

Supporting Information

Planarization of tetracarboxamides. Tuning the self-assembly of polycyclic aromatic hydrocarbons

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1. Supplementary Figures and Tables

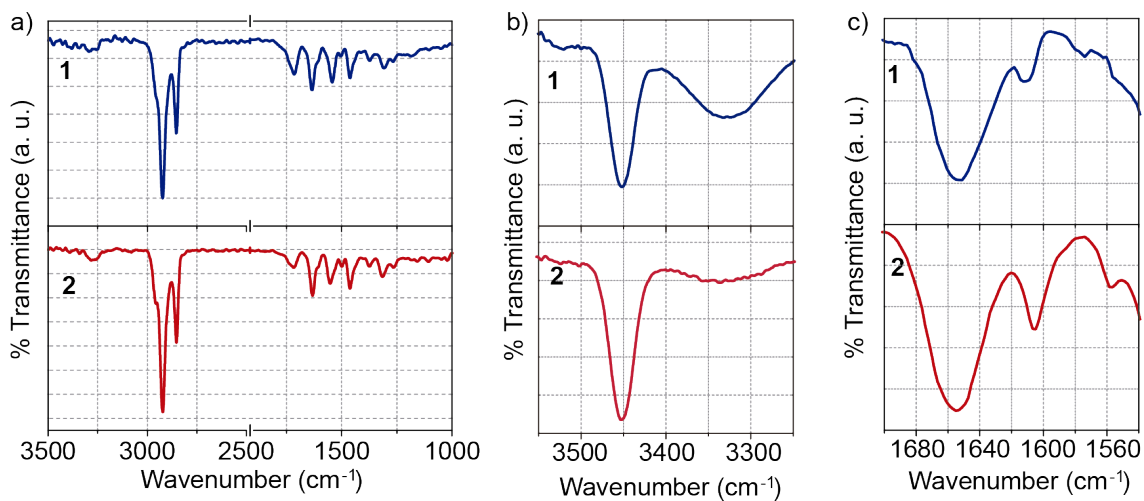


Figure S1. FTIR spectra of **1** and **2** in film (a) and in CHCl₃ at 8 mM (b and c). The FTIR spectra in (b) and (c) show the region on which the stretching N-H and Amide I bands are observed.

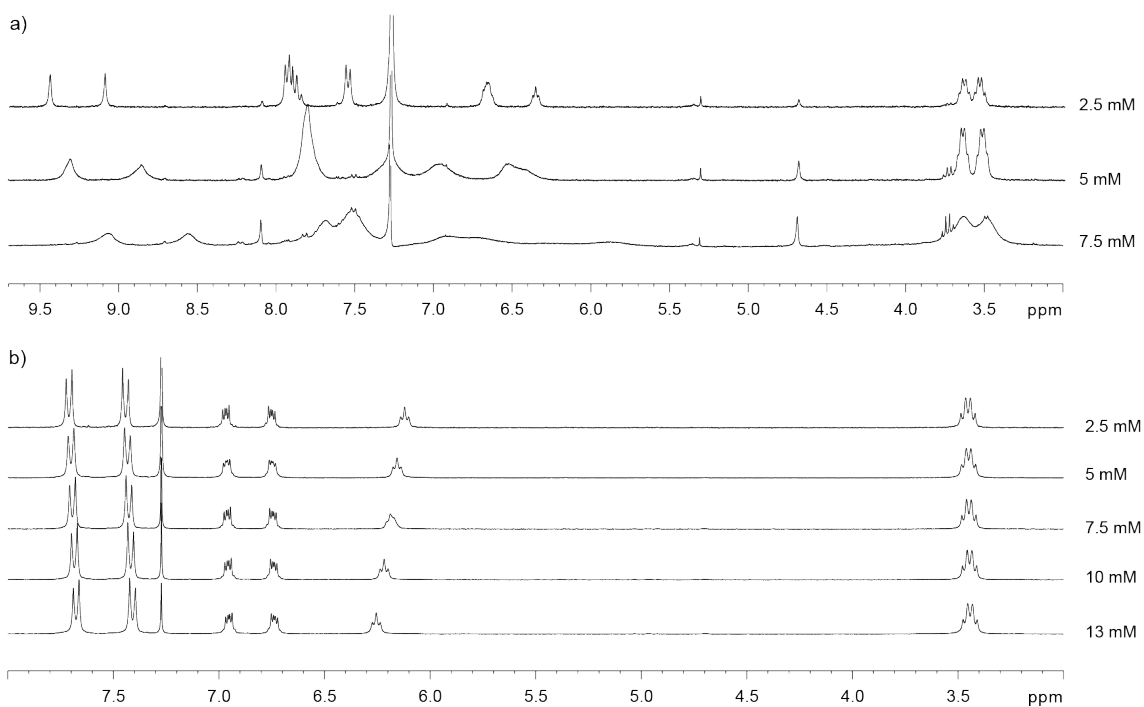


Figure S2. Partial ¹H NMR spectra of **1** (a) and **2** (b) in CDCl₃ at different concentrations (300 MHz, 303 K).

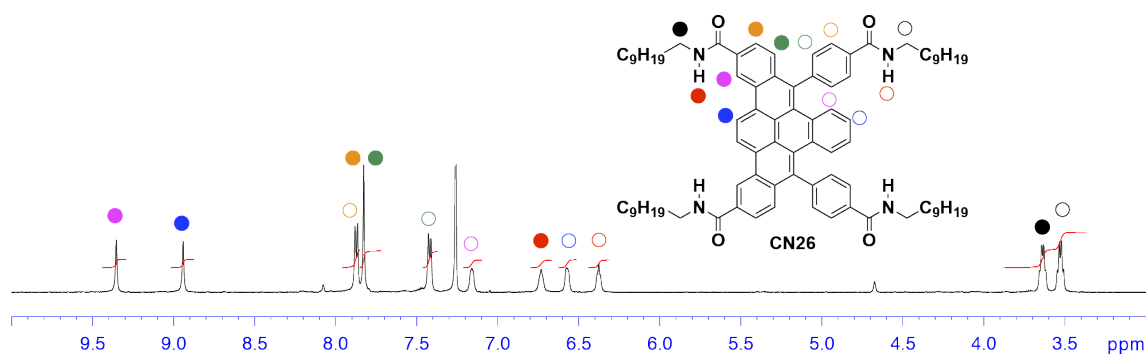


Figure S3. ^1H NMR spectrum of **1** (CDCl_3 , 500 MHz, 328 K).

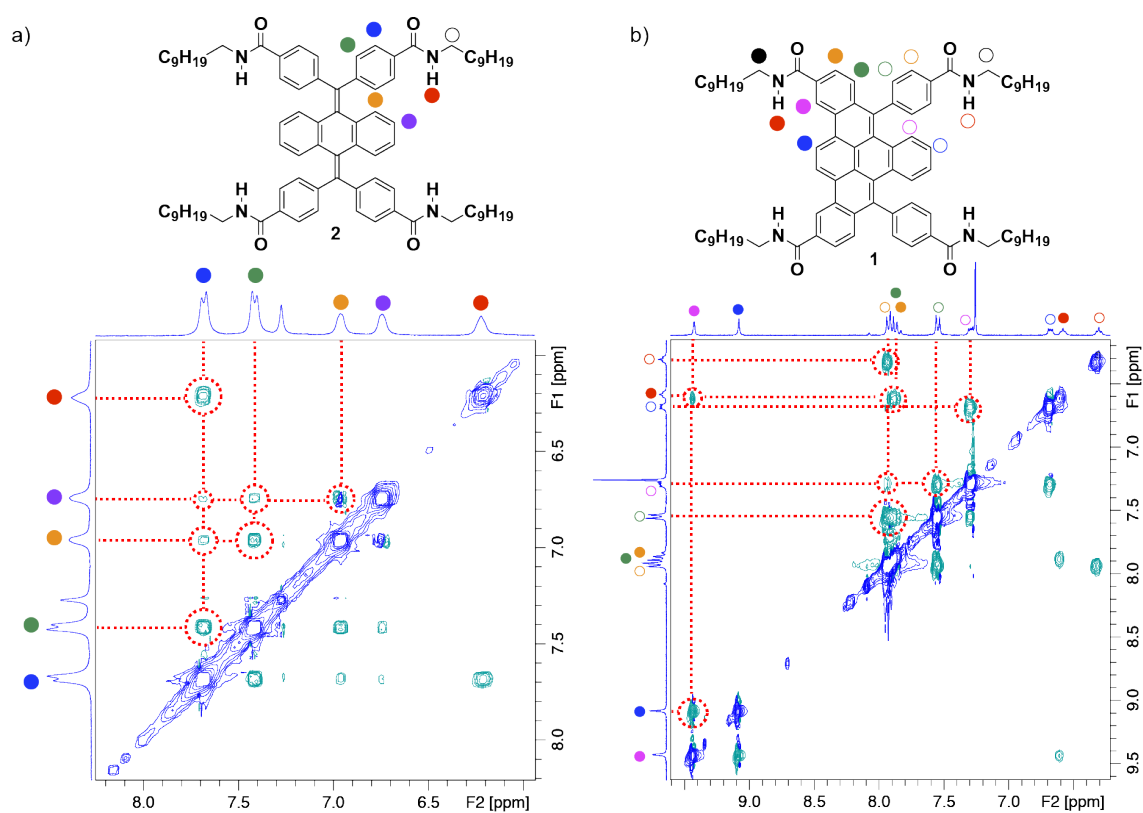


Figure S4. ROESY NMR spectra of (a) **2** (CDCl_3 , 300 MHz, 30 mM, 303 K) and (b) **1** (CDCl_3 , 300 MHz, 4 mM, 318 K). The red, dotted lines show the intramolecular through-space coupling signals between the different resonances

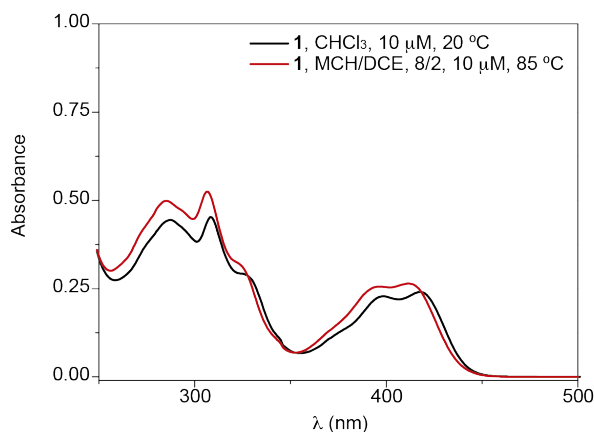


Figure S5. UV-Vis spectra of **1** in CHCl_3 at 20 °C (black line) and MCH/DCE (8/2) at 85 °C. The concentration of both samples is 10 μM .

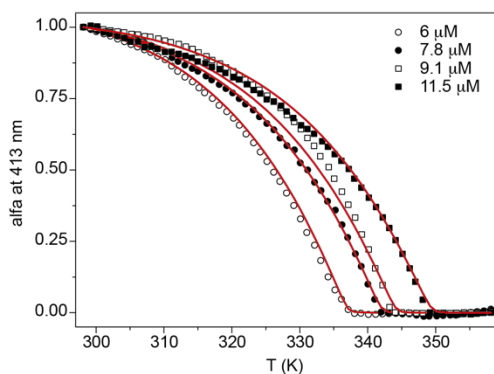


Figure S6. UV-Vis cooling curves of compound **1** (MCH/DCE 8/2, measured at $\lambda = 413$ nm). The red lines correspond to the fit to the EQ model.^{4a}

	1
ΔH_e [kJ/mol] ^a	-55.1 ± 5
ΔS [J/Kmol]	-63 ± 1
ΔH_n [kJ/mol] ^a	-23.9 ± 2
σ [-] ^b	3.4×10^{-6}
K_e [L/mol] ^b	2.4×10^6
K_n [L/mol] ^b	8.2

Table S1. ^aThe nucleation penalty ΔH_n is negative, which implies that the enthalpy gain is smaller for nucleation compared to elongation. ^bThe equilibrium constants for elongation and nucleation, K_e and K_n , and the cooperativity factor σ ($= K_n/K_e$) are calculated at 298 K.

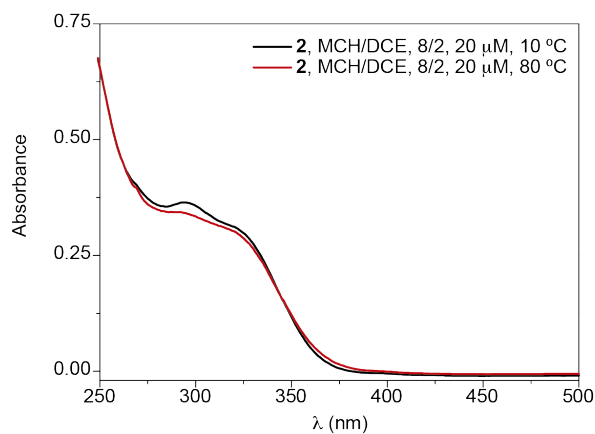


Figure S7. UV-Vis spectra of **2** in MCH/DCE (8/2) at 20 °C and 80 °C. The concentration is 20 μM .

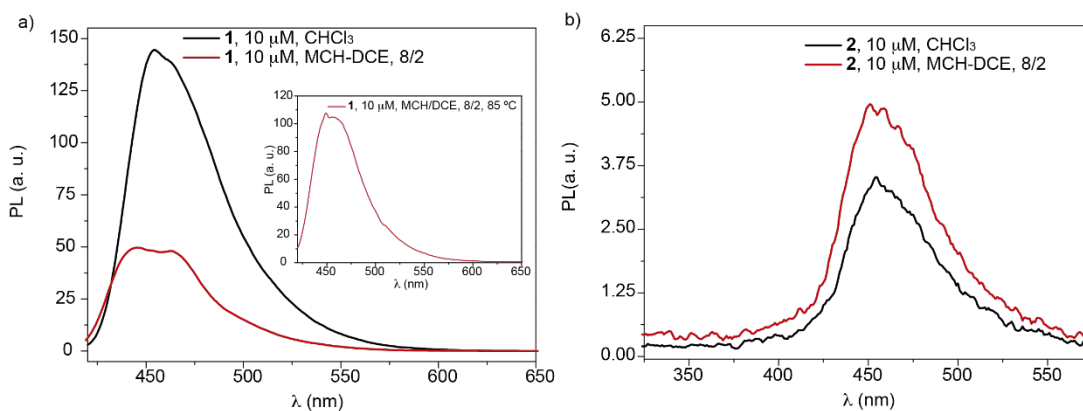


Figure S8. Emission spectra of **1** (a) and **2** (b) in MCH/DCE, 8/2, and CHCl_3 (20 °C, 10 μM ; $\lambda_{\text{exc}} = 334 \text{ nm}$). The inset in (a) shows the PL spectrum of **1** in MCH/DCE, 8/2, 10 μM , at 85 °C.

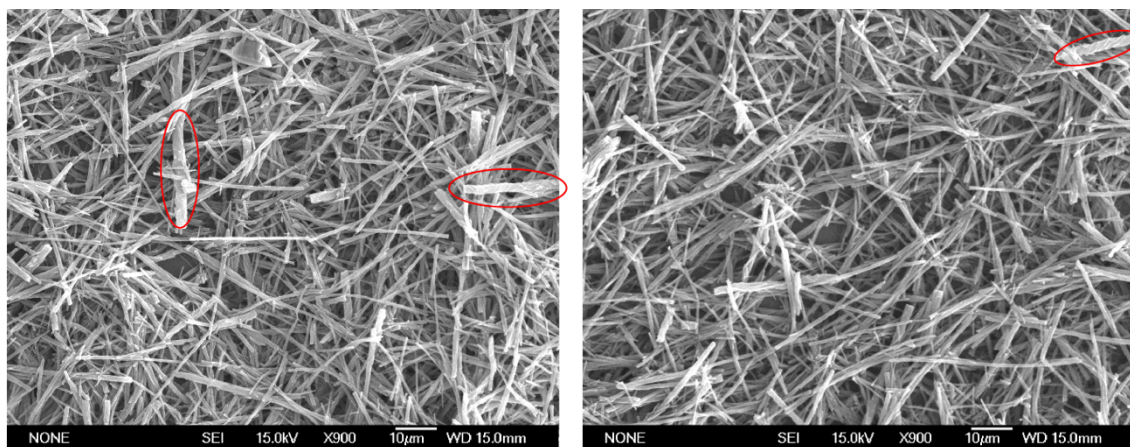


Figure S9. SEM images of the rod-like supramolecular structures formed by the self-assembly of **1** onto a glass substrate.

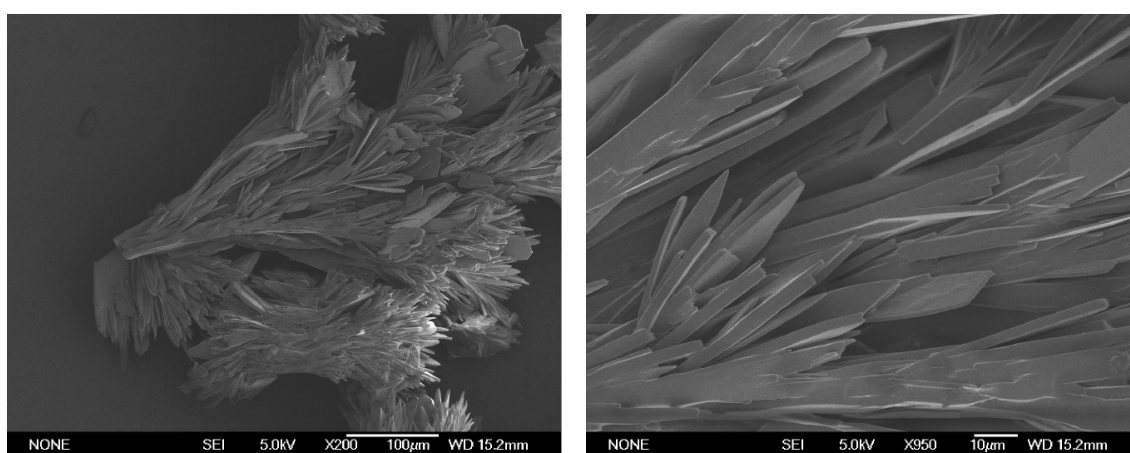


Figure S10. SEM images of the lamellar supramolecular structures formed by the self-assembly of **2** onto a glass substrate.

2. Experimental section

General. All solvents were dried according to standard procedures. Reagents were used as purchased. All air-sensitive reactions were carried out under argon atmosphere. Flash chromatography was performed using silica gel (Merck, Kieselgel 60, 230-240 mesh or Scharlau 60, 230-240 mesh). Analytical thin layer chromatography (TLC) was performed using aluminium-coated Merck Kieselgel 60 F254 plates. NMR spectra were recorded on a Bruker Avance 300 (^1H : 300 MHz; ^{13}C : 75 MHz) and on a Bruker Avance 500 (^1H : 500 MHz; ^{13}C : 126 MHz) spectrometer using partially deuterated solvents as internal standards. Coupling constants (J) are denoted in Hz and chemical shifts (δ) in ppm. Multiplicities are denoted as follows: s = singlet, d = doublet, t = triplet, q = quadruplet, m = multiplet, br = broad. FT-IR spectra in film were recorded on a Bruker Tensor 27 (ATR device) spectrometer. The samples for the FTIR spectra in film were prepared as follows: a 1 mM solution of the corresponding tetracarboxamide in CHCl_3 was deposited in the ATR device and the solvent was gently evaporated under a nitrogen stream. FT-IR spectra in solution were recorded on a SHIMADZU FTIR-8300 Fourier transform infrared spectrophotometer. UV-Vis spectra were registered on a Jasco-V630 spectrophotometer equipped with a Peltier thermoelectric temperature controller. The spectra were recorded in the continuous mode between 200 and 500 nm, with a wavelength increment of 1 nm, a response time of 4 s, and a bandwidth of 1 nm. A 1 mm path length quartz cuvette (Hellma) was used. Thermal experiments were performed at constant heating rates of 1 K min^{-1} . Fluorescence spectra were recorded on a Jasco FP-6500 spectrophotometer. SEM images were obtained from on a JEOL JSM 6335F microscope working at 10kV. Matrix Assisted Laser Desorption Ionization (coupled to a Time-Of-Flight analyzer) experiments (MALDI-TOF) were recorded on a Bruker REFLEX spectrometer.

3. X-Ray data collection and structure refinement

Data collection for compound **5** was carried out at room temperature on a Bruker Smart CCD diffractometer using graphite-monochromated Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$) operating at 50 kV and 40 mA.

Data were collected over a sphere of the reciprocal space by combination of five exposure sets. Each exposure was of 30 s covered 0.3 in ω . The cell parameters were determined and refinement by least-squares fit of all reflections. The 50 frames were recollected at the end of the data collections to monitor crystal decay, and no appreciable decay was observed.

The structure was solved by direct methods and refined by full-matrix least-square procedures on F2 (SHELXL-97)1. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were included in their calculated positions and refined riding on the respective carbon atoms. Mercury CSD 4.02 was used for molecular graphics (see Figure S11).

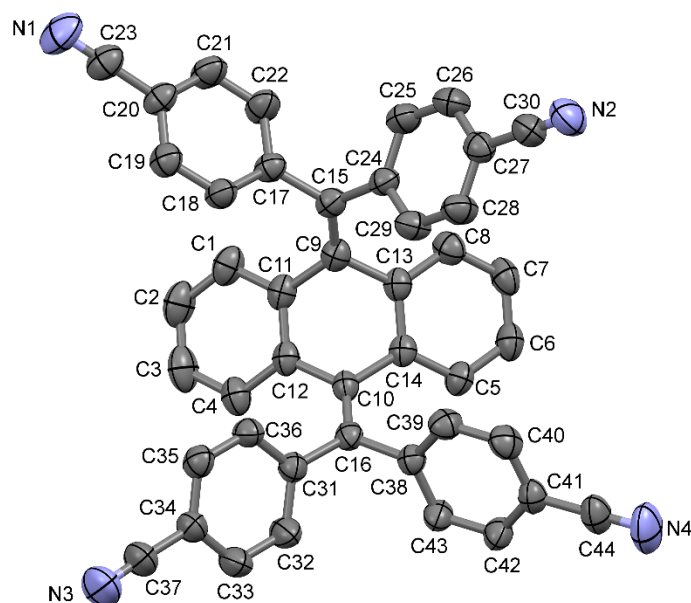


Figure S11. ORTEP diagram (50% probability level) of compound **5**. Hydrogen atoms are omitted for clarity.

Further crystallographic details for the structure reported in this paper may be obtained from the Cambridge Crystallographic Data Center, on quoting the depository number CCDC: 1851673

Table S2. Crystal data and structure refinement for compound **5**.

Identification code	p21c
Empirical formula	C ₄₄ H ₂₄ N ₄
Formula weight	608.67
Temperature/K	296.15
Crystal system	monoclinic
Space group	<i>P21/c</i>
<i>a</i> /Å	10.3313(6)
<i>b</i> /Å	10.8088(6)
<i>c</i> /Å	29.7134(2)
α /°	90
β /°	93.6640(9)
γ /°	90
Volume/Å ³	3311.3(3)
<i>Z</i>	4
$\rho_{\text{calc}}/\text{cm}^3$	1.221
μ/mm^{-1}	0.072
<i>F</i> (000)	1264.0
Crystal size/mm ³	0.52 × 0.18 × 0.12
Radiation	MoK α (λ = 0.71073)
2 θ range for data collection/°	2.746 to 57.838
Index ranges	-14 ≤ <i>h</i> ≤ 13, -14 ≤ <i>k</i> ≤ 14, -40 ≤ <i>l</i> ≤ 40
Reflections collected	33972
Independent reflections	8213 [<i>R</i> _{int} = 0.0720, <i>R</i> _{sigma} = 0.0839]
Data/restraints/parameters	8213/0/433
Goodness-of-fit on <i>F</i> ²	0.988
Final <i>R</i> indexes [<i>I</i> ≥ 2 σ (<i>I</i>)] ^a	<i>R</i> 1 = 0.0533, <i>wR</i> 2 = 0.1070
Final <i>R</i> indexes [all data] ^b	<i>R</i> 1 = 0.1698, <i>wR</i> 2 = 0.1537
Largest diff. peak/hole / e Å ⁻³	0.16/-0.17

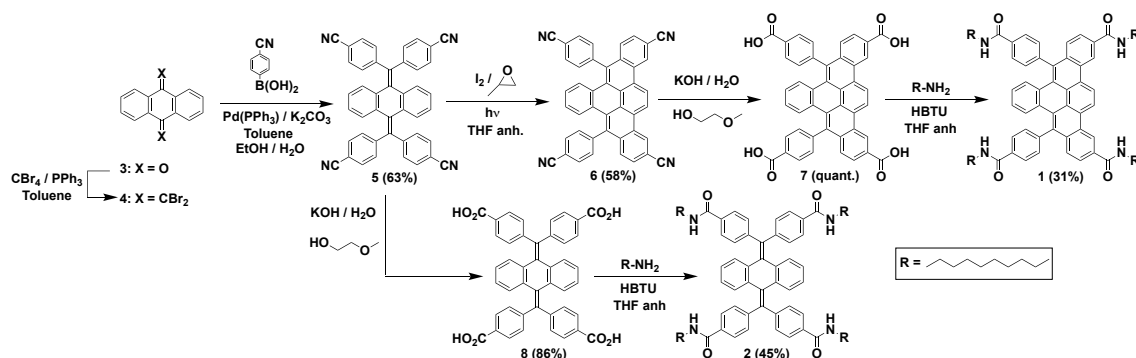
$$^a R1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

$$^b wR2 = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right\}$$

1. Siemens, SMART, SAINT and SHELXTL, Siemens Analytical X-ray Instruments INC., Madison, Wisconsin, USA, **2002**.

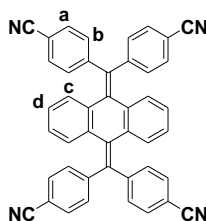
2. I.J. Bruno, J.A. Chisholm, P. R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. Van de Streek and P. A. Wood, *J. APPL. Crystallogr.*, **2008**, 41, 466-470.

4. Synthetic details and characterization



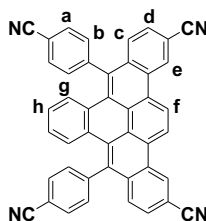
Compound **4** was prepared according to previously reported synthetic procedures (see: R. Neidlein and M. Winter, *Synthesis*, 1998, 1362) and showed identical spectroscopic properties to those reported therein.

9,10-bis(di-4'-cyanophenylmethylene)-9,10-dihydroanthracene (**5**)



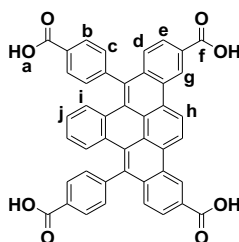
To a stirred suspension of **4** (1.0 g, 1.92 mmol) and 4-cyanophenylboronic acid (1.07 g, 11.5 mmol) in 55 mL of toluene, potassium carbonate (2.11 g, 15.3 mmol) was added in 16 mL of a EtOH:H₂O mixture (1:1) under argon atmosphere. After fifteen minutes, *tetrakis*(triphenylphosphine)palladium(0) (89 mg, 1.68 mmol) was added and the reaction mixture was heated at reflux overnight. The solvent was evaporated under reduced pressure and the crude was dissolved in dichloromethane, washed with brine and dried over MgSO₄. After removal of the solvent, the raw product was purified by silica gel column chromatography (CH₂Cl₂ as eluent), affording **5** as pale pink flakes in a 63 % yield (730 mg, 1.2 mmol). ¹H NMR (300 MHz, CDCl₃) (δ / ppm): 7.64 (d, 8H, H_a, *J*=8.3), 7.46 (d, 8H, H_b, *J*=8.3), 6.93 (m, 4H, H_c), 6.85 (m, 4H, H_d). ¹³C NMR (75 MHz, CDCl₃) (δ / ppm): 145.75, 137.68, 136.94, 136.28, 132.74, 130.61, 128.55, 126.78, 118.36, 111.71. FTIR (ν /cm⁻¹): 571, 642, 668, 730, 760, 828, 862, 951, 1020, 1044, 1110, 1179, 1267, 1401, 1452, 1498, 1601, 1923, 2226, 3061. MALDI-MS: C₄₄H₂₄N₄ [M+1]⁺ calcd. 609.178; found, 609.080.

5,10-bis(4-cyanophenyl)-5b,9a-dihydrodibenzo[h,rst]pentaphene-2,13-dicarbonitrile (6)



To a stirred suspension of **5** (330 mg, 0.54 mmol) in 180 mL of anhydrous tetrahydrofuran, iodine (680 mg, 2.71 mmol) and 15 mL of propylene oxide were added portion wise under argon atmosphere. The reaction mixture was stirred overnight at room temperature and under UV-light irradiation ($\lambda = 360$ nm). The solvent was evaporated under reduced pressure and the resulting crude was washed with methanol and filtered affording compound **6** as yellow flakes in a 58 % yield (190 mg, 0.31 mmol). ^1H NMR (700 MHz, $\text{C}_2\text{D}_2\text{Cl}_4$) (δ / ppm): 9.31 (s, 2H, H_e), 9.13 (s, 2H, H_i), 7.88 (d, 4H, H_a , $J=8.2$), 7.86 (d, 2H, H_d), 7.77 (d, 2H, H_c , $J=8.5$), 7.69 (d, 4H, H_b , $J=7.6$), 7.29 (m, 2H, H_g), 6.88 (m, 2H, H_h). ^{13}C NMR (150 MHz, $\text{C}_2\text{D}_2\text{Cl}_4$) (δ / ppm): 145.59, 133.50, 133.30, 133.11, 132.69, 131.33, 130.99, 129.23, 129.10, 128.69, 128.18, 127.57, 125.80, 123.14, 120.60, 119.61, 118.98, 112.35, 110.42, 99.81. FTIR (ν / cm^{-1}): 626, 703, 742, 802, 827, 887, 948, 1005, 1094, 1260, 1375, 1460, 1605, 2229, 2854, 2924, 2957. MALDI-MS: $\text{C}_{44}\text{H}_{20}\text{N}_4$ $[\text{M}+1]^+$ calcd. 605.1688; found, 604.896.

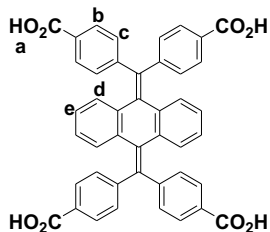
5,10-bis(4-carboxyphenyl)-5b,9a-dihydrodibenzo[h,rst]pentaphene-2,13-dicarboxylic acid (7)



To a stirred suspension of **6** (190 mg, 0.31 mmol) in 15 mL of 2-methoxyethanol, potassium hydroxide (4.74 mL, 12 M) was added portion wise and the reaction mixture was stirred overnight at reflux. To the resulting crude, HCl (1 M) was added and the precipitate was filtrated affording **7** as a yellow solid in a quantitative yield (213 mg, 0.31 mmol). ^1H NMR (500 MHz, DMSO) (δ / ppm): 13.29 (s, 4H, $\text{H}_{a,f}$), 9.54 (s, 2H, H_g), 9.09 (s, 2H, H_h), 8.19 (s, 4H, H_b), 8.18 (d, 2H, H_e , $J=9.1$), 7.90 (d, 2H, H_d , $J=8.8$), 7.78 (d, 4H, H_c , $J=8.1$), 7.47 (m, 2H, H_i), 6.94 (m, 2H, H_j). ^{13}C NMR (126 MHz, DMSO) (δ / ppm): 167.35, 167.16, 145.17, 133.31, 132.95, 132.73, 132.41, 130.72, 130.23, 130.14, 129.07, 128.53, 128.02, 127.12, 127.03, 126.52, 125.11, 124.37, 122.57, 79.18. FTIR (ν

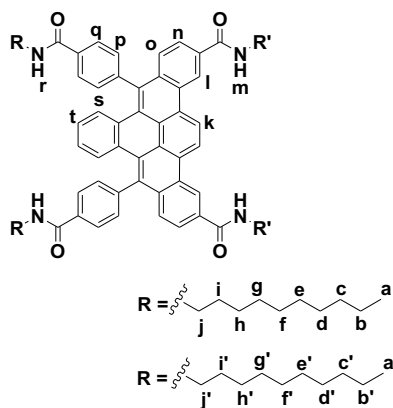
/cm⁻¹): 705, 742, 764, 819, 1008, 1026, 1072, 1123, 1153, 1290, 1379, 1463, 1601, 1730, 3309, 3395, 3671. MALDI-MS: C₄₄H₂₄O₈ [M+1]⁺ calcd.681.1471; found, 681.1584.

9,10-bis(di-4'-carboxyphenylmethylene)-9,10-dihydroanthracene (8)



To a stirred suspension of **5** (330 mg, 0.542 mmol) in 25 mL of 2-methoxyethanol, potassium hydroxide (8.25, 12 M) was added portion wise and the reaction was heated overnight at reflux. To the resulting organic mixture, HCl (1 M) was added and the precipitate was filtrated affording **8** as a brown solid in 86 % yield (320 mg, 0.46 mmol). ¹H NMR (300 MHz, DMSO) (δ / ppm): 12.98 (s, 4H, H_a), 7.95 (d, 8H, H_b, J=8.4), 7.65 (d, 8H, H_c, J=8.3), 6.94 (m, 4H, H_d), 6.80 (m, 4H, H_e). ¹³C NMR (75 MHz, DMSO) (δ / ppm): 167.04, 146.26, 138.91, 136.57, 135.17, 129.65, 129.62, 129.28, 127.63, 125.80. FTIR (ν /cm⁻¹): 542, 631, 662, 714, 750, 767, 793, 874, 927, 956, 1019, 1112, 1128, 1179, 1285, 1311, 1424, 1454, 1562, 1606, 1703, 2550, 2674, 2923, 3003, 3444. MALDI-MS: C₄₄H₂₈O₈ [M+1]⁺ calcd. 685.1784; found, 685.1854.

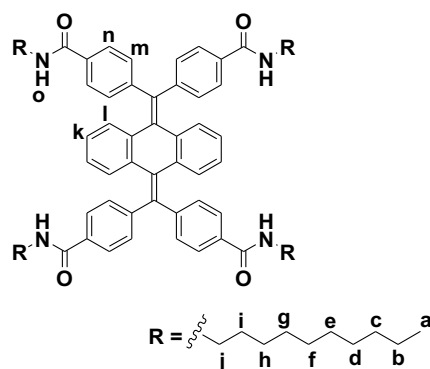
N²,N¹³-didecyl-5,10-bis(4-(decylcarbamoyl)phenyl)-5b,9a-dihydrodibenzo-[h,rst]pentaphene-2,13-dicarboxamide (1)



To a stirred solution of **7** (100 mg, 0.14 mmol) in 15 mL of anhydrous tetrahydrofuran, decylamine (140 mg, 0.88 mmol) and HBTU (280 mg, 0.73 mmol) were added portion wise under argon atmosphere. The reaction mixture was stirred overnight at room temperature. The resulting crude was washed with HCl (1 M), NaOH (1M) and brine and dried over MgSO₄. After filtration and removal of the solvent, the residue was purified by column chromatography (silica gel, CH₂Cl₂:MeOH 10:0.2) and subsequent precipitation

from CH₂Cl₂/MeOH afforded compound **2** as yellow flakes in a 31 % yield. (10 mg, 0.008 mmol) ¹H NMR (500 MHz, CDCl₃) (δ / ppm): 9.36 (s, 2H, H_l), 8.95 (s, 2H, H_k), 7.88 (d, 4H, H_q, *J*=7.8), 7.83 (s, 4H, H_{n+o}), 7.43 (d, 4H, H_p, *J*=7.5), 7.17 (m, 2H, H_s), 6.72 (m, 2H, H_m), 6.58 (m, 2H, H_t), 6.37 (m, 2H, H_r), 3.64 (q, 4H, H_j), 3.53 (q, 4H, H_i), 1.91 – 1.10 (br, 64H, H_{b+b'+c+c'+d+d'+e+e'+f+f'+g+g'+h+h'+i+i'}), 0.89 (t, 12H, H_{a+a'}, *J*=2.3). ¹³C NMR (126 MHz, CDCl₃) (δ / ppm): 167.89, 167.29, 144.60, 134.33, 133.45, 133.06, 132.60, 130.89, 129.22, 129.12, 128.45, 127.85, 127.74, 127.58, 127.42, 126.10, 125.52, 125.04, 124.40, 122.33, 40.79, 40.54, 32.14, 32.08, 29.99, 29.86, 29.82, 29.78, 29.75, 29.63, 29.57, 29.50, 29.47, 27.41, 27.32, 22.83, 14.20. FTIR (ν /cm⁻¹): 733, 1267, 1308, 1373, 1462, 1504, 1543, 1634, 1715, 2853, 2922, 3294, 3841. HRMS (MALDI-TOF) *m/z* for C₈₄H₁₀₈N₄O₄ [M+1]⁺ calcd. 1237.8371; found, 1237.8387.

9,10-bis(di-4'-N-decylcarbamoylphenylmethylene)-9,10-dihydroanthracene (**2**)

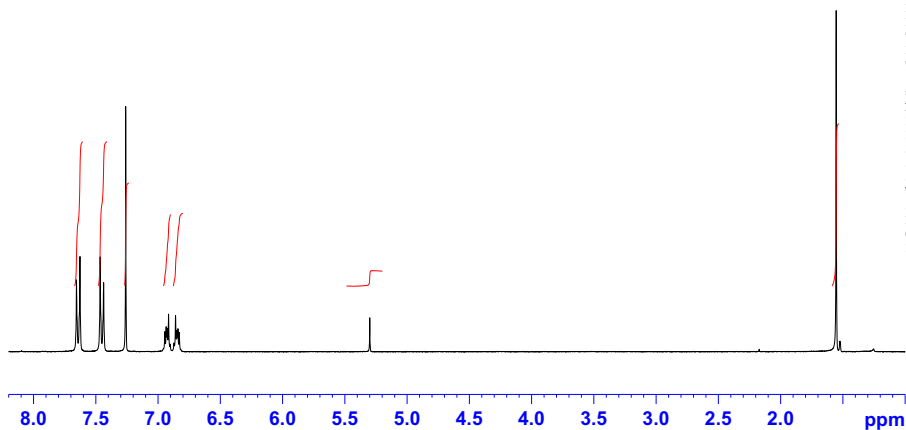


Tetrabenzoic acid **9** (230 mg, 0.33 mmol) was dissolved in anhydrous THF (15 mL) under argon atmosphere. After fifteen minutes, decylamine (316 mg, 2.01 mmol) and HBTU (635 mg, 1.67 mmol) were added. The reaction mixture was stirred overnight at room temperature. The resulting crude was washed with HCl (1 M), NaOH (1 M) and brine solution and dried over MgSO₄. After filtration and removal of the solvent, the residue was purified by column chromatography (silica gel, CH₂Cl₂:MeOH 10:0.2) and subsequent precipitation from CH₂Cl₂/MeOH afforded compound **2** as white flakes in 45 % yield (280 mg, 0.22 mmol). ¹H NMR (500 MHz, CDCl₃) (δ / ppm): 7.68 (d, 8H, H_n, *J*=8.1), 7.41 (d, 8H, H_m, *J*=8.1), 6.95 (m, 4H, H_i), 6.73 (m, 4H, H_k), 6.17 (t, 4H, H_o, *J*=5.5), 3.44 (q, 8H, H_j), 1.58-1.31 (br, 64H, H_{b+c+d+e+f+g+h+i}), 0.87 (t, 12H, H_a, *J*=6.7). ¹³C NMR (126 MHz, CDCl₃) (δ / ppm): 167.40, 145.31, 138.28, 137.23, 136.82, 133.63, 130.06, 128.14, 127.24, 126.07, 40.66, 32.30, 30.14, 30.11, 29.96, 29.74, 29.69, 27.46, 23.06, 14.46. FTIR (ν /cm⁻¹): 608, 631, 684, 728, 869, 1021, 1107, 1158, 1265, 1314, 1374, 1462, 1499, 1552, 1632, 1717, 2852, 2922, 3281. HRMS (MALDI-TOF) *m/z* for C₈₄H₁₁₂N₄O₄ [M+H]⁺ calcd. 1241.8684; found, 1241.8705.

5. Collection of spectra



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PROCNO    1
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PULPROG   zg30
TD        32768
SOLVENT   CDCl3
NS        16
DS        2
SWH       4496.403 Hz
FIDRES    0.137219 Hz
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DE        6.50 usec
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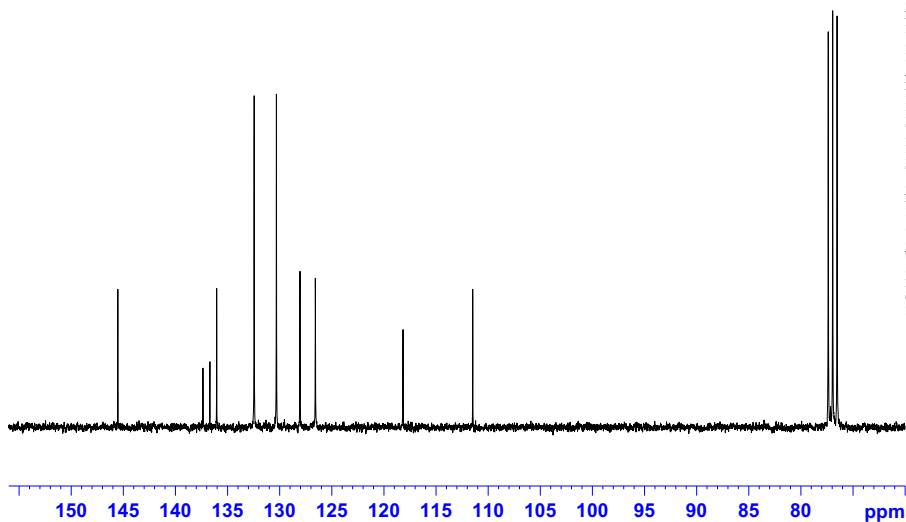


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WDW       EM
SSB       0
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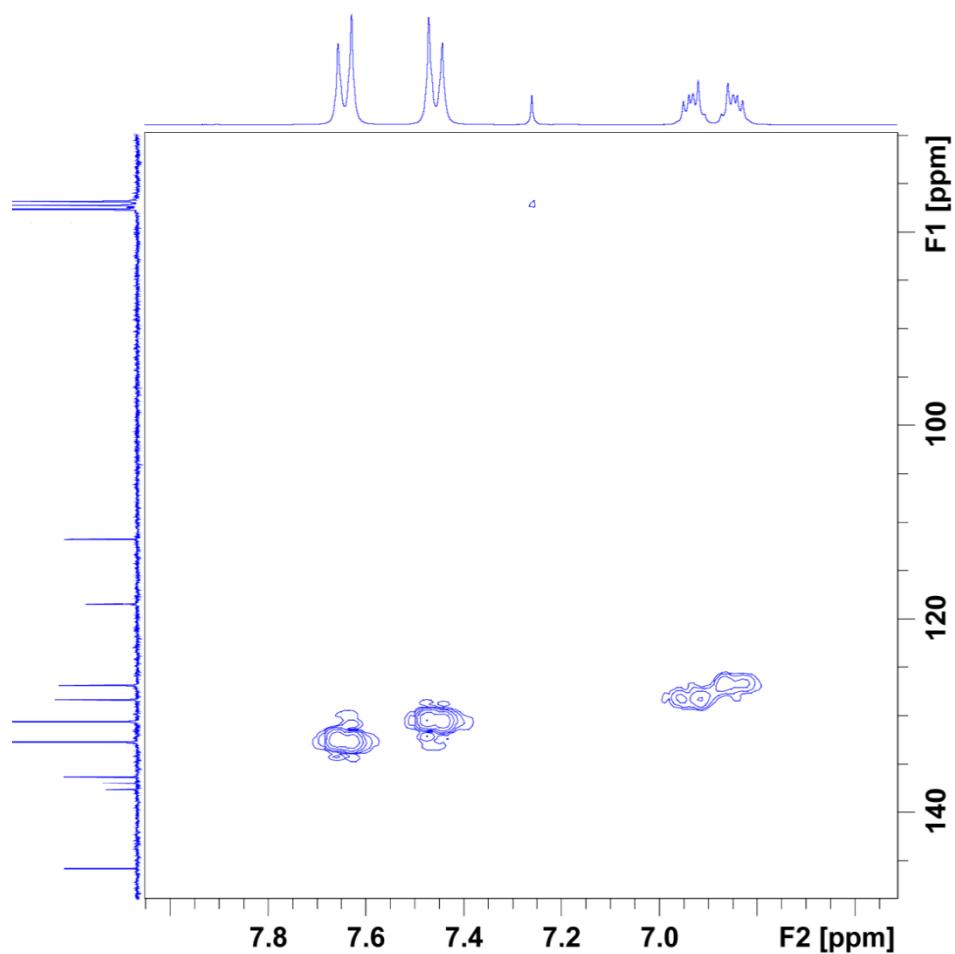
^1H NMR spectrum (CDCl_3 , 300 MHz, 298 K) of compound **5**.



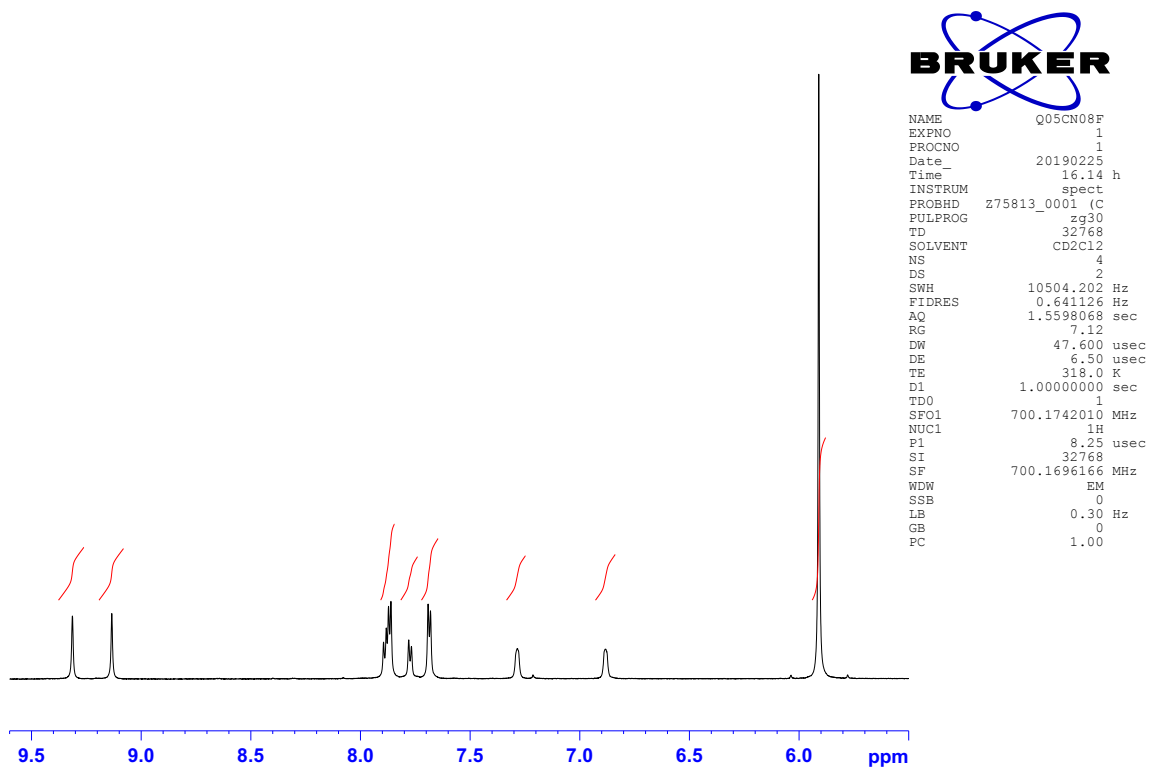
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Date_     20190214
Time      20.18 h
INSTRUM   spect
PROBHD    Z104275_0393 (
PULPROG   zgpg30
TD        65536
SOLVENT   C2D2Cl4
NS        800
DS        4
SWH       18115.941 Hz
FIDRES    0.552855 Hz
AQ        1.8088436 sec
RG        13.95
DW        27.600 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
SFO1     75.4835188 MHz
NUC1      13C
P1        10.00 usec
SI        32768
SF        75.4752055 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
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^{13}C NMR spectrum (CDCl_3 , 75 MHz, 298 K) of compound **5**.



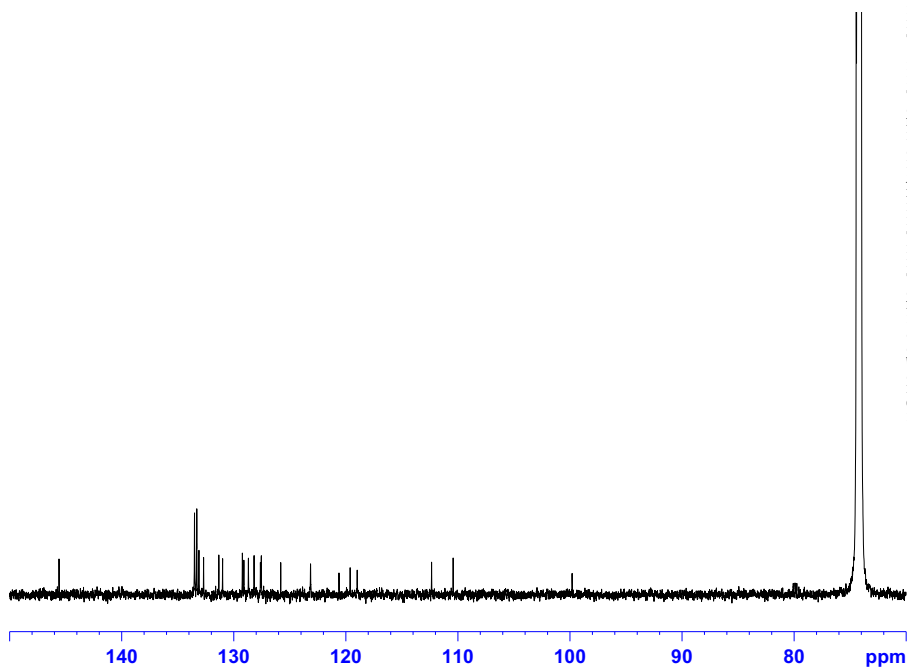
^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound 5.



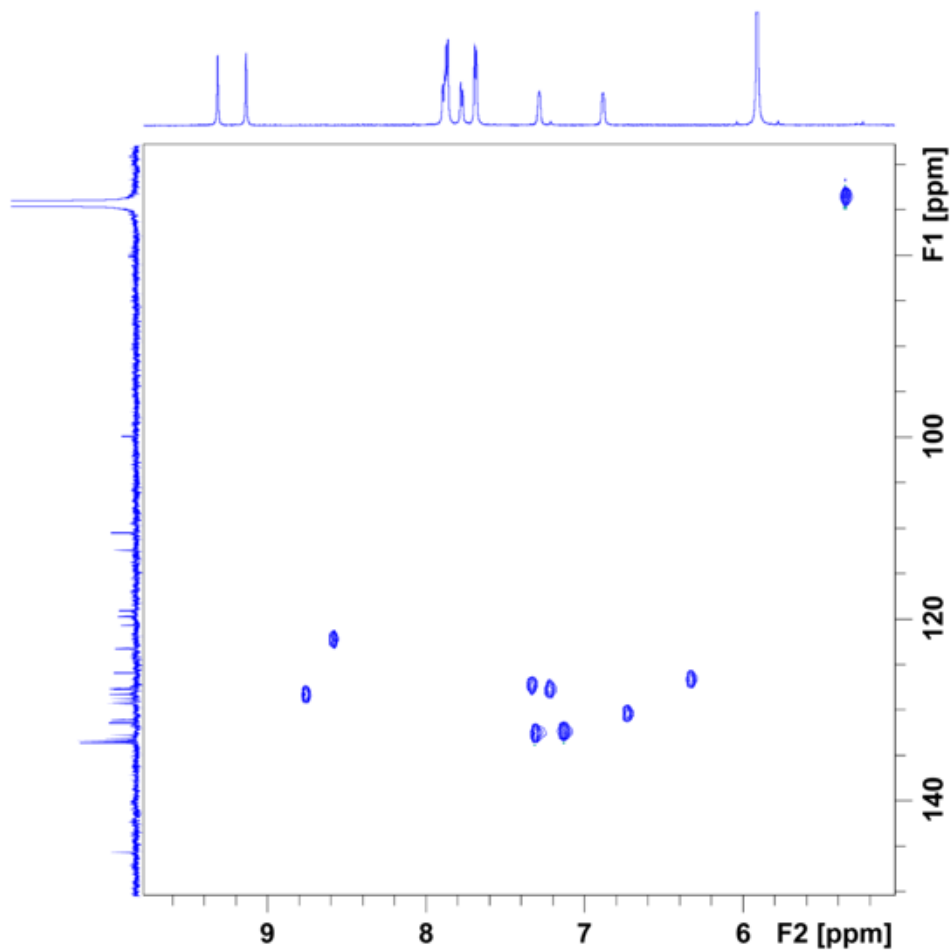
^1H NMR spectrum ($\text{C}_2\text{D}_2\text{Cl}_4$, 700 MHz, 298 K) of compound 6.



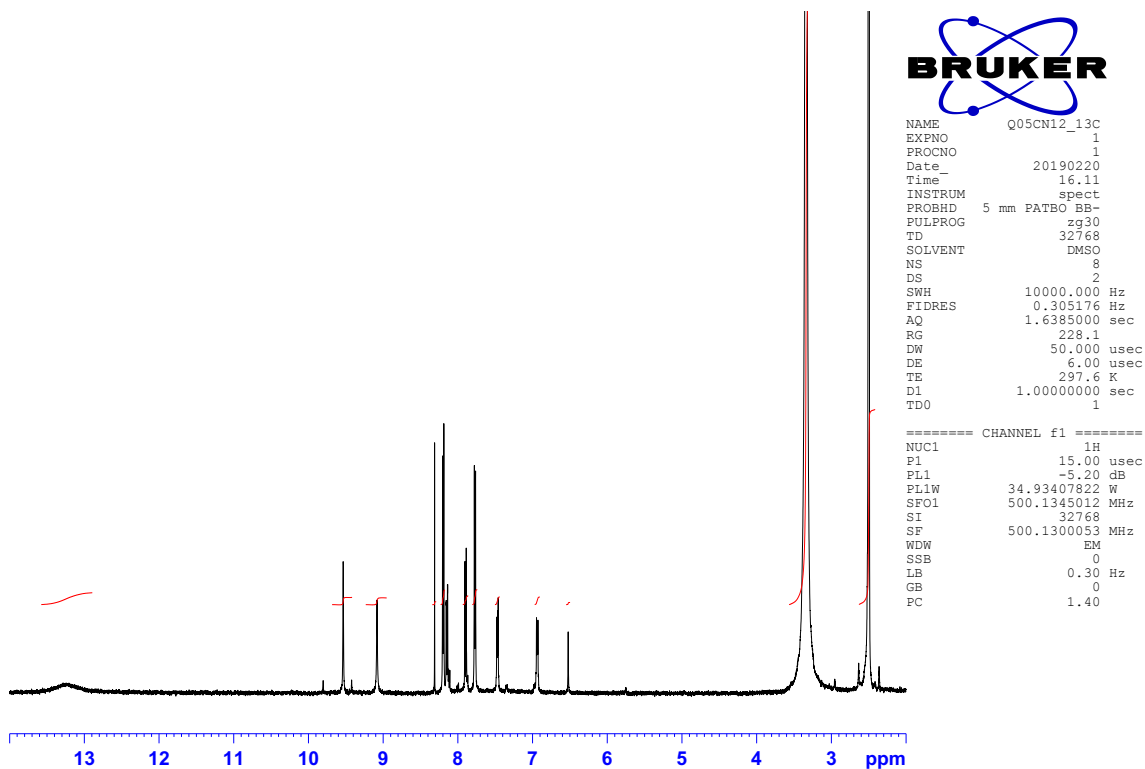
NAME Q05CN08F
EXPNO 2
PROCNO 1
Date_ 20190225
Time_ 17.10 h
INSTRUM spect
PROBHD Z75813_0001 (C
PULPROG zgpg30
TD 65356
SOLVENT CD2Cl2
NS 1200
DS 4
SWH 48076.922 Hz
FIDRES 1.471232 Hz
AQ 0.6797524 sec
RG 1030
DW 10.400 usec
DE 18.00 usec
TE 318.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
SF01 176.0772534 MHz
NUC1 13C
P1 13.75 usec
SI 32768
SF 176.0577395 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



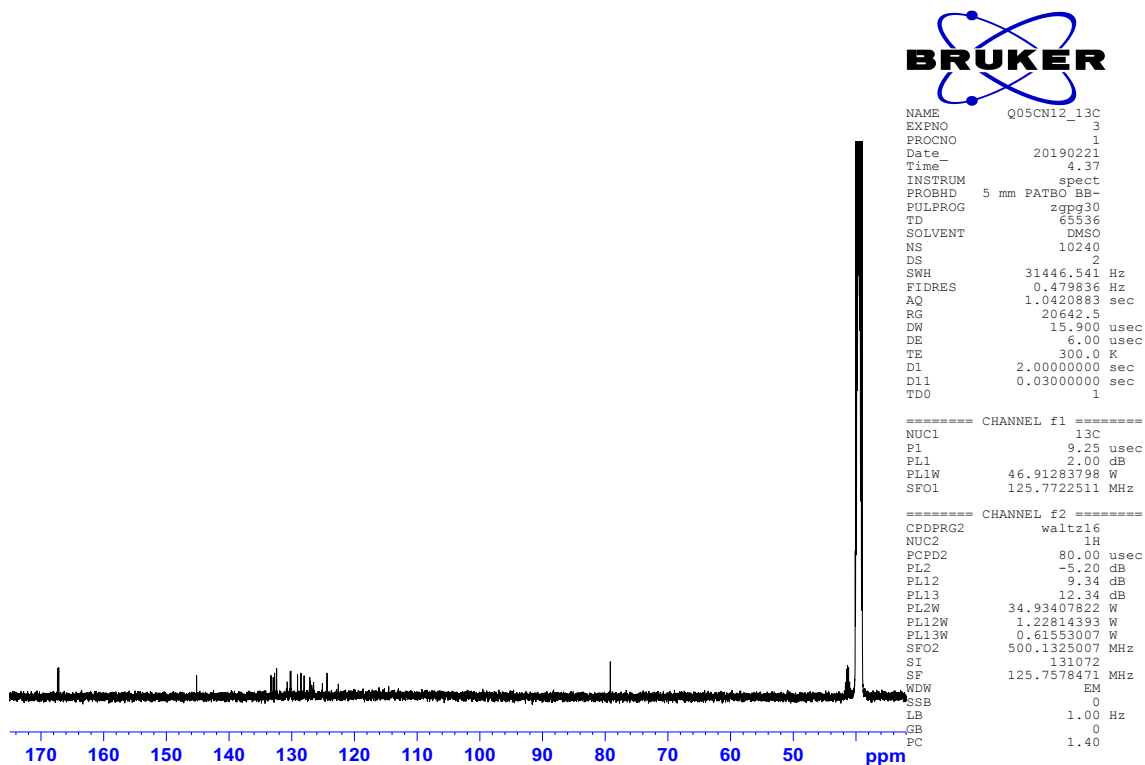
^{13}C NMR spectrum ($\text{C}_2\text{D}_2\text{Cl}_4$, 150 MHz, 298 K) of compound **6**.



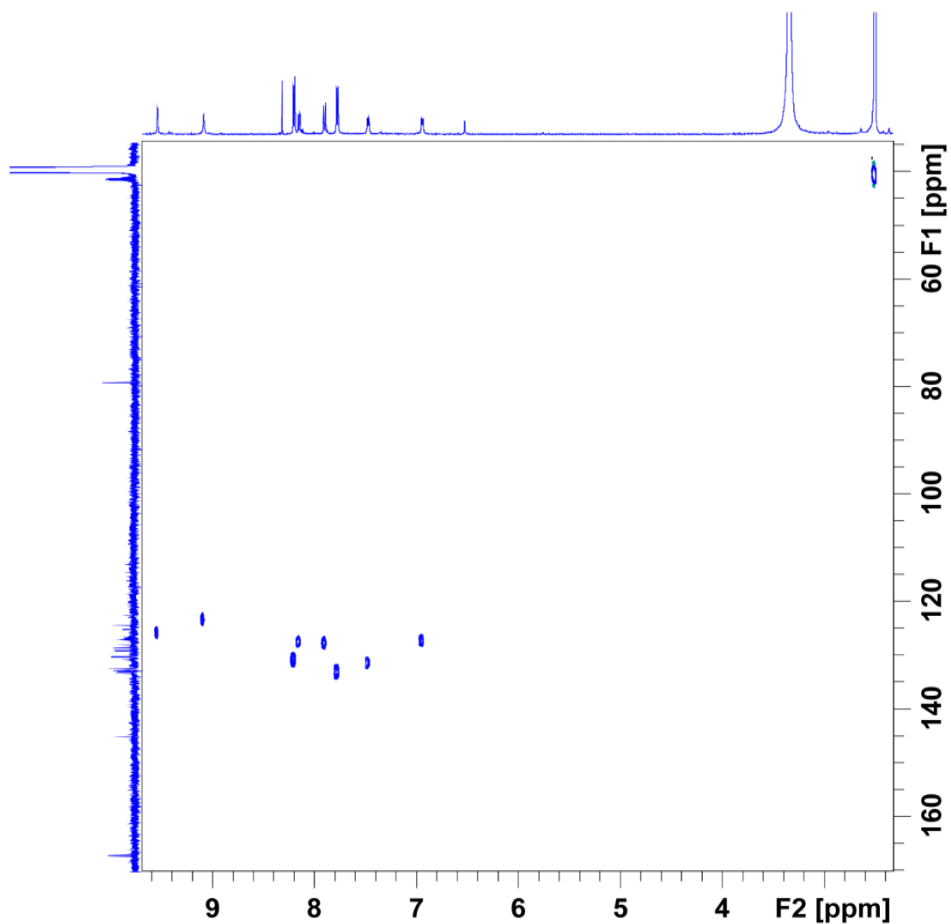
^1H , ^{13}C -HMQC spectrum ($\text{C}_2\text{D}_2\text{Cl}_4$, 298 K) of compound 6.



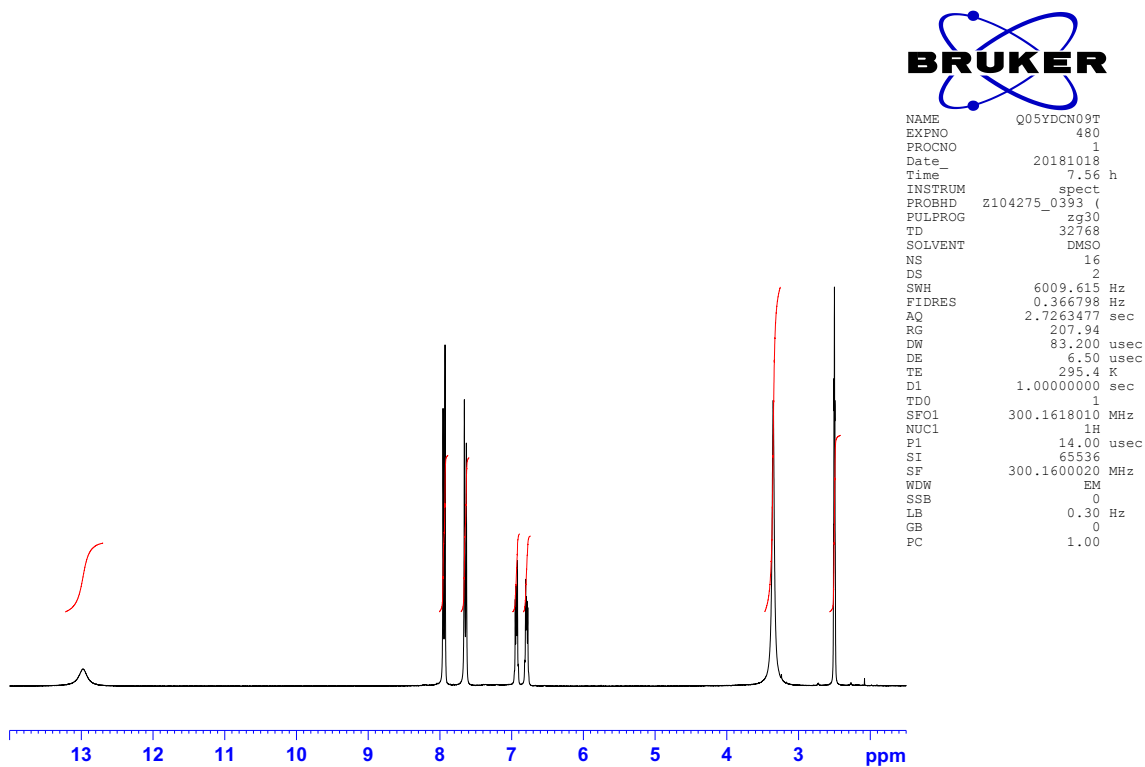
^1H NMR spectrum (CDCl_3 , 500 MHz, 298 K) of compound 7.



^{13}C NMR spectrum (CDCl_3 , 126 MHz, 298 K) of compound 7.



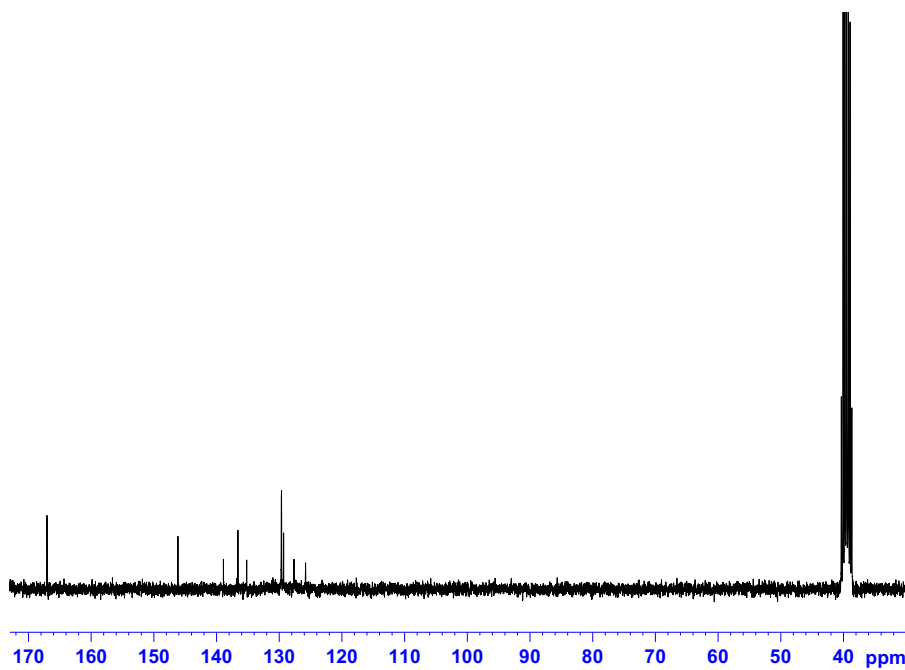
^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound 7.



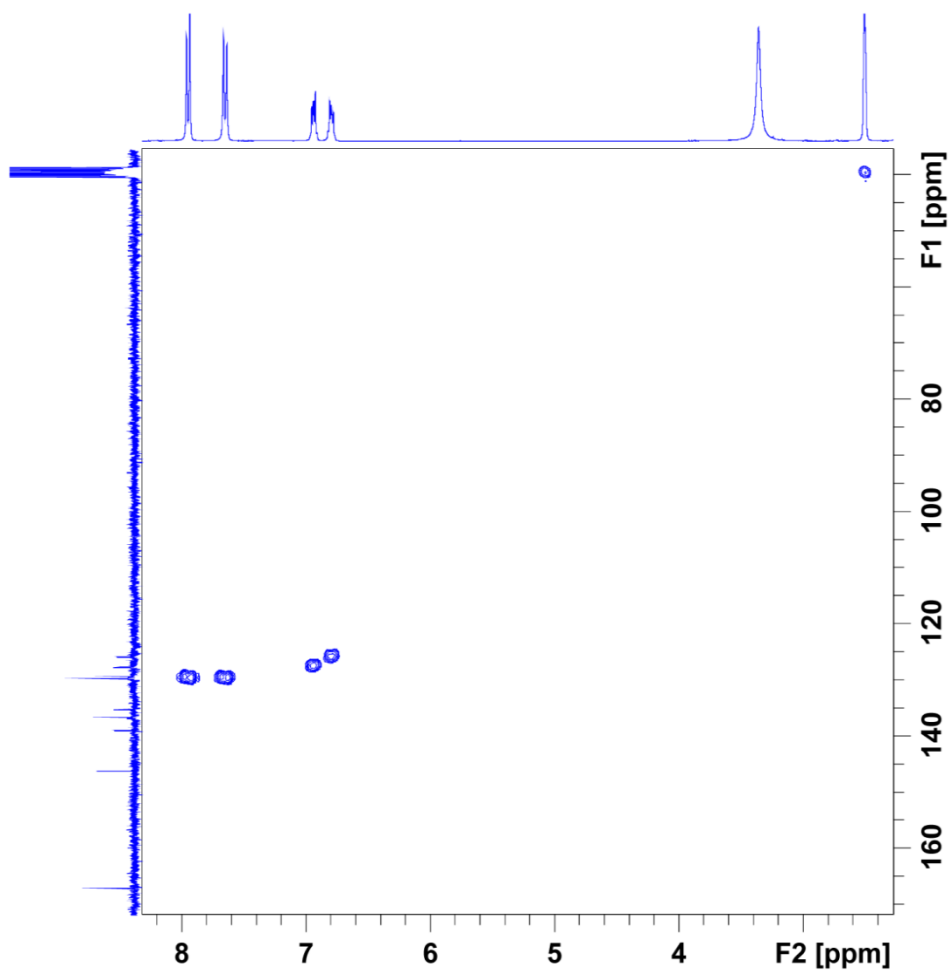
^1H NMR spectrum (DMSO, 300 MHz, 298 K) of compound **8**.



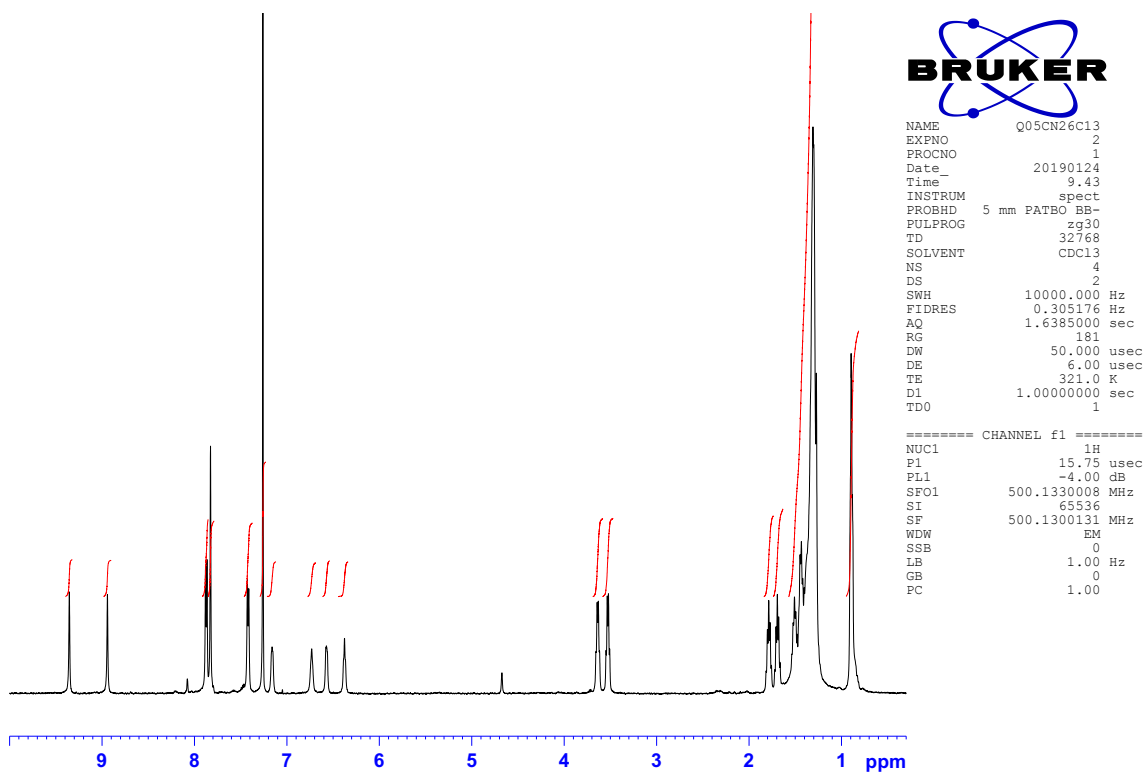
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EXPNO         481
PROCNO        1
Date_         20181018
Time          2.05 h
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PROBHD        z104275_0393 (
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            800
DS            4
SWH           18115.941 Hz
FIDRES        0.552855 Hz
AQ            1.8088436 sec
RG            13.95
DW            27.600 usec
DE            6.50 usec
TE            295.8 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
SF01          75.4835188 MHz
NUC1          13C
P1            10.00 usec
SI            32768
SF            75.4756850 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
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^{13}C NMR spectrum (DMSO, 75 MHz, 298 K) of compound **8**.



^1H , ^{13}C -HMQC spectrum (DMSO, 298 K) of compound **8**.



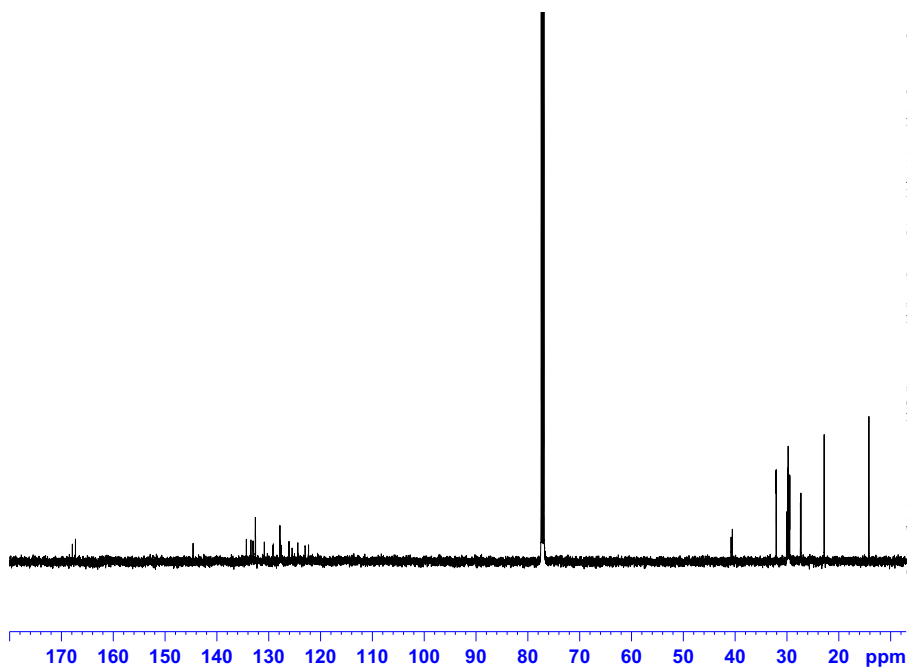
^1H NMR (CDCl_3 , 500 MHz, 298 K) of compound **1**.



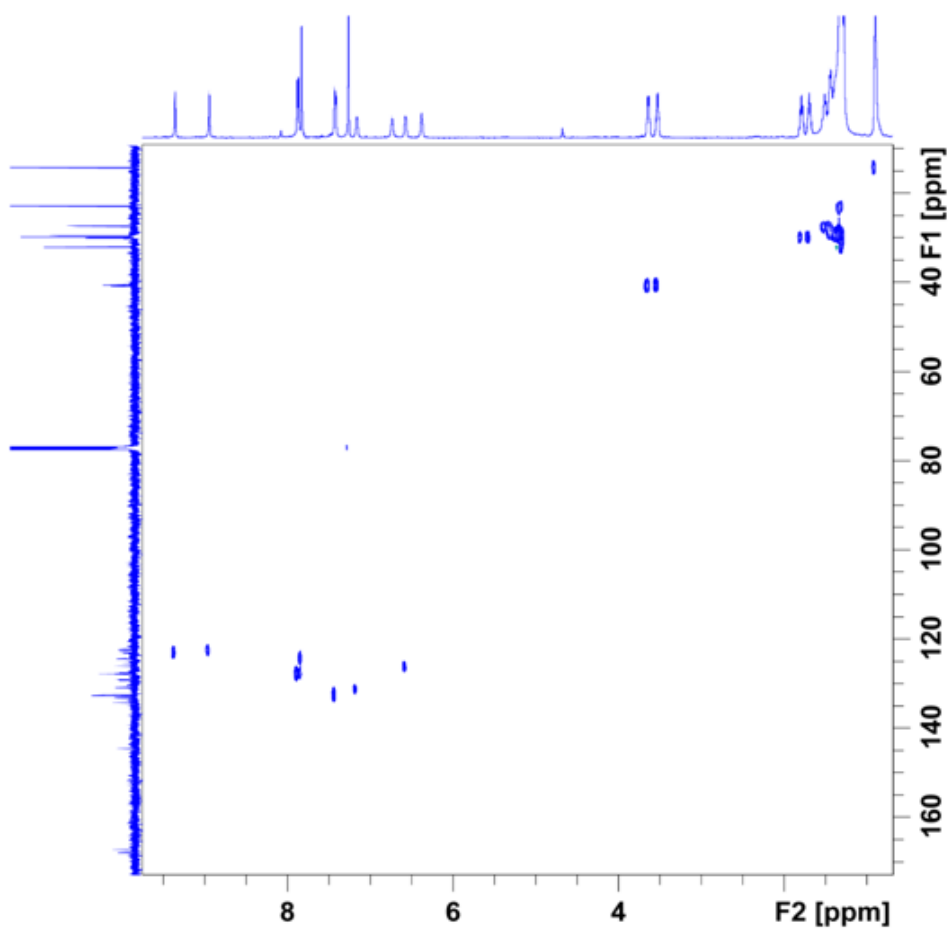
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NAME Q05CN26c13
EXPNO 3
PROCNO 1
Date_ 20190124
Time_ 9.47
INSTRUM spect
PROBHD 5 mm PATBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1432
DS 2
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 20642.5
DW 15.900 usec
DE 6.00 usec
TE 321.4 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 5.00 dB
SFO1 125.772511 MHz

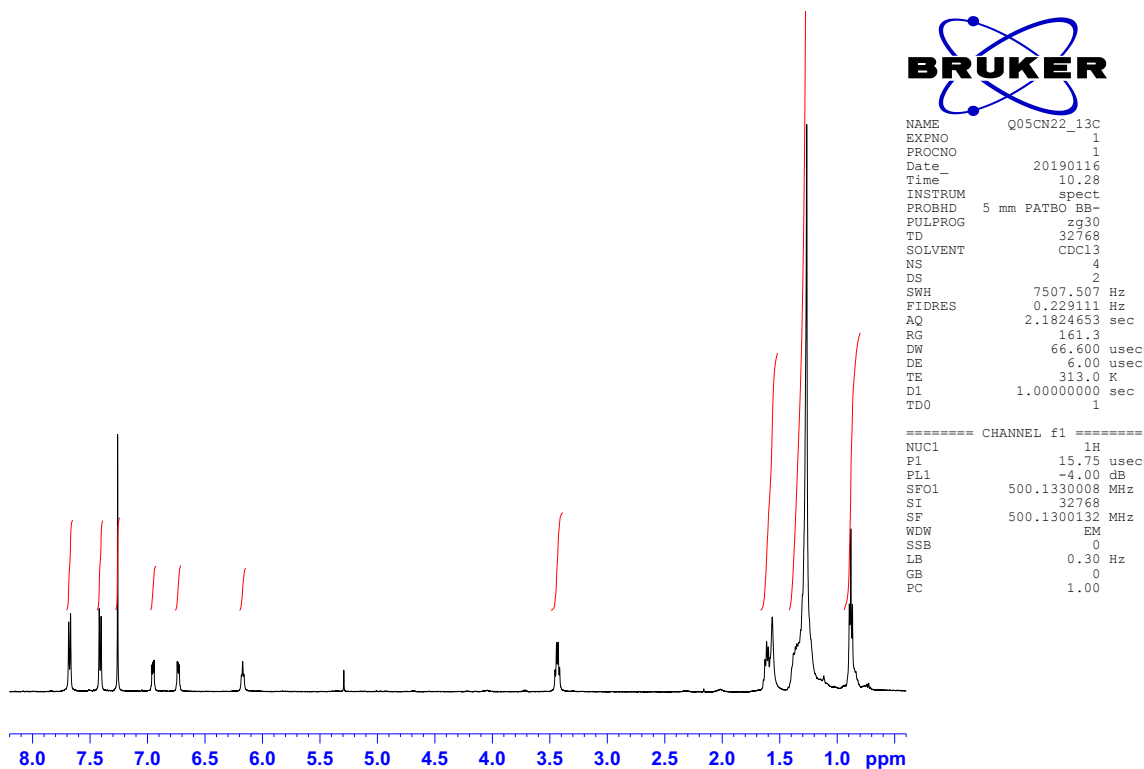
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 11.50 dB
PL13 11.50 dB
SFO2 500.1325007 MHz
SI 131072
SF 125.7577606 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
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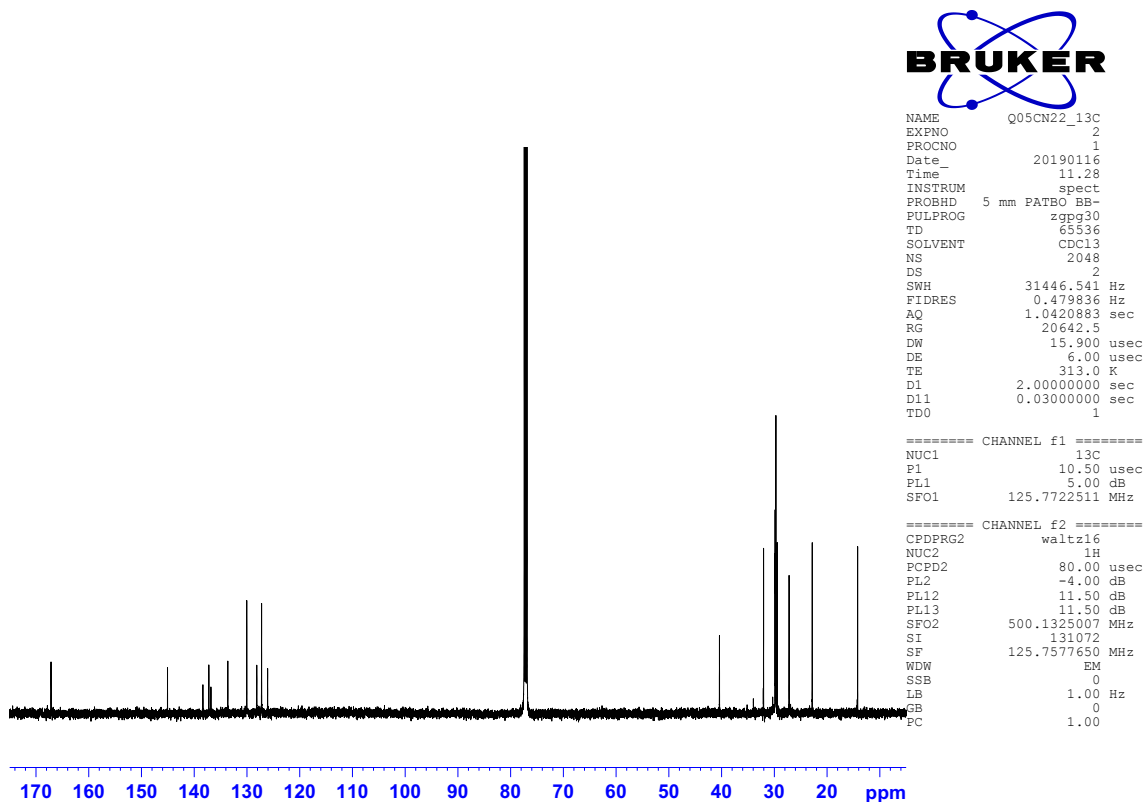
^{13}C NMR (CDCl_3 , 126 MHz, 298 K) of compound 1.



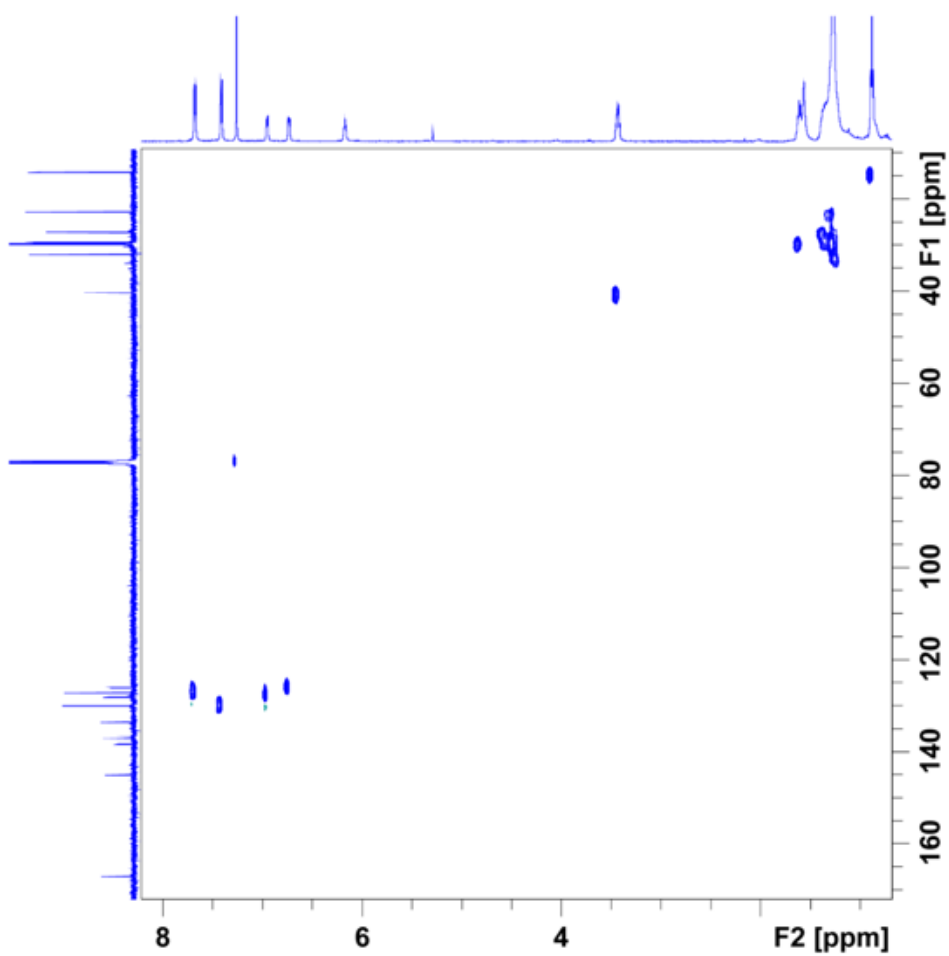
^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound 1.



^1H NMR spectrum (CDCl_3 , 500 MHz, 298 K) of compound 2.



^{13}C NMR spectrum (CDCl_3 , 126 MHz, 298 K) of compound 2.



^1H , ^{13}C -HMQC spectrum (CDCl_3 , 298 K) of compound **2**.