Supporting Information for

Synthesis and Properties of the *anti* and *syn* Isomers of Dibenzothieno[*b*,*d*]pyrrole

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Contents

1. Synthesis

General: Chemicals were purchased from Aldrich, Alfa Aesar and used as received. Solvents and other common reagents were obtained from the Beijing Chemical Plant. Diethyl ether and *o*-DCB were dried and distillated immediately prior to use. 2,2'-Bibenzo[*b*]thiophene[S1] (**2**), 3-bromobenzo[*b*]thiophene[S2] (**4**) and 3,3'-bibenzo[*b*]thiophene[S3] (**5**) was synthesized according to the literatures.

Melting points were measured with a WRR melting point apparatus. ^{1}H-NMR (400

MHz) and ^{13}C-NMR (75 and 100 MHz) spectra were obtained on a Bruker DMX-400 and DMX-300 NMR Spectrometer using tetramethylsilane as internal standard. High-resolution mass spectra (HRMS) and EI MS were both recorded on Micromass GCT-MS spectrometer. FT-IR spectra were determined using a Perkin-Elmer Tensor 27 spectrometer. Elemental analyses were performed on a Carlo Erba model 1160 elemental analyzer.

2,2'-Bibenzo[*b*]thiophene[S1] (**2**)

To a stirred solution of benzo[*b*]thiophene (4.15 g, 31 mmol) in 170 ml of anhydrous diethyl ether at room temperature was added dropwise a solution of n-BuLi (15.4 ml, 38.5 mmol) under argon atmosphere during 20 minutes. The reaction mixture was heated at reflux with stirring under an inert gas atmosphere for 6 h and then allowed to cool to room temperature. Anhydrous copper (II) chloride (4.16 g, 31 mmol) was then added in four portions and the mixture was again heated under reflux for 4 h. After being allowed to cool to room temperature, the mixture was left to stand for 1h. The dirty brown-green precipitate was filtered off, and the organic layer was washed with dilute HCl solution and water. The precipitate was washed several times with THF and the filtrate combined with the ether phase. The combined organic phases were dried with anhydrous $MgSO_4$, concentrated, and the residue was purified by recrystallization from toluene to get a light yellow solid 3.01 g (73%) of **2**: ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.82 (dd, J = 17.20 Hz, J = 7.20 Hz, 4H), 7.51 (s, 2H), 7.38 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 140.4, 139.6, 137.4, 125.1, 124.9, 123.9, 122.3, 121.6; EI-MS m/z (%) 266 (M^+, 100%).

3-Nitro-2-(benzothiophen-2-yl)benzothiophene (3).

To a solution of compound **2** (1.26 g, 4.7 mmol) in glacial AcOH (150 mL) at 60 °C was added slowly a mixture of fuming HNO_3 (100%, 0.4 mL) and glacial AcOH (20 mL). On heating the reaction mixture for 30 min at 60 °C, the initially formed precipitate redissolved. The solution was cooled down and poured into H_2O, and then the yellow precipitate was formed rapidly and collected by filtration. The obtained solid was washed with water and dried in vacuo to get a yellow solid (1.42 g, 97%): mp 133−135 °C; ^{1}HNMR (400 MHz, Acetone-d$_6$) δ 8.27 (d, J = 8.36 Hz, 1H), 8.15 (d, J = 7.96 Hz, 1H), 8.07 (m, 3H), 7.72 (m, 2H), 7.52 (m, 2H); ^{13}CNMR (75 MHz, CDCl$_3$) δ 128.3, 127.3, 127.1, 126.5, 125.4, 125.1, 124.1, 122.5, 122.3; EI-MS *m*/*z* (%) 311 (M$^+$, 100%); HRMS *m*/*z* Calcd for $C_{16}H_9NS_2$: 311.0075, Found: 311.0073; Anal. Calcd for $C_{16}H_9NO_2S_2$: C, 61.72; H, 2.91; N, 4.50. Found: C, 61.60; H, 3.03; N, 4.42.

3-bromobenzo[*b*]thiophene^{S2} (**4**)

NBS (4.48 g, 25 mmol) was added in small portions under darkness to a solution of benzo[*b*]thiophene (3.38 g, 25 mmol) in 100 mL of CHCl$_3$/glacial AcOH (1:1) at room temperature. The reaction mixture was stirred at room temperature for another 10 hours before being poured into water. After extraction with methylene chloride, the organic phase was separated, dried over anhydrous MgSO$_4$, and concentrated. The residue was purified by column chromatography (silica gel, eluent: petroleum) to get a colorless liquid 4.35 g (82%) of **4**: ^{1}H NMR (400 MHz, CDCl$_3$, ppm) δ 8.04 (d, J = 8.09 Hz, 1H), 7.84 (m, 2H), 7.57 (m, 2H); ^{13}C NMR (75 MHz, CDCl$_3$, ppm) δ 139.0, 137.9, 125.7, 125.4, 123.9, 123.5, 123.1, 108.1; EI-MS *m*/*z* (%) 212 (M$^+$, 100%).

3,3’-bibenzo[*b*]thiophene^{S3} (**5**)

2.5 M n-BuLi (8.8 ml, 22.0 mmol) was added dropwise to a solution of **4** (3.78 g, 17.7 mmol) in anhydrous diethyl ether (150 ml) at −78 °C under nitrogen with stirring. After stirring for 1 h at −78 °C, anhydrous copper (II) chloride (2.38 g, 17.7 mmol) was added in three portions. The reaction mixture was then warmed to room temperature and stirred overnight. The dirty deep gray precipitate was filtered off and washed with diethyl ether three times. The obtained solution was washed with dilute HCl solution and water, and then dried with anhydrous MgSO$_4$, concentrated. The residue was purified by column chromatography (silica gel, eluent: petroleum) to get a light yellow solid 1.60 g (68%) of **5**: ^{1}H NMR (400 MHz, CDCl$_3$, ppm) δ 7.96 (d, J = 8.24 Hz, 2H), 7.75 (d, J = 7.16 Hz, 2H), 7.55 (s, 2H), 7.41 (m, 4H); ^{13}C NMR (75

MHz, $CDCl_3$, ppm) δ140.4, 138.8, 131.6, 124.9, 124.8, 124.5, 123.4, 123.0; EI-MS *m*/*z* (%) 266 (M^+, 100%).

2-Nitro-3-(benzothiophen-3-yl)benzothiophene (6).

This compound was prepared from **5** according to the procedure for **3**. The reaction solution was poured into H_2O and extracted with CH_2Cl_2. The combined organic layers were washed with water, dried over anhydrous $MgSO_4$, filtered, and the solvent was removed in vacuo. The crude product was purified by column chromatography (silica gel, eluent: petroleum) to get a yellow solid in 95% yield: mp 146−148 °C; ^{1}HNMR (400 MHz, Acetone-d_6) δ 8.20 (d, *J* = 8.17 Hz, 1H), 8.13 (d, *J* = 7.38 Hz, 1H), 7.98 (s, 1H), 7.75 (t, *J* = 7.64 Hz, 1H), 7.62 (d, *J* = 8.08 Hz, 1H), 7.54 (t, *J* = 7.22 Hz, 1H), 7.47 (t, *J* = 8.52 Hz, 2H), 7.38 (t, *J* = 7.04 Hz, 1H); ^{13}CNMR (75 MHz, $CDCl_3$) δ 130.0, 127.5, 127.2, 126.4, 125.3, 125.1, 123.3, 123.1, 122.9; EI-MS *m*/*z* (%) 311 (M^+, 100%); HRMS *m*/*z* Calcd for $C_{16}H_9NS_2$: 311.0075, Found: 311.0072; Anal. Calcd for $C_{16}H_9NO_2S_2$: C, 61.72; H, 2.91; N, 4.50. Found: C, 61.74; H, 3.05; N, 4.51.

Dibenzothieno[3,2-*b*:2',3'-*d*]pyrrole (*anti*-1).

A mixture of compound **3** (280 mg, 0.90 mmol), PPh_3 (590 mg, 2.25 mmol) and *o*-DCB (7 mL) was heated under reflux in an inert atmosphere. After 20 h, the reaction was cooled to R. T. The solvent was distilled off and the crude product was purified by column chromatography (silica gel, eluent: petroleum/CH_2Cl_2 = 1:1) to provide 196 mg of the title compound as a light yellow solid (78%): mp 264−268 °C; IR (KBr) 3413, 3049, 1592, 1503, 1463, 1435, 1396, 1292, 1098, 1054, 1018, 851, 746, 720, 441 cm^{-1}; ^{1}HNMR (400 MHz, Acetone-d_6) δ 11.94 (s, 1H), 7.98 (dd, *J* = 19.04 Hz, *J* = 7.88 Hz, 4H), 7.43 (t, *J* = 7.54 Hz, 2H), 7.32 (t, *J* = 7.60 Hz, 2H); ^{13}CNMR (75 MHz, Acetone-d_6) δ 143.3, 139.5, 129.6, 126.5, 126.1, 125.6, 120.9, 116.7; EI-MS *m*/*z* (%) 279 (M^+, 100%); HRMS *m*/*z* Calcd for $C_{16}H_9NS_2$: 279.0176, Found: 279.0179. Anal. Calcd for $C_{16}H_9NS_2$: C, 68.79; H, 3.25; N, 5.01. Found: C, 68.78; H, 3.29; N, 4.96.

Dibenzothieno[2,3-*b*:3',2'-*d*]pyrrole (*syn*-1).

This compound was prepared from **6** according to the procedure for ***anti*-1**. The crude product was purified by column chromatography (silica gel, eluent: petroleum/CH_2Cl_2 = 1:1) to get the title compound as a light yellow solid (51%): mp 179−181 °C; IR (KBr) 3061, 2920, 2235, 1577, 1533, 1473, 1433, 1366, 1311, 1260, 1183, 1135, 1097, 1068, 1032, 995, 930, 842, 753, 728, 679, 605, 460, 419 cm^{-1}; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.04 Hz, 2H), 7.57 (m, 1H), 7.50 (m, 2H), 7.40 (m, 3H), 5.63 (m, 1H); ^{13}CNMR (75 MHz, Acetone-d_6) δ 143.3, 139.5, 129.6, 126.5, 126.1, 125.5, 120.9, 116.7, 31.2; EI-MS *m*/*z* (%) 278 ($(M-1)^+$, 100%), 279 (M^+, 75%); HRMS *m*/*z* Calcd for $C_{16}H_9NS_2$: 279.0176, Found: 279.0172. Anal. Calcd for $C_{16}H_9NS_2$: C, 68.79; H, 3.25; N, 5.01. Found: C, 68.56; H, 3.21; N, 5.12.

_N_-Hexyldibenzothieno[3,2-_b_:2',3'-_d_]pyrrole (7).

To a solution of compound ***anti*-1** (157 mg, 0.56 mmol) in 50 mL of DMF was added KOH (158 mg, 2.82 mmol). After the solution was stirred at 50 °C for 2 h under nitrogen, 1-bromohexane (0.1 ml, 0.71 mmol) was added. The mixture was stirred at 50 °C for 24 h and then quenched with water. The aqueous layer was extracted three times with diethyl ether. The organic fractions were dried over $MgSO_4$ and the solvent was removed under reduced pressure. The residue was purified by column chromatography (silica gel, eluent: petroleum) to get a light yellow solid 128 mg (63%) of **7**: mp 134−136 °C; ^{1}HNMR (400 MHz, Acetone-d_6) δ 8.15 (d, J = 8.04 Hz, 2H), 7.97 (d, J = 8.04 Hz, 2H), 7.48 (t, J = 7.60 Hz, 2H), 7.35 (t, J = 7.62 Hz, 2H), 4.98 (t, J = 7.42 Hz, 2H), 2.13 (m, 2H), 1.61 (m, 2H), 1.40 (m, 4H), 0.83 (t, J = 7.23 Hz, 3H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 142.0, 137.6, 127.7, 124.6, 124.5, 123.3, 118.9, 114.7, 47.6, 31.7, 31.3, 26.7, 22.7, 14.1; EI-MS *m*/*z* (%) 363 (M^+, 100%); Anal. Calcd for $C_{16}H_9NS_2$: C, 72.68; H, 5.82; N, 3.85. Found: C, 72.53; H, 5.90; N, 4.02.

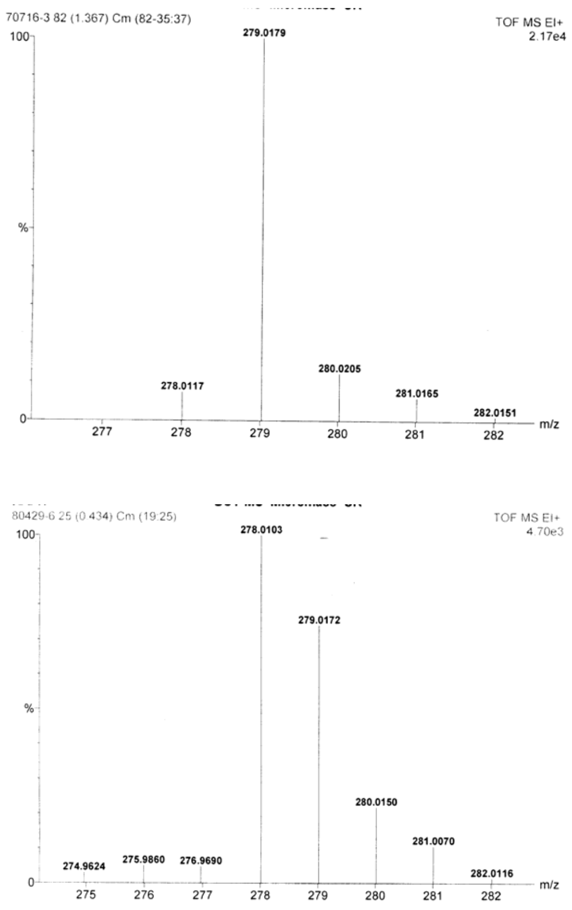


Figure S1. High-resolution mass spectra of ***anti*-1** (top) and ***syn*-1** (bottom).

Seen from the conjugated degree of the whole molecule, the conjugated degree of ***anti*-1** is larger than that of ***syn*-1**. ***anti*-1** molecule has stable electronic structure, but ***syn*-1** molecule is not the most stable structure. To extend the conjugated degree of ***syn*-1**, it forms the ***syn*-1'** structure. Seen from Figure S1, we found the difference of their mass spectra. ***anti*-1** forms the strongest molecular ion peak, while ***syn*-1** forms the strongest fractional ion peak when a hydrogen atom leaves. This indicates the ***syn*-1'** structure, because only the hydrogen at the carbon near to the nitrogen is easier to leave in mass spectrum. ***syn*-1'** without one H atom forms a stable electronic structure, while the ***syn*-1** structure could not obtain the more stable structure by this way. For ***anti*-1**, the structure with the H at N atom is most stable and has the largest conjugation, so other forms are all not reasonable.

We simulated the ^{1}H-NMR spectrum of ***syn*-1'** structure by ChemDraw (Figure S2), and obviously, we can classify four groups of aromatic H atoms ([δ= 7.02(1H), 7.04(1H)]; [δ= 7.18 (2H)]; [δ= 7.26 (1H)]; [δ= 7.32(1H), 7.33(2H)]), which is consistent with our reported result (four groups of aromatic H atoms). Therefore, it can further confirm that the speculated ***syn*-1'** structure is reasonable.

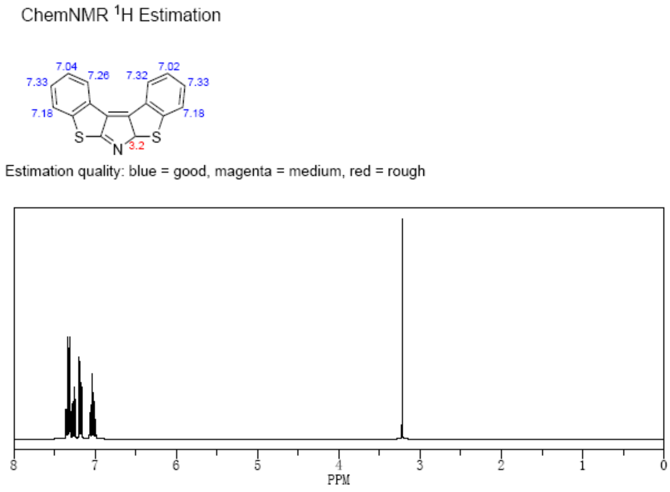


Figure S2. The simulated ^{1}H-NMR spectrum of ***syn*-1'** structure by ChemDraw.

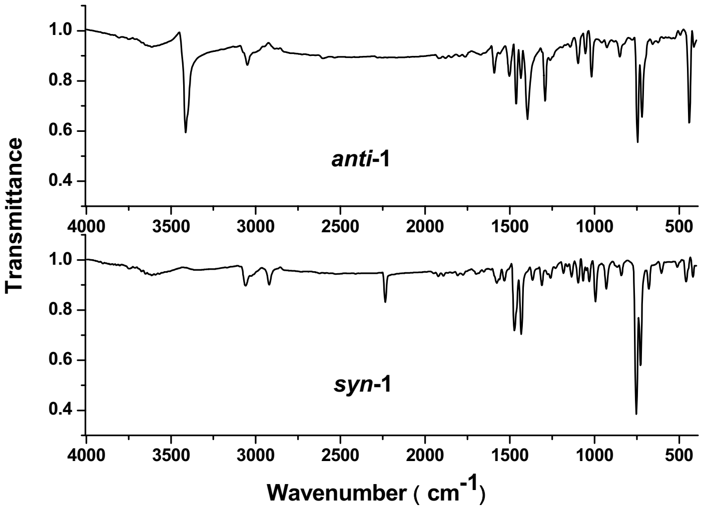


Figure S3. Infrared spectra of ***anti*-1** and ***syn*-1**.

2. Single-crystal X-ray analysis of *anti*-1 and 7

The measurement was made on a Rigaku MM-007 diffractometer with Mo-Kα radiation (λ = 0.71073 Å). The structure was solved by direct methods and

SHELXS-97, and refined by using SHELXL-97. Hydrogen atoms were located at the calculated positions. Absorption correction was applied using semi-empirical from equivalents.

***anti*-1**, $C_{16}H_9NS_2$, M = 279.36, crystal dimension 0.39 × 0.36 × 0.15 mm, monoclinic, space group $P2_1/n$, a = 9.916(2), b = 10.619(2), c = 23.425(5) Å, α = 90.00, β = 96.04(3), γ = 90.00°, V = 2453.0(8) Å^3, Z = 8, D_c = 1.513 g·cm^{-3}, T = 173(2) K, μ = 0.415 mm^{-1}, θ range 2.11–25.00°, 7940 reflection collected, 4328 of which were independent (R_{int} = 0.0199), GOF = 1.168, 343 parameters, R_1 = 0.0623, wR_2 = 0.0993 for all reflections. CCDC 697266.

7, $C_{22}H_{21}NS_2$, M = 363.52, crystal dimension 0.14 × 0.12 × 0.04 mm, monoclinic, space group $P2_1/c$, a = 11.582(2), b = 8.1596(16), c = 19.770(4) Å, α = 90.00, β = 106.89(3), γ = 90.00°, V = 1787.7(6) Å^3, Z = 4, D_c = 1.351 g·cm^{-3}, T = 113(2) K, μ = 0.302 mm^{-1}, θ range 1.84– 27.87°, 12637 reflection collected, 4248 of which were independent (R_{int} = 0.0717), GOF = 1.033, 227 parameters, R_1 = 0.0567, wR_2 = 0.1106 for all reflections. CCDC 697267.

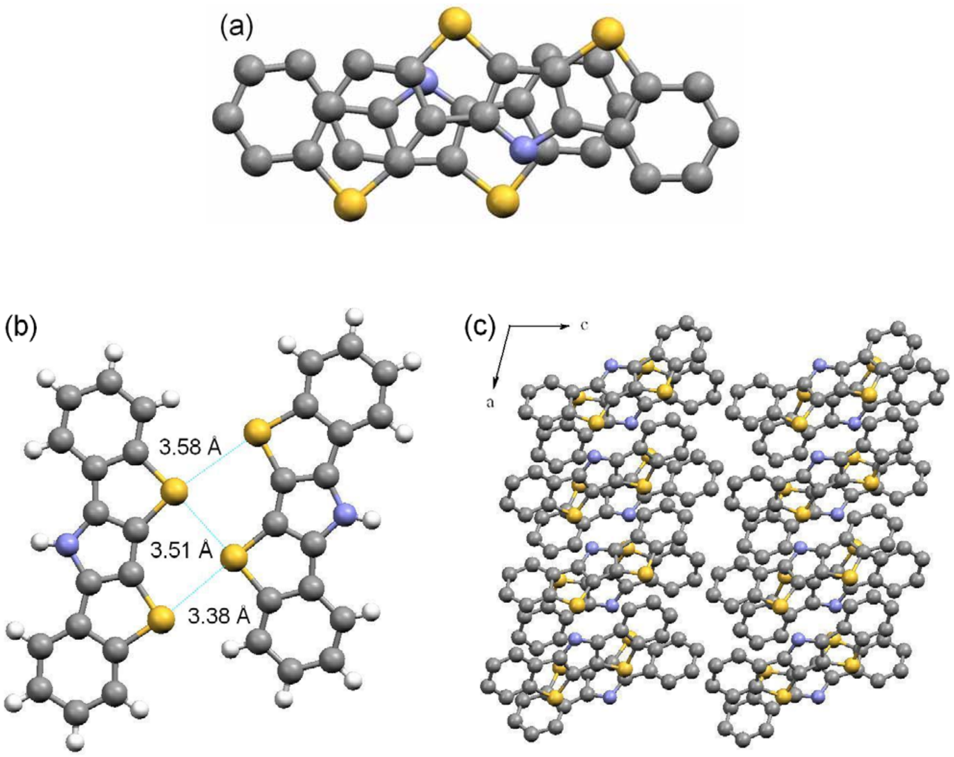


Figure S4. The crystal structure of ***anti*-1**, (a,c) hydrogen atoms removed for clarity.

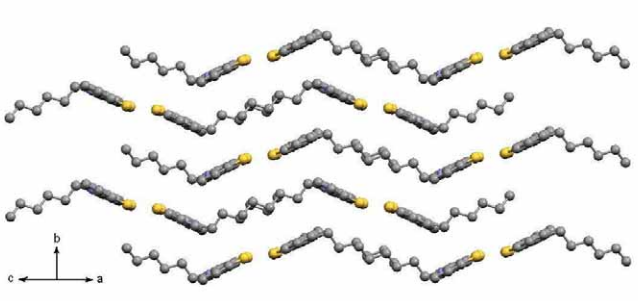


Figure S5. The crystal packing structure of **7**, hydrogen atoms removed for clarity.

3. Physicochemical properties

Methods: TGA measurements were carried out on a TA SDT 2960 instruments under a dry nitrogen flow, heating from room temperature (R. T.) to 500 °C, with a heating rate of 10 °C/min. Electronic absorption spectra were measured on a Jasco V570 UV-vis spectrophotometer. Emission spectra were recorded on Hitachi F-4500 fluorescence spectrometer. Cyclic voltammetric measurements were carried out in a conventional three-electrode cell using Pt button working electrodes of 2 mm diameter, a platinum wire counter electrode, and a Ag/AgCl reference electrode on a computer-controlled CHI660C instruments at R. T. All the potentials were calibrated with the standard ferrocene/ferrocenium redox couple ($E^{1/2}$ = +0.46 V measured under identical conditions).

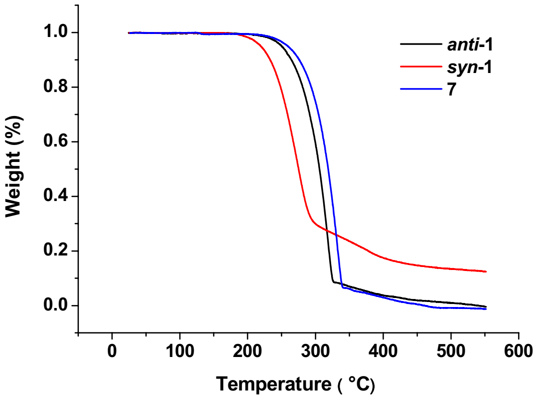


Figure S6. TGA scan of ***anti*-1**, ***syn*-1** and **7**.

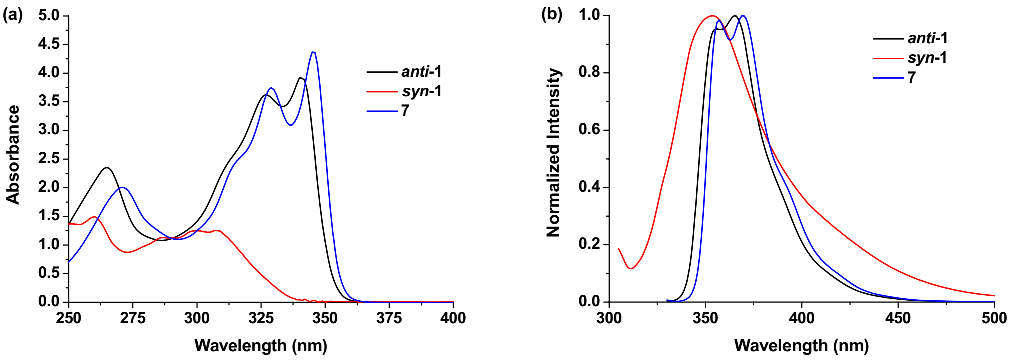


Figure S7. Absorption (a) and fluorescence (b) spectra of ***anti*-1**, ***syn*-1** and **7** in CH_2Cl_2.

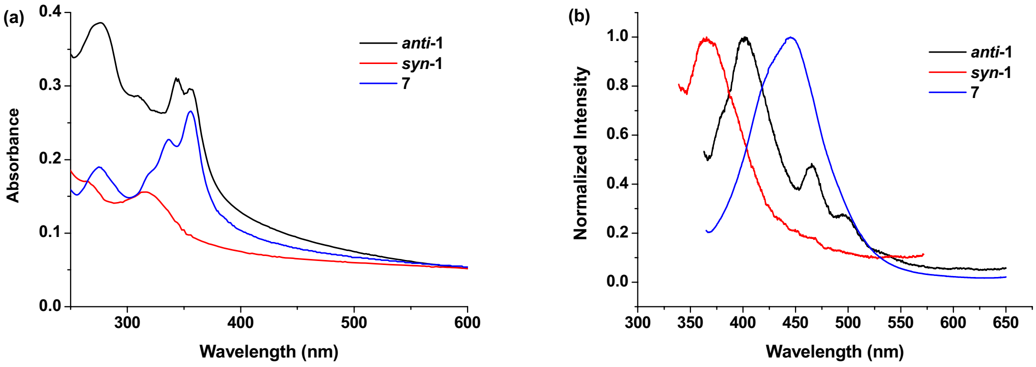


Figure S8. Absorption (a) and fluorescence (b) spectra of ***anti*-1**, ***syn*-1** and **7** for vapor deposited films on the quartz substrate.

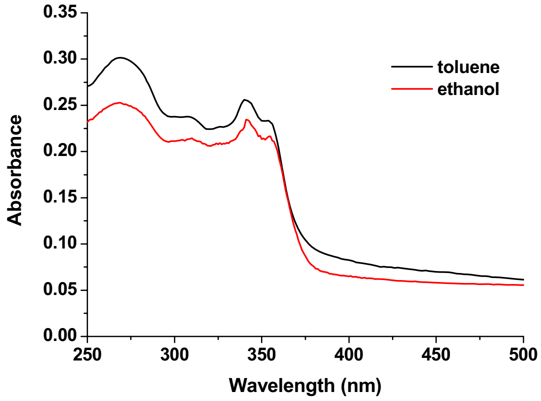


Figure S9. Absorption spectra of ***anti*-1** for drop coating films on the quartz substrate.

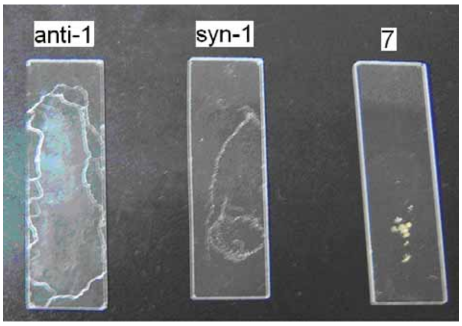


Figure S10. The picture of ***anti*-1**, ***syn*-1** and **7** for drop coating films from toluene solution (1 mg/mL) on the quartz substrate.

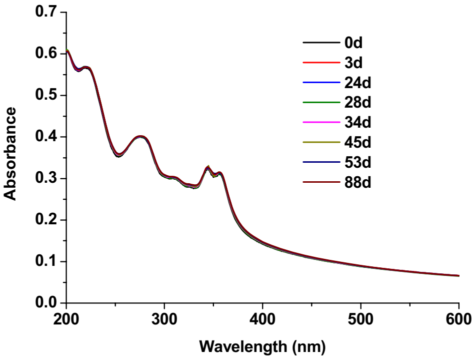


Figure S11. The photostability of ***anti*-1** thin film deposited on quartz.

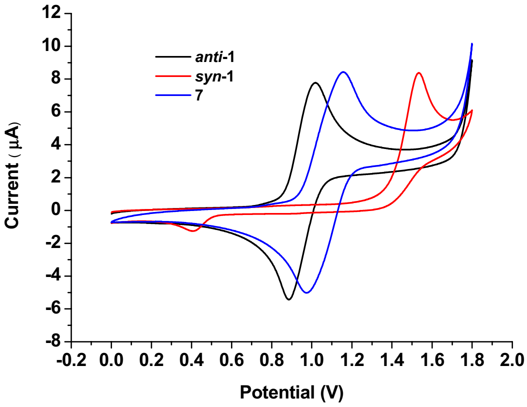


Figure S12. Cyclic voltammograms of ***anti*-1**, ***syn*-1** and **7** ($c = 10^{-3}$ M) in CH_2Cl_2 containing Bu_4NPF_6 (0.01 M) as supporting electrolyte under nitrogen at room temperature, with a scan rate of 100 mV/s.

4. X-ray Diffraction Studies

X-ray diffraction (XRD) measurements were carried out with a 2-kW Rigaku x-ray diffraction system. XRD patterns were obtained using bragg-Brentano Geometry (θ−2θ) with Cu Kαradiation as an X-ray source in the reflection mode at 45 kV and 300 mA.

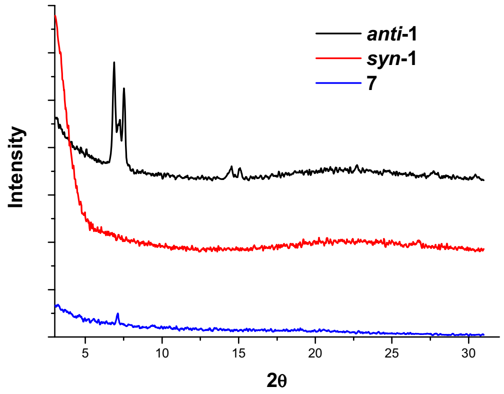


Figure S13. X-ray diffraction of thin films deposited on OTS-treated Si/SiO_2 substrate (film thickness: 50 nm).

5. AFM Studies

AFM measurements were made in air using a Digital Instruments Nanoscope III in contacting mode.

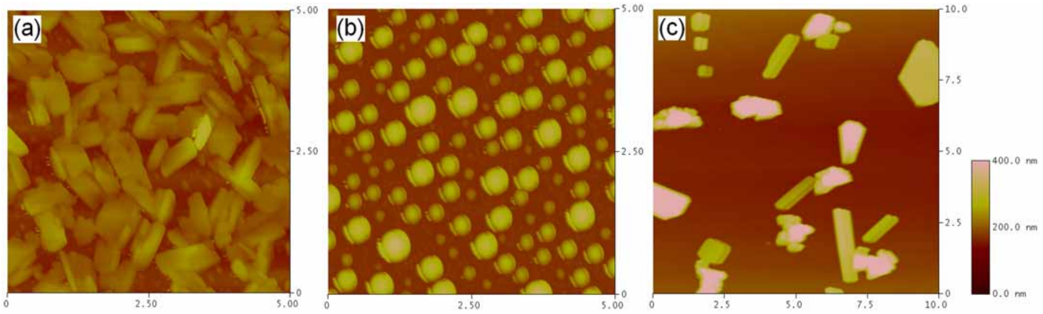


Figure S14. AFM images of thin films deposited on OTS-treated Si/SiO_2 substrate (film thickness: 50 nm): (a) ***anti*-1**, (b) ***syn*-1** and (c) **7**.

6. Device fabrications and evaluations

FET devices were fabricated with a top-contact configuration. A thin film of about 50 nm was vacuum deposited on octadecyltrichlorosilane (OTS) treated Si/SiO_2 substrate at room temperature. An n-type Si wafer with a SiO_2 layer of 500 nm and a capacitance of 7.5 nF cm^{-2} was used as the gate, and gold source and drain contacts (50 nm) were deposited on the organic layer through a shadow mask. The channel length (*L*) and width (*W*) were 50 μm and 3 mm, respectively. The FET measurements were carried out at room temperature in air using a HP 4140B semiconductor parameter analyzer. The mobility of devices based on ***anti*-1** was calculated in the saturation regime. The equation was listed as follow:

$$I_{\mathrm{DS}} = (W/2L)C_{\mathrm{i}}\mu(V_{\mathrm{GS}} - V_{\mathrm{th}})^2$$

Where μ is the field-effect mobility, L and W are the channel length and width, respectively, C_{i} is the insulator capacitance per unit area, and V_{GS} and V_{th} is the gate voltage and threshold voltage, respectively.

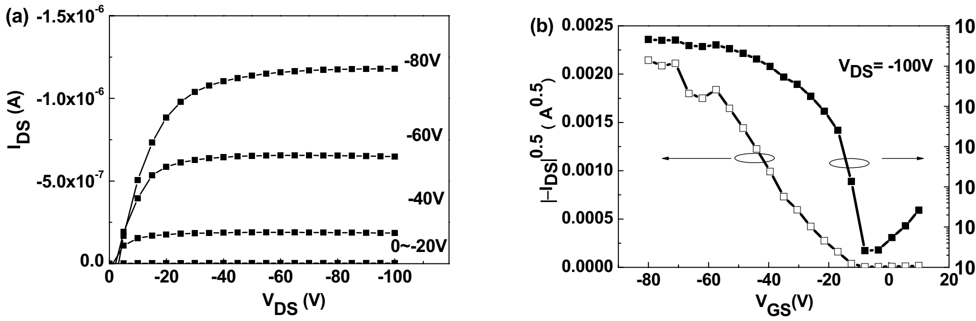


Figure S15. FET characteristics of device based on ***anti*-1** thin film deposited on OTS/SiO$_2$/Si substrate under ambient conditions. (a) Output curves at different gate voltages. (b) Transfer curve in the saturated regime at a constant source–drain voltage of −100 V (black squares) and the square root of the absolute value of the current as a function of the gate voltage (white squares).

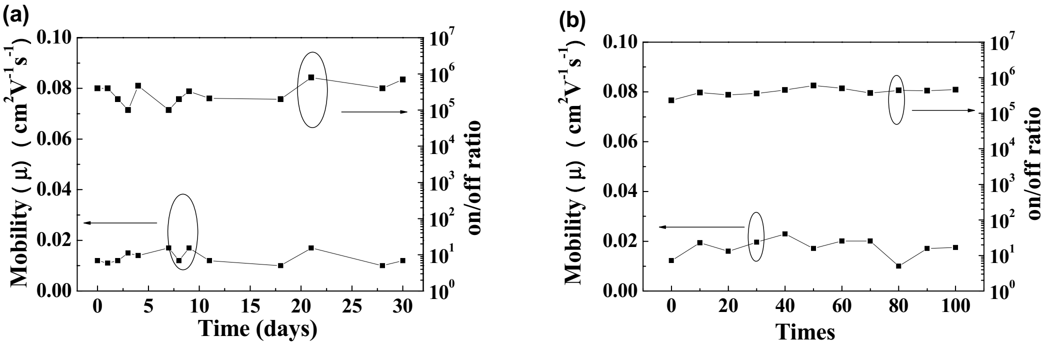


Figure S16. Mobilities and on/off ratios for a FET device based on ***anti*-1** tested (a) over a period of one month in air; (b) for 100 times.

7. References

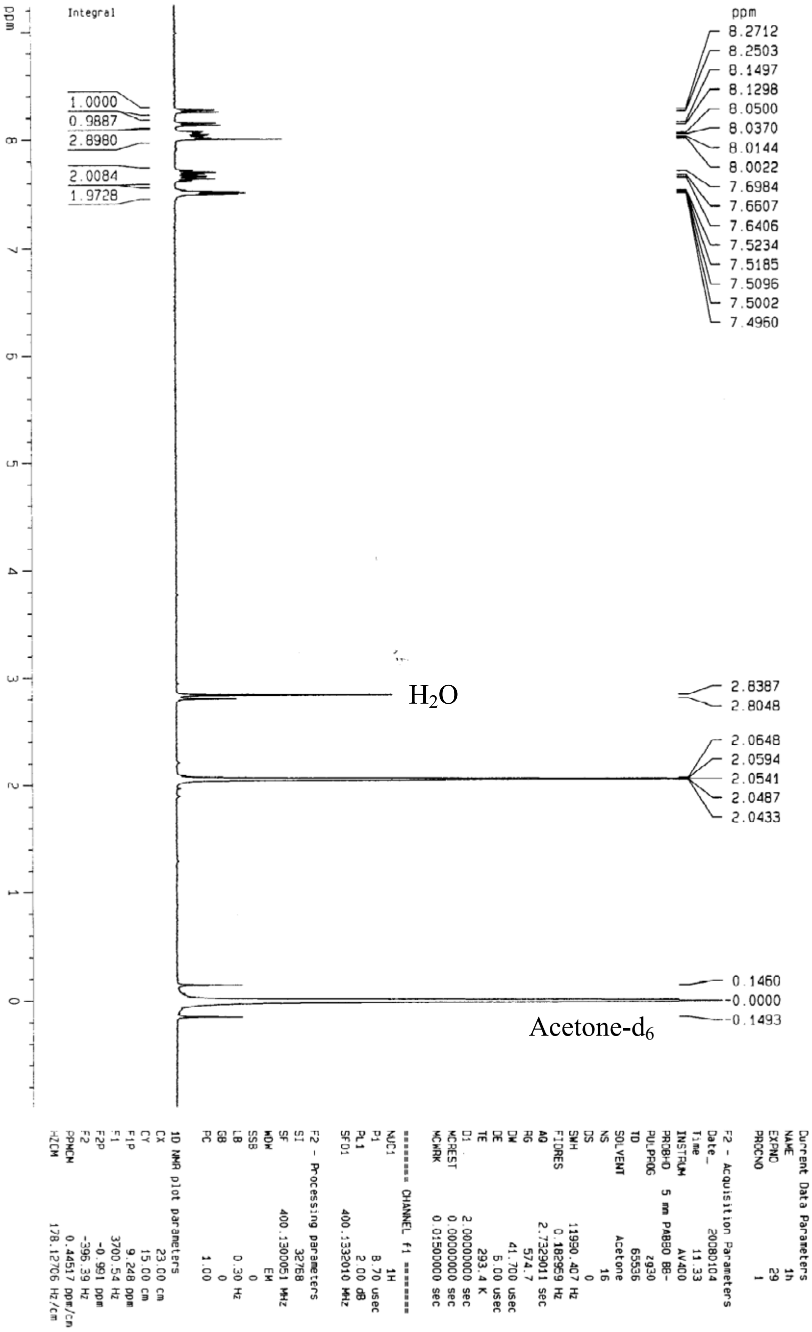
S1. Pandya et al. *J. Sci. Ind. Res.* **1959**, *18 B*, 516.

S2. Samusakovics, J.; Modest, E. *J. Am. Chem. Soc.* **1950**, *72*, 571.

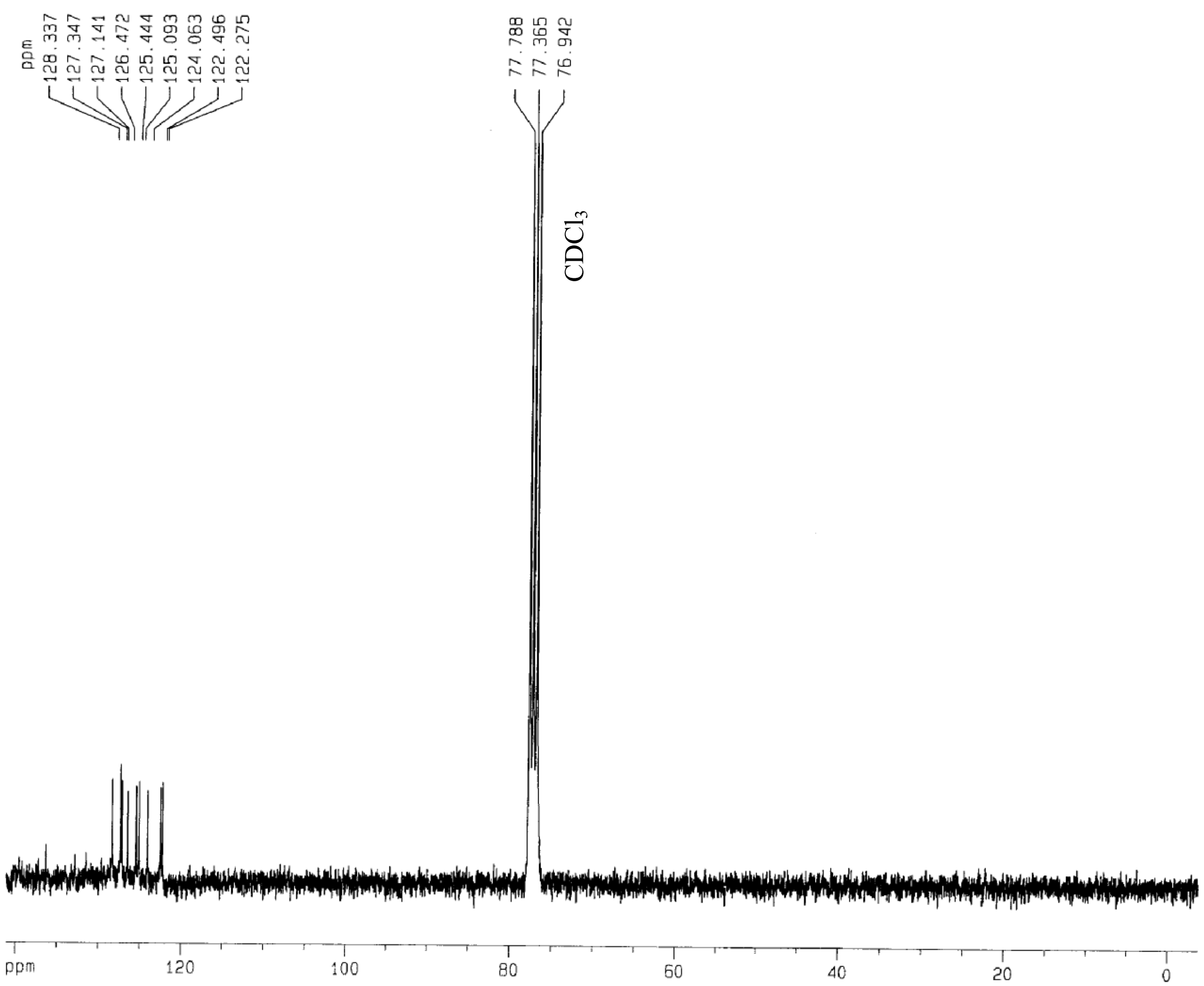
S3. Benincori, T.; Brenna, E.; Sannicolo, F.; Trimarco, L.; Antognazza, P.; Cesarotti, E.; Demartin, F.; Pilati, T. *J. Org. Chem.* **1996**, *61*, 6244.

8. ^{1}H and ^{13}C NMR spectra

^{1}H NMR spectrum of **3**



^{13}C NMR spectrum of **3**



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PROCNO                1

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TD                65536
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DS                    2
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FIDRES         0.274439 Hz
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DW               27.800 usec
DE                30.00 usec
TE                  0.0 K
D1           2.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
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PL1                4.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
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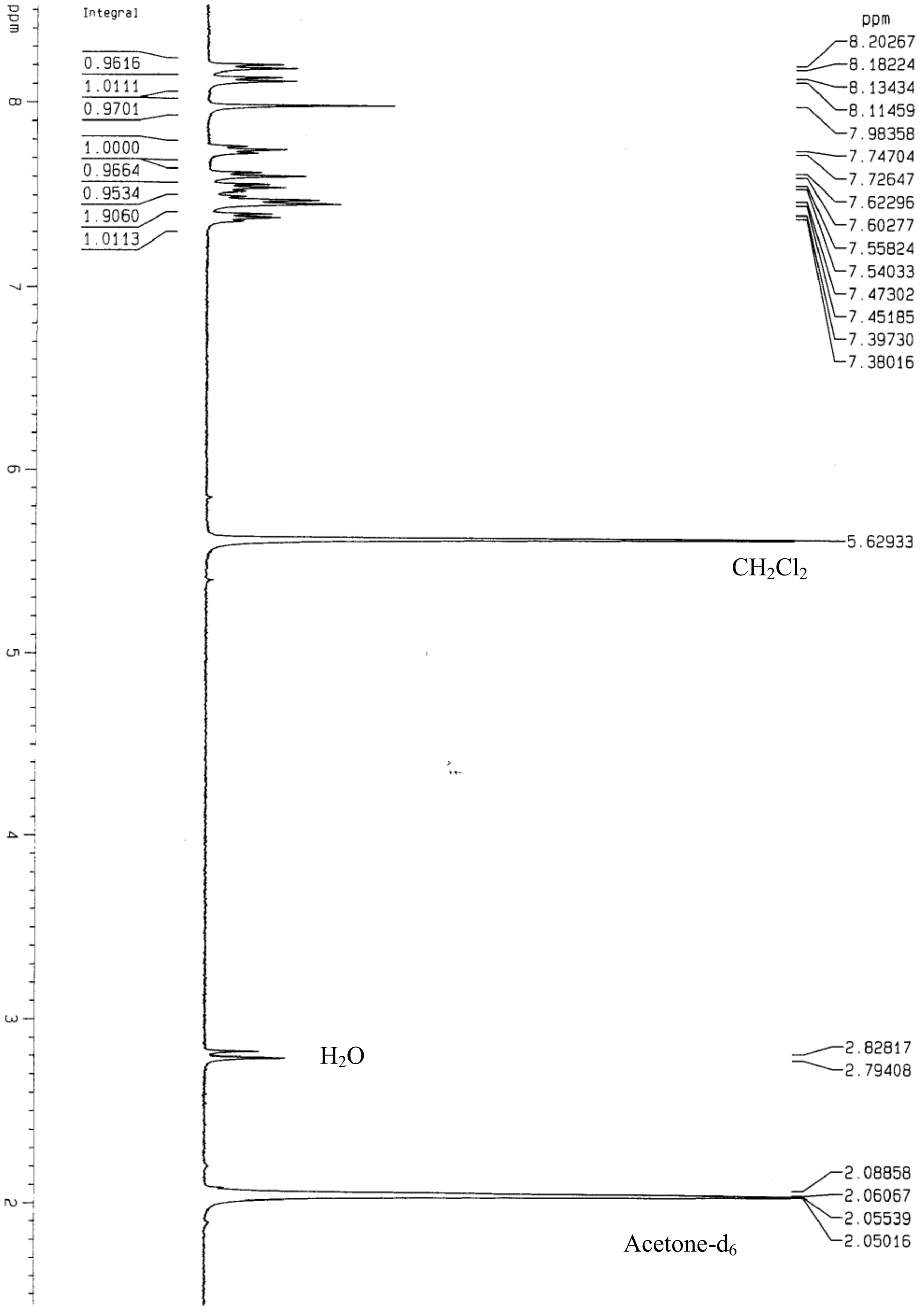
NUC2                 1H
PCPD2             88.00 usec
PL2                4.00 dB
PL12              19.08 dB
PL13              19.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF           75.4677210 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 2.00 cm
F1P             141.199 ppm
F1             10655.99 Hz
F2P              -3.752 ppm
F2              -283.16 Hz
PPMCM           7.24756 ppm/cm
HZCM          546.95721 Hz/cm
```

^{1}H NMR spectrum of **6**



```
Current Data Parameters
NAME                 1h
EXPNO                22
PROCNO                1

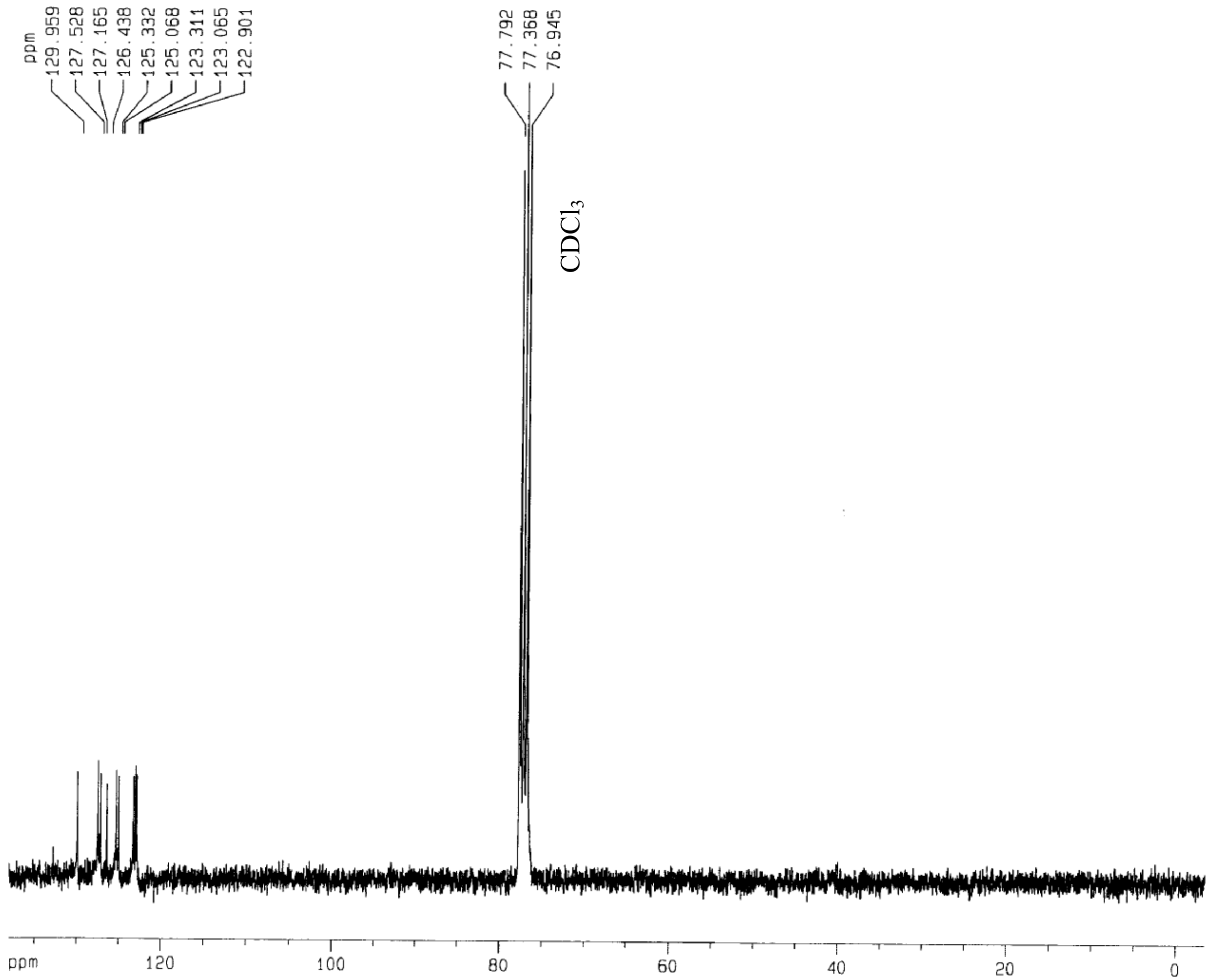
F2 - Acquisition Parameters
Date_          20071214
Time              17.39
INSTRUM           AV400
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                   16
DS                    0
SWH          11990.407 Hz
FIDRES        0.182959 Hz
AQ           2.7329011 sec
RG               1149.4
DW               41.700 usec
DE                 6.00 usec
TE                295.4 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.70 usec
PL1                2.00 dB
SFO1        400.1332010 MHz

F2 - Processing parameters
SI                32768
SF          400.1300049 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                23.00 cm
CY                25.00 cm
F1P               8.528 ppm
F1              3412.43 Hz
F2P               1.442 ppm
F2               577.03 Hz
PPMCM           0.30810 ppm/cm
HZCM          123.27856 Hz/cm
```

^{13}C NMR spectrum of **6**



```
Current Data Parameters
NAME                 13c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080516
Time               22.12
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   808
DS                     2
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                 16384
DW                27.800 usec
DE                 30.00 usec
TE                   0.0 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

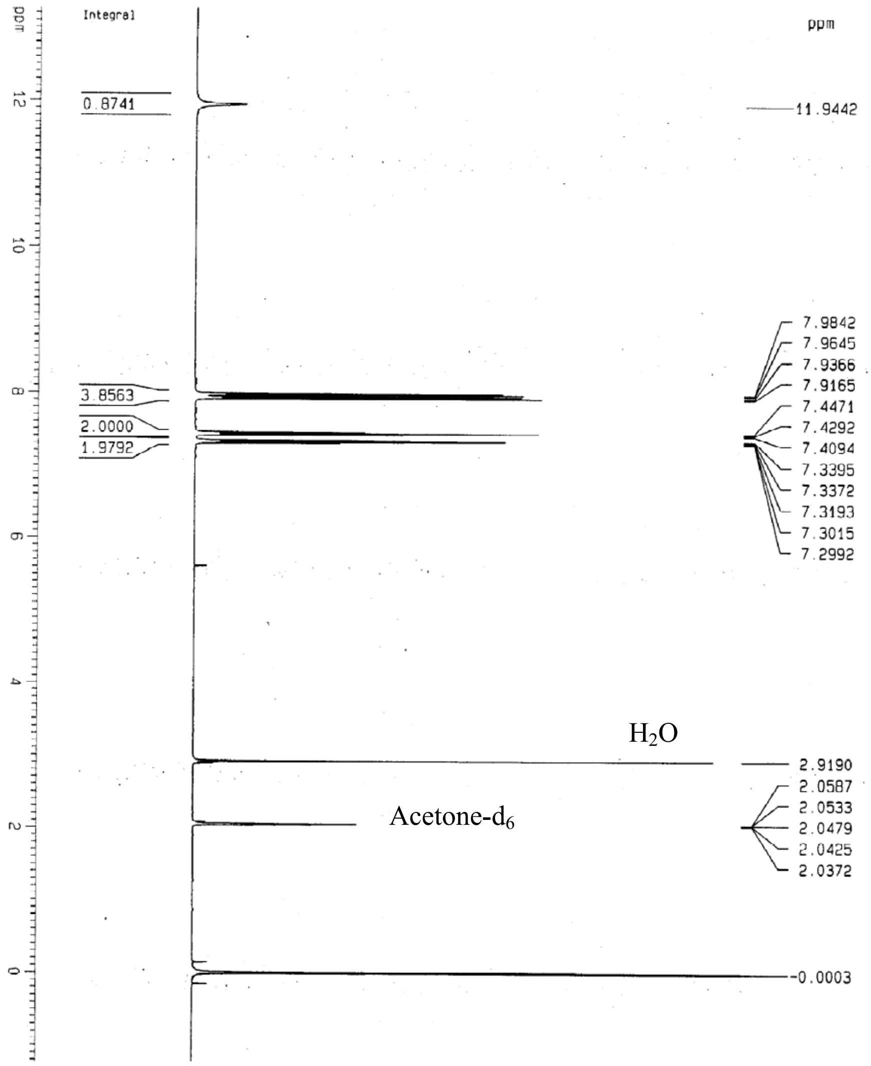
======== CHANNEL f1 ========
NUC1                 13C
P1                 12.00 usec
PL1                 4.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              88.00 usec
PL2                 4.00 dB
PL12               19.08 dB
PL13               19.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677210 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  2.00 cm
F1P              138.095 ppm
F1              10421.71 Hz
F2P               -3.513 ppm
F2               -265.14 Hz
PPMCM            7.08041 ppm/cm
HZCM           534.34210 Hz/cm
```

^{1}H NMR spectrum of ***anti*-1**



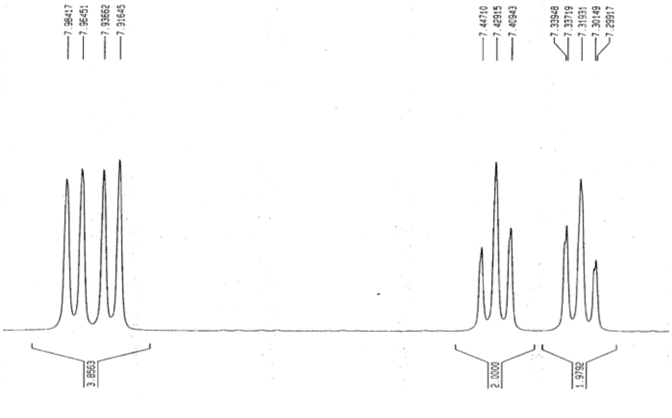
```
Current Data Parameters
NAME                 1h
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080703
Time              16.17
INSTRUM           AV400
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                55536
SOLVENT         Acetone
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ            3.9584243 sec
RG                  114
DW               60.400 usec
DE                 6.00 usec
TE                300.4 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

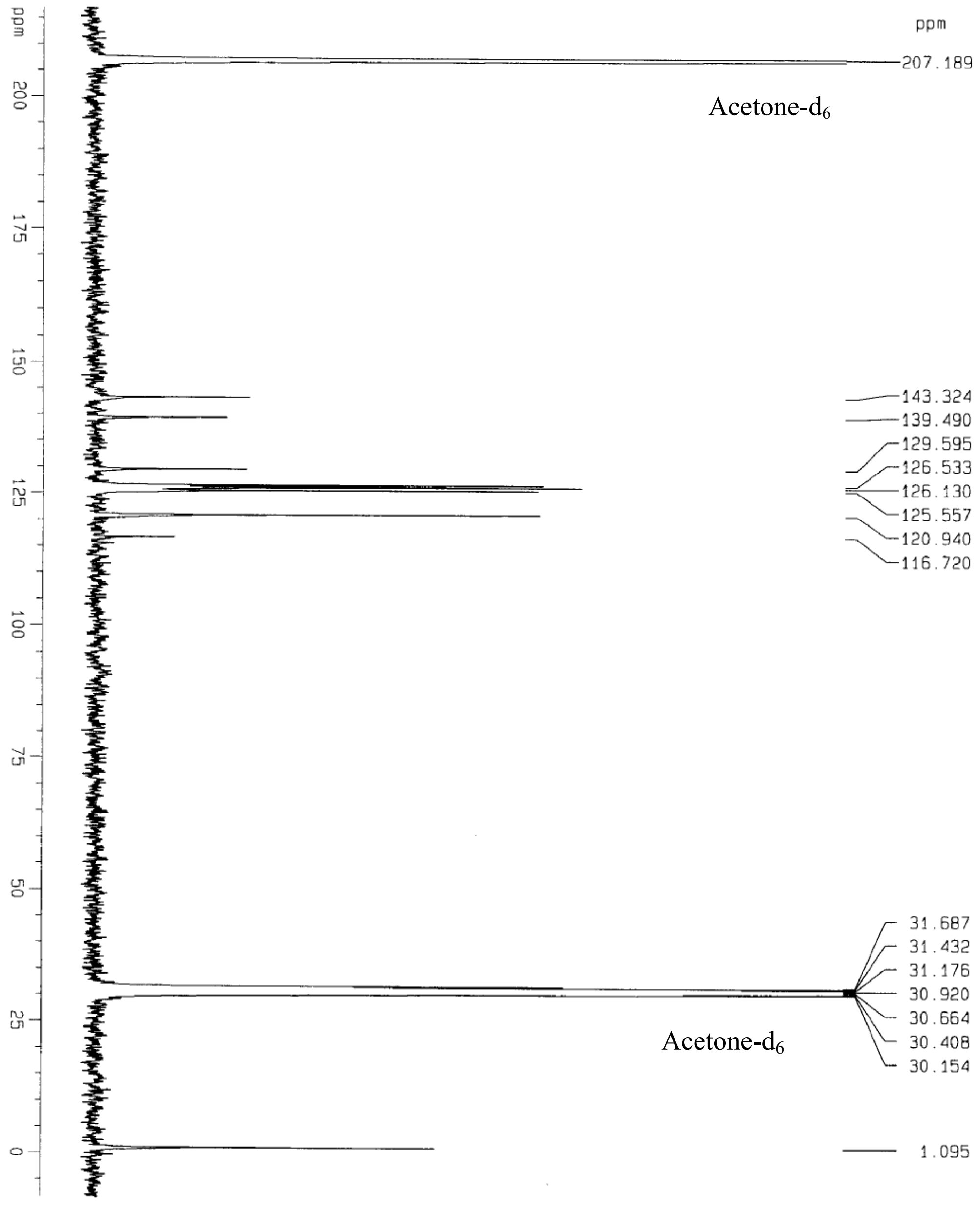
======== CHANNEL f1 ========
NUC1                 1H
P1                13.50 usec
PL1               -1.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300076 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P              13.267 ppm
F1              5308.48 Hz
F2P              -1.223 ppm
F2              -489.54 Hz
PPMCM           0.72452 ppm/cm
HZCM          289.90097 Hz/cm
```



^{13}C NMR spectrum of ***anti*-1**



```
Current Data Parameters
NAME                 13c
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080519
Time               12.00
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgig30
TD                 65536
SOLVENT          Acetone
NS                  1448
DS                     2
SWH            18939.395 Hz
FIDRES          0.288992 Hz
AQ             1.7302004 sec
RG                 16384
DW                26.400 usec
DE                 30.00 usec
TE                   0.0 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

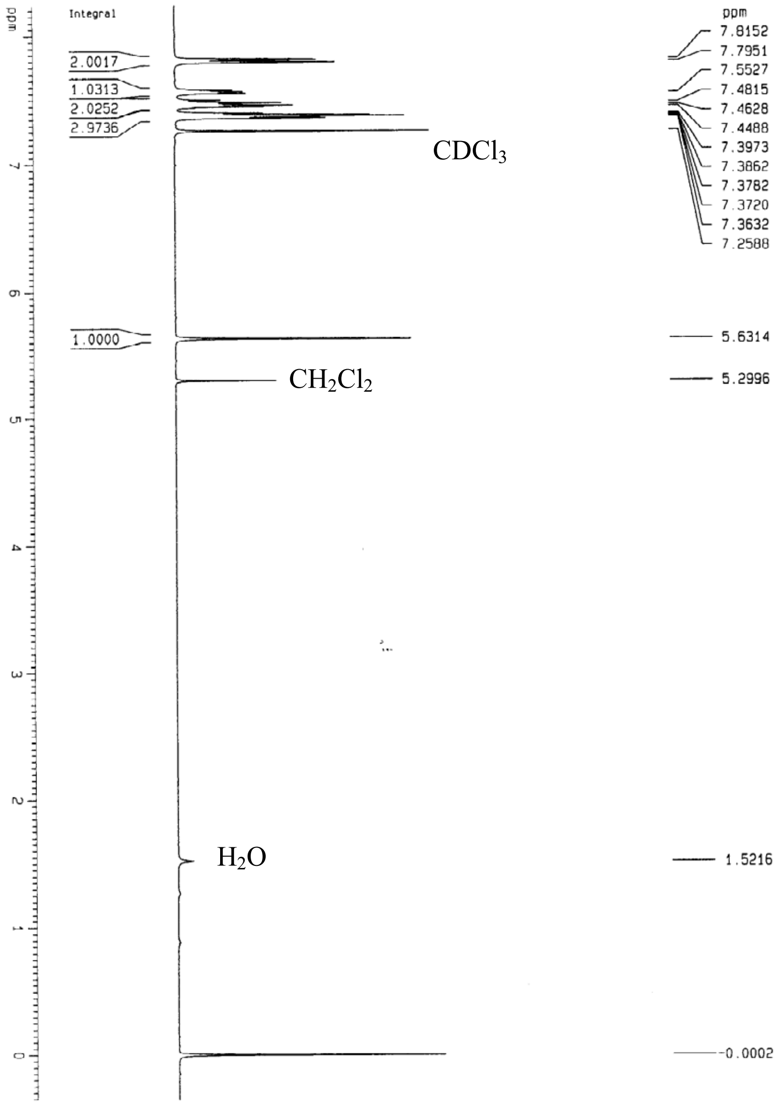
======== CHANNEL f1 ========
NUC1                 13C
P1                 12.00 usec
PL1                 4.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              88.00 usec
PL2                 4.00 dB
PL12               19.08 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4675993 MHz
WDW                   EM
SSB                    0
LB                  5.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                100.00 cm
F1P              216.896 ppm
F1              16368.65 Hz
F2P               -8.415 ppm
F2               -635.06 Hz
PPMCM           11.26557 ppm/cm
HZCM           850.18518 Hz/cm
```

^{1}H NMR spectrum of ***syn*-1**



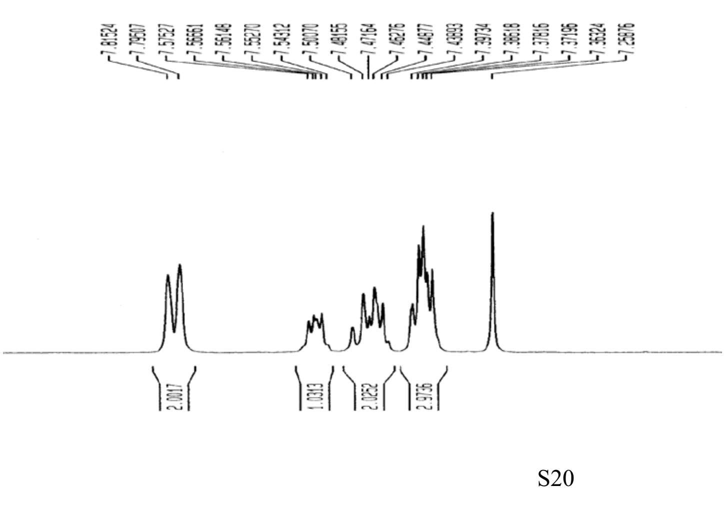
```
Current Data Parameters
NAME                1h
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20080110
Time              9.24
INSTRUM          AV400
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  32
DS                   0
SWH          11990.407 Hz
FIDRES        0.182959 Hz
AQ           2.7329011 sec
RG               912.3
DW              41.700 usec
DE                6.00 usec
TE               292.8 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

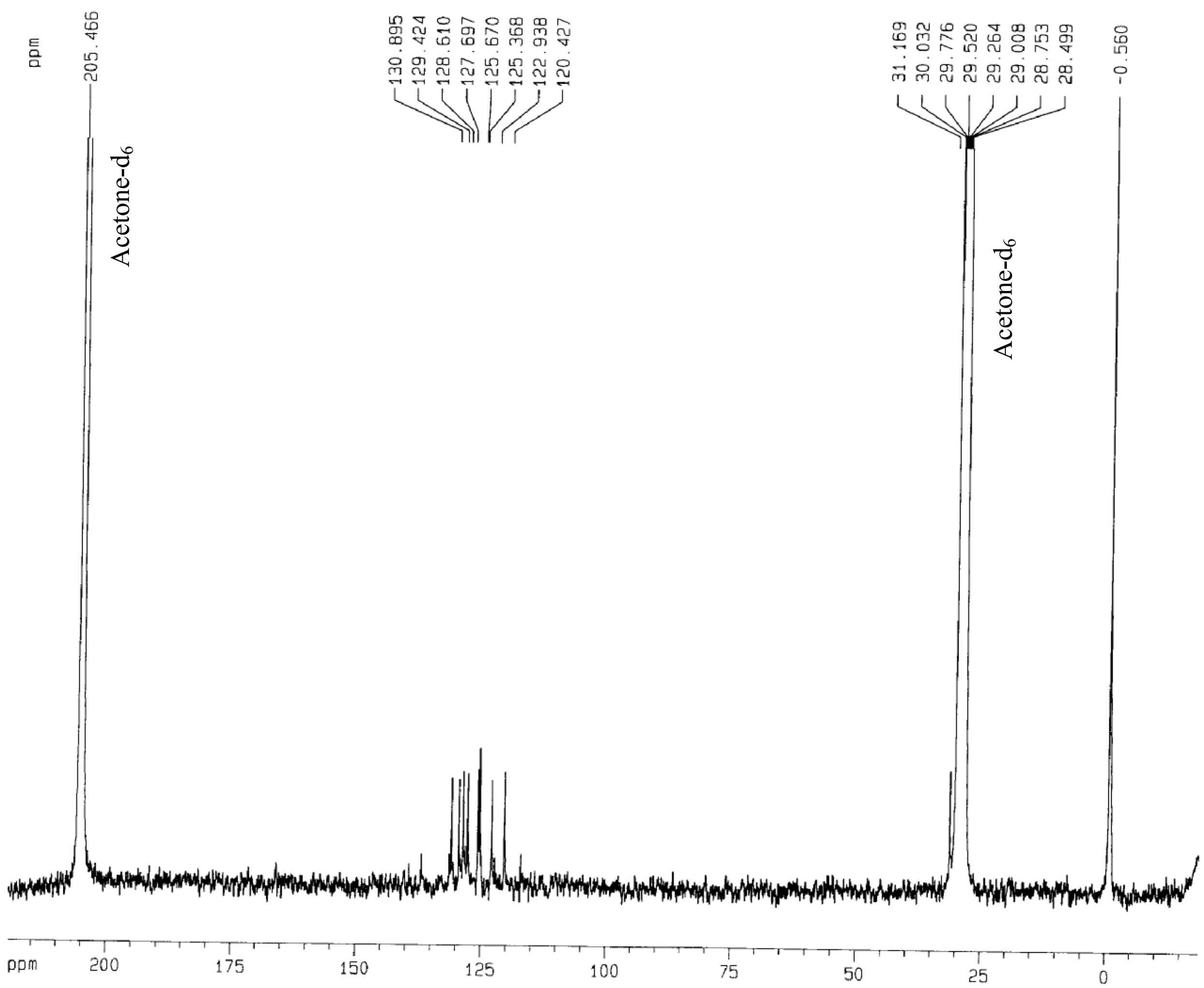
======== CHANNEL f1 ========
NUC1                1H
P1                8.70 usec
PL1               2.00 dB
SFO1       400.1332010 MHz

F2 - Processing parameters
SI               32768
SF         400.1300102 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               23.00 cm
CY                5.00 cm
F1P              8.237 ppm
F1             3295.93 Hz
F2P             -0.353 ppm
F2             -141.21 Hz
PPMCM          0.37348 ppm/cm
HZCM         149.44089 Hz/cm
```



^{13}C NMR spectrum of ***syn*-1**



```
Current Data Parameters
NAME                13c
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080516
Time              23.32
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 8397
DS                    2
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG                16384
DW               27.800 usec
DE                30.00 usec
TE                  0.0 K
D1           2.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

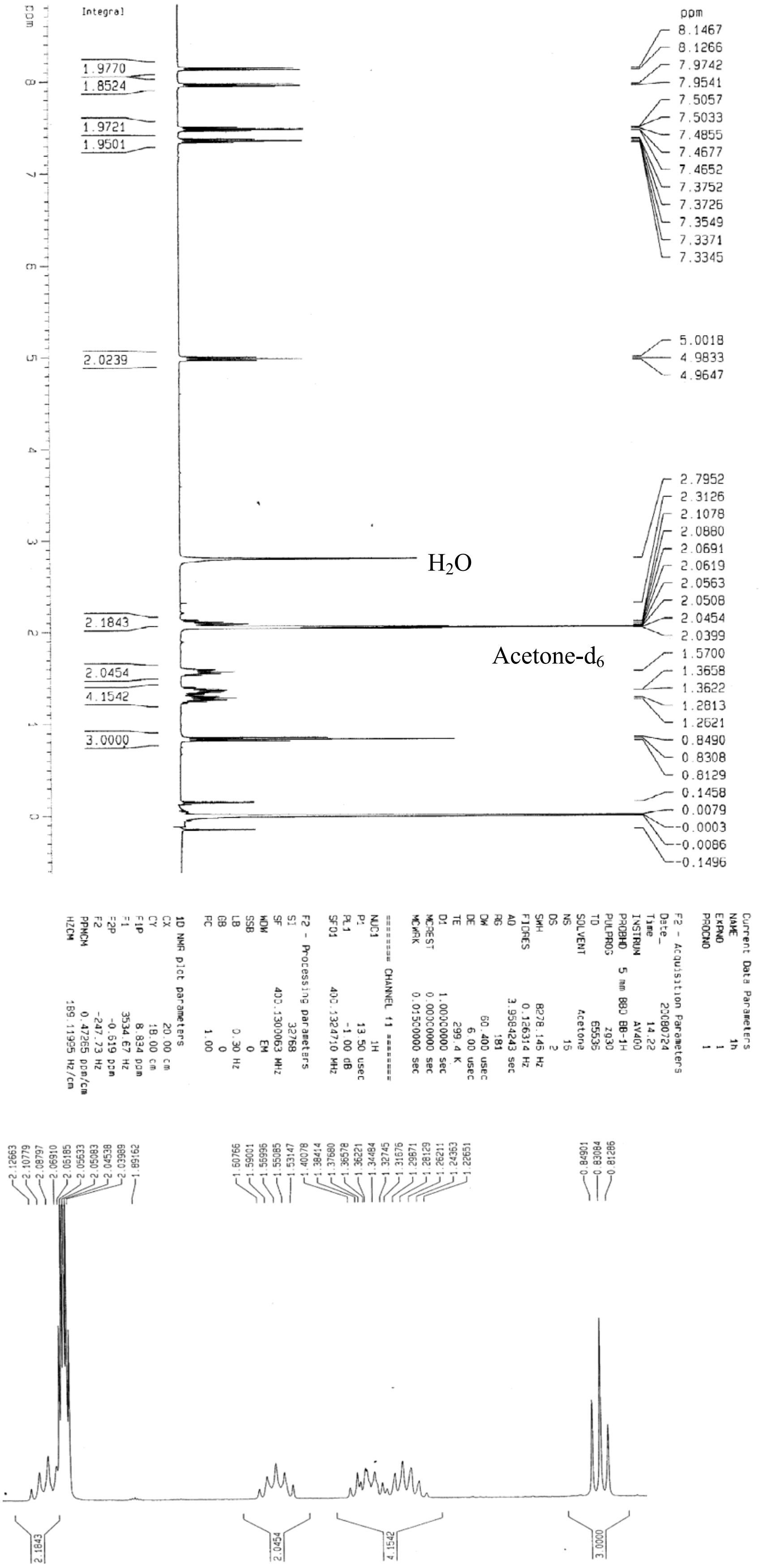
======== CHANNEL f1 ========
NUC1                13C
P1                12.00 usec
PL1                4.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             88.00 usec
PL2                4.00 dB
PL12              19.08 dB
PL13              19.00 dB
SFO2        300.1312005 MHz

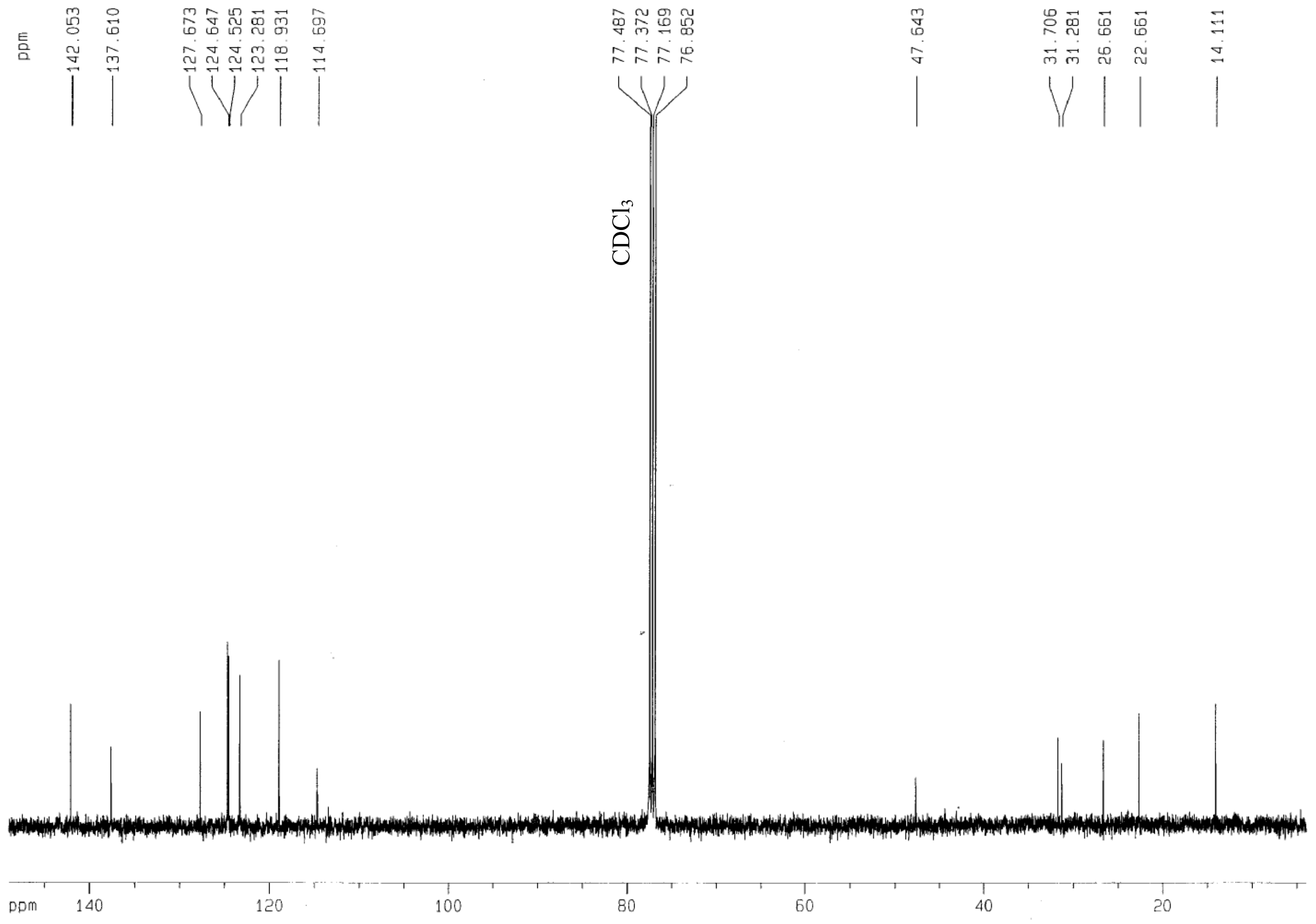
F2 - Processing parameters
SI                32768
SF           75.4677210 MHz
WDW                  EM
SSB                   0
LB                 5.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY               250.00 cm
F1P             219.525 ppm
F1             16567.09 Hz
F2P             -18.796 ppm
F2             -1418.52 Hz
PPMCM          11.91610 ppm/cm
HZCM          899.28058 Hz/cm
```

^{1}H NMR spectrum of **7**



^{13}C NMR spectrum of **7**



```
Current Data Parameters
NAME               13c
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20080731
Time             12.15
INSTRUM          AV400
PROBHD   5 mm BBO BB-1H
PULPROG         zgig30
TD               65536
SOLVENT          CDCl3
NS                 370
DS                   0
SWH          25125.629 Hz
FIDRES        0.383387 Hz
AQ           1.3042164 sec
RG             20642.5
DW              19.900 usec
DE                6.00 usec
TE               300.1 K
D1          2.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               12.20 usec
PL1              -1.00 dB
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             17.30 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127539 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX               23.00 cm
CY               20.00 cm
F1P            148.980 ppm
F1            14989.30 Hz
F2P              3.905 ppm
F2              392.86 Hz
PPMCM          6.30763 ppm/cm
HZCM         634.62787 Hz/cm
```