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Supporting Information

for

Three-component reactions of conjugated dienes, CH acids and formaldehyde under diffusion mixing conditions

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^{1}H and ^{13}C NMR spectra of synthesized compounds

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1. Synthesis

2,4-dibenzoyl-1,5-diphenylpentane-1,5-dione (**3**) [1]. From 1,3-diphenylpropane-1,3-dione (112 mg, 0.50 mmol) and L-proline (3 mg, 0.025 mmol) compound **3** (227 mg, 99%) was obtained as a white crystalline solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20-8.12 (m, 8H), 7.63-7.57 (m, 4H), 7.54-7.45 (m, 8H), 5.76 (t, *J* = 7.0 Hz, 2H), 2.77 (t, *J* = 7.0 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 196.7 (4C), 135.6 (4C), 134.0 (4C), 129.2 (8C), 128.9 (8C), 54.1 (2C), 29.1.

HRMS (ESI+) m/z calcd. for ($C_{31}H_{25}O_4$, M+H): 461.1747, found: (M+H): 461.1753.

Ethyl (1S,2S*,4S*)-2-cyanobicyclo[2.2.1]hept-5-ene-2-carboxylate* (**4a**) and *ethyl (1S*,2R*,4S*)-2-cyanobicyclo[2.2.1]hept-5-ene-2-carboxylate* (**4b**) [2]. From cyanoacetic acid ester (113 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and cyclopentadiene (330 mg, 5.0 mmol) the mixture of compounds **4a** and **4b** in 78/22 ratio (189 mg, 99%) was obtained as a light orange oil.

Major isomer 4a: ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.31 (dd, *J* = 5.5, 2.9 Hz, 1H), 5.86 (dd, *J* = 5.5, 2.9 Hz, 1H), 4.19 (qd, *J* = 7.2, 3.2 Hz, 2H), 3.56-3.51 (m, 1H), 3.10-3.06 (m, 1H), 2.23 (dd, *J* = 12.5, 3.7 Hz, 1H), 2.05 (dd, *J* = 12.5, 2.9 Hz, 1H), 1.79-1.73 (m, 1H), 1.67-1.59 (m, 1H), 1.29 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 167.4, 140.3, 131.0, 121.7, 62.7, 53.6, 49.2 (2C), 42.9, 37.2, 14.1.

Minor isomer 4b: ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.43 (dd, *J* = 5.8, 3.0 Hz, 1H), 6.33-6.27 (m, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 3.38–3.35 (m, 1H), 3.10-3.06 (m, 1H), 2.54 (dd, *J* = 12.4, 3.4 Hz, 1H), 1.69 (dd, *J* = 12.4, 2.7 Hz, 1H), 1.67–1.59 (m, 1H), 1.54-1.49 (m, 1H), 1.33 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 167.4, 141.0, 134.1, 121.1, 63.0, 52.4, 46.8, 46.5, 42.7, 39.1, 14.1.

HRMS (ESI+) m/z calcd. for ($C_{11}H_{14}NO_2$, M+H): 192.1019, found: (M+H): 192.1020.

Ethyl (1S,2S*,4S*)-2-acetylbicyclo[2.2.1]hept-5-ene-2-carboxylate* (**5a**), *ethyl (1S*,2R*,4S*)-2-acetylbicyclo[2.2.1]hept-5-ene-2-carboxylate* (**5b**) and *ethyl (4aR*,7aS*)-2-methyl-4,4a,5,7a-tetrahydrocyclopenta[b]pyran-3-carboxylate* (**6**). From acetoacetic ester (130 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and cyclopentadiene (330 mg, 5.0 mmol) the mixture of compounds **5a**, **5b** and **6** in 27/67/6 ratio (196 mg, 94%) was obtained as a colorless oil.

Minor isomer 5a: ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.23 (dd, *J* = 5.6, 3.0 Hz, 1H), 5.96 (dd, *J* = 5.6, 2.9 Hz, 1H), 4.20-4.05 (m, 2H), 3.33 (s, 1H), 2.85 (s, 1H), 2.15 (s, 3H), 2.05-1.97 (m, 2H), 1.45-1.41 (m, 1H), 1.38-1.33 (m, 1H), 1.24-1.17 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 204.4, 171.7, 140.0, 133.9, 67.9, 61.3, 48.4, 48.3, 42.2, 34.4, 27.2, 14.1.

Major isomer 5b: ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.17 (dd, *J* = 5.6, 3.1 Hz, 1H), 5.88 (dd, *J* = 5.6, 2.8 Hz, 1H), 4.20-4.05 (m, 2H), 3.41 (s, 1H), 2.85 (s, 1H), 2.08 (s, 3H), 2.02-1.97 (m, 1H), 1.95-1.92 (m,

1H), 1.62-1.58 (m, 1H), 1.51-1.46 (m, 1H), 1.24-1.17 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 202.5, 172.9, 140.1, 132.3, 67.6, 61.6, 49.5, 49.1, 42.2, 33.9, 27.5, 14.1.

Minor isomer 6: ^{1}H NMR (400 MHz, $CDCl_3$) *(due to the low content of compound **6** in the mixture and the overlap of most signals with the signals of the main isomer, only characteristic peaks are indicated):* δ 5.96-5.93 (m, 1H), 5.82-5.77 (m, 1H), 4.93 (d, *J* = 6.9 Hz, 1H).

HRMS (ESI+) m/z calcd. for ($C_{12}H_{17}O_3$, M+H): 209.1172, found: (M+H): 209.1175.

1,1'-(Bicyclo[2.2.1]hept-5-ene-2,2-diyl)bis(ethan-1-one) (**7**). From acetylacetone (100 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and cyclopentadiene (330 mg, 5.0 mmol) compound **7** (63 mg, 38%) was obtained as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.17 (dd, *J* = 5.7, 3.1 Hz, 1H), 5.92 (dd, *J* = 5.7, 2.9 Hz, 1H), 3.52-3.46 (m, 1H), 2.86-2.79 (m, 1H), 2.09-2.03 (m, 1H), 2.07 (s, 3H), 2.01 (s, 3H), 1.87 (dd, *J* = 12.2, 3.8 Hz, 1H), 1.48-1.42 (m, 1H), 1.27 (d, *J* = 8.8 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 206.0, 204.5, 140.3, 132.8, 77.4, 48.8, 47.7, 42.3, 33.1, 27.8, 27.6.

HRMS (ESI+) m/z calcd. for ($C_{11}H_{15}O_2$, M+H): 179.1067, found: (M+H): 179.1065.

((1S,4S*)-Bicyclo[2.2.1]hept-5-ene-2,2-diyl)bis(phenylmethanone)* (**8**) and *phenyl((4aR*,7aS*)-2-phenyl-4,4a,5,7a-tetrahydrocyclopenta[b]pyran-3-yl)methanone* (**9**) [3].From 1,3-diphenylpropane-1,3-dione (224 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and cyclopentadiene (330 mg, 5.0 mmol) compound **8** (136 mg, 45%) and compound **9** (97 mg, 32%) were obtained as a white crystalline solids.

Major isomer 8: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98-7.89 (m, 4H), 7.45-7.38 (m, 2H), 7.36-7.28 (m, 4H), 6.30 (dd, *J* = 5.7, 3.0 Hz, 1H), 5.74 (dd, *J* = 5.7, 2.9 Hz, 1H), 3.94-3.90 (m, 1H), 3.00-2.95 (m, 1H), 2.84 (dd, *J* = 12.1, 2.9 Hz, 1H), 2.18 (dd, *J* = 12.1, 3.7 Hz, 1H), 1.76-1.71 (m, 1H), 1.63-1.54 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 200.0, 197.0, 140.1, 137.5, 136.6, 133.1, 133.0, 132.7, 129.9 (2C), 129.2 (2C), 128.6 (2C), 128.5 (2C), 71.9, 51.6, 49.3, 43.0, 36.9.

HRMS (ESI+) m/z calcd. for ($C_{21}H_{19}O_2$, M+H): 303.1380, found: (M+H): 303.1382.

Minor isomer 9: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.49 (m, 2H), 7.22-7.15 (m, 3H), 7.11-6.99 (m, 5H), 6.22-6.16 (m, 1H), 6.09-6.04 (m, 1H), 5.44-5.39 (m, 1H), 3.18-3.08 (m, 1H), 2.75 (dd, *J* = 14.3, 6.1 Hz, 1H), 2.71-2.62 (m, 1H), 2.58 (dd, *J* = 14.3, 4.8 Hz, 1H), 2.34-2.25 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.4, 165.1, 139.1, 137.5, 135.6, 131.4, 130.9, 129.7 (2C), 129.6 (2C), 129.5 (2C), 127.7 (3C), 115.0, 85.6, 39.3, 37.8, 27.4.

HRMS (ESI+) m/z calcd. for ($C_{21}H_{19}O_2$, M+H): 303.1380, found: (M+H): 303.1383.

(1S,4S*)-2',2'-dimethylspiro[bicyclo[2.2.2]octane-2,5'-[1,3]dioxan]-5-ene-4',6'-dione* (**10**) [4]. From Meldrum's acid (144 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and 1,3-cyclohexadiene (160 mg, 2.0 mmol) compound **10** (179 mg, 76%) was obtained as a white crystalline solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.45-6.39 (m, 1H), 6.10-6.04 (m, 1H), 2.99-2.93 (m, 1H), 2.81-2.73 (m, 1H), 2.17 (dd, *J* = 12.8, 2.7 Hz, 1H), 2.02-1.93 (m, 1H), 1.78 (s, 3H), 1.76-1.70 (m, 2H), 1.63 (s, 3H), 1.28-1.10 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 169.0, 168.9, 136.3, 128.9, 105.2, 54.5, 39.7, 32.5, 30.5, 29.2, 28.1, 22.3, 22.1.

HRMS (ESI+) m/z calcd. for ($C_{13}H_{17}O_4$, M+H): 237.1121, found: (M+H): 237.1123.

3,3,8,9-Tetramethyl-2,4-dioxaspiro[5.5]undec-8-ene-1,5-dione (**12**) [5]. From Meldrum's acid (144 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and 2,3-dimethyl-1,3-butadiene (164 mg, 2.0 mmol) compound **12** (231 mg, 97%) was obtained as a white crystalline solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.49-2.48 (m, 2H), 2.12-2.11 (m, 4H), 1.75 (s, 3H), 1.72 (s, 3H), 1.68 (s, 3H), 1.65 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 169.9 (2C), 124.9, 121.1, 104.8, 48.6, 36.7, 31.4, 29.7, 28.6, 28.0, 19.1, 18.7.

HRMS (ESI+) m/z calcd. for ($C_{13}H_{19}O_4$, M+H): 239.1278, found: (M+H): 239.1270.

1,1'-(3,4-dimethylcyclohex-3-ene-1,1-diyl)bis(ethan-1-one) (**13**). From acetylacetone (100 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and 2,3-dimethyl-1,3-butadiene (164 mg, 2.0 mmol) compound **13** (80 mg, 41%) was obtained as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.34 (bs, 2H), 2.04 (s, 6H), 2.04-2.01 (m, 2H), 1.92-1.85 (m, 2H), 1.61 (s, 3H), 1.50 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 206.7 (2C), 125.4, 122.8, 67.1, 35.1, 28.8, 27.3, 26.1 (2C), 19.2, 18.7.

HRMS (ESI+) m/z calcd. for ($C_{12}H_{19}O_2$, M+H): 195.1380, found: (M+H): 195.1381.

3,3,9-Trimethyl-2,4-dioxaspiro[5.5]undec-8-ene-1,5-dione (**14a**) and *3,3,8-trimethyl-2,4-dioxaspiro[5.5]undec-8-ene-1,5-dione* (**14b**) [6]. From Meldrum's acid (144 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and isoprene (340 mg, 5.0 mmol) the mixture of compounds **14a** and **14b** in 95/5 ratio (204 mg, 91%) was obtained as a white crystalline solid.

Major isomer 14a: ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.38-5.33 (m, 1H), 2.59-2.55 (m, 2H), 2.17-2.12 (m, 2H), 2.11-2.06 (m, 2H), 1.74-1.71 (m, 3H), 1.70-1.66 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 169.8 (2C), 133.1, 116.1, 104.7, 46.9, 31.5, 30.9, 29.5, 28.5, 26.4, 23.4.

Minor isomer 14b: ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.53-5.51 (m, 1H), 2.46-2.44 (m, 2H), 2.17-2.12 (m, 2H), 2.11-2.06 (m, 2H), 1.74-1.71 (m, 3H), 1.70-1.66 (m, 6H).

HRMS (ESI+) m/z calcd. for ($C_{12}H_{17}O_4$, M+H): 225.1121, found: (M+H): 225.1114.

Ethyl 1-acetyl-4-methylcyclohex-3-ene-1-carboxylate (**15a**) and *ethyl 1-acetyl-3-methylcyclohex-3-ene-1-carboxylate* (**15b**). From acetoacetic ester (130 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and isoprene (340 mg, 5.0 mmol) the mixture of compounds **15a** and **15b** >95/5 ratio (19 mg, 9%) was obtained as a colorless oil.

Major isomer 15a: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.40-5.31 (m, 1H), 4.22-4.13 (m, 2H), 2.58-2.47 (m, 1H), 2.46-2.36 (m, 1H), 2.17 (s, 3H), 2.16-2.10 (m, 1H), 2.08-1.89 (m, 3H), 1.61 (s, 3H), 1.24 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 205.4, 172.3, 133.7, 118.1, 61.5, 59.0, 30.1, 27.3, 27.2, 25.9, 23.4, 14.2.

HRMS (ESI+) m/z calcd. for ($C_{12}H_{19}O_3$, M+H): 211.1329, found: (M+H): 211.1337.

1,1'-(4-Methylcyclohex-3-ene-1,1-diyl)bis(ethan-1-one) (**16a**) and *1,1'-(3-methylcyclohex-3-ene-1,1-diyl)bis(ethan-1-one)* (**16b**). From acetylacetone (100 mg, 1.0 mmol), L-proline (6 mg, 0.05 mmol) and isoprene (340 mg, 5.0 mmol) the mixture of compounds **16a** and **16b** in 87/13 ratio (50 mg, 28%) was obtained as a light yellow oil.

Major isomer 16a: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.38-5.33 (m, 1H), 2.48-2.44 (m, 2H), 2.14-2.07 (m, 8H), 1.96-1.90 (m, 2H), 1.59 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 206.8 (2C), 134.1, 118.1, 66.7, 29.3, 27.3, 27.1, 26.2 (2C), 23.3.

Minor isomer 16b: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.35-5.30 (m, 1H), 2.37-2.35 (m, 2H), 2.14-2.07 (m, 8H), 1.96-1.90 (m, 2H), 1.70 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 206.6 (2C), 131.3, 120.7, 67.2, 33.7, 29.3, 26.5 (2C), 23.7, 22.6.

HRMS (ESI+) m/z calcd. for ($C_{11}H_{17}O_2$, M+H): 181.1223, found: (M+H): 181.1222.

2. ^{1}H and ^{13}C NMR spectra of the compounds 3–10 and 12–16

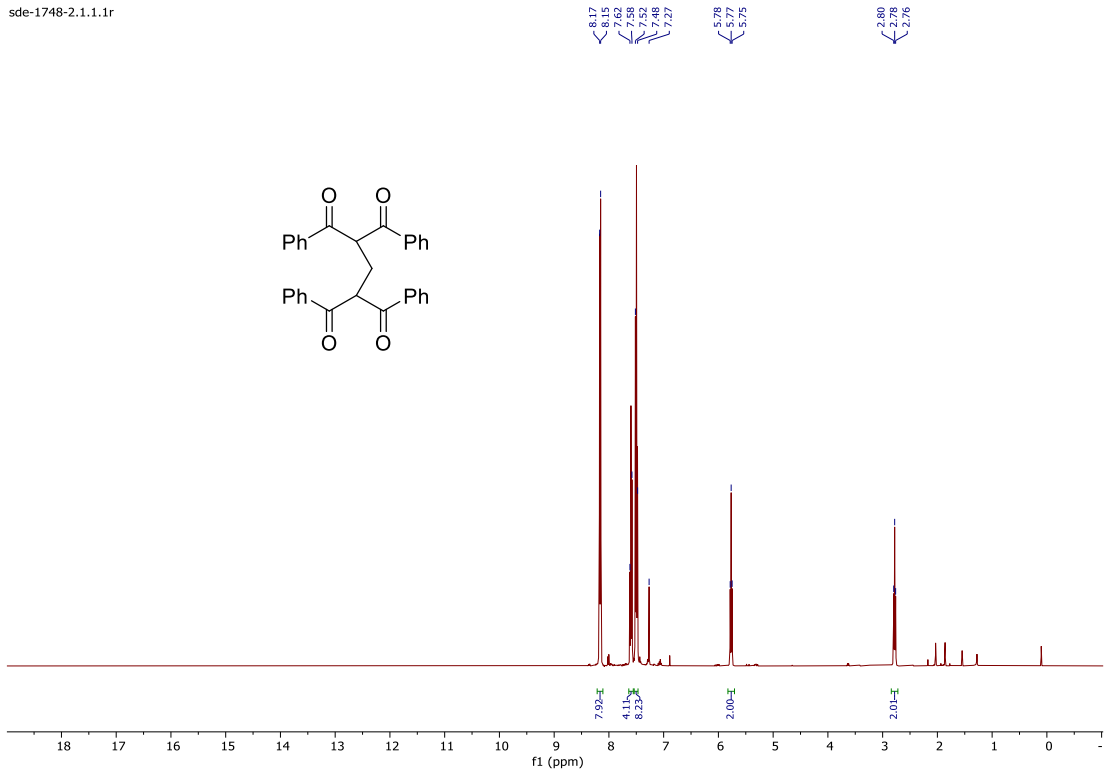

Figure S1. ^{1}H NMR spectra of compound **3.**

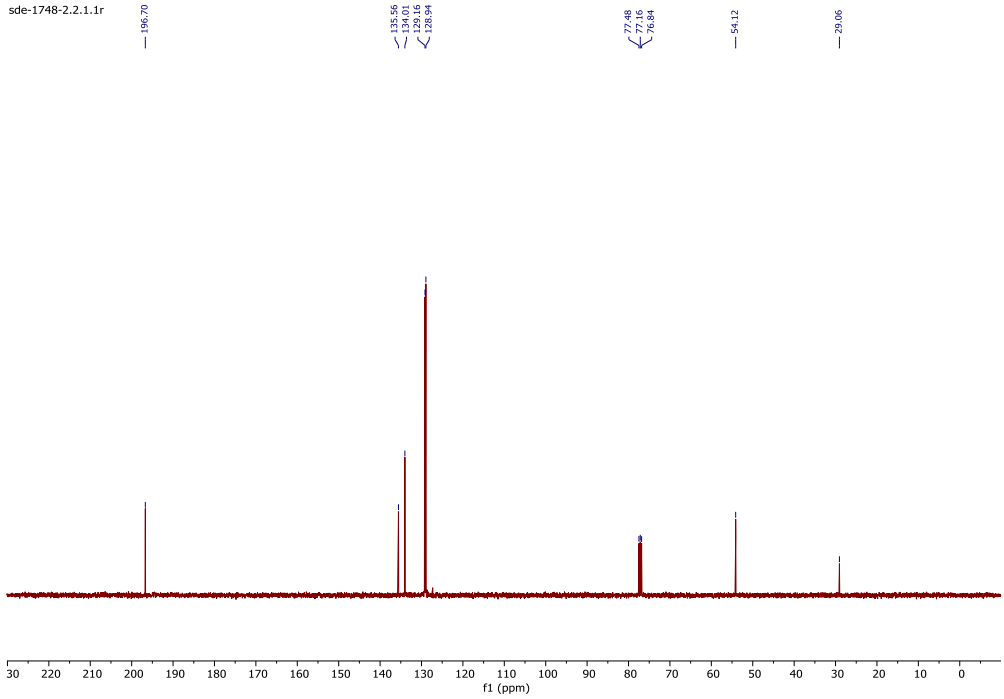

Figure S2. ^{13}C NMR spectra of compound **3.**

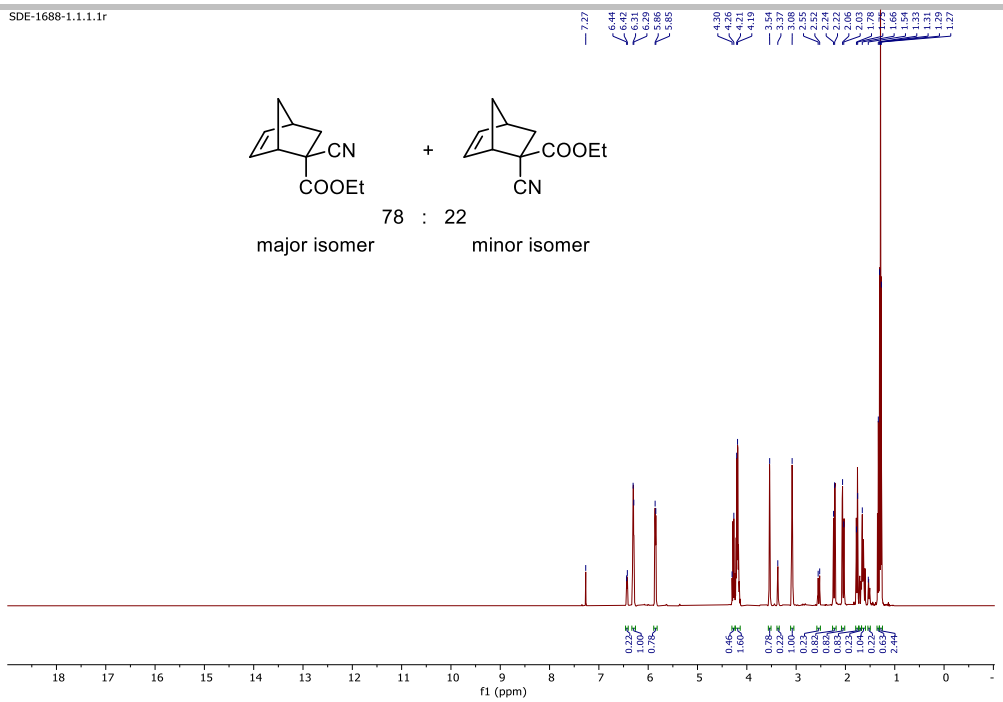

Figure S3. ^{1}H NMR spectra of the mixture of compounds **4a** and **4b.**

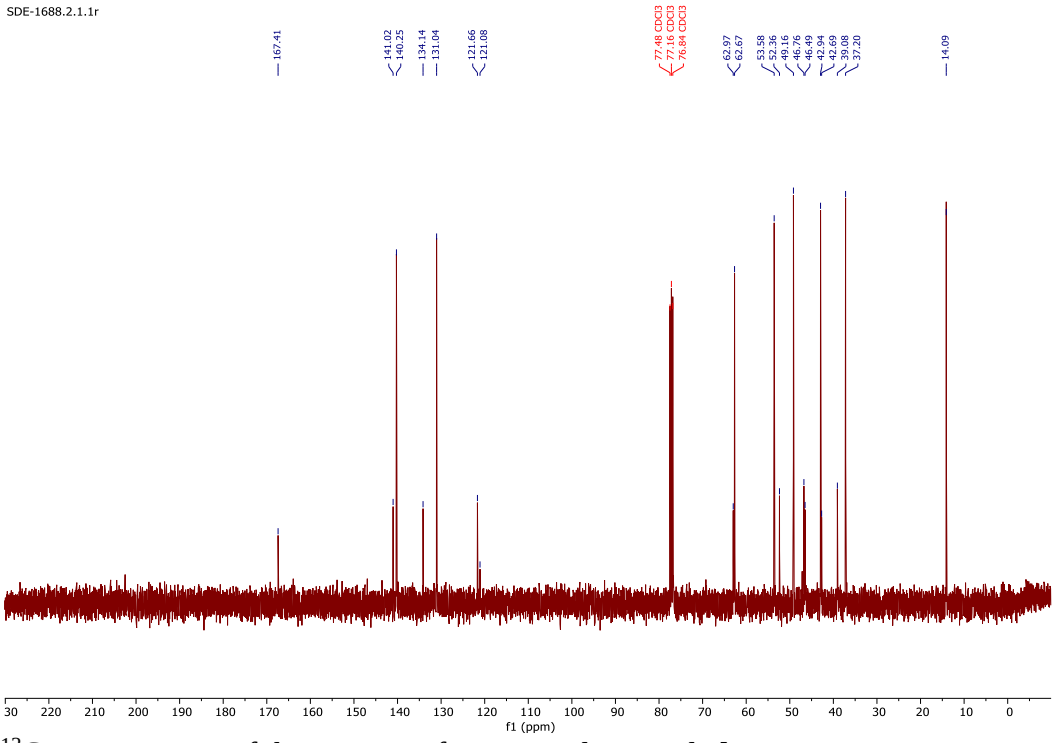

Figure S4. ^{13}C NMR spectra of the mixture of compounds **4a** and **4b.**

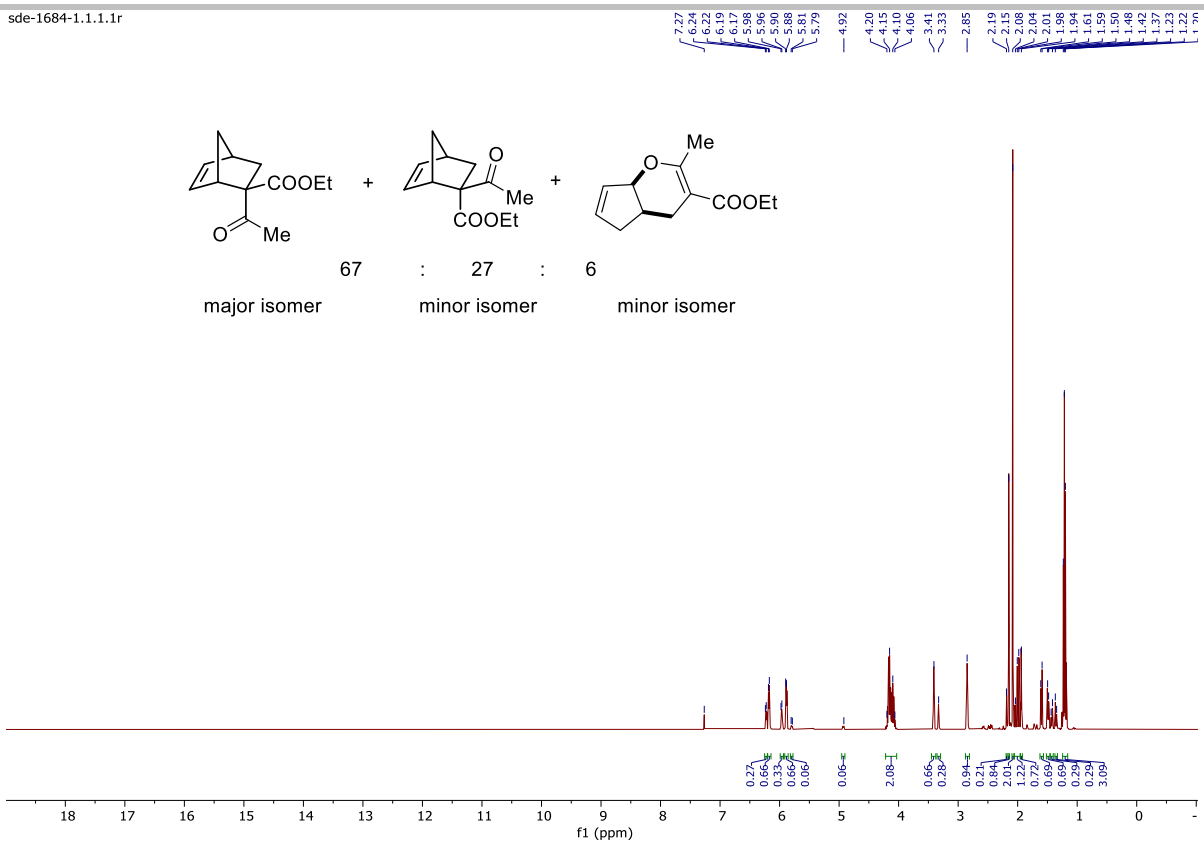

Figure S5. ^{1}H NMR spectra of the mixture of compounds **5a**, **5b** and **6.**

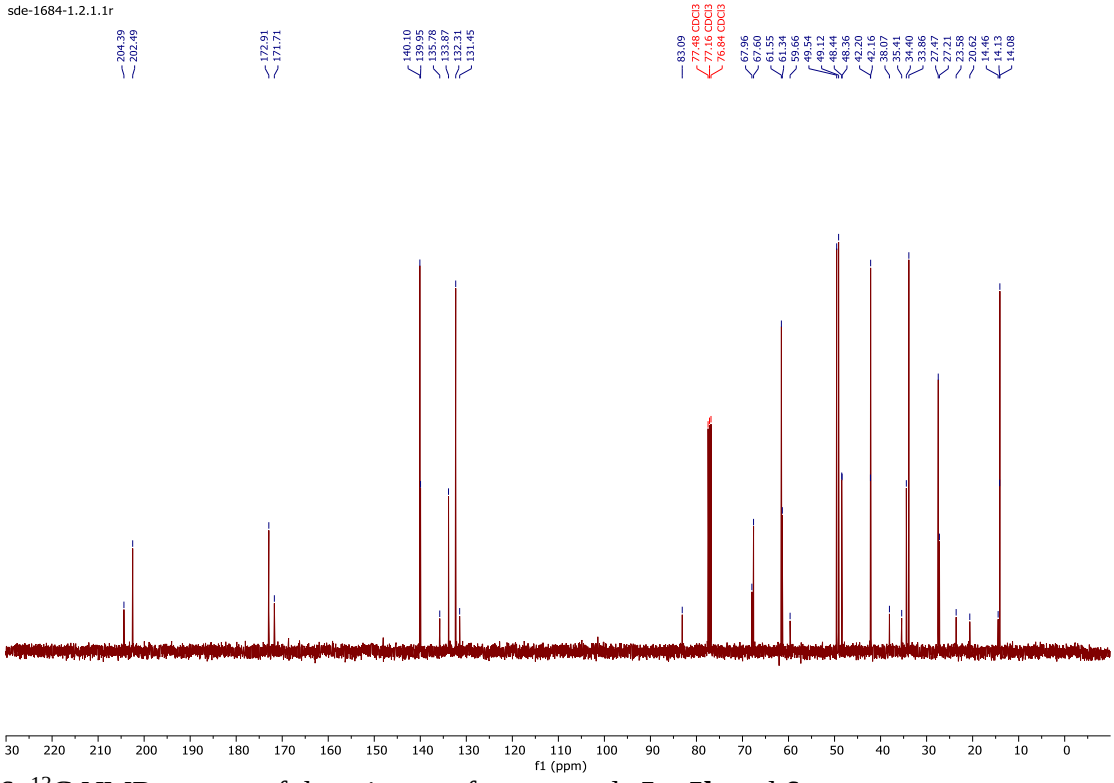

Figure S6. ^{13}C NMR spectra of the mixture of compounds **5a**, **5b** and **6.**

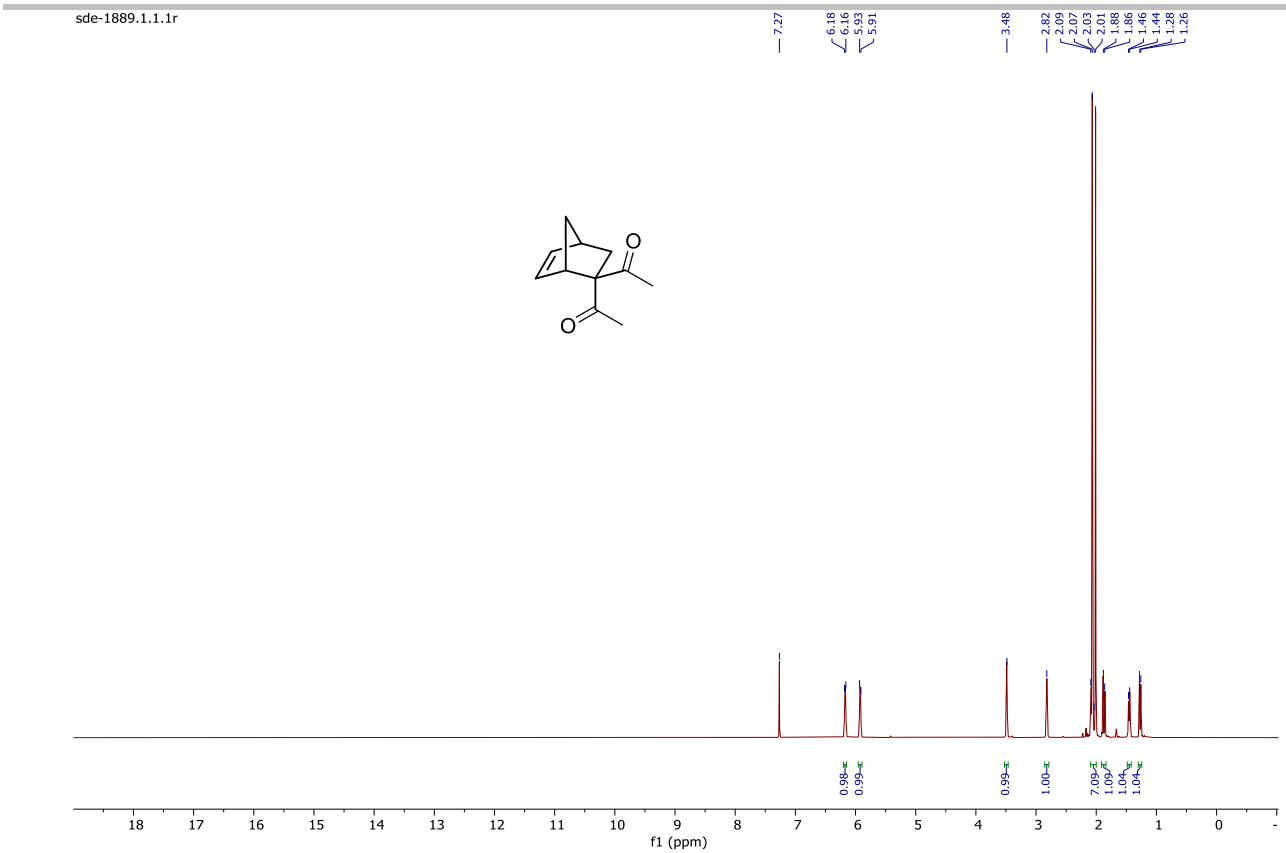

Figure S7. ^{1}H NMR spectra of compound **7.**

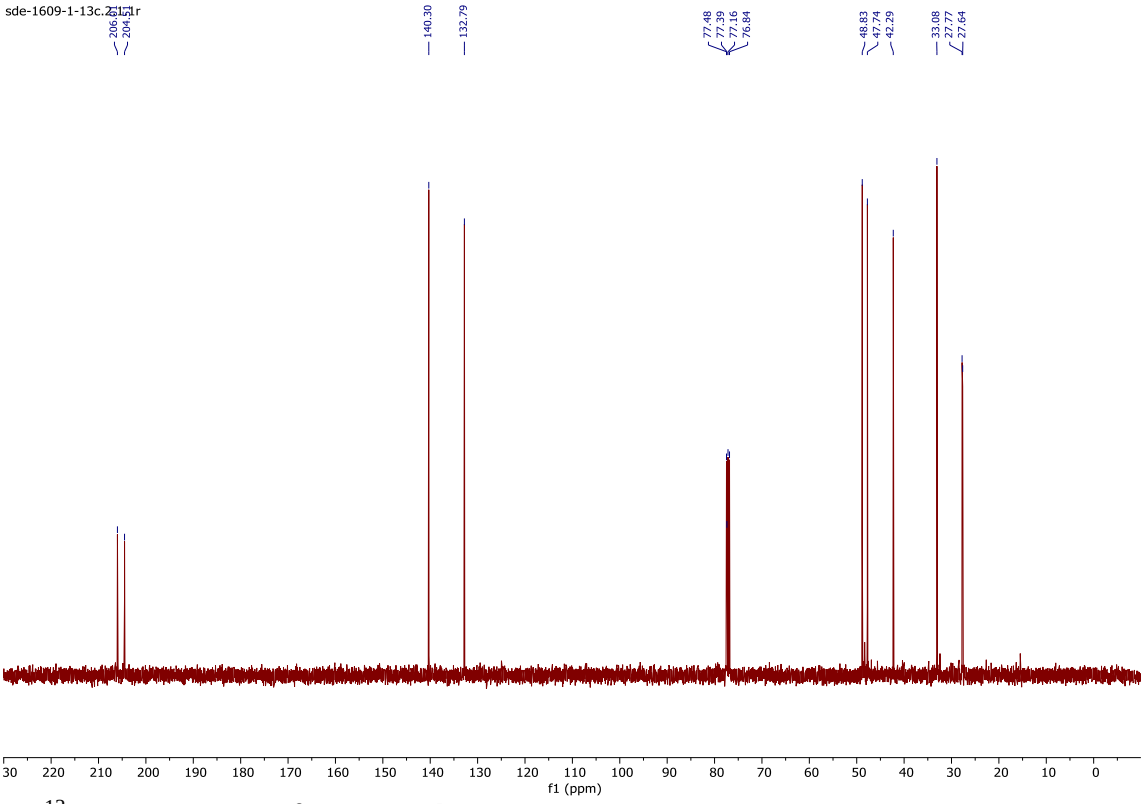

Figure S8. ^{13}C NMR spectra of compound **7.**

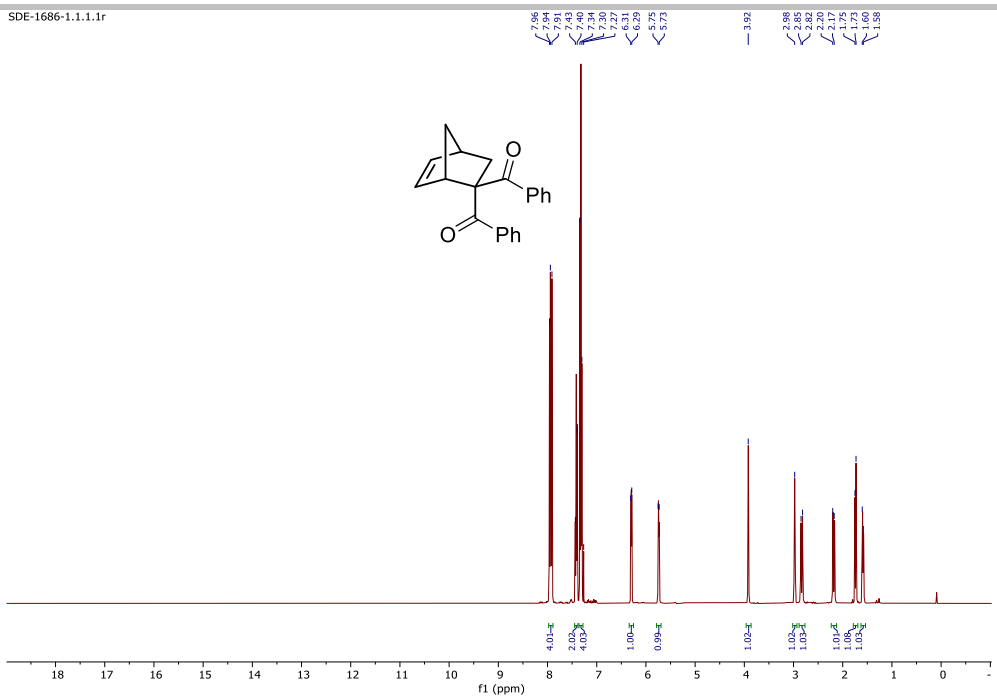

Figure S9. ^{1}H NMR spectra of compound **8.**

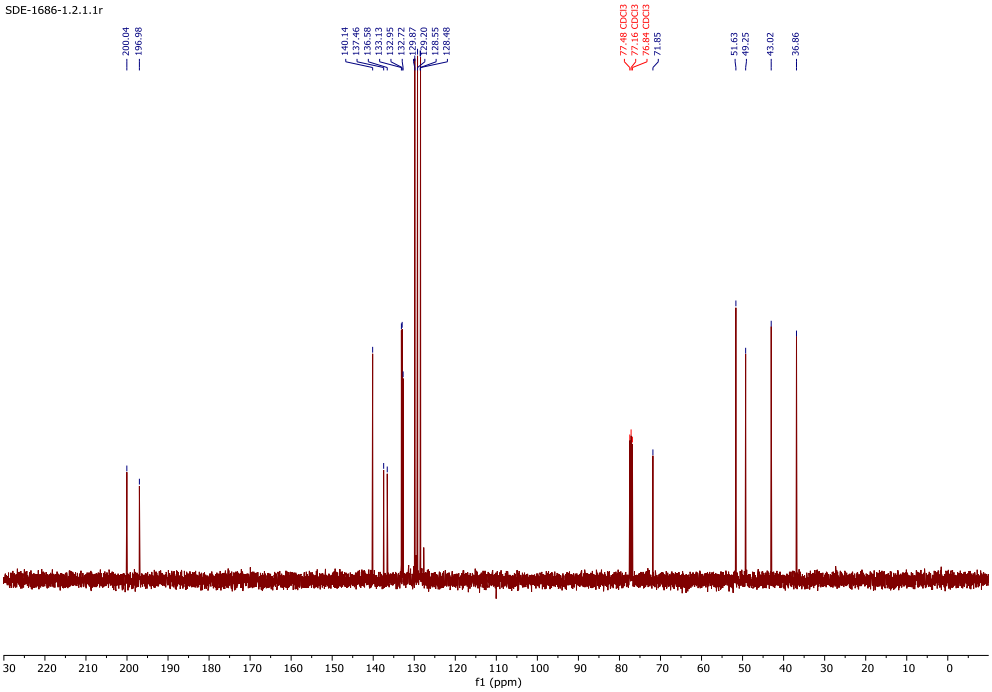

Figure S10. ^{13}C NMR spectra of compound **8.**

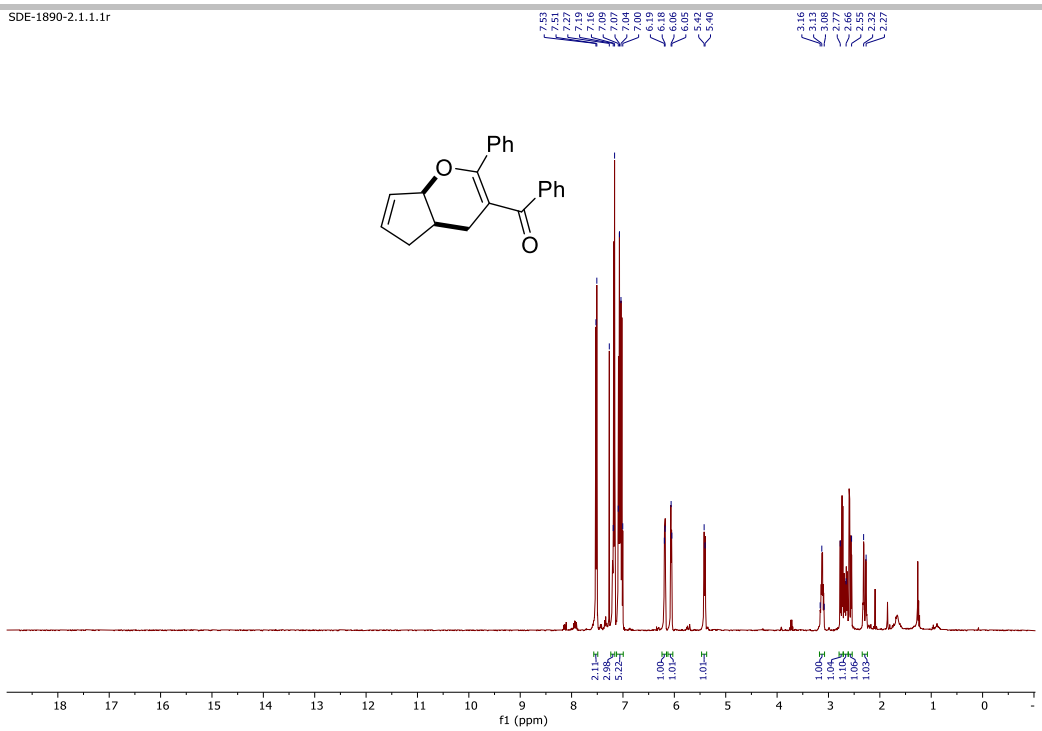

Figure S11. ^{1}H NMR spectra of compound **9.**

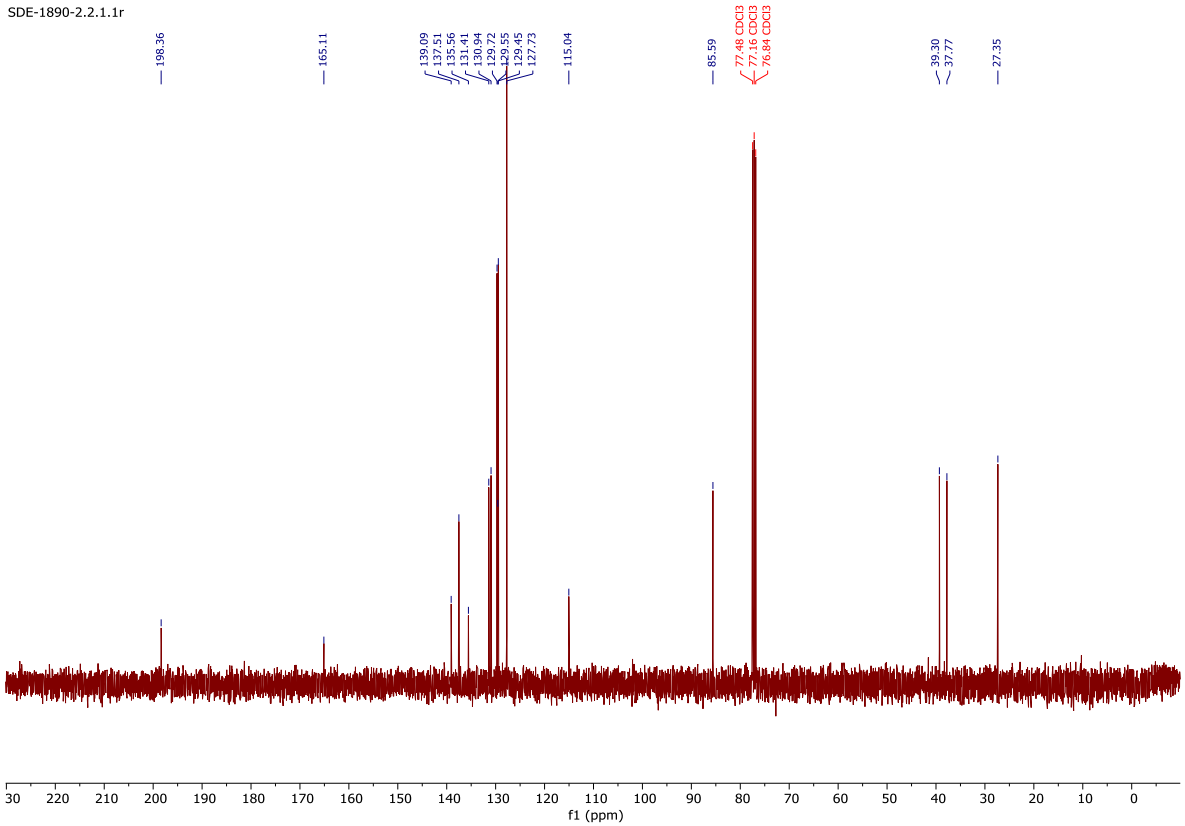

Figure S12. ^{13}C NMR spectra of compound **9.**

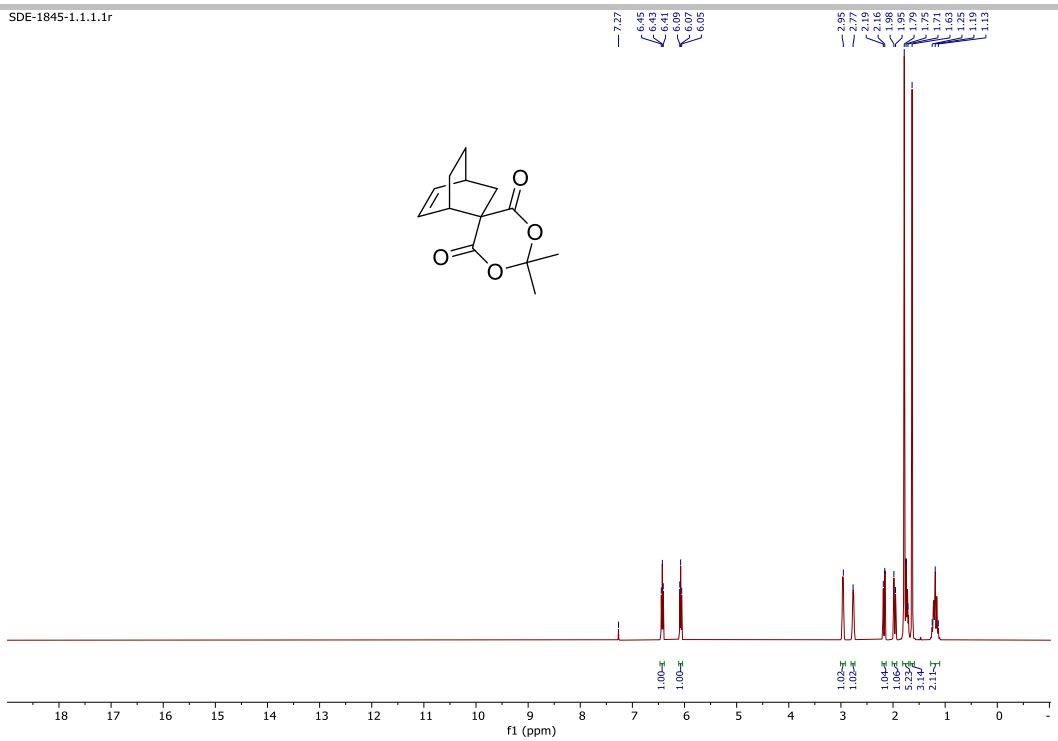

Figure S13. ^{1}H NMR spectra of compound **10.**

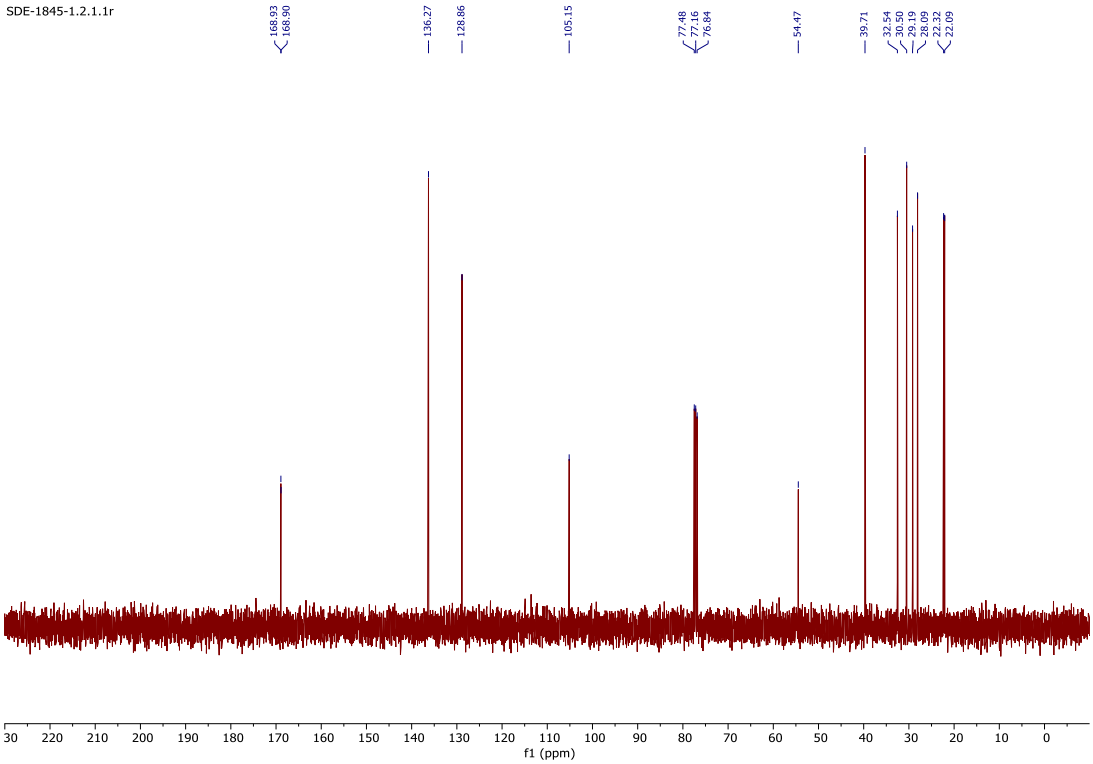

Figure S14. ^{13}C NMR spectra of compound **10.**

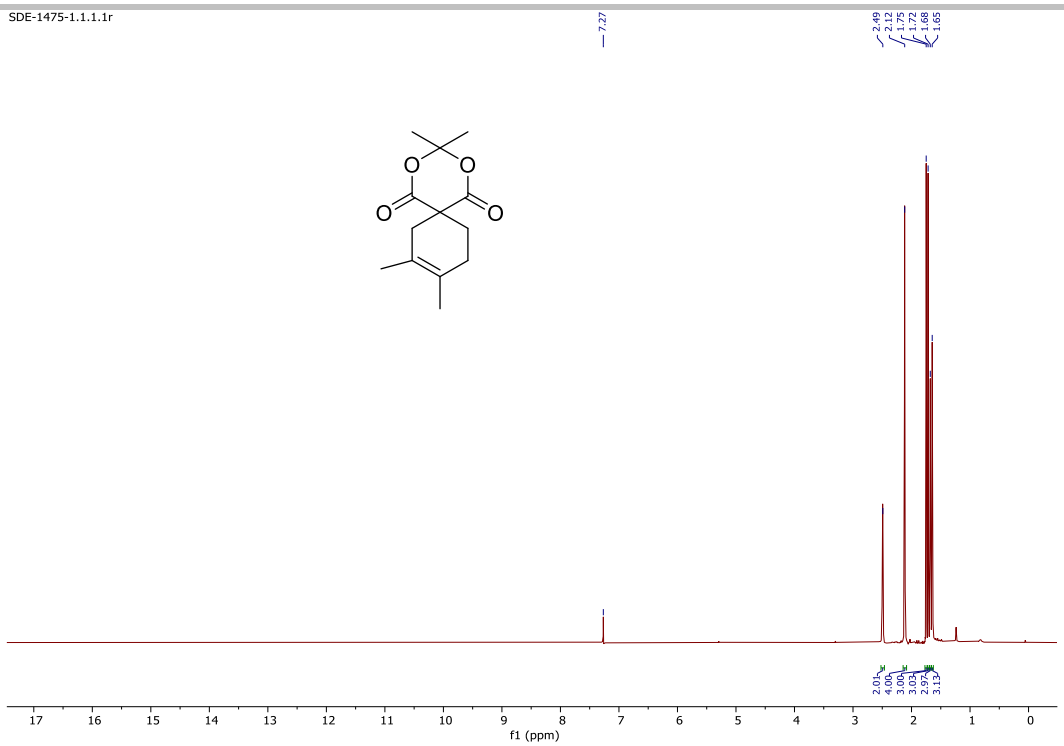

Figure S15. ^{1}H NMR spectra of compound **12.**

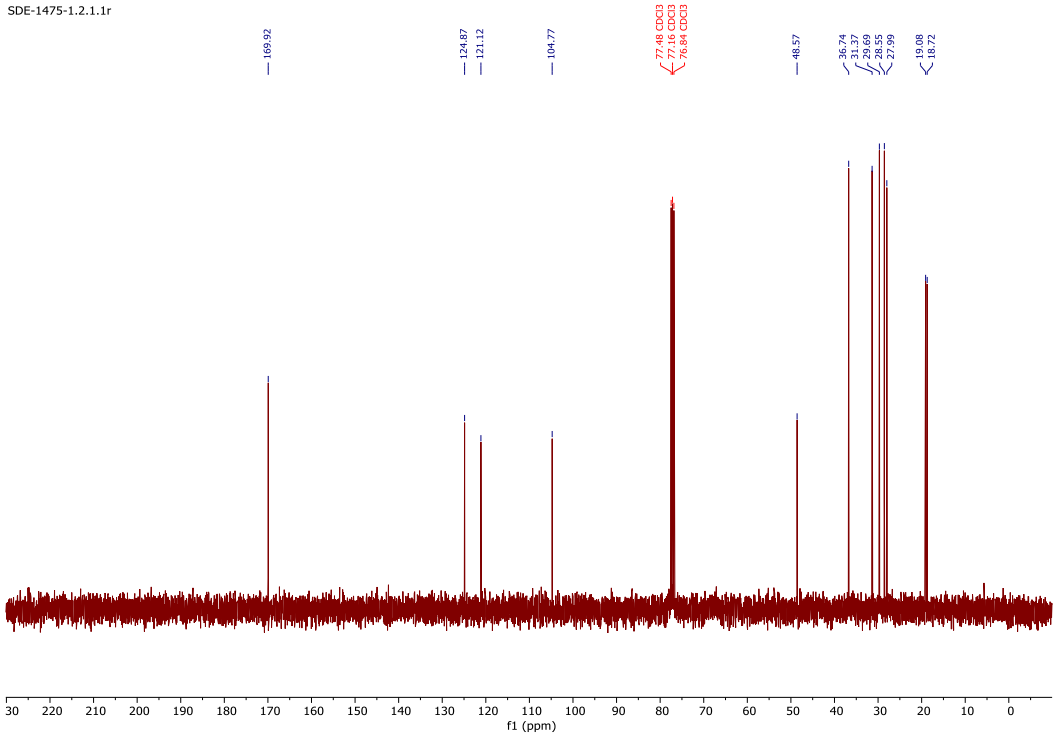

Figure S16. ^{13}C NMR spectra of compound **12.**

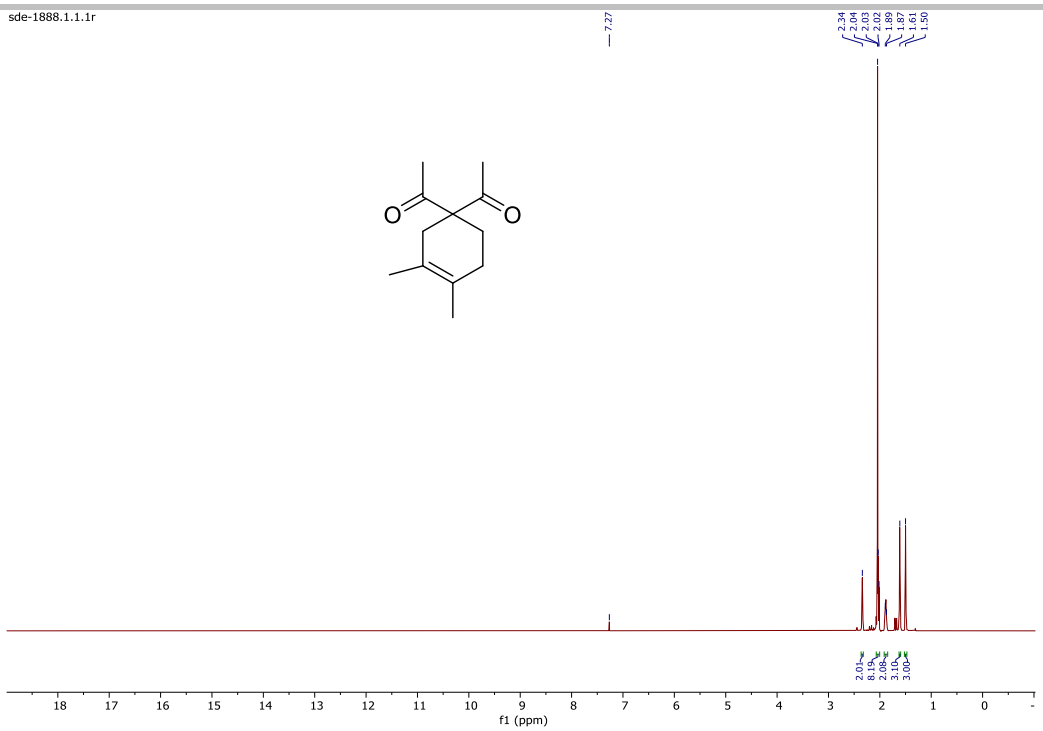

Figure S17. ^{1}H NMR spectra of compound **13.**

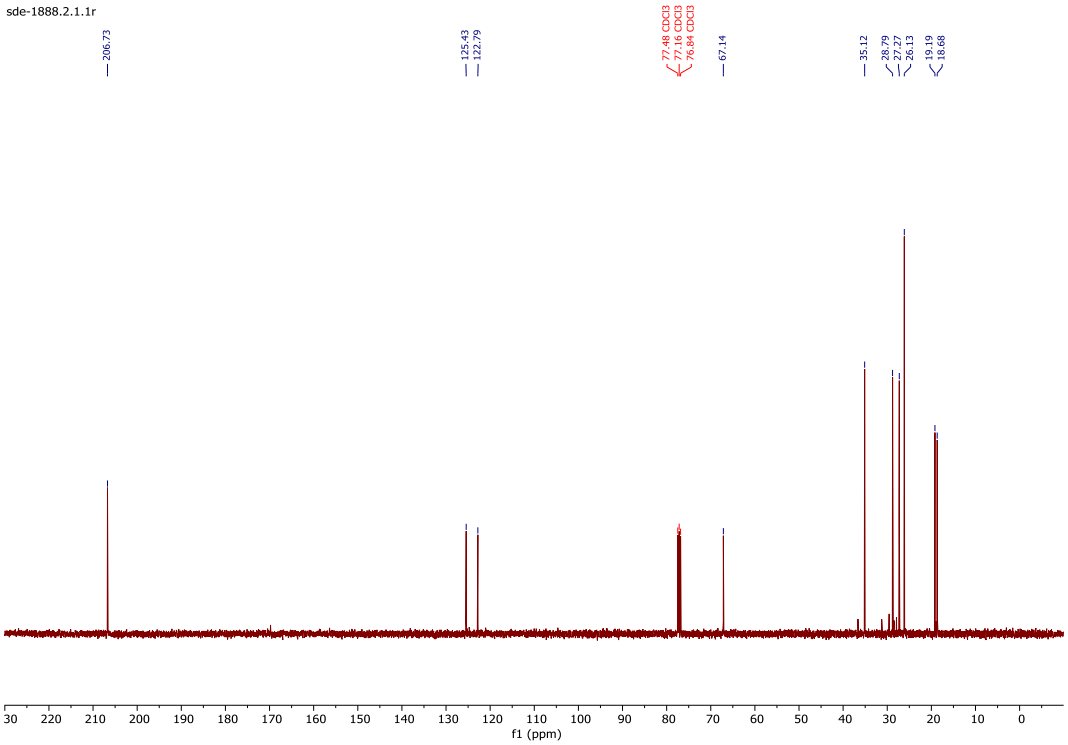

Figure S18. ^{13}C NMR spectra of compound **13.**

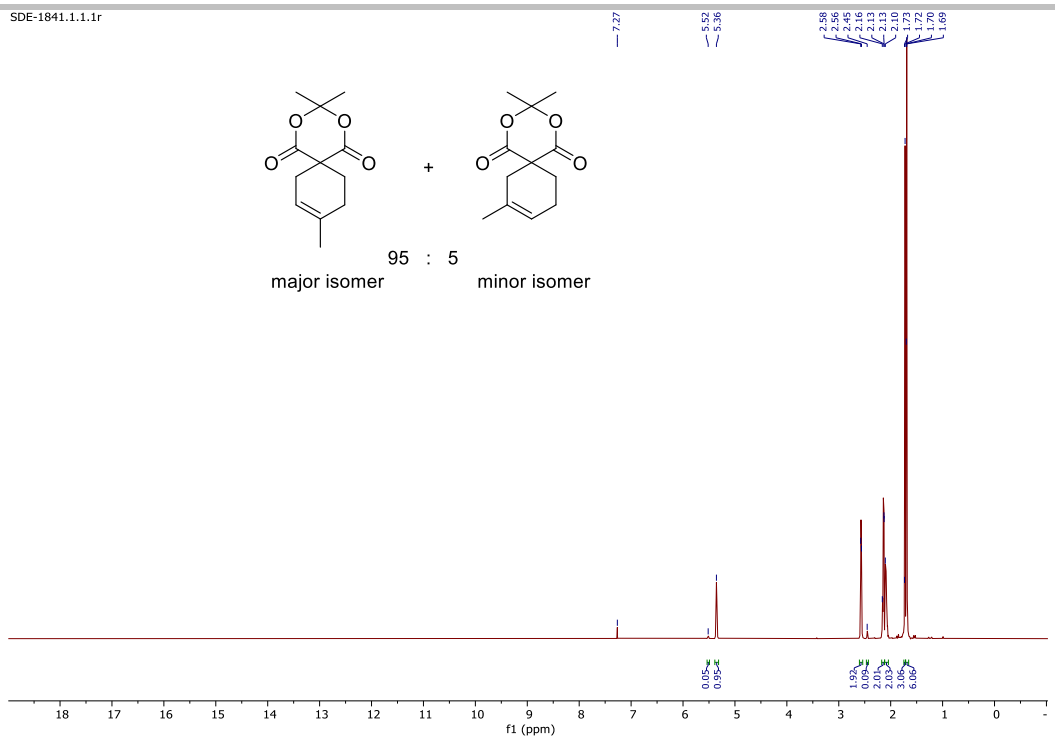

Figure S19. ^{1}H NMR spectra of the mixture of compounds **14a** and **14b.**

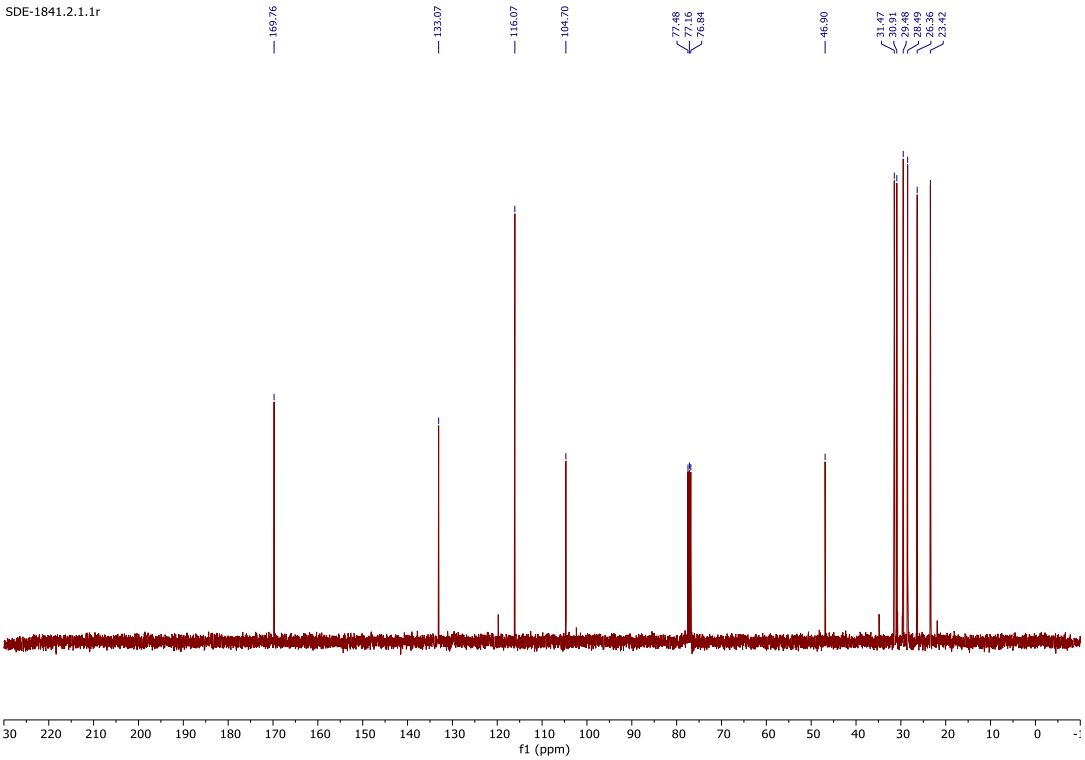

Figure S20. ^{13}C NMR spectra of the mixture of compounds **14a** and **14b.**

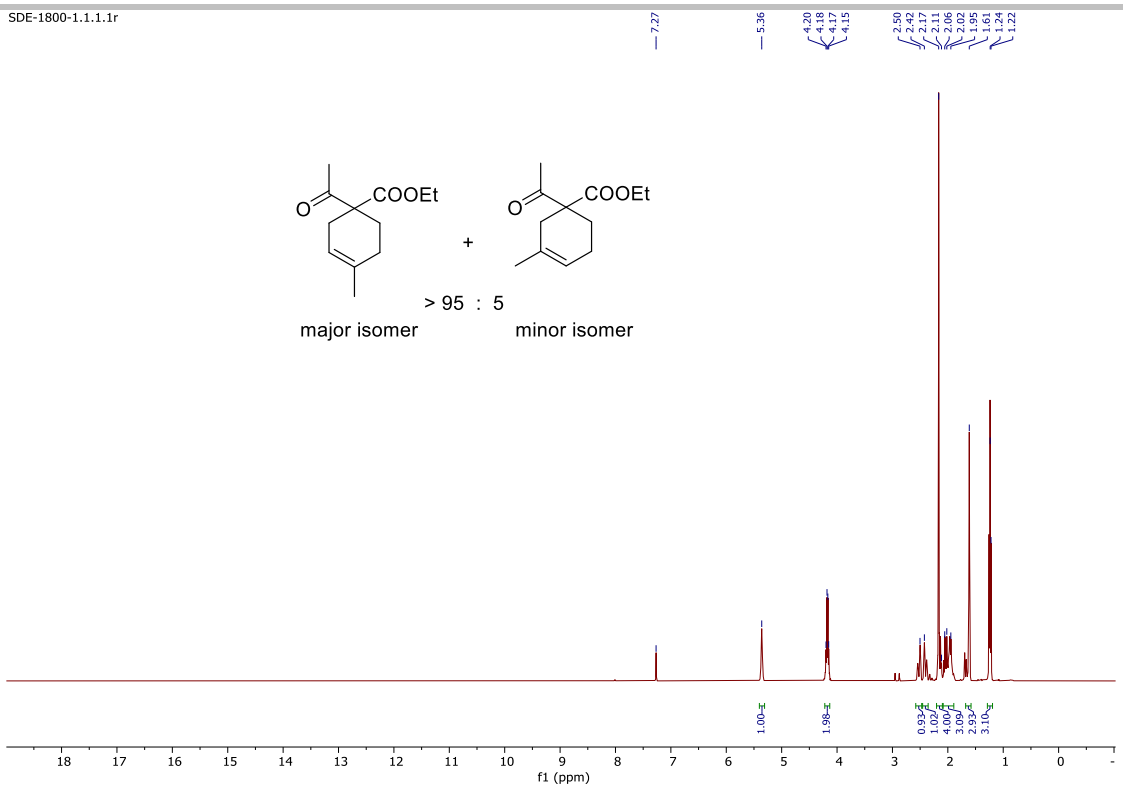

Figure S21. ^{1}H NMR spectra of the mixture of compounds **15a** and **15b.**

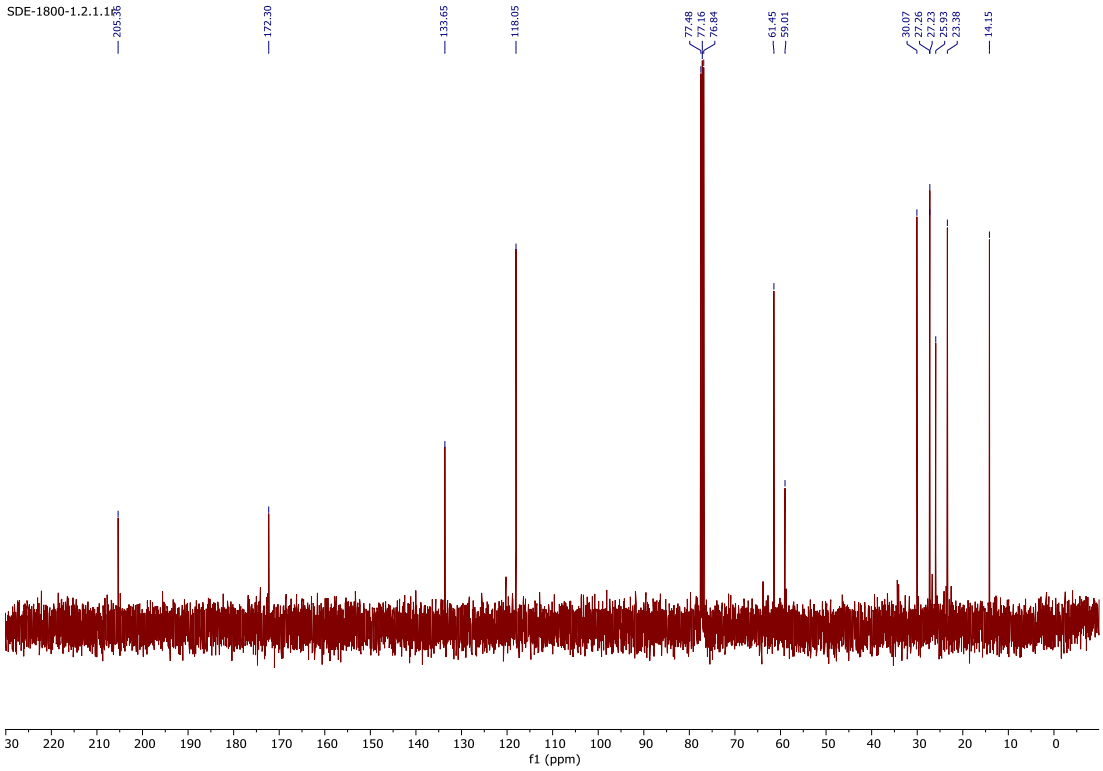

Figure S22. ^{13}C NMR spectra of the mixture of compounds **15a** and **15b.**

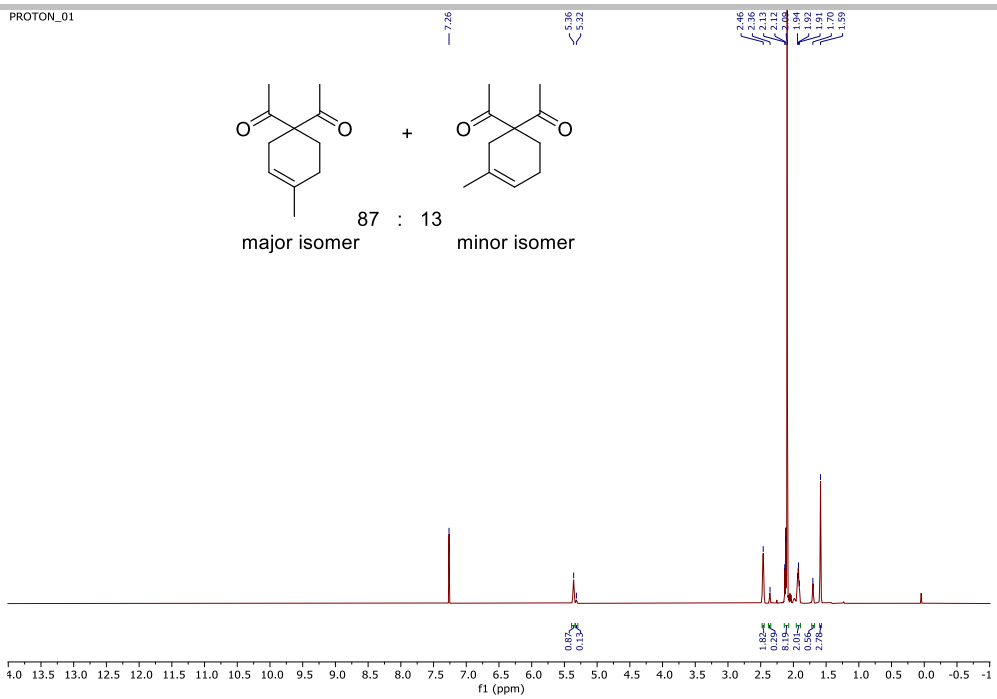

Figure S23. ^{1}H NMR spectra of the mixture of compounds **16a** and **16b.**

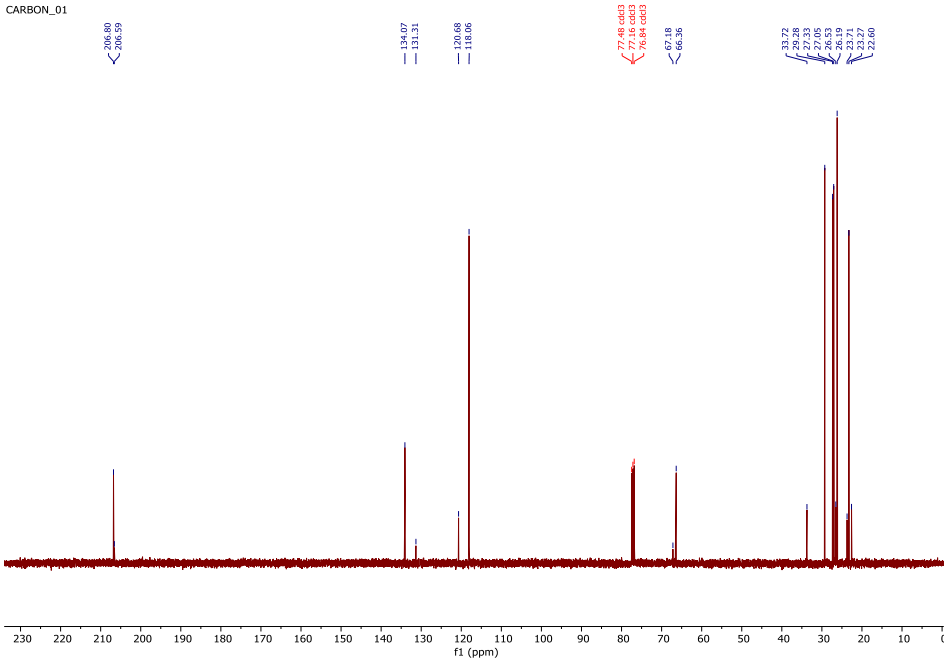

Figure S24. ^{13}C NMR spectra of the mixture of compounds **16a** and **16b.**

3. ^{1}H NMR spectra of reaction mixtures formed during bromination of compounds 4 and 5

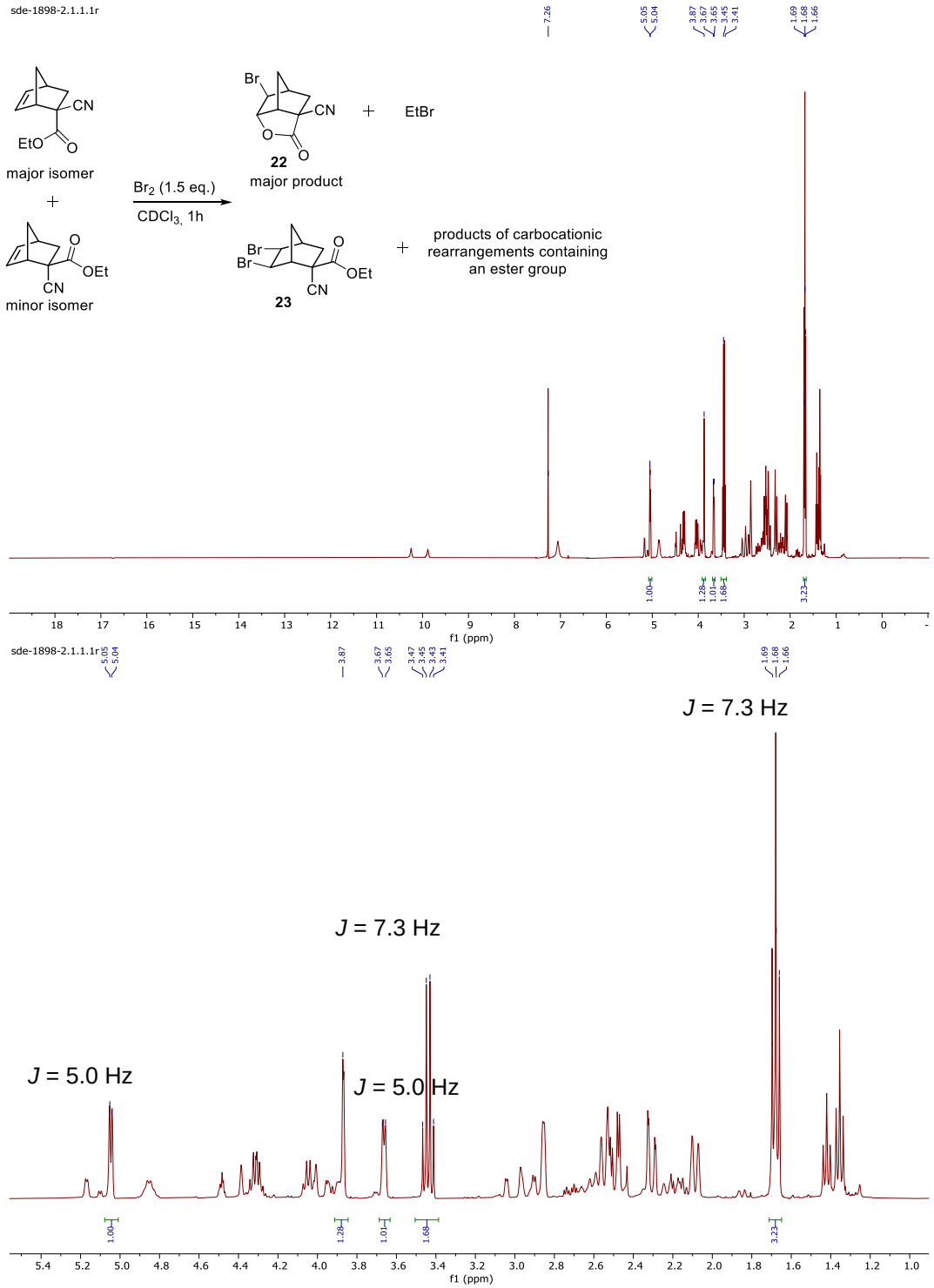

Figure S25. ^{1}H NMR spectrum of the reaction mixture of bromination of compounds **4a** and **4b**. The characteristic signals of the main reaction products, as well as EtBr, are labeled.

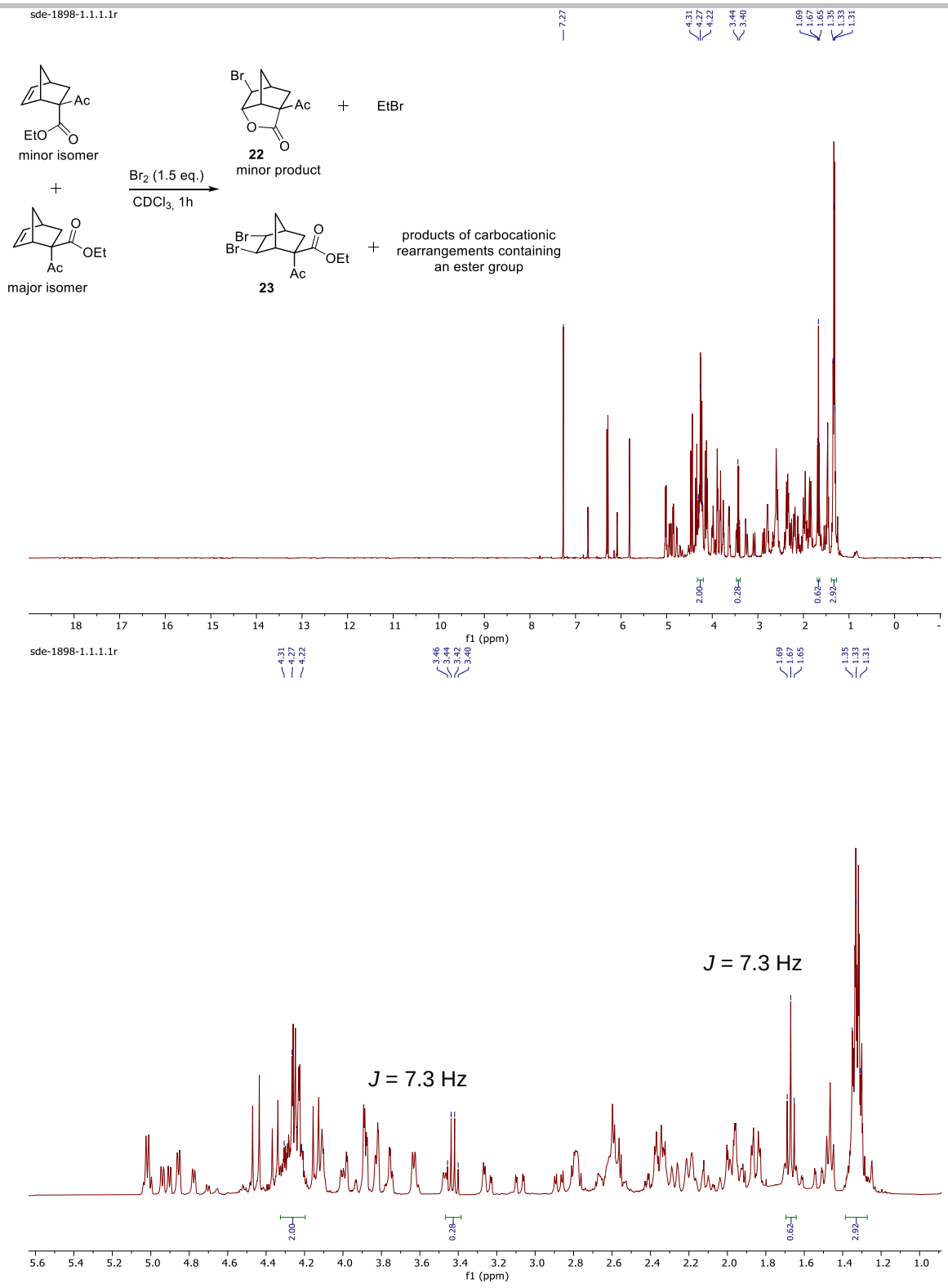

Figure S26. ^{1}H NMR spectrum of the reaction mixture of bromination of compounds **5a** and **5b**. The characteristic signals of the main reaction products, as well as EtBr, are labeled.

4. LC–MS/MS spectra of compound mixtures

HPLC-MS/MS method parameters for compound analysis

Agilent 1290 Infinity II – 5500 QTRAP (Sciex)				
Chromatographic parameters				
Solvent A	0,1% HCOOH in H_2O			
Solvent B	0,1% HCOOH in CH_3CN			
Chromatographic column	Eclipse XDB C18, 150*3 mm, 5 μm (Agilent)			
Gradient	Time, min	Flow rate, mL/min	%A	%B
	0,00	0,4	80,0	80,0
	1,00	0,4	80,0	80,0
	10,0	0,4	20,0	20,0
	12,0	0,4	20,0	20,0
	12,1	0,4	80,0	80,0
	15,0	0,4	80,0	80,0
Column temperature	40ºC			
Autosampler temperature	8ºC			
Injection volume	10 μL			
Total analysis time	15 min			
MS/MS parameters				
Ion source type	Turbo Spray			
Ionization mode	Positive/Negative			
Source temperature, ºC	630			
Nebulizer voltage (IS), V	5500			
Curtain gas (CUR), psi	30			
Nebulizer gas (GS1), psi	70			
Heater gas (GS2), psi	70			
Mass range	100 - 1000			
Scanning quadrupole	Q3			
Declustering potential, V	60			

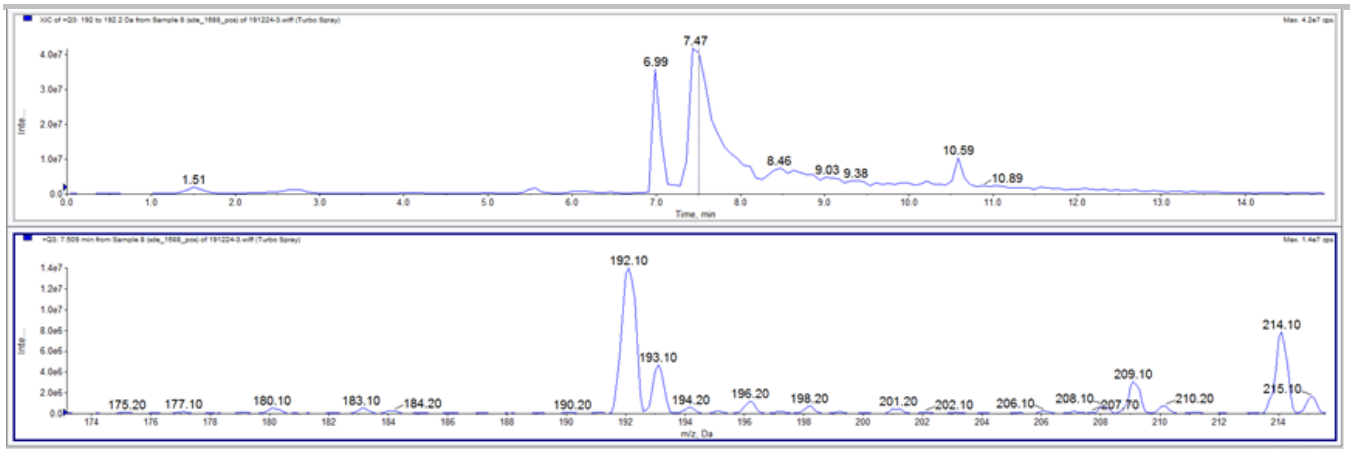

Figure S27. LC-MS/MS spectra of the compounds **4a** and **4b** mixture.

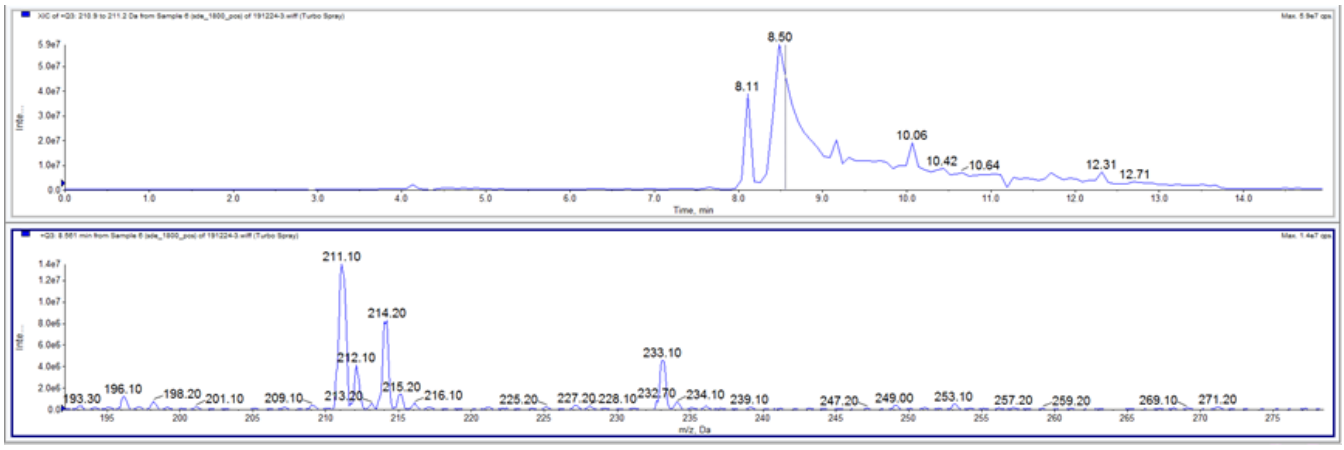

Figure S28. LC-MS/MS spectra of the compounds **5a**, **5b** and **6** mixture.

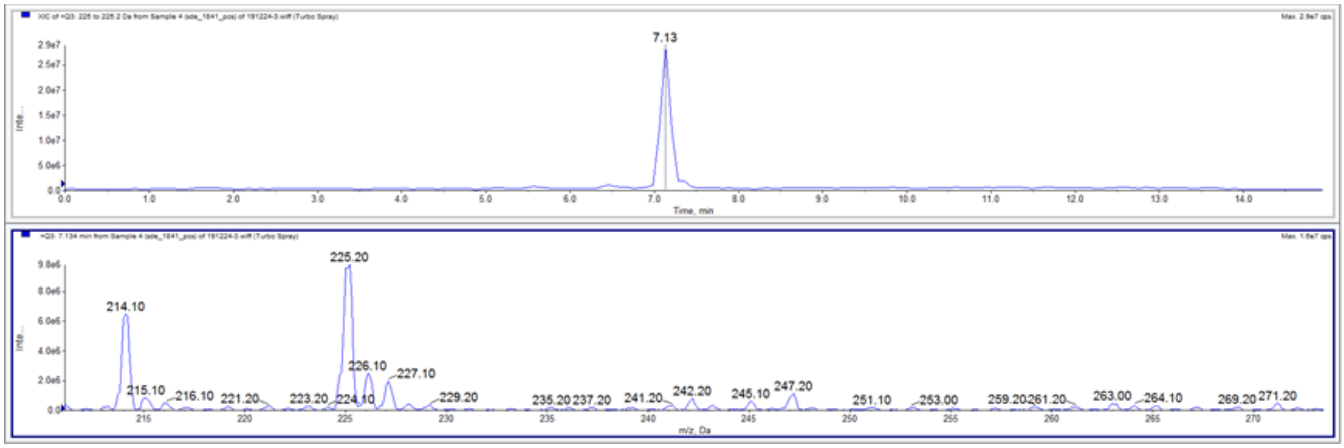

Figure S29. LC-MS/MS spectra of the compounds **14a** and **14b** mixture.

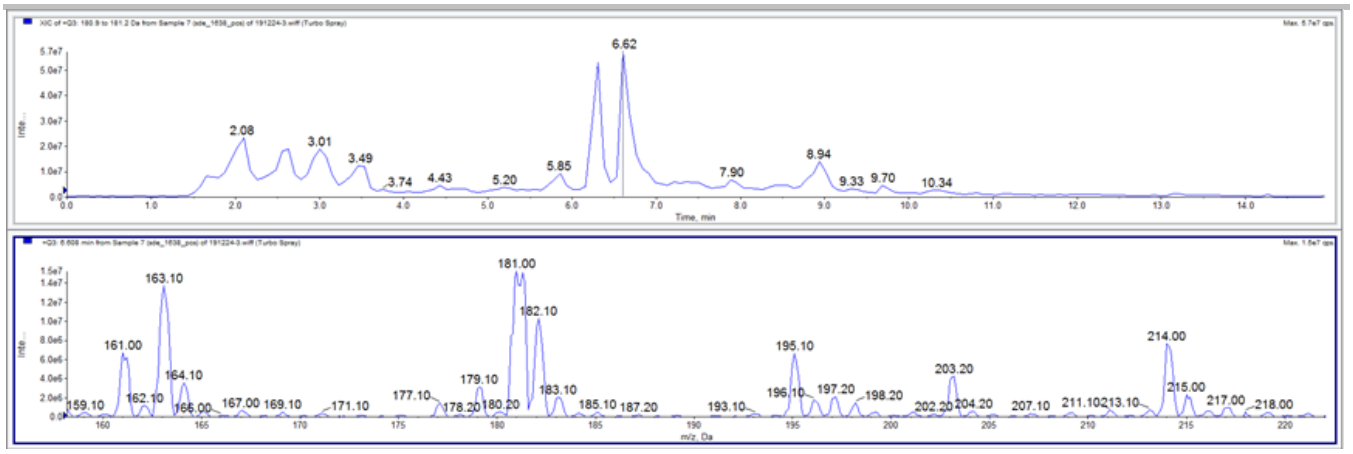

Figure S30. LC-MS/MS spectra of the compounds **16a** and **1b** mixture.

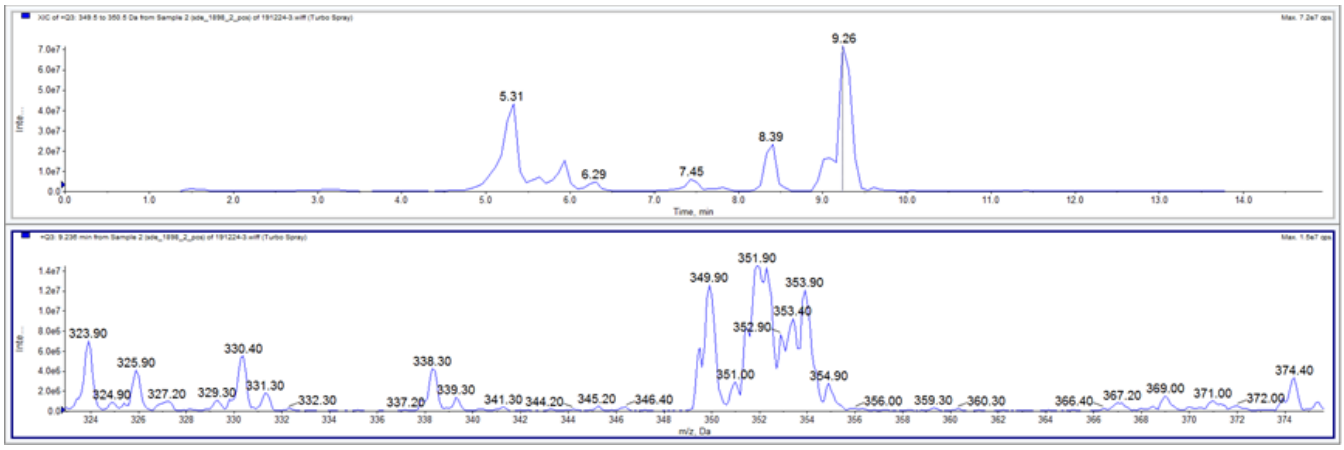

Figure S31. LC-MS/MS spectra of the the reaction mixture **4a** + **4b** + Br_2.

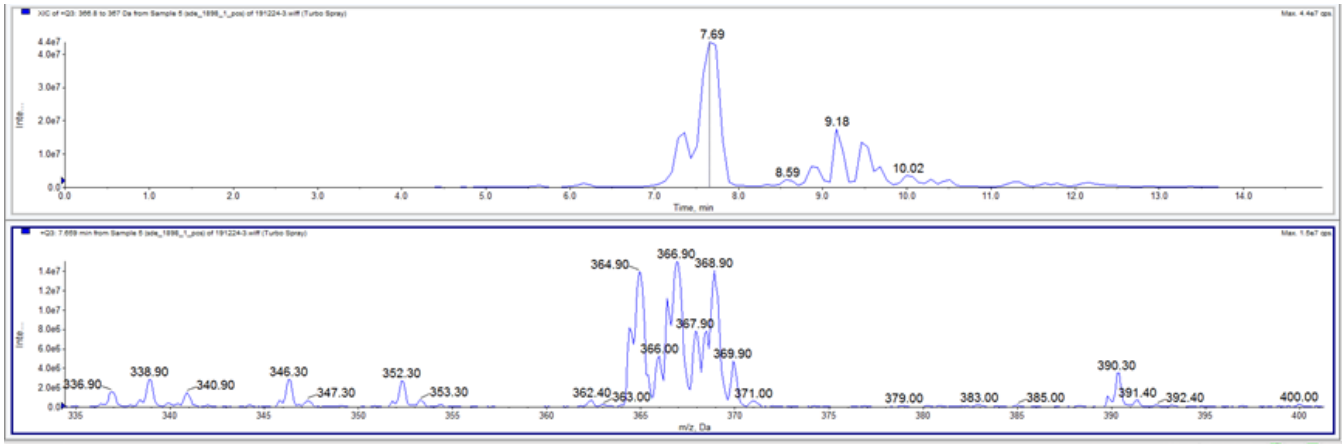

Figure S32. LC-MS/MS spectra of the the reaction mixture **5a** + **5b** + Br_2.

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