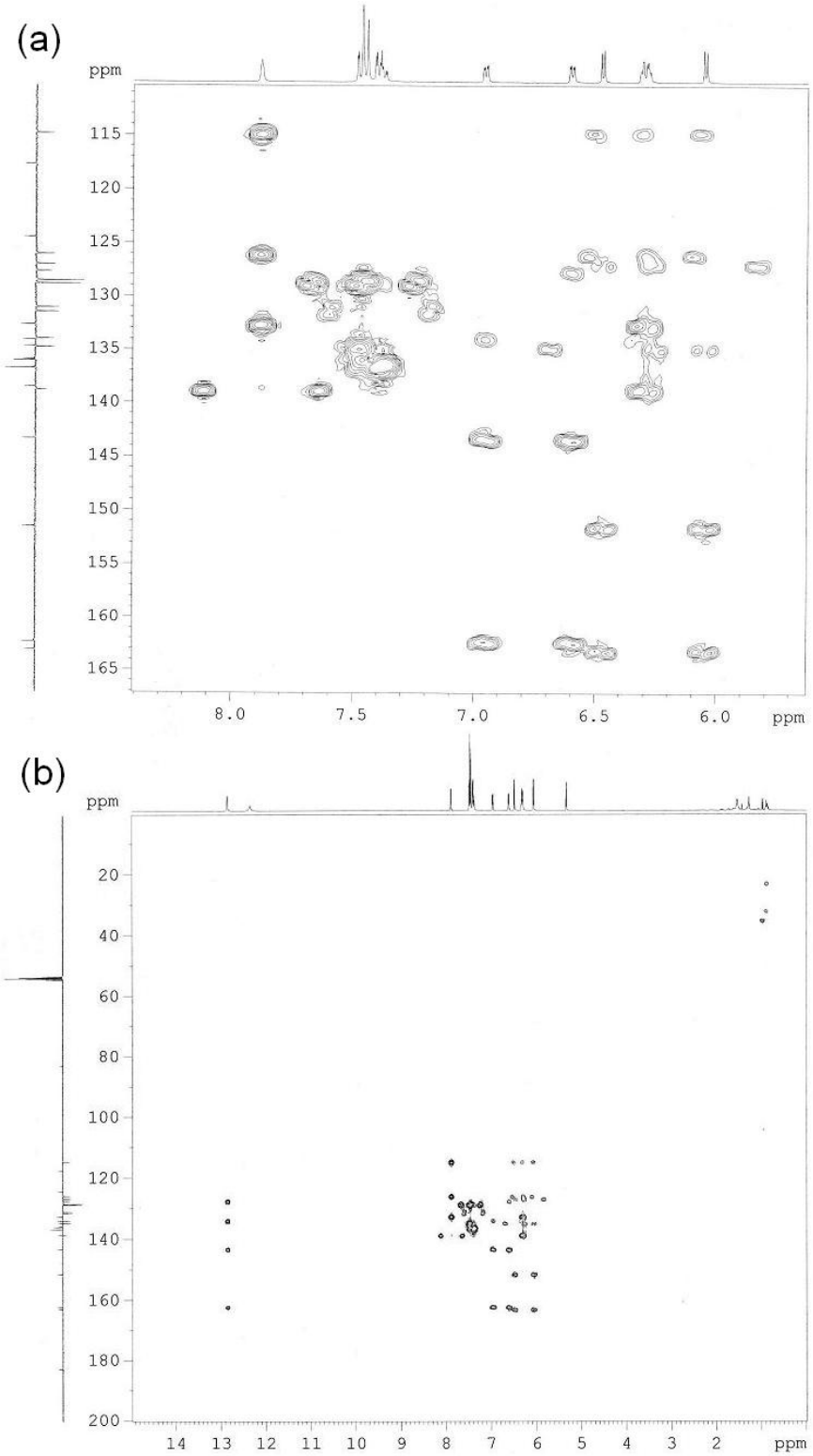

Fig. S21 HMBC (F1: 400 MHz and F2: 100 MHz) spectrum of **7** in CD_2Cl_2; zoom in (a) and out (b).

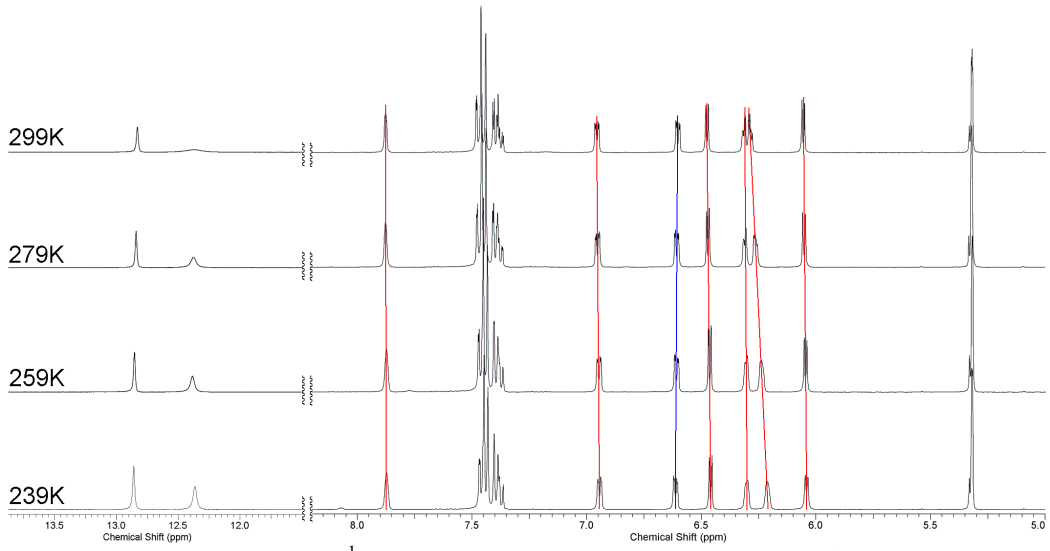

Fig. S22 VT ^{1}H NMR (400 MHz) spectra of **7** in CD_2Cl_2.

2. X-ray crystallographic data.

For Compound 3

The crystals of $C_{26}H_9F_{10}N_3O{\cdot}½CH_2Cl_2$ were grown by diffusion of hexane into a CH_2Cl_2 solution of **3**. A blue prism having dimensions approximately 0.05 x 0.10 x 0.30 mm was mounted on a glass fiber. The data were collected at a temperature of -100.0 ± 0.1ºC to a maximum 2θ value of 50.2º. Data were collected in a series of ϕ and ω scans in 0.50º oscillations with 10.0 second exposures. The crystal-to-detector distance was 36.00 mm.

Of the 16039 reflections that were collected, 8018 were unique; equivalent reflections were merged. Data were collected and integrated using the Bruker SAINT[S1] software package. The linear absorption coefficient, μ, for Mo-Kα radiation is 2.69 cm^{-1}. Data were corrected for absorption effects using the multi-scan technique (SADABS[S2]), with minimum and maximum transmission coefficients of 0.681 and 0.987, respectively. The data were corrected for Lorentz and polarization effects.

The structure was solved by direct methods[S3]. Refinements converged around R1 (I > 2sig(I)) = 0.16, and PLATON/ROTAX program was used to investigate possible twinning. An HKLF5 dataset containing unique data from one component and overlapping data from the second, reflecting 180 degree rotation about the 1 0 0 reciprocal axis lowered R1 (I > 2sig(I)) to 0.084. The material crystallizes with one, disordered half-molecule of CH_2Cl_2 in the asymmetric unit. All non-hydrogen atoms were refined anisotropically, while all hydrogen atoms except H1N and H3N were placed in calculated positions and not refined. All N-H hydrogen atoms were located in difference maps and refined isotropically. The final cycle of full-matrix least-squares refinement[S4] on F^2 was based on 13171 reflections and 399 variable parameters (R1 = 0.162 and wR2 = 0.246).

The standard deviation of an observation of unit weight[S5] was 1.07. The weighting scheme was based on counting statistics. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.71 and -0.64 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[S6]. Anomalous dispersion effects were included in Fcalc[S7]; the values for Δf' and Δf'' were those of Creagh and McAuley[S8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[S9]. All refinements were performed using the SHELXTL[S10] crystallographic software package of Bruker-AXS.

References

S1. **SAINT.** Version 7.03A. Bruker AXS Inc., Madison, Wisconsin, USA. (1997-2003)
S2. **SADABS.** Bruker Nonius area detector scaling and absorption correction – V2.10, Bruker AXS Inc., Madison, Wisconsin, USA. (2003)
S3. **SIR97.** A. Altomare, M. C. Burla, M. Camalli, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori and R. Spagna, *J. Appl. Cryst.*, 1999, **32**, 115-119.
S4. Least Squares function minimized: $\Sigma w(F_o^2-F_c^2)^2$
S5. Standard deviation of an observation of unit weight: $[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$
where: N_o = number of observations, N_v = number of variables
S6. D. T. Cromer, J. T. Waber, "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
S7. Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).
S8. D. C. Creagh, W.J. McAuley, "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
S9. D. C. Creagh, J. H. Hubbell, "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).
S10. **SHELXTL.** Version 5.1, Bruker AXS Inc., Madison, Wisconsin, USA. (1997).

A. Crystal Data

Empirical Formula	$C_{26.5}H_{10}N_3F_{10}OCl$
Formula Weight	611.83
Crystal Color, Habit	blue, tablet
Crystal Dimensions	0.05 X 0.10 X 0.30 mm
Crystal System	C-centred
Lattice Type	monoclinic
Lattice Parameters	a = 29.240(1) Å b = 23.151(9) Å c = 7.016(2) Å α = 90.0º β = 93.06(1)º γ = 90.0º V = 4743(3) Å^3
Space Group	*C* 2/*c* (#15)
Z value	8
D_{calc}	1.714 g/cm^3
F_{000}	2440.00
μ(MoKα)	2.69

B. Intensity Measurements

Diffractometer	Bruker X8 APEX II
Radiation	MoKα (λ = 0.71073 Å) graphite monochromated
Data Images	697 exposures @ 60.0 seconds
Detector Position	36.00 mm
$2\theta_{max}$	50.2º
No. of Reflections Measured	Total: 16039 Unique: 8018 (R_{int} = 0.000)
Corrections	Absorption (T_{min} = 0.681, T_{max}= 0.987) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w=1/(\sigma^2(Fo^2)+(0.0479P)^2+ 40.18398P)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>0.00σ(I))	13171
No. Variables	399
Reflection/Parameter Ratio	33.01
Residuals (refined on F^2, all data): R1; wR2	0.162; 0.246
Goodness of Fit Indicator	1.20
No. Observations (I>2.00σ(I))	7238
Residuals (refined on F): R1; wR2	0.084; 0.219
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.71 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.64 $e^-/Å^3$

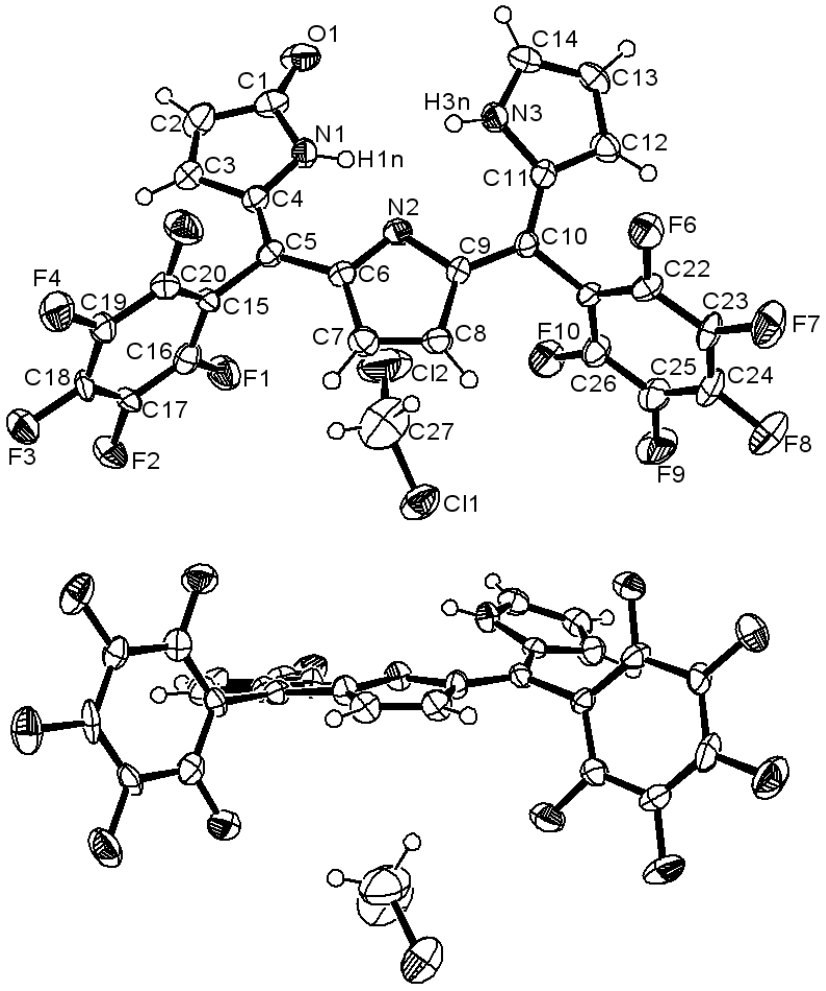

Fig. S23 Crystal structure of **3** with numbering; top-view (above), side-view (below). Thermal ellipsoids are scaled to the 50 % probability level.

For Compound 7

The crystals were grown by diffusion of *iso*-propyl alcohol into a CH_2Cl_2 solution of **7**. Single crystal X-ray data were collected from a sample of the material. The crystal was a particularly weak diffractor, with recognizable data out only as far as 37.5 degrees 2-theta (Mo radiation). Although the data was weak and the initial structure was obtained and isotropic refinements could be carried out (see the CIF data below). Subsequent anisotropic refinements were, predictably, unreliable, and they resulted in unreasonable geometries and anisotropic displacement parameters. The isotropic model is, however, sufficient to unambiguously determine the three-dimensional connectivity of the material. Based on the structure, we calculated from the beginning and found that even the data of the NMR and optical spectra matched reasonably well with the observed ones.

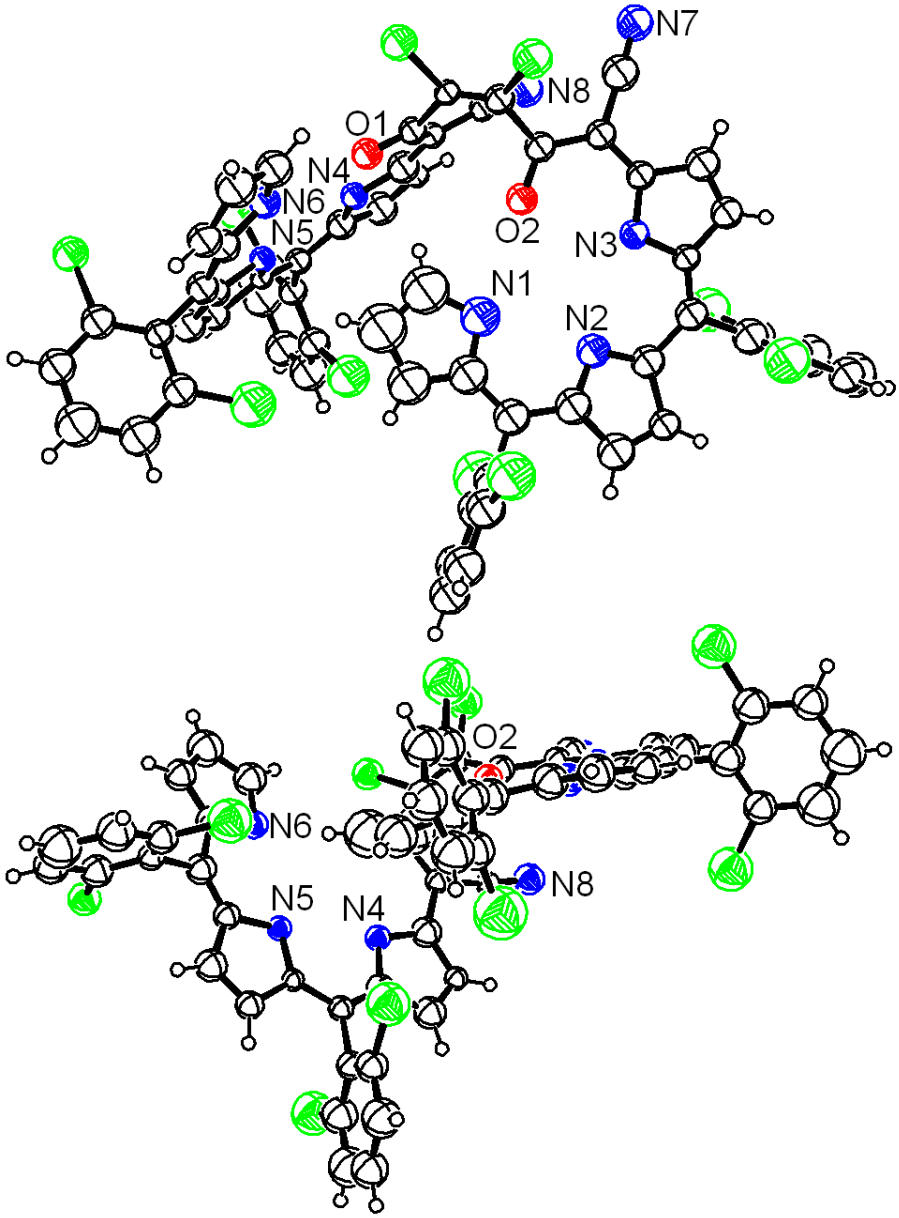

Fig. S24 Crystal structure (isotrophic model) of **7**; top-view (above), side-view (below). Thermal ellipsoids are scaled to the 50 % probability level.

CIF data

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 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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 '-y, x-y, z+2/3'
 '-x+y, -x, z+1/3'

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_cell_angle_gamma                 120.00
_cell_volume                      5339.7(6)
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_cell_measurement_temperature     173(2)
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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C13 C 0.999(2) -0.0190(19) 1.1300(5) 0.060(5) Uiso 1 1 d . . .
H13 H 0.9918 0.0145 1.1499 0.072 Uiso 1 1 calc R . .
C14 C 0.9632(16) 0.0093(17) 1.0985(4) 0.042(4) Uiso 1 1 d . . .
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C16 C 0.8756(17) 0.1080(18) 1.0621(4) 0.046(5) Uiso 1 1 d . . .
C17 C 0.7985(16) 0.1757(17) 1.0588(4) 0.044(4) Uiso 1 1 d . . .
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C19 C 0.9183(16) 0.3012(16) 1.0102(4) 0.039(4) Uiso 1 1 d . . .
C20 C 1.0413(15) 0.3543(15) 1.0170(3) 0.035(4) Uiso 1 1 d . . .
C21 C 1.1424(19) 0.4028(19) 0.9930(4) 0.056(5) Uiso 1 1 d . . .
C22 C 1.2723(15) 0.4590(15) 0.9956(4) 0.033(4) Uiso 1 1 d . . .
H22 H 1.3192 0.4803 1.0150 0.039 Uiso 1 1 calc R . .
C23 C 1.320(2) 0.4777(19) 0.9671(5) 0.063(5) Uiso 1 1 d . . .
H23 H 1.4079 0.5165 0.9622 0.075 Uiso 1 1 calc R . .
C24 C 1.2197(17) 0.4311(18) 0.9443(4) 0.049(5) Uiso 1 1 d . . .
C25 C 1.2249(16) 0.4237(16) 0.9120(4) 0.037(4) Uiso 1 1 d . . .
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C26 C 1.1203(14) 0.3703(14) 0.8913(3) 0.027(3) Uiso 1 1 d . . .
C27 C 1.1334(17) 0.3700(16) 0.8569(4) 0.045(4) Uiso 1 1 d . . .
H27 H 1.2108 0.4077 0.8451 0.054 Uiso 1 1 calc R . .
C28 C 1.0170(19) 0.3073(18) 0.8450(5) 0.058(5) Uiso 1 1 d . . .
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C29 C 0.9258(16) 0.2707(16) 0.8703(4) 0.041(4) Uiso 1 1 d . . .
C30 C 0.8037(18) 0.2110(18) 0.8690(4) 0.052(5) Uiso 1 1 d . . .
N6 N 0.7451(13) 0.2350(14) 0.9241(3) 0.047(4) Uiso 1 1 d D . .
C31 C 0.7057(17) 0.1866(18) 0.8935(4) 0.052(5) Uiso 1 1 d D . .
C32 C 0.5778(17) 0.1299(18) 0.8900(4) 0.057(5) Uiso 1 1 d D . .
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C33 C 0.532(2) 0.145(2) 0.9201(5) 0.080(7) Uiso 1 1 d D . .
H33 H 0.4465 0.1190 0.9255 0.096 Uiso 1 1 calc R . .
C34 C 0.6371(18) 0.204(2) 0.9399(4) 0.066(6) Uiso 1 1 d D . .
H34 H 0.6334 0.2216 0.9619 0.079 Uiso 1 1 calc R . .
C35 C 0.9789(14) -0.2939(13) 0.9363(3) 0.066(6) Uiso 1 1 d G . .
C36 C 1.0883(11) -0.2602(13) 0.9187(3) 0.076(6) Uiso 1 1 d G . .
C37 C 1.0820(12) -0.3294(15) 0.8916(3) 0.089(7) Uiso 1 1 d G . .
H37 H 1.1568 -0.3064 0.8796 0.107 Uiso 1 1 calc R . .
C38 C 0.9663(15) -0.4324(14) 0.8822(3) 0.081(7) Uiso 1 1 d G . .
H38 H 0.9620 -0.4797 0.8637 0.097 Uiso 1 1 calc R . .
C39 C 0.8569(12) -0.4661(12) 0.8998(3) 0.083(7) Uiso 1 1 d G . .
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C41 C 1.1253(13) -0.2675(13) 1.0980(3) 0.068(6) Uiso 1 1 d G . .
C42 C 1.0490(10) -0.3879(13) 1.1109(3) 0.072(6) Uiso 1 1 d G . .
C43 C 1.1035(14) -0.4448(11) 1.1288(3) 0.066(6) Uiso 1 1 d G . .
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H44 H 1.2716 -0.4204 1.1459 0.132 Uiso 1 1 calc R . .
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C46 C 1.2561(13) -0.2041(11) 1.1029(3) 0.059(5) Uiso 1 1 d G . .
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C48 C 1.0958(15) 0.3770(15) 1.0486(4) 0.036(4) Uiso 1 1 d . . .
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C2 C 0.912(3) 0.022(3) 0.9350(6) 0.128(11) Uiso 1 1 d D . .
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C3 C 0.958(3) -0.065(3) 0.9296(5) 0.098(8) Uiso 1 1 d D . .
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C4 C 0.967(2) -0.111(2) 0.9587(5) 0.079(7) Uiso 1 1 d D . .
N2 N 1.0112(15) -0.1840(15) 1.0222(4) 0.058(4) Uiso 1 1 d . . .
N3 N 0.9834(13) -0.0635(13) 1.0774(3) 0.043(4) Uiso 1 1 d . . .
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N7 N 0.9077(16) 0.2179(16) 1.1432(4) 0.066(5) Uiso 1 1 d . . .
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 into account individually in the estimation of esds in distances, angles
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 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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C6 C7 1.46(3) . ?
C7 C8 1.32(3) . ?
C7 H7 0.9500 . ?
C8 C9 1.51(3) . ?
C8 H8 0.9500 . ?
C9 N2 1.34(2) . ?
C9 C10 1.50(3) . ?
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C12 H12 0.9500 . ?
C13 C14 1.48(2) . ?
C13 H13 0.9500 . ?
C14 C15 1.34(2) . ?
C14 N3 1.36(2) . ?
C15 C16 1.46(2) . ?
C15 C47 1.48(3) . ?
C16 O2 1.256(19) . ?
C16 C17 1.53(3) . ?
C17 C18 1.24(2) . ?
C17 Cl5 1.720(18) . ?
C18 C19 1.57(2) . ?
C18 Cl6 1.727(17) . ?
C19 O1 1.236(18) . ?
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C20 C21 1.46(2) . ?
C20 C48 1.44(2) . ?
C21 N4 1.38(2) . ?
C21 C22 1.37(2) . ?
C22 C23 1.30(2) . ?
C22 H22 0.9500 . ?
C23 C24 1.42(3) . ?
C23 H23 0.9500 . ?
C24 N4 1.38(2) . ?
C24 C25 1.36(2) . ?
C25 C26 1.40(2) . ?
C25 C49 1.526(19) . ?
C26 N5 1.348(19) . ?
C26 C27 1.45(2) . ?
C27 C28 1.32(3) . ?
C27 H27 0.9500 . ?
C28 C29 1.43(3) . ?
C28 H28 0.9500 . ?
C29 C30 1.28(2) . ?
C29 N5 1.47(2) . ?
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C30 C31 1.48(3) . ?
N6 C31 1.392(19) . ?
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C33 C34 1.38(2) . ?
C33 H33 0.9500 . ?
C34 H34 0.9500 . ?
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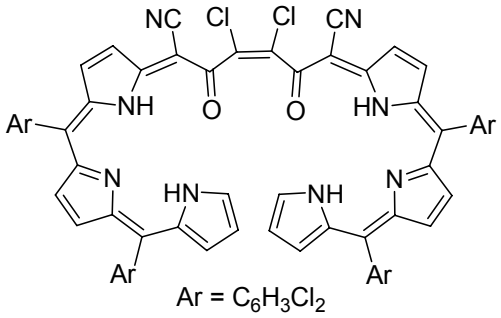
3. Computational data.



3-1. General

Gaussian 03[S11] was used for all calculations. Geometry optimizations were performed using the popular Kohn-Sham method B3LYP[S12a,b] with Pople's 6-31G(d,p)[S13a-c] basis set. Solvent effects were considered by employing an integral equation formalism polarizable continuum model (PCM)[S14] appropriate for methylene chloride with the MP2 energies. The ^{1}H and ^{13}C NMR chemical shifts for the molecules were calculated with the gauge-independent atomic orbital (GIAO)[S15] method at the B3LYP/6-31+G(df,p) level of theory (with PCM solvent effects) using the B3LYP/6-31G** optimized geometries. Time-dependent density functional theory (TDDFT)[S16] with B3LYP was utilized to calculate the optical spectra.

References

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3-2. Geometry Optimization

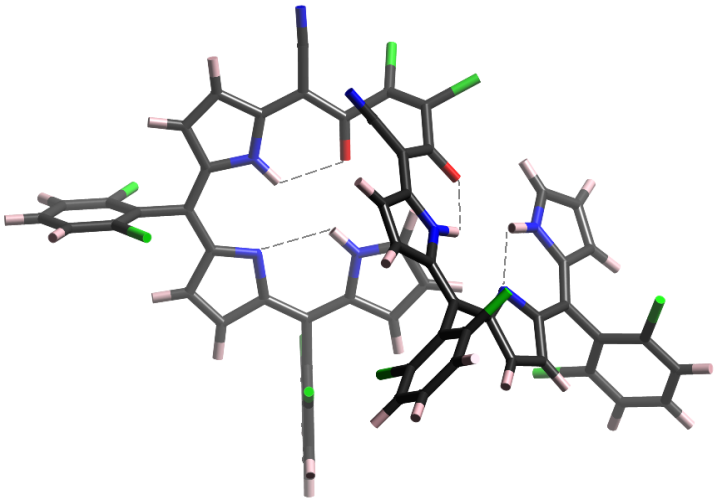


Fig. S25 Optimized structure of **7** in C_1 symmetry. Hydrogen bonds are indicated with dashed lines.

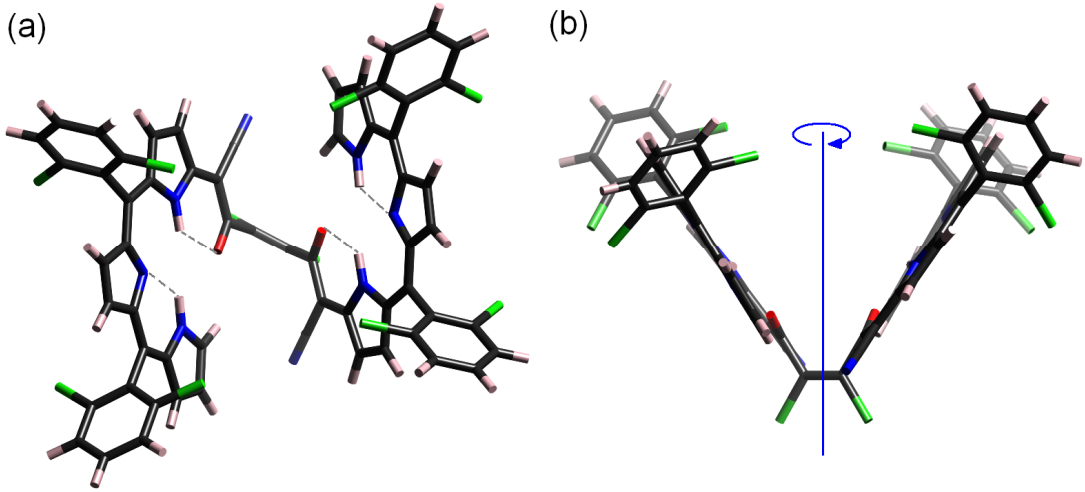
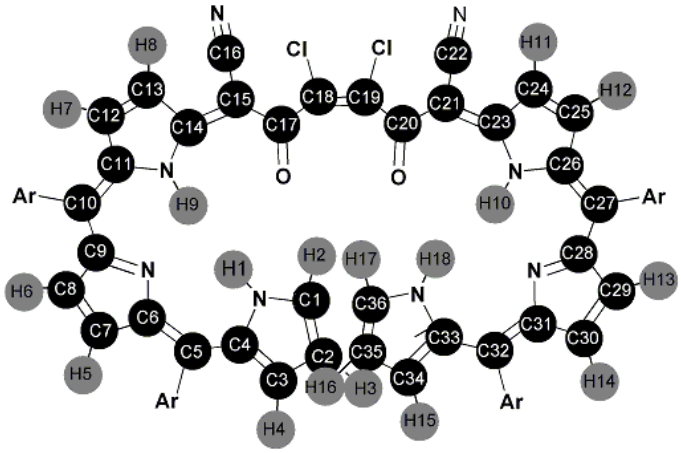


Fig. S26 Optimized structure of **7** in C_2 symmetry. Hydrogen bonds are indicated with dashed lines; (a) top-view and (b) side-view.

Table S1. Selected NMR data comparison of observed and calculated data for **7**; CD_2Cl_2 was used as a solvent.



Atomic number	Calculation data (Structure C)		Observed data
	C_1 symmetry	C_2 symmetry	
C1, C36	140.73, 139.28	141.35	138.86
C2, C35	114.20, 114.38	114.68	114.82
C3, C34	126.53, 125.63	127.03	126.09
C4, C33	132.98, 133.78	133.01	132.71
C5, C32	140.48, 137.20	139.75	138.57
C6, C31	151.14, 153.31	152.00	151.55
C7, C30	131.58, 131.53	130.40	134.87
C8, C29	124.93, 125.78	123.90	127.03
C9, C28	160.57, 161.61	158.96	163.12
C10, C27	130.90, 128.34	129.14	134.16
C11, C26	140.28, 142.05	140.09	143.38
C12, C25	131.83, 132.66	131.70	134.07
C13, C24	123.56, 125.31	124.26	127.72
C14, C23	159.48, 159.51	159.97	162.38
C15, C22	83.94, 84.28	82.88	83.03
C16, C21	117.16, 115.75	116.35	117.68
C17, C20	181.33,181.84	181.13	183.02
C18, C19	143.76, 142.42	140.37	135.94
H1, H18	12.40, 13.12	12.67	12.35
H9, H10	12.56, 12.92	12.62	12.85

3-3. Geometric Parameters obtained from optimizations (Table S2).

#	Atom	X	Y	Z
1	C	3.25944	-0.085908	3.832845
2	C	-2.418554	0.939219	3.066748
3	C	-1.018806	1.083014	3.380874
4	C	-3.25944	0.085908	3.832845
5	C	-0.48958	0.458545	4.658576
6	C	0.48958	-0.458545	4.658576
7	C	1.018806	-1.083014	3.380874
8	C	2.418554	-0.939219	3.066748
9	N	3.962923	0.630756	4.426783
10	N	-3.962923	-0.630756	4.426783
11	O	-0.223292	1.71405	2.665734
12	O	0.223292	-1.71405	2.665734
13	Cl	-1.155338	1.119818	6.128853
14	Cl	1.155338	-1.119818	6.128853
15	C	-4.375472	1.57455	1.540589
16	C	-3.189166	2.925562	0.142343
17	C	-4.491745	2.375067	0.445548
18	H	-5.148703	1.01118	2.041947
19	H	-5.389006	2.588391	-0.116372
20	C	4.375472	-1.57455	1.540589
21	C	3.189166	-2.925562	0.142343
22	C	4.491745	-2.375067	0.445548
23	H	5.148703	-1.01118	2.041947
24	H	5.389006	-2.588391	-0.116372
25	C	-2.990582	1.598955	1.962535
26	C	2.990582	-1.598955	1.962535
27	N	-2.32691	2.409506	1.099223
28	N	2.32691	-2.409506	1.099223
29	H	-1.330324	2.586153	1.126103
30	H	1.330324	-2.586153	1.126103
31	C	2.835668	-3.787971	-0.870871
32	C	-2.835668	3.787971	-0.870871
33	C	1.205703	-5.178018	-2.238382
34	C	-0.114399	-5.486122	-2.125954
35	H	1.894973	-5.4916	-3.008791
36	C	-0.585386	-4.828907	-0.924527
37	H	-0.707118	-6.103959	-2.784818
38	C	-1.205703	5.178018	-2.238382
39	C	0.114399	5.486122	-2.125954
40	H	-1.894973	5.4916	-3.008791
41	C	0.585386	4.828907	-0.924527
42	H	0.707118	6.103959	-2.784818
43	C	1.519309	-4.326323	-1.107035
44	C	-1.519309	4.326323	-1.107035
45	N	-0.438772	4.118697	-0.324354
46	N	0.438772	-4.118697	-0.324354
47	C	-1.898637	-4.934449	-0.43987
48	C	1.898637	4.934449	-0.43987
49	C	-3.735027	-4.451321	1.26738
50	C	-3.765642	-3.724552	2.453885
51	H	-4.551994	-4.988583	0.808821
52	C	-2.476751	-3.206239	2.649864
53	H	-0.719734	-3.348929	1.499922
54	H	-4.605204	-3.560719	3.112544
55	H	-2.089828	-2.579737	3.43687
56	C	3.735027	4.451321	1.26738
57	C	3.765642	3.724552	2.453885
58	H	4.551994	4.988583	0.808821
59	C	2.476751	3.206239	2.649864
60	H	0.719734	3.348929	1.499922
61	H	4.605204	3.560719	3.112544
62	H	2.089828	2.579737	3.43687
63	N	1.691883	3.598124	1.630984
64	N	-1.691883	-3.598124	1.630984
65	C	-2.419167	-4.370941	0.739652
66	C	2.419167	4.370941	0.739652
67	C	3.928316	-4.21433	-1.805545
68	C	4.684518	-5.37804	-1.573352
69	C	4.24652	-3.482791	-2.964023
70	C	5.700353	-5.791351	-2.434033
71	C	5.257751	-3.876422	-3.839883
72	C	5.982479	-5.033901	-3.568015
73	H	6.256535	-6.6942	-2.210887
74	H	5.466095	-3.278693	-4.719397
75	H	6.771366	-5.347857	-4.244077
76	C	-3.928316	4.21433	-1.805545
77	C	-4.684518	5.37804	-1.573352
78	C	-4.24652	3.482791	-2.964023
79	C	-5.700353	5.791351	-2.434033
80	C	-5.257751	3.876422	-3.839883
81	C	-5.982479	5.033901	-3.568015
82	H	-6.256535	6.6942	-2.210887
83	H	-5.466095	3.278693	-4.719397
84	H	-6.771366	5.347857	-4.244077
85	Cl	4.358795	-6.358767	-0.15501
86	Cl	3.351641	-2.021748	-3.345565
87	Cl	-4.358795	6.358767	-0.15501
88	Cl	-3.351641	2.021748	-3.345565
89	C	-2.87255	-5.738792	-1.252502
90	C	-3.056727	-7.116045	-1.038116
91	C	-3.654334	-5.148424	-2.260924
92	C	-3.962923	-7.86922	-1.78366
93	C	-4.566581	-5.881876	-3.018466
94	C	-4.716114	-7.244573	-2.774288
95	H	-4.069582	-8.928805	-1.583259
96	H	-5.1467	-5.383536	-3.78618
97	H	-5.423875	-7.823121	-3.359347
98	C	2.87255	5.738792	-1.252502
99	C	3.056727	7.116045	-1.038116

100	C	3.654334	5.148424	-2.260924	107	Cl	-3.49509	-3.433794	-2.597967
101	C	3.962923	7.86922	-1.78366	108	Cl	-2.12441	-7.939705	0.199349
102	C	4.566581	5.881876	-3.018466	109	Cl	3.49509	3.433794	-2.597967
103	C	4.716114	7.244573	-2.774288	110	Cl	2.12441	7.939705	0.199349
104	H	4.069582	8.928805	-1.583259					
105	H	5.1467	5.383536	-3.78618					
106	H	5.423875	7.823121	-3.359347					

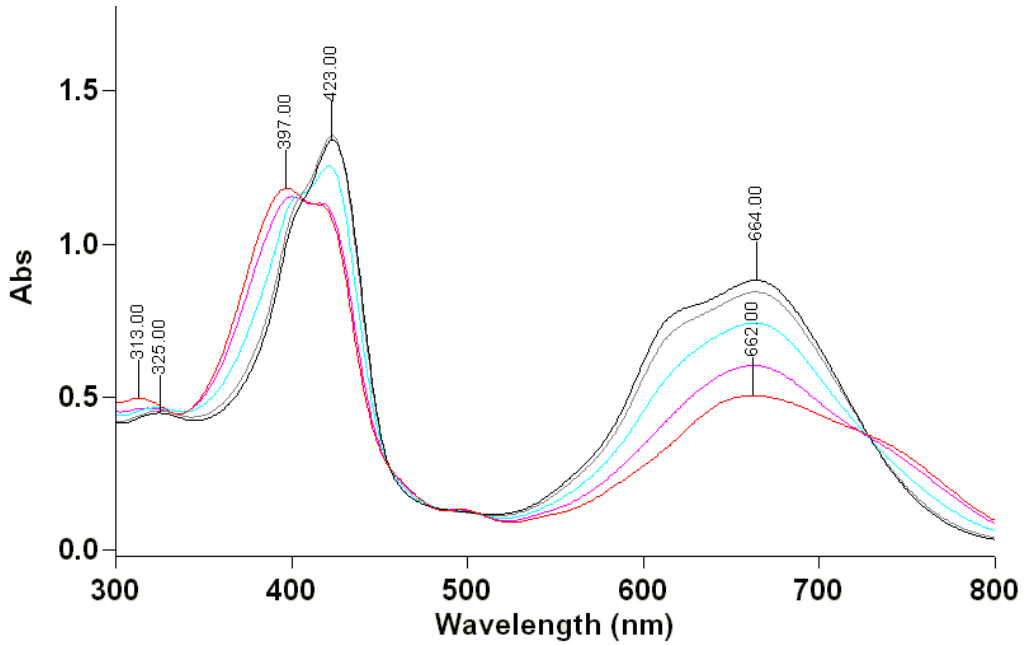


Fig. S27 Optical spetrum change of **7** in CH_2Cl_2 by addition of TFA.