

Supporting Information for

Gold-catalyzed π -directed regioselective cyclization of bis(*o*-alkynyl benzyl alcohols): a rapid access to dihydroisobenzofuran derivatives

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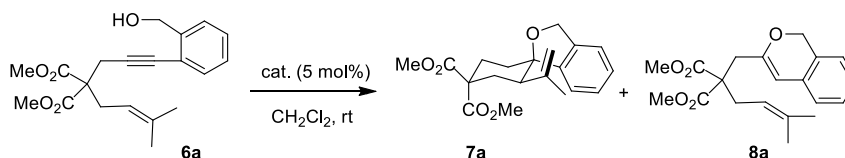
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-The details for the reaction of enynol 6a by gold catalysis.

A variety of gold catalysts were screened. As seen from the below Table 1, $\text{Ph}_3\text{PAuNTf}_2$ was proved to be effective and selective.

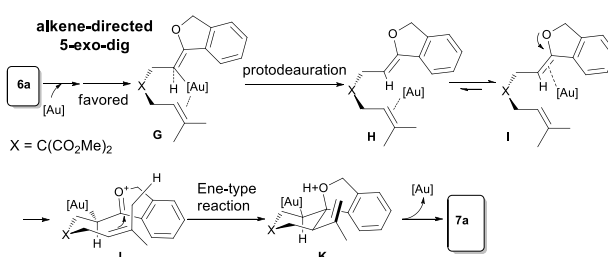
Table 1. Effects of the Catalysts^a



Entry	catalyst	Time (h)	Yield of 7a+8a (%) ^b	7a/8a ^c
1	$\text{PPh}_3\text{AuNTf}_2$	1	92	>20/1
2	$(o\text{-CH}_3\text{C}_6\text{H}_4)_3\text{P AuNTf}_2$	3	75	8/1
3	$(p\text{-CH}_3\text{OC}_6\text{H}_4)_3\text{P AuNTf}_2$	2	82	14/1
4	$(2,4\text{-}^t\text{Bu}_2\text{C}_6\text{H}_4\text{O})_3\text{P AuNTf}_2$	1	60	5/1
5	$\text{Cy}_3\text{PAuNTf}_2$	6	81	1.2/1
6	$\text{CyJohnPhos AuNTf}_2$	6	86	1/2
7	XPhos AuNTf_2	2	79	1/4
8	JohnPhosAuNTf_2	1	92	0/1
9	IPrAuNTf_2	2	73	1/15

^a All reactions were performed using **6a** (0.1 mmol), catalyst (5 mol%) in CH_2Cl_2 (1 mL) at room temperature unless otherwise noted. ^b Yield of isolated products (**7a+8a**) via column chromatography. ^c Judged by ^1H NMR spectroscopic analysis of the crude products.

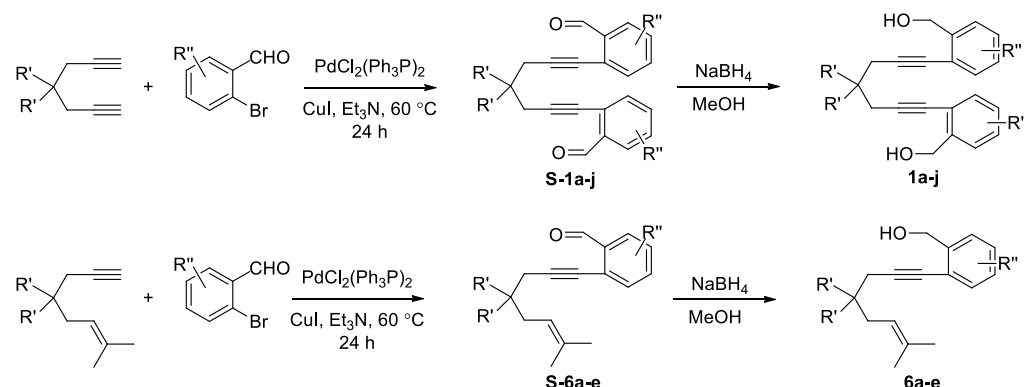
A possible mechanism for this reaction is shown in Scheme 1. Initially, the coordination of a gold catalyst to the alkyne and then 5-exo-dig cyclization could give intermediate **G**, which led to complex **H** by protodeauration. Complex **I** could be equilibrium with complex **H**. The auroated complex **J** can be formed by tautomerization of the enol ether **I**, followed by an Ene type reaction by a chair-like conformation to afford the complex **K**. After protodeauration, the desired product **7a** can be provided.



Scheme 1 Possible mechanism for the formation of **7a**.

-Synthesis of the substrates 1a-1j and 6a-e.

General route



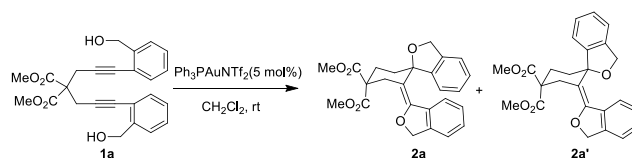
A schlenk flask charged with a stirring bar and $\text{PdCl}_2(\text{Ph}_3\text{P})_2$ (4 mol%) was evacuated and filled with argon three times, then Et_3N (6 mL), the diyne (2 mmol) and *o*-bromoarylaldehyde (4.4 mmol) was added successively. The mixture was stirred at room temperature for 2 minutes before CuI (4 mol%) was added. The resulting reaction mixture was stirred at 60 °C for 24 hours and quenched with aqueous NH_4Cl (6 mL), extracted with EtOAc (3 x 15 mL), washed by brine (6 mL), the organic phase was dried over MgSO_4 , and then filtered. After removal of the solvent, the residue was purified by flash column chromatography (silica gel 300 mesh: petrol ether/ethyl acetate = 20:1) to give the pure **S-1a-j** in 55-90% yields without further optimization.

The **S-6a-e** was obtained from the reaction of the corresponding enyne (3 mmol), *o*-bromoarylaldehyde (2 mmol), $\text{PdCl}_2(\text{Ph}_3\text{P})_2$ (2 mol%) and CuI (2 mol%) in Et_3N (6 mL) in 45-85% yields usually without further optimization.

To a solution of the above dialdehyde **S-1a-j** (1 mmol) in methanol (3 mL) at -10 °C, NaBH_4 (2.1 eq) was added in batches. The reaction was stirred for additional 10 minutes at the same temperature and then quenched with water (1 mL). The resulting mixture was extracted with EtOAc (3 X 10 mL). The organic phase was combined and washed with brine (10 mL), dried over MgSO_4 , and filtered. After removal of the solvent under reduced pressure, the residue was purified by flash column chromatography (silica gel 300 mesh: petrol ether/ethyl acetate = 3:1) to give the pure **1a-j** in 35-70% yields.

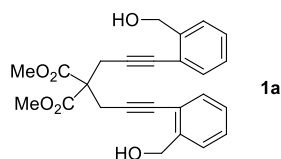
The substrate **6a-e** was obtained from the reaction of the corresponding aldehyde **S-6a-e** (1 mmol) and NaBH_4 (1.05 eq) in methanol in 40-85% yields.

-Typical procedure for gold catalysis.



To a solution of the substrate **1a** (0.2 mmol) in dichloromethane (2 mL), $\text{Ph}_3\text{PAuNTf}_2$ (5 mol%) was added. The resulting solution was stirred at room temperature. After finishing the reaction, the solvent was removed. The crude residue was purified by flash column chromatography on silica gel (petrol ether/ethyl acetate = 20:1) to give the desired product **2a** in 84% yield with a 14/1 mixture of the isomers (**2a/2a'** = 14/1).

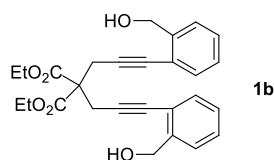
-Characterization for the substrates 1a-1j and 6a-e.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.40 (dd, $J = 7.5, 7.5$ Hz, 4H), 7.31 (ddd, $J = 7.5, 7.5, 1.3$ Hz, 2H), 7.23 (ddd, $J = 7.5, 7.5, 1.4$ Hz, 2H), 4.75 (s, 4H), 3.83 (s, 6H), 3.32 (s, 4H), 2.52 (b, 2H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 169.7, 142.6, 132.5, 128.6, 127.7, 127.5, 121.3, 88.3, 81.7, 63.9, 56.9, 53.4, 24.3.

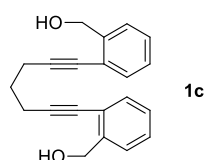
HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{25}\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 421.1646, Found: 421.1650.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.44-7.37 (m, 4H), 7.30 (ddd, $J = 7.5, 7.5, 1.3$ Hz, 2H), 7.22 (ddd, $J = 7.5, 7.4, 1.2$ Hz, 2H), 4.75 (s, 4H), 4.29 (q, $J = 7.1$ Hz, 4H), 3.31 (s, 4H), 2.60 (b, 2H), 1.29 (t, $J = 7.1$ Hz, 6H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 169.3, 142.6, 132.5, 128.5, 127.8, 127.4, 121.4, 88.5, 81.6, 63.9, 62.3, 56.8, 24.2, 14.0.

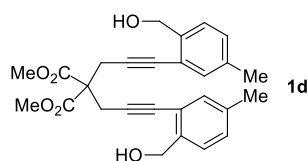
HRMS (ESI) calcd. For $\text{C}_{27}\text{H}_{29}\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 449.1959, Found: 449.1959.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.42 (d, $J = 7.6$ Hz, 2H), 7.39 (d, $J = 7.4$ Hz, 2H), 7.29 (ddd, $J = 7.5, 7.4, 1.2$ Hz, 2H), 7.22 (ddd, $J = 7.5, 7.5, 1.3$ Hz, 2H), 4.80 (s, 4H), 2.67 (t, $J = 6.9$ Hz, 4H), 2.30 (b, 2H), 1.93 (quint, $J = 6.9$ Hz, 2H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 142.4, 132.2, 128.1, 127.4, 127.3, 121.9, 93.8, 79.1, 64.0, 27.5, 18.6.

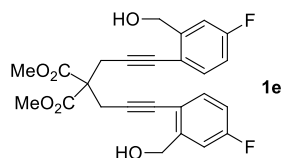
HRMS (ESI) calcd. For $\text{C}_{21}\text{H}_{21}\text{O}_2$ ($\text{M} + \text{H}$) $^+$: 305.1536, Found: 305.1537.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.26 (d, $J = 7.8$ Hz, 2H), 7.22 (s, 2H), 7.10 (d, $J = 7.8$ Hz, 2H), 4.70 (s, 4H), 3.82 (s, 6H), 3.31 (s, 4H), 2.35 (b, 2H), 2.30 (s, 6H).

^{13}C NMR (100 M, CDCl_3): δ 169.7, 139.8, 137.2, 133.0, 129.4, 127.8, 121.2, 87.8, 81.9, 63.7, 57.0, 53.3, 24.3, 20.8.

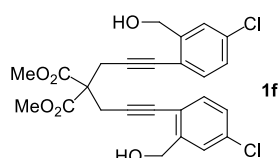
HRMS (ESI) calcd. For $\text{C}_{27}\text{H}_{29}\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 449.1959, Found: 449.1960.



The product was obtained as colorless oil; **^1H NMR** (400 M, CDCl_3): δ 7.37 (dd, J = 8.5, 5.6 Hz, 2H), 7.15 (dd, J = 9.3, 2.6 Hz, 2H), 6.91 (dd, J = 8.5, 8.3, 2.6 Hz, 2H), 4.73 (d, J = 5.3 Hz, 4H), 3.82 (s, 6H), 3.29 (s, 4H), 2.43 (t, J = 5.3 Hz, 2H).

^{13}C NMR (100 M, CDCl_3): δ 169.6, 162.7 (d, J = 248.5 Hz), 145.4 (d, J = 7.3 Hz), 134.2 (d, J = 8.2 Hz), 116.9 (d, J = 2.6 Hz), 114.6 (d, J = 18.2 Hz), 114.4 (d, J = 17.5 Hz), 88.0, 80.7, 63.4, 56.9, 53.3, 24.3.

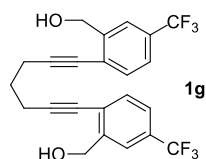
HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{23}\text{F}_2\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 457.1457, Found: 457.1460.



The product was obtained as colorless oil; **^1H NMR** (400 M, CDCl_3): δ 7.40 (d, J = 2.0 Hz, 2H), 7.30 (d, J = 8.2 Hz, 2H), 7.18 (dd, J = 8.2, 2.0 Hz, 2H), 4.70 (s, 4H), 3.81 (s, 6H), 3.28 (s, 4H), 2.72 (b, 2H).

^{13}C NMR (100 M, CDCl_3): δ 169.6, 144.4, 134.6, 133.5, 127.6, 127.5, 119.4, 89.2, 80.7, 63.2, 56.7, 53.4, 24.3.

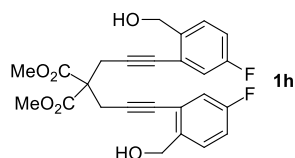
HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{23}\text{Cl}_2\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 489.0866, Found: 489.0865.



The product was obtained as colorless oil; **^1H NMR** (400 M, CDCl_3): δ 7.72 (s, 2H), 7.50 (d, J = 8.1 Hz, 2H), 7.47 (dd, J = 8.2, 1.3 Hz, 2H), 4.86 (s, 4H), 2.70 (t, J = 6.9 Hz, 4H), 2.33 (b, 2H), 1.96 (quint, J = 6.9 Hz, 2H).

^{13}C NMR (100 M, CDCl_3): δ 143.2, 132.4, 129.9 (q, J = 32.3 Hz), 125.2, 124.0 (q, J = 3.8 Hz), 123.7 (q, J = 3.7 Hz), 122.5, 96.4, 78.1, 63.3, 27.2, 18.7.

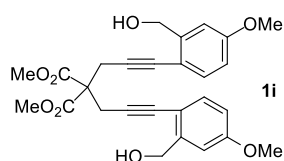
HRMS (ESI) calcd. For $\text{C}_{23}\text{H}_{19}\text{F}_6\text{O}_2$ ($\text{M} + \text{H}$) $^+$: 441.1284, Found: 441.1286.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.35 (dd, $J = 8.5, 5.8$ Hz, 2H), 7.09 (dd, $J = 9.1, 2.7$ Hz, 2H), 7.00 (ddd, $J = 8.5, 8.4, 2.7$ Hz, 2H), 4.70 (s, 4H), 3.83 (s, 6H), 3.30 (s, 4H), 2.54 (b, 2H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 169.5, 161.7 (d, $J = 245.2$ Hz), 138.6 (d, $J = 3.0$ Hz), 129.6 (d, $J = 8.6$ Hz), 123.2 (d, $J = 9.6$ Hz), 119.1 (d, $J = 22.9$ Hz), 115.8 (d, $J = 21.0$ Hz), 89.2, 80.8 (d, $J = 3.0$ Hz), 63.2, 56.8, 53.4, 24.3.

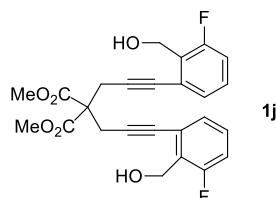
HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{23}\text{F}_2\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 457.1457, Found: 457.1460.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.33 (d, $J = 8.5$ Hz, 2H), 6.95 (d, $J = 2.6$ Hz, 2H), 6.75 (dd, $J = 8.5, 2.6$ Hz, 2H), 4.72 (s, 4H), 3.812 (s, 6H), 3.810 (s, 6H), 3.29 (s, 4H), 2.54 (b, 2H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 169.8, 159.8, 144.4, 133.8, 113.3, 113.2, 113.0, 86.8, 81.5, 64.0, 57.0, 55.3, 53.3, 24.3.

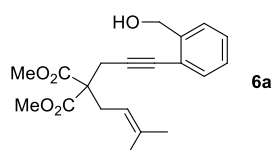
HRMS (ESI) calcd. For $\text{C}_{27}\text{H}_{29}\text{O}_8$ ($\text{M} + \text{H}$) $^+$: 481.1857, Found: 481.1856



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.24-7.17 (m, 4H), 7.07-7.00 (m, 2H), 4.81 (s, 4H), 3.84 (s, 6H), 3.32 (s, 4H), 2.58 (b, 2H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 169.6, 161.0 (d, $J = 245.6$ Hz), 129.5 (d, $J = 16.5$ Hz), 129.3 (d, $J = 9.4$ Hz), 128.5 (d, $J = 3.2$ Hz), 124.6 (d, $J = 5.4$ Hz), 116.0 (d, $J = 22.8$ Hz), 88.7, 81.1 (d, $J = 4.0$ Hz), 56.8, 56.7 (d, $J = 4.9$ Hz), 53.5, 24.4.

HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{23}\text{F}_2\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 457.1457, Found: 457.1457.

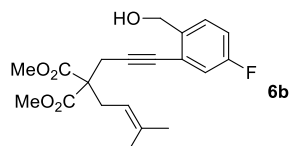


The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.41-7.36 (m, 2H), 7.29 (ddd, $J = 7.6, 7.5, 1.2$ Hz, 1H), 7.22 (ddd, $J = 7.6, 7.4, 1.2$ Hz, 1H), 4.94 (tt, $J = 7.7, 1.3$ Hz, 1H),

4.73 (d, $J = 6.3$ Hz, 2H), 3.76 (s, 6H), 3.03 (s, 2H), 2.83 (d, $J = 7.7$ Hz, 2H), 2.55 (t, $J = 6.3$ Hz, 1H), 1.71 (s, 3H), 1.66 (s, 3H).

^{13}C NMR (100 M, CDCl_3): δ 170.8, 142.6, 137.1, 132.4, 128.4, 127.8, 127.5, 121.7, 116.9, 89.4, 81.1, 64.0, 57.5, 52.8, 31.1, 26.1, 23.8, 18.0.

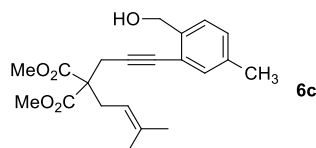
HRMS (ESI) calcd. For $\text{C}_{20}\text{H}_{25}\text{O}_5$ ($\text{M} + \text{H}$) $^+$: 345.1697, Found: 345.1698.



The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.34 (dd, $J = 8.5, 5.8$ Hz, 1H), 7.07 (dd, $J = 9.2, 2.7$ Hz, 1H), 6.98 (ddd, $J = 8.5, 8.4, 2.7$ Hz, 1H), 4.94 (tt, $J = 7.7, 1.4$ Hz, 1H), 4.68 (s, 2H), 3.76 (s, 6H), 3.02 (s, 2H), 2.81 (d, $J = 7.7$ Hz, 2H), 2.57 (b, 1H), 1.71 (s, 3H), 1.65 (s, 3H).

^{13}C NMR (100 M, CDCl_3): δ 170.8, 161.7 (d, $J = 244.8$ Hz), 138.7 (d, $J = 3.0$ Hz), 137.2, 129.6 (d, $J = 8.6$ Hz), 123.5 (d, $J = 9.7$ Hz), 119.0 (d, $J = 22.8$ Hz), 116.8, 115.5 (d, $J = 21.1$ Hz), 90.5, 80.1 (d, $J = 3.2$ Hz), 63.2, 57.4, 52.8, 31.2, 26.0, 23.8, 18.0.

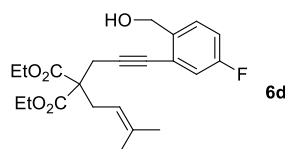
HRMS (ESI) calcd. For $\text{C}_{20}\text{H}_{24}\text{FO}_5$ ($\text{M} + \text{H}$) $^+$: 363.1602, Found: 363.1601.



The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.25 (d, $J = 8.0$ Hz, 1H), 7.20 (s, 1H), 7.09 (d, $J = 8.0$ Hz, 1H), 4.95 (tt, $J = 7.7, 1.3$ Hz, 1H), 4.68 (s, 2H), 3.76 (s, 6H), 3.02 (s, 2H), 2.83 (d, $J = 7.7$ Hz, 2H), 2.51 (b, 1H), 2.30 (s, 3H), 1.71 (s, 3H), 1.66 (s, 3H).

^{13}C NMR (100 M, CDCl_3): δ 170.8, 139.8, 137.2, 137.1, 132.9, 129.2, 127.9, 121.6, 116.9, 88.8, 81.2, 63.8, 57.5, 52.8, 31.1, 26.1, 23.8, 20.9, 18.0.

HRMS (ESI) calcd. For $\text{C}_{21}\text{H}_{27}\text{O}_5$ ($\text{M} + \text{H}$) $^+$: 359.1853, Found: 359.1856.

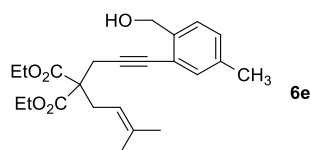


The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.34 (dd, $J = 8.5, 5.8$ Hz, 1H), 7.07 (dd, $J = 9.2, 2.7$ Hz, 1H), 6.98 (ddd, $J = 8.5, 8.4, 2.7$ Hz, 1H), 4.96 (tt, $J = 7.6, 1.4$ Hz, 1H), 4.69 (s, 2H), 4.23 (dq, $J = 7.1, 1.8$ Hz, 4H), 3.02 (s, 2H), 2.81 (d, $J = 7.6$ Hz, 2H), 1.71 (s, 3H), 1.66 (s, 3H), 1.65 (b, 1H), 1.26 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (100 M, CDCl_3): δ 170.4, 161.7 (d, $J = 244.6$ Hz), 138.7 (d, $J = 3.2$ Hz), 136.9, 129.7 (d, $J = 8.7$ Hz), 123.7 (d, $J = 9.9$ Hz), 118.9 (d, $J = 22.8$ Hz), 116.9, 115.4 (d, $J = 21.0$ Hz), 90.7, 80.1

(d, $J = 2.9$ Hz), 63.3, 61.7, 57.3, 31.0, 26.1, 23.7, 18.0, 14.0.

HRMS (ESI) calcd. For $C_{22}H_{28}FO_5$ ($M + H$)⁺: 391.1915, Found: 391.1921.

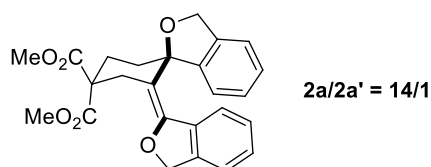


The product was obtained as colorless oil; ¹H NMR (400 M, CDCl₃): δ 7.25 (d, $J = 7.8$ Hz, 1H), 7.20 (s, 1H), 7.09 (d, $J = 7.8$ Hz, 1H), 4.97 (tt, $J = 7.7, 1.3$ Hz, 1H), 4.69 (s, 2H), 4.22 (dq, $J = 7.1, 2.1$ Hz, 4H), 3.02 (s, 2H), 2.83 (d, $J = 7.6$ Hz, 2H), 2.56 (b, 1H), 2.30 (s, 3H), 1.71 (s, 3H), 1.67 (s, 3H), 1.26 (t, $J = 7.1$ Hz, 6H).

¹³C NMR (100 M, CDCl₃): δ 170.4, 139.8, 137.2, 136.7, 132.9, 129.1, 128.0, 121.7, 117.1, 89.1, 81.2, 63.8, 61.6, 57.4, 31.0, 26.0, 23.7, 20.8, 18.0, 14.0.

HRMS (ESI) calcd. For $C_{23}H_{31}O_5$ ($M + H$)⁺: 387.2166, Found: 387.2168.

-Characterization for the products 2a-2g, 2h and 7a-e, 8a.

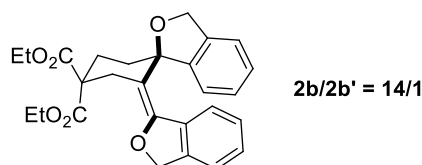


The product was obtained as colorless oil; ¹H NMR (400 M, CDCl₃): δ 7.59 (d, $J = 7.8$ Hz, 1H), 7.39-7.33 (m, 1H), 7.32-7.27 (m, 1H), 7.25-7.16 (m, 4H), 6.89 (d, $J = 7.6$ Hz, 1H), 5.27 (d, $J = 12.5$ Hz, 1H), 5.11 (d, $J = 12.5$ Hz, 1H), 4.96 (d, $J = 12.6$ Hz, 1H), 4.43 (d, $J = 12.6$ Hz, 1H), 3.68 (s, 3H), 3.51 (s, 3H), 3.20-3.08 (m, 1H), 2.99-2.91 (m, 1H), 2.50-2.38 (m, 2H), 2.33 (d, $J = 17.8$ Hz, 1H), 2.12-2.02 (m, 1H).

¹³C NMR (100 M, CDCl₃): δ 172.3, 171.1, 140.5, 140.0, 138.8, 134.1, 131.2, 128.9, 127.7, 127.3, 127.2, 126.8, 126.6, 122.4, 121.0, 114.9, 71.6, 66.6, 53.2, 52.7, 52.3, 33.7, 30.0, 28.0.

HRMS (ESI) calcd. For $C_{25}H_{25}O_6$ ($M + H$)⁺: 421.1646, Found: 421.1646.

H⁺

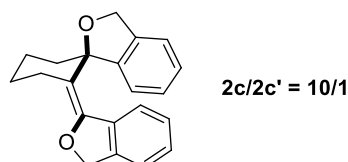


The product was obtained as colorless oil; ¹H NMR (500 M, CDCl₃): δ 7.59 (d, $J = 8.0$ Hz, 1H), 7.38-7.32 (m, 1H), 7.31-7.15 (m, 5H), 6.90 (d, $J = 7.7$ Hz, 1H), 5.26 (d, $J = 12.6$ Hz, 1H), 5.10 (d, $J = 12.6$ Hz, 1H), 4.96 (d, $J = 12.6$ Hz, 1H), 4.43 (d, $J = 12.6$ Hz, 1H), 4.14 (q, $J = 7.1$ Hz, 2H),

4.13-4.07 (m, 1H), 3.89-3.81 (m, 1H), 3.23-3.12 (m, 1H), 2.98 (dd, $J = 18.1, 2.4$ Hz, 1H), 2.49-2.36 (m, 2H), 2.31 (d, $J = 17.9$ Hz, 1H), 2.11-2.01 (m, 1H), 1.20 (t, $J = 7.1$ Hz, 3H), 1.06 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (125.8 M, CDCl_3): δ 171.9, 170.8, 140.5, 140.1, 140.0, 138.9, 134.2, 131.3, 128.9, 127.7, 127.4, 127.2, 126.7, 126.6, 122.3, 120.9, 114.9, 71.6, 66.6, 61.4, 61.1, 53.1, 33.5, 30.0, 27.9, 14.0, 13.9.

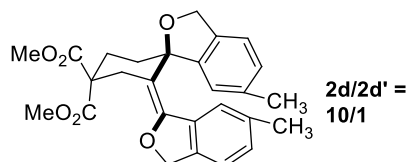
HRMS (ESI) calcd. For $\text{C}_{27}\text{H}_{29}\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 449.1959, Found: 449.1964.



The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.70 (d, $J = 7.7$ Hz, 1H), 7.22-7.15 (m, 3H), 7.14-7.06 (m, 3H), 7.03 (d, $J = 7.1$ Hz, 1H), 5.01 (d, $J = 12.3$ Hz, 1H), 4.96 (d, $J = 16.2$ Hz, 1H), 4.91 (d, $J = 16.2$ Hz, 1H), 4.82 (d, $J = 12.3$ Hz, 1H), 3.87 (d, $J = 17.0$ Hz, 1H), 2.51 (d, $J = 17.0$ Hz, 1H), 2.34-2.29 (m, 2H), 2.26 (ddd, $J = 14.1, 6.3, 2.5$ Hz, 1H), 1.95-1.82 (m, 1H), 1.74-1.64 (m, 1H), 1.50-1.37 (m, 1H).

^{13}C NMR (100 M, CDCl_3): δ 157.0, 133.5, 133.1, 130.4, 129.5, 129.2, 127.7, 126.5, 125.8, 125.5, 124.2, 123.9, 123.6, 113.4, 73.4, 68.6, 62.4, 36.3, 31.0, 28.4, 17.2.

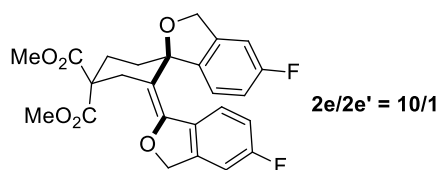
HRMS (ESI) calcd. For $\text{C}_{21}\text{H}_{21}\text{O}_2$ ($\text{M} + \text{H}$) $^+$: 305.1536, Found: 305.1540.



The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.42 (s, 1H), 7.12-7.07 (m, 3H), 7.03 (d, $J = 7.7$ Hz, 1H), 6.67 (s, 1H), 5.22 (d, $J = 12.3$ Hz, 1H), 5.06 (d, $J = 12.3$ Hz, 1H), 4.91 (d, $J = 12.6$ Hz, 1H), 4.39 (d, $J = 12.6$ Hz, 1H), 3.68 (s, 3H), 3.49 (s, 3H), 3.24-3.11 (m, 1H), 2.95 (dd, $J = 17.6, 2.1$ Hz, 1H), 2.50-2.37 (m, 2H), 2.40 (s, 3H), 2.37-2.28 (m, 1H), 2.26 (s, 3H), 2.11-2.01 (m, 1H).

^{13}C NMR (100 M, CDCl_3): δ 172.3, 171.2, 140.2, 138.6, 137.8, 137.2, 137.1, 137.0, 134.0, 131.1, 129.9, 128.0, 127.4, 126.6, 122.6, 120.7, 114.9, 71.5, 66.3, 53.2, 52.7, 52.2, 33.7, 29.9, 28.0, 21.5, 21.2.

HRMS (ESI) calcd. For $\text{C}_{27}\text{H}_{29}\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 449.1959, Found: 449.1958.

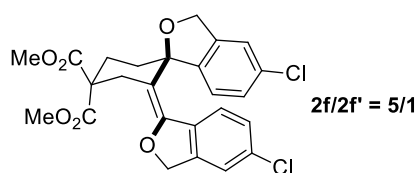


The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.53 (dd, $J = 8.8, 5.5$ Hz,

1H), 7.04 (ddd, $J = 8.6, 8.5, 2.8$ Hz, 1H), 6.95-6.83 (m, 4H), 5.21 (d, $J = 13.0$ Hz, 1H), 5.05 (d, $J = 13.0$ Hz, 1H), 4.91 (d, $J = 12.8$ Hz, 1H), 4.36 (d, $J = 12.8$ Hz, 1H), 3.69 (s, 3H), 3.56 (s, 3H), 3.11-2.99 (m, 1H), 2.89 (dd, $J = 17.8, 2.9$ Hz, 1H), 2.47-2.37 (m, 2H), 2.30 (d, $J = 17.6$ Hz, 1H), 2.12-2.03 (m, 1H).

^{13}C NMR (100 M, CDCl_3): δ 172.1, 171.0, 163.6 (d, $J = 245.0$ Hz), 161.1 (d, $J = 246.9$ Hz), 142.6 (d, $J = 7.0$ Hz), 142.4 (d, $J = 8.9$ Hz), 135.6 (d, $J = 2.1$ Hz), 134.8 (d, $J = 4.1$ Hz), 134.2 (d, $J = 13.7$ Hz), 133.4 (d, $J = 2.0$ Hz), 130.6, 129.1 (dd, $J = 20.2, 8.3$ Hz), 124.0 (d, $J = 9.5$ Hz), 114.6 (dd, $J = 34.4, 23.3$ Hz), 114.5, 113.5 (d, $J = 21.3$ Hz), 108.2 (d, $J = 23.9$ Hz), 71.8 (d, $J = 2.5$ Hz), 66.0, 53.2, 52.7, 52.4, 33.6, 30.2, 27.9.

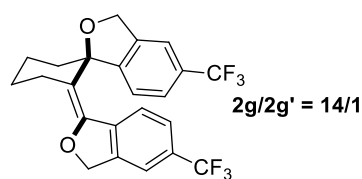
HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{23}\text{F}_2\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 457.1457, Found: 457.1459.



The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.49 (d, $J = 8.5$ Hz, 1H), 7.31 (dd, $J = 8.5, 2.3$ Hz, 1H), 7.23 (d, $J = 1.0$ Hz, 1H), 7.21-7.16 (m, 2H), 6.83 (d, $J = 8.1$ Hz, 1H), 5.20 (d, $J = 13.0$ Hz, 1H), 5.05 (d, $J = 13.0$ Hz, 1H), 4.89 (d, $J = 12.8$ Hz, 1H), 4.36 (d, $J = 12.8$ Hz, 1H), 3.68 (s, 3H), 3.56 (s, 3H), 3.10-2.98 (m, 1H), 2.88 (dd, $J = 17.8, 2.6$ Hz, 1H), 2.46-2.36 (m, 2H), 2.28 (d, $J = 17.8$ Hz, 1H), 2.11-2.01 (m, 1H).

^{13}C NMR (100 M, CDCl_3): δ 172.0, 171.0, 142.1, 142.0, 138.5, 137.1, 135.0, 134.1, 132.5, 130.7, 128.7, 127.8, 127.7, 126.6, 123.6, 121.4, 114.5, 71.0, 66.0, 53.1, 52.8, 52.5, 33.7, 30.0, 27.8.

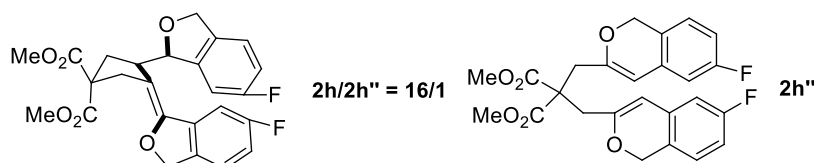
HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{23}\text{Cl}_2\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 489.0866, Found: 489.0870.



The product was obtained as colorless oil; ^1H NMR (400 M, CDCl_3): δ 7.74 (d, $J = 8.3$ Hz, 1H), 7.45 (d, $J = 8.0$ Hz, 1H), 7.41 (d, $J = 8.3$ Hz, 1H), 7.37 (s, 1H), 7.30 (s, 1H), 7.21 (d, $J = 8.0$ Hz, 1H), 5.08 (d, $J = 12.5$ Hz, 1H), 5.00 (d, $J = 16.2$ Hz, 1H), 4.94 (d, $J = 16.2$ Hz, 1H), 4.84 (d, $J = 12.5$ Hz, 1H), 3.81 (d, $J = 17.4$ Hz, 1H), 2.59 (d, $J = 17.4$ Hz, 1H), 2.37-2.31 (m, 2H), 2.24 (ddd, $J = 14.0, 6.2, 2.4$ Hz, 1H), 1.95-1.82 (m, 1H), 1.72-1.67 (m, 1H), 1.50-1.40 (m, 1H).

^{13}C NMR (100 M, CDCl_3): δ 159.6, 136.9, 134.1, 133.8, 130.0, 129.4, 128.5 (d, $J = 32.6$ Hz), 127.5 (d, $J = 32.2$ Hz), 125.5 (d, $J = 12.0$ Hz), 124.7 (q, $J = 3.7$ Hz), 123.4, 123.3 (q, $J = 3.7$ Hz), 122.8 (d, $J = 12.2$ Hz), 121.3 (q, $J = 3.8$ Hz), 120.9 (q, $J = 3.8$ Hz), 112.7, 73.2, 68.2, 62.1, 36.3, 30.9, 28.4, 16.9.

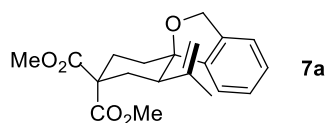
HRMS (ESI) calcd. For $\text{C}_{23}\text{H}_{19}\text{F}_6\text{O}_2$ ($\text{M} + \text{H}$) $^+$: 441.1284, Found: 441.1284.



The product was obtained as colorless oil; $^1\text{H NMR}$ (500 M, CDCl_3): δ 7.41 (dd, $J = 8.3, 6.0$ Hz, 1H), 7.21 (dd, $J = 8.3, 4.7$ Hz, 1H), 7.07-6.86 (m, 2H), 6.90 (dd, $J = 9.5, 2.2$ Hz, 1H), 6.82 (dd, $J = 8.0, 1.8$ Hz, 1H), 6.72 (s, 1H), 5.14 (d, $J = 12.3$ Hz, 1H), 5.01 (d, $J = 12.3$ Hz, 1H), 4.89 (d, $J = 12.8$ Hz, 1H), 4.46 (d, $J = 12.8$ Hz, 1H), 3.71 (s, 3H), 3.66 (s, 3H), 3.35 (d, $J = 14.4$ Hz, 1H), 3.05 (d, $J = 14.4$ Hz, 1H), 2.84 (dd, $J = 8.1, 7.8$ Hz, 1H), 2.42 (dd, $J = 13.6, 10.0$ Hz, 1H), 1.67 (dd, $J = 13.8, 7.0$ Hz, 1H).

$^{13}\text{C NMR}$ (125.8 M, CDCl_3): δ 171.6, 171.5, 162.8 (d, $J = 246.1$ Hz), 161.9 (d, $J = 247.3$ Hz), 143.8, 142.9 (d, $J = 7.7$ Hz), 139.2 (d, $J = 8.2$ Hz), 135.2 (d, $J = 1.4$ Hz), 133.3 (d, $J = 8.7$ Hz), 132.3 (d, $J = 2.4$ Hz), 124.3 (d, $J = 1.5$ Hz), 122.5 (d, $J = 8.6$ Hz), 116.2 (d, $J = 23.4$ Hz), 114.8 (d, $J = 21.6$ Hz), 114.2 (d, $J = 20.9$ Hz), 109.0 (d, $J = 23.6$ Hz), 107.1 (d, $J = 2.6$ Hz), 71.6, 64.1, 58.0, 52.8, 52.7, 47.0, 44.8, 38.2.

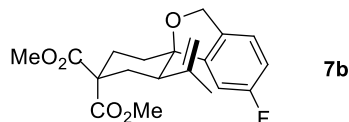
HRMS (ESI) calcd. For $\text{C}_{25}\text{H}_{23}\text{F}_2\text{O}_6$ ($\text{M} + \text{H}$) $^+$: 457.1457, Found: 457.1458.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.25-7.20 (m, 2H), 7.16-7.12 (m, 1H), 7.06-7.02 (m, 1H), 5.03 (s, 2H), 4.69 (b, 1H), 4.61 (dd, $J = 1.6, 1.6$ Hz, 1H), 3.82 (s, 3H), 3.74 (s, 3H), 2.49 (dd, $J = 13.1, 2.8$ Hz, 1H), 2.41 (dd, $J = 13.0, 12.6$ Hz, 1H), 2.36-2.24 (m, 3H), 1.84-1.76 (m, 2H), 1.43 (s, 3H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 172.4, 171.8, 145.2, 143.8, 138.9, 127.5, 126.9, 121.4, 120.9, 113.7, 88.6, 72.1, 55.0, 52.7, 52.6, 48.5, 36.0, 32.8, 27.2, 23.0.

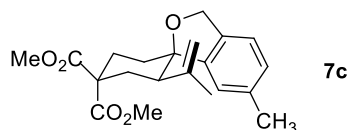
HRMS (ESI) calcd. For $\text{C}_{20}\text{H}_{25}\text{O}_5$ ($\text{M} + \text{H}$) $^+$: 345.1697, Found: 345.1701.



The product was obtained as colorless oil; $^1\text{H NMR}$ (400 M, CDCl_3): δ 7.07 (dd, $J = 8.2, 4.8$ Hz, 1H), 6.92 (ddd, $J = 8.8, 8.5, 2.3$ Hz, 1H), 6.72 (dd, $J = 8.5, 2.3$ Hz, 1H), 4.98 (s, 2H), 4.70 (b, 1H), 4.64 (dd, $J = 1.6, 1.6$ Hz, 1H), 3.82 (s, 3H), 3.74 (s, 3H), 2.46 (dd, $J = 13.0, 2.6$ Hz, 1H), 2.39 (dd, $J = 12.4, 12.9$ Hz, 1H), 2.36-2.22 (m, 3H), 1.80-1.73 (m, 2H), 1.47 (s, 3H).

$^{13}\text{C NMR}$ (100 M, CDCl_3): δ 172.3, 171.6, 162.5 (d, $J = 242.7$ Hz), 146.3 (d, $J = 7.5$ Hz), 144.9, 134.1 (d, $J = 1.6$ Hz), 122.1 (d, $J = 8.8$ Hz), 114.7 (d, $J = 22.8$ Hz), 114.0, 108.5 (d, $J = 23.2$ Hz), 88.6, 71.7, 54.9, 52.7, 52.6, 48.4, 35.8, 32.6, 27.1, 22.8.

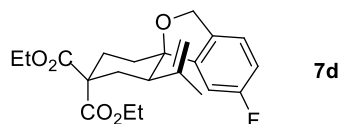
HRMS (ESI) calcd. For $C_{20}H_{24}FO_5$ ($M + H$)⁺: 363.1602, Found: 363.1600.



The product was obtained as colorless oil; **1H NMR** (400 M, $CDCl_3$): δ 7.04 (d, $J = 8.3$ Hz, 1H), 7.01 (d, $J = 8.1$ Hz, 1H), 6.83 (s, 1H), 4.98 (s, 2H), 4.70 (b, 1H), 4.62 (dd, $J = 1.8, 1.6$ Hz 1H), 3.83 (s, 3H), 3.74 (s, 3H), 2.47 (dd, $J = 13.2, 2.7$ Hz 1H), 2.40 (dd, $J = 13.0, 12.4$ Hz 1H), 2.34 (s, 3H), 2.32-2.20 (m, 3H), 1.81-1.74 (m, 2H), 1.43 (s, 3H).

^{13}C NMR (100 M, $CDCl_3$): δ 172.5, 171.8, 145.3, 144.1, 136.6, 136.0, 128.4, 121.9, 120.6, 113.6, 88.4, 72.0, 55.0, 52.7, 52.6, 48.4, 36.0, 32.8, 27.2, 23.1, 21.3.

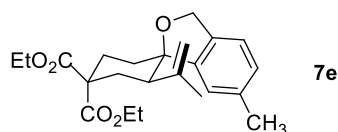
HRMS (ESI) calcd. For $C_{21}H_{27}O_5$ ($M + H$)⁺: 359.1853, Found: 359.1855.



The product was obtained as colorless oil; **1H NMR** (400 M, $CDCl_3$): δ 7.07 (dd, $J = 8.2, 4.8$ Hz, 1H), 6.92 (ddd, $J = 8.8, 8.6, 2.3$ Hz, 1H), 6.72 (dd, $J = 7.7, 2.3$ Hz, 1H), 4.98 (s, 2H), 4.71 (b, 1H), 4.64 (dd, $J = 1.6, 1.5$ Hz 1H), 4.37-4.21 (m, 2H), 4.19 (q, $J = 7.1$ Hz, 2H), 2.49 (dd, $J = 13.0, 3.4$ Hz, 1H), 2.38 (dd, $J = 13.0, 13.0$ Hz, 1H), 2.34-2.17 (m, 3H), 1.82-1.78 (m, 1H), 1.77 (d, $J = 3.6$ Hz, 1H), 1.48 (s, 3H), 1.31 (t, $J = 7.1$ Hz, 3H), 1.26 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (100 M, $CDCl_3$): δ 171.9, 171.2, 162.5 (d, $J = 242.4$ Hz), 146.4 (d, $J = 7.1$ Hz), 145.0, 134.2 (d, $J = 2.5$ Hz), 122.1 (d, $J = 8.8$ Hz), 114.7 (d, $J = 22.7$ Hz), 113.9, 108.5 (d, $J = 23.2$ Hz), 88.6, 71.7, 61.4, 61.2, 54.8, 48.4, 35.8, 32.6, 27.0, 22.8, 14.2, 14.1.

HRMS (ESI) calcd. For $C_{22}H_{28}FO_5$ ($M + H$)⁺: 391.1915, Found: 391.1915.

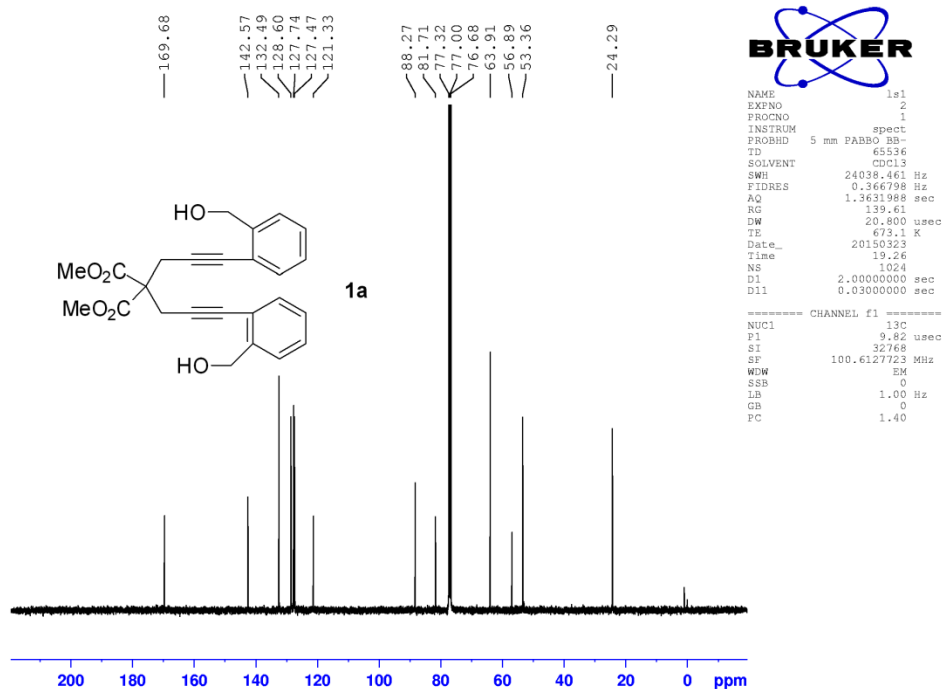
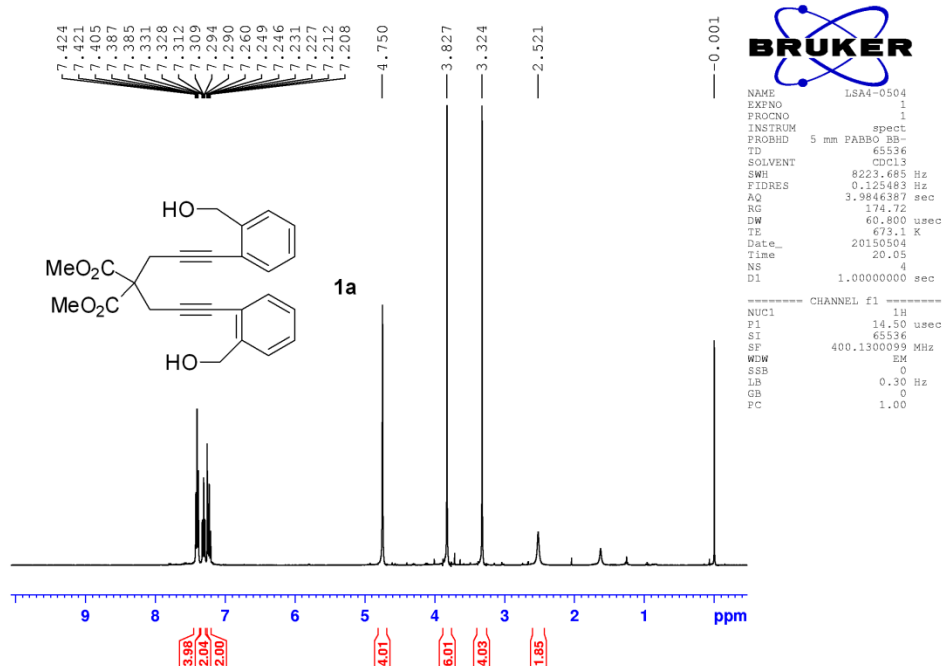


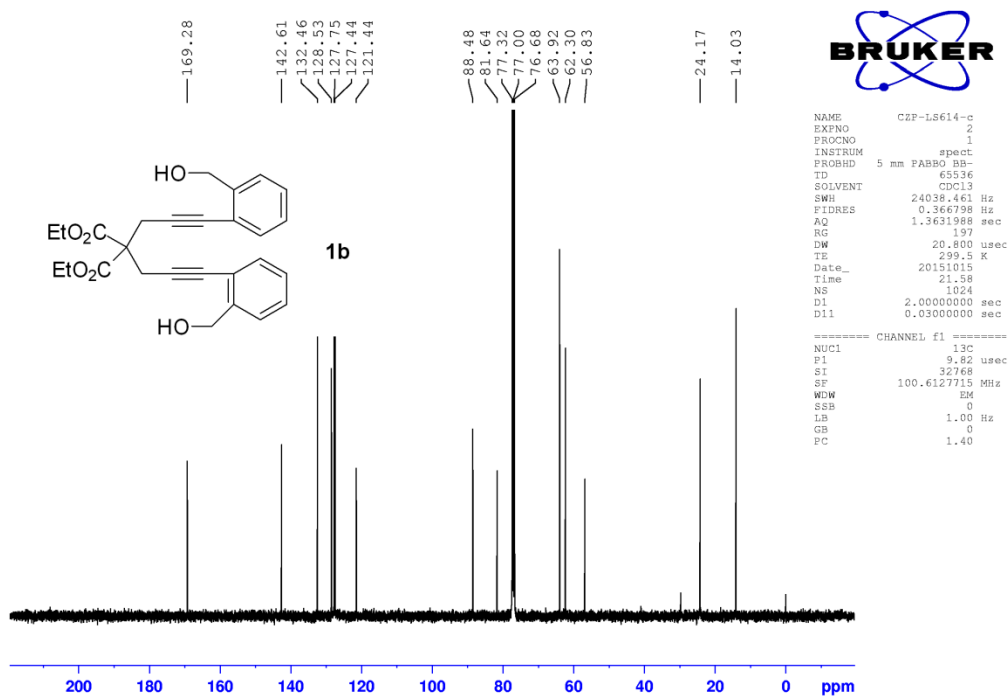
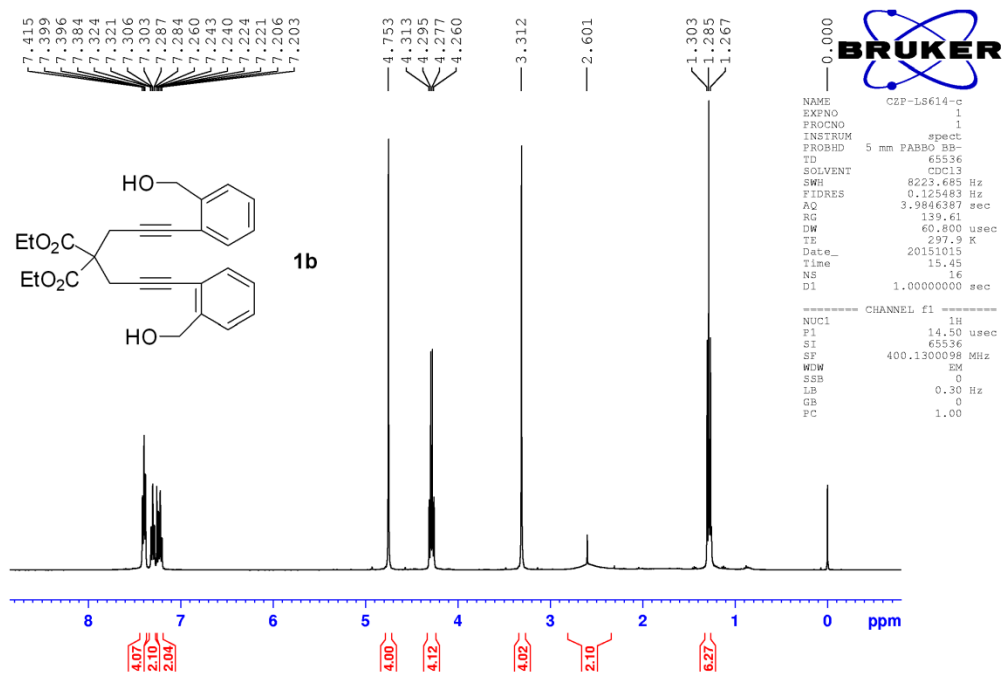
The product was obtained as colorless oil; **1H NMR** (400 M, $CDCl_3$): δ 7.06-6.99 (m, 2H), 6.82 (s, 1H), 4.98 (s, 2H), 4.70 (b, 1H), 4.61 (dd, $J = 1.8, 1.6$ Hz, 1H), 4.37-4.21 (m, 2H), 4.19 (q, $J = 7.1$ Hz, 2H), 2.50 (dd, $J = 13.1, 3.3$ Hz, 1H), 2.39 (dd, $J = 13.0, 13.0$ Hz 1H), 2.34 (s, 3H), 2.31-2.18 (m, 3H), 1.87-1.72 (m, 2H), 1.44 (s, 3H), 1.31 (t, $J = 7.1$ Hz, 3H), 1.26 (t, $J = 7.1$ Hz, 3H).

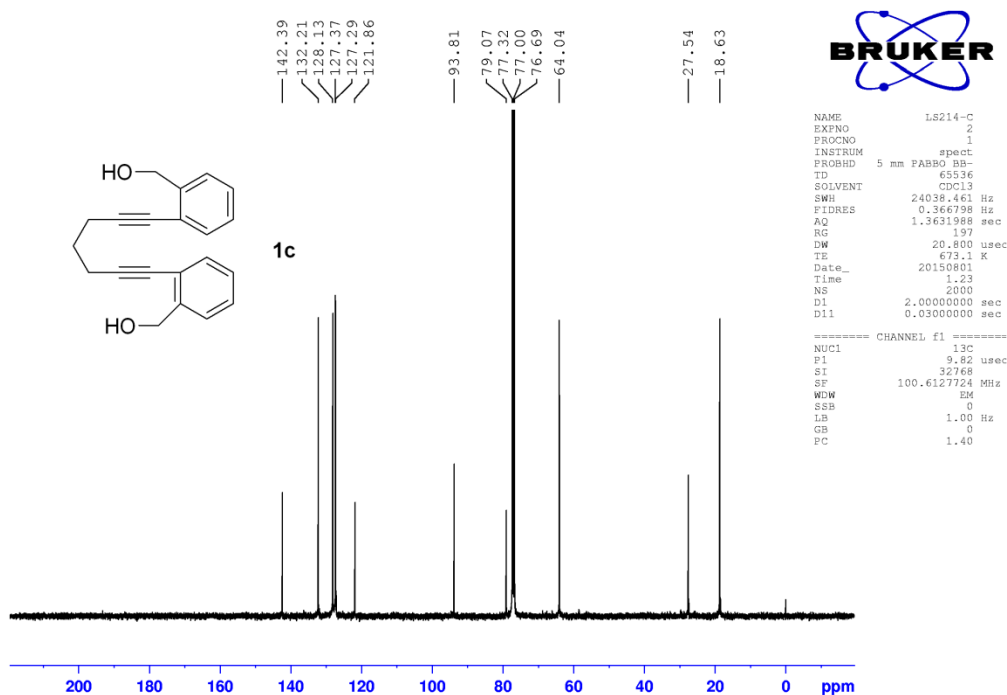
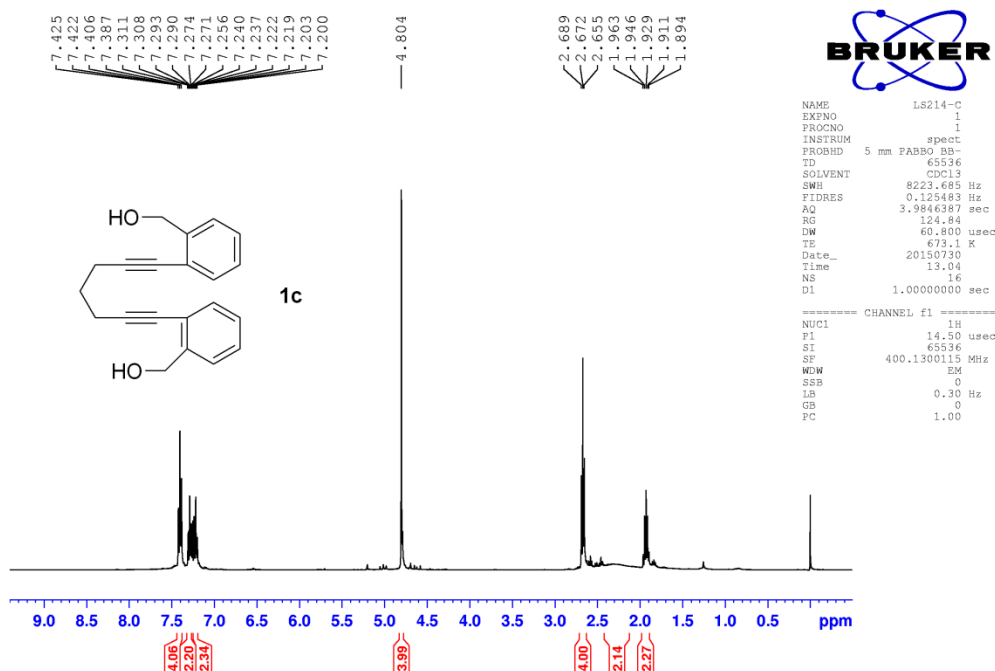
^{13}C NMR (100 M, $CDCl_3$): δ 172.0, 171.3, 145.5, 144.3, 136.6, 136.1, 128.4, 121.9, 120.6, 113.4, 88.5, 72.0, 61.3, 61.1, 55.0, 48.4, 36.0, 32.8, 27.2, 23.0, 21.3, 14.2, 14.1.

HRMS (ESI) calcd. For $C_{23}H_{31}O_5$ ($M + H$)⁺: 387.2166, Found: 387.2164.

-Copies of NMR spectra.









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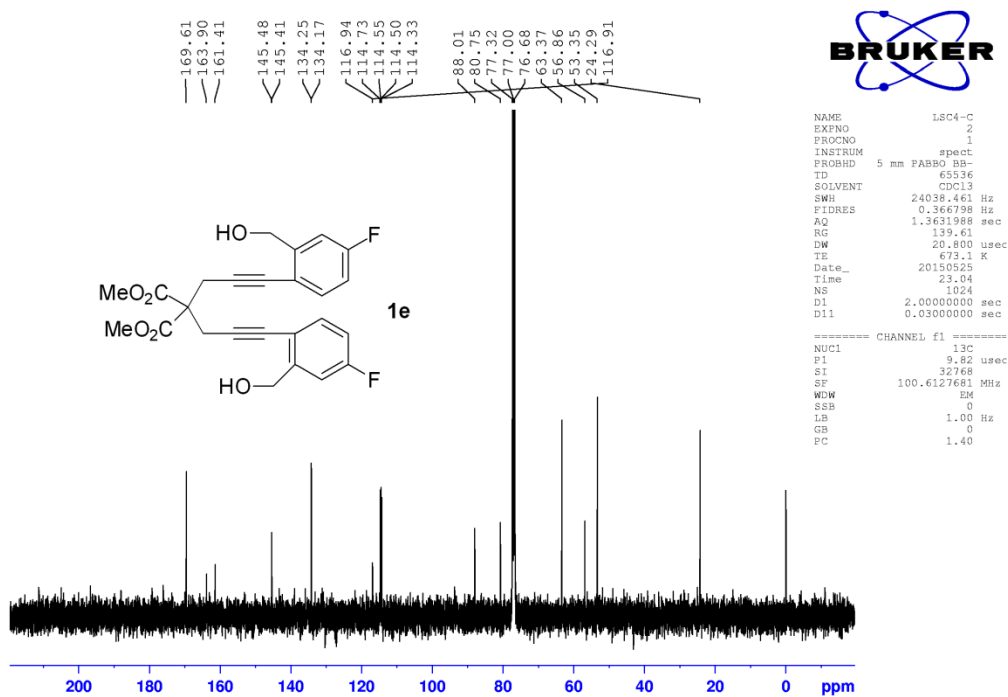
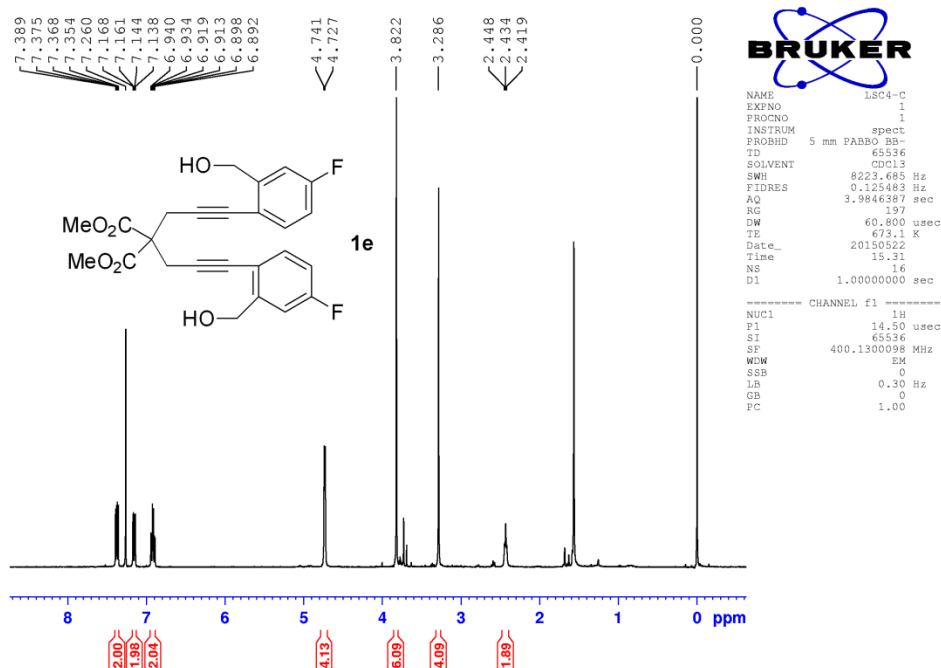
NAME                               LSE4
EXPNO                               2
PROCNO                              1
INSTRUM                             spect
PROBHD 5 mm PABBO BB-
TD                                   65536
SOLVENT                             CDCl3
SWH                                  240.3641 Hz
FIDRES                               0.366798 Hz
AQ                                   1.363198 sec
RG                                   197
DW                                   20.800 usec
TE                                   673.1 K
Date_                               20150602
Time                                 19.54
NS                                   1024
D1                                   2.00000000 sec
D11                                 0.03000000 sec

```

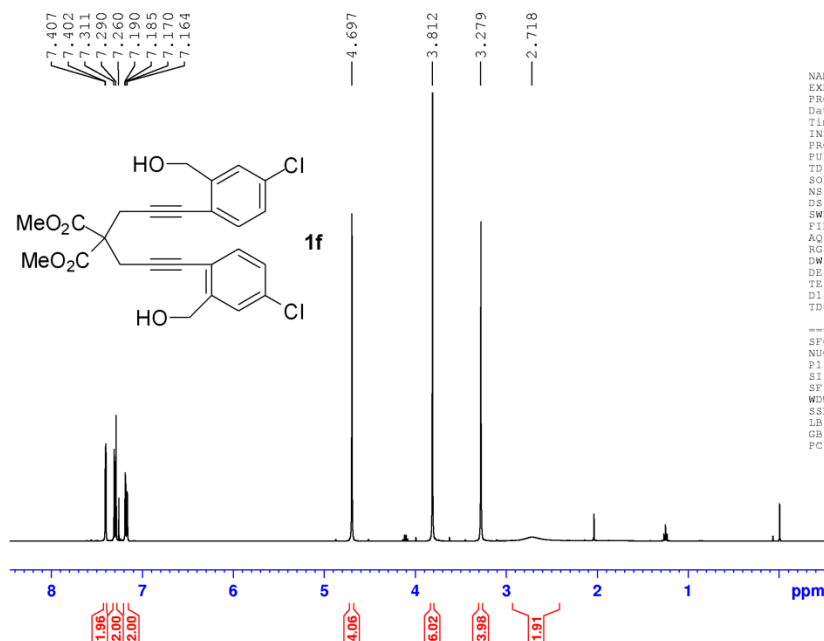
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===== CHANNEL f1 =====
NUC1              13C
F1                9.82 usec
SI               32768
SF             100.6127723 MHz
WDW              EM
SSB              0
LB              1.00 Hz
GB              0
PC              1.40

```



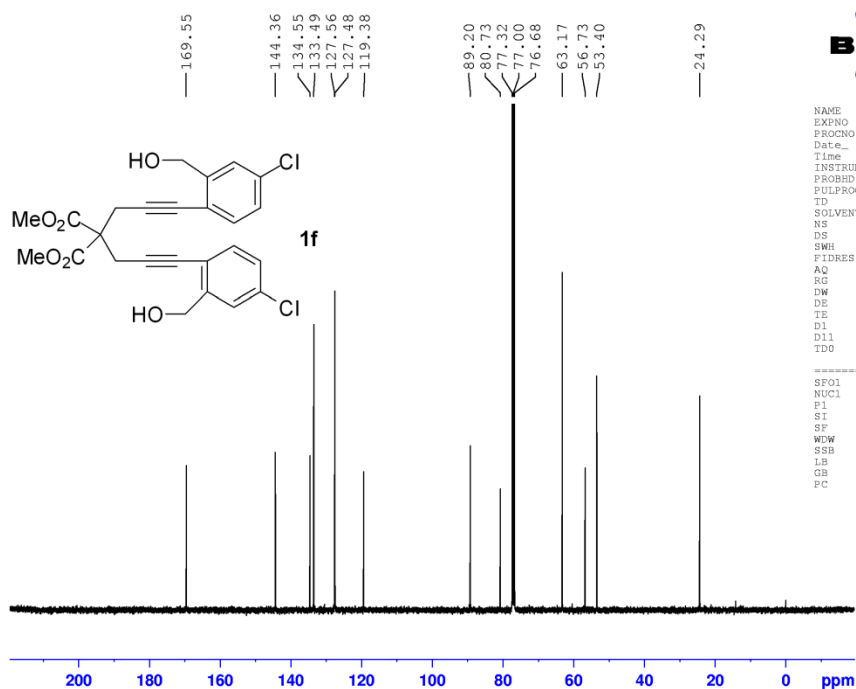
wyz-LSG43



```

NAME      wyz-LSG43
EXPNO     1
PROCNO    1
Date_     20150617
Time      23.06
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         97.67
DE         62.400 usec
TE         295.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SF01      400.1324710 MHz
NUC1       1H
P1         13.79 usec
SI         65536
SF         400.1300099 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

NAME      wyz-LSG43
EXPNO     2
PROCNO    1
Date_     20150617
Time      23.41
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         600
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         193.76
DE         20.800 usec
TE         296.4 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SF01      100.6228293 MHz
NUC1      13C
P1         10.11 usec
SI         32768
SF         100.6127732 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

¹H NMR spectrum (400 MHz, CDCl₃) of compound **1g**. The chemical structure of **1g** is shown above the spectrum. The spectrum displays peaks in the aromatic region (7.2-7.8 ppm), a singlet for the CF₃ groups (4.86 ppm), and aliphatic signals (1.9-2.7 ppm). Integration values are provided below the baseline.

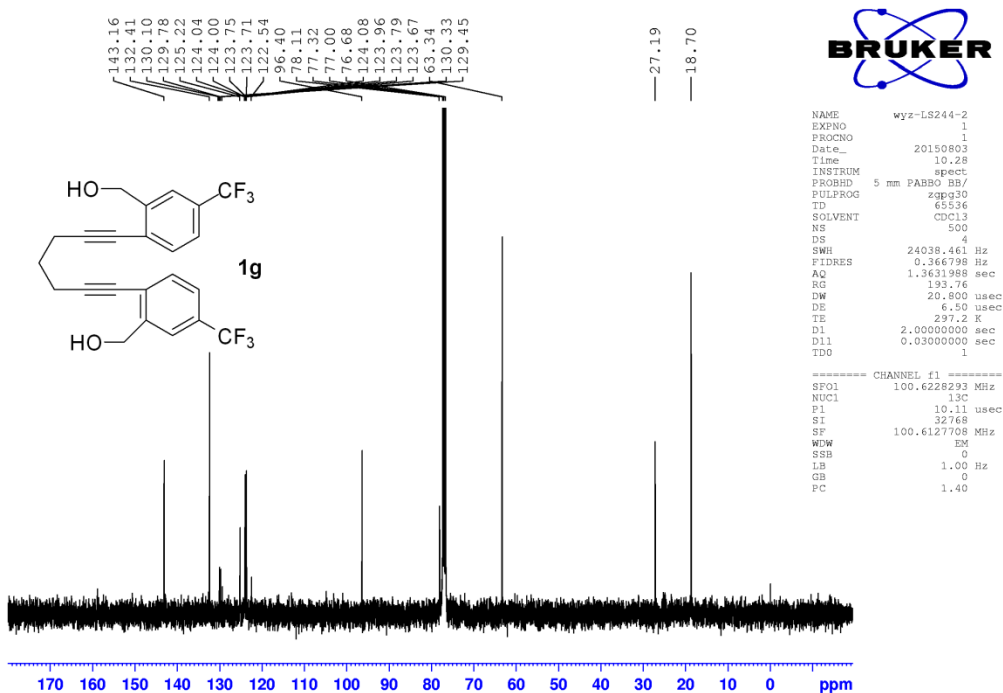
Chemical Shift (ppm)	Integration
7.720, 7.510, 7.490, 7.482, 7.479, 7.461, 7.458, 7.262	2.00, 1.91, 2.18
4.862	3.98
2.713, 2.696, 2.678, 2.334, 1.994, 1.977, 1.960, 1.943, 1.925	4.06, 1.87, 2.16

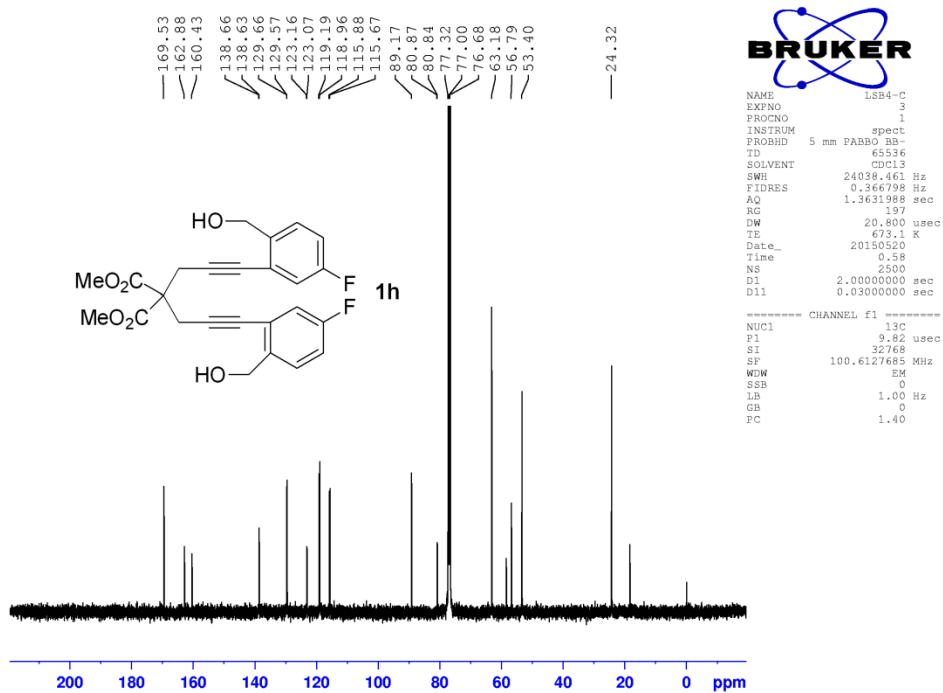
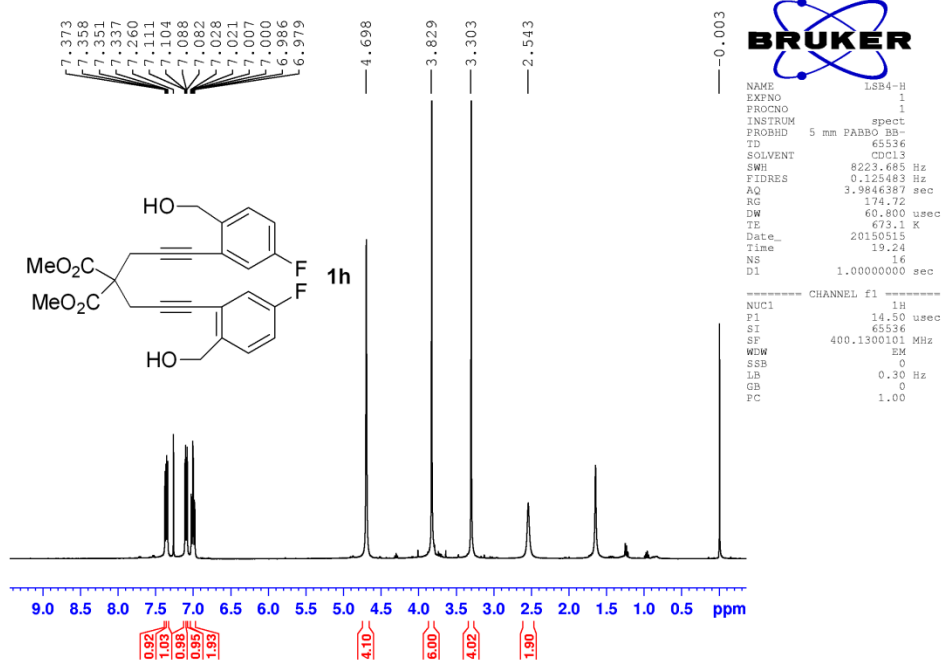
Additional data from the spectrum:

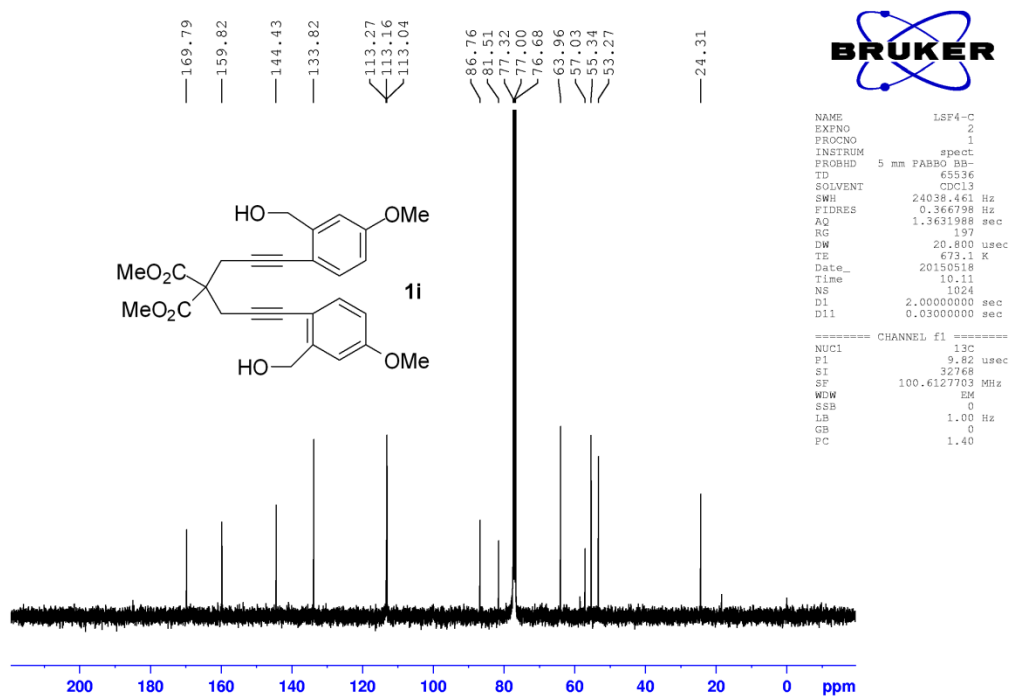
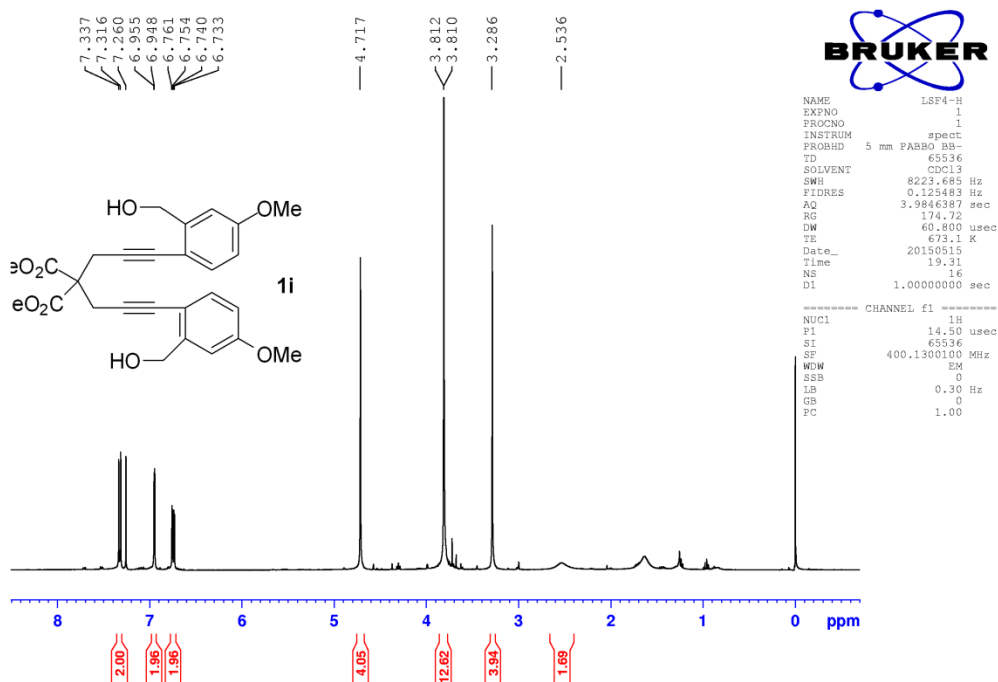
- NAME: EXPNO
- PROCNO: 1
- Date_:
- Time: 11:00
- INSTRUM: spect
- PROBHD: 5
- PULPROG: zgpg30
- TD: 65536
- SOLVENT: CHL
- NS: 2048
- DS: 4
- SWH: 100.620 MHz
- FIDRES: 0.188 Hz
- AQ: 0.48000000
- RG: 327.680
- DW: 12.000 ns
- DE: 2.000 dB
- TE: 300.2 K
- D1: 1.500 s
- D11: 0.050 s
- TD0: 1

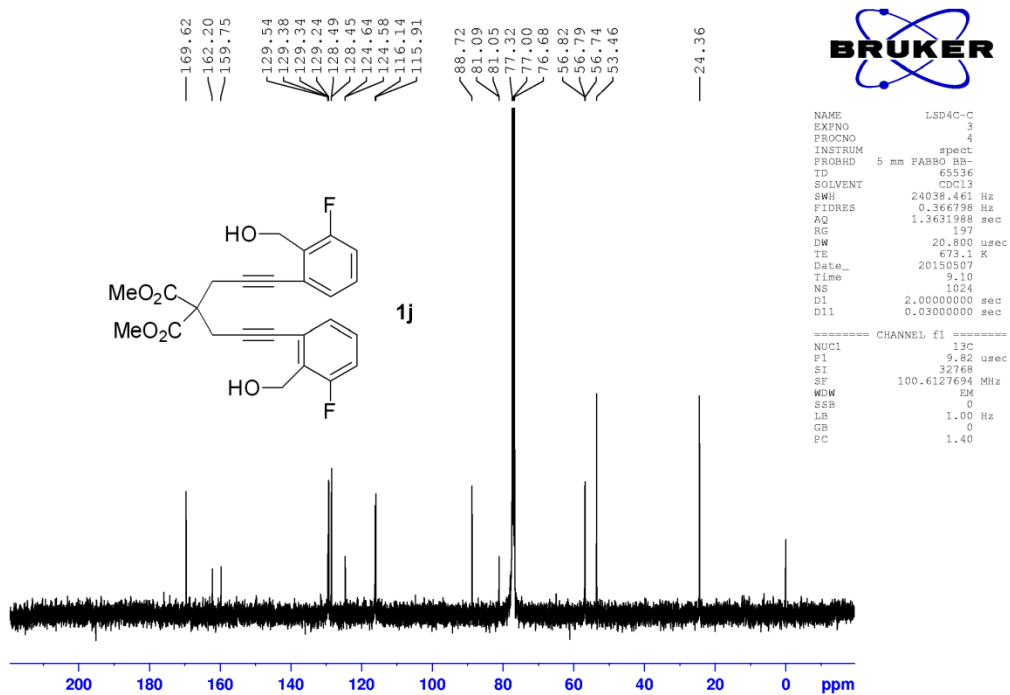
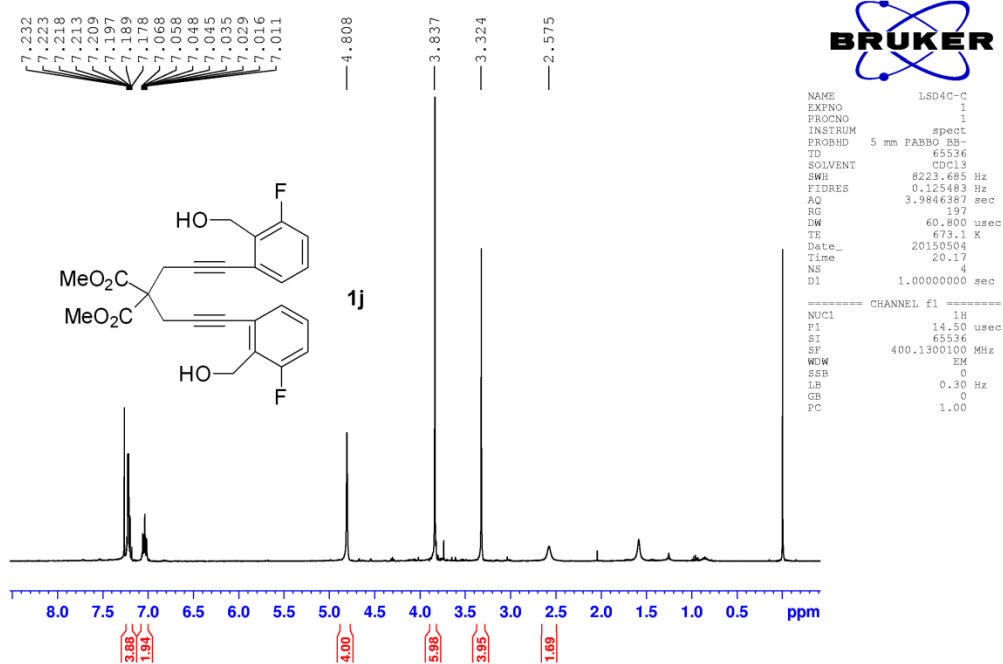
Processing parameters:

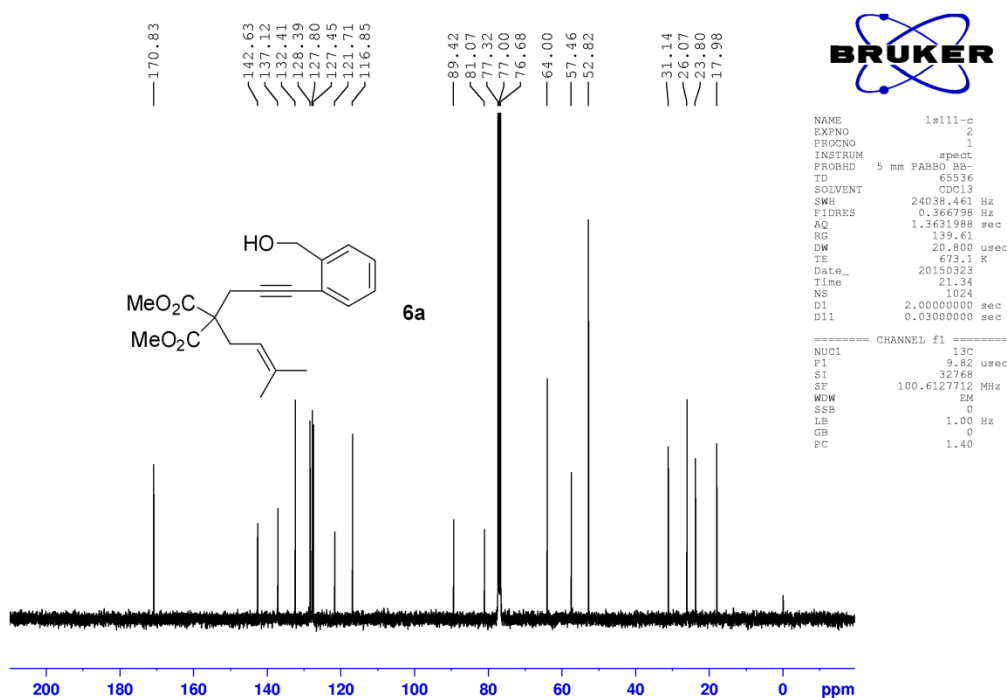
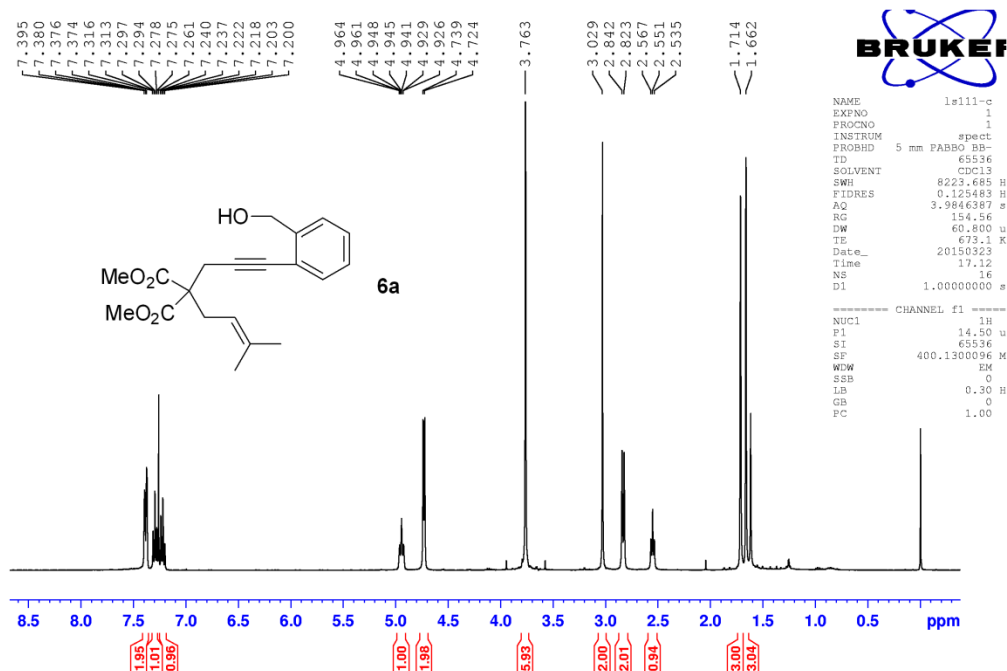
- SI: 32768
- SF: 400.146 MHz
- WDW: EM
- SSB: 0
- LB: 3.00 Hz
- GB: 0
- PC: 1.00

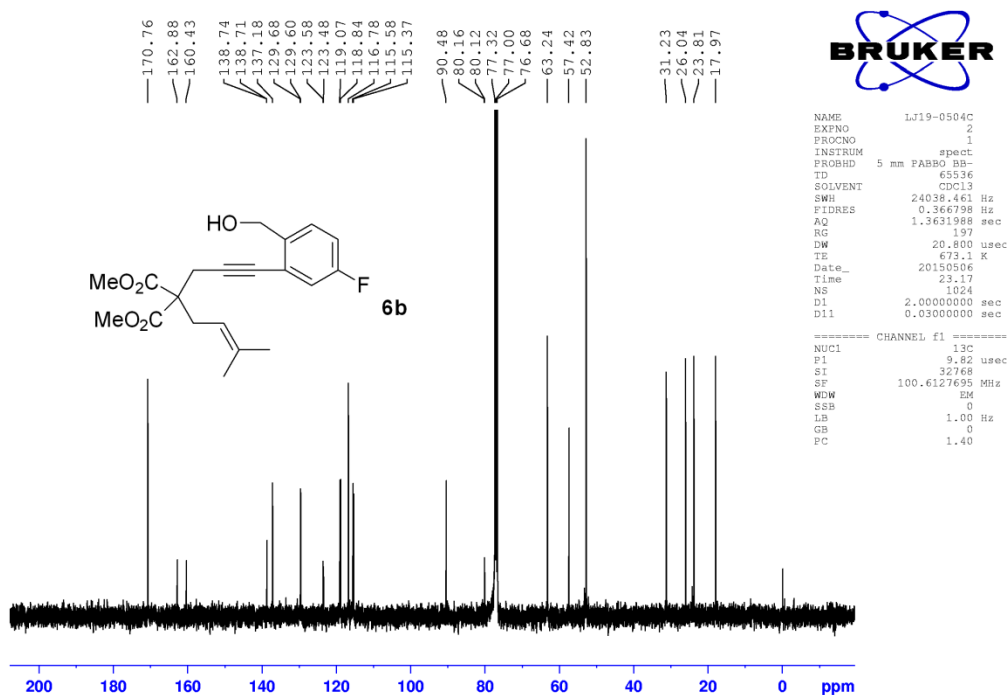
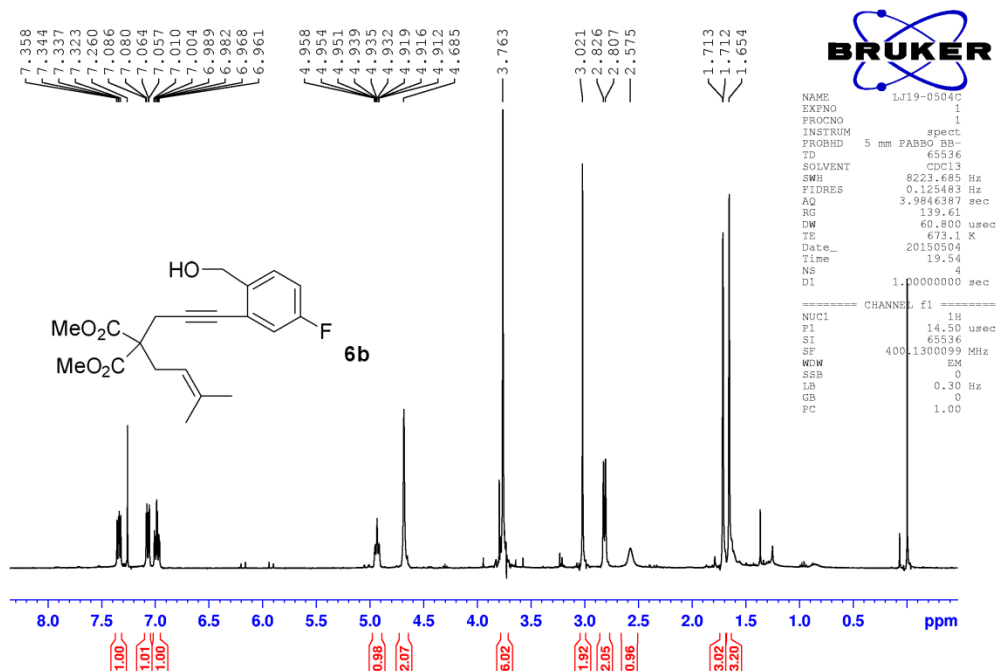




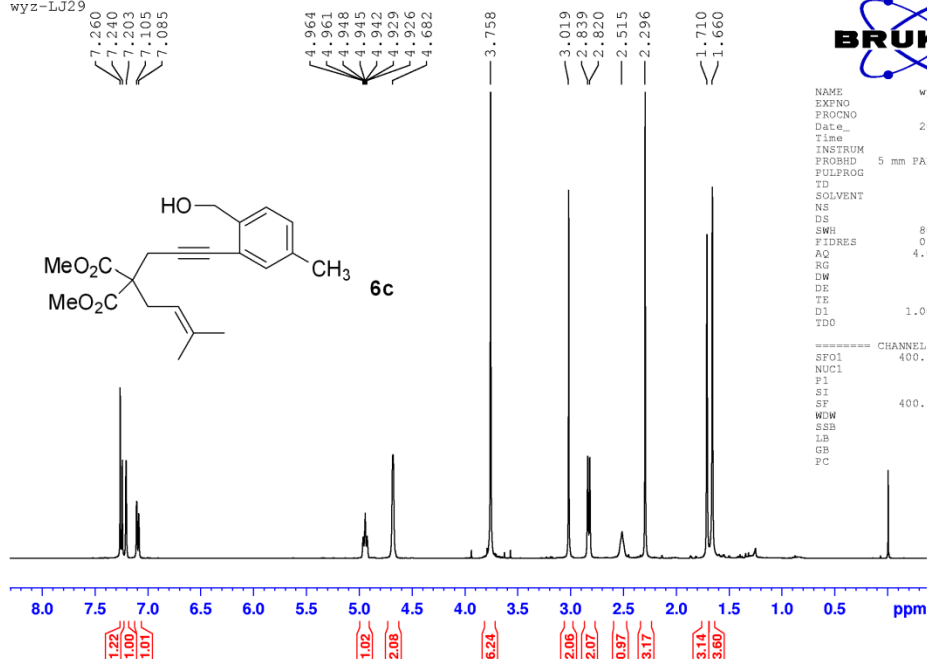






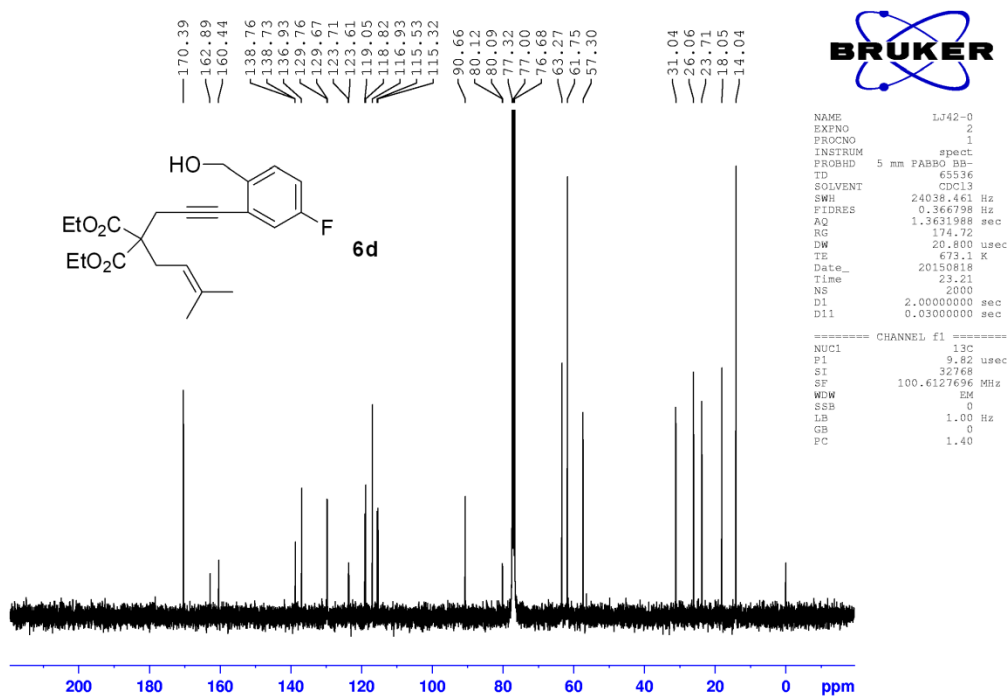
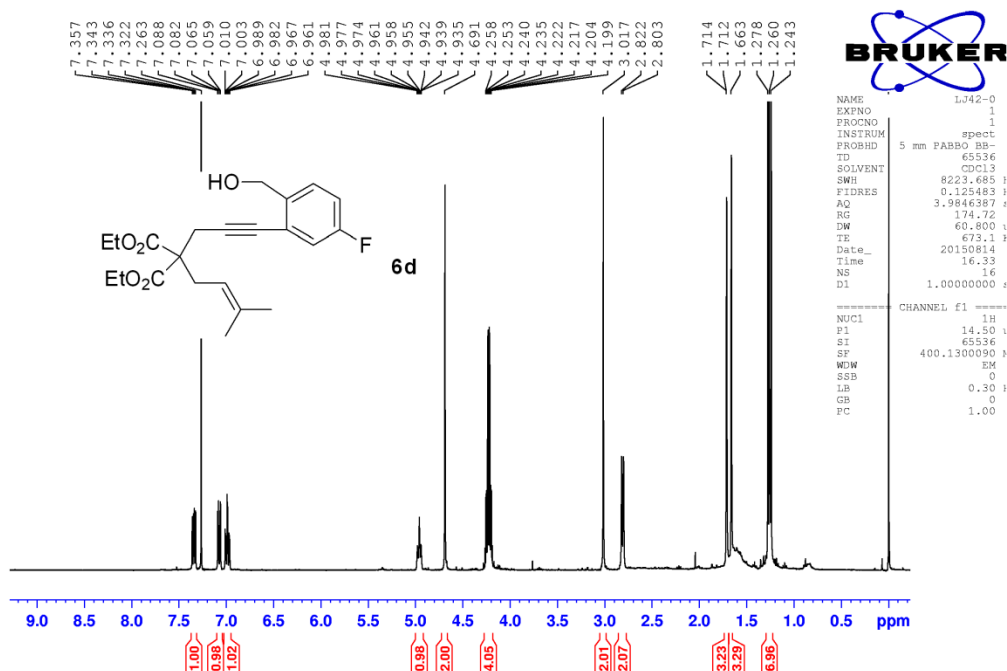


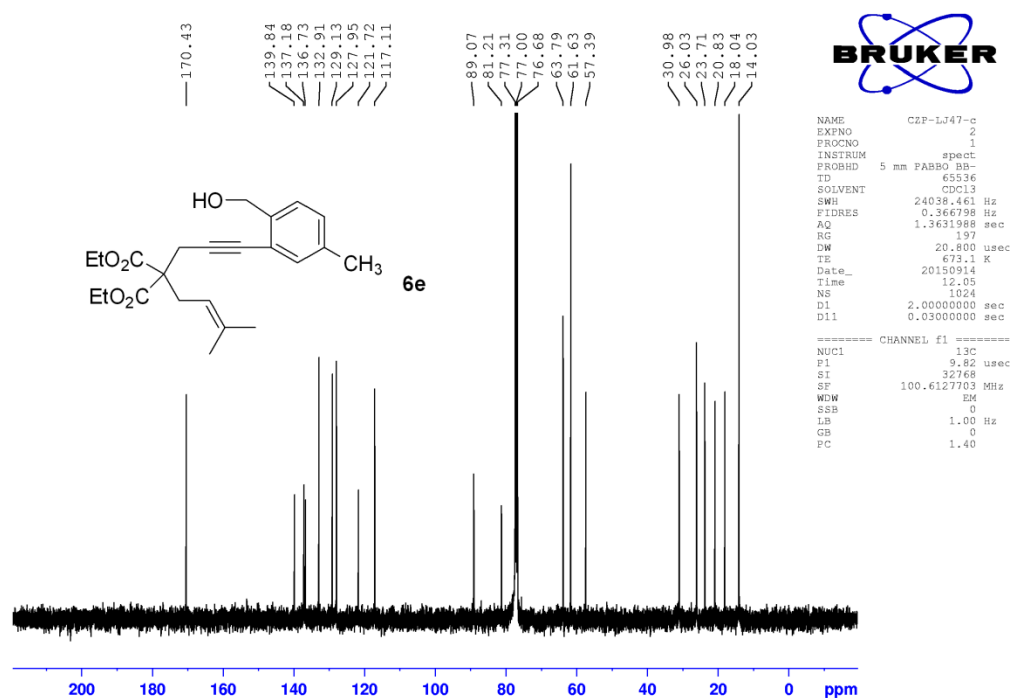
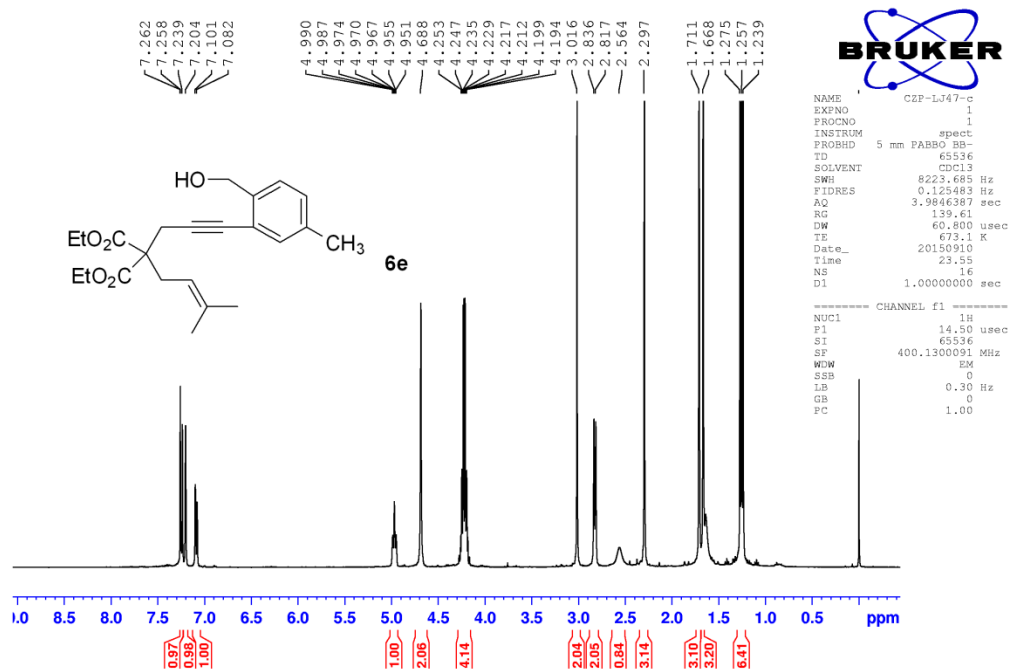
wyz-LJ29

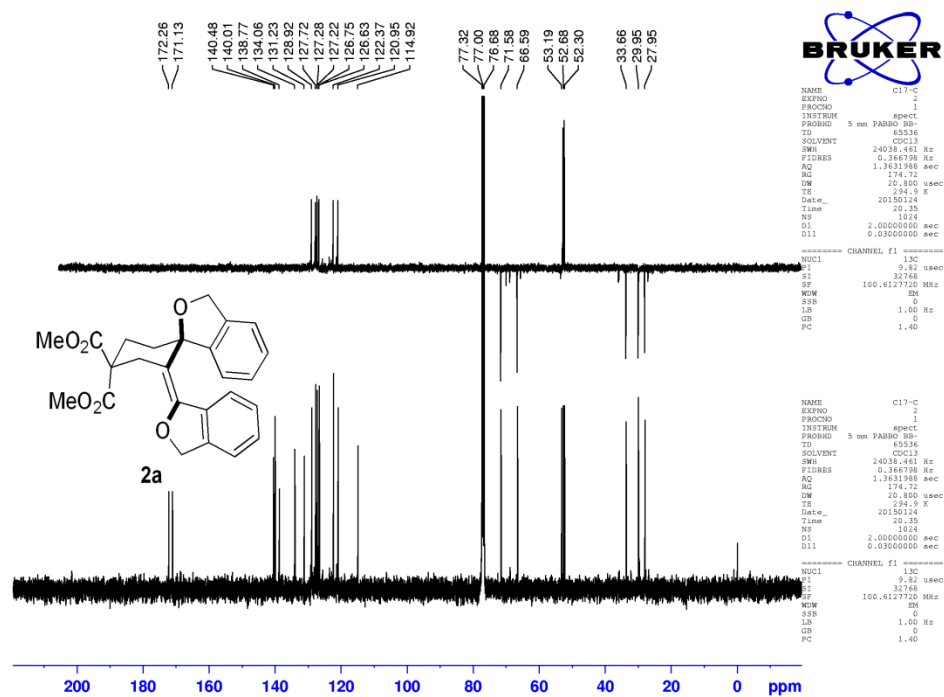
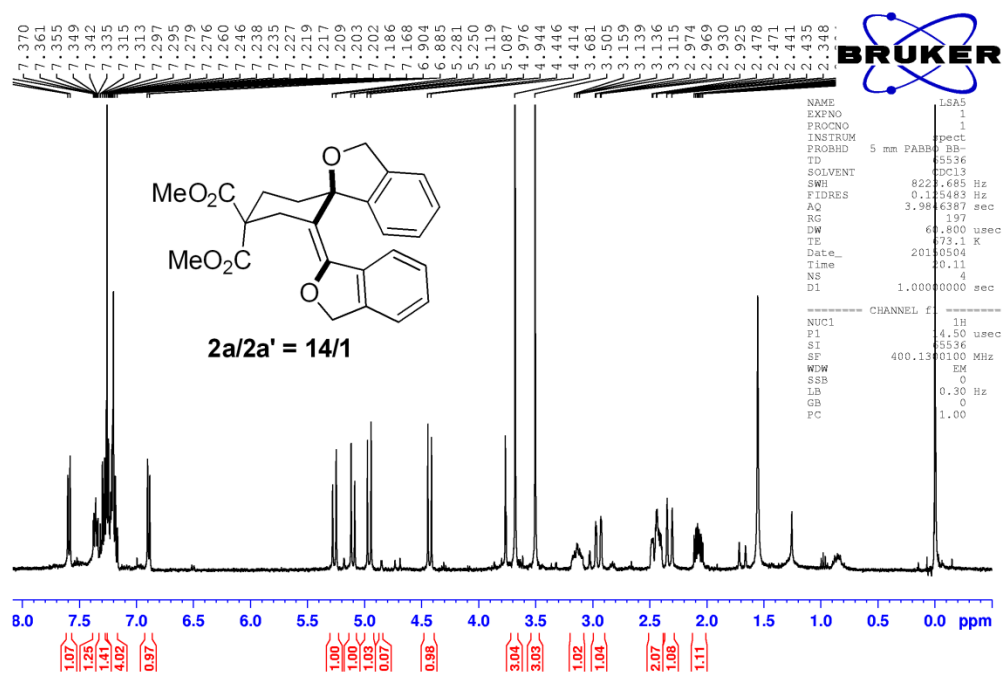


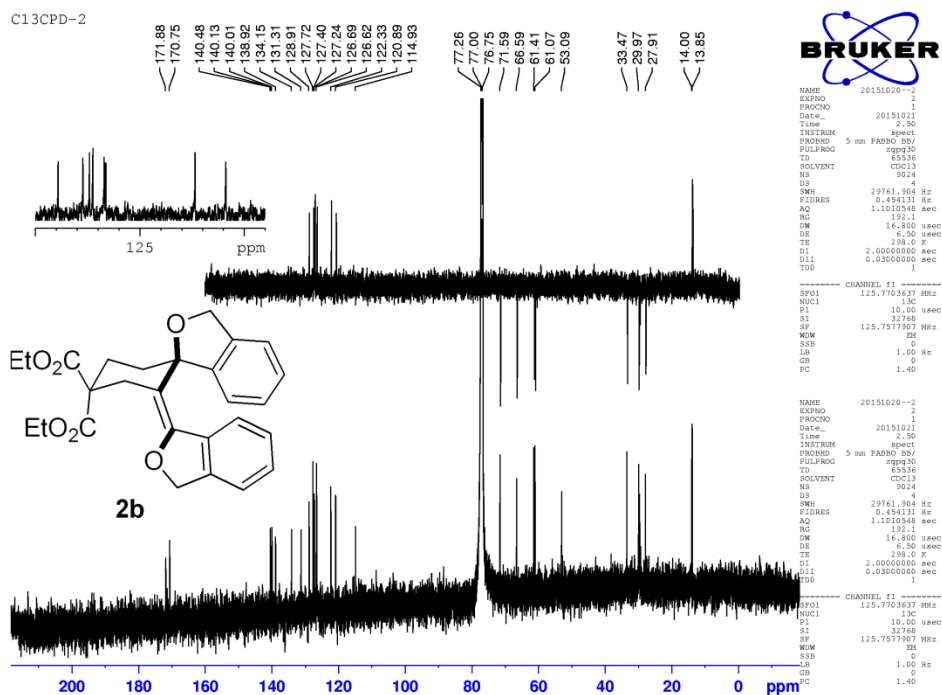
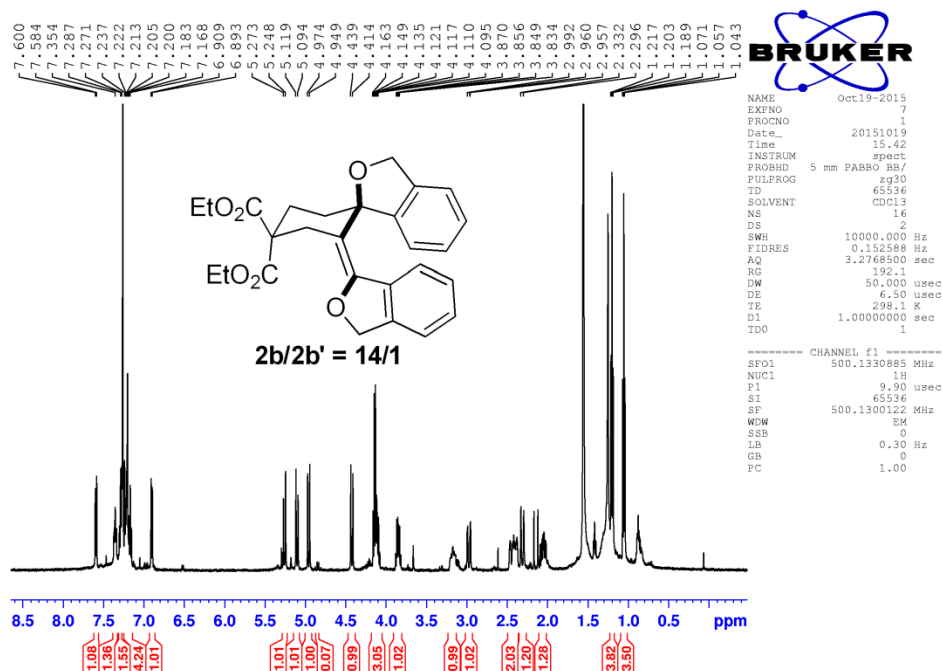
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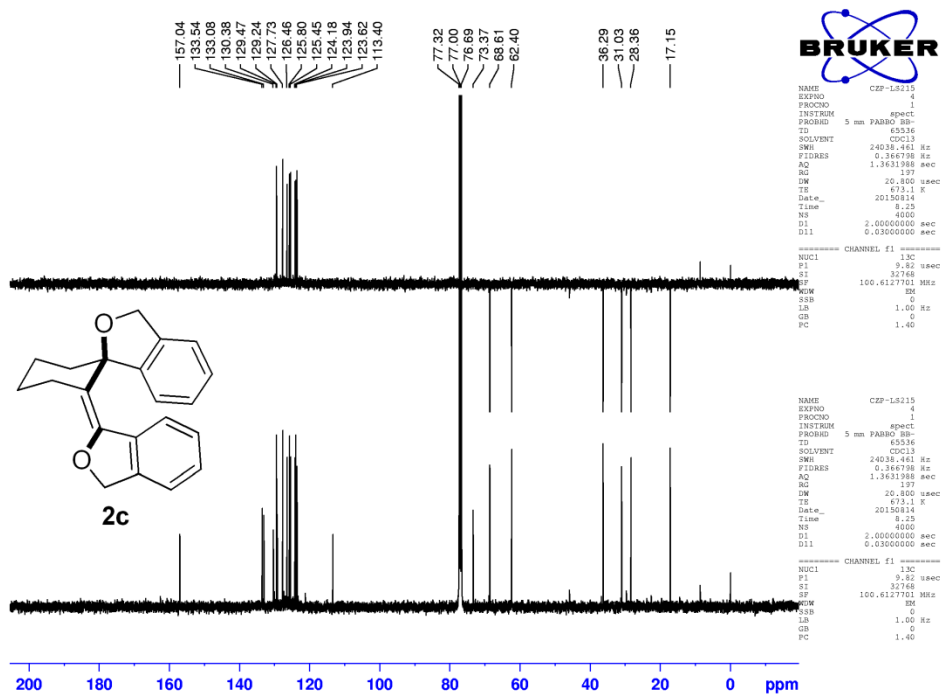
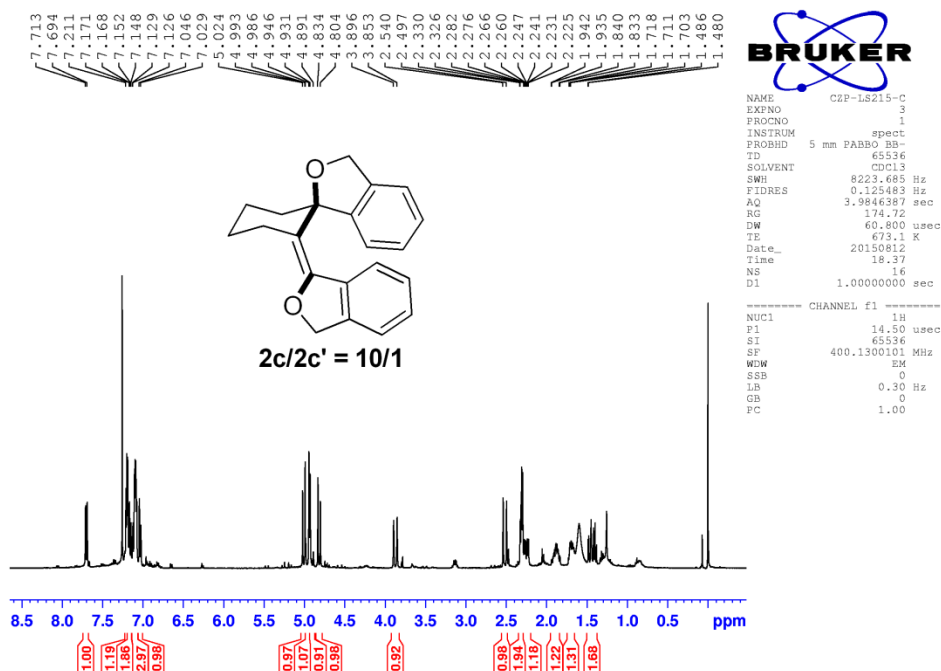
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EXPNO     1
PROCNO    1
Date_     20150618
Time      1.20
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         97.67
DW         62.400 usec
DE         6.50 usec
TE         295.3 K
D1         1.00000000 sec
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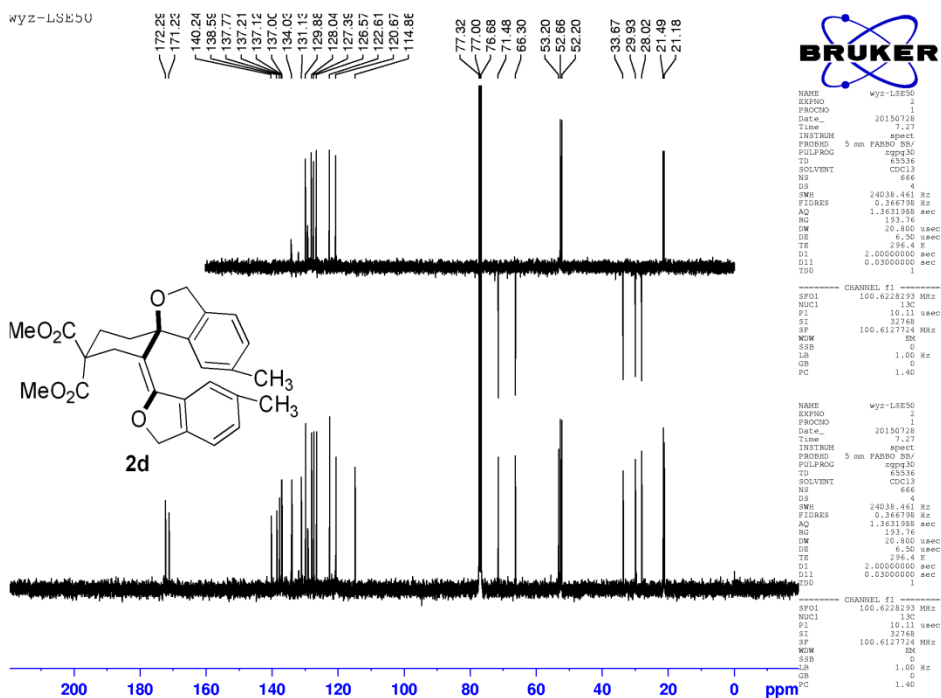
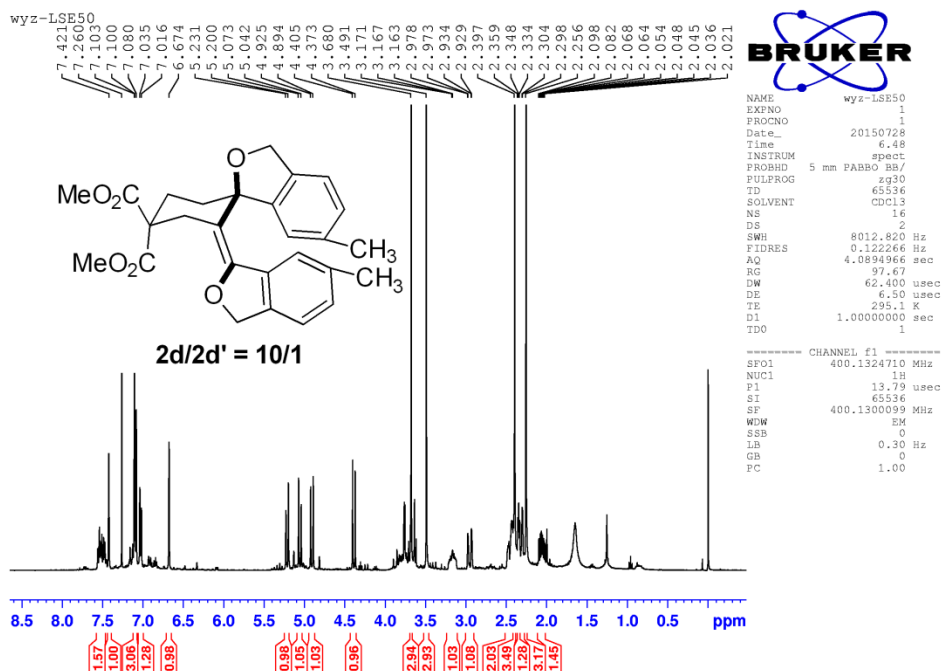


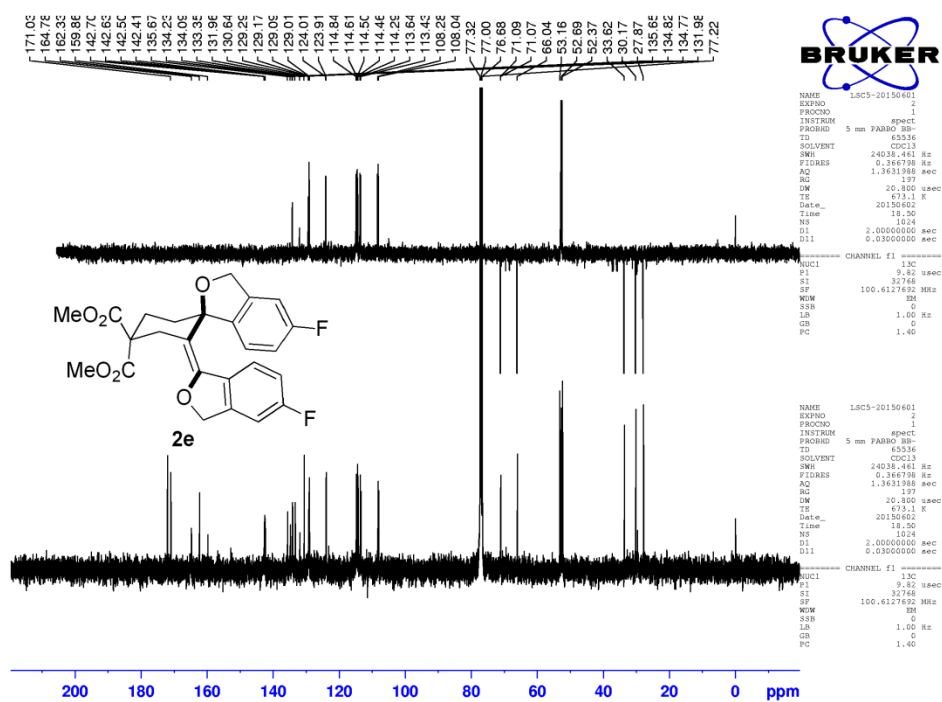
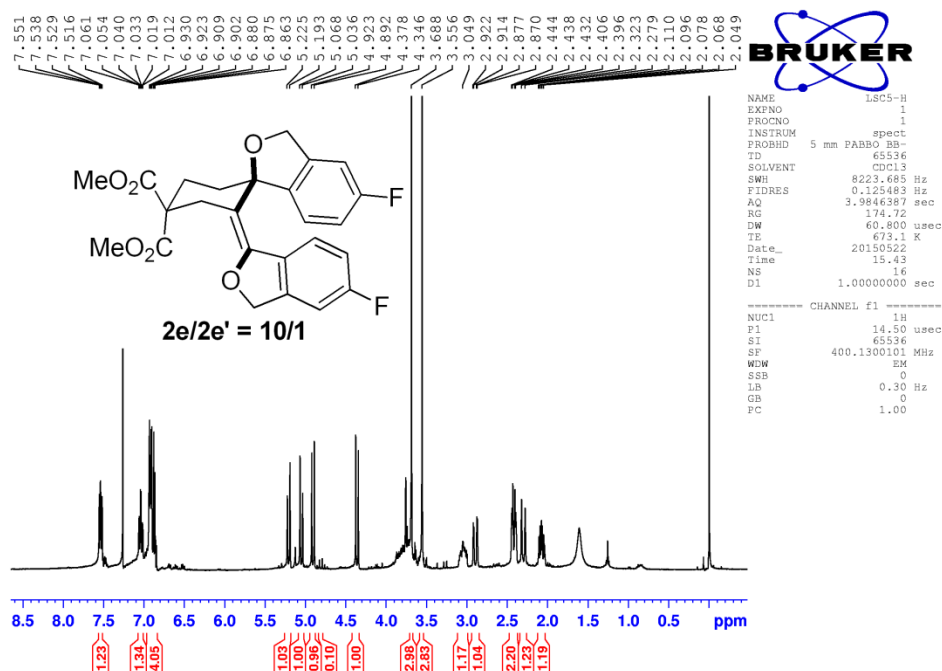


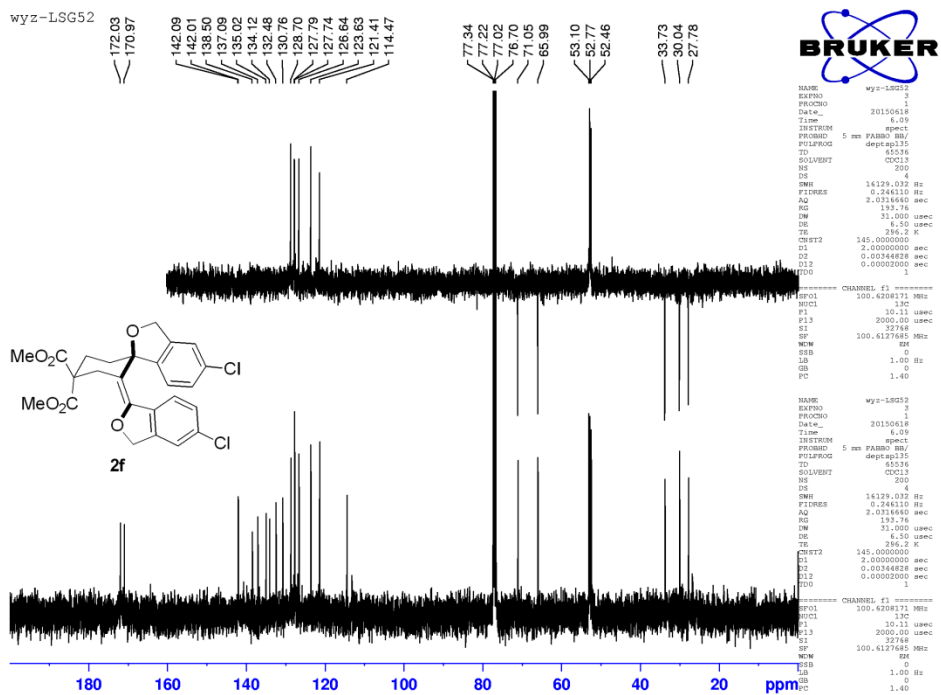
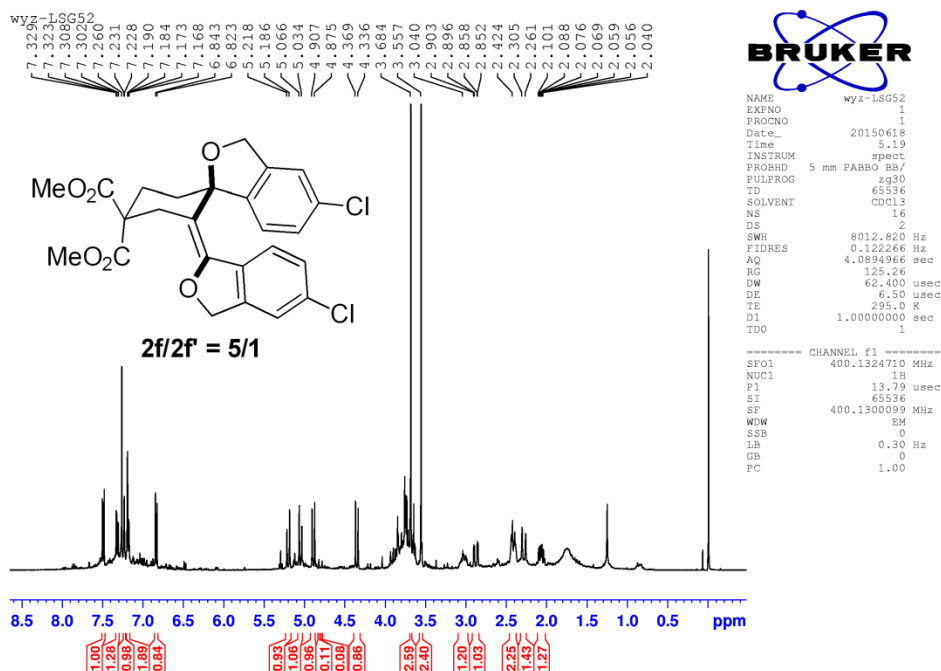


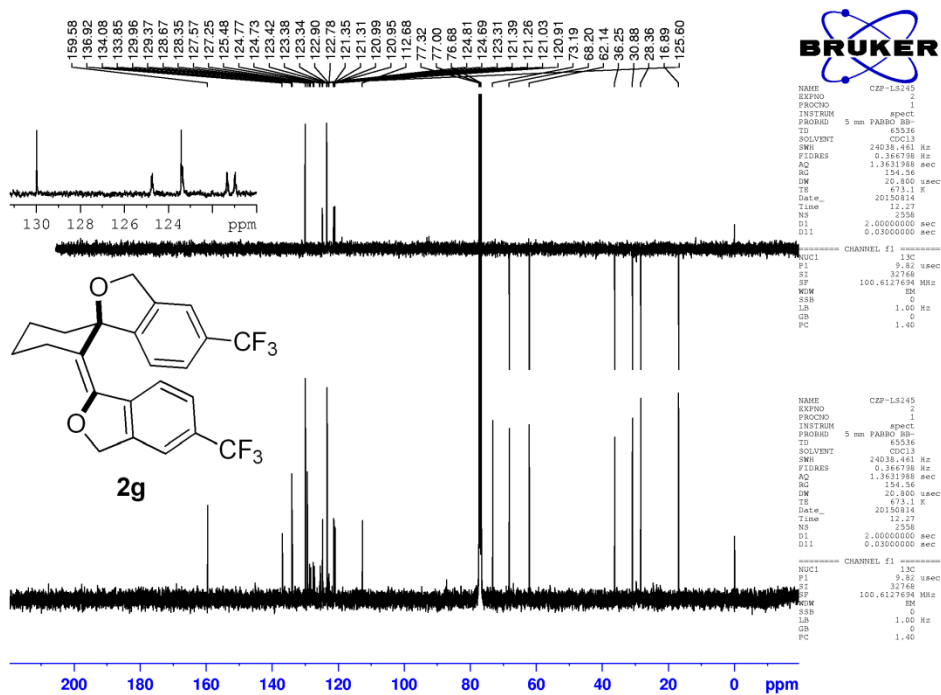
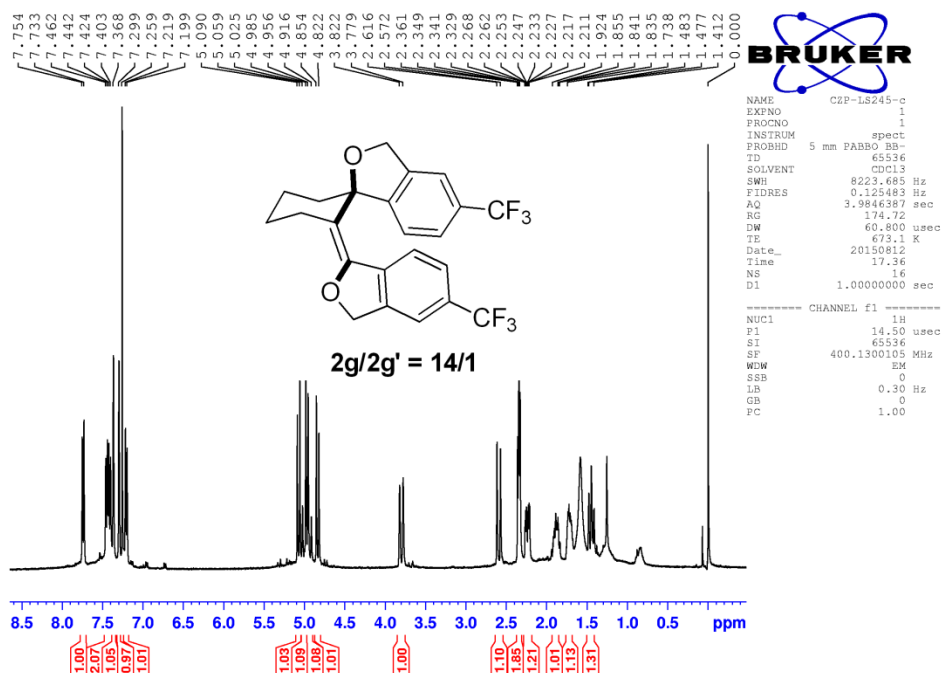


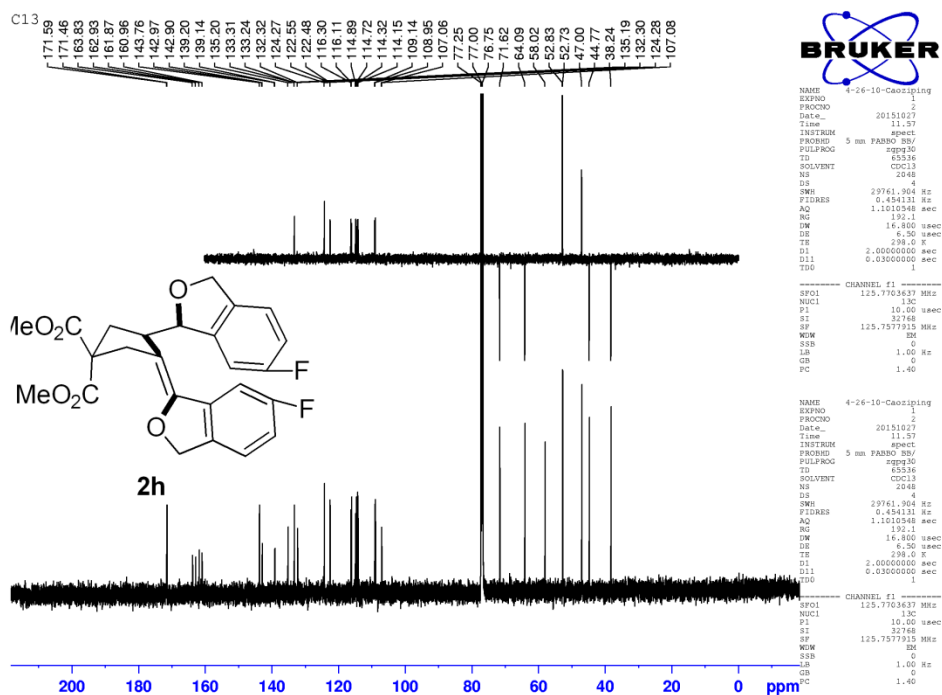
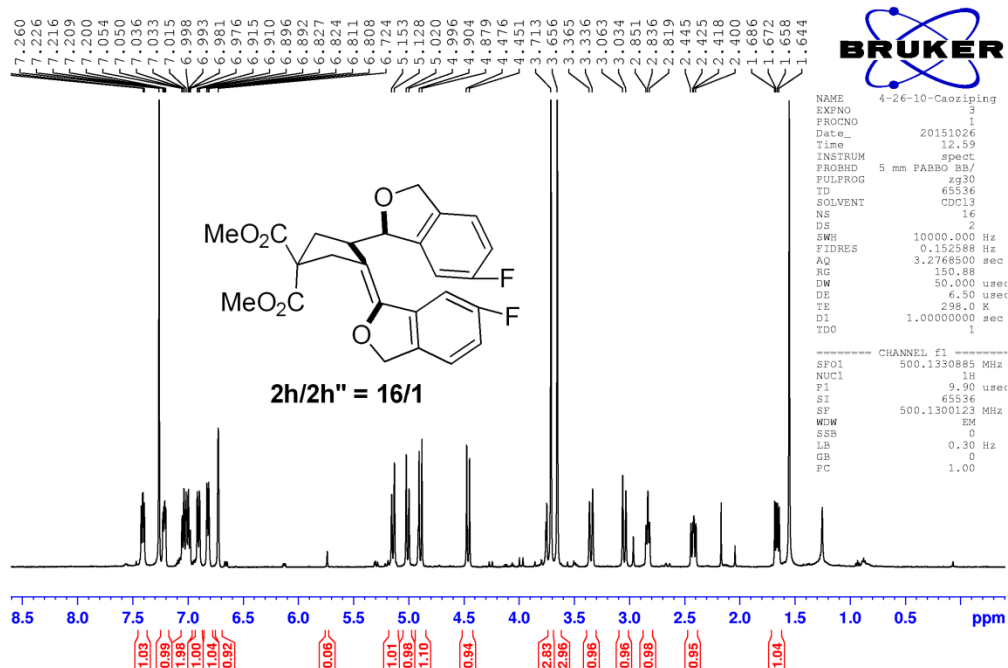


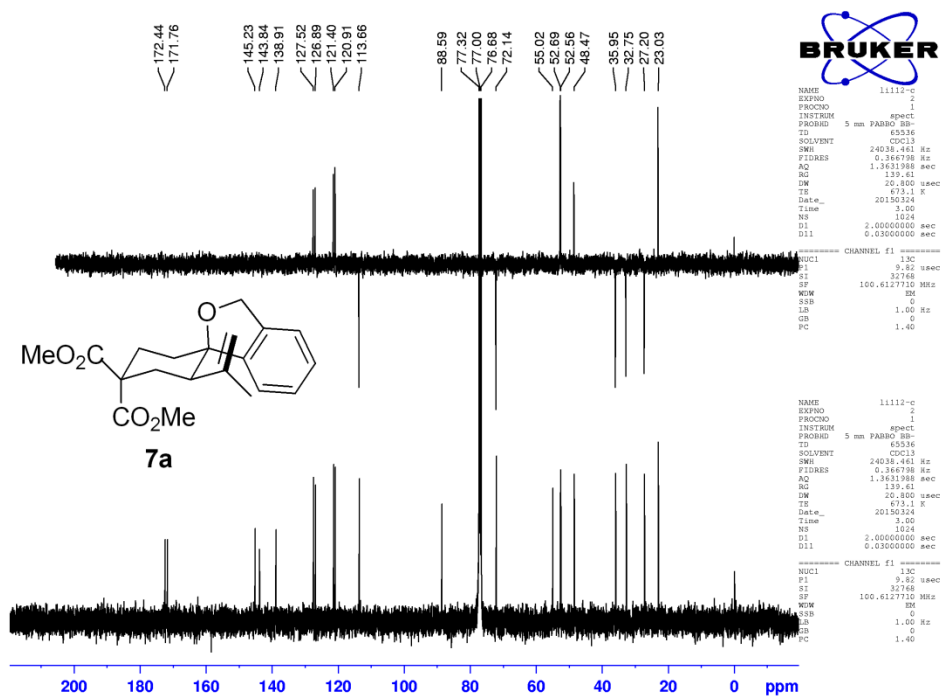
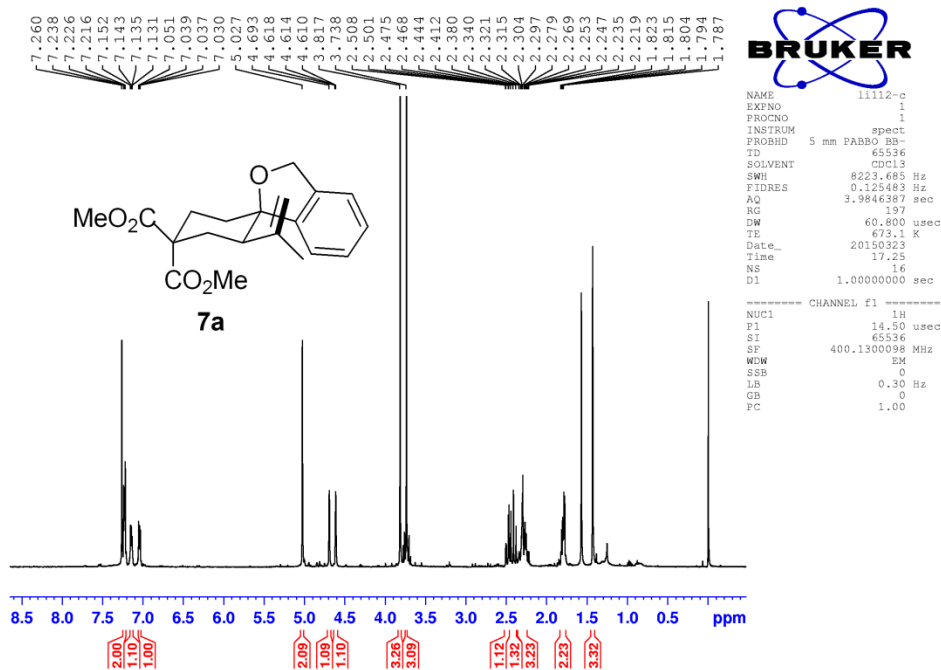


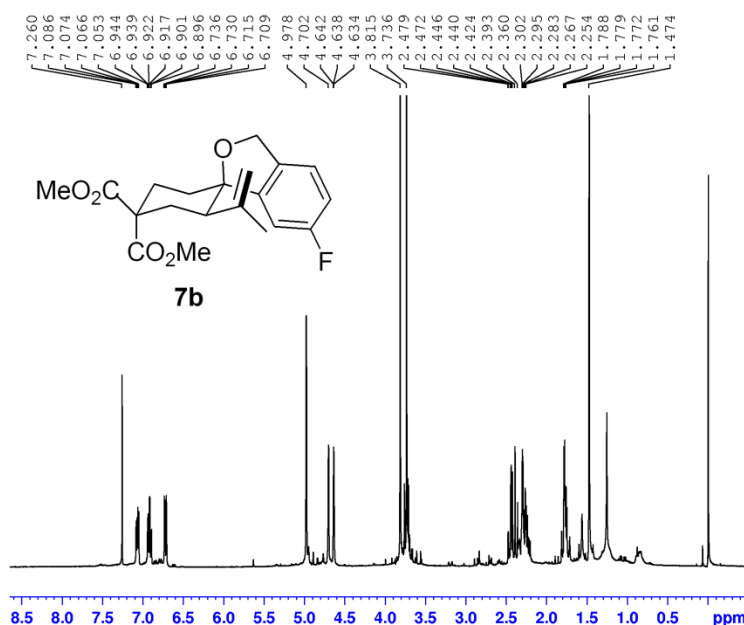










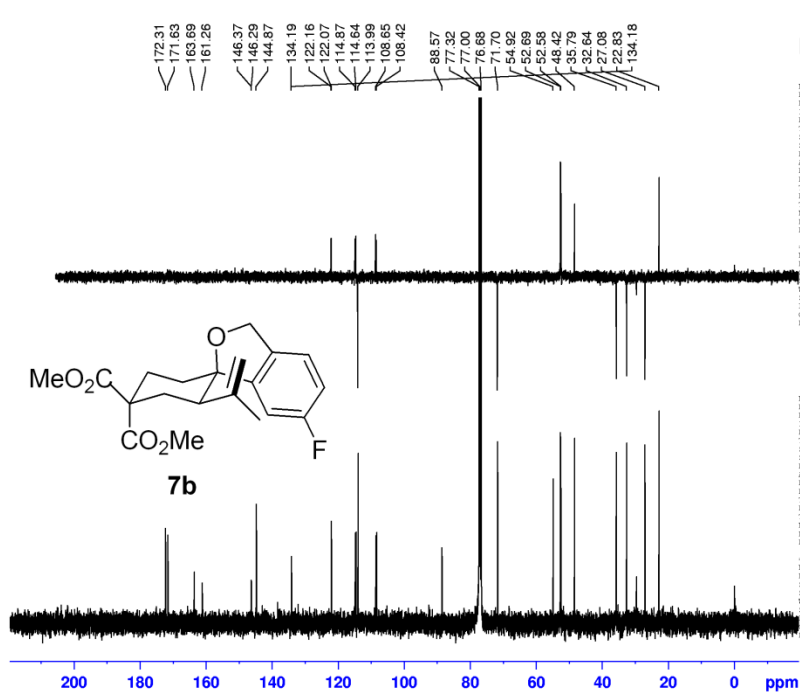


BRUKER

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NAME      1j20-0505C
EXPNO     1
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INSTRUM    spect
PROBHD     5 mm PABBO BB-
TD         65536
SOLVENT    CDCl3
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         154.56
DW         60.800 usec
TE         293.1 K
Date_      20150505
Time       18.55
NS         16
D1         1.00000000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         14.50 usec
SI         65536
SF         400.1300099 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
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BRUKER

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NAME      1j20-0505C
EXPNO     2
PROCNO    1
INSTRUM    spect
PROBHD     5 mm PABBO BB-
TD         65536
SOLVENT    CDCl3
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         197
DW         20.800 usec
TE         293.1 K
Date_      20150507
Time       4.39
NS         1024
D1         2.00000000 sec
D11        0.03000000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         9.82 usec
SI         32768
SF         100.6127694 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

===== CHANNEL f2 =====
NUC1       13C
P1         9.82 usec
SI         32768
SF         100.6127694 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
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