

Methyl 12-Methyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxo-dibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate and Related Compounds

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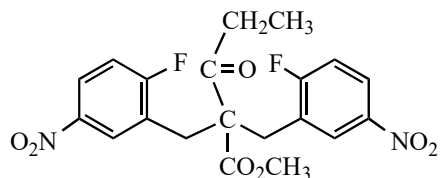
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Supplemental Information–Spectra of Related Compounds

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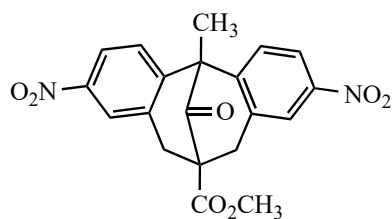
carboxylate (**1g**)

Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxohexanoate (**5a**)



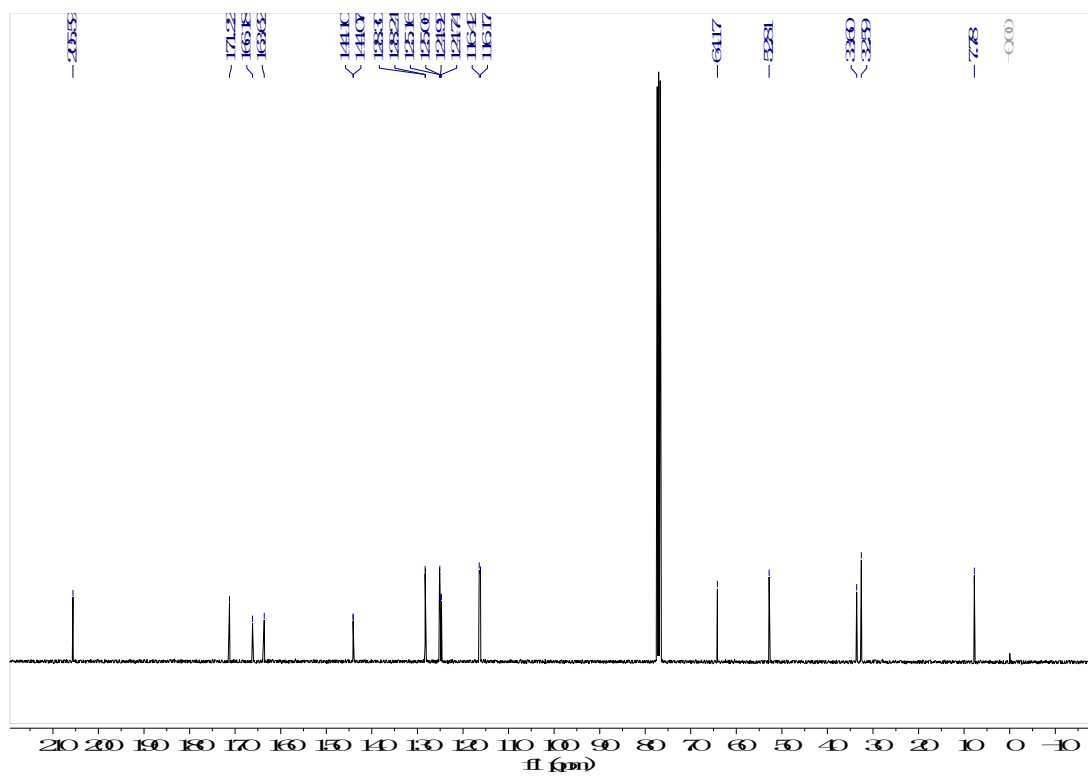
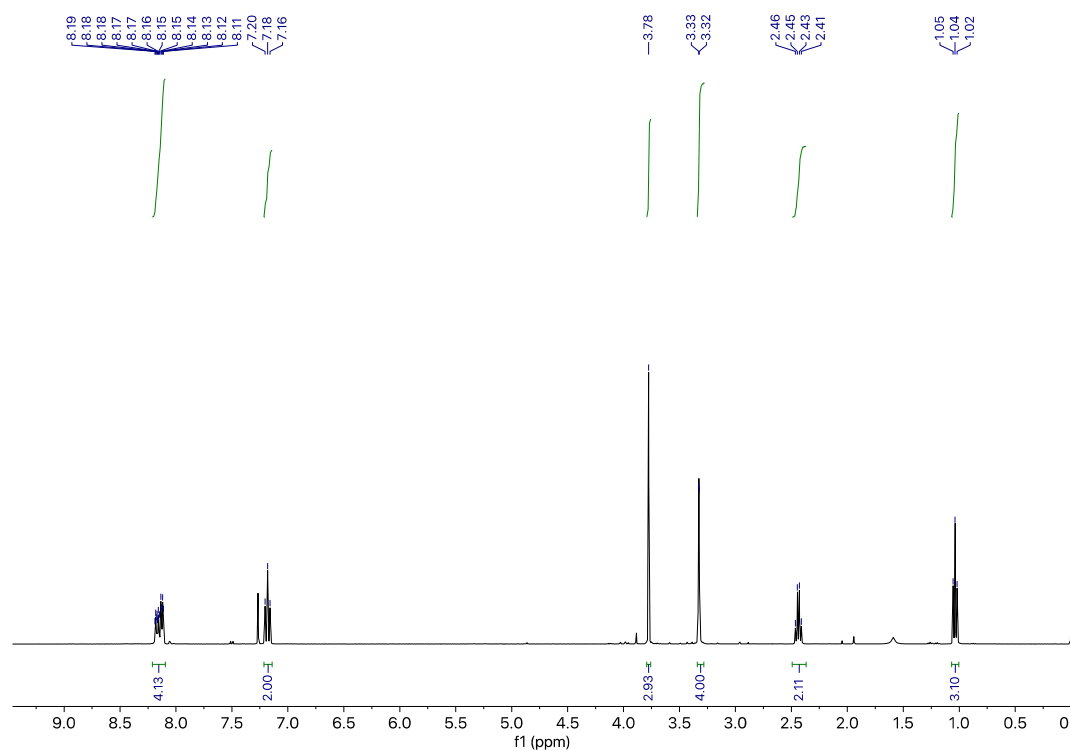
Yield: 140 mg (64%) as a yellow solid, m.p. 112-114 °C. IR: 1741, 1716, 1532, 1345 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.17 (ddd, *J* = 9.0, 4.4, 2.8 Hz, 2H), 8.13 (dd, *J* = 6.4, 2.8 Hz, 2H), 7.18 (t, *J* = 9.0 Hz, 2H), 3.78 (s, 3H), 3.33 (apparent s, 4H), 2.44 (q, *J* = 7.1 Hz, 2H), 1.04 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 205.6, 171.2, 164.9 (d, *J* = 256.8 Hz), 144.1 (d, *J* = 2.8 Hz), 128.3 (d, *J* = 6.1 Hz), 125.1 (d, *J* = 10.4 Hz), 124.8 (d, *J* = 18.2 Hz), 116.3 (d, *J* = 25.8 Hz), 64.2, 52.8, 33.6, 32.6, 7.8; HRMS (*m/z*): Calcd for C₂₀H₁₈F₂N₂O₇: 436.1082; Found: 436.1074.

Methyl 12-Methyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b,g*]bicyclo[3.3.1]nonane-6-carboxylate (**1a**)

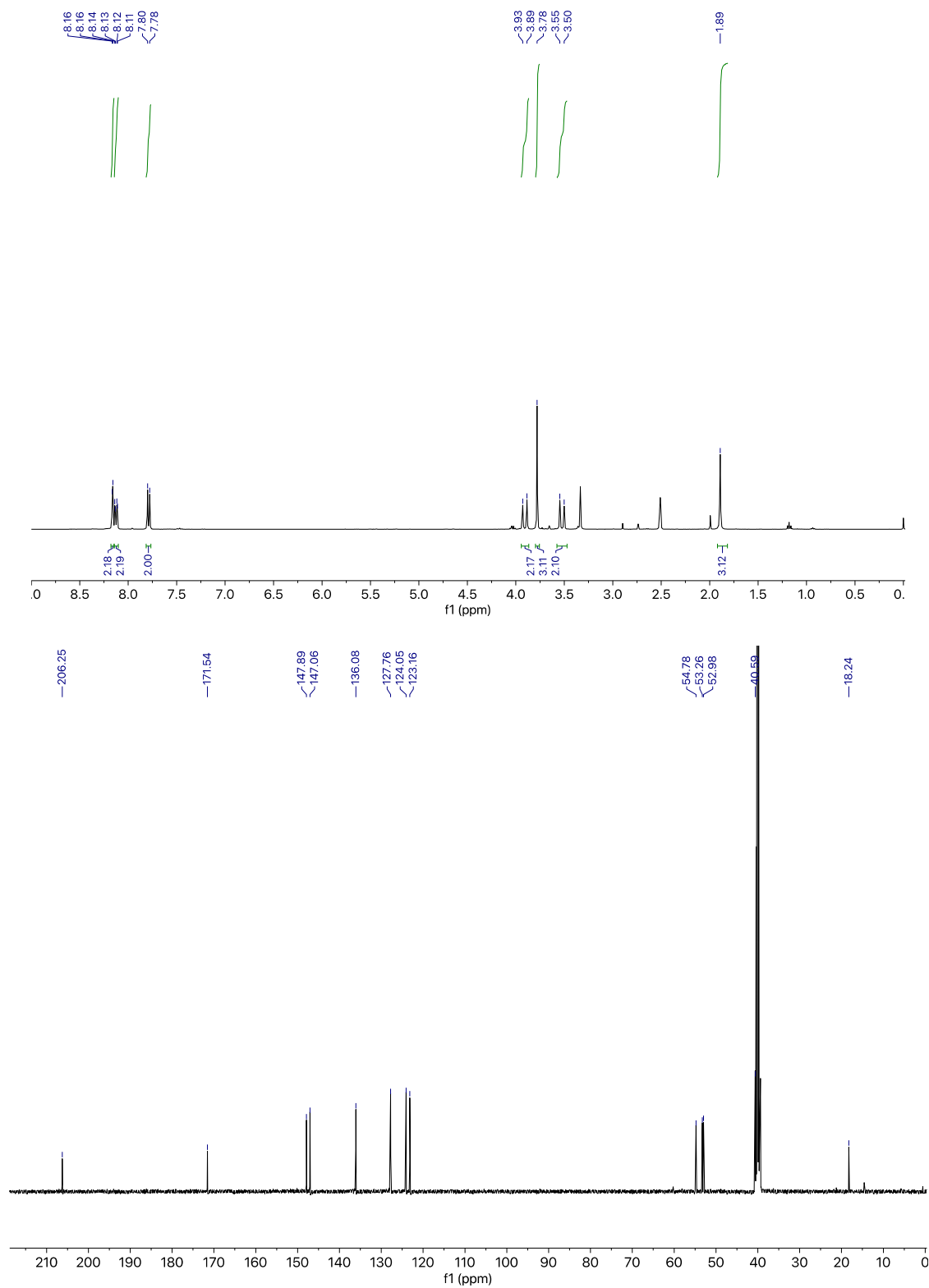


Yield: 104 mg (82%) as a yellow solid, m.p. 267-268 °C; IR: 1739, 1732, 1530, 1351 cm⁻¹; ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.16 (d, *J* = 2.7 Hz, 1H), 8.12 (dd, *J* = 8.8, 2.7 Hz, 2H), 7.79 (d, *J* = 8.8 Hz, 2H), 3.90 (ABd, *J* = 16.2 Hz, 2H), 3.77 (s, 3H), 3.52 (ABd, *J* = 16.2 Hz, 2H), 1.89 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆): δ 206.3, 171.5, 147.9, 147.1, 136.1, 127.8, 124.1, 123.2, 54.8, 53.3, 53.0, 40.6, 18.2; HRMS (*m/z*): Calcd for C₂₀H₁₆N₂O₇: 396.0958; Found: 396.0955. Anal. Calcd for C₂₀H₁₆N₂O₇: C, 60.61; H, 4.07; N, 7.07, Found: C, 60.47; H, 4.11; N, 6.98.

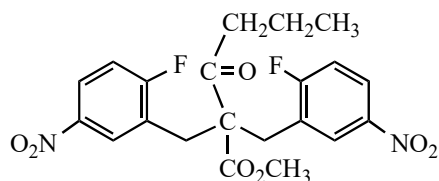
Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxohexanoate (**5a**)



Methyl 12-Methyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b,g*]bicyclo[3.3.1]nonane-6-carboxylate (**1a**)

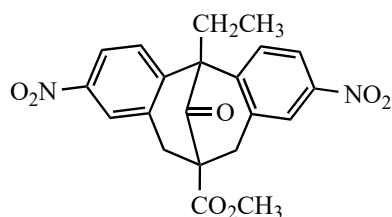


Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxohexanoate (**5b**)



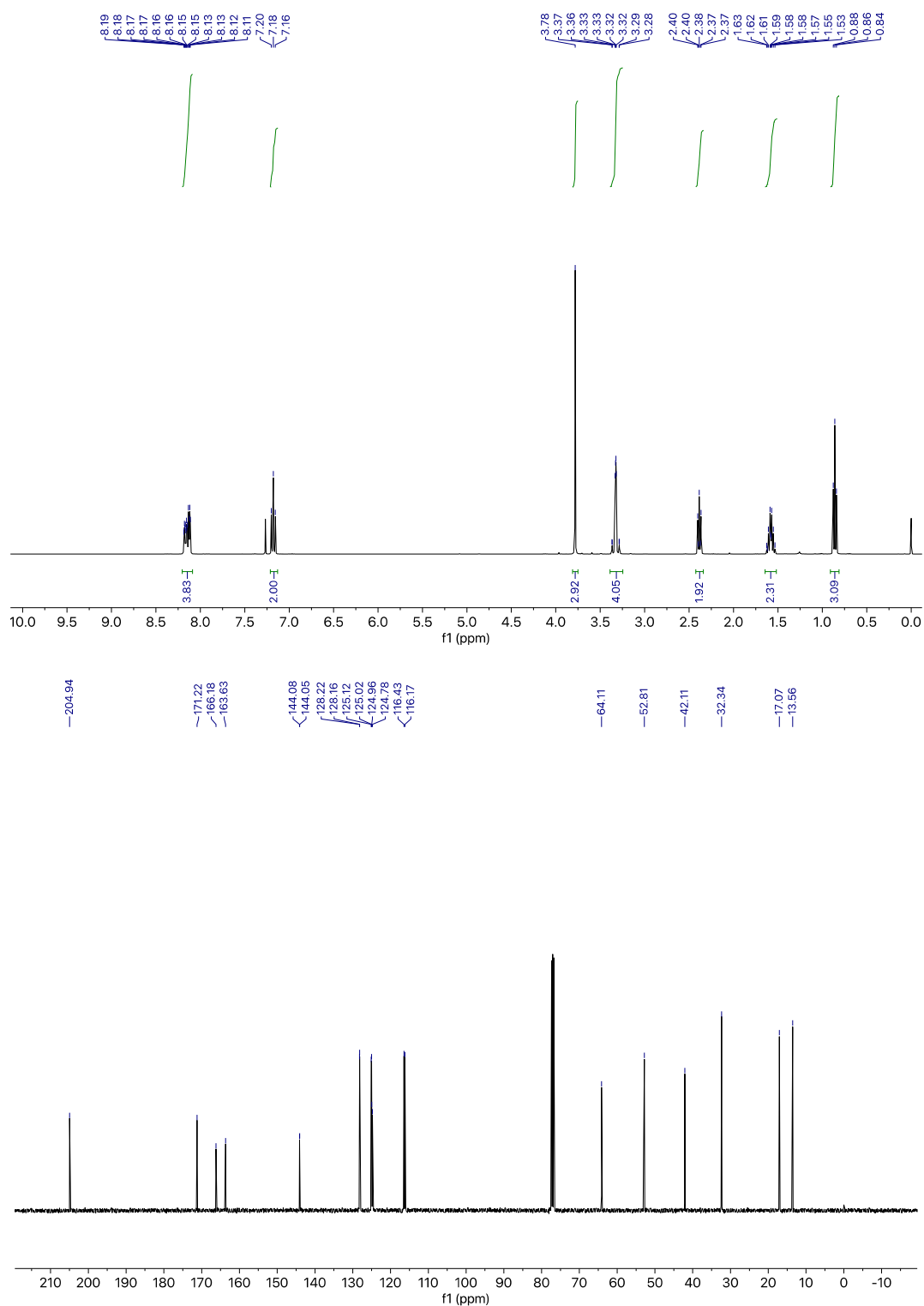
Yield: 173 mg (77%) as a light yellow waxy solid, m.p. 83-84 °C; IR: 1735, 1710, 1532, 1351 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.16 (m, 2H), 8.12 (dd, *J* = 6.3, 2.9 Hz, 2H), 7.18 (t, *J* = 8.9 Hz, 2H), 3.78 (s, 3H), 3.332 (ABd, *J* = 14.4 Hz, 2H), 3.327 (ABd, *J* = 14.4 Hz, 2H), 2.38 (t, *J* = 7.4 Hz, 2H), 1.58 (pentet, *J* = 7.4 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 204.9, 171.2, 164.9 (d, *J* = 256.8 Hz), 144.1 (d, *J* = 2.9 Hz), 128.2 (d, *J* = 6.1 Hz), 125.1 (d, *J* = 10.4 Hz), 124.9 (d, *J* = 17.7 Hz), 116.3 (d, *J* = 25.9 Hz), 64.1, 52.8, 42.1, 32.3, 17.1, 13.6; HRMS (*m/z*): Calcd for C₂₁H₂₀F₂N₂O₇: 450.1239; Found: 450.1233.

Methyl 12-Ethyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b,g*]bicyclo[3.3.1]nonane-6-carboxylate (**1b**)

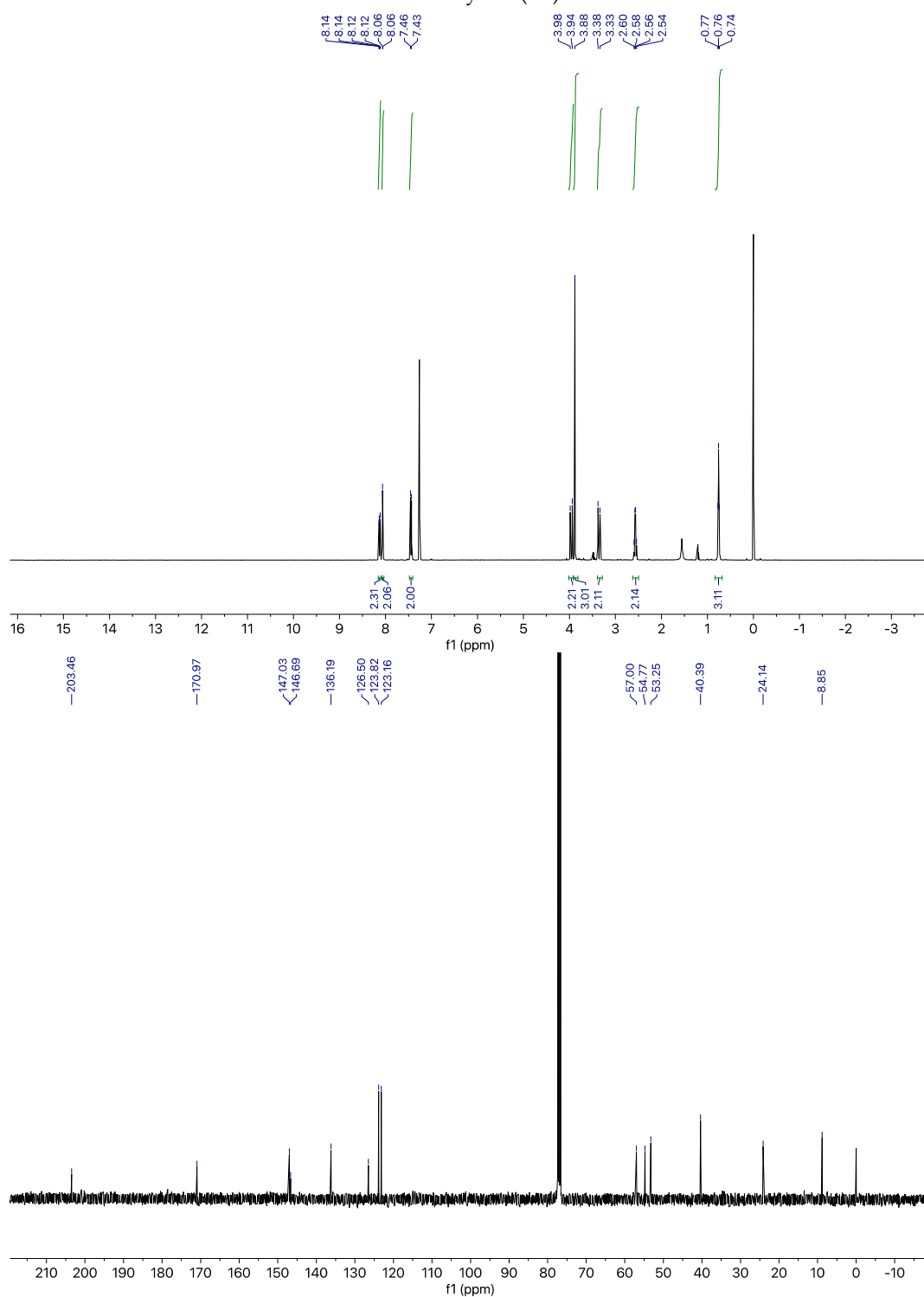


Yield: 128 mg (81%) as a light yellow solid, m.p. 238-239 °C; IR: 1748, 1719, 1528, 1352 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.13 (dd, *J* = 8.7, 2.4 Hz, 2H), 8.06 (d, *J* = 2.4 Hz, 2H), 7.45 (d, *J* = 8.7 Hz, 2H), 3.96 (ABd, *J* = 17.4 Hz, 2H), 3.88 (s, 3H), 3.36 (ABd, *J* = 17.4 Hz, 2H), 2.57 (q, *J* = 7.2 Hz, 2H), 0.76 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 203.5, 171.0, 147.0, 146.7, 136.2, 126.5, 123.8, 123.2, 57.0, 54.8, 53.3, 40.4, 24.1, 8.9; HRMS (*m/z*): Calcd for C₂₁H₁₈N₂O₇: 410.1114; Found: 410.1108.

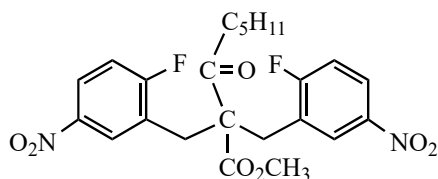
Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxohexanoate (**5b**)



Methyl 12-Ethyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1b**)

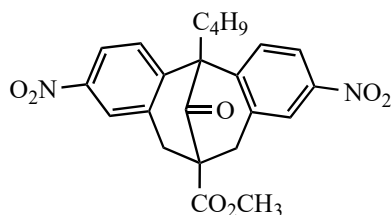


Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxooctanoate (**5c**)



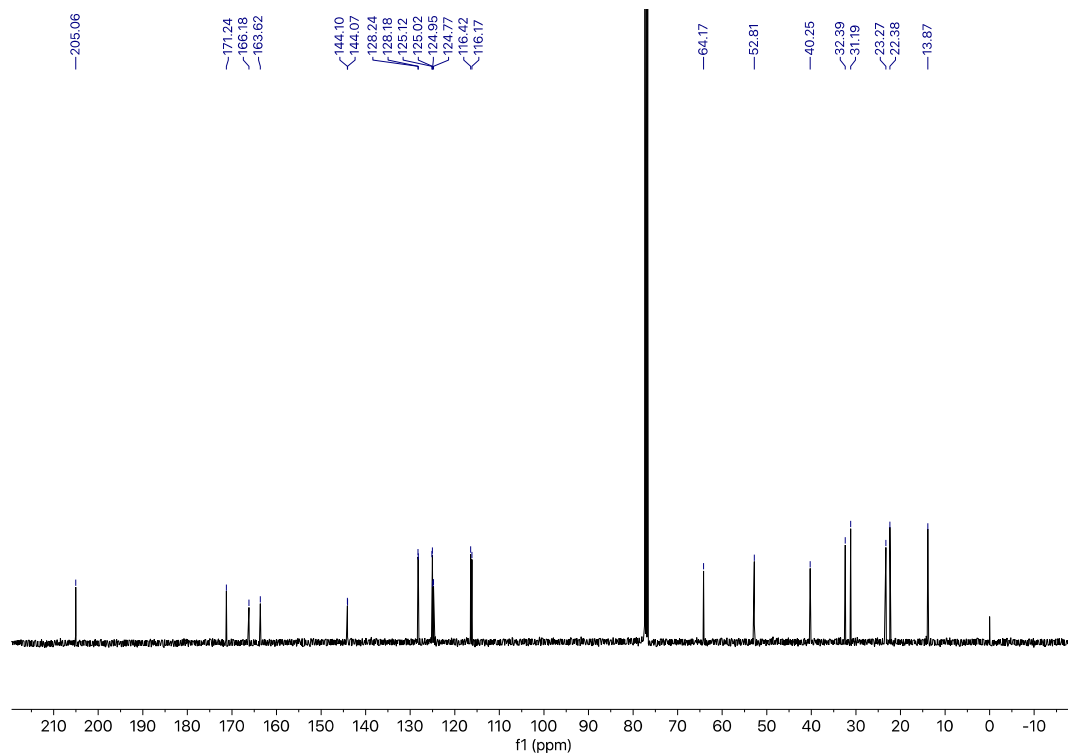
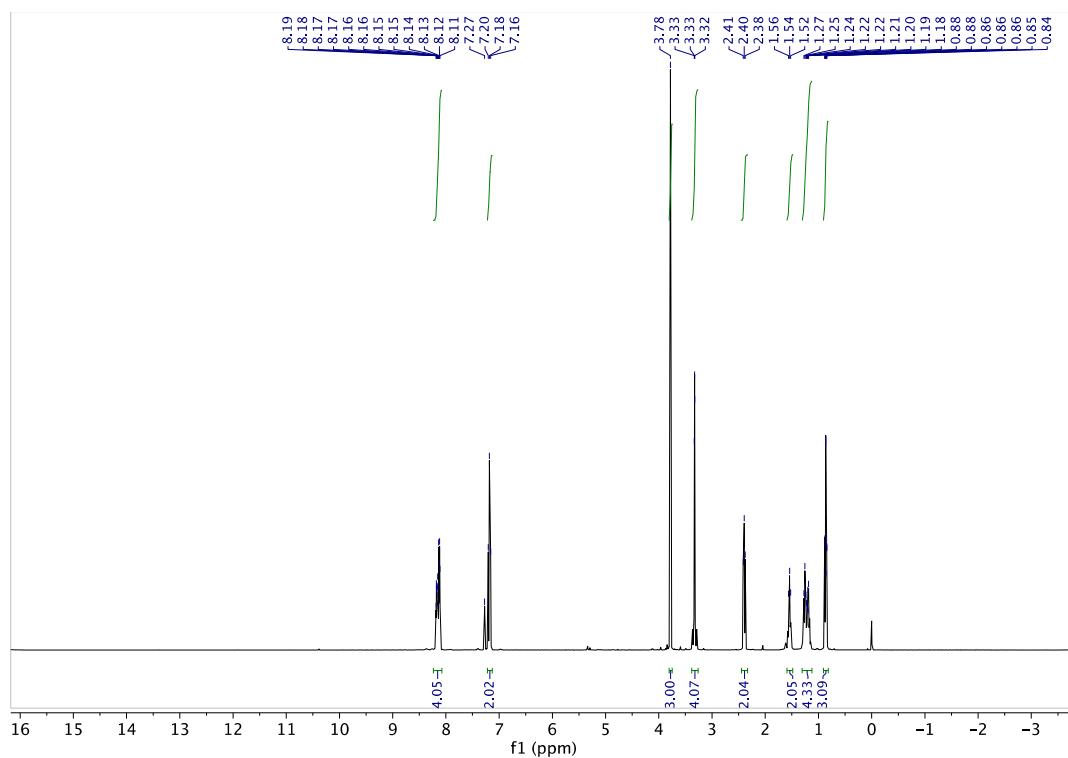
Yield: 153 mg (64%) as a light yellow solid, m.p. 96-98 °C; IR: 1735, 1710, 1532, 1351 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.16 (m, 2H), 8.12 (dd, J = 6.3, 2.9 Hz, 2H), 7.18 (t, J = 8.9 Hz, 2H), 3.78 (s, 3H), 3.332 (ABd, J = 1.9 Hz, 2H), 3.327 (ABd, J = 1.9 Hz, 2H), 2.40 (t, J = 7.5 Hz, 2H), 1.54 (pentet, J = 7.5 Hz, 2H), 1.30-1.15 (complex, 4H), 0.86 (t, J = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 205.1, 171.2, 164.9 (d, J = 256.8 Hz), 144.1 (d, J = 2.9 Hz), 128.2 (d, J = 6.2 Hz), 125.2 (d, J = 10.5 Hz), 124.9 (d, J = 17.8 Hz), 116.3 (d, J = 25.8 Hz), 64.2, 52.8, 40.3, 32.4, 31.2, 23.3, 22.4, 13.9; HRMS (m/z): Calcd for $\text{C}_{23}\text{H}_{24}\text{F}_2\text{N}_2\text{O}_7$: 478.1552; Found: 478.1559.

Methyl 12-Butyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1c**)

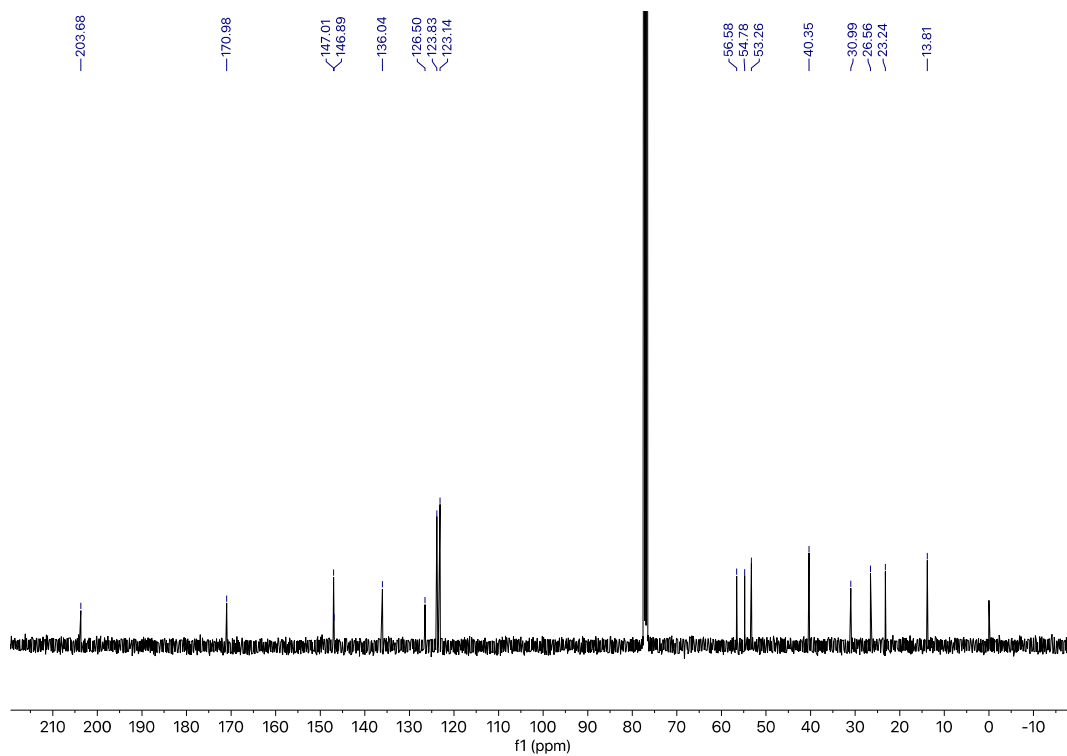
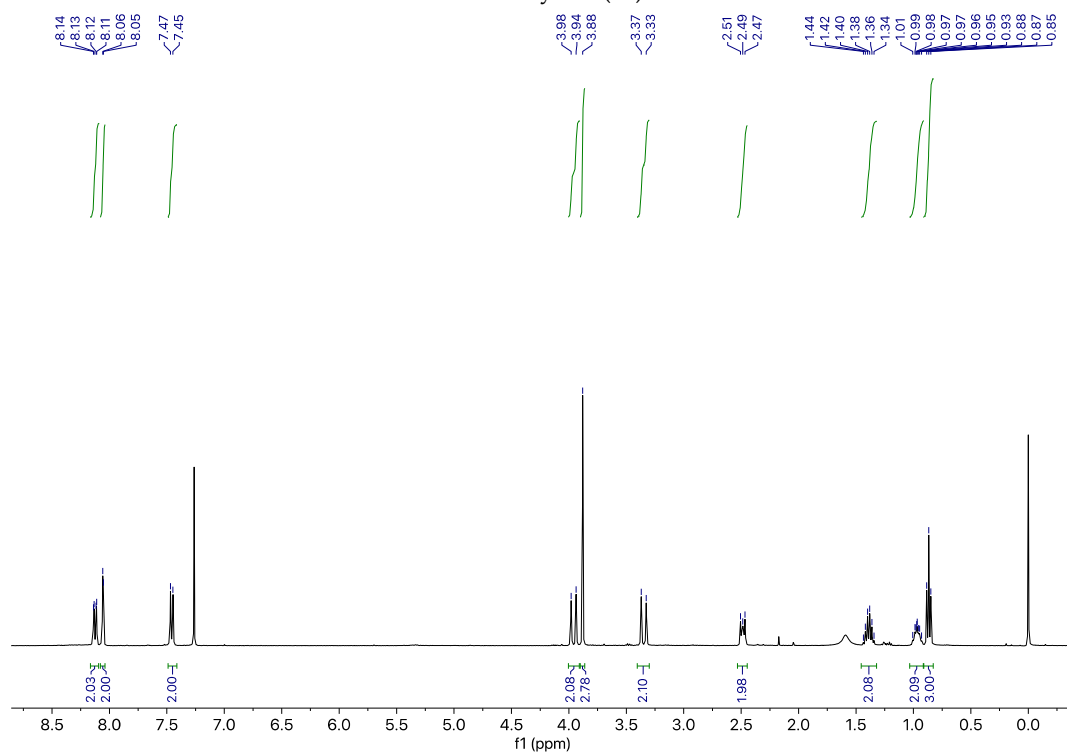


Yield: 122 mg (87%) as a tan solid, m.p. 209-210 °C; IR: 1744, 1712, 1532, 1348 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.13 (dd, J = 8.7, 2.4 Hz, 2H), 8.06 (d, J = 2.4 Hz, 2H), 7.46 (d, J = 8.7 Hz, 2H), 3.92 (ABd, J = 17.4 Hz, 2H), 3.88 (s, 3H), 3.35 (ABd, J = 17.4 Hz, 2H), 2.49 (m, 2H), 1.39 (sextet, J = 7.4 Hz, 2H), 0.96 (m, 2H), 0.87 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 203.7, 171.0, 147.0, 146.9, 136.1, 126.5, 123.8, 123.1, 56.6, 54.8, 53.3, 40.3, 31.0, 26.6, 23.2, 13.8; HRMS (m/z): Calcd for $\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_7$: 438.1427; Found: 438.1418.

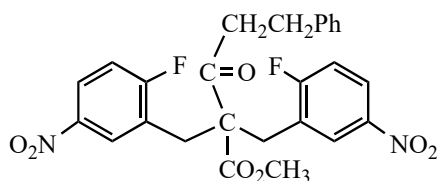
Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxooctanoate (**5c**)



Methyl 12-Butyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1c**)

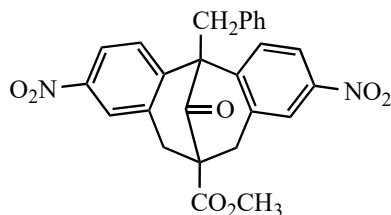


Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxo-5-phenylpentanoate (**5d**)



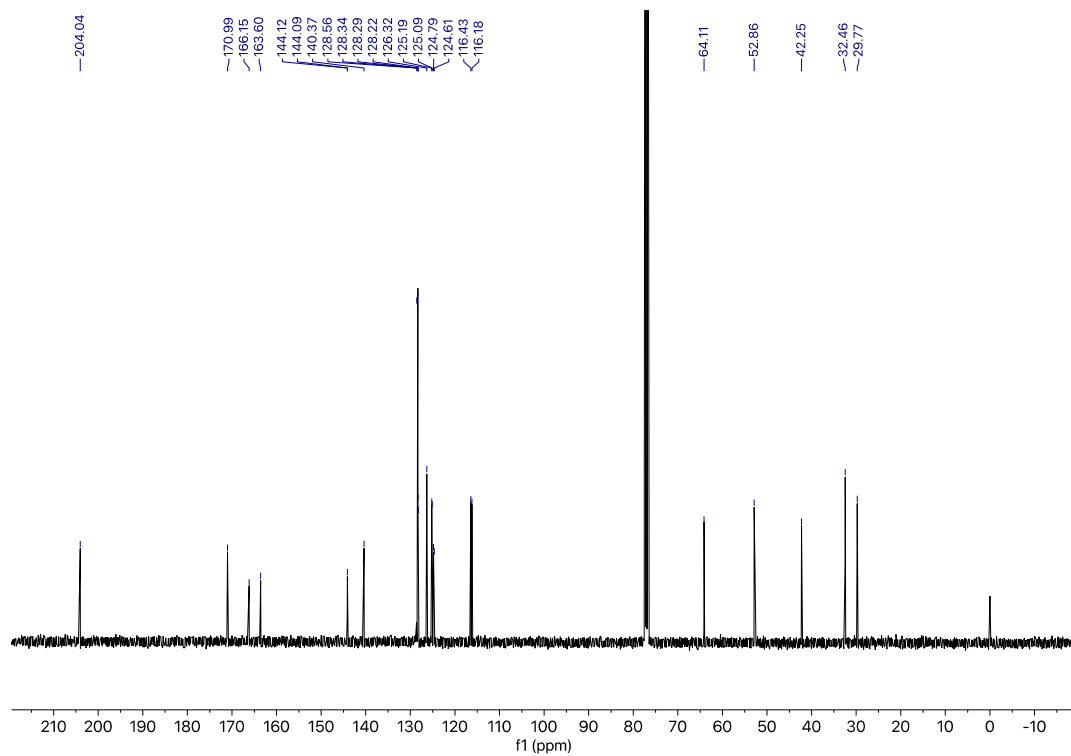
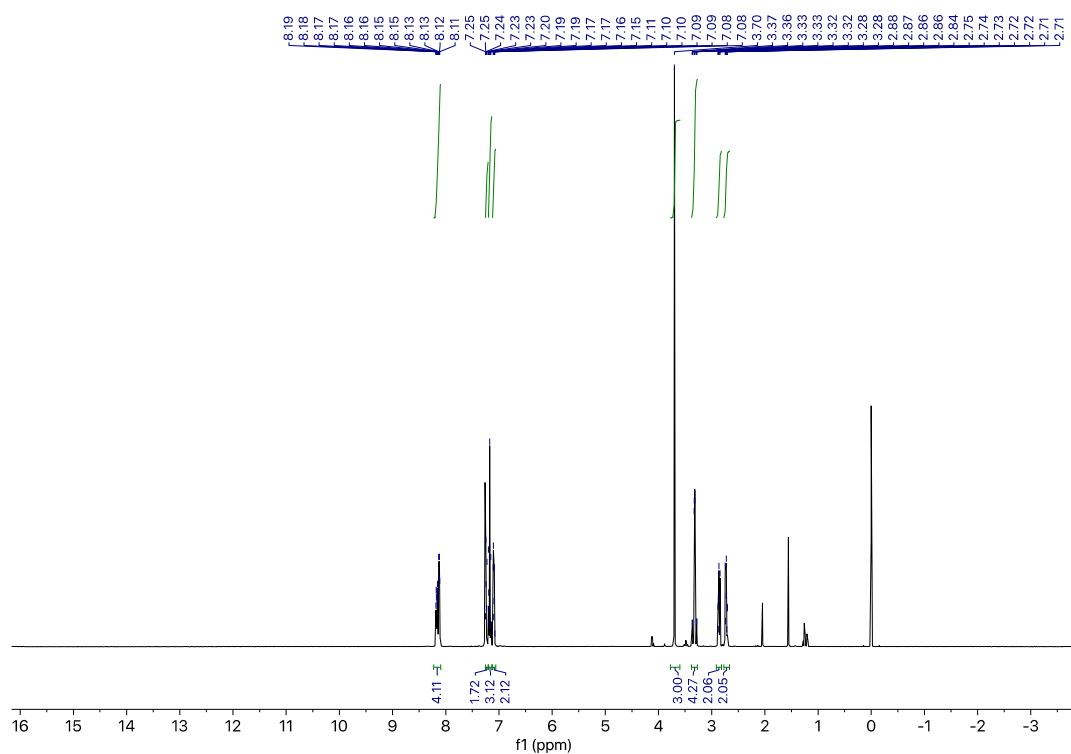
Yield: 166 mg (65%) as a light yellow solid, m.p. 151-152 °C; IR 1746, 1716, 1531, 1349 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.16 (m, 2H), 8.12 (dd, *J* = 6.4, 2.8 Hz, 2H), 7.24 (t, *J* = 7.7 Hz, 2H), 7.17 (overlapping t, *J* = 9.0 Hz, 2H and obscured aromatic, 1H), 7.10 (d, *J* = 7.6 Hz, 2H), 3.70 (s, 3H), 3.33 (ABd, *J* = 14.4 Hz, 2H), 3.30 (ABd, *J* = 14.4 Hz, 2H), 2.87 (t, *J* = 8.0 Hz, 2H), 2.73 (t, *J* = 8.0 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃): δ 204.0, 171.0, 164.9 (d, *J* = 256.6 Hz), 144.1 (d, *J* = 2.9 Hz), 140.4, 128.6, 128.4, 128.3 (d, *J* = 5.7 Hz), 126.3, 125.1 (d, *J* = 10.4 Hz), 124.7 (d, *J* = 17.7 Hz), 116.3 (d, *J* = 25.9 Hz), 64.1, 52.9, 42.3, 32.5, 29.8; HRMS (*m/z*): Calcd for C₂₆H₂₂F₂N₂O₇: 512.1395; Found: 512.1381.

Methyl 12-Benzyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1d**)

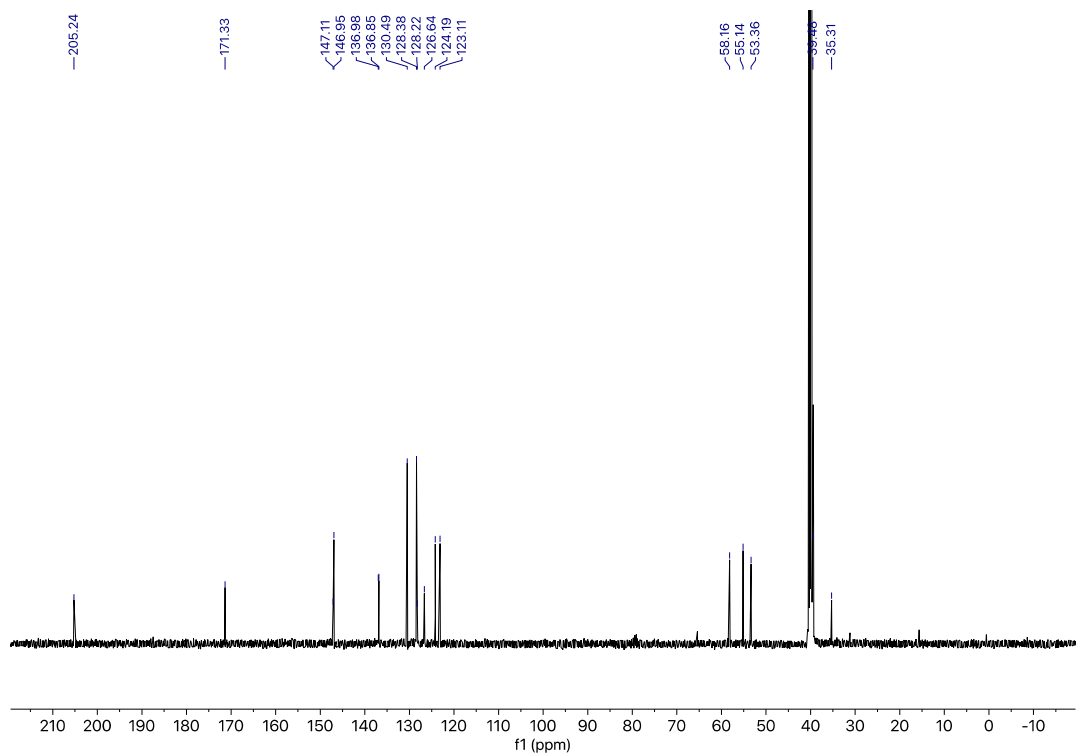
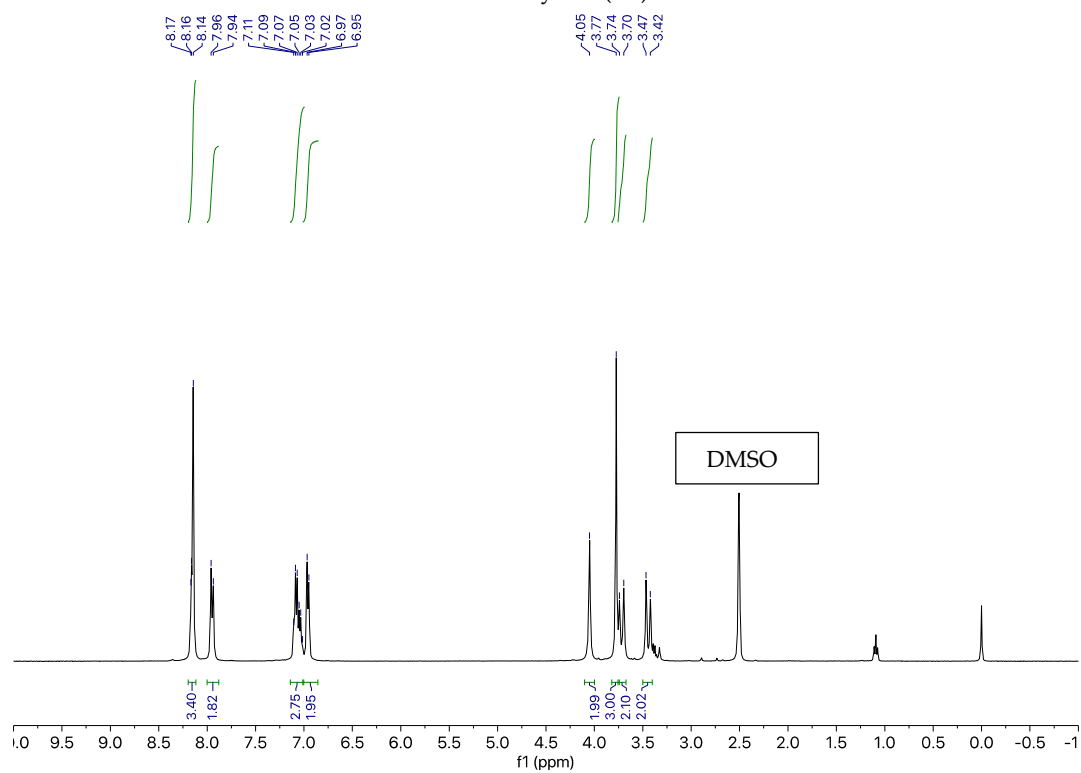


Yield: 130 mg (85%) as a light yellow solid, m.p. 240-241 °C; IR 1745, 1718, 1546, 1349 cm⁻¹; ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.16 (overlapping aromatic, 4H), 7.95 (d, *J* = 9.2 Hz, 2H), 7.15-7.01 (complex, 3H), 6.96 (d, *J* = 7.4 Hz, 2H), 4.05 (s, 2H), 3.77 (s, 3H), 3.72 (ABd, *J* = 17.8 Hz, 2H), 3.45 (ABd, *J* = 17.8 Hz, 2H); ¹³C NMR (101 MHz, DMSO-*d*₆): δ 205.2, 171.3, 147.1, 147.0, 137.0, 136.9, 130.5, 128.4, 128.2, 126.6, 124.2, 123.1, 58.2, 55.1, 53.4, 39.5, 35.3; HRMS (*m/z*): Calcd for C₂₆H₂₀N₂O₇: 472.1271; Found: 472.1266,

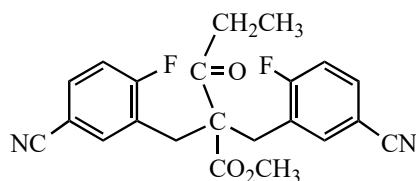
Methyl 2,2-Bis(2-fluoro-5-nitrobenzyl)-3-oxo-5-phenylpentanoate (**5d**)



Methyl 12-Benzyl-3,9-dinitro-5,6,7,12-tetrahydro-13-oxodibenzo[*b,g*]bicyclo[3.3.1]nonane-6-carboxylate (**1d**)

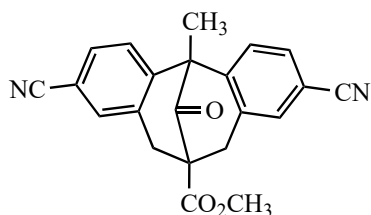


Methyl 2-Bis(5-cyano-2-fluorobenzyl)-3-oxopentanoate (**5e**)



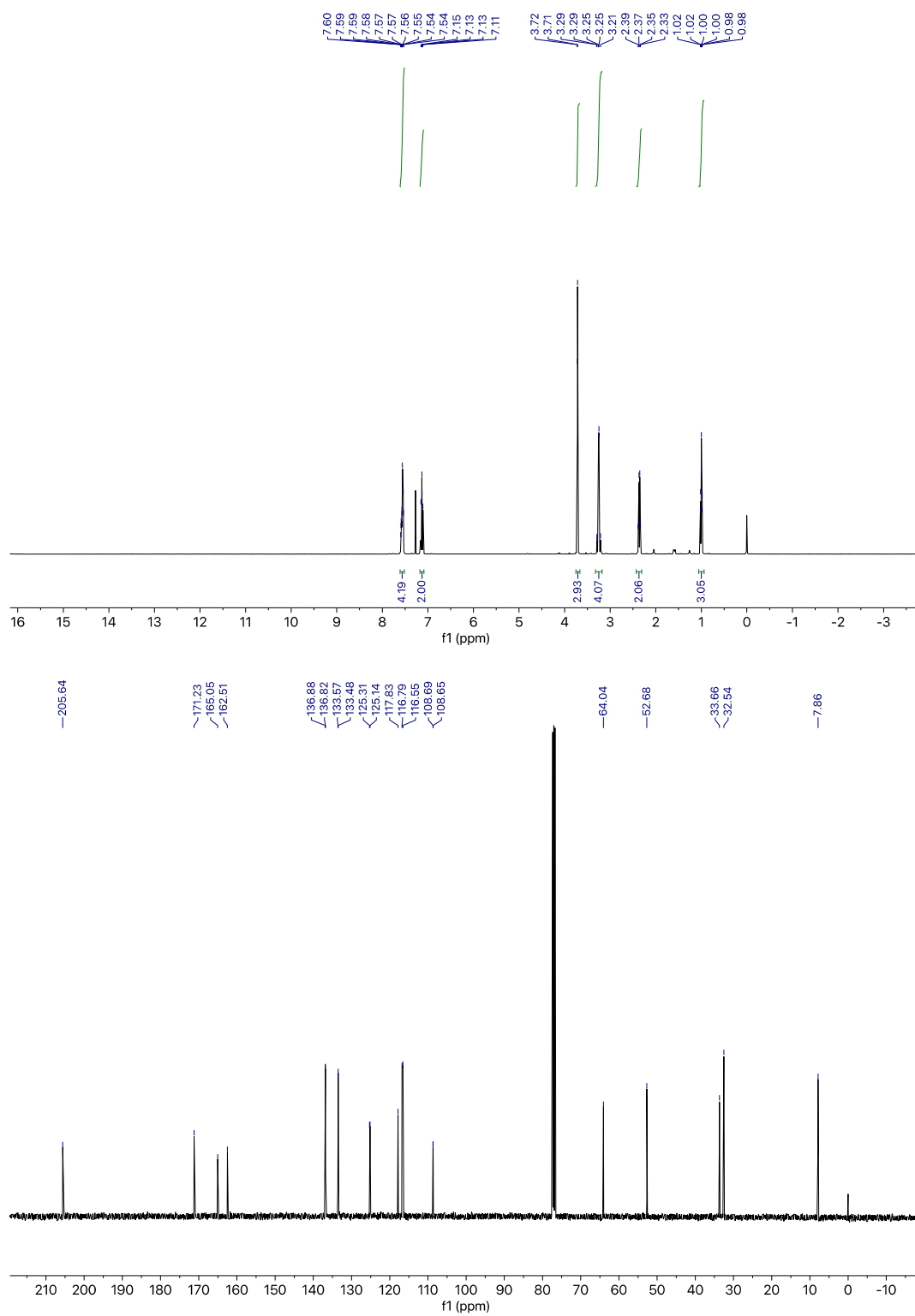
Yield: 162 mg (82%) as a white solid, m.p. 103-104 °C; IR: 2237, 1745, 1717 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.58 (m, 2H), 7.54 (dd, J = 6.9, 2.2 Hz, 2H), 7.13 (t, J = 8.8 Hz, 2H), 3.72 (s, 3H), 3.27 (ABd, J = 14.4 Hz, 2H), 3.23 (ABd, J = 14.4 Hz, 2H), 2.36 (q, J = 7.1 Hz, 2H), 1.00 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 205.6, 171.2, 163.8 (d, J = 255.6 Hz), 136.9 (d, J = 5.5 Hz), 133.5 (d, J = 9.9 Hz), 125.2 (d, J = 16.9 Hz), 117.8, 116.8 (d, J = 24.7 Hz), 108.7 (d, J = 3.8 Hz), 64.0, 52.7, 33.7, 32.5, 7.9; HRMS (m/z): Calcd for $\text{C}_{22}\text{H}_{18}\text{F}_2\text{N}_2\text{O}_3$: 396.1285; Found: 396.1274.

Methyl 12-Methyl-3,9-dicyano-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1e**)

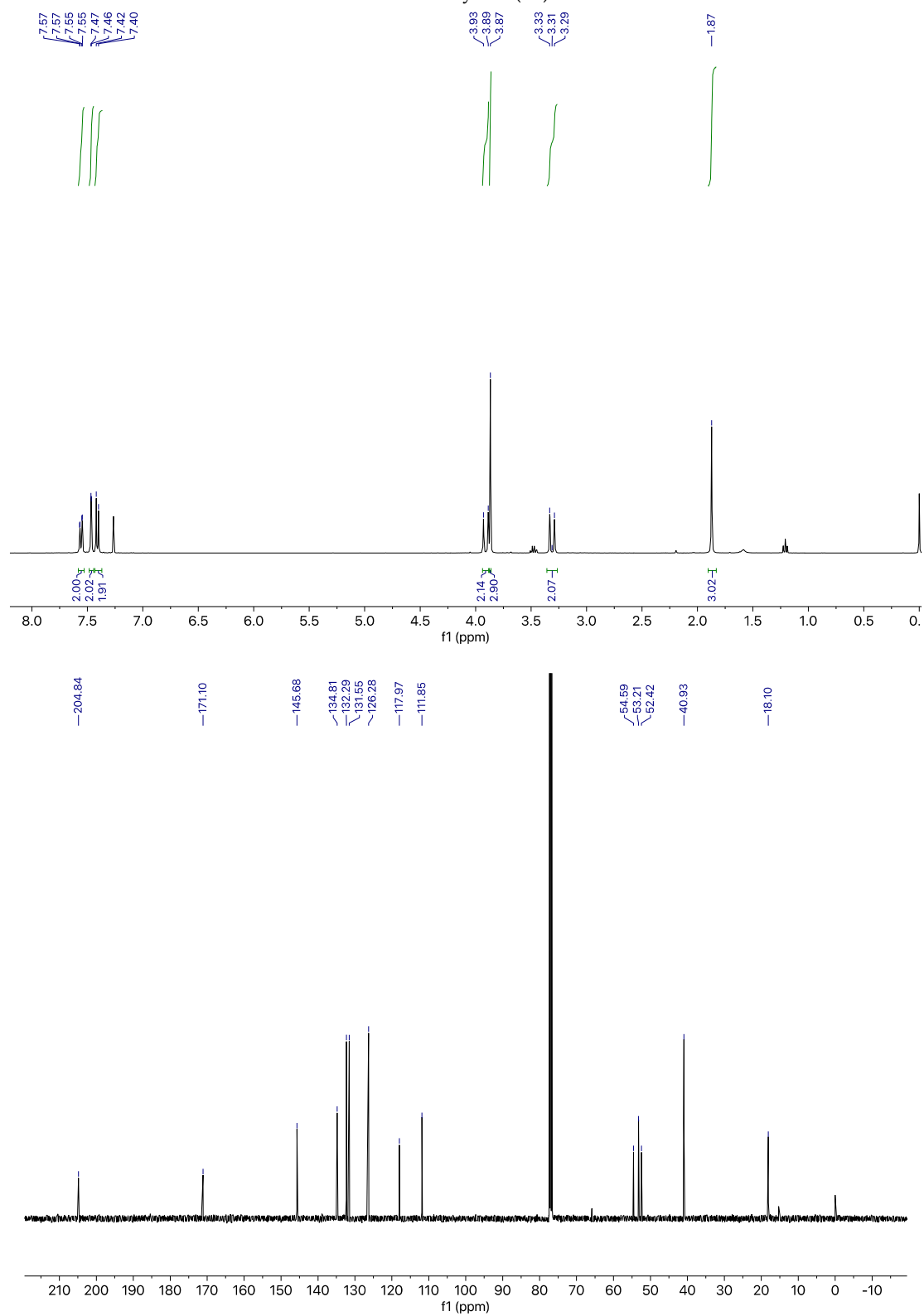


Yield: 105 mg (72%) as an off-white solid, m.p. 243-245 °C; IR: 2233, 1749, 1732 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.56 (dd, J = 8.2, 1.7 Hz, 2H), 7.47 (d, J = 1.7 Hz, 2H), 7.41 (d, J = 8.2 Hz, 2H), 3.91 (ABd, J = 17.4 Hz, 2H), 3.87 (s, 3H), 3.31 (ABd, J = 17.4 Hz, 2H), 1.87 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 204.8, 171.1, 145.9, 134.8, 132.3, 131.6, 126.3, 118.0, 111.9, 54.6, 53.2, 52.4, 40.9, 18.1; HRMS (m/z): Calcd for $\text{C}_{22}\text{H}_{16}\text{N}_2\text{O}_3$: 356.1161; Found: 356.1157.

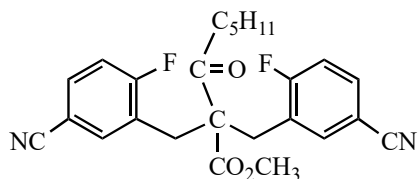
Methyl 2-Bis(5-cyano-2-fluorobenzyl)-3-oxopentanoate (**5e**)



Methyl 12-Methyl-3,9-dicyano-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1e**)

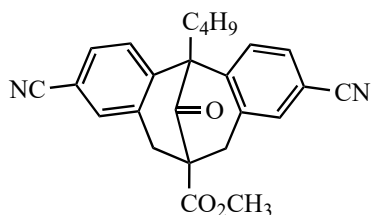


Methyl 2-Bis(5-cyano-2-fluorobenzyl)-3-oxooctanoate (**5f**)



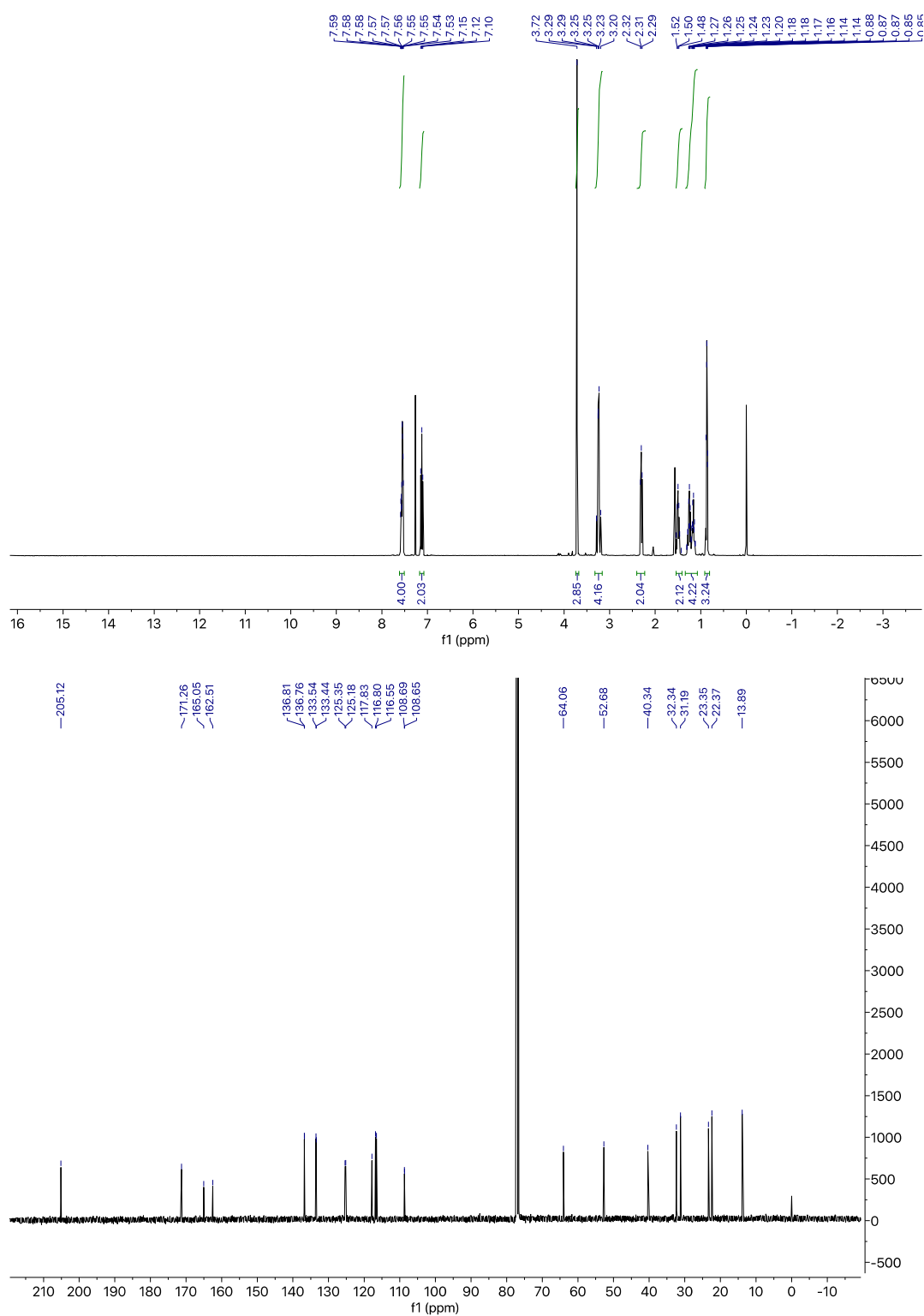
Yield: 142 mg (65%) as a colorless oil; IR: 2237, 1744, 1715 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.58 (m, 2H), 7.54 (dd, J = 6.9, 2.2 Hz, 2H), 7.13 (t, J = 8.9 Hz, 2H), 3.72 (s, 3H), 3.27 (ABd, J = 14.2 Hz, 2H), 3.22 (ABd, J = 14.2 Hz, 2H), 2.31 (t, J = 7.4 Hz, 2H), 1.50 (pentet, J = 7.4 Hz, 2H), 1.25 (m, 2H), 1.16 (m, 2H), 0.86 (t, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 205.1, 171.3, 163.8 (d, J = 255.6 Hz), 136.8 (d, J = 5.5 Hz), 133.4, 125.3 (d, J = 16.9 Hz), 117.8, 116.7 (d, J = 24.8 Hz), 108.7 (d, J = 3.8 Hz), 64.1, 52.7, 40.3, 32.3, 31.2, 23.4, 22.4, 13.9; HRMS (m/z): Calcd for $\text{C}_{25}\text{H}_{24}\text{F}_2\text{N}_2\text{O}_3$: 438.1755; Found: 438.1746.

Methyl 12-Butyl-3,9-dicyano-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1f**)

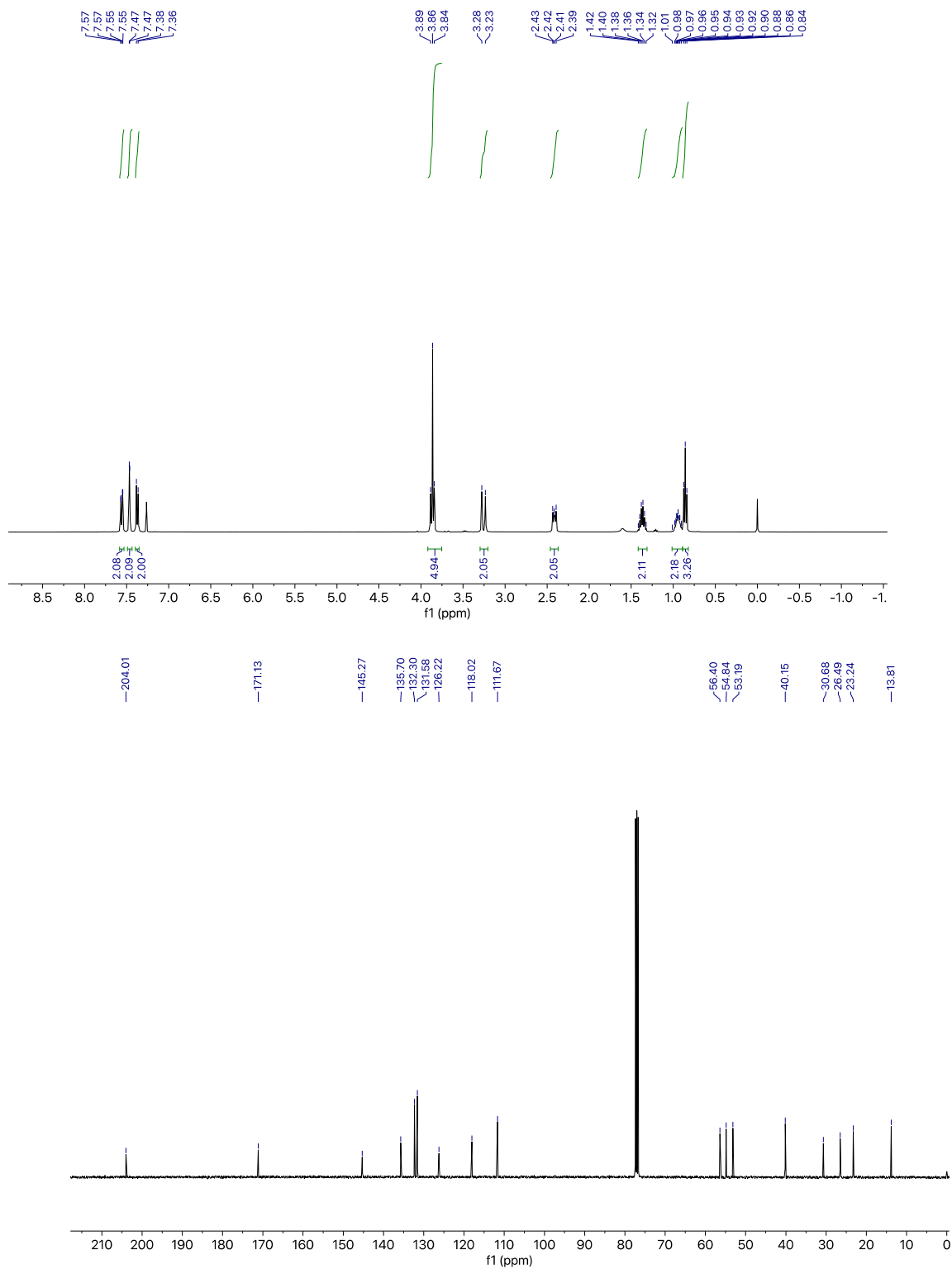


Yield: 106 mg (82%) as a tan solid, m.p. 190-191 $^{\circ}\text{C}$; IR: 2237, 1744, 1715 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.56 (dd, J = 8.2, 1.7 Hz, 2H), 7.47 (d, J = 1.7 Hz, 2H), 7.38 (d, J = 8.2 Hz, 2H), 3.87 (ABd, J = 17.3 Hz, 2H), 3.86 (s, 3H), 3.26 (ABd, J = 17.3 Hz, 2H), 2.41 (m, 2H), 1.36 (sextet, J = 7.4 Hz, 2H), 0.95 (m, 2H), 0.86 (t, J = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 204.0, 171.1, 145.3, 135.7, 132.3, 131.6, 126.2, 118.0, 111.7, 56.4, 55.0, 53.2, 40.2, 30.7, 26.5, 23.2, 13.8; HRMS (m/z): Calcd for $\text{C}_{25}\text{H}_{22}\text{N}_2\text{O}_3$: 398.1630; Found: 398.1623.

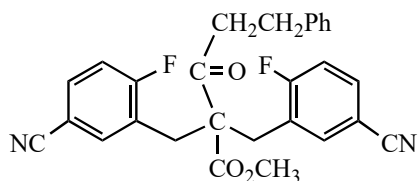
Methyl 2-Bis(5-cyano-2-fluorobenzyl)-3-oxooctanoate (**5f**)



Methyl 12-Butyl-3,9-dicyano-5,6,7,12-tetrahydro-13-oxodibenzo[*b,g*]bicyclo[3.3.1]nonane-6-carboxylate (**1f**)

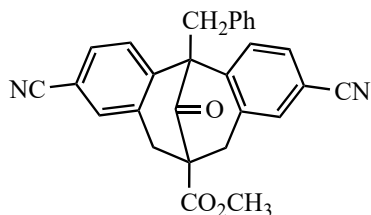


Methyl 2-Bis(5-cyano-2-fluorobenzyl)-3-oxo-5-phenylpentanoate (**5g**)



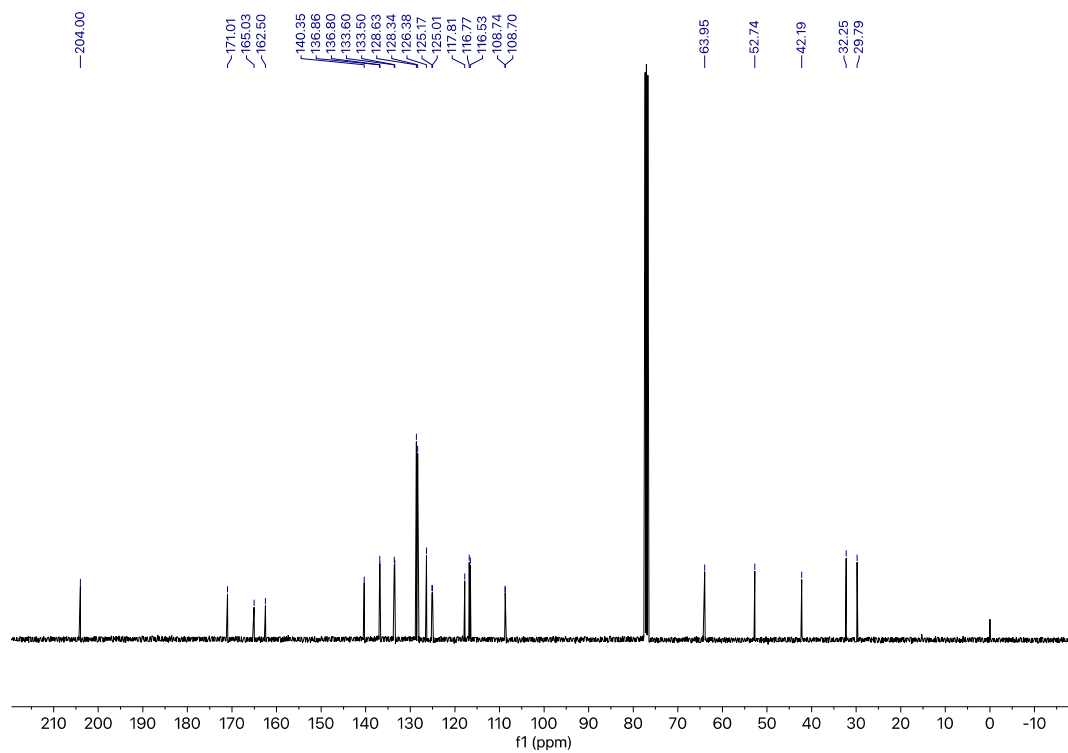
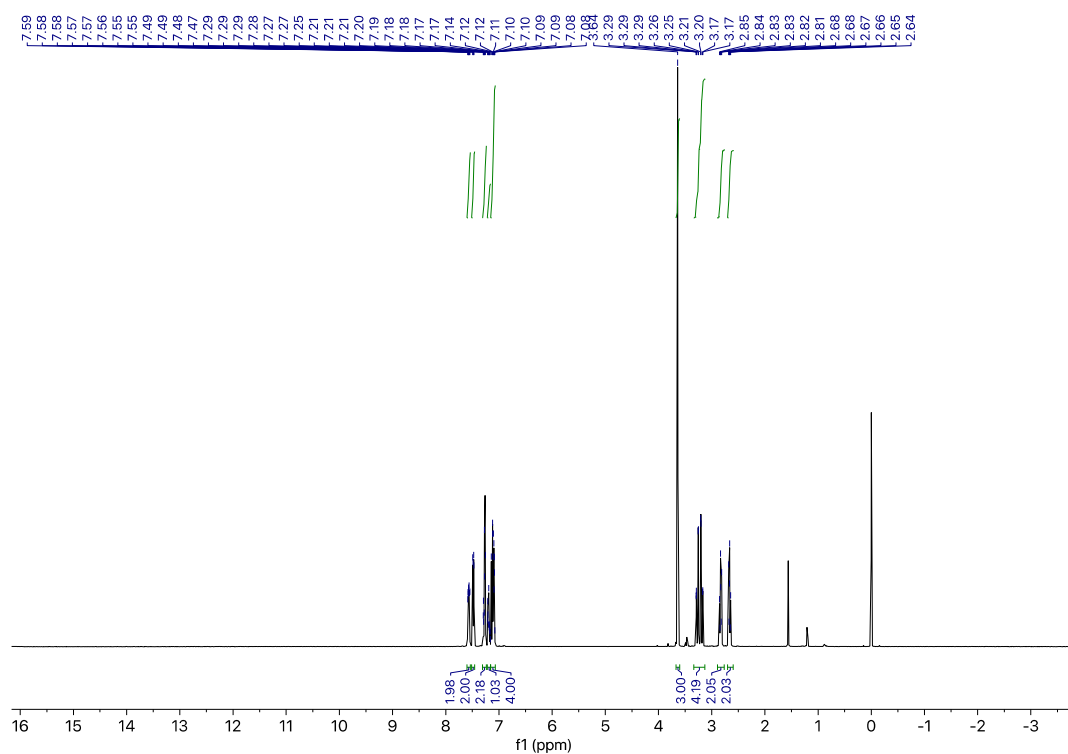
Yield: 189 mg (80%) as an off-white solid, m.p. 120-121 °C; IR: 2236, 1744, 1714 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.57 (ddd, J = 8.6, 4.8, 2.2 Hz, 2H), 7.48 (dd, J = 6.9, 2.2 Hz, 2H), 7.30-7.24 (complex, 2H), 7.19 (tt, J = 7.4, 1.4 Hz, 1H), 7.12 (t, J = 8.6 Hz, 2H), 7.10 (d, J = 7.6 Hz, 1H), 3.64 (s, 3H), 3.28 (ABd, J = 14.8 Hz, 2H), 3.19 (ABd, J = 14.8 Hz, 2H), 2.83 (t, J = 7.2 Hz, 2H), 2.66 (t, J = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ 204.0, 171.0, 163.8 (d, J = 255.4 Hz), 140.4, 136.8 (d, J = 5.4 Hz), 133.6 (d, J = 9.9 Hz), 128.6, 128.3, 126.4, 125.1 (d, J = 16.9 Hz), 117.8, 116.7 (d, J = 24.6 Hz), 108.7 (d, J = 3.8 Hz), 64.0, 52.7, 42.2, 32.3, 29.8; HRMS (m/z): Calcd for $\text{C}_{28}\text{H}_{22}\text{F}_2\text{N}_2\text{O}_3$: 472.1598; Found: 472.1605.

Methyl 12-Benzyl-3,9-dicyano-5,6,7,12-tetrahydro-13-oxodibenzo[*b,g*]bicyclo[3.3.1]nonane-6-carboxylate (**1g**)



Yield: 128 mg (74%) as an off-white solid, m.p. 272-274 °C; IR: 2234, 1747, 1714 cm^{-1} ; ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 7.84 (d, J = 8.3 Hz, 2H), 7.79 (dd, J = 8.3, 1.7 Hz, 2H), 7.75 (d, J = 1.7 Hz, 2H), 7.11-7.00 (complex 3 H), 6.92 (d, J = 8.0 Hz, 2H), 3.97 (s, 2H), 3.75 (s, 3H), 3.60 (ABd, J = 17.6 Hz, 2H), 3.32 (ABd, J = 17.6 Hz, 2H); ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$): δ 205.5, 171.4, 145.5, 137.2, 136.2, 133.0, 131.8, 130.5, 128.3, 127.8, 126.6, 118.7, 110.8, 58.0, 55.2, 53.3, 35.0 (one aliphatic C obscured); HRMS (m/z): Calcd for $\text{C}_{28}\text{H}_{20}\text{N}_2\text{O}_3$: 432.1474; Found: 432.1468.

Methyl 2-Bis(5-cyano-2-fluorobenzyl)-3-oxo-5-phenylpentanoate (5g)



Methyl 12-Benzyl-3,9-dicyano-5,6,7,12-tetrahydro-13-oxodibenzo[*b.g*]bicyclo[3.3.1]nonane-6-carboxylate (**1g**)

