Electronic Supplementary Material (ESI) for Polymer Chemistry.

Supplementary Information

for

Lactone-Fused Electron-Deficient Building Blocks for *n*-Type Polymer Field-Effect Transistors: Synthesis, Properties, and Impact of Alkyl Substitution Positions

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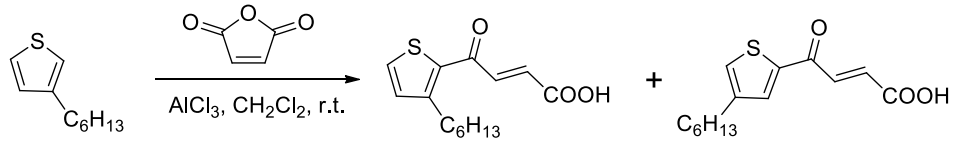
1. Synthetic Procedures.

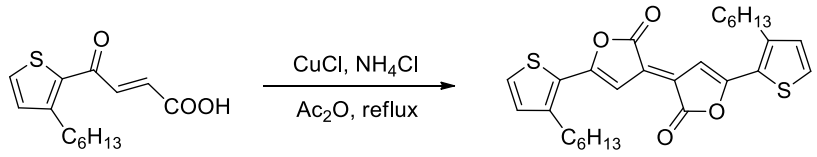


1-H **2a-H** **2b-H**

(*E*)-4-(3-hexylthiophen-2-yl)-4-oxobut-2-enoic acid (2a-H) and (*E*)-4-(4-hexylthiophen-2-yl)-4-oxobut-2-enoic acid (2b-H). 3-Hexylthiophene (**1-H**) (10.0 g, 59.4 mmol) and maleic anhydride (5.83 g, 59.4 mmol) were dissolved in dichloromethane (150 mL) and the mixture was cooled in an ice bath. $AlCl_3$ (11.9 g, 89.1 mmol) was added portion-wise and the mixture was stirred at room temperature for 16 h. Then the mixture was poured into ice-cooled 10% aqueous HCl (200 mL) and stirred for 30 min. The organic layer was separated and the aqueous layer was extracted with dichloromethane for 3 times. The combined organic layers were washed with water and saturated aqueous NaCl successively and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was subjected to column chromatography (80 g normal phase silica gel cartridge, eluent: petroleum ether/dichloromethane/acetic acid = 50 : 50 : 1) to afford 3.13 g (20 %) of compound **2a-H**, which eluted first, as a light yellow solid and 2.43 g (15 %) of compound **2b-H**, which followed, as a light yellow solid.

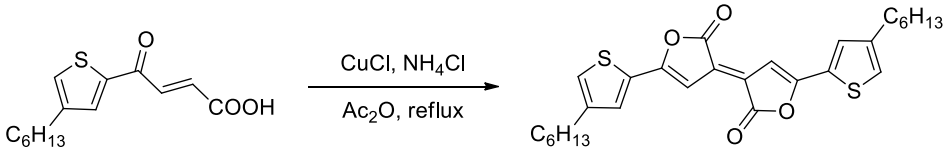
Compound **2a-H**: ^{1}H NMR (400 MHz, $CDCl_3$, 298 K, ppm): δ 7.79 (d, J = 15.2 Hz, 1H), 7.58 (d, J = 4.8 Hz, 1H), 7.09 (d, J = 4.8 Hz, 1H), 6.89 (d, J = 15.2 Hz, 1H), 3.06 (t, J = 7.8 Hz, 2H), 1.69 – 1.58 (m, 2H), 1.44 – 1.26 (m, 6H), 0.89 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm): δ 181.1, 170.4, 153.4, 140.8, 134.8, 132.2, 131.8, 130.3, 31.6, 30.7, 30.2, 29.2, 22.6, 14.0; HRMS (ESI) *m/z*: Calcd for $C_{14}H_{19}O_3S$: 267.1049; Found: 267.1049 $[M + H]^+$.

Compound **2b-H**: ^{1}H NMR (400 MHz, $CDCl_3$, 298 K, ppm): δ 7.85 (d, J = 15.6 Hz, 1H), 7.71 (s, 1H), 7.40 (s, 1H), 6.94 (d, J = 15.6 Hz, 1H), 2.65 (t, J = 7.6 Hz, 2H), 1.70 – 1.59 (m, 2H), 1.40 – 1.26 (m, 6H), 0.89 (t, J = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm): δ 180.6, 170.5, 145.3, 143.6, 138.0, 134.7, 131.4, 130.8, 31.6, 30.4, 30.2, 28.8, 22.5, 14.0. HRMS (ESI) *m/z*: Calcd for $C_{14}H_{19}O_3S$: 267.1049; Found: 267.1049 $[M + H]^+$.



2a-H **3a-H**

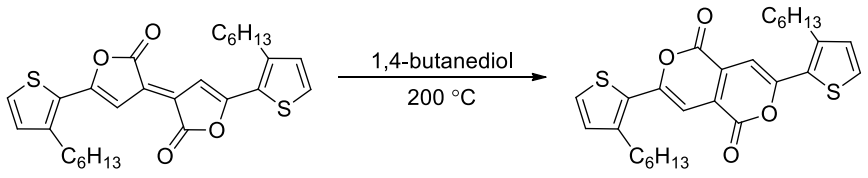
(*E*)-5,5'-(3-hexylthiophen-2-yl)-3,3'-bifuranylidene-2,2'-dione (3a-H). A 50 mL round bottomed flask was charged with **2a-H** (0.90 g, 3.8 mmol), CuCl (0.14 g, 1.4 mmol), NH_4Cl (0.15 g, 2.8 mmol) and acetic anhydride (8 mL). The mixture was heated to reflux for 2 h after a dark purple color appeared. Then the mixture was cooled thoroughly, whereupon dark purple solids precipitated. The precipitate was filtrated and washed with acetic acid and ethanol successively and was purified by column chromatography over silica gel (eluent: petroleum ether/CH_2Cl_2 = 4 : 1) to give 0.30 g (36 %) of compound **3a-H** as a dark purple solid. ^{1}H NMR (400 MHz, $CDCl_3$, 298 K, ppm): δ 7.47 (d, J = 5.2 Hz, 2H), 7.27 (s, 2H), 7.02 (d, J = 5.2 Hz, 2H), 2.90 (t, J = 7.6 Hz, 4H), 1.75 – 1.65 (m, 4H), 1.49 – 1.29 (m, 12H), 0.90 (t, J = 6.8 Hz,6H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm): δ 166.9, 154.8, 147.6, 131.6, 129.9, 125.3, 124.8, 103.8, 31.6, 30.4, 29.8, 29.2, 22.6, 14.0; HRMS (ESI) *m/z*: Calcd for $C_{28}H_{33}O_4S_2$: 497.1815; Found: 497.1801 $[M + H]^+$.



2b-H **3b-H**

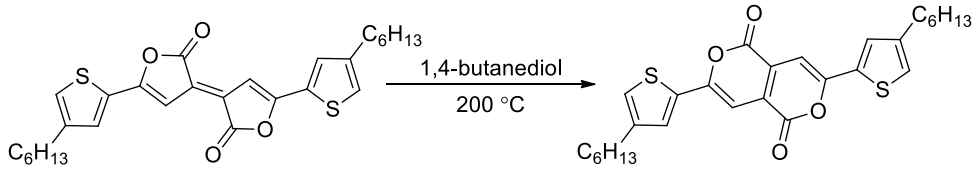
(*E*)-5,5'-(4-hexylthiophen-2-yl)-3,3'-bifuranylidene-2,2'-dione (3b-H). A 50 mL round bottomed flask was charged with **2b-H** (640 mg, 2.4 mmol), CuCl (88 mg, 0.9 mmol), NH_4Cl (94 mg, 1.8 mmol) and acetic anhydride (4 mL). The mixture was heated to reflux for 2 h after a dark purple color appeared. Then the mixture was cooled thoroughly and subjected to ultrasonication to give a dark purple precipitate. The precipitate was filtrated and washed with acetic acid and ethanol successively. The residue was purified by column chromatography over silica gel (eluent: petroleum ether/CH_2Cl_2 = 4 : 1) to give 85 mg (14 %) of compound **3b-H** as a dark purple solid. ^{1}H NMR (400 MHz, $CDCl_3$, 298 K, ppm): δ 7.46 (s, 2H), 7.31 (s, 2H), 7.18 (s, 2H), 2.63 (t, J = 7.8 Hz, 4H), 1.69 – 1.58 (m, 4H), 1.40 – 1.24 (m, 12H), 0.89

(t, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm): δ 167.2, 154.6, 145.5, 130.7, 130.4, 126.5, 124.9, 102.8, 31.6, 30.3, 30.2, 28.8, 22.6, 14.1; HRMS (ESI) *m/z*: Calcd for $C_{28}H_{32}O_4S_2$: 496.1736; Found: 496.1725 $[M]^+$.



3a-H **4a-H** (P_{66}2T-3H)

3,7-bis(3-hexylthiophen-2-yl)pyrano[4,3-c]pyran-1,5-dione (4a-H). Compound **3a-H** (330 mg, 0.66 mmol) was suspended in 1,4-butanediol (20 mL) and the mixture was heated to 200 ℃ under nitrogen for 1 h and then cooled down to room temperature. 20 mL of methanol and 40 mL of saturated aqueous NaCl were added. The mixture was extracted with chloroform for 3 times and the combined organic layers were washed with water and saturated aqueous NaCl successively and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was subjected to column chromatography over silica gel (eluent: petroleum ether/CH_2Cl_2 = 4 : 1 to 2 : 1) to give 100 mg (30 %) of compound **4a-H** as an orange solid. ^{1}H NMR (400 MHz, $CDCl_3$, 298 K, ppm): δ 7.39 (d, J = 5.2 Hz, 2H), 7.00 (s, 2H), 6.98 (s, 2H), 2.92 (t, J = 7.8 Hz, 4H), 1.75 – 1.65 (m, 4H), 1.47 – 1.29 (m, 12H), 0.89 (t, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm): δ 159.2, 154.2, 145.2, 131.3, 128.3, 127.9, 126.5, 98.8, 31.6, 30.22, 30.19, 29.2, 22.6, 14.04; HRMS (ESI) *m/z*: Calcd for $C_{28}H_{33}O_4S_2$: 497.1815; Found: 497.1819 $[M + H]^+$.



3b-H **4b-H** P_{66}2T-4H

3,7-bis(4-hexylthiophen-2-yl)pyrano[4,3-c]pyran-1,5-dione (4b-H). Compound **3b-H** (155 mg, 0.31 mmol) was suspended in 1,4-butanediol (30 mL) and the mixture was heated to 200 ℃ under nitrogen for 0.5 h and then cooled down to room temperature. 20 mL of methanol and 40 mL of saturated aqueous NaCl was added.

The mixture was extracted with chloroform for 3 times and the combined organic layers were washed with water and saturated aqueous NaCl successively and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was subjected to column chromatography over silica gel (eluent: petroleum ether/CH_2Cl_2 = 4 : 1 to 2 : 1) to give 78 mg (50 %) of compound **4b-H** as an orange solid. ^{1}H NMR (400 MHz, $CDCl_3$, 298 K, ppm): δ 7.49 (s, 2H), 7.08 (s, 2H), 7.07 (s, 2H), 2.62 (t, J = 7.6 Hz, 4H), 1.67 – 1.58 (m, 4H), 1.40 – 1.25 (m, 12H), 0.90 (t, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm): δ 159.2, 153.6, 145.0, 134.3, 128.8, 126.7, 124.5, 96.7, 31.6, 30.34, 30.30, 28.9, 22.6, 14.1; HRMS (ESI) *m/z*: Calcd for $C_{28}H_{33}O_4S_2$: 497.1815; Found: 497.1828 $[M + H]^+$.

2. Thermal Properties (TGA and DSC)

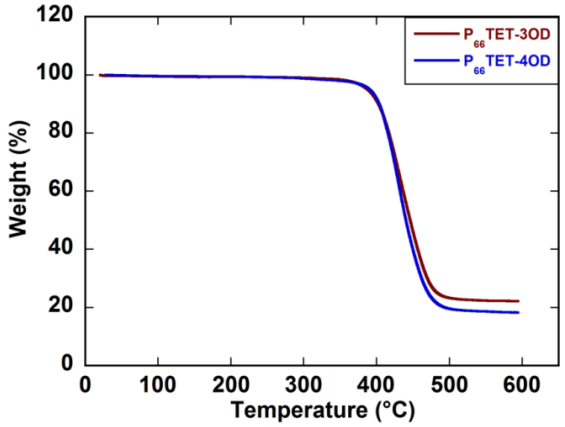


Fig. S1 TGA curves of **P_{66}TET-3OD** and **P_{66}TET-4OD**. T_d with 5% weight loss: 389 ℃ (**P_{66}TET-3OD**), 391 ℃ (**P_{66}TET-4OD**).

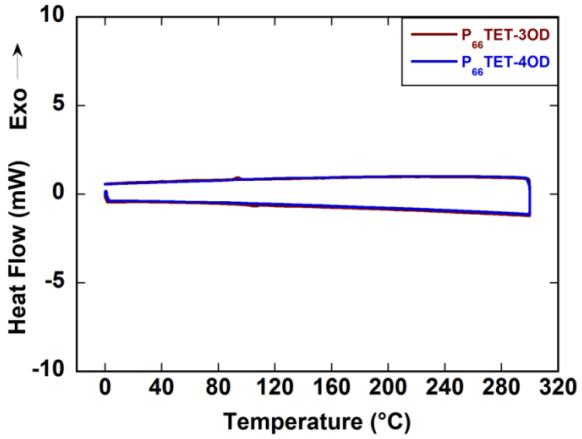


Fig. S2 DSC traces of polymers **P_{66}TET-3OD** and **P_{66}TET-4OD**.

3. Photoelectron Spectroscopy (PES)

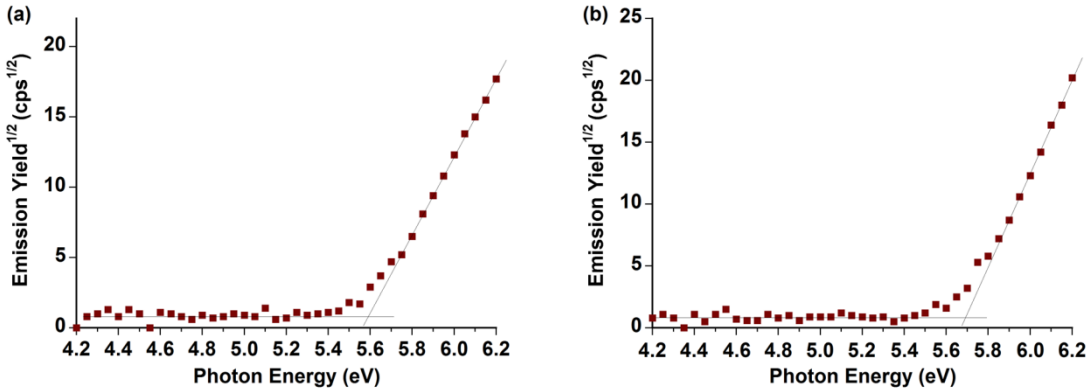


Fig. S3 PES spectra of (a) **P_{66}TET-3OD** and (b) **P_{66}TET-4OD** in thin films (work function: **P_{66}TET-3OD**, 5.59 eV; **P_{66}TET-4OD**, 5.69 eV).

4. X-Ray Diffraction (XRD)

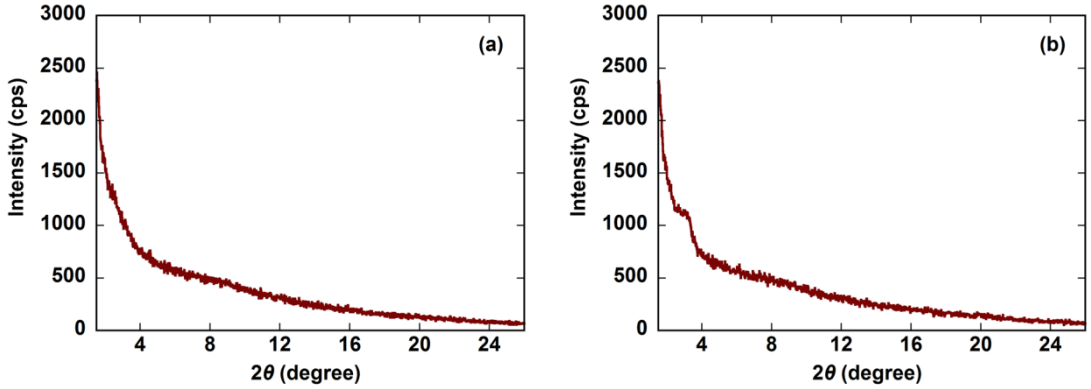


Fig. S4 Out-of-plane XRD patterns of (a) **P_{66}TET-3OD** and (b) **P_{66}TET-4OD** films.

5. Single-Crystal X-Ray Analysis

Table S1. Crystal data and structure refinement for P_{66}2T-3H.

Empirical formula	$C_{28}H_{32}O_4S_2$
Formula weight	496.65
Temperature/K	180.01(10)
Crystal system	triclinic
Space group	P-1
a/Å	6.0485(7)
b/Å	6.9813(4)
c/Å	15.2806(16)
α/°	94.625(6)
β/°	94.955(9)
γ/°	95.942(7)
Volume/Å^3	636.77(10)
Z	1
ρ_{calc}mg/mm^3	1.295
m/mm^{-1}	0.241
F(000)	264.0
Crystal size/mm^3	0.2 × 0.1 × 0.1
2θ range for data collection	5.892 to 52.042 °
Index ranges	$-6 \le h \le 7$, $-8 \le k \le 8$, $-18 \le l \le 18$
Reflections collected	4143
Independent reflections	2507[R(int) = 0.0364]
Data/restraints/parameters	2507/0/155
Goodness-of-fit on F^2	1.025

Final R indexes [I>=2σ (I)]	R_1 = 0.0745, wR_2 = 0.1791
Final R indexes [all data]	R_1 = 0.1171, wR_2 = 0.2069
Largest diff. peak/hole / e Å^{-3}	0.83/-0.37

6. Computational Details

DFT calculations were performed using the Gaussian 09 software package.[1] The geometries and energies were calculated at the B3LYP/DGDZVP level.

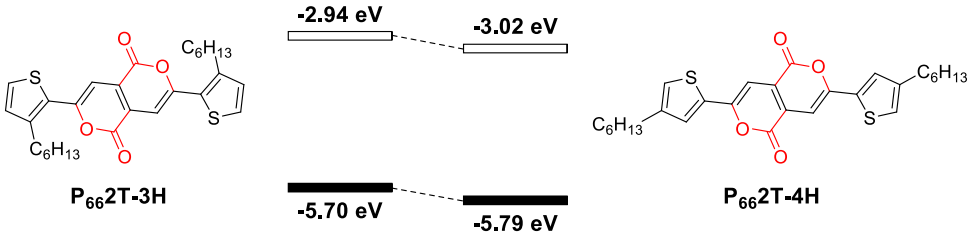


Fig. S5 DFT-calculated HOMO and LUMO energy levels of **P_{66}2T-3H** and **P_{66}2T-4H**. Hexyl chains were replaced with methyl groups during the calculation.

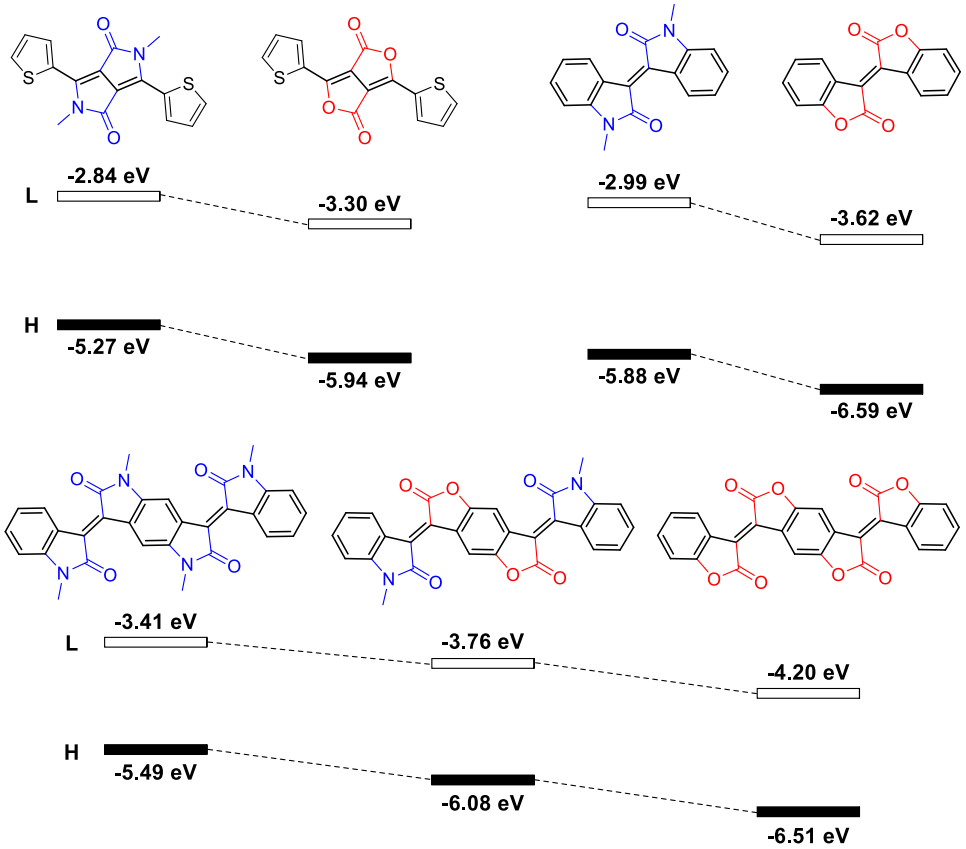
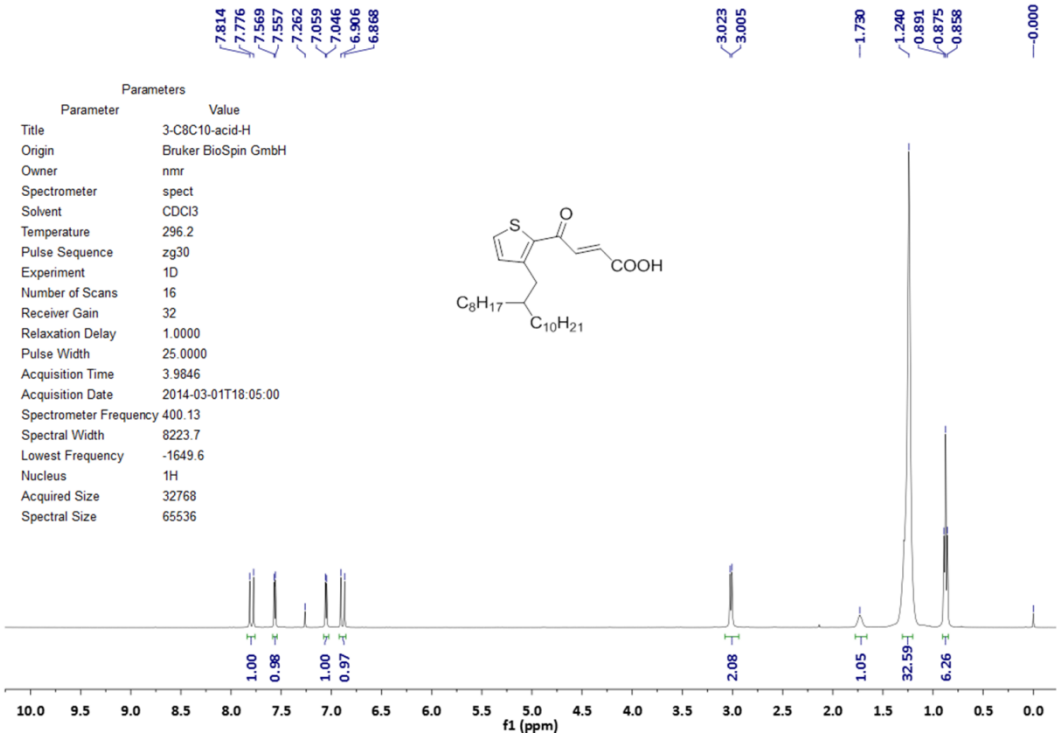


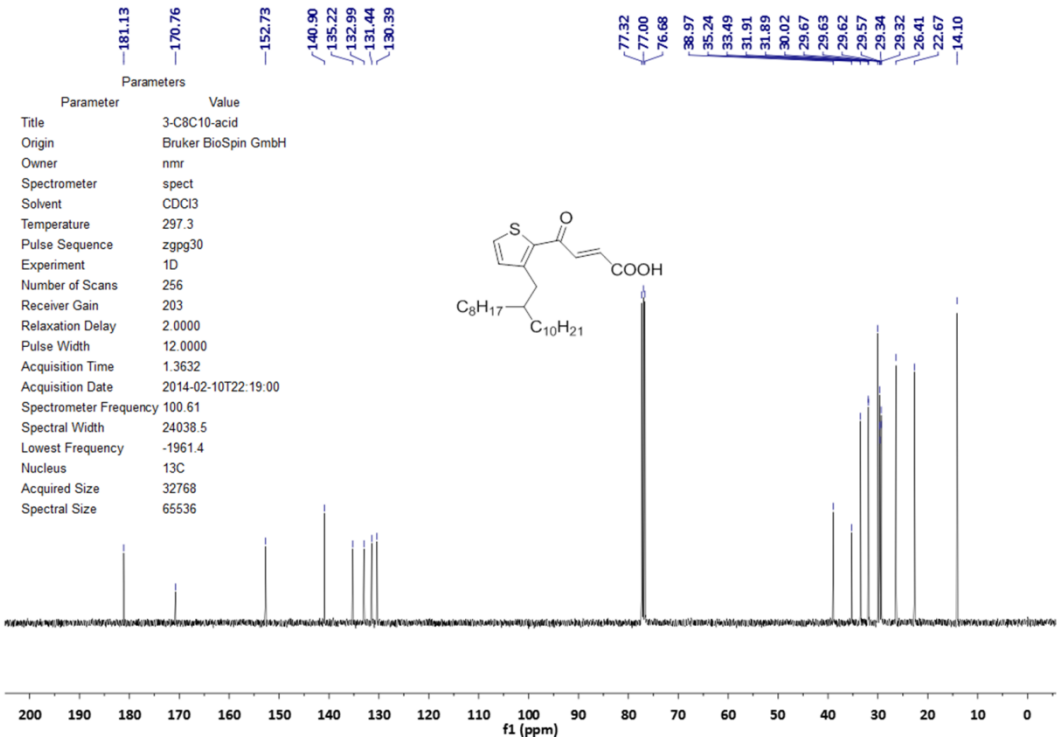
Fig. S6 DFT-calculated HOMO (H) and LUMO (L) energy levels of DPP, isoindigo, and their corresponding lactone-based counterparts (top) and OPV derivatives fused with lactams and/or lactones (bottom).

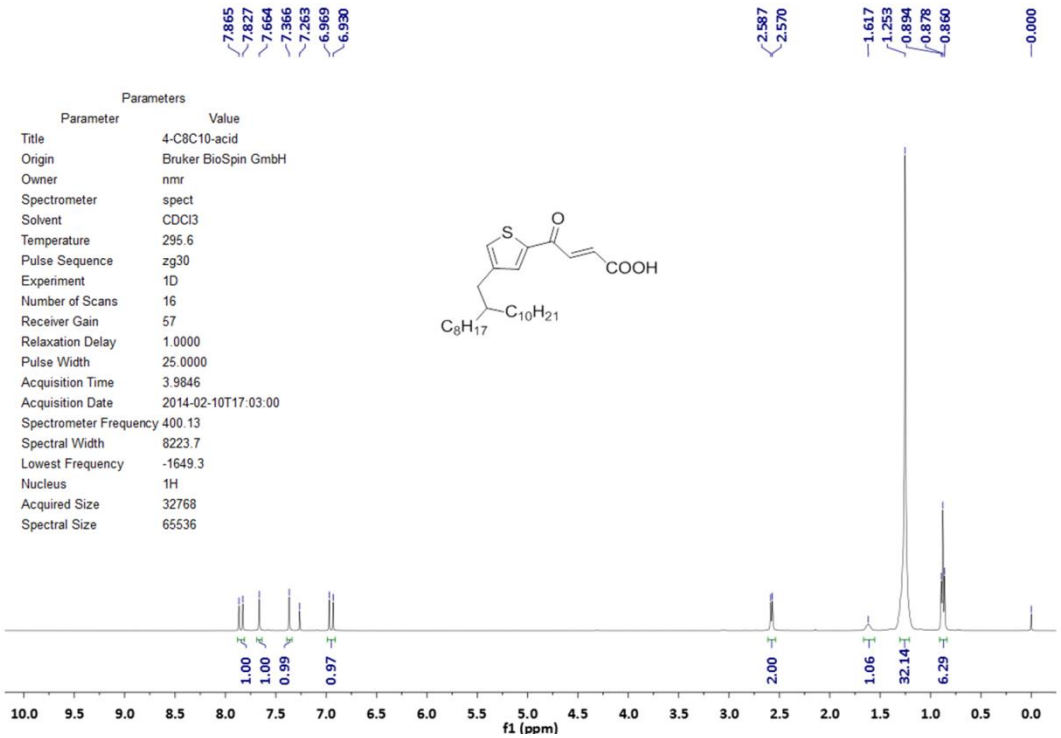
Reference:

1. Gaussian 09, Revision D.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2013.

7. NMR Spectra



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Owner nmr
Spectrometer spect
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C8H17
C10H21
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1.00
0.99
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2.00
1.06
32.14
6.29
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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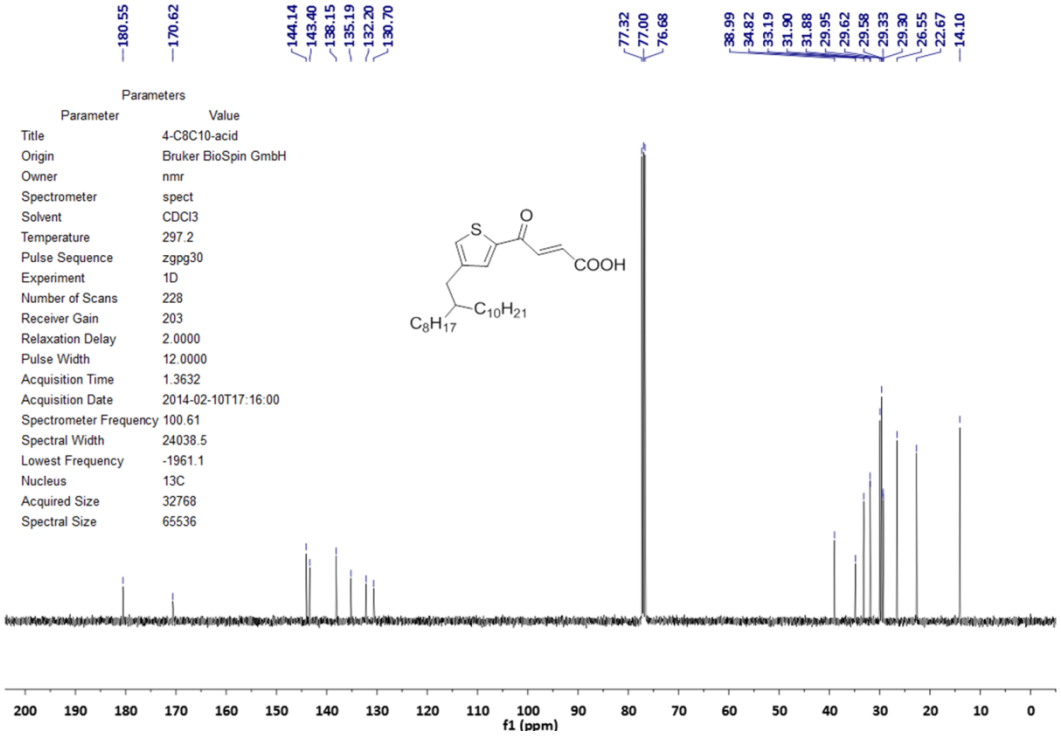
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33.19
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29.30
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14.10
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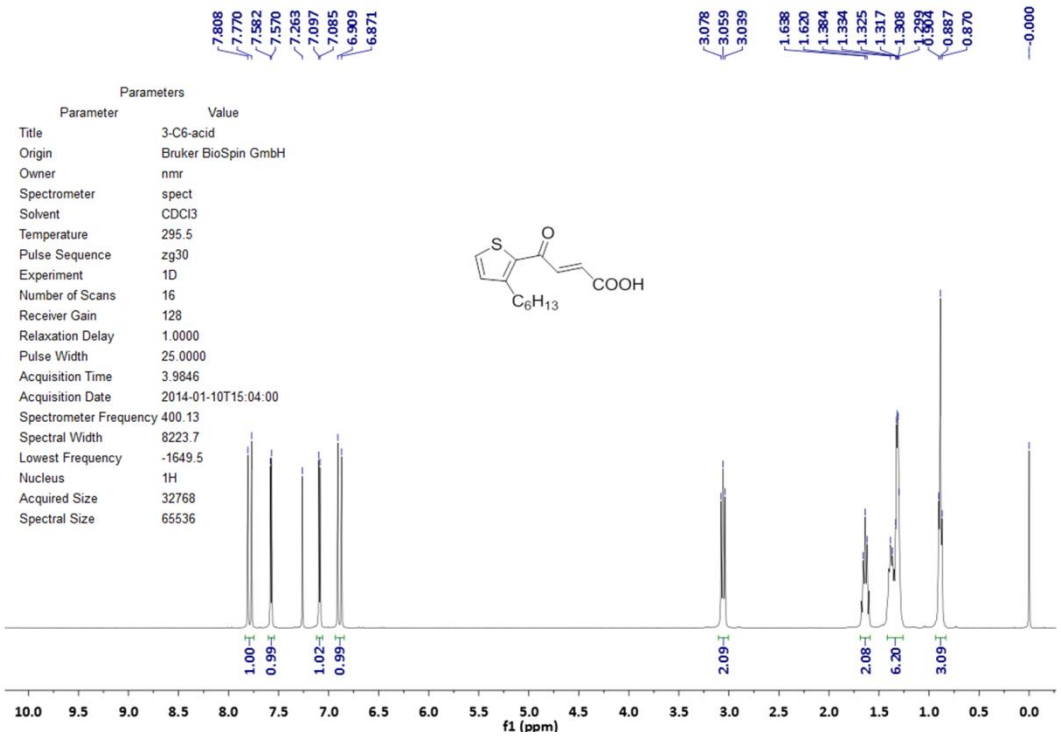
f1 (ppm)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Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
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Temperature 295.5
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Nucleus 1H
Acquired Size 32768
Spectral Size 65536
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C6H13
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0.99
1.02
0.99
2.09
2.08
6.20
3.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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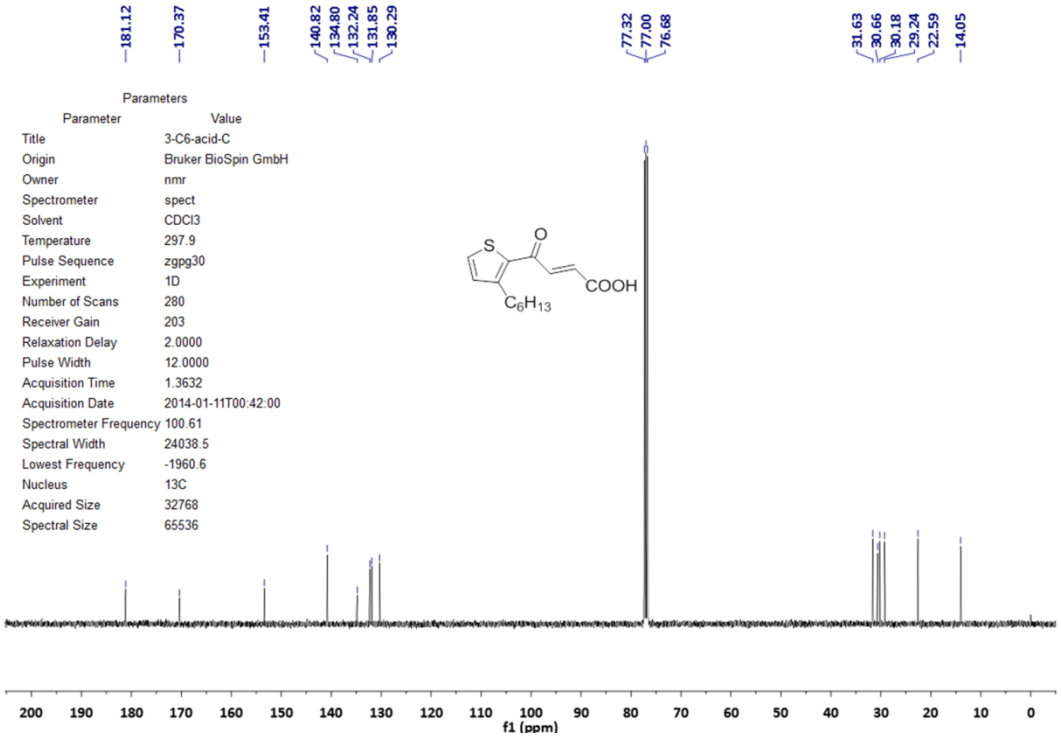
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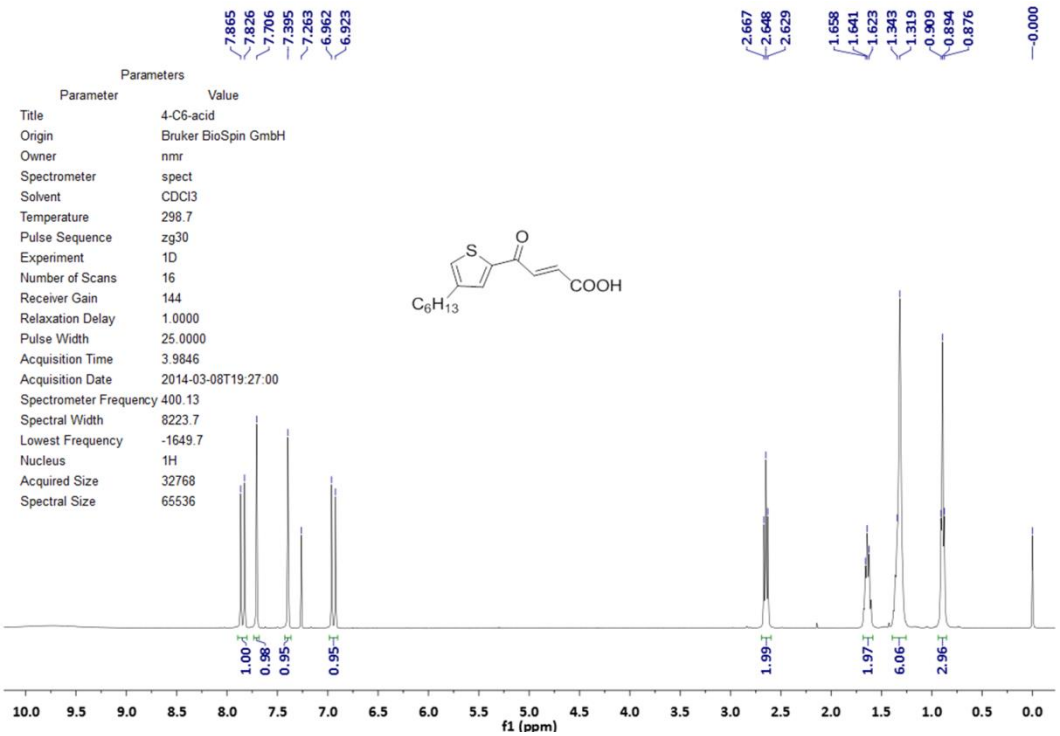
f1 (ppm)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0.876
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Parameter Value
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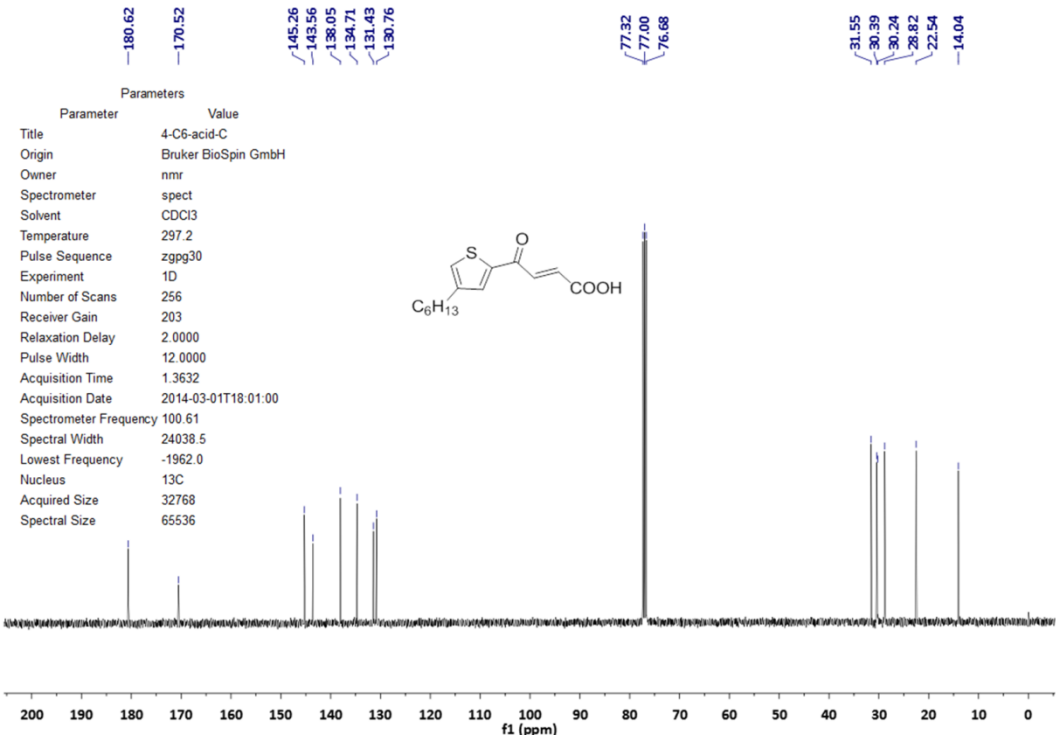
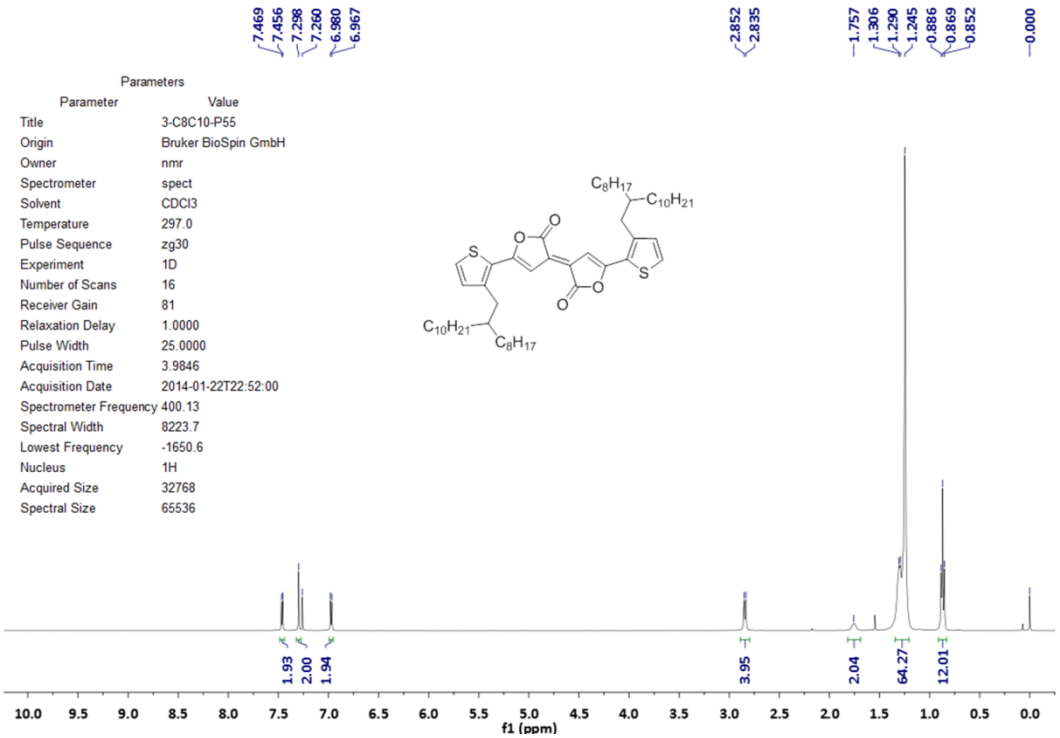
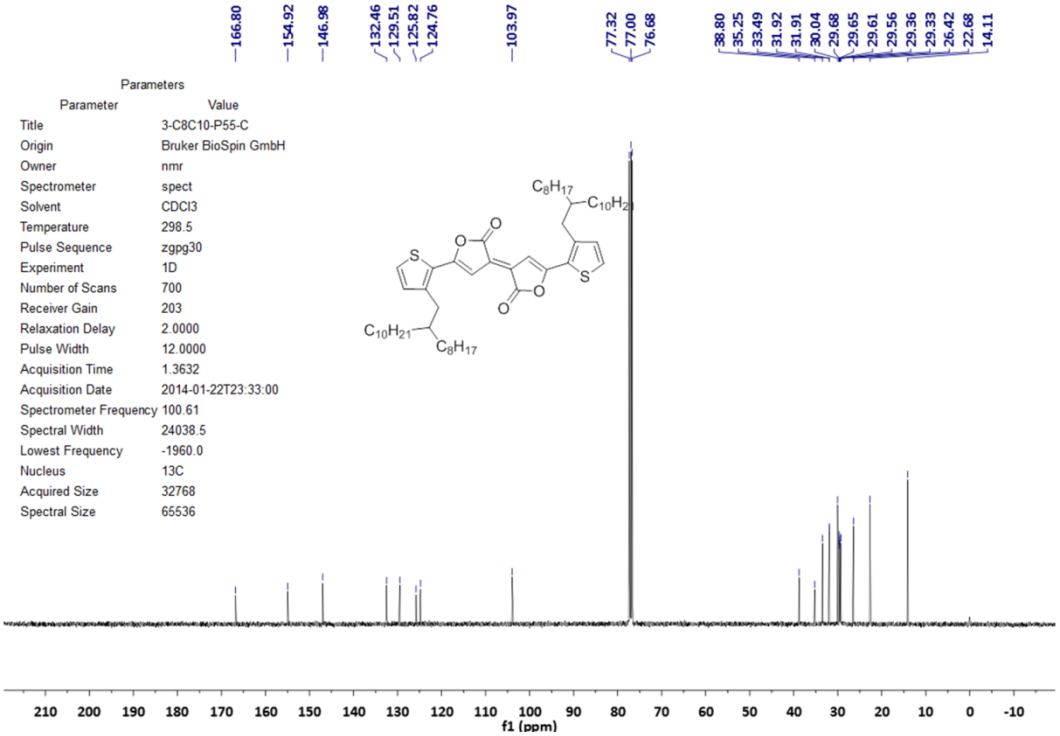
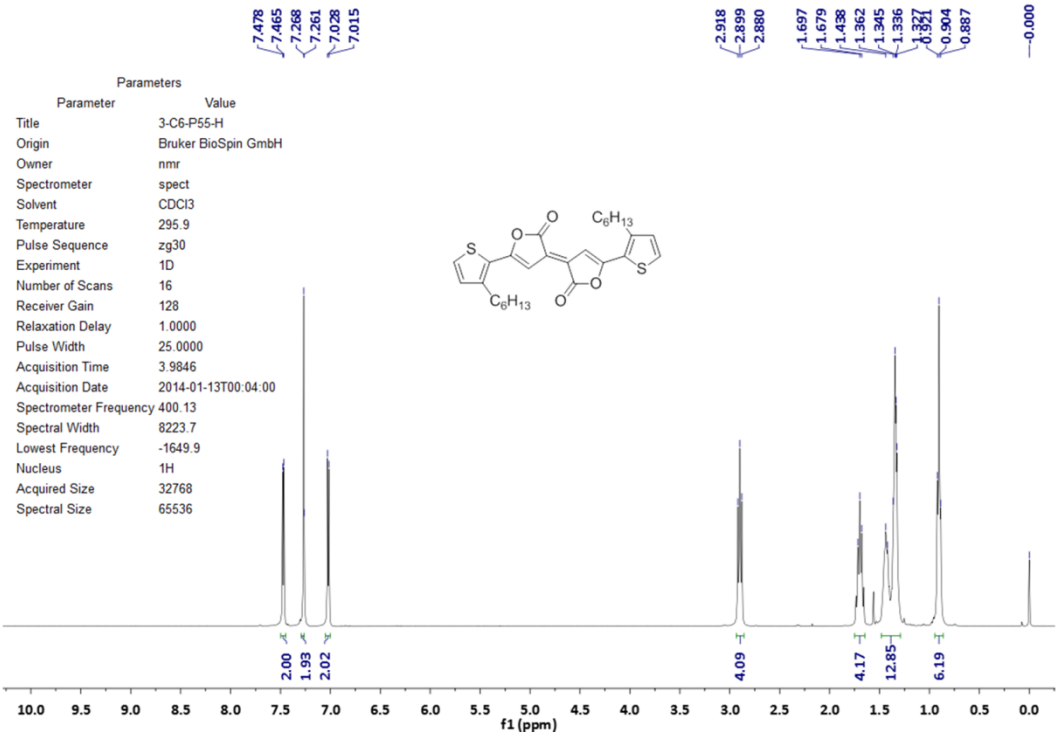
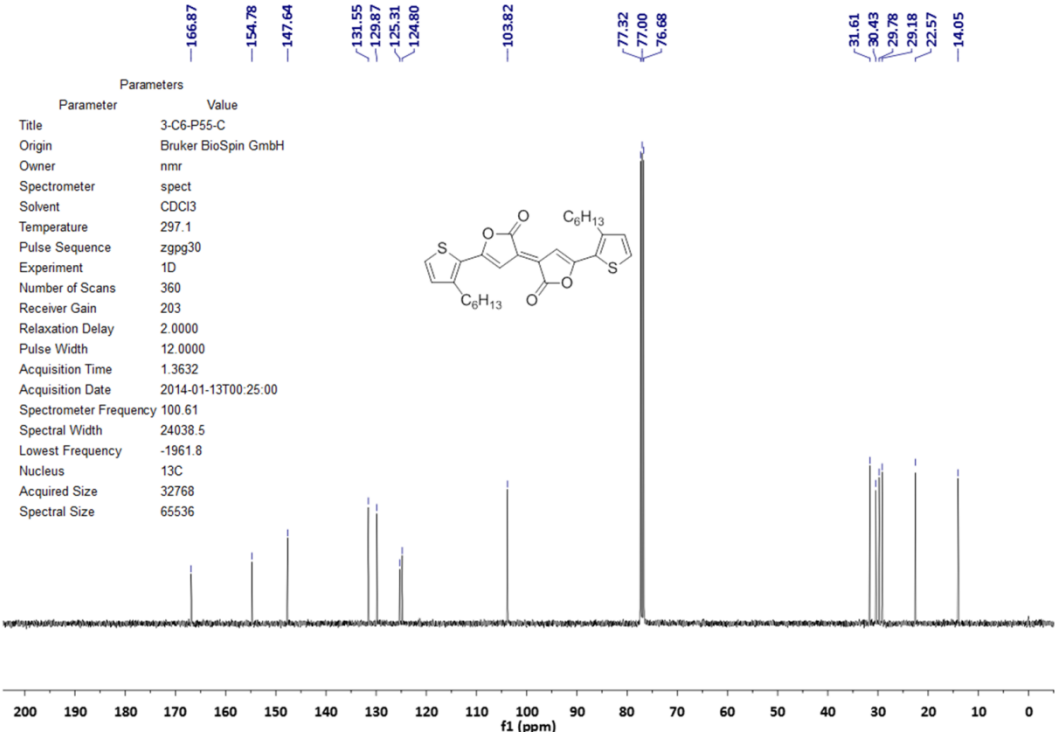
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 298.7
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 144
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-03-08T19:27:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1649.7
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
S
O
COOH
C6H13
1.00
0.98
0.95
0.95
1.99
1.97
6.06
2.96
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

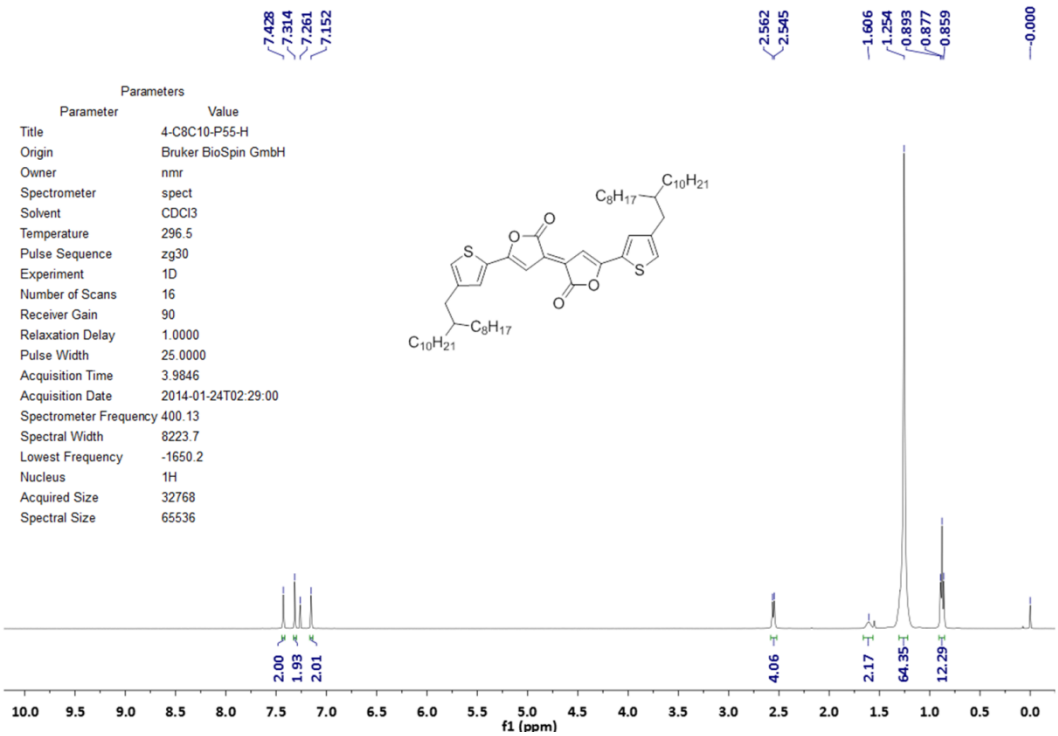

180.62
170.52
145.26
143.56
138.05
134.71
131.43
130.76
77.32
77.00
76.68
31.55
30.39
30.24
28.82
22.54
14.04
Parameters
Parameter Value
Title 4-C6-acid-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 297.2
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 256
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-03-01T18:01:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1962.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
S
O
COOH
C6H13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

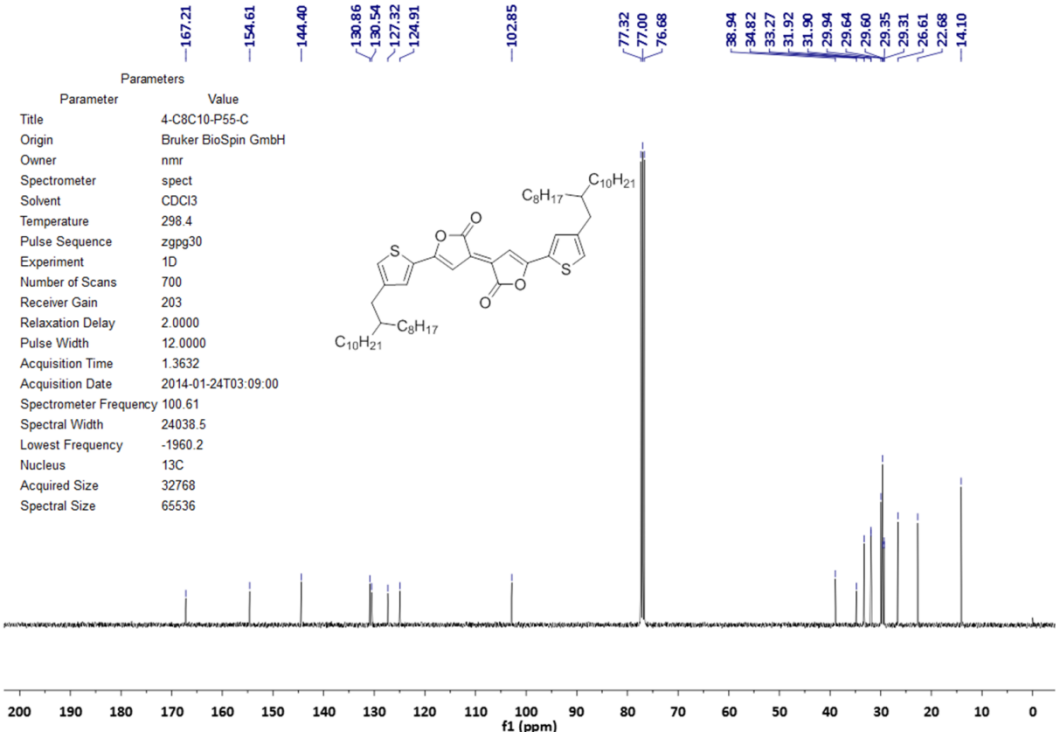

7.469
7.456
7.298
7.260
6.980
6.967
2.852
2.835
1.757
1.306
1.290
1.245
0.886
0.869
0.852
0.000
Parameters
Parameter Value
Title 3-C8C10-P55
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 297.0
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 81
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-01-22T22:52:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1650.6
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
1.93
2.00
1.94
3.95
2.04
64.27
12.01
f1 (ppm)

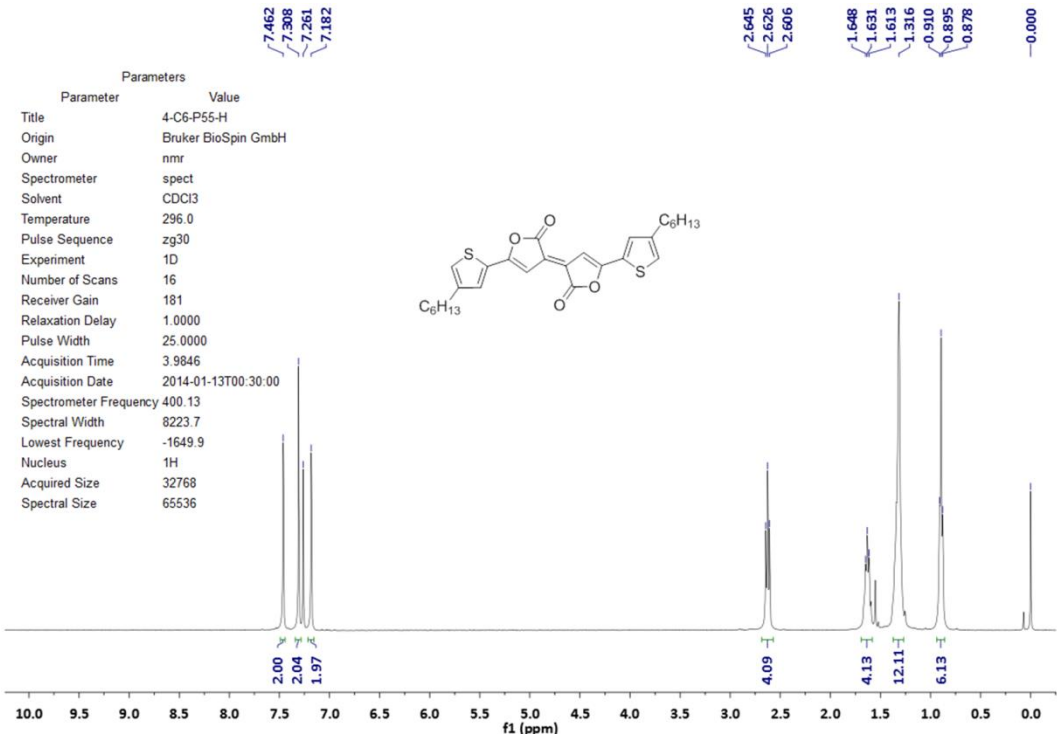

166.80
154.92
146.98
132.46
129.51
125.82
124.76
103.97
77.32
77.00
76.68
38.80
35.25
33.49
31.92
31.91
30.04
29.68
29.65
29.61
29.56
29.36
29.33
26.42
22.68
14.11
Parameters
Parameter Value
Title 3-C8C10-P55-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 298.5
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 700
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-01-22T23:33:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1960.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)

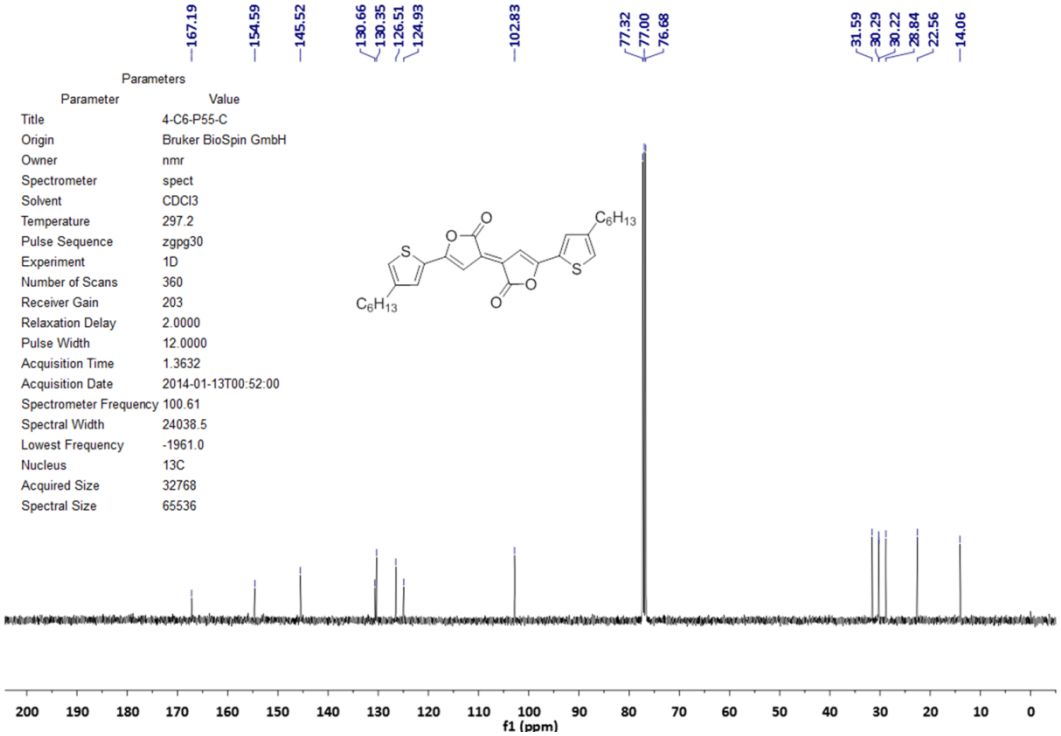

7.478
7.465
7.268
7.261
7.028
7.015
2.918
2.899
2.880
1.697
1.679
1.438
1.362
1.345
1.336
0.921
0.904
0.887
0.000
Parameters
Parameter Value
Title 3-C6-P55-H
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 295.9
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 128
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-01-13T00:04:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1649.9
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
2.00
1.93
2.02
4.09
4.17
12.85
6.19
f1 (ppm)

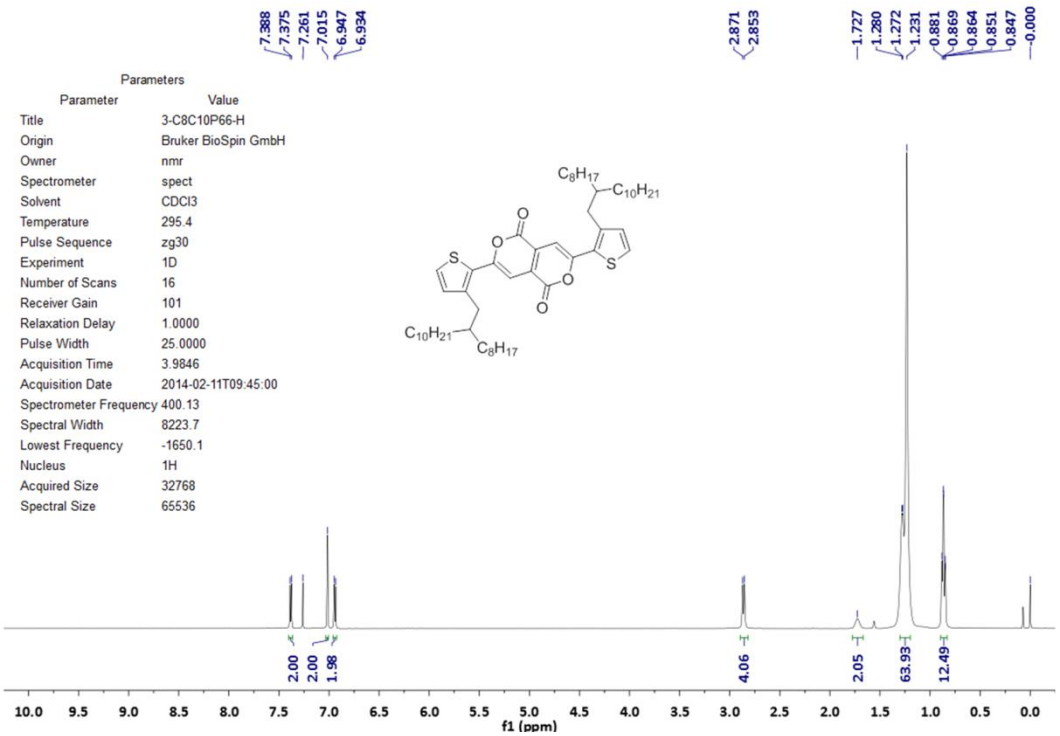

166.87
154.78
147.64
131.55
129.87
125.31
124.80
103.82
77.32
77.00
76.68
31.61
30.43
29.78
29.18
22.57
14.05
Parameters
Parameter Value
Title 3-C6-P55-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 297.1
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 360
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-01-13T00:25:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1961.8
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)

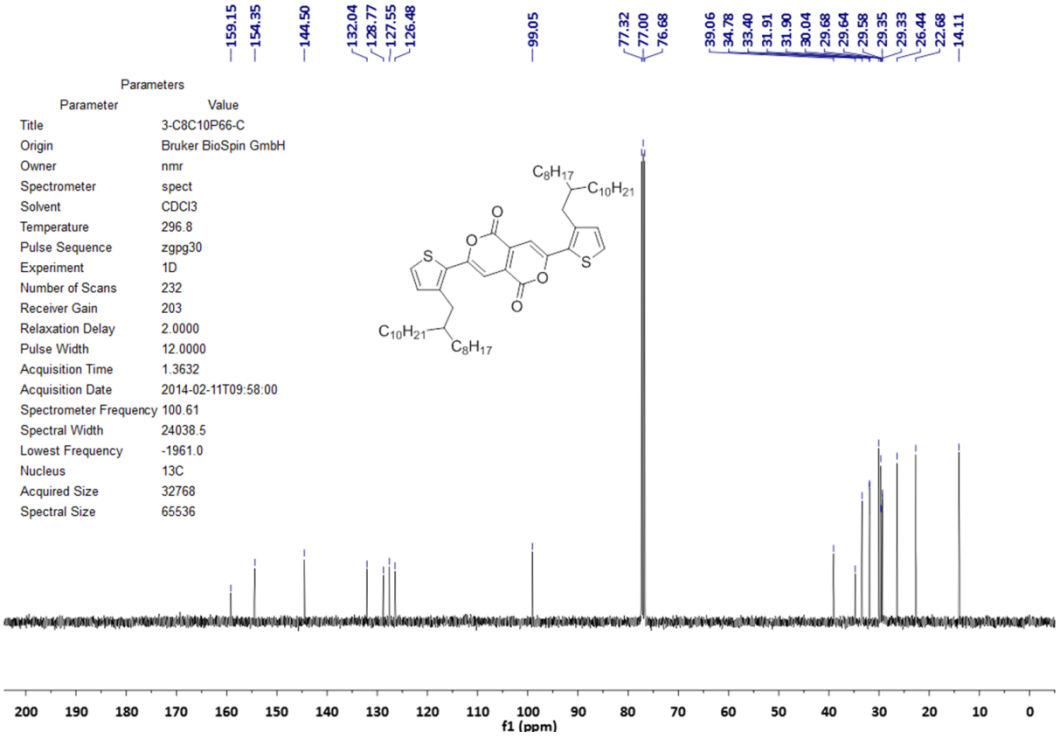

7.428
7.314
7.261
7.152
2.562
2.545
1.606
1.254
0.893
0.877
0.859
0.000
Parameters
Parameter Value
Title 4-C8C10-P55-H
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 296.5
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 90
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-01-24T02:29:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1650.2
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
2.00
1.93
2.01
4.06
2.17
64.35
12.29
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

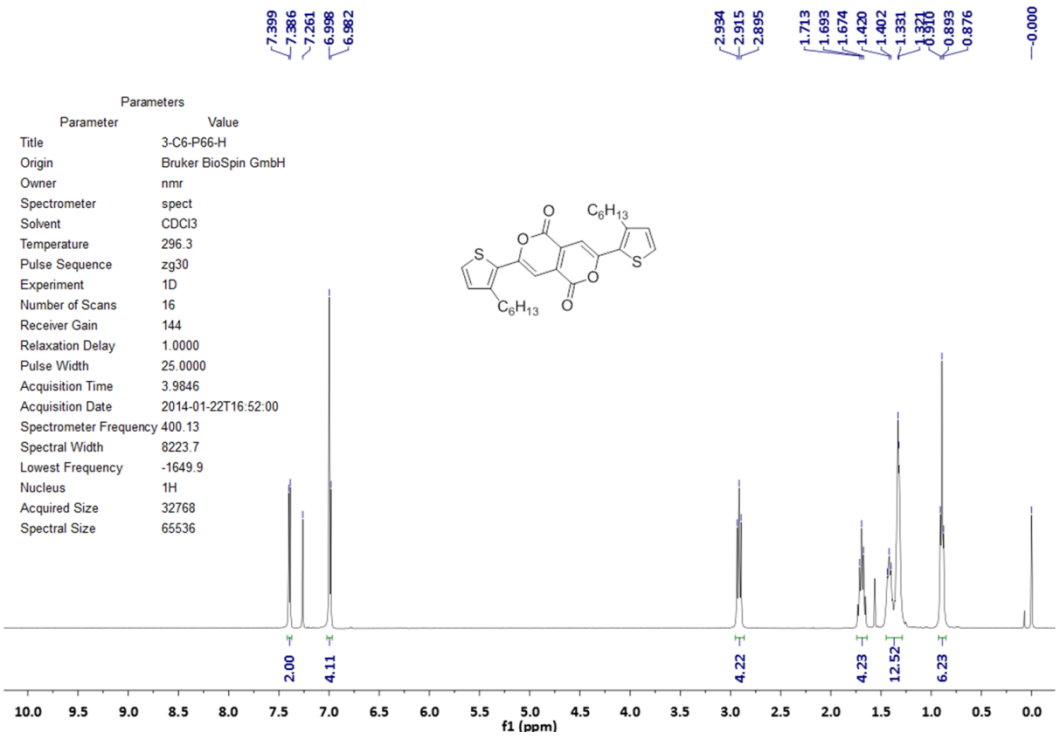

167.21
154.61
144.40
130.86
130.54
127.32
124.91
102.85
77.32
77.00
76.68
38.94
34.82
33.27
31.92
31.90
29.94
29.64
29.60
29.35
29.31
26.61
22.68
14.10
Parameters
Parameter Value
Title 4-C8C10-P55-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 298.4
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 700
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-01-24T03:09:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1960.2
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.462
7.308
7.261
7.182
2.645
2.626
2.606
1.648
1.631
1.613
1.316
0.910
0.895
0.878
0.000
Parameters
Parameter Value
Title 4-C6-P55-H
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 296.0
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 181
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-01-13T00:30:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1649.9
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
2.00
2.04
1.97
4.09
4.13
12.11
6.13
f1 (ppm)


167.19
154.59
145.52
130.66
130.35
126.51
124.93
102.83
77.32
77.00
76.68
31.59
30.29
30.22
28.84
22.56
14.06
Parameters
Parameter Value
Title 4-C6-P55-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 297.2
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 360
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-01-13T00:52:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1961.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)


7.388
7.375
7.261
7.015
6.947
6.934
2.871
2.853
1.727
1.280
1.272
1.231
0.881
0.869
0.864
0.851
0.847
0.000
Parameters
Parameter Value
Title 3-C8C10P66-H
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 295.4
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 101
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-02-11T09:45:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1650.1
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
2.00
2.00
1.98
4.06
2.05
63.93
12.49
f1 (ppm)


159.15
154.35
144.50
132.04
128.77
127.55
126.48
99.05
77.32
77.00
76.68
39.06
34.78
33.40
31.91
31.90
30.04
29.68
29.64
29.58
29.35
29.33
26.44
22.68
14.11
Parameters
Parameter Value
Title 3-C8C10P66-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 296.8
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 232
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-02-11T09:58:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1961.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)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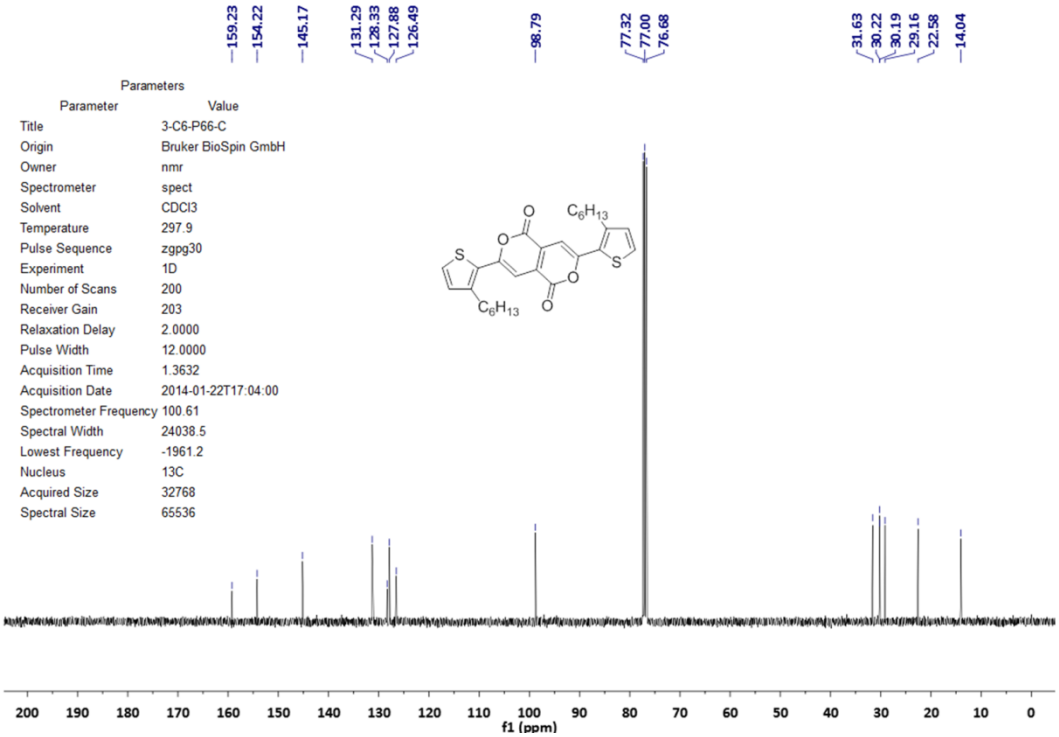
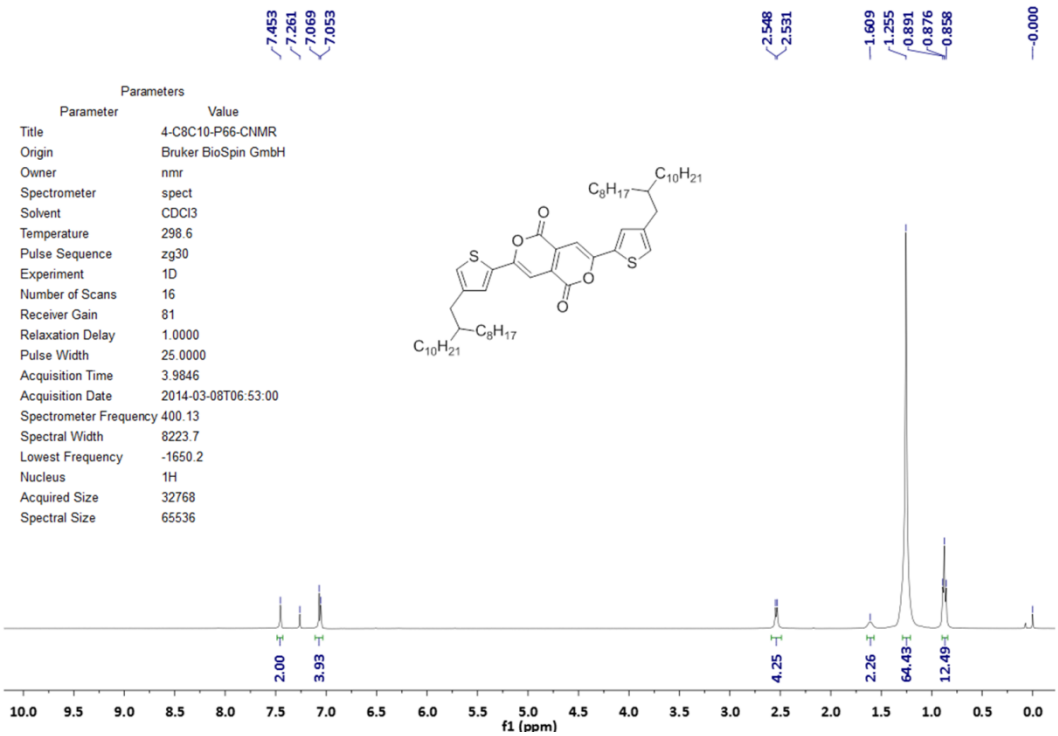
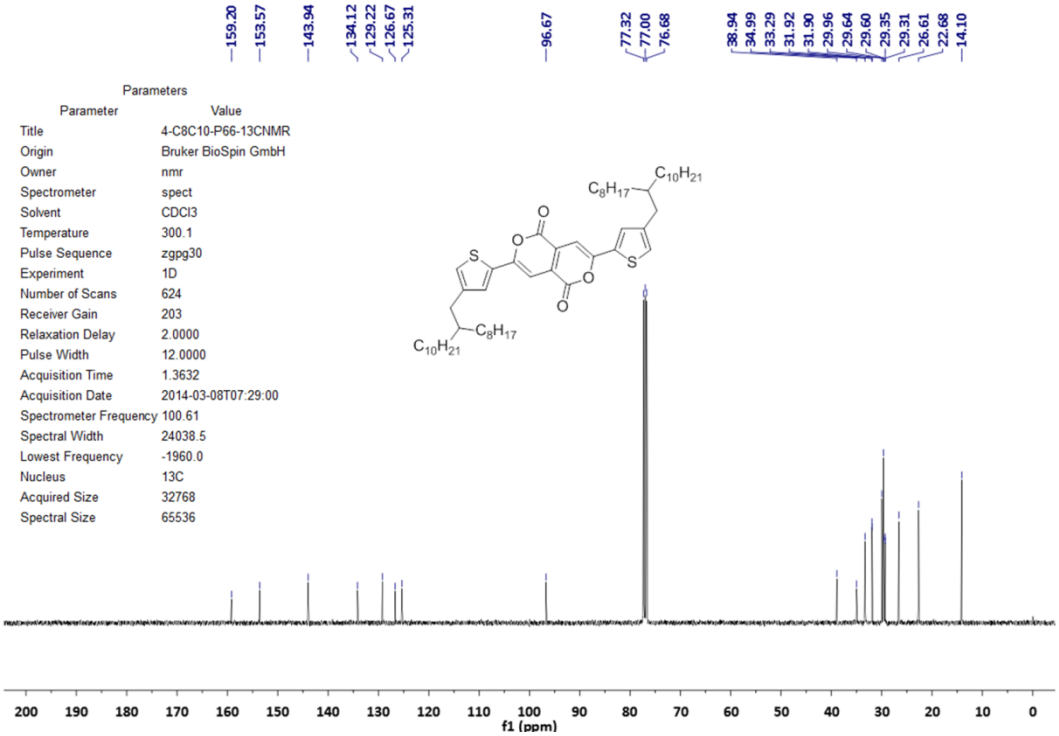
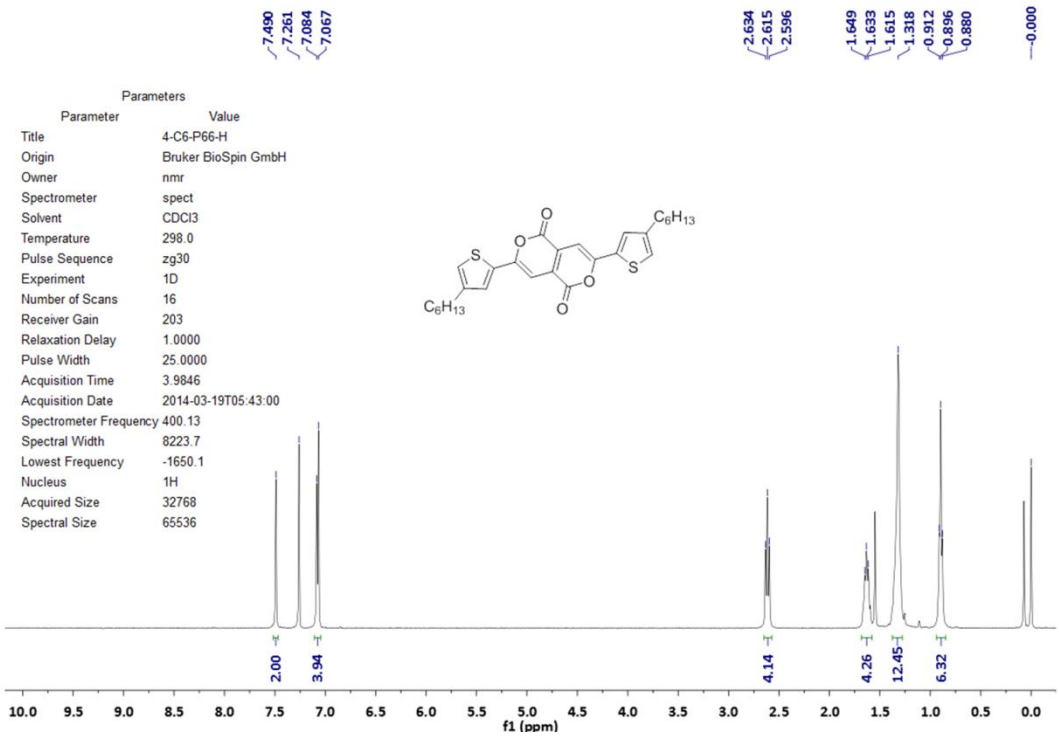
7.386
7.261
6.998
6.982
2.934
2.915
2.895
1.713
1.693
1.674
1.420
1.402
1.331
1.321
0.910
0.893
0.876
0.000
Parameters
Parameter Value
Title 3-C6-P66-H
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 296.3
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 144
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-01-22T16:52:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1649.9
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
C6H13
C6H13
2.00
4.11
4.22
4.23
12.52
6.23
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.23
154.22
145.17
131.29
128.33
127.88
126.49
98.79
77.32
77.00
76.68
31.63
30.22
30.19
29.16
22.58
14.04
Parameters
Parameter Value
Title 3-C6-P66-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 297.9
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 200
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-01-22T17:04:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1961.2
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
C6H13
C6H13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.453
7.261
7.069
7.053
2.548
2.531
1.609
1.255
0.891
0.876
0.858
0.000
Parameters
Parameter Value
Title 4-C8C10-P66-CNMR
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 298.6
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 81
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-03-08T06:53:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1650.2
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
2.00
3.93
4.25
2.26
64.43
12.49
f1 (ppm)


159.20
153.57
143.94
134.12
129.22
126.67
125.31
96.67
77.32
77.00
76.68
38.94
34.99
33.29
31.92
31.90
29.96
29.64
29.60
29.35
29.31
26.61
22.68
14.10
Parameters
Parameter Value
Title 4-C8C10-P66-13CNMR
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 300.1
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 624
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-03-08T07:29:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1960.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)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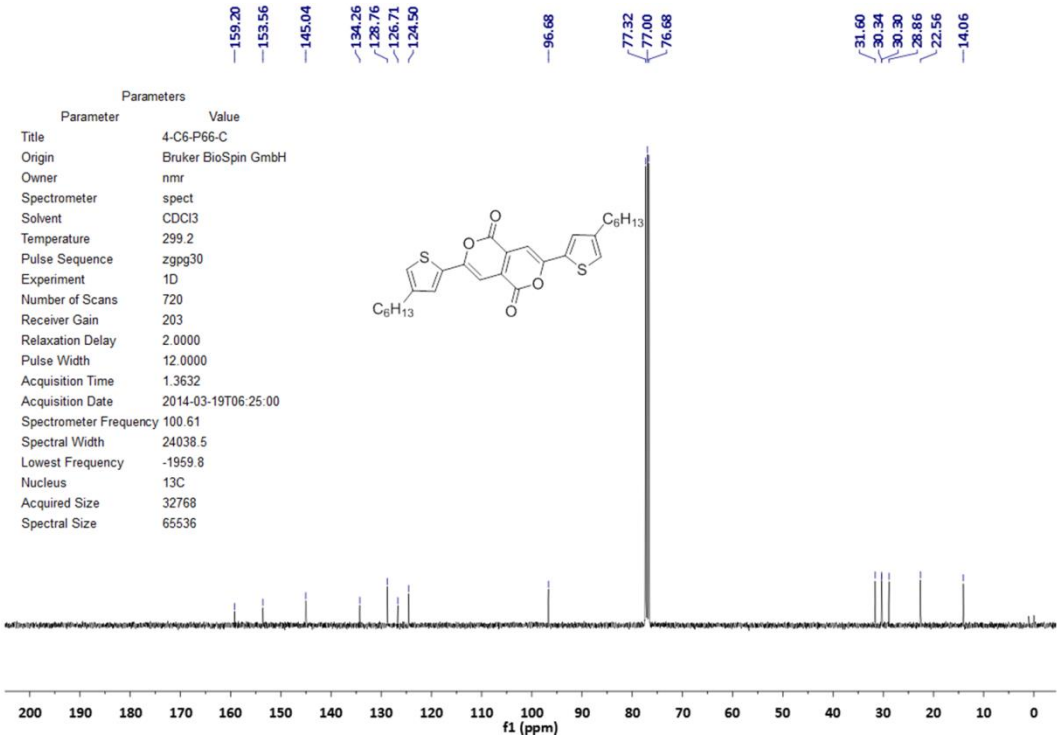
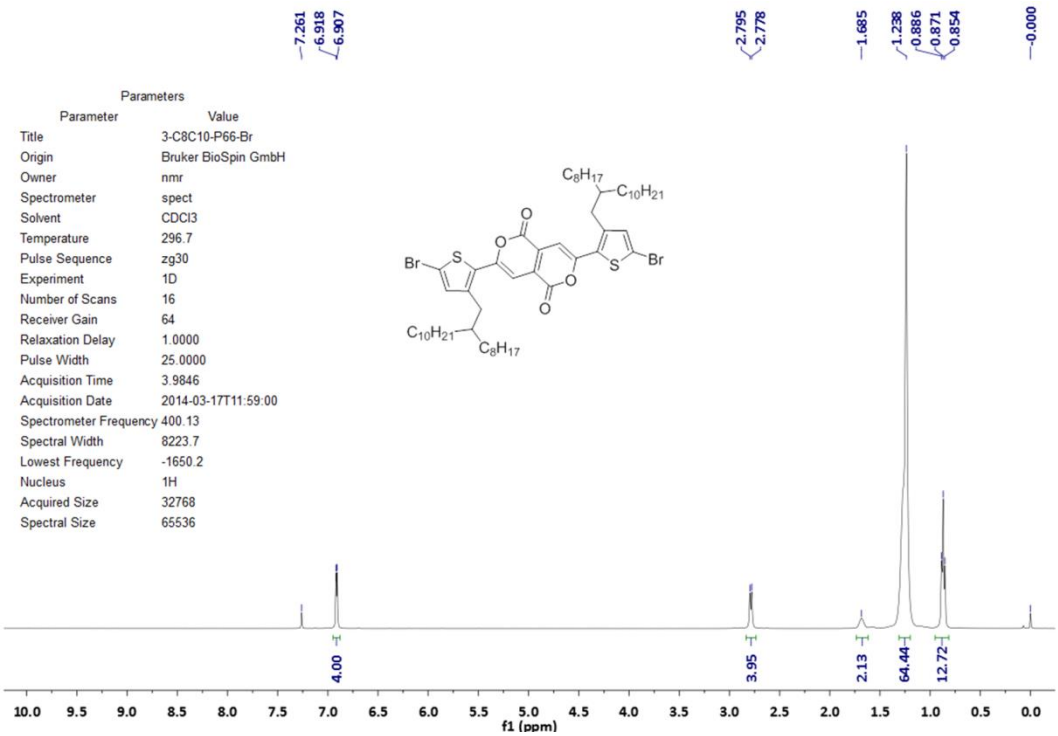
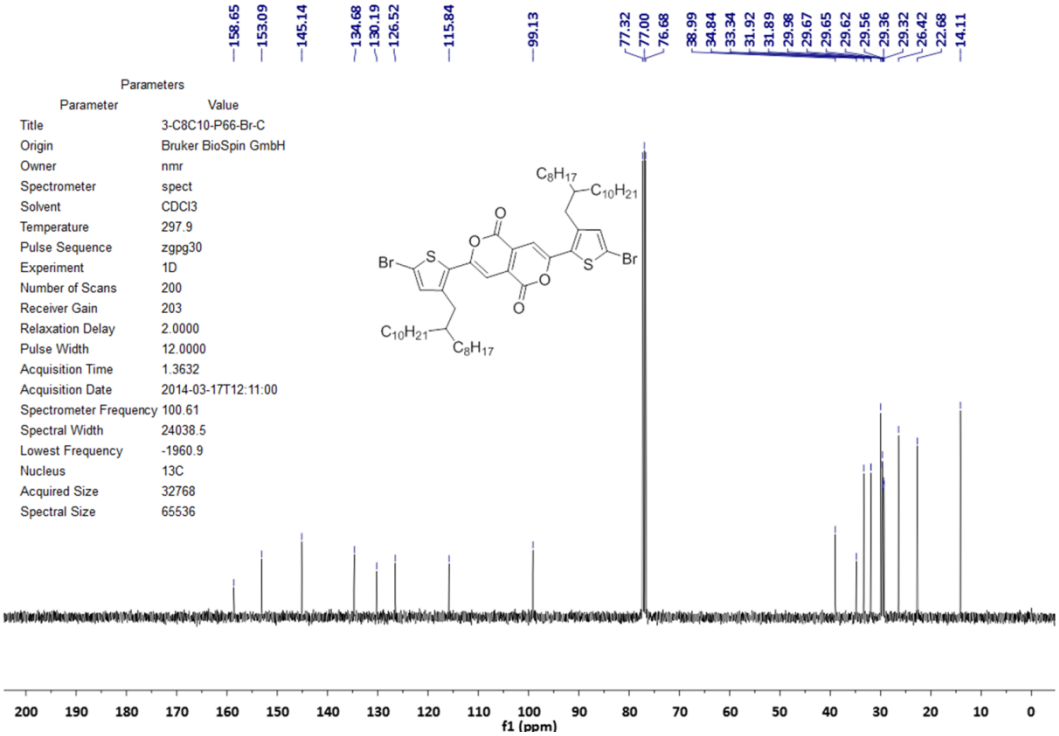
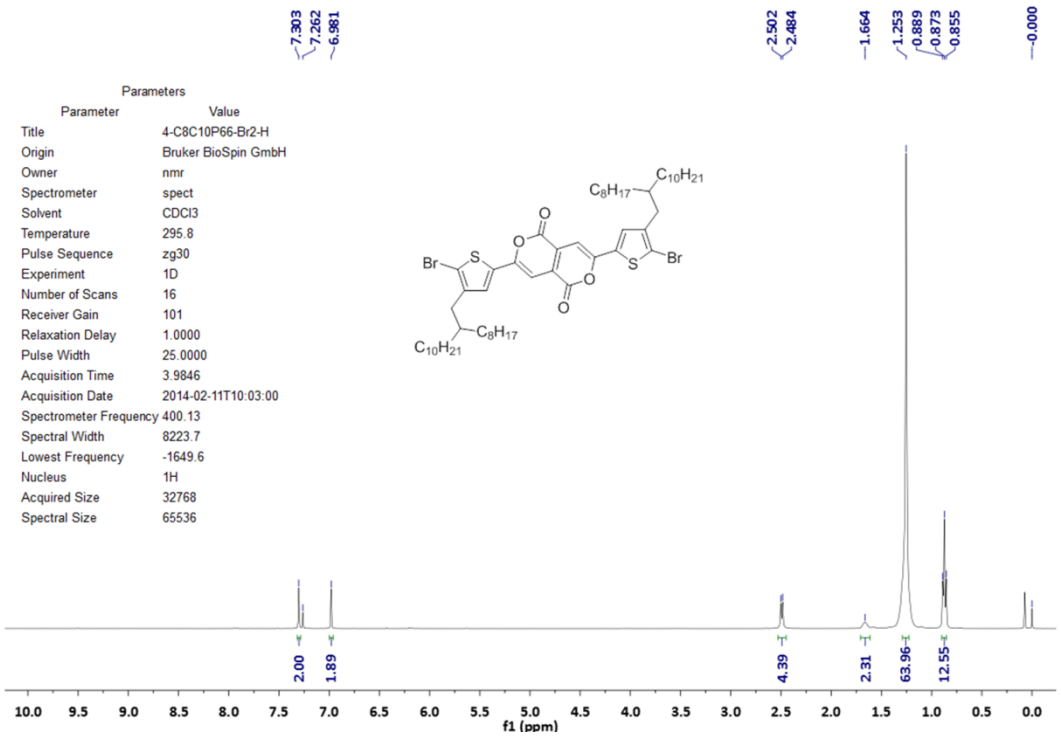
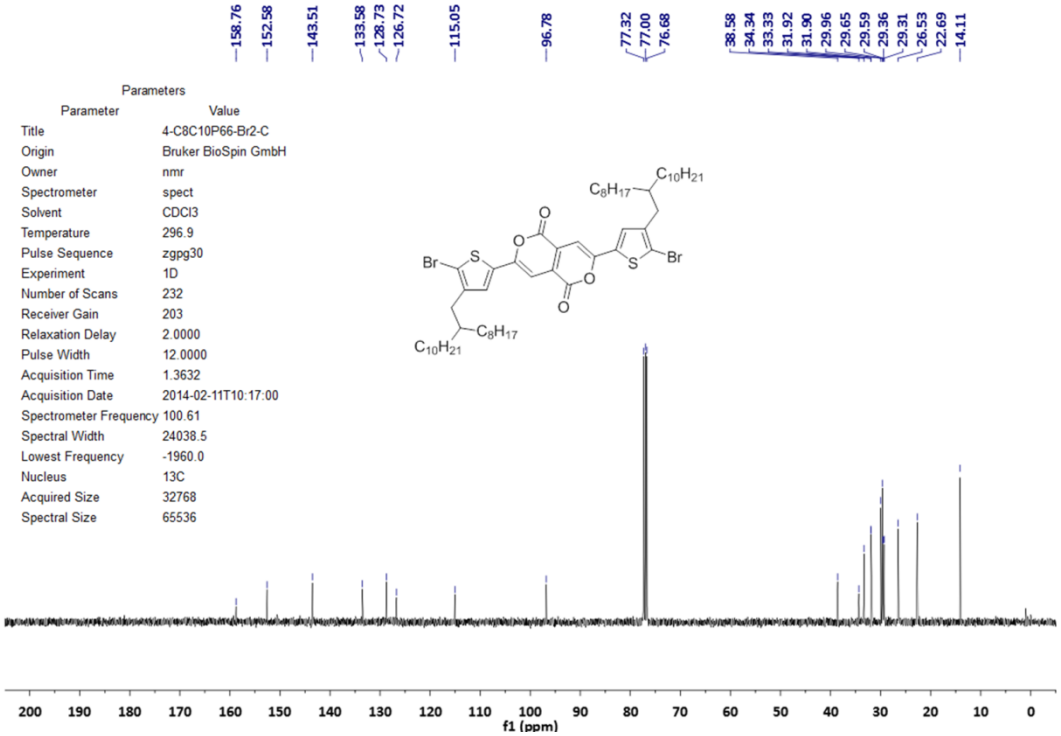
7.261
7.084
7.067
2.634
2.615
2.596
1.649
1.633
1.615
1.318
0.912
0.896
0.880
0.000
Parameters
Parameter Value
Title 4-C6-P66-H
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 298.0
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 203
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-03-19T05:43:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1650.1
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
2.00
3.94
4.14
4.26
12.45
6.32
f1 (ppm)


159.20
153.56
145.04
134.26
128.76
126.71
124.50
96.68
77.32
77.00
76.68
31.60
30.34
30.30
28.86
22.56
14.06
Parameters
Parameter Value
Title 4-C6-P66-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 299.2
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 720
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-03-19T06:25:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1959.8
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)


7.261
6.918
6.907
2.795
2.778
1.685
1.238
0.886
0.871
0.854
0.000
Parameters
Parameter Value
Title 3-C8C10-P66-Br
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 296.7
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 64
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-03-17T11:59:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1650.2
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
4.00
3.95
2.13
64.44
12.72
f1 (ppm)


158.65
153.09
145.14
134.68
130.19
126.52
115.84
99.13
77.32
77.00
76.68
38.99
34.84
33.34
31.92
31.89
29.98
29.67
29.65
29.62
29.56
29.36
29.32
26.42
22.68
14.11
Parameters
Parameter Value
Title 3-C8C10-P66-Br-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 297.9
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 200
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-03-17T12:11:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1960.9
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)


7.303
7.262
6.981
2.502
2.484
1.664
1.253
0.889
0.873
0.855
0.000
Parameters
Parameter Value
Title 4-C8C10P66-Br2-H
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 295.8
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 101
Relaxation Delay 1.0000
Pulse Width 25.0000
Acquisition Time 3.9846
Acquisition Date 2014-02-11T10:03:00
Spectrometer Frequency 400.13
Spectral Width 8223.7
Lowest Frequency -1649.6
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
2.00
1.89
4.39
2.31
63.96
12.55
f1 (ppm)


158.76
152.58
143.51
133.58
128.73
126.72
115.05
96.78
77.32
77.00
76.68
38.58
34.34
33.33
31.92
31.90
29.96
29.65
29.59
29.36
29.31
26.53
22.69
14.11
Parameters
Parameter Value
Title 4-C8C10P66-Br2-C
Origin Bruker BioSpin GmbH
Owner nmr
Spectrometer spect
Solvent CDCl3
Temperature 296.9
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 232
Receiver Gain 203
Relaxation Delay 2.0000
Pulse Width 12.0000
Acquisition Time 1.3632
Acquisition Date 2014-02-11T10:17:00
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1960.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
f1 (ppm)