

Supporting Information

Ag-Catalyzed Ring-Opening of Tertiary Cycloalkanols for C-H Functionalization of Cyclic Aldimines

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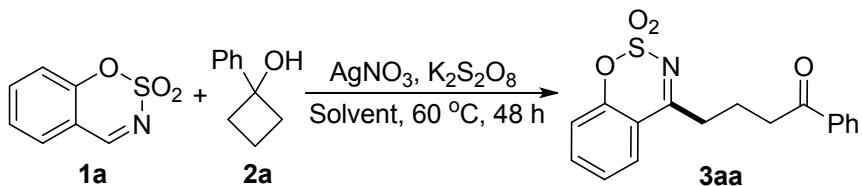
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General information:

¹H, and ¹³C were recorded at Bruker 400 MHz (¹H NMR) and 100 MHz (¹³C NMR). Chemical shifts were reported in ppm from the solvent resonance as the internal standard (CDCl₃: 7.26 ppm, 77.0 ppm). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br (broad). Coupling constants were reported in Hertz (Hz). Infrared spectra were obtained with a AVATAR 360 FT-IR spectrometer. The melting points were measured with a XT-4 melting point apparatus. X-ray structural analysis was conducted on the XtaLAB mini. The high resolution ESI-MS spectra were tested with a Waters Vion IMS QT of high resolution mass spectrometer.

Materials: All commercially available reagents and solvent were used without further purification. Analytical thin layer chromatography was performed on 0.25 mm silica gel plates. Silica gel (200-300 mesh) was used for flash chromatography. Cyclic aldimines were prepared according to the literatures.^[1] Cycloalkanols were prepared according to the literatures.^[2]

Extended optimization table of the ratio of the solvent:

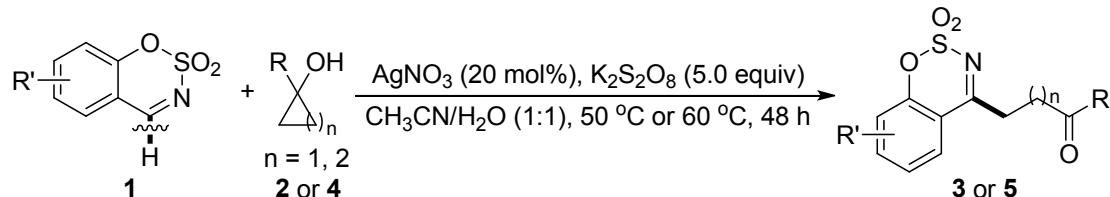


Entry	Solvent (ratio)	Yield (%) ^b
1	CH ₃ CN	40
2	CH ₃ CN/H ₂ O (2:1)	83
3	CH ₃ CN/H ₂ O (3:1)	81
4	CH ₃ CN/H ₂ O (4:1)	80
5	CH ₃ CN/H ₂ O (1:2)	81
6	CH ₃ CN/H ₂ O (1:3)	78
7	CH ₃ CN/H ₂ O (1:4)	75

8	H_2O	10
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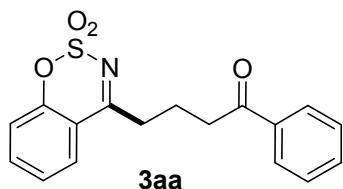
^a Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), AgNO₃ (20 mol%), K₂S₂O₈ (1.0 mmol), solvent (2 mL), 48 h. ^b Isolated yield.

General Procedure for the ring-opening C–H functionalization of cycloalkanols with cyclic aldimines:



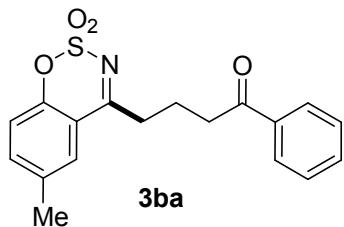
To a 10 mL Schlenk charged with cyclic aldimines **1** (0.2 mmol), AgNO₃ (6.8 mg, 0.04 mmol), K₂S₂O₈ (270 mg, 1.0 mmol) and cycloalkanols **2** or **4** (0.3 mmol) was added CH₃CN (1.0 mL) and distilled H₂O (1.0 mL) *via* a syringe. The reaction mixture was vigorously stirred at 50 °C or 60 °C for 48 h. After the reaction was complete, the mixture was diluted with water (10 mL) and extracted with ethyl acetate (3 × 10 mL). The organic layers were combined and washed with a saturated brine (15 mL), dried anhydrous Mg₂SO₄, and then concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent) to afford the desired products **3** or **5**.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



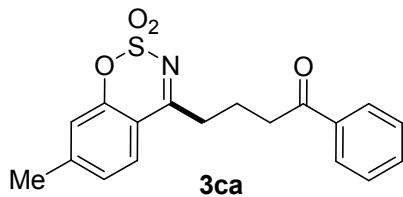
85% yield, mp 72–73 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.25–2.32 (m, 2H), 3.16–3.22 (m, 4H), 7.29 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.46–7.50 (m, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.68–7.72 (m, 1H), 7.95–7.99 (m, 3H); **13C NMR** (100 MHz, CDCl₃) δ 199.3, 179.7, 153.6, 136.9, 136.5, 133.3, 128.7, 128.2, 128.0, 125.9, 119.1, 116.0, 36.8, 35.0, 20.1; **IR** (KBr) ν 2958, 1685, 1650, 1508, 1458, 1385, 1188 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₆NO₄S [M+H]⁺ 330.0795, found 330.0799.

4-(6-methyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



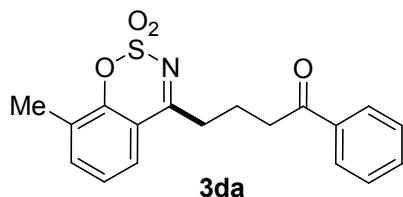
75% yield, mp 100–101 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.24–2.31 (m, 2H), 2.43 (s, 3H), 3.14–3.21 (m, 4H), 7.17 (d, *J* = 8.4 Hz, 1H), 7.45–7.50 (m, 3H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.70 (s, 1H), 7.97–7.99 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 199.3, 179.7, 151.5, 137.6, 136.5, 135.9, 133.3, 128.6, 128.1, 128.0, 118.8, 115.7, 36.8, 34.8, 20.9, 19.9; **IR** (KBr) ν 2918, 1682, 1599, 1562, 1385, 1264, 1189, 827, 738 cm⁻¹; **HRMS** (ESI): calcd for C₁₈H₁₈NO₄S [M+H]⁺ 344.0951, found 344.0952.

4-(7-methyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



68% yield, mp 142–143 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.22–2.29 (m, 2H), 2.46 (s, 3H), 3.13 (t, *J* = 7.6 Hz, 2H), 3.18 (t, *J* = 6.8 Hz, 2H), 7.08 (s, 1H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.45–7.49 (m, 2H), 7.75 (t, *J* = 7.6 Hz, 1H), 7.82 (d, *J* = 8.4 Hz, 1H), 7.95–7.98 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 199.3, 179.6, 153.5, 149.3, 136.5, 133.3, 128.6, 128.0, 127.9, 126.9, 119.2, 113.6, 36.8, 34.8, 22.0, 20.1; **IR** (KBr) ν 2928, 1681, 1561, 1385, 1193, 748 cm⁻¹; **HRMS** (ESI): calcd for C₁₈H₁₈NO₄S [M+H]⁺ 344.0951, found 344.0952.

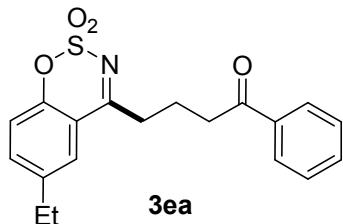
4-(8-methyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



65% yield, mp 110–111 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.23–2.30 (m, 2H), 2.39 (s, 3H), 3.14–3.20 (m, 4H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.53–7.59 (m, 2H), 7.79 (d, *J* = 7.2 Hz, 1H), 7.96–7.98 (m, 2H); **13C NMR** (100 MHz,

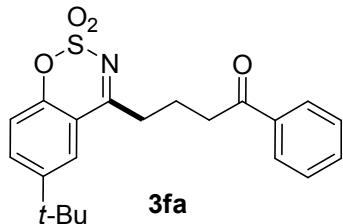
CDCl_3) δ 199.2, 180.1, 151.9, 138.3, 136.5, 133.3, 128.8, 128.6, 127.9, 125.7, 125.1, 115.8, 36.8, 35.0, 20.2, 14.9; **IR** (KBr) ν 2928, 1686, 1594, 1561, 1389, 1199, 1093, 867, 771 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_4\text{S}$ $[\text{M}+\text{H}]^+$ 344.0951, found 344.0952.

4-(6-ethyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



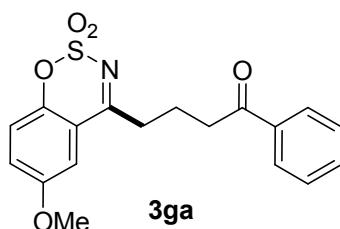
66% yield, mp 79–80 °C; **1H NMR** (400 MHz, CDCl_3) δ 1.26 (t, $J = 7.6$ Hz, 3H), 2.22–2.29 (m, 2H), 7.72 (q, $J = 15.2$ Hz, 7.6 Hz, 2H), 3.15–3.20 (m, 4H), 7.18 (d, $J = 8.4$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 7.52 (dd, $J = 8.4$ Hz, 1.6 Hz, 1H), 7.56 (t, $J = 7.6$ Hz, 1H), 7.73 (s, 1H), 7.97 (d, $J = 7.6$ Hz, 2H); **13C NMR** (100 MHz, CDCl_3) δ 199.2, 179.9, 151.5, 142.2, 136.6, 136.5, 133.2, 128.6, 127.9, 127.0, 118.7, 115.7, 36.8, 34.8, 28.1, 19.9, 15.3; **IR** (KBr) ν 2928, 1681, 1655, 1556, 1385, 1262, 1188, 1096, 802 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{19}\text{H}_{20}\text{NO}_4\text{S}$ $[\text{M}+\text{H}]^+$ 358.1108, found 358.1116.

4-(6-(tert-butyl)-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



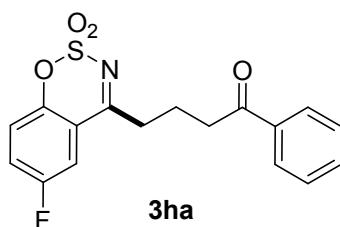
64% yield, mp 98–99 °C; **1H NMR** (400 MHz, CDCl_3) δ 1.37 (s, 9H), 2.24–2.31 (m, 2H), 3.16–3.23 (m, 4H), 7.21 (d, $J = 8.8$ Hz, 1H), 7.46–7.50 (m, 2H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.73 (dd, $J = 8.8$ Hz, 2.4 Hz, 1H), 7.94 (d, $J = 2.4$ Hz, 1H), 7.97–8.00 (m, 2H); **13C NMR** (100 MHz, CDCl_3) δ 199.1, 180.1, 151.3, 149.3, 136.5, 134.4, 133.3, 128.6, 128.0, 124.6, 118.5, 115.4, 36.8, 34.9, 34.8, 31.1, 20.2; **IR** (KBr) ν 2928, 1682, 1598, 1557, 1385, 1191, 844, 787 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{21}\text{H}_{24}\text{NO}_4\text{S}$ $[\text{M}+\text{H}]^+$ 386.1421, found 386.1410.

4-(6-methoxy-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



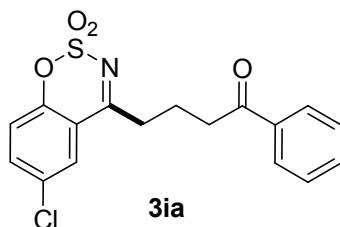
77% yield, mp 135–136 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.23–2.30 (m, 2H), 3.13 (t, *J* = 7.6 Hz, 2H), 3.21 (t, *J* = 6.4 Hz, 2H), 3.93 (s, 3H), 7.19–7.26 (m, 2H), 7.46–7.50 (m, 3H), 7.58 (t, *J* = 7.2 Hz, 1H), 7.98 (d, *J* = 7.6 Hz, 2H); **13C NMR** (100 MHz, CDCl₃) δ 199.2, 179.7, 156.9, 147.3, 136.4, 133.3, 128.6, 127.9, 123.7, 120.0, 116.3, 111.2, 56.1, 36.8, 35.1, 20.2; **IR** (KBr) ν 2926, 1685, 1650, 1560, 1458, 1384, 1186, 833 cm⁻¹; **HRMS** (ESI): calcd for C₁₈H₁₈NO₅S [M+H]⁺ 360.0900, found 360.0889.

4-(6-fluoro-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



68% yield, mp 112–113 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.23–2.30 (m, 2H), 3.14 (t, *J* = 7.6 Hz, 2H), 3.20 (t, *J* = 6.4 Hz, 2H), 7.29 (dd, *J* = 13.2 Hz, 4.4 Hz, 1H), 7.39–7.44 (m, 1H), 7.45–7.49 (m, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.68 (dd, *J* = 8.0 Hz, 2.8 Hz, 1H), 7.95–7.98 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 199.1, 178.8, 159.0 (d, *J*_{C-F} = 246.9 Hz), 149.4, 136.4, 133.3, 128.6, 127.9, 124.1 (d, *J*_{C-F} = 24.0 Hz), 120.9 (d, *J*_{C-F} = 8.0 Hz), 116.7 (d, *J*_{C-F} = 7.8 Hz), 114.3 (d, *J*_{C-F} = 25.0 Hz), 36.6, 35.0, 19.8; **IR** (KBr) ν 2928, 1686, 1655, 1509, 1451, 1189 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₅FNO₄S [M+H]⁺ 348.0700, found 348.0697.

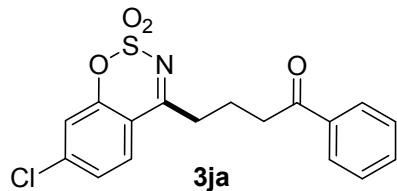
4-(6-chloro-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



83% yield, mp 129–130 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.25–2.31 (m, 2H), 3.14–3.22 (m, 4H), 7.25 (d, *J* = 8.8 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.58 (t, *J* = 7.2 Hz,

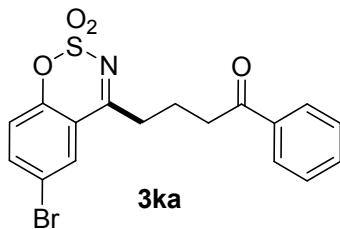
1H), 7.65 (dd, J = 8.8 Hz, 2.4 Hz, 1H), 7.93 (d, J = 2.4 Hz, 1H), 7.97–7.99 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 199.1, 178.6, 151.8, 136.6, 136.4, 133.4, 131.4, 128.6, 128.0, 127.7, 120.6, 116.9, 36.6, 34.9, 19.6; **IR** (KBr) ν 2927, 1681, 1655, 1599, 1556, 1391, 1191, 1101, 798 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{17}\text{H}_{15}\text{ClNO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 364.0405, found 364.0407.

4-(7-chloro-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



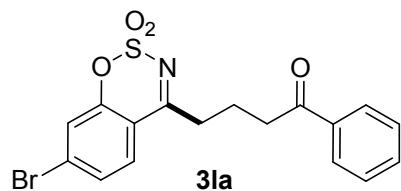
95% yield, mp 120–121 °C; **^1H NMR** (400 MHz, CDCl_3) δ 2.22–2.29 (m, 2H), 3.14 (t, J = 7.6 Hz, 2H), 3.20 (t, J = 6.4 Hz, 2H), 7.31 (d, J = 2.0 Hz, 1H), 7.39 (dd, J = 8.8 Hz, 1.6 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 7.57–7.61 (m, 1H), 7.94–7.99 (m, 3H); **^{13}C NMR** (100 MHz, CDCl_3) δ 199.2, 179.0, 154.0, 143.0, 136.4, 133.4, 129.2, 128.7, 127.9, 126.5, 119.5, 114.4, 36.7, 35.1, 20.1; **IR** (KBr) ν 2928, 1686, 1655, 1560, 1508, 1458, 1189 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{17}\text{H}_{15}\text{ClNO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 364.0405, found 364.0407.

4-(6-bromo-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



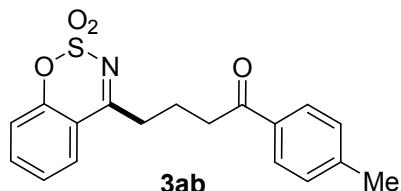
70% yield, mp 146–147 °C; **^1H NMR** (400 MHz, CDCl_3) δ 2.24–2.31 (m, 2H), 3.14–3.22 (m, 4H), 7.18 (d, J = 8.8 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 7.58 (t, J = 7.2 Hz, 1H), 7.79 (dd, J = 8.8 Hz, 2.0 Hz, 1H), 7.98 (d, J = 7.2 Hz, 2H), 8.06 (d, J = 2.4 Hz, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 199.1, 178.5, 152.3, 139.5, 136.4, 133.3, 130.7, 128.6, 127.9, 120.8, 118.6, 117.3, 36.6, 34.8, 19.6; **IR** (KBr) ν 2928, 1686, 1654, 1542, 1458, 1188, 753 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{17}\text{H}_{15}\text{BrNO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 407.9900, found 407.9905.

4-(7-bromo-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-1-one:



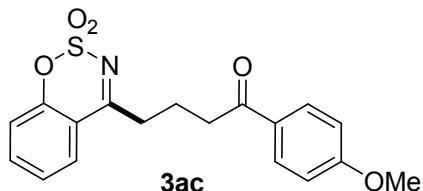
76% yield, mp 149–150 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.24–2.30 (m, 2H), 3.14–3.22 (m, 4H), 7.18 (d, *J* = 8.8 Hz, 1H), 7.47 (t, *J* = 7.2 Hz, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.78 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 7.98 (d, *J* = 7.2 Hz, 2H), 8.06 (d, *J* = 2.0 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 199.1, 178.5, 152.3, 139.5, 136.4, 133.3, 130.7, 128.6, 127.9, 120.8, 118.6, 117.3, 36.6, 34.9, 19.6; **IR** (KBr) ν 2926, 1697, 1650, 1556, 1391, 1262, 1190, 805 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₅BrNO₄S [M+H]⁺ 407.9900, found 407.9879.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(p-tolyl)butan-1-one:



78% yield, mp 105–106 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.23–2.30 (m, 2H), 2.41 (s, 3H), 3.17 (t, *J* = 6.8 Hz, 4H), 7.28 (t, *J* = 8.0 Hz, 3H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.70 (dt, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 2H), 7.97 (d, *J* = 8.0 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 198.9, 179.8, 153.5, 144.2, 136.9, 134.1, 129.3, 128.2, 128.1, 125.9, 119.1, 116.0, 36.7, 35.0, 21.6, 20.2; **IR** (KBr) ν 2929, 1685, 1650, 1385, 1188, 751 cm⁻¹; **HRMS** (ESI): calcd for C₁₈H₁₈NO₄S [M+H]⁺ 344.0951, found 344.0952.

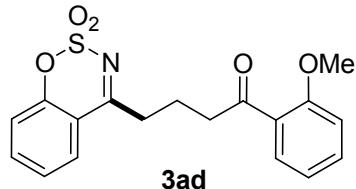
4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(4-methoxyphenyl)butan-1-one:



68% yield, mp 108–109 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.22–2.29 (m, 2H), 3.12–3.18 (m, 4H), 3.87 (s, 3H), 6.92–6.96 (m, 2H), 7.28 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.40 (dt, *J* = 8.0 Hz, 1.2 Hz, 1H), 7.70 (dt, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.94–7.99 (m, 3H); **13C NMR** (100 MHz, CDCl₃) δ 197.8, 179.8, 163.6, 153.5, 136.9, 130.2, 129.6, 128.3,

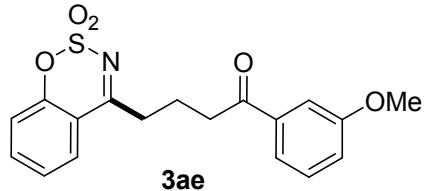
125.9, 119.1, 116.0, 113.8, 55.4, 36.4, 35.0, 20.3; **IR** (KBr) ν 2928, 1673, 1600, 1556, 1510, 1385, 1262, 1189, 1029, 738 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_5\text{S}$ $[\text{M}+\text{H}]^+$ 360.0900, found 360.0889.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(2-methoxyphenyl)butan-1-one:



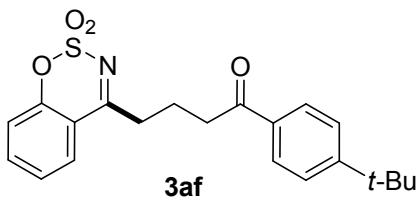
80% yield, mp 118–119 $^\circ\text{C}$; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 2.17–2.24 (m, 2H), 3.13 (t, $J = 7.6$ Hz, 2H), 3.19 (t, $J = 6.8$ Hz, 2H), 3.90 (s, 3H), 6.96–7.02 (m, 2H), 7.28 (dd, $J = 8.4$ Hz, 0.8 Hz, 1H), 7.39 (dt, $J = 7.6$ Hz, 0.8 Hz, 1H), 7.47 (dt, $J = 7.6$ Hz, 2.0 Hz, 1H), 7.67–7.73 (m, 2H), 7.97 (dd, $J = 8.0$ Hz, 1.6 Hz, 1H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 201.4, 180.1, 158.7, 153.5, 136.8, 133.8, 130.2, 128.3, 127.6, 125.9, 120.6, 119.0, 115.9, 111.5, 55.4, 42.1, 35.2, 20.7; **IR** (KBr) ν 2927, 1684, 1556, 1385, 1188, 755 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_5\text{S}$ $[\text{M}+\text{H}]^+$ 360.0900, found 360.0914.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(3-methoxyphenyl)butan-1-one:



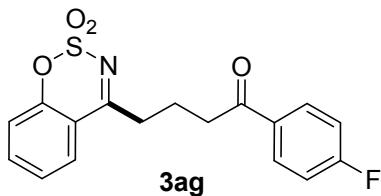
95% yield, mp 113–114 $^\circ\text{C}$; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 2.17–2.24 (m, 2H), 3.13 (t, $J = 7.6$ Hz, 2H), 3.19 (t, $J = 6.4$ Hz, 2H), 3.89 (s, 3H), 6.96–7.02 (m, 2H), 7.27 (d, $J = 8.0$ Hz, 1H), 7.39 (t, $J = 7.6$ Hz, 1H), 7.47 (dt, $J = 8.4$ Hz, 1.6 Hz, 1H), 7.67–7.73 (m, 2H), 7.97 (dd, $J = 8.0$ Hz, 0.8 Hz, 1H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 201.4, 180.1, 158.7, 153.4, 136.8, 133.7, 130.2, 128.3, 127.6, 125.9, 120.6, 119.0, 115.9, 111.5, 55.4, 42.1, 35.2, 20.7; **IR** (KBr) ν 2927, 1684, 1597, 1560, 1458, 1386, 1188, 755 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_5\text{S}$ $[\text{M}+\text{H}]^+$ 360.0900, found 360.0889.

1-(4-(tert-butyl)phenyl)-4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)butan-1-one:



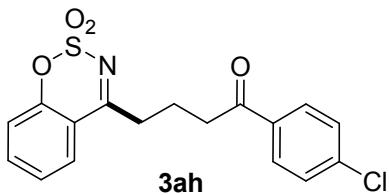
90% yield, mp 83–84 °C; **1H NMR** (400 MHz, CDCl₃) δ 1.34 (s, 9H), 2.22–2.29 (m, 2H), 3.15–3.19 (m, 4H), 7.27 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.40 (dt, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.47–7.50 (m, 2H), 7.70 (dt, *J* = 8.4 Hz, 1.6 Hz, 1H), 7.91–7.93 (m, 2H), 7.98 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 198.9, 179.8, 157.0, 153.4, 136.9, 133.9, 128.2, 127.9, 125.9, 125.5, 119.0, 115.9, 36.7, 35.1, 35.0, 30.9, 20.2; **IR** (KBr) ν 2965, 1681, 1605, 1556, 1385, 1188, 1108, 852, 774 cm⁻¹; **HRMS** (ESI): calcd for C₂₁H₂₄NO₄S [M+H]⁺ 386.1421, found 386.1410.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(4-fluorophenyl)butan-1-one:



82% yield, mp 125–126 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.24–2.31 (m, 2H), 3.15–3.20 (m, 4H), 7.11–7.17 (m, 2H), 7.29 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.40 (dt, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.71 (dt, *J* = 8.4 Hz, 1.6 Hz, 1H), 7.95 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.98–9.03 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 197.6, 179.5, 165.9 (d, *J*_{C-F} = 253.7 Hz), 153.6, 136.9, 130.7 (d, *J*_{C-F} = 9.4 Hz), 128.1, 125.9, 119.2, 116.0, 115.8 (d, *J*_{C-F} = 21.8 Hz), 36.8, 34.9, 20.0; **IR** (KBr) ν 2928, 1688, 1650, 1537, 1454, 1384, 1188 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₅FNO₄S [M+H]⁺ 348.0700, found 348.0697.

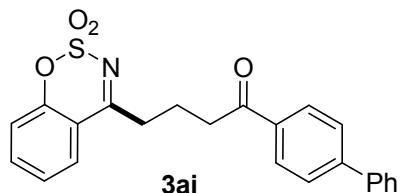
1-(4-chlorophenyl)-4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)butan-1-one:



87% yield, mp 142–143 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.23–2.30 (m, 2H), 3.14–3.19 (m, 4H), 7.28 (d, *J* = 8.4 Hz, 1H), 7.38–7.45 (m, 3H), 7.70 (t, *J* = 7.6 Hz, 1H), 7.90–7.95 (m, 3H); **13C NMR** (100 MHz, CDCl₃) δ 198.0, 179.6, 153.4, 139.7, 136.9, 134.8, 129.4, 128.9, 128.1, 125.9, 119.1, 115.9, 36.8, 34.8, 19.8; **IR** (KBr) ν 2928,

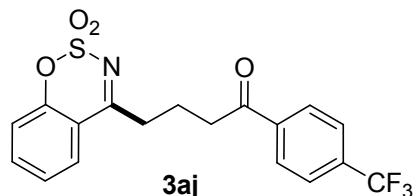
1686, 1655, 1560, 1508, 1458, 1388, 1189 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₅ClNO₄S [M+H]⁺ 364.0405, found 364.0407.

1-([1,1'-biphenyl]-4-yl)-4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)butan-1-one:



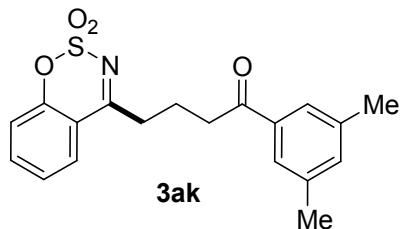
80% yield, mp 104–105 °C; **¹H NMR** (400 MHz, CDCl₃) δ 2.18–2.20 (m, 2H), 3.09–3.13 (m, 4H), 7.16–7.19 (m, 1H), 7.25–7.32 (m, 2H), 7.35–7.40 (m, 2H), 7.48–7.54 (m, 2H), 7.55–7.69 (m, 3H), 7.85–7.90 (m, 1H), 7.94–8.00 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 198.8, 179.8, 153.4, 145.8, 139.5, 136.9, 135.1, 128.9, 128.5, 128.2, 127.2, 127.1, 125.9, 119.0, 115.9, 36.8, 34.9, 20.0; **IR** (KBr) ν 2928, 1681, 1655, 1556, 1504, 1454, 1385, 1188, 767 cm⁻¹; **HRMS** (ESI): calcd for C₂₃H₂₀NO₄S [M+H]⁺ 406.1108, found 406.1103.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(4-(trifluoromethyl)phenyl)butan-1-one:



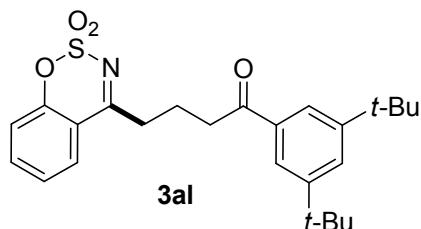
70% yield, mp 101–102 °C; **¹H NMR** (400 MHz, CDCl₃) δ 2.26–2.33 (m, 2H), 3.17–3.24 (m, 4H), 7.29 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.41 (dt, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.69–7.75 (m, 3H), 7.94 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 8.08 (d, *J* = 8.4 Hz, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 198.3, 179.5, 153.5, 139.1, 137.0, 134.5 (q, *J*_{C-F} = 32.7 Hz), 128.3, 128.0, 125.9, 125.7 (q, *J*_{C-F} = 3.7 Hz), 123.5 (q, *J*_{C-F} = 271.2 Hz), 119.1, 115.9, 37.1, 34.7, 19.6; **IR** (KBr) ν 2927, 1689, 1654, 1560, 1458, 1325, 1066, 740 cm⁻¹; **HRMS** (ESI): calcd for C₁₈H₁₅F₃NO₄S [M+H]⁺ 398.0668, found 398.0662.

1-(3,5-dimethylphenyl)-4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)butan-1-one:



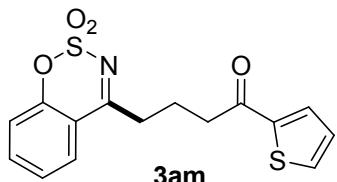
61% yield, mp 102–103 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.20–2.27 (m, 2H), 2.35 (s, 3H), 2.50 (s, 3H), 3.10 (t, *J* = 6.8 Hz, 2H), 3.15 (t, *J* = 7.6 Hz, 2H), 7.07–7.09 (m, 2H), 7.29 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.40 (dt, *J* = 8.0 Hz, 1.2 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 1H), 7.70 (dt, *J* = 7.6 Hz, 1.6 Hz, 1H), 7.94 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 202.3, 179.7, 153.5, 142.3, 138.8, 136.9, 134.2, 133.0, 129.2, 128.2, 126.4, 125.9, 119.1, 116.0, 39.2, 35.0, 21.7, 21.3, 20.5; **IR** (KBr) ν 2928, 1683, 1605, 1556, 1504, 1454, 1382, 1188 cm⁻¹; **HRMS** (ESI): calcd for C₁₉H₂₀NO₄S [M+H]⁺ 358.1108, found 358.1116.

1-(3,5-di-tert-butylphenyl)-4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)butan-1-one:



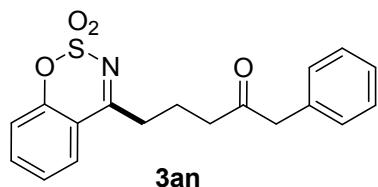
89% yield, mp 123–124 °C; **1H NMR** (400 MHz, CDCl₃) δ 1.36 (s, 18H), 2.24–2.31 (m, 2H), 3.17–3.24 (m, 4H), 7.27 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.41 (dt, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.67–7.72 (m, 2H), 7.83 (d, *J* = 1.6 Hz, 2H), 8.02 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 199.9, 179.9, 153.4, 151.3, 136.8, 136.2, 128.3, 127.5, 125.9, 122.1, 119.0, 115.9, 36.9, 35.0, 34.9, 31.2, 20.3; **IR** (KBr) ν 2964, 1682, 1599, 1556, 1478, 1391, 1190, 860, 771 cm⁻¹; **HRMS** (ESI): calcd for C₂₅H₃₂NO₄S [M+H]⁺ 442.2047, found 442.2054.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(thiophen-2-yl)butan-1-one:



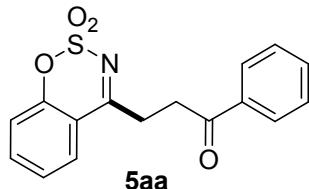
75% yield, mp 92–93 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.22–2.29 (m, 2H), 3.12–3.19 (m, 4H), 7.14 (dd, *J* = 4.8 Hz, 4.0 Hz, 1H), 7.28 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.40 (dt, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.65 (dd, *J* = 4.8 Hz, 0.8 Hz, 1H), 7.68–7.72 (m, 1H), 7.75 (dd, *J* = 4.0 Hz, 1.2 Hz, 1H), 7.95 (dd, *J* = 8.0 Hz, 1.2 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 192.2, 179.6, 153.4, 143.7, 136.9, 133.9, 132.2, 128.3, 128.2, 125.9, 119.0, 115.9, 37.4, 34.8, 20.2; **IR** (KBr) ν 2929, 1686, 1650, 1601, 1556, 1384, 1187, 1059 cm⁻¹; **HRMS** (ESI): calcd for C₁₅H₁₄NO₄S₂ [M+H]⁺ 336.0359, found 336.0359.

5-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpentan-2-one:



67% yield, mp 87–88 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.01–2.08 (m, 2H), 2.68 (t, *J* = 6.4 Hz, 2H), 2.97 (t, *J* = 7.6 Hz, 2H), 3.71 (s, 2H), 7.20–7.34 (m, 6H), 7.37 (dt, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.69 (dt, *J* = 7.6 Hz, 1.6 Hz, 1H), 7.84 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 207.6, 179.6, 153.3, 136.9, 133.7, 129.3, 128.7, 128.1, 127.0, 125.8, 118.9, 115.7, 50.1, 39.9, 34.5, 19.5; **IR** (KBr) ν 2929, 1682, 1654, 1599, 1560, 1508, 1458, 1381, 1189 cm⁻¹; **HRMS** (ESI): calcd for C₁₈H₁₈NO₄S [M+H]⁺ 344.0951, found 344.0952.

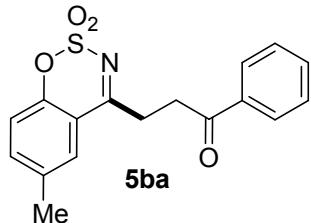
3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



88% yield, mp 113–114 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.53 (t, *J* = 5.6 Hz, 2H), 3.60 (t, *J* = 5.6 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.71 (t, *J* = 7.2 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 8.01 (d, *J* = 7.6 Hz, 2H); **13C NMR** (100 MHz, CDCl₃) δ 197.3, 178.9, 153.3, 136.9, 136.1, 133.5, 128.6, 128.0, 127.9, 125.9, 119.0, 116.2, 33.2, 29.3; **IR** (KBr) ν 2928, 1682, 1650, 1600, 1556, 1384, 1190 cm⁻¹; **HRMS** (ESI): calcd for C₁₆H₁₄NO₄S

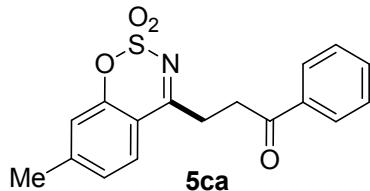
$[M+H]^+$ 316.0638, found 316.0629.

3-(6-methyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



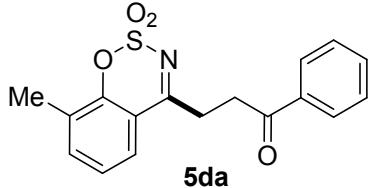
57% yield, mp 138–139 °C; **1H NMR** (400 MHz, CDCl_3) δ 2.45 (s, 3H), 3.05–3.54 (m, 2H), 3.58–3.62 (m, 2H), 7.19 (d, $J = 8.4$ Hz, 1H), 7.47–7.52 (m, 3H), 7.58–7.62 (m, 1H), 7.75 (d, $J = 1.2$ Hz, 1H), 8.01–8.04 (m, 2H); **13C NMR** (100 MHz, CDCl_3) δ 197.6, 178.9, 151.4, 137.7, 136.3, 136.0, 133.5, 128.7, 128.1, 127.8, 118.8, 116.0, 33.3, 29.4, 20.9; **IR** (KBr) ν 2926, 1681, 1655, 1556, 1506, 1385, 1190 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{17}\text{H}_{16}\text{NO}_4\text{S}$ $[M+H]^+$ 330.0795, found 330.1007.

3-(7-methyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



88% yield, mp 124–125 °C; **1H NMR** (400 MHz, CDCl_3) δ 2.47 (s, 3H), 3.48–3.51 (m, 2H), 3.56–3.60 (m, 2H), 7.09 (s, 1H), 7.20 (d, $J = 8.0$ Hz, 1H), 7.48 (t, $J = 7.2$ Hz, 2H), 7.59 (t, $J = 7.2$ Hz, 1H), 7.84 (d, $J = 8.4$ Hz, 1H), 8.00–8.03 (m, 2H); **13C NMR** (100 MHz, CDCl_3) δ 197.5, 178.7, 153.5, 149.3, 136.2, 133.4, 128.6, 128.0, 127.7, 126.8, 119.1, 113.9, 33.3, 29.3, 22.0; **IR** (KBr) ν 2929, 1681, 1621, 1593, 1544, 1449, 1385, 1265, 1196, 1135, 788 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{17}\text{H}_{16}\text{NO}_4\text{S}$ $[M+H]^+$ 330.0795, found 330.1007.

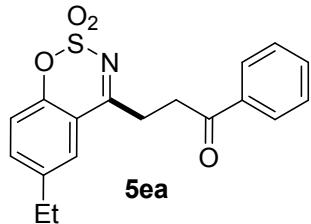
3-(8-methyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



75% yield, mp 117–118 °C; **1H NMR** (400 MHz, CDCl_3) δ 2.40 (s, 3H), 3.51–3.54 (m, 2H), 3.57–3.61 (m, 2H), 7.30 (t, $J = 8.0$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 2H), 7.55–

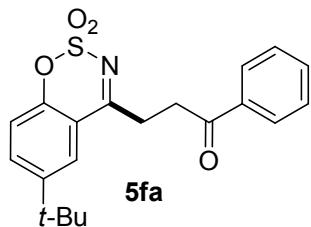
7.62 (m, 2H), 7.80 (dd, J = 8.0 Hz, 0.8 Hz, 1H), 8.01–8.04 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 197.4, 179.2, 151.8, 138.3, 136.2, 133.4, 128.8, 128.6, 128.1, 125.4, 125.1, 116.1, 33.4, 29.5, 14.9; **IR** (KBr) ν 3057, 2927, 1688, 1596, 1568, 1387, 1266, 1202, 1091, 1002, 871, 746 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₆NO₄S [M+H]⁺ 330.0795, found 330.0799.

3-(6-ethyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



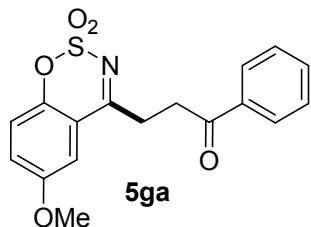
67% yield, mp 142–143 °C; **¹H NMR** (400 MHz, CDCl₃) δ 1.29 (t, J = 7.6 Hz, 3H), 2.75 (q, J = 15.2 Hz, 7.6 Hz, 2H), 3.52–3.56 (m, 2H), 3.59–3.62 (m, 2H), 7.21 (d, J = 8.4 Hz, 1H), 7.49 (t, J = 7.2 Hz, 2H), 7.54 (dd, J = 8.4 Hz, 2.0 Hz, 1H), 7.58–7.62 (m, 1H), 7.75 (d, J = 2.0 Hz, 1H), 8.02–8.05 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 197.5, 178.9, 151.5, 142.3, 136.6, 136.3, 133.5, 128.7, 128.1, 126.7, 118.9, 116.1, 33.3, 29.4, 28.3, 15.4; **IR** (KBr) ν 2927, 1681, 1650, 1556, 1504, 1385, 1190 cm⁻¹; **HRMS** (ESI): calcd for C₁₈H₁₈NO₄S [M+H]⁺ 344.0951, found 344.1141.

3-(6-(tert-butyl)-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



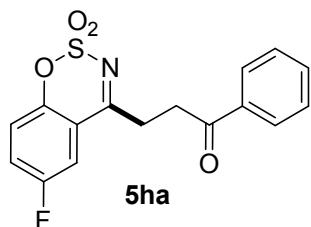
82% yield, mp 118–119 °C; **¹H NMR** (400 MHz, CDCl₃) δ 1.37 (s, 9H), 3.54–3.62 (m, 4H), 7.22 (d, J = 8.8 Hz, 1H), 7.49 (t, J = 7.2 Hz, 2H), 7.57–7.62 (m, 1H), 7.75 (dd, J = 8.4 Hz, 2.4 Hz, 1H), 7.90 (d, J = 2.0 Hz, 1H), 8.02–8.05 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 197.5, 179.1, 151.2, 149.3, 136.2, 134.5, 133.4, 128.6, 128.0, 124.0, 118.5, 115.7, 34.8, 33.2, 31.1, 29.3; **IR** (KBr) ν 2966, 1686, 1598, 1561, 1449, 1386, 1236, 1162, 842, 787 cm⁻¹; **HRMS** (ESI): calcd for C₂₀H₂₂NO₄S [M+H]⁺ 372.1264, found 372.1435.

3-(6-methoxy-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



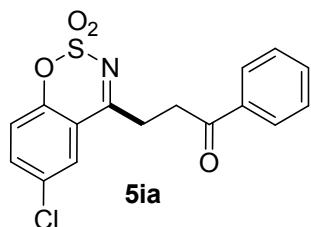
94% yield, mp 135–136 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.54–3.57 (m, 2H), 3.63–3.66 (m, 2H), 3.93 (s, 3H), 7.28–7.32 (m, 2H), 7.41 (d, *J* = 2.8 Hz, 1H), 7.54 (t, *J* = 7.2 Hz, 2H), 7.64 (t, *J* = 7.2 Hz, 1H), 8.06–8.08 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 197.5, 178.7, 156.8, 147.1, 136.2, 133.5, 128.6, 128.1, 123.3, 120.0, 116.7, 111.1, 56.1, 33.3, 29.4; **IR** (KBr) ν 2926, 1682, 1603, 1562, 1485, 1385, 1291, 1187, 1037, 832 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₆NO₅S [M+H]⁺ 346.0744, found 346.0926.

3-(6-fluoro-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



70% yield, mp 125–126 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.47 (t, *J* = 6.8 Hz, 2H), 3.61 (t, *J* = 6.4 Hz, 2H), 7.31 (dd, *J* = 13.2 Hz, 4.4 Hz, 1H), 7.41–7.46 (m, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.58–7.62 (m, 1H), 7.67 (dd, *J* = 8.0 Hz, 2.8 Hz, 1H), 8.00–8.03 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 197.2, 178.0, 159.0 (d, *J*_{C-F} = 246.7 Hz), 149.3, 136.1, 133.6, 128.7, 128.1, 124.1 (d, *J*_{C-F} = 24.0 Hz), 120.9 (d, *J*_{C-F} = 8.0 Hz), 117.0 (d, *J*_{C-F} = 7.6 Hz), 114.1 (d, *J*_{C-F} = 25.0 Hz), 33.3, 29.4; **IR** (KBr) ν 2925, 1686, 1599, 1542, 1504, 1396, 1199 cm⁻¹; **HRMS** (ESI): calcd for C₁₆H₁₃FNO₄S [M+H]⁺ 334.0544, found 334.0539.

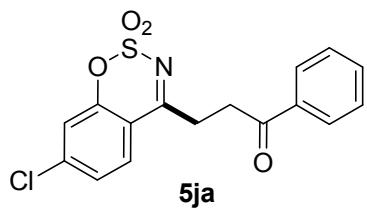
3-(6-chloro-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



68% yield, mp 151–152 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.49 (t, *J* = 6.8 Hz, 2H),

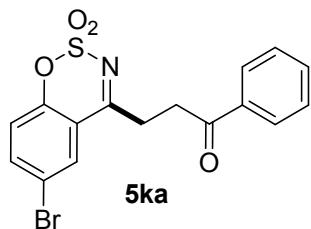
3.61 (t, $J = 6.4$ Hz, 2H), 7.26 (d, $J = 8.8$ Hz, 1H), 7.59 (t, $J = 7.2$ Hz, 2H), 7.60 (t, $J = 7.6$ Hz, 1H), 7.87 (dd, $J = 8.8$ Hz, 2.4 Hz, 1H), 7.96 (d, $J = 2.4$ Hz, 1H), 8.00–8.03 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.1, 177.9, 151.8, 136.7, 136.1, 133.6, 131.4, 128.7, 128.1, 127.5, 120.6, 117.2, 33.3, 29.4; IR (KBr) ν 2926, 1681, 1599, 1556, 1504, 1392, 1193, 800 cm^{-1} ; HRMS (ESI): calcd for $\text{C}_{16}\text{H}_{13}\text{ClNO}_4\text{S}$ [M+H] $^+$ 350.0248, found 350.0453.

3-(7-chloro-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



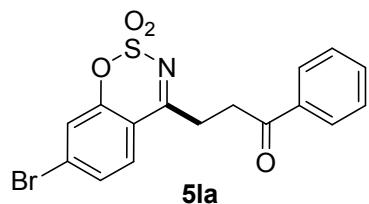
72% yield, mp 124–125 °C; ^1H NMR (400 MHz, CDCl_3) δ 3.49 (t, $J = 6.4$ Hz, 2H), 3.59 (t, $J = 6.0$ Hz, 2H), 7.31 (d, $J = 1.6$ Hz, 1H), 7.39 (dd, $J = 8.4$ Hz, 2.0 Hz, 1H), 7.48 (t, $J = 7.2$ Hz, 2H), 7.58–7.61 (m, 1H), 7.92 (d, $J = 8.4$ Hz, 1H), 7.99–8.02 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.3, 178.2, 153.8, 143.0, 136.1, 133.6, 128.9, 128.7, 128.1, 126.5, 119.4, 114.7, 33.3, 29.4; IR (KBr) ν 2928, 1681, 1655, 1599, 1556, 1504, 1396, 1193, 1102, 911 cm^{-1} ; HRMS (ESI): calcd for $\text{C}_{16}\text{H}_{13}\text{ClNO}_4\text{S}$ [M+H] $^+$ 350.0248, found 350.0239.

3-(6-bromo-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



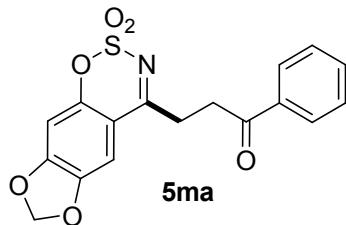
65% yield, mp 127–128 °C; ^1H NMR (400 MHz, CDCl_3) δ 3.49 (t, $J = 6.4$ Hz, 2H), 3.61 (t, $J = 6.0$ Hz, 2H), 7.20 (d, $J = 8.8$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 2H), 7.58–7.63 (m, 1H), 7.81 (dd, $J = 8.8$ Hz, 2.0 Hz, 1H), 8.00–8.03 (m, 2H), 8.10 (d, $J = 2.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.2, 177.8, 152.3, 139.5, 136.1, 133.6, 130.5, 128.7, 128.1, 120.8, 118.6, 117.6, 33.3, 29.4; IR (KBr) ν 2926, 1686, 1650, 1556, 1392, 1192 cm^{-1} ; HRMS (ESI): calcd for $\text{C}_{16}\text{H}_{13}\text{BrNO}_4\text{S}$ [M+H] $^+$ 393.9743, found 393.9737.

3-(7-bromo-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



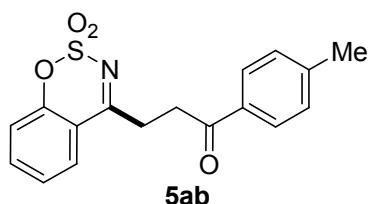
66% yield, mp 129–130 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.49 (t, *J* = 6.4 Hz, 2H), 3.60 (t, *J* = 6.4 Hz, 2H), 7.20 (d, *J* = 8.8 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.58–7.62 (m, 1H), 7.81 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 8.00–8.03 (m, 2H), 8.10 (d, *J* = 2.0 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 197.2, 177.8, 152.2, 139.5, 136.0, 133.5, 130.5, 128.7, 128.1, 120.8, 118.6, 117.6, 33.2, 29.4; **IR** (KBr) ν 2924, 1682, 1638, 1556, 1459, 1391, 1191, 786 cm⁻¹; **HRMS** (ESI): calcd for C₁₆H₁₃BrNO₄S [M+H]⁺ 393.9743, found 393.9737.

3-(2,2-dioxido-[1,3]dioxolo[4',5':4,5]benzo[1,2-e][1,2,3]oxathiazin-4-yl)-1-phenylpropan-1-one:



50% yield, mp 175–176 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.40 (t, *J* = 6.8 Hz, 2H), 3.60 (t, *J* = 6.8 Hz, 2H), 6.15 (s, 2H), 6.75 (s, 1H), 7.28 (s, 1H), 7.49 (t, *J* = 7.2 Hz, 2H), 7.59 (t, *J* = 7.2 Hz, 1H), 8.00–8.03 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 197.5, 177.9, 154.6, 151.5, 145.7, 136.2, 133.5, 128.7, 128.1, 110.1, 105.1, 103.3, 100.1, 33.4, 29.7; **IR** (KBr) ν 2928, 1680, 1633, 1556, 1504, 1385, 1195 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₄NO₆S [M+H]⁺ 360.0536, found 360.0721.

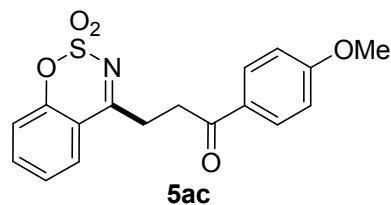
3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(p-tolyl)propan-1-one:



96% yield, mp 115–116 °C; **1H NMR** (400 MHz, CDCl₃) δ 2.36 (s, 3H), 3.44–3.53

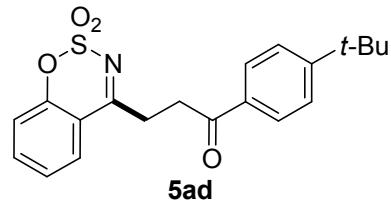
(m, 4H), 7.21–7.24 (m, 3H), 7.35 (t, J = 7.6 Hz, 1H), 7.65 (t, J = 7.6 Hz, 1H), 7.85 (d, J = 8.0 Hz, 2H), 7.91 (d, J = 8.0 Hz, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 196.9, 179.0, 153.3, 144.3, 136.9, 133.7, 129.3, 128.2, 127.9, 125.9, 119.0, 116.2, 33.2, 29.4, 21.6; **IR** (KBr) ν 2927, 1698, 1655, 1633, 1556, 1385, 1189 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{17}\text{H}_{16}\text{NO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 330.0795, found 330.1007.

3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(4-methoxyphenyl)propan-1-one:



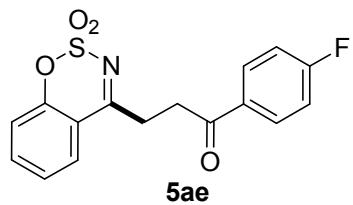
72% yield, mp 121–122 °C; **^1H NMR** (400 MHz, CDCl_3) δ 3.49–3.57 (m, 4H), 3.87 (s, 3H), 6.93–6.97 (m, 2H), 7.29 (dd, J = 8.4 Hz, 0.8 Hz, 1H), 7.41 (dt, J = 8.0 Hz, 1.2 Hz, 1H), 7.71 (dt, J = 8.4 Hz, 1.6 Hz, 1H), 7.96–8.02 (m, 3H); **^{13}C NMR** (100 MHz, CDCl_3) δ 195.8, 179.1, 163.8, 153.3, 136.9, 130.4, 129.3, 127.9, 125.9, 119.0, 116.3, 113.8, 55.4, 33.0, 29.5; **IR** (KBr) ν 2928, 1686, 1655, 1556, 1504, 1454, 1385, 1185 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{17}\text{H}_{16}\text{NO}_5\text{S}$ [$\text{M}+\text{H}]^+$ 346.0744, found 346.0737.

1-(4-(tert-butyl)phenyl)-3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)propan-1-one:



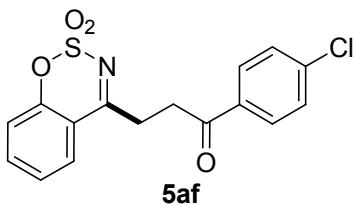
69% yield, mp 111–112 °C; **^1H NMR** (400 MHz, CDCl_3) δ 1.35 (s, 9H), 3.50–3.54 (m, 2H), 3.56–3.60 (m, 2H), 7.29 (d, J = 8.4 Hz, 1H), 7.41 (dt, J = 8.0 Hz, 0.8 Hz, 1H), 7.50 (d, J = 8.8 Hz, 2H), 7.71 (dt, J = 8.4 Hz, 1.2 Hz, 1H), 7.94–8.00 (m, 3H); **^{13}C NMR** (100 MHz, CDCl_3) δ 197.0, 179.0, 157.3, 153.4, 136.9, 133.6, 128.0, 127.9, 125.9, 125.6, 119.0, 116.3, 35.1, 33.2, 31.0, 29.4; **IR** (KBr) ν 2926, 1682, 1650, 1600, 1556, 1386, 1189, 821 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{20}\text{H}_{22}\text{NO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 372.1264, found 372.1435.

3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(4-fluorophenyl)propan-1-one:



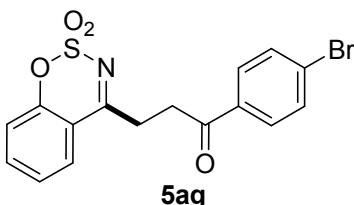
87% yield, mp 127–128 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.50–3.58 (m, 4H), 7.12–7.17 (m, 2H), 7.28 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.41 (dt, *J* = 8.0 Hz, 1.2 Hz, 1H), 7.69–7.73 (m, 1H), 7.97 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 8.01–8.07 (m, 2H); **13C NMR** (100 MHz, CDCl₃) δ 195.8, 178.7, 165.9 (d, *J*_{C–F} = 253.8 Hz), 153.3, 137.0, 132.6 (d, *J*_{C–F} = 3.0 Hz), 130.8 (d, *J*_{C–F} = 9.4 Hz), 127.9, 125.9, 119.0, 116.2, 115.8 (d, *J*_{C–F} = 30.8 Hz), 33.1, 29.3; **IR** (KBr) ν 2919, 1687, 1599, 1557, 1508, 1390, 1266, 1190, 856, 743 cm⁻¹; **HRMS** (ESI): calcd for C₁₆H₁₃FNO₄S [M+H]⁺ 334.0544, found 334.0539.

1-(4-chlorophenyl)-3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)propan-1-one:



73% yield, mp 132–133 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.50–3.56 (m, 4H), 7.28 (d, *J* = 8.4 Hz, 1H), 7.39–7.46 (m, 3H), 7.71 (dt, *J* = 8.8 Hz, 2.0 Hz, 1H), 7.93–7.98 (m, 3H); **13C NMR** (100 MHz, CDCl₃) δ 196.2, 178.7, 153.3, 139.9, 137.0, 134.5, 129.4, 128.9, 127.8, 125.9, 119.0, 116.1, 33.1, 29.2; **IR** (KBr) ν 2928, 1681, 1633, 1599, 1556, 1504, 1385, 1190 cm⁻¹; **HRMS** (ESI): calcd for C₁₆H₁₃ClNO₄S [M+H]⁺ 350.0248, found 350.0453.

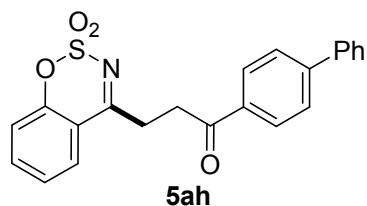
1-(4-bromophenyl)-3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)propan-1-one:



85% yield, mp 158–159 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.51–3.58 (m, 4H), 7.30 (d, *J* = 8.4 Hz, 1H), 7.42 (dt, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.72

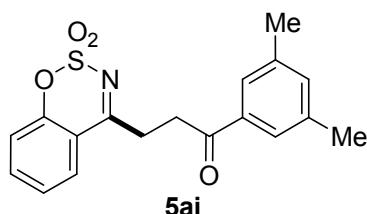
(dt, $J = 8.4$ Hz, 1.2 Hz, 1H), 7.88 (d, $J = 8.8$ Hz, 2H), 7.96 (dd, $J = 8.0$ Hz, 1.2 Hz, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 196.4, 178.5, 153.4, 137.0, 134.9, 132.0, 129.6, 128.7, 127.8, 125.9, 119.1, 116.2, 33.2, 29.3; **IR** (KBr) ν 2926, 1681, 1655, 1631, 1556, 1385, 1188, 1070, 755 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{16}\text{H}_{13}\text{BrNO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 393.9743, found 393.9888.

1-([1,1'-biphenyl]-4-yl)-3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)propan-1-one:



90% yield, mp 207–208 °C; **^1H NMR** (400 MHz, CDCl_3) δ 3.56 (t, $J = 6.0$ Hz, 2H), 3.63 (t, $J = 6.0$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 1H), 7.39–7.50 (m, 4H), 7.64 (d, $J = 7.6$ Hz, 2H), 7.70–7.75 (m, 3H), 7.99 (d, $J = 7.6$ Hz, 1H), 8.10 (d, $J = 8.0$ Hz, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 197.0, 178.8, 153.5, 146.2, 139.7, 136.9, 134.9, 128.9, 128.7, 128.3, 127.9, 127.3, 127.2, 125.9, 119.1, 116.3, 33.4, 29.5; **IR** (KBr) ν 2919, 1680, 1655, 1600, 1545, 1503, 1389, 1189, 751 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{22}\text{H}_{18}\text{NO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 392.0951, found 392.0959.

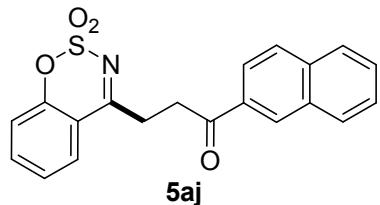
1-(3,5-dimethylphenyl)-3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)propan-1-one:



95% yield, mp 127–128 °C; **^1H NMR** (400 MHz, CDCl_3) δ 2.38 (s, 6H), 3.49–3.59 (m, 4H), 7.22 (s, 1H), 7.29 (d, $J = 8.4$ Hz, 1H), 7.41 (dt, $J = 7.6$ Hz, 0.8 Hz, 1H), 7.62 (s, 2H), 7.71 (dt, $J = 8.4$ Hz, 1.2 Hz, 1H), 7.98 (dd, $J = 8.0$ Hz, 1.6 Hz, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 197.7, 179.0, 153.3, 128.3, 136.9, 136.3, 135.1, 127.9, 125.8, 119.0, 116.3, 33.4, 29.5, 21.1; **IR** (KBr) ν 2928, 1681, 1650, 1599, 1556, 1385, 1189, 859, 768 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_4\text{S}$ [$\text{M}+\text{H}]^+$ 344.0951, found

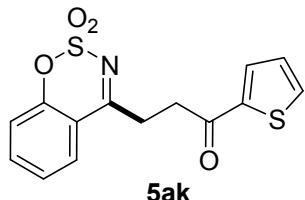
344.1141.

3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(naphthalen-2-yl)propan-1-one:



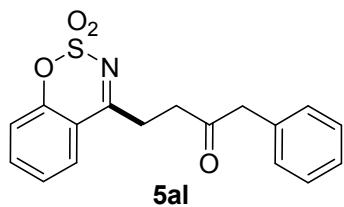
92% yield, mp 166–167 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.58 (t, *J* = 6.4 Hz, 2H), 3.73 (t, *J* = 6.4 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.55–7.64 (m, 2H), 7.72 (dt, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.89 (t, *J* = 13.6 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 8.05 (dd, *J* = 8.4 Hz, 1.2 Hz, 1H), 8.56 (s, 1H); **13C NMR** (100 MHz, CDCl₃) δ 197.3, 178.9, 153.4, 136.9, 135.7, 133.5, 132.4, 130.0, 129.6, 128.6, 128.5, 127.9, 127.7, 126.9, 125.9, 123.6, 119.1, 116.3, 33.4, 29.5; **IR** (KBr) ν 2926, 1680, 1601, 1556, 1504, 1385, 1187 cm⁻¹; **HRMS** (ESI): calcd for C₂₀H₁₆NO₄S [M+H]⁺ 366.0795, found 366.0975.

3-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-(thiophen-2-yl)propan-1-one:



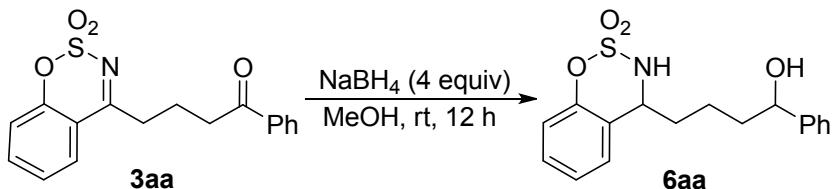
60% yield, mp 132–133 °C; **1H NMR** (400 MHz, CDCl₃) δ 3.50–3.56 (m, 4H), 7.16 (dd, *J* = 13.2 Hz, 4.0 Hz, 1H), 7.16 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.41 (dt, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.68 (dd, *J* = 8.8 Hz, 1.2 Hz, 1H), 7.72 (dt, *J* = 8.4 Hz, 1.6 Hz, 1H), 7.83 (dd, *J* = 3.6 Hz, 1.2 Hz, 1H), 7.95 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H); **13C NMR** (100 MHz, CDCl₃) δ 190.2, 178.6, 153.3, 143.1, 137.0, 134.0, 132.4, 128.2, 127.9, 125.9, 119.1, 116.2, 33.7, 29.3; **IR** (KBr) ν 2919, 1686, 1655, 1599, 1542, 1504, 1385, 1187 cm⁻¹; **HRMS** (ESI): calcd for C₁₄H₁₂NO₄S₂ [M+H]⁺ 322.0202, found 322.0418.

4-(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)-1-phenylbutan-2-one:



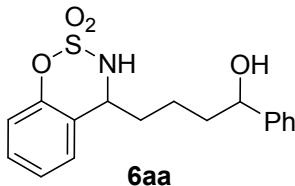
87% yield, mp 90–91 °C; **¹H NMR** (400 MHz, CDCl₃) δ 3.06 (t, *J* = 6.4 Hz, 2H), 3.35 (t, *J* = 6.4 Hz, 2H), 3.84 (s, 2H), 7.27–7.32 (m, 4H), 7.36–7.42 (m, 3H), 7.69–7.74 (m, 1H), 7.87 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 206.0, 178.5, 153.2, 136.9, 133.6, 129.4, 128.7, 127.7, 127.1, 125.8, 119.0, 116.0, 50.0, 36.0, 29.2; **IR** (KBr) ν 2928, 1715, 1655, 1633, 1556, 1385, 1188, 743 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₆NO₄S [M+H]⁺ 330.0795, found 330.1007.

The procedure for the further synthetic transformations:



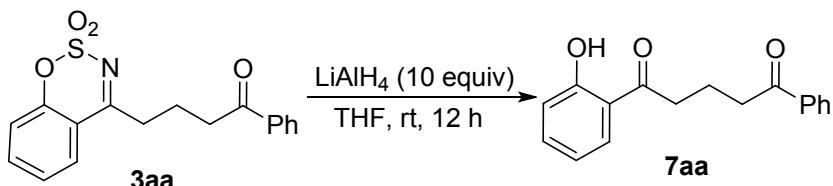
To a solution of the **3aa** (99 mg, 0.3 mmol) in MeOH (5 mL) was added NaBH₄ (46 mg, 1.2 mmol) a few times at room temperature. The reaction mixture was stirred at room temperature for 12 h. The reaction was diluted with water (10 mL) and EtOAc (10 mL). The aqueous layer was separated and extracted with EtOAc (2 × 10 mL). The organic layers were combined and washed with saturated brine (15 mL), dried anhydrous Mg₂SO₄, filtered, and then concentrated in vacuo. The residue was purified by column chromatography (petroleum ether/EtOAc as the eluent) to give the product **6aa**.

4-(4-hydroxy-4-phenylbutyl)benzo[e][1,2,3]oxathiazine 2,2-dioxide:



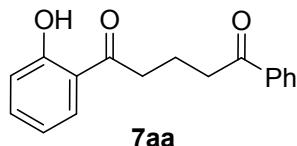
oil, 96% yield; **¹H NMR** (400 MHz, CDCl₃) δ 1.14–1.35 (m, 1H), 1.47–1.77 (m, 3H), 1.84–1.98 (m, 2H), 2.65 (s, 1H), 4.52–4.54 (m, 2H), 5.35 (dd, *J* = 36.8 Hz, 7.2 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 7.00–7.09 (m, 2H), 7.14–7.25 (m, 6H); **¹³C NMR** (100

MHz, CDCl₃) δ 151.0, 144.1 (d, *J* = 3.2 Hz), 129.1, 128.4, 127.6 (d, *J* = 4.3 Hz), 126.3, 125.8 (d, *J* = 7.1 Hz), 125.1, 122.8 (d, *J* = 5.4 Hz), 118.6, 74.1 (d, *J* = 26.1 Hz), 57.0 (d, *J* = 3.9 Hz), 37.9 (d, *J* = 5.0 Hz), 33.4, 21.6 (d, *J* = 11.6 Hz); **IR** (KBr) ν 3432, 2929, 1607, 1560, 1508, 1454, 1421, 1372, 1262, 1174, 1107, 883, 762, 702 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₈NO₃S [M-H₂O+H]⁺ 316.1002, found 316.1013.



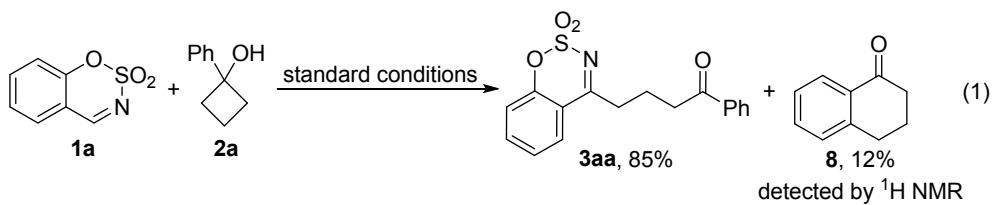
To a solution of the **3aa** (99 mg, 0.3 mmol) in THF (5 mL) was added LiAlH₄ (114 mg, 3.0 mmol) a few times at room temperature. The reaction mixture was stirred at room temperature for 12 h. The reaction was diluted with water (10 mL) and EtOAc (10 mL). The aqueous layer was separated and extracted with EtOAc (2 × 10 mL). The organic layers were combined and washed with saturated brine (15 mL), dried anhydrous Mg₂SO₄, filtered, and then concentrated in vacuo. The residue was purified by column chromatography (petroleum ether/EtOAc as the eluent) to give the product **7aa**.

1-(2-hydroxyphenyl)-5-phenylpentane-1,5-dione:



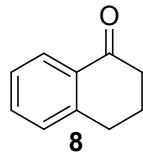
White solid, 55% yield, mp 83–84 °C; **¹H NMR** (400 MHz, CDCl₃) δ 2.17–2.24 (m, 2H), 3.11–3.16 (m, 4H), 6.87 (dt, *J* = 8.4 Hz, 1.2 Hz, 1H), 6.97 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 7.44–7.48 (m, 3H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.81 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.96–7.99 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 206.1, 199.5, 162.4, 136.7, 136.3, 133.1, 130.0, 128.6, 127.9, 119.2, 118.9, 118.4, 37.4, 37.3, 18.6; **IR** (KBr) ν 3450, 2928, 1686, 1650, 1556, 1504, 1260, 873, 721 cm⁻¹; **HRMS** (ESI): calcd for C₁₇H₁₇O₃ [M+H]⁺ 269.1172, found 269.1164.

General Procedure for the Mechanism studies (1):



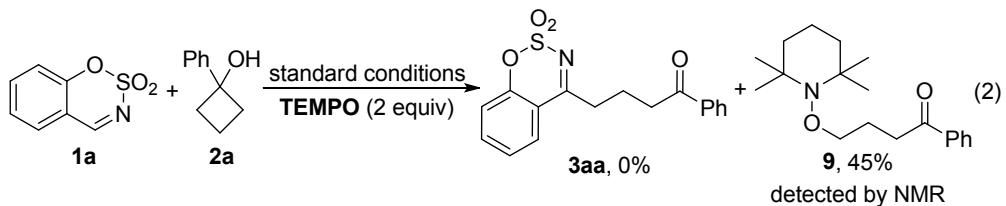
To a 10 mL Schlenk charged with cyclic aldimine **1a** (36.6 mg, 0.2 mmol), AgNO_3 (6.8 mg, 0.04 mmol), $\text{K}_2\text{S}_2\text{O}_8$ (270 mg, 1.0 mmol) and 1-phenylcyclobutanol **2a** (44.5 mg, 0.3 mmol) was added CH_3CN (1.0 mL) and distilled H_2O (1.0 mL) *via* a syringe. The reaction mixture was vigorously stirred at 60 °C for 48 h. After the reaction was complete, the mixture was diluted with water (10 mL) and extracted with ethyl acetate (3×10 mL). The organic layers were combined and washed with saturated brine (15 mL), dried anhydrous Mg_2SO_4 , and then concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent) to afford the desired product **3aa** in 85% yield and the byproduct **8** in 12% yield.

3,4-dihydronaphthalen-1(2H)-one:



oil, 12% yield; ^1H NMR (400 MHz, CDCl_3) δ 2.11–2.17 (m, 2H), 2.66 (t, $J = 6.0$ Hz, 2H), 2.97 (t, $J = 6.0$ Hz, 2H), 7.25 (d, $J = 6.4$ Hz, 1H), 7.31 (t, $J = 7.6$ Hz, 1H), 7.47 (dt, $J = 7.6$ Hz, 1.2 Hz, 1H), 8.03 (d, $J = 8.0$ Hz, 1H).

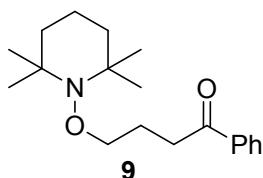
General Procedure for the Mechanism studies (2):



To a 10 mL Schlenk charged with cyclic aldimine **1a** (36.6 mg, 0.2 mmol), AgNO_3 (6.8 mg, 0.04 mmol), $\text{K}_2\text{S}_2\text{O}_8$ (270 mg, 1.0 mmol), 1-phenylcyclobutanol **2a** (44.5 mg, 0.3 mmol) and TEMPO (62.5 mg, 0.4 mmol) was added CH_3CN (1.0 mL) and distilled H_2O (1.0 mL) *via* a syringe. The reaction mixture was vigorously stirred at 60 °C for 48 h. After the reaction was complete, the mixture was diluted with water

(10 mL) and extracted with ethyl acetate (3×10 mL). The organic layers were combined and washed with saturated brine (15 mL), dried anhydrous Mg_2SO_4 , and concentrated in vacuo. The desired product **3aa** was not detected by TLC. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent) to give the compound **9** in 45% yield as white solid.

1-phenyl-4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)butan-1-one:

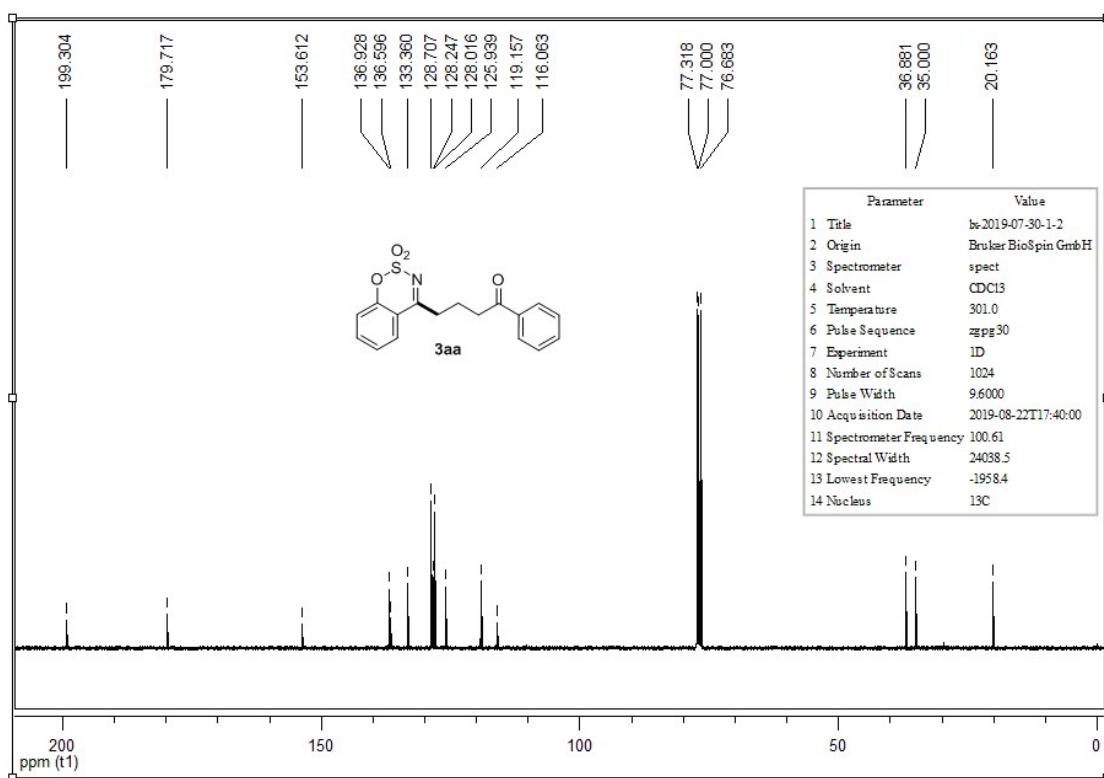
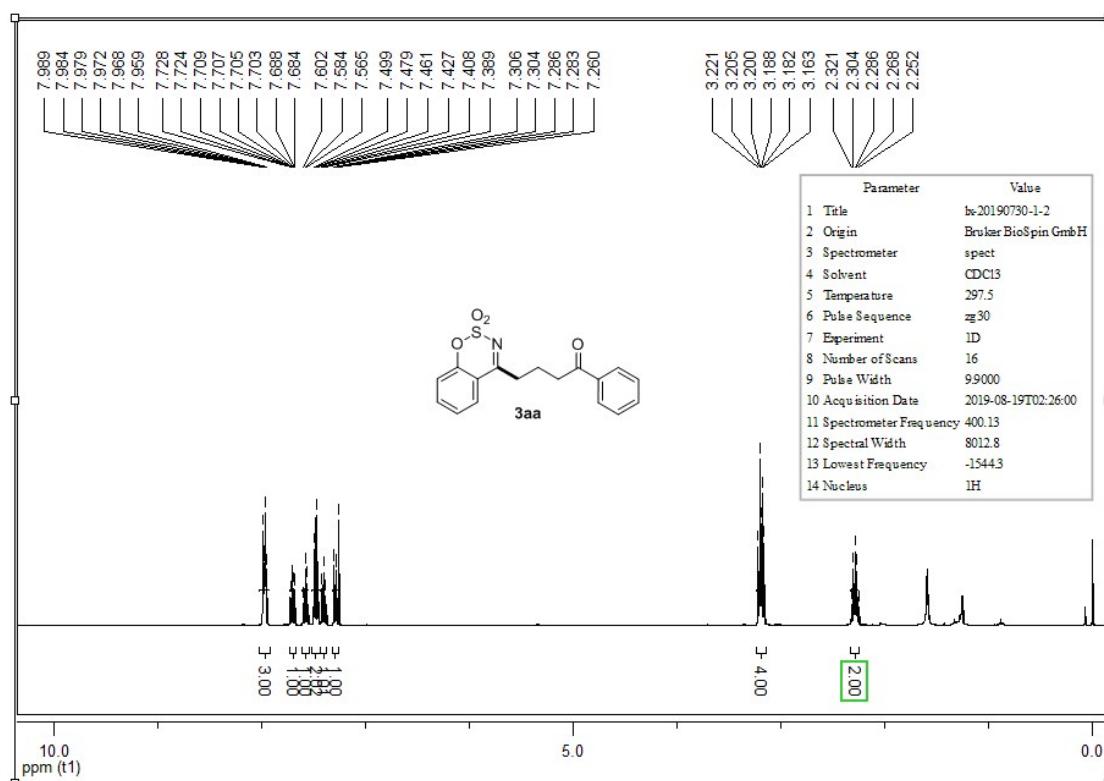


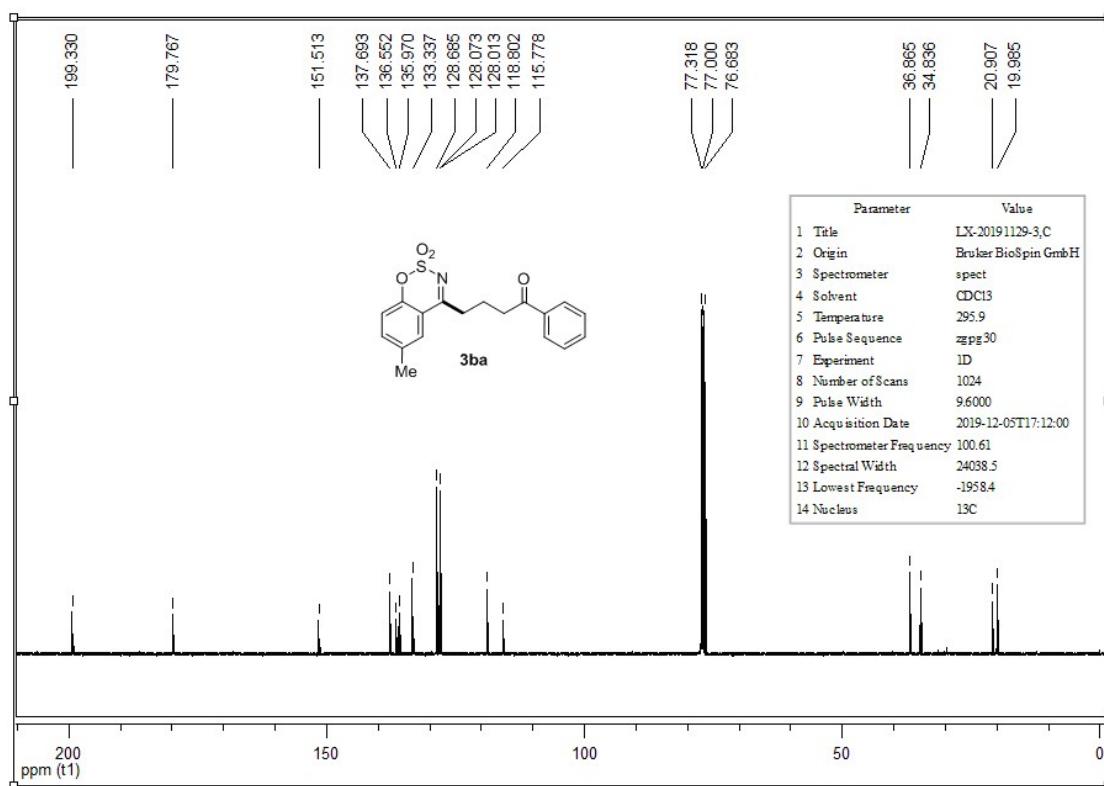
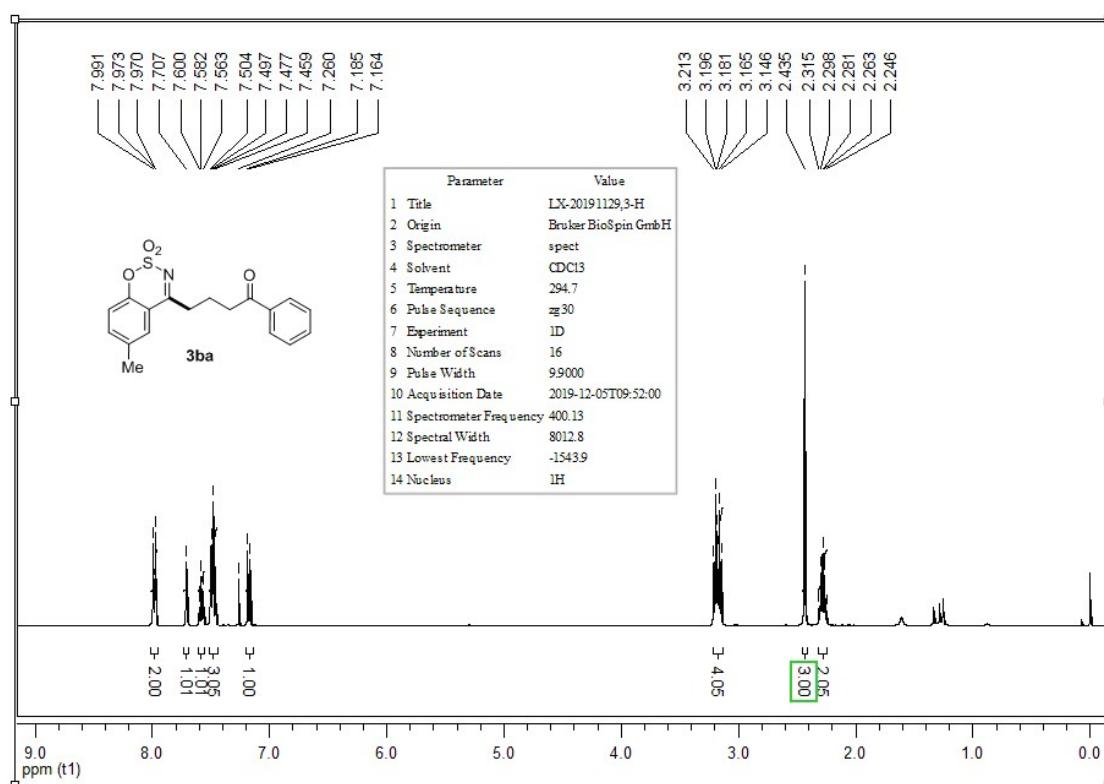
45% yield, mp 82–83 °C; **1H NMR** (400 MHz, CDCl_3) δ 1.08 (s, 6H), 1.14 (s, 6H), 1.28–1.57 (m, 6H), 1.94–2.01 (m, 2H), 3.09 (t, $J = 7.6$ Hz, 2H), 3.82 (t, $J = 6.4$ Hz, 3H), 7.46 (t, $J = 7.2$ Hz, 2H), 7.55 (t, $J = 7.2$ Hz, 1H), 7.96–7.99 (m, 2H); **13C NMR** (100 MHz, CDCl_3) δ 200.1, 137.0, 132.8, 128.5, 128.0, 75.4, 59.6, 39.5, 35.4, 33.0, 23.6, 20.0, 17.0; **IR** (KBr) ν 2929, 1682, 1650, 1561, 1509, 1450, 1359, 1263, 1208, 1132, 741, 691 cm^{-1} ; **HRMS** (ESI): calcd for $\text{C}_{19}\text{H}_{30}\text{NO}_2$ [$\text{M}+\text{H}]^+$ 304.2271, found 304.2269.

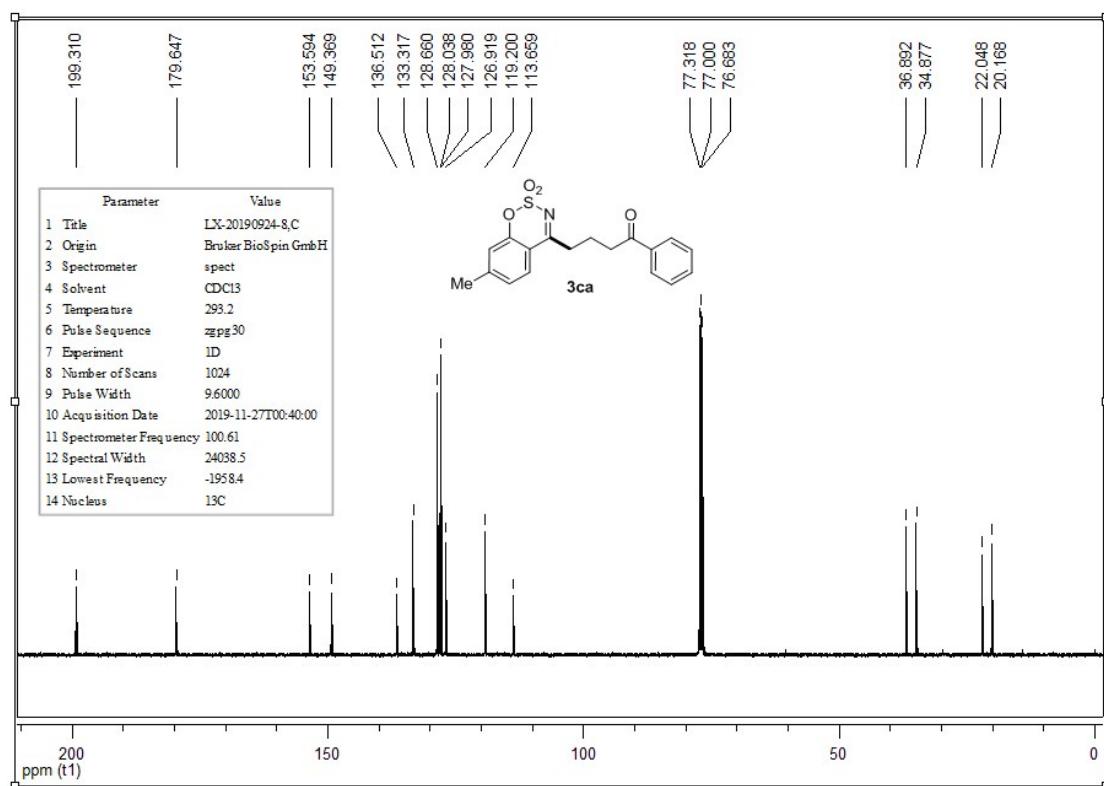
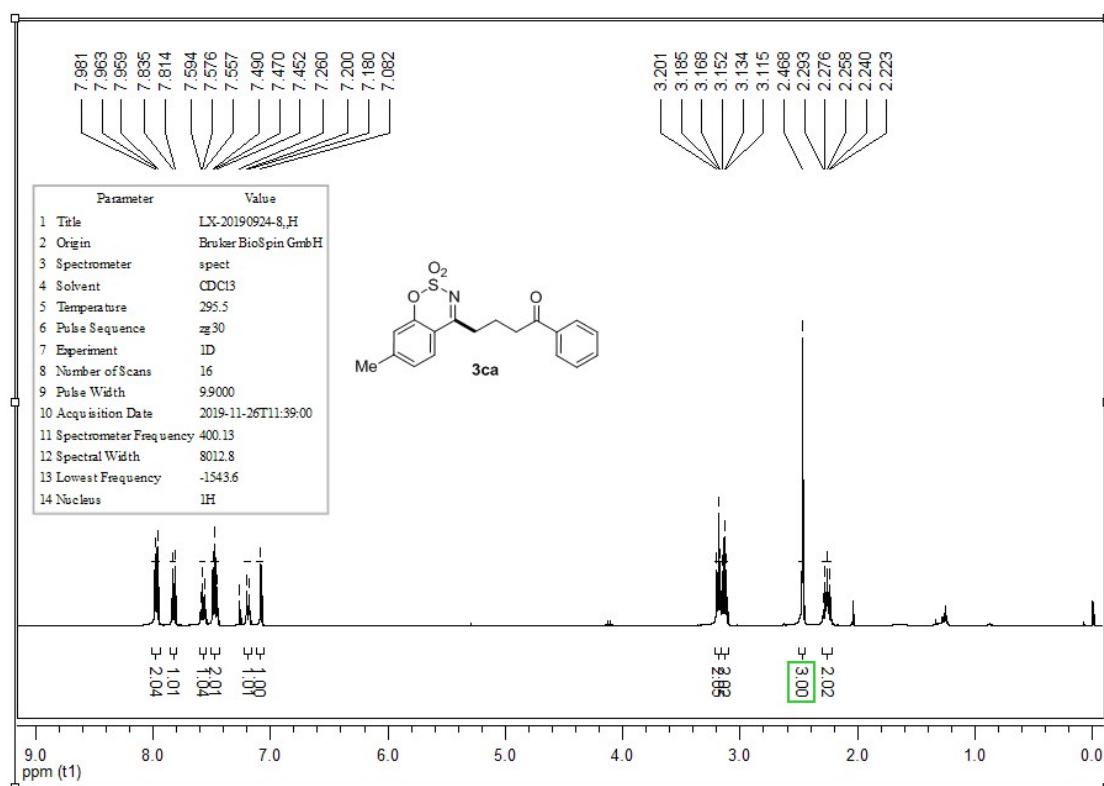
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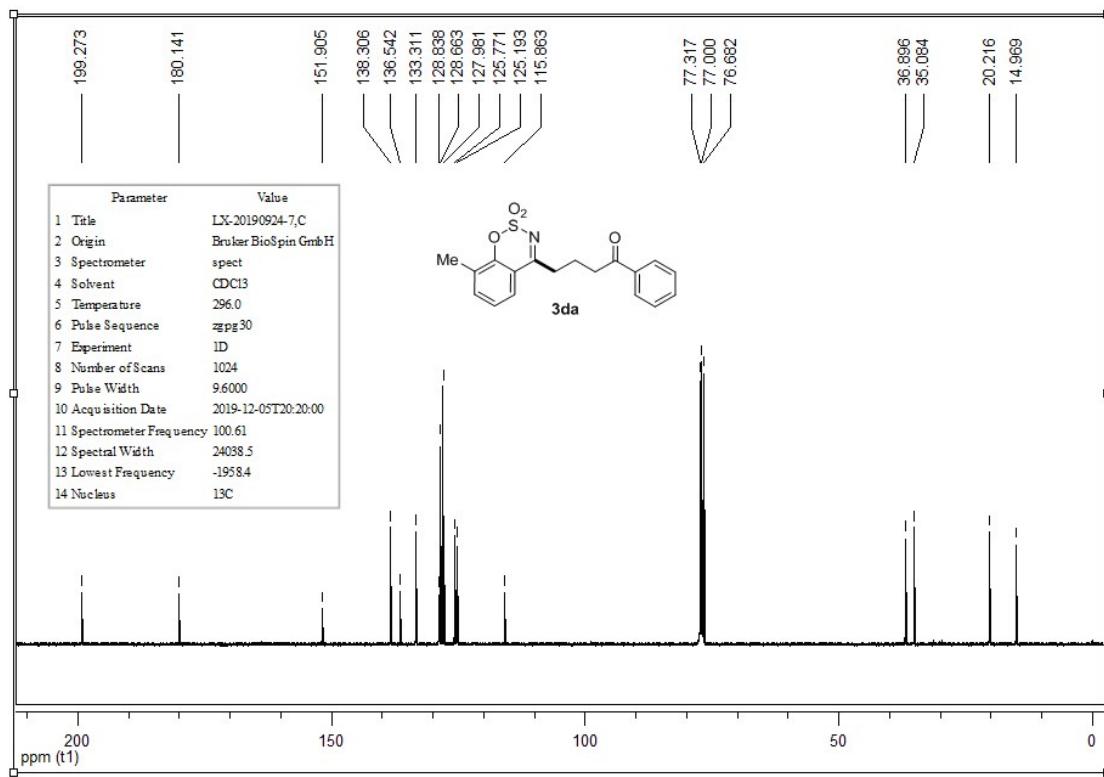
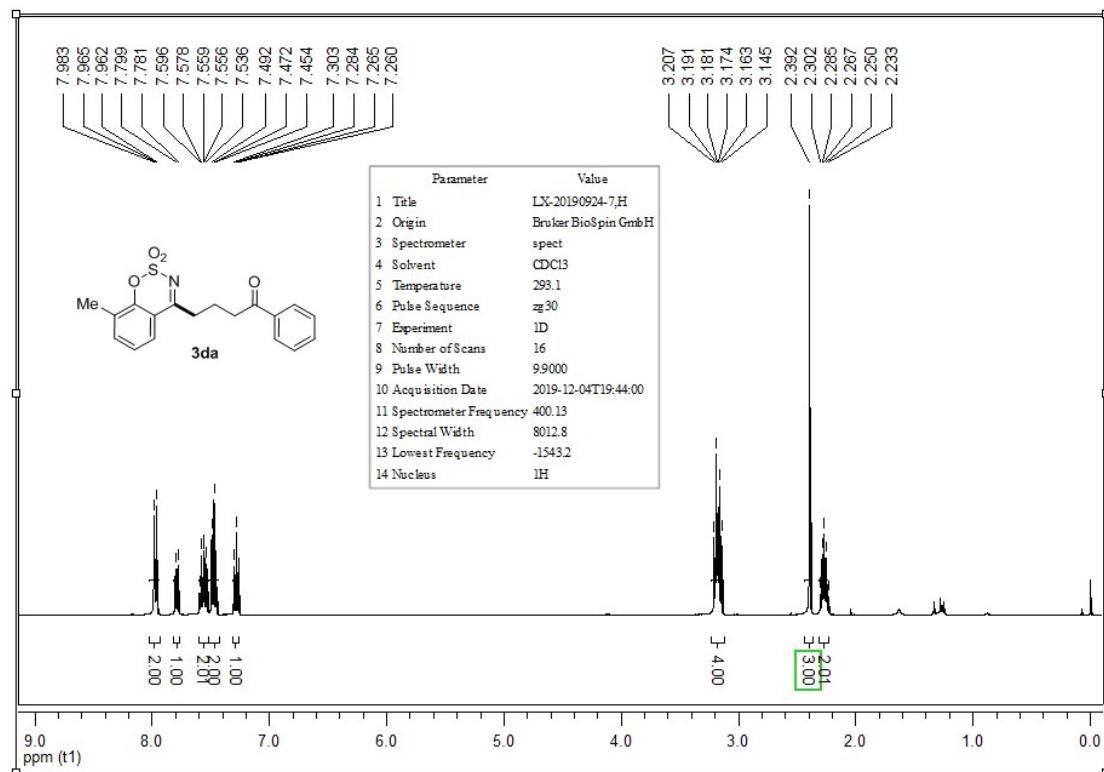
- [1] (a) N. D. Litvinas, B. H. Brodsky, J. D. Bois, *Angew. Chem. Int. Ed.*, **2009**, *48*, 4513. (b) H. Yu, L. Zhang, Z. Yang, Z. Li, Y. Zhao, Y. Xiao, H. Guo, *J. Org. Chem.*, **2013**, *78*, 8427.
- [2] (a) J. Jiao , L. X. Nguyen, D. R. Patterson, R. A. Flowers, *Org. Lett.*, **2007**, *9*, 1323–1326. (b) Y.-F. Wang, S. Chiba, *J. Am. Chem. Soc.*, **2009**, *131*, 12570–12572. (c) A. Ilangovan, S. Saravanakumar, S. Malayappasamy, *Org. Lett.*, **2013**, *15*, 19, 4968–4971. (d) B. M. Casey, C. A. Eakin, R. A. Flowers II, *Tetrahedron Lett.*, **2009**, *50*, 1264–1266. (e) R. Ren, H. Zhao, L. Huan, C. Zhu, *Angew. Chem. Int. Ed.*, **2015**, *54*, 12692–12696.

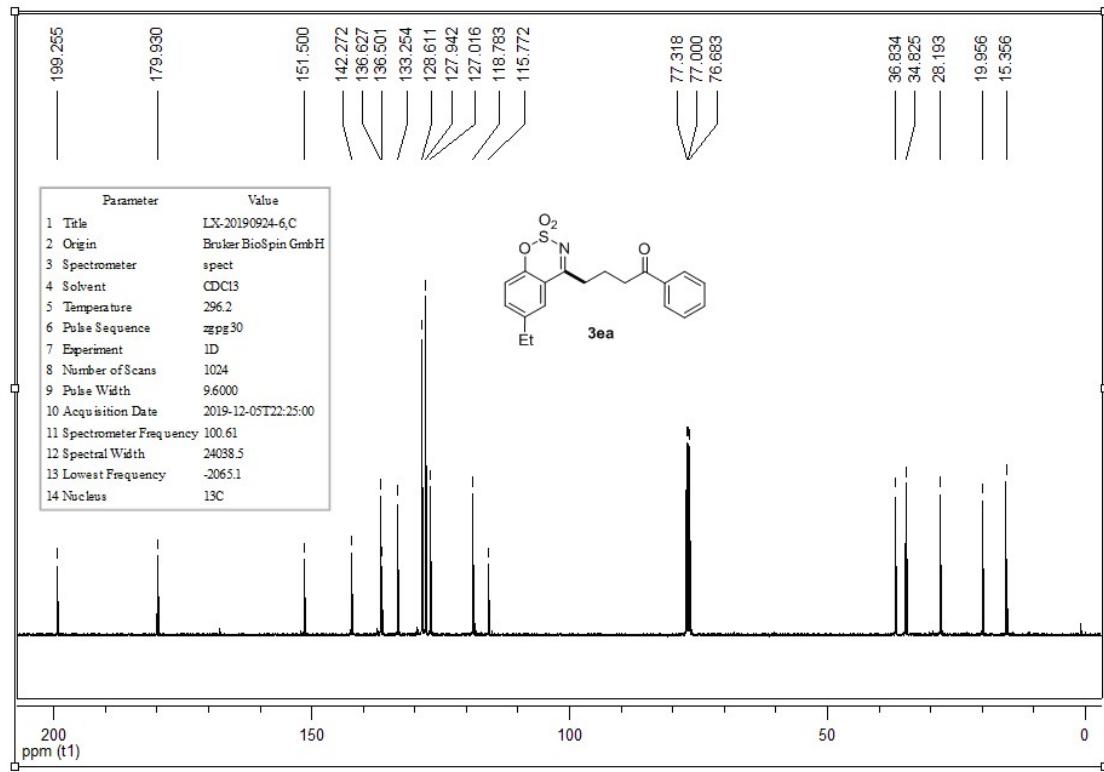
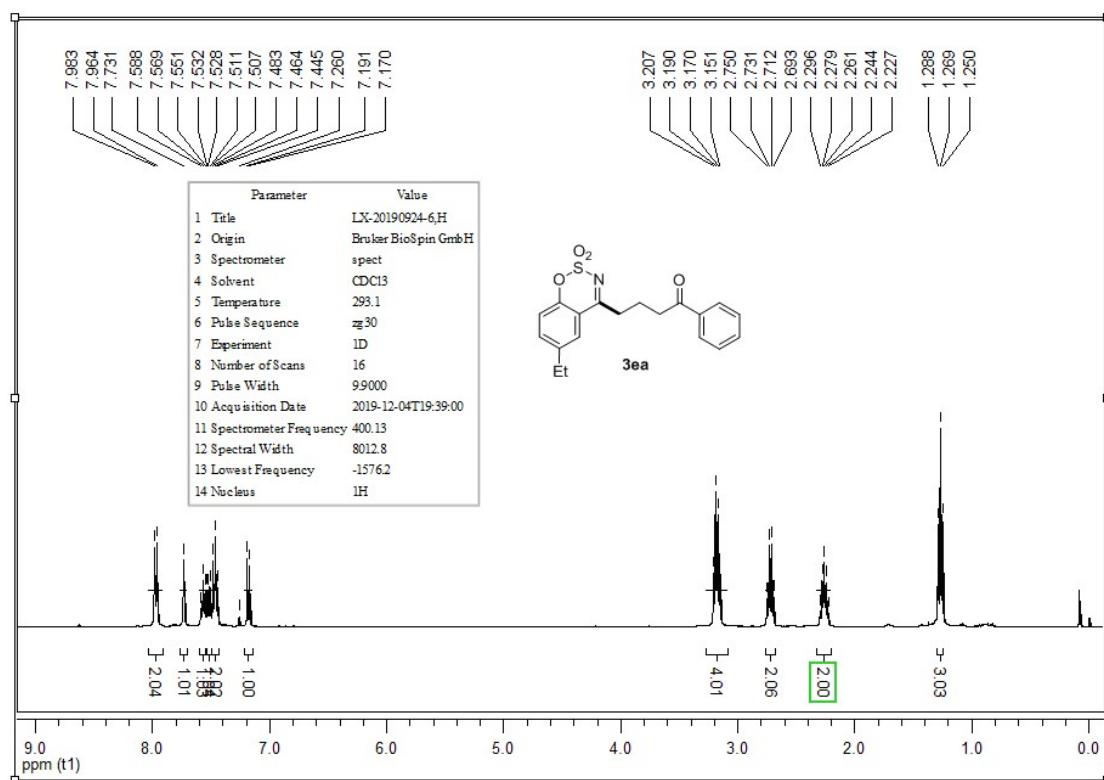
Copies of NMR spectra of the products:

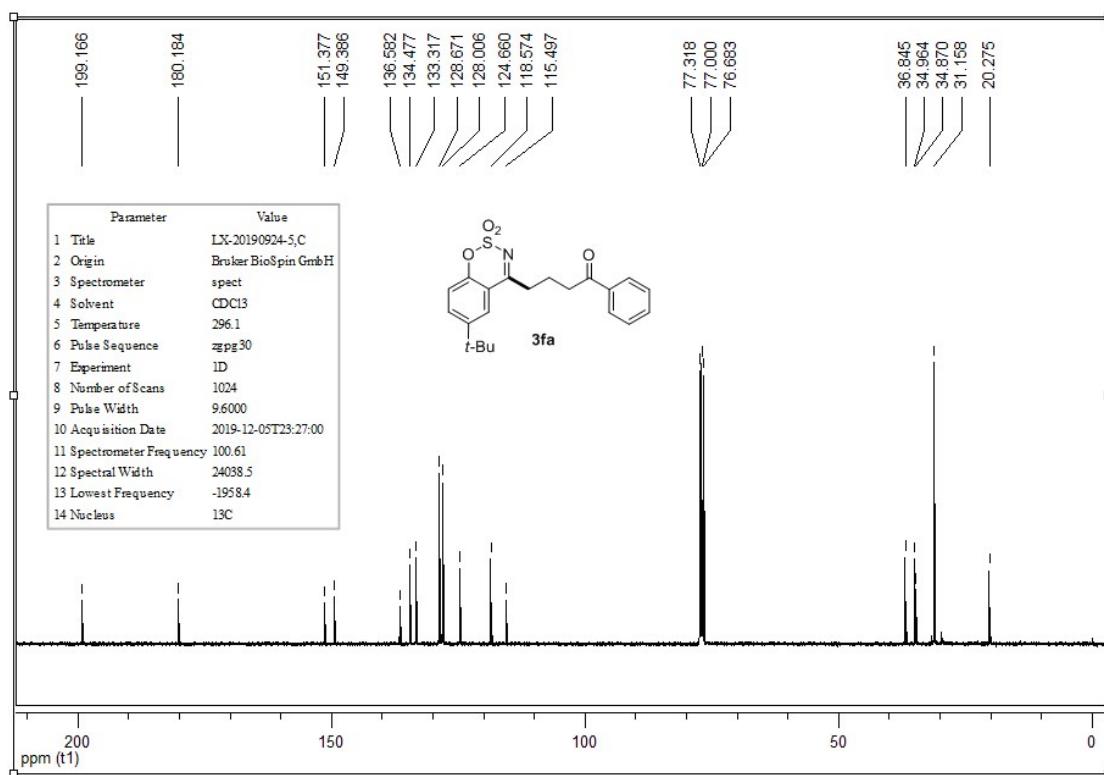
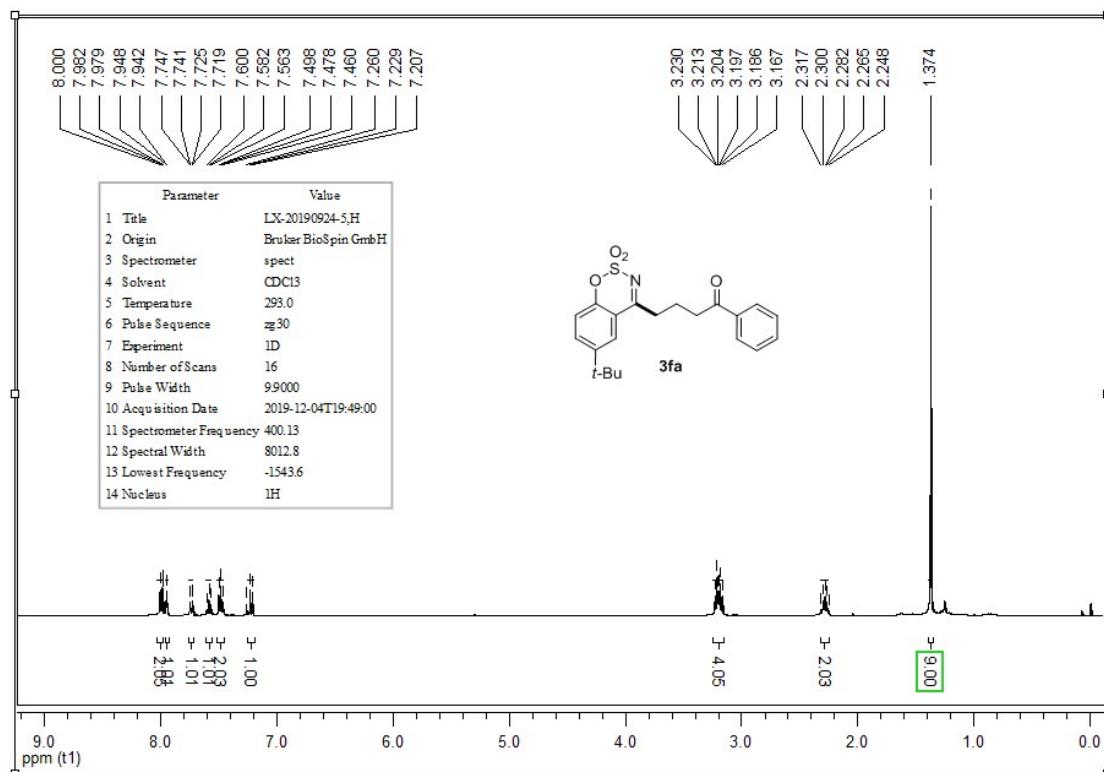


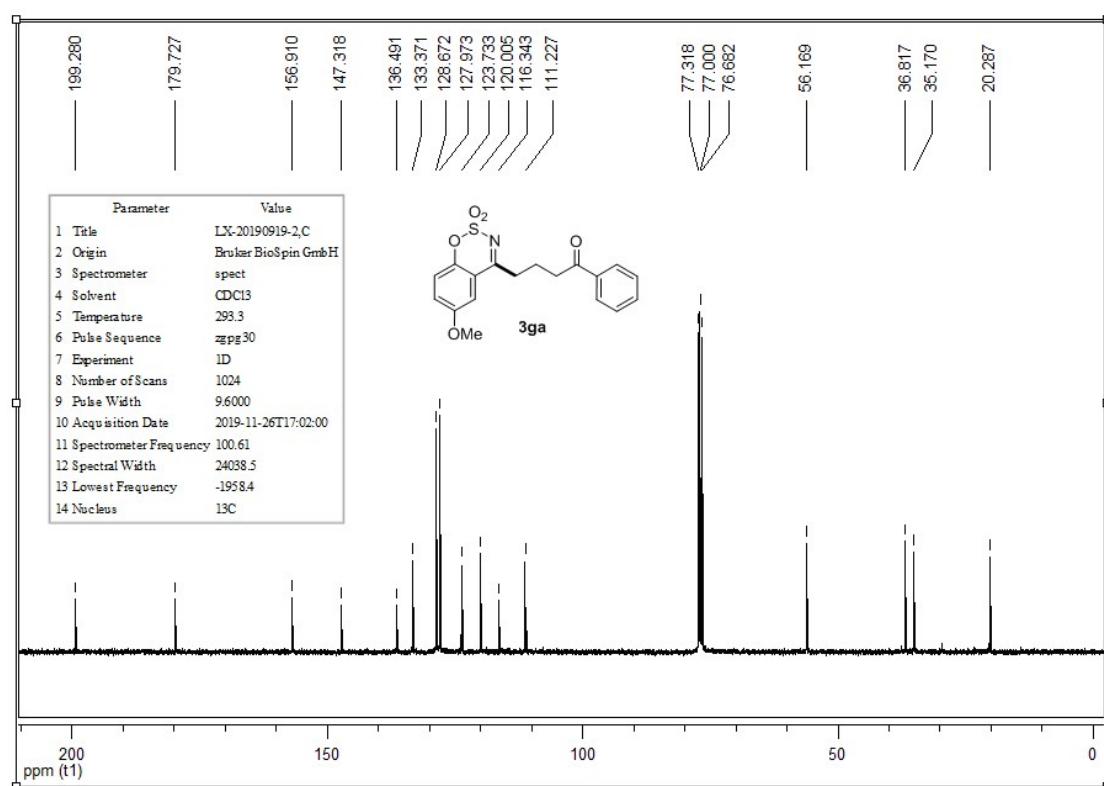
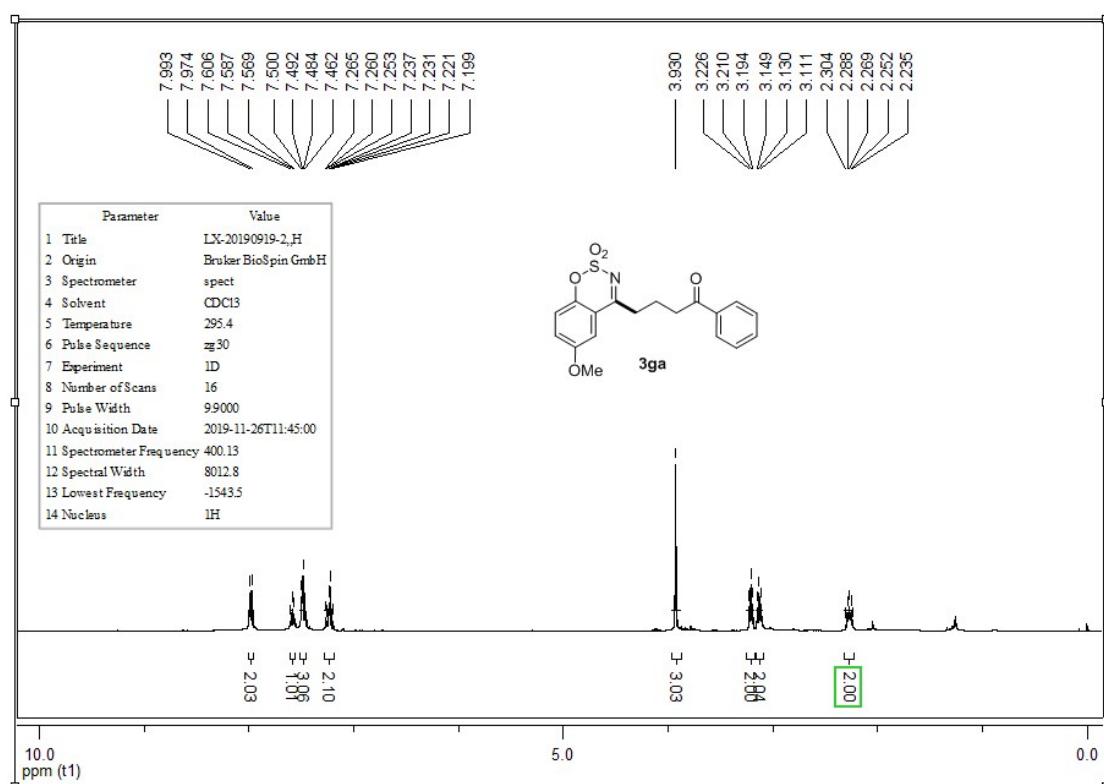


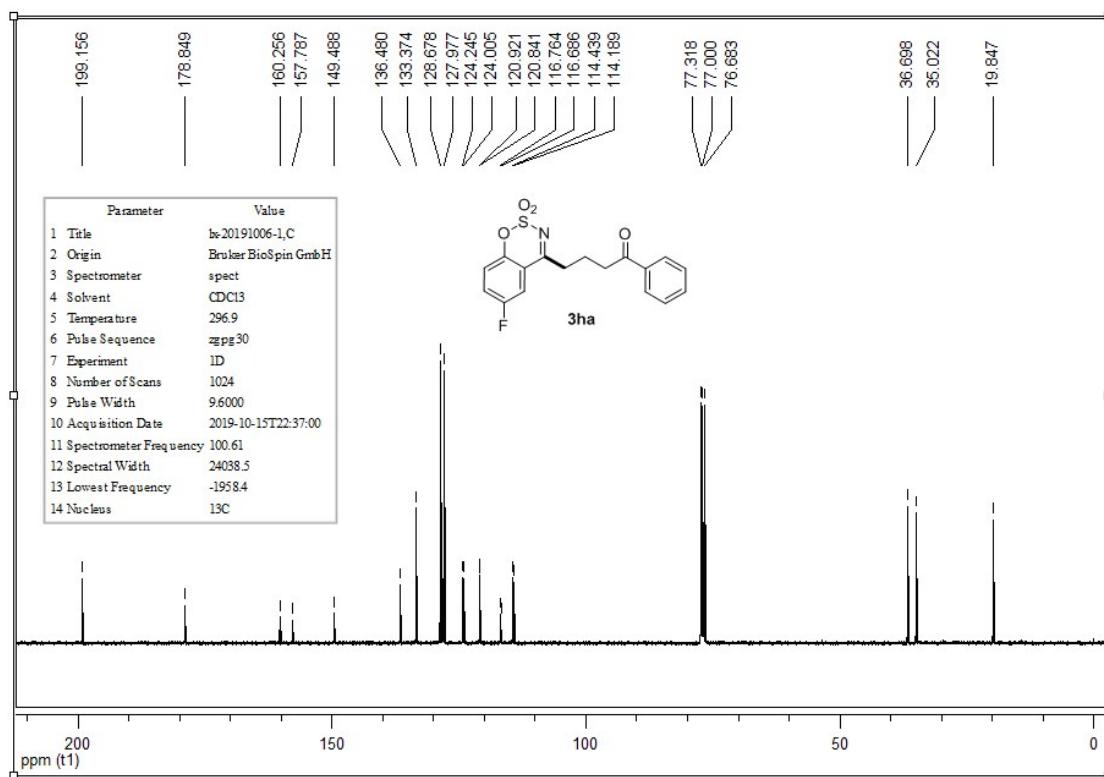
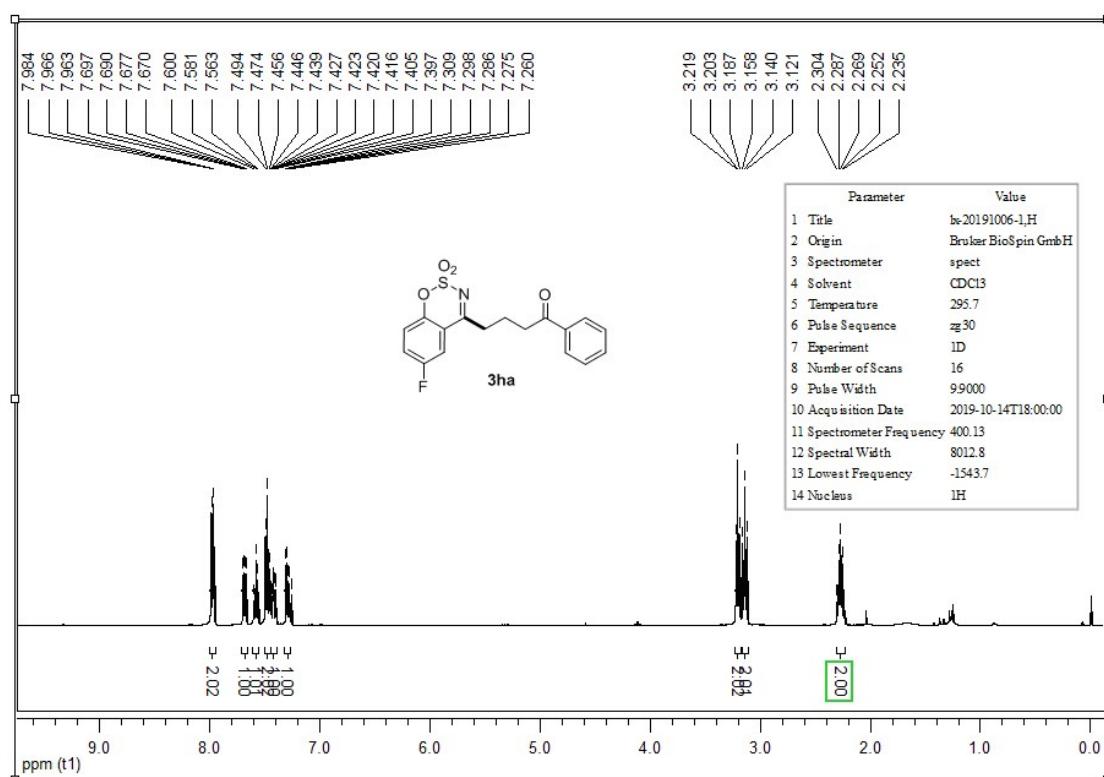


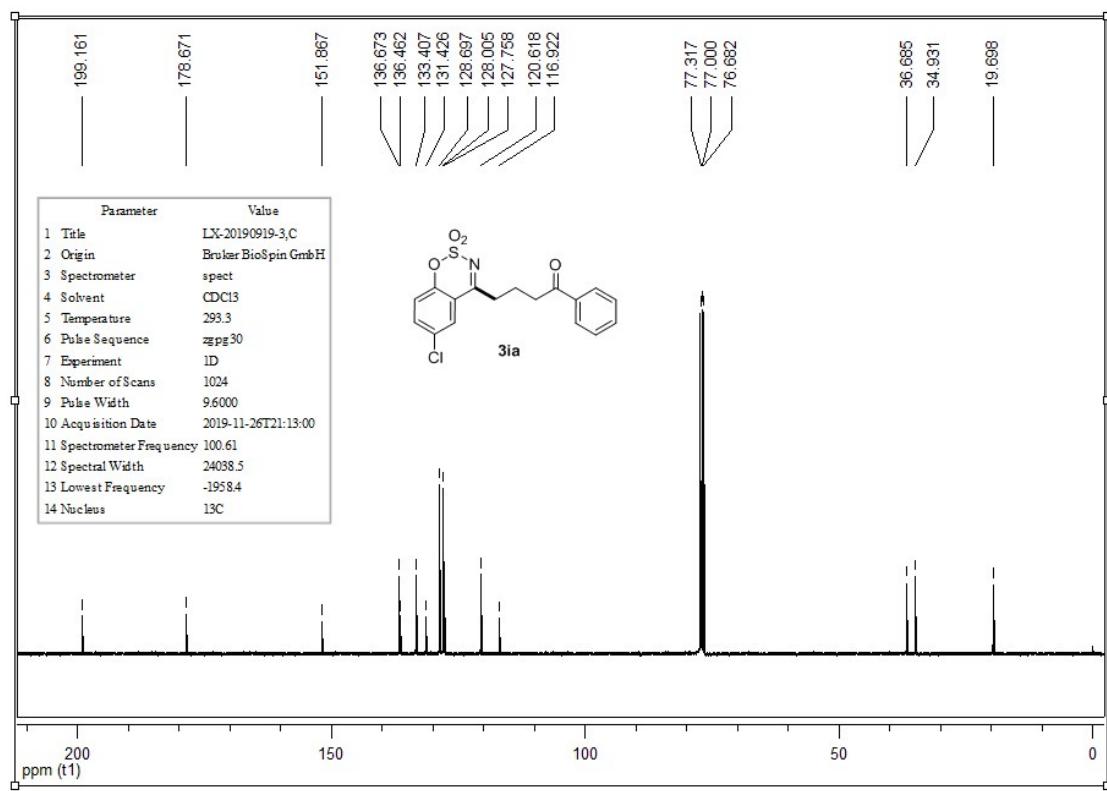
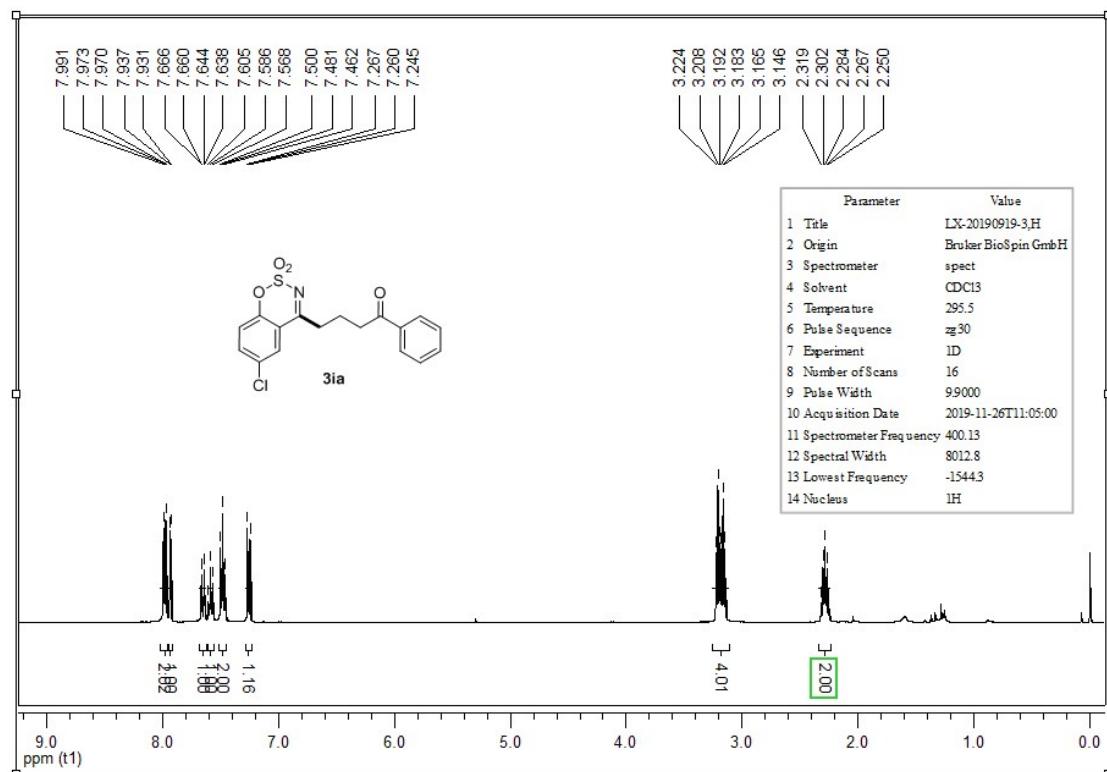


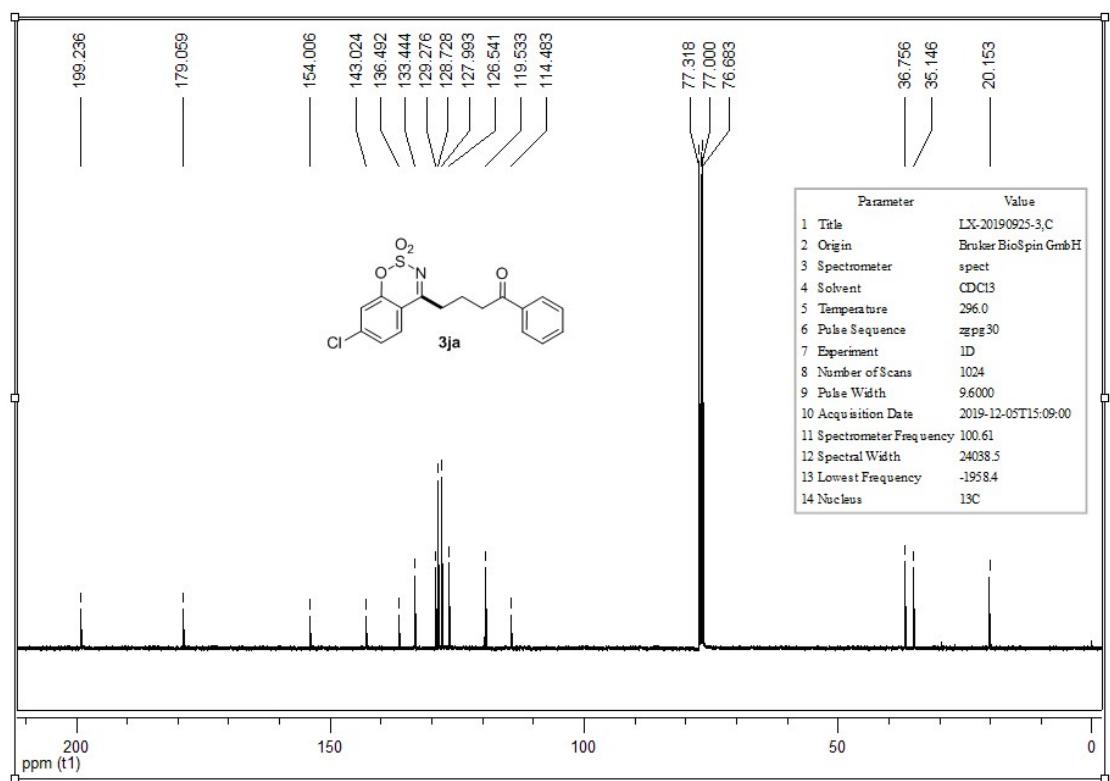
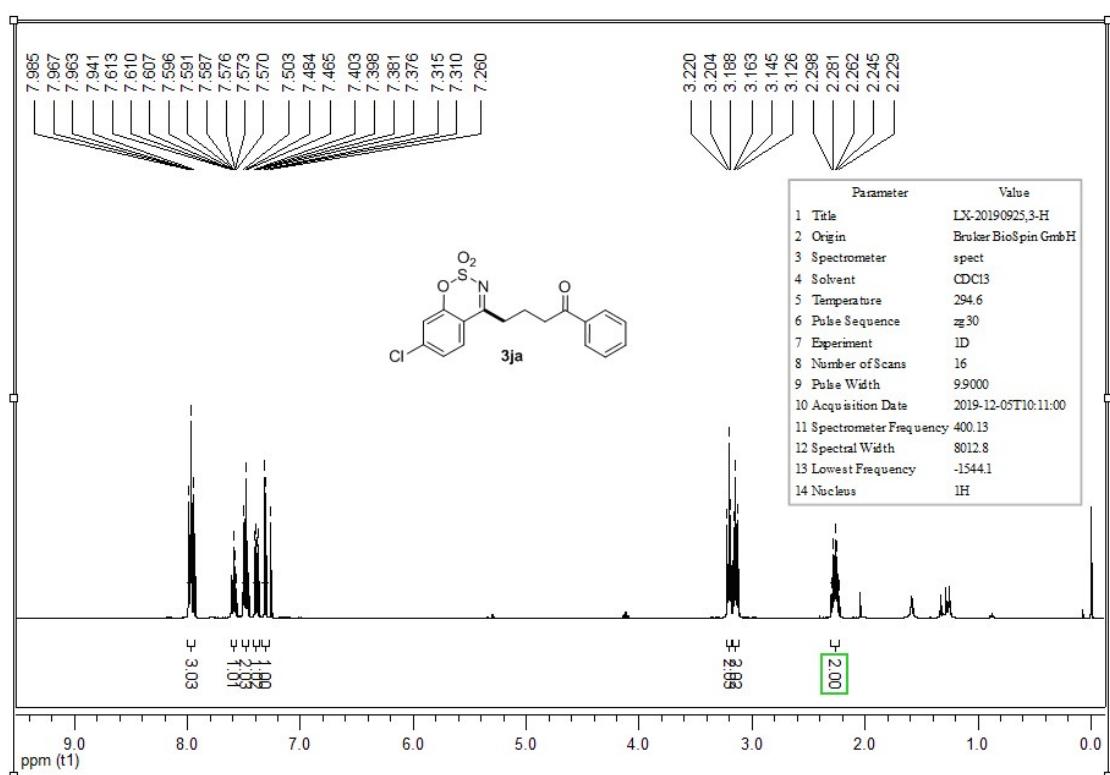


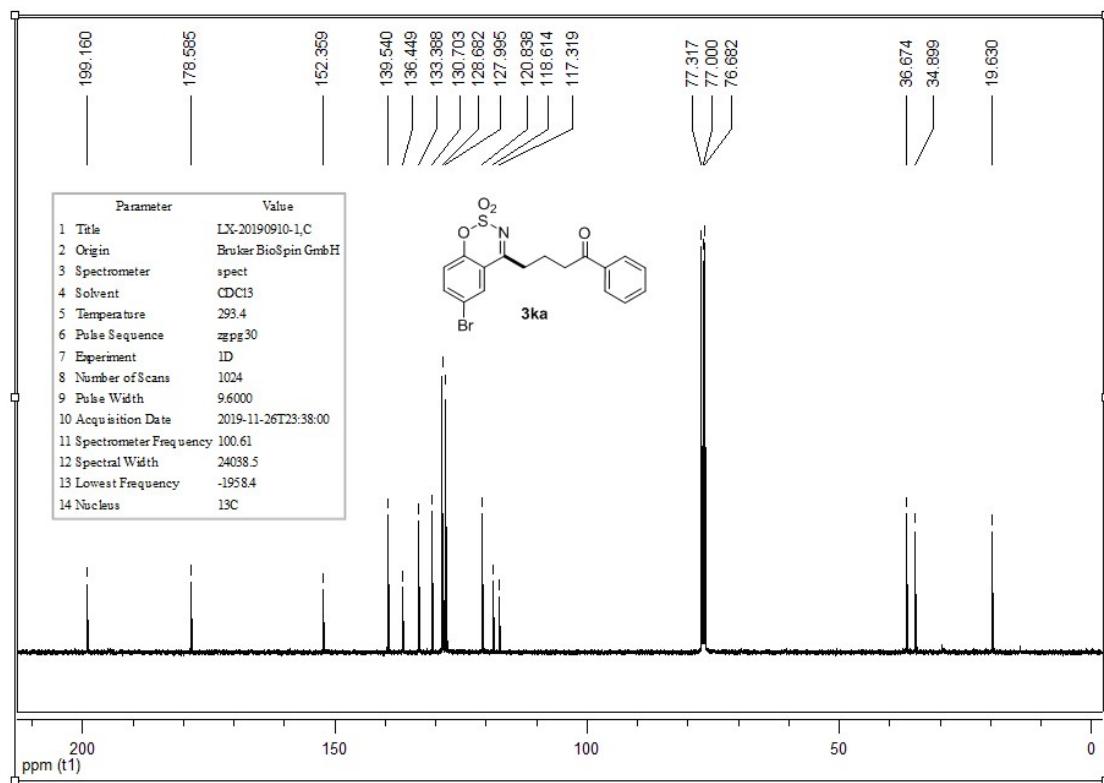
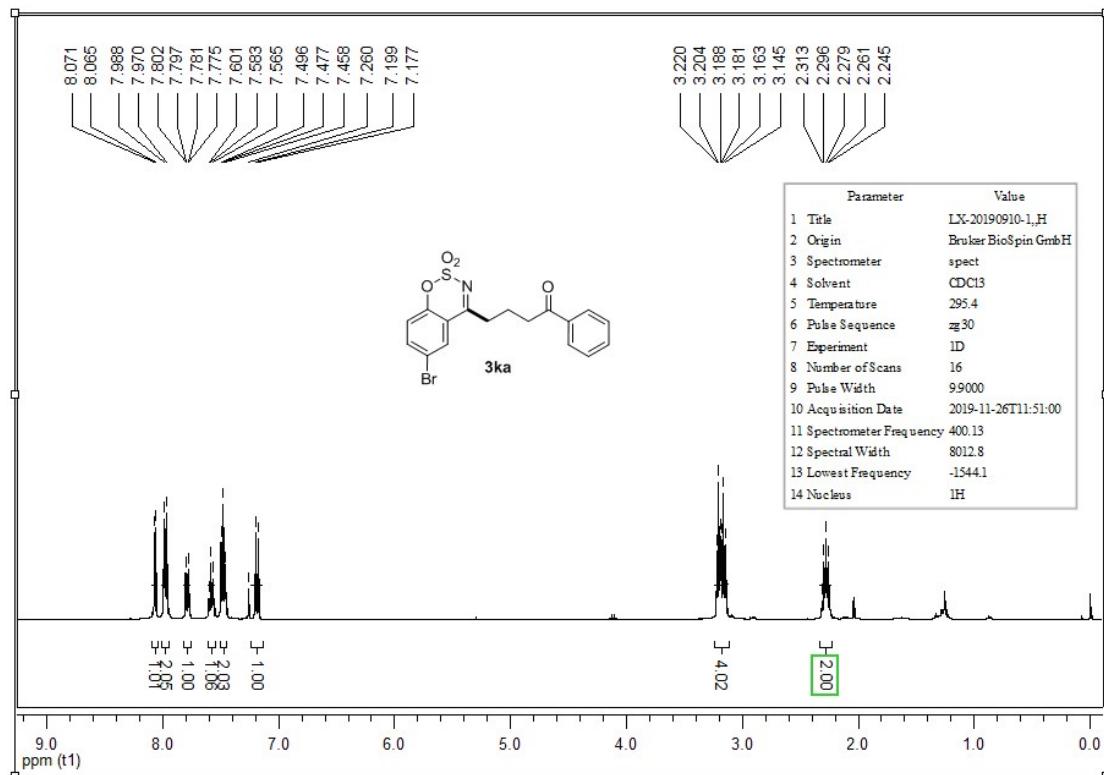


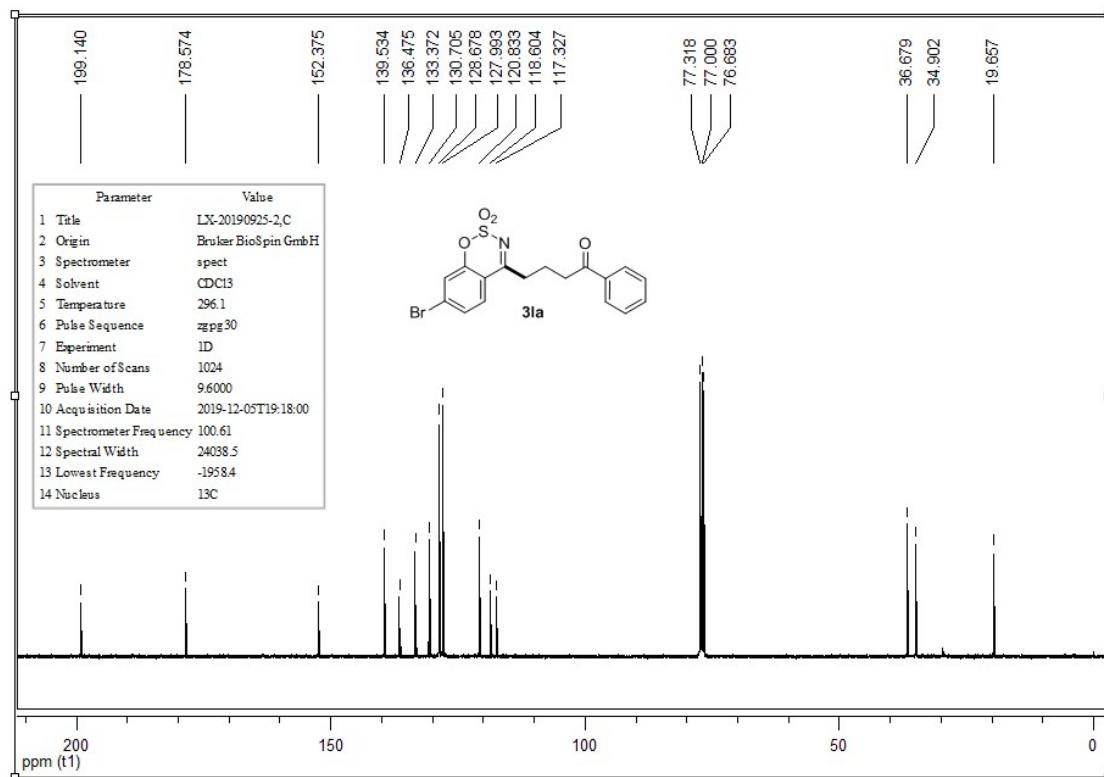
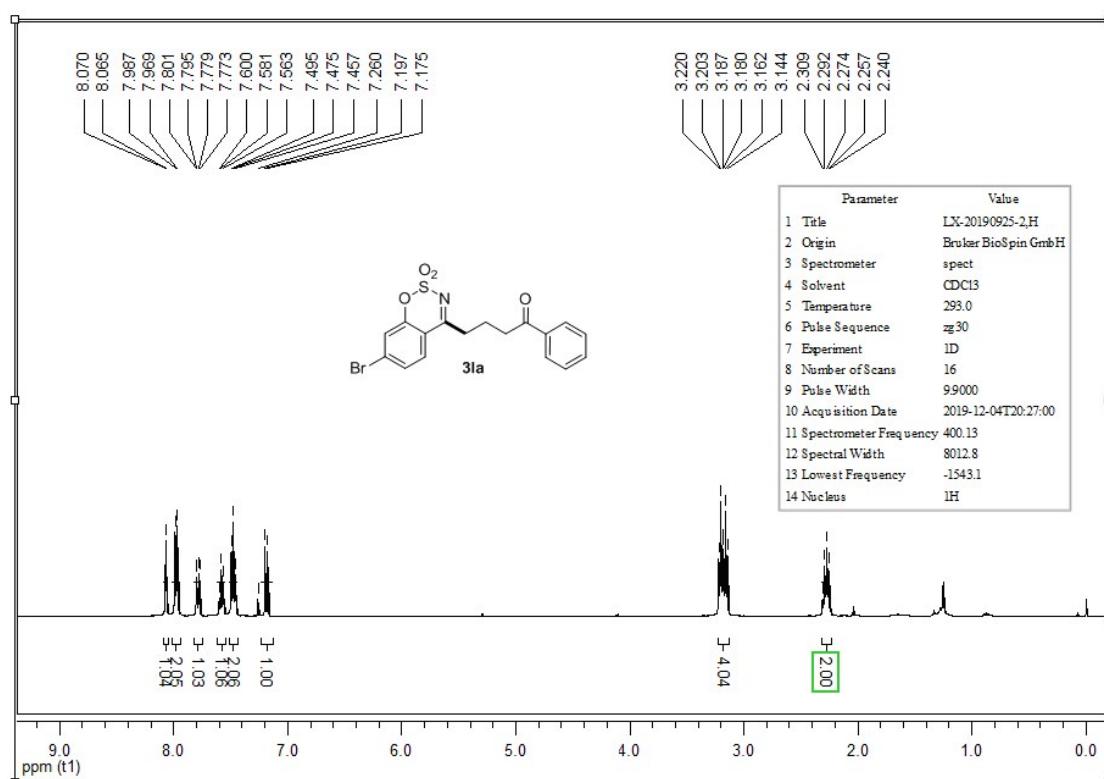


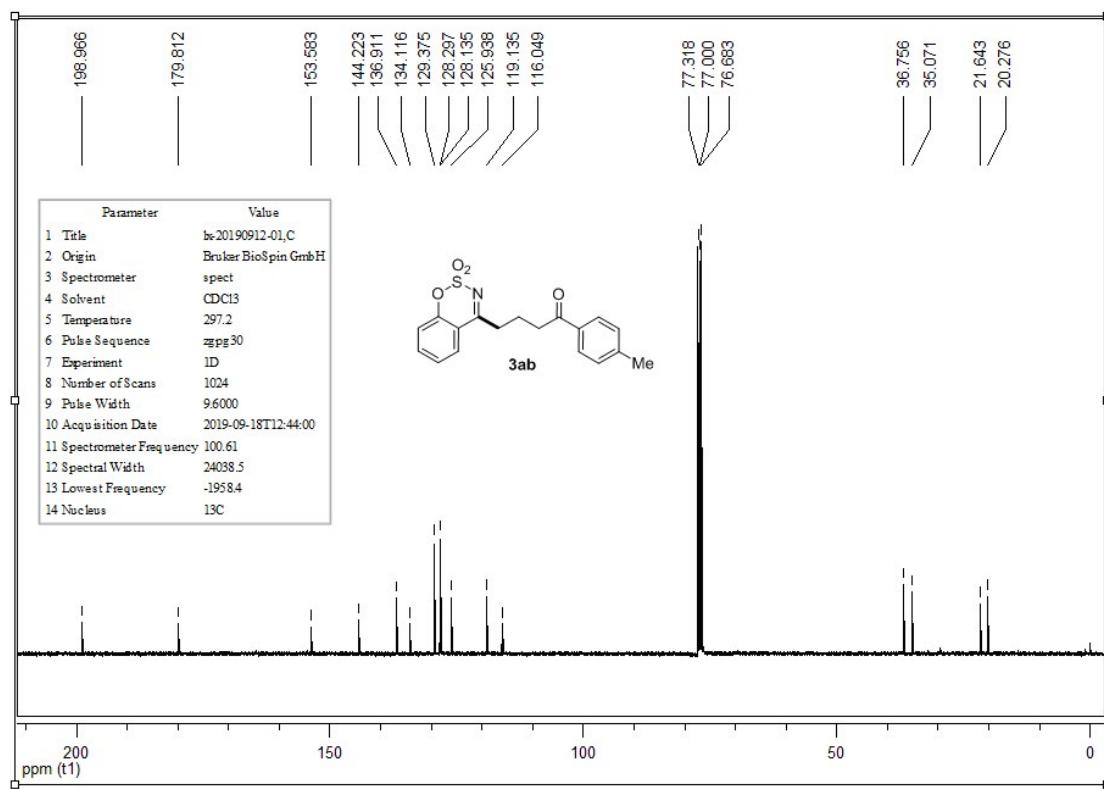
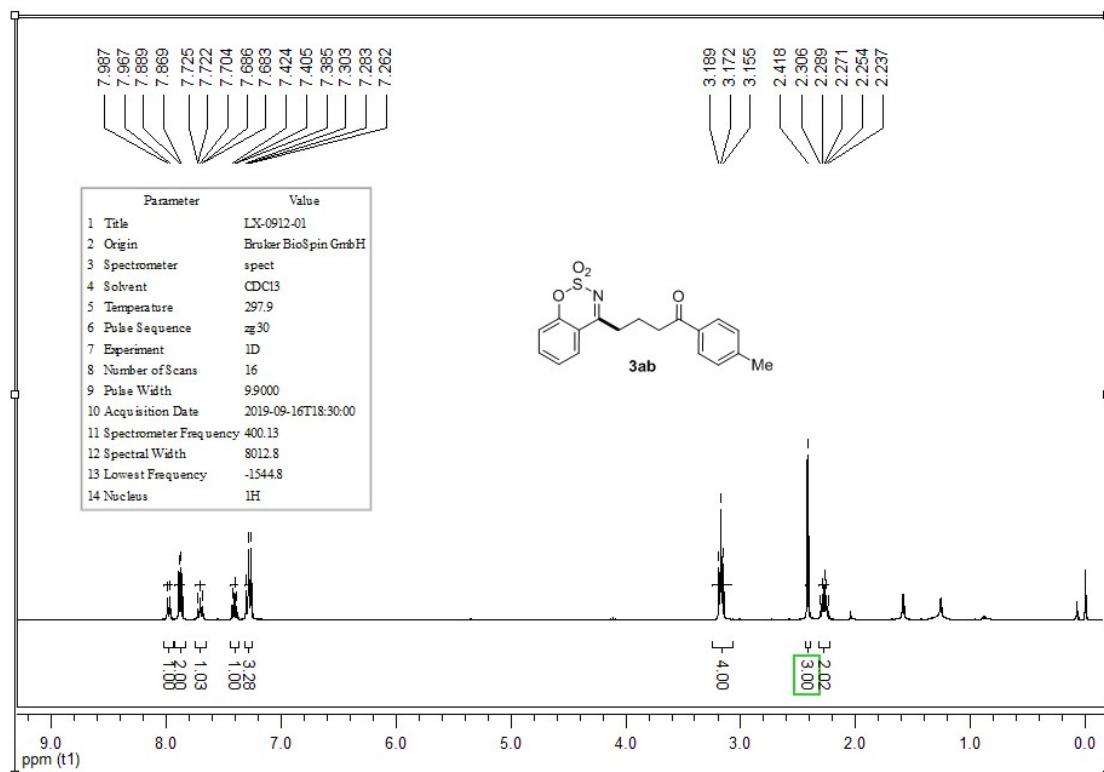


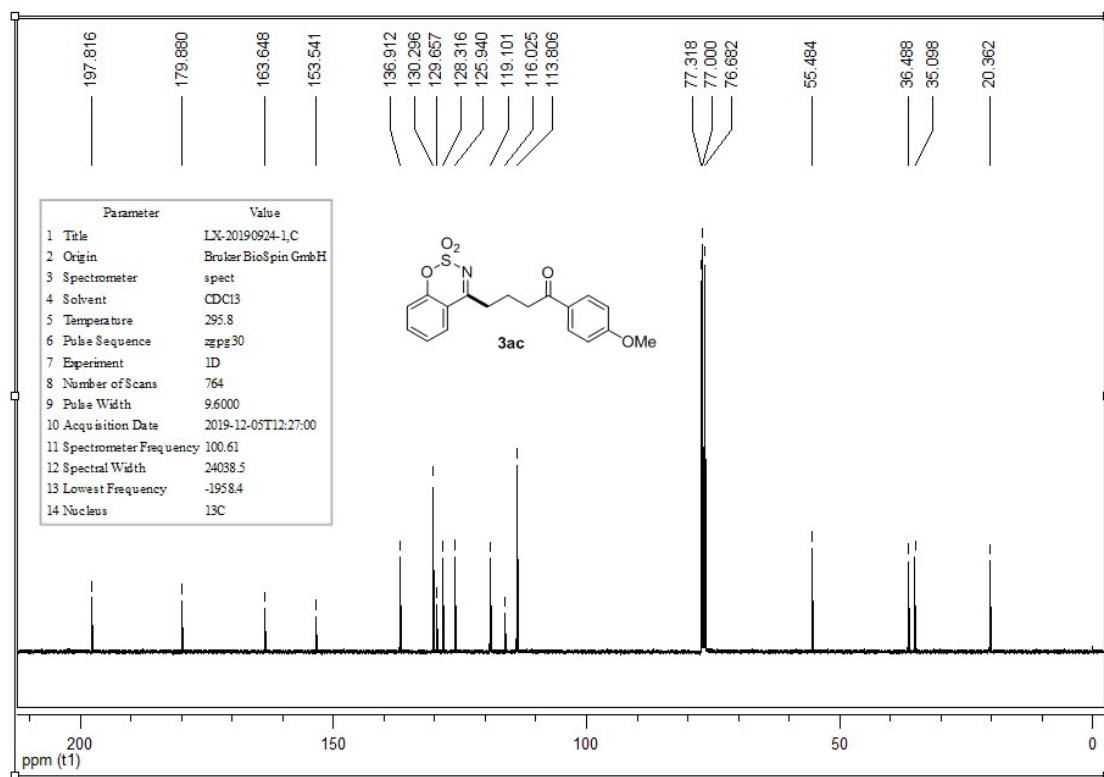
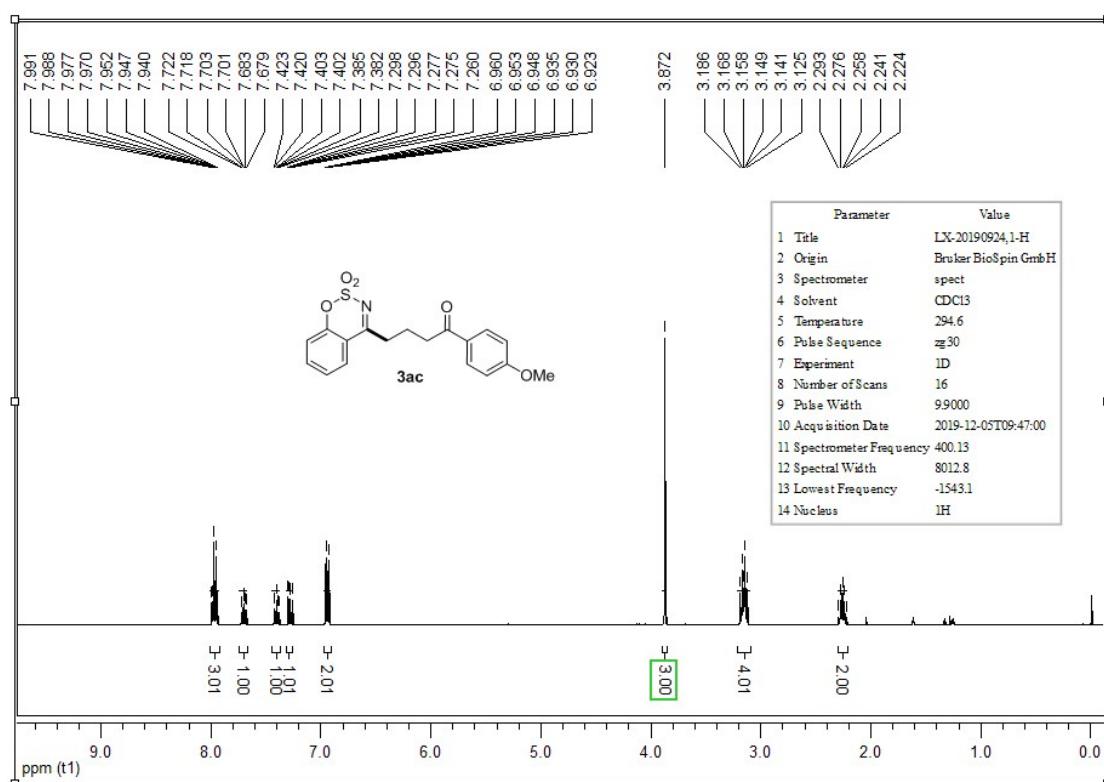


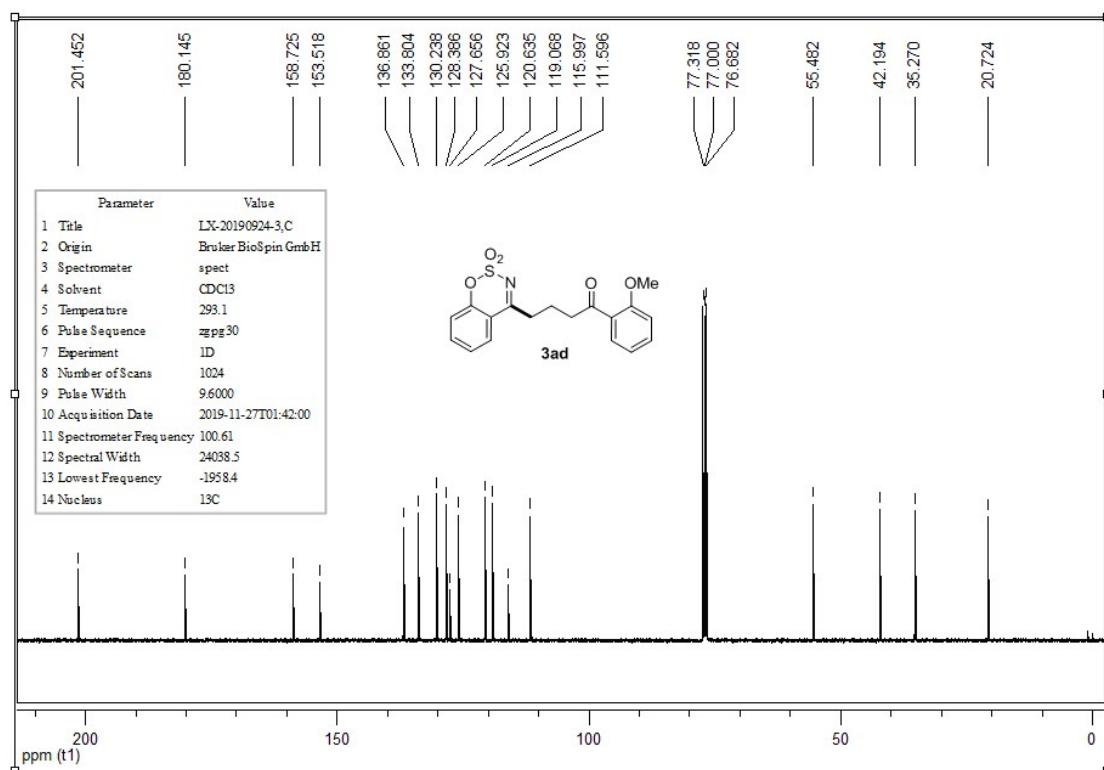
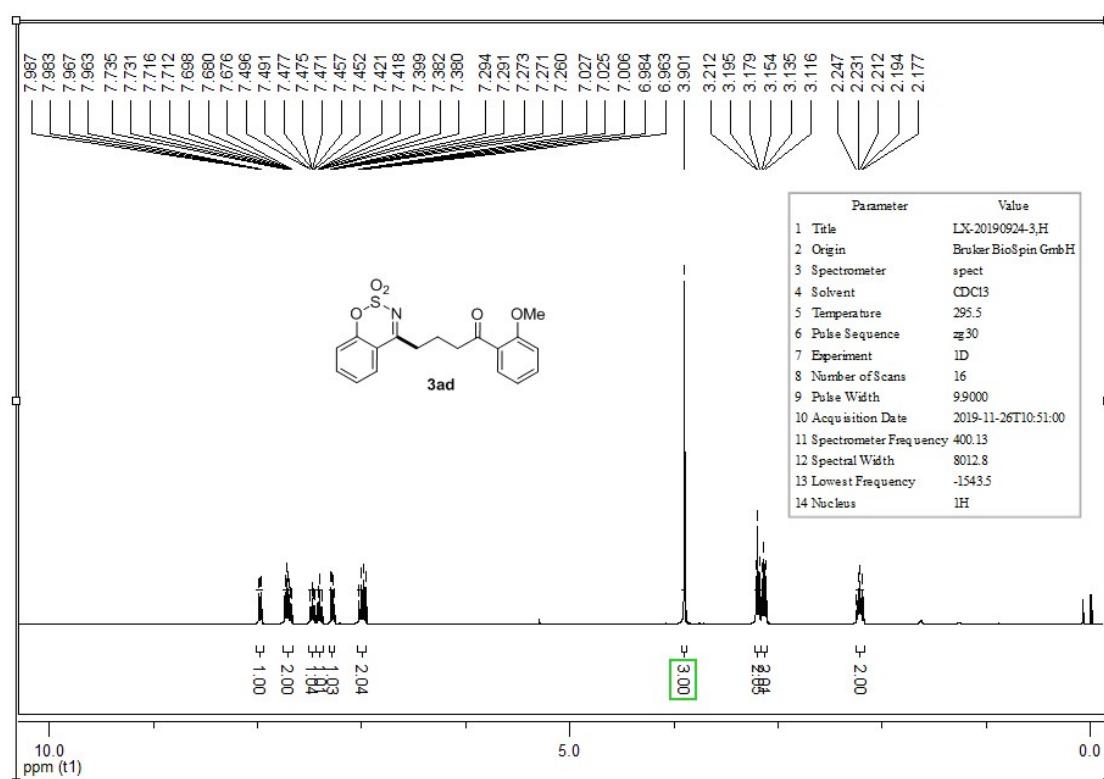


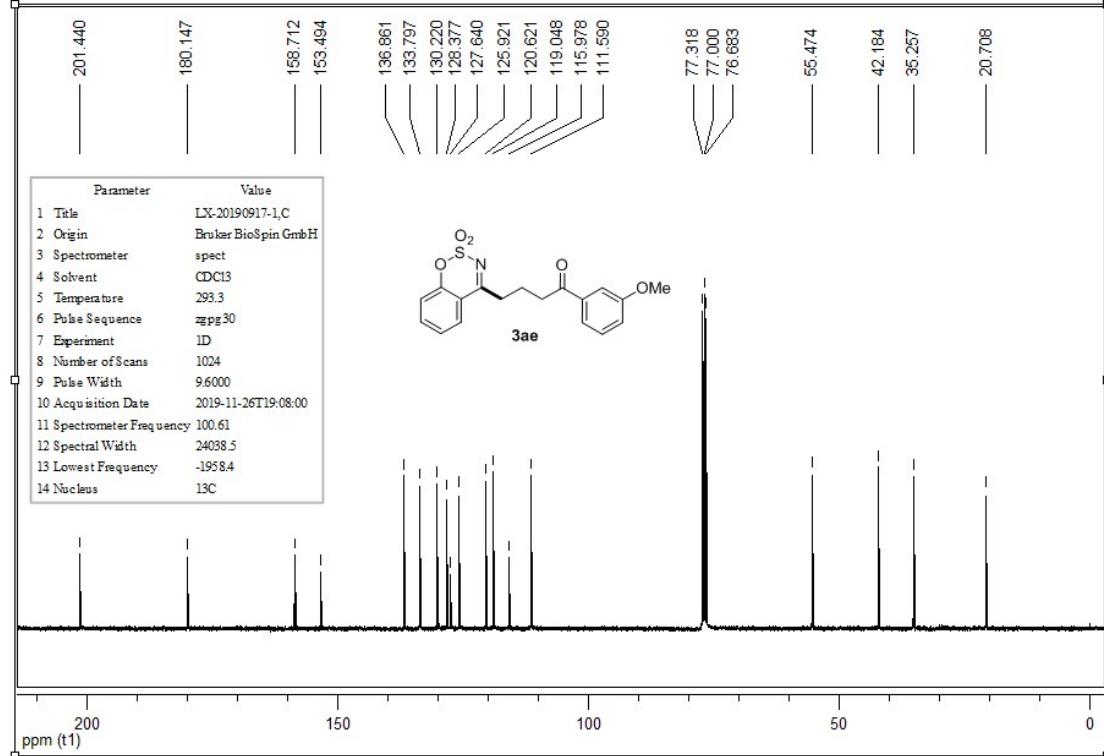
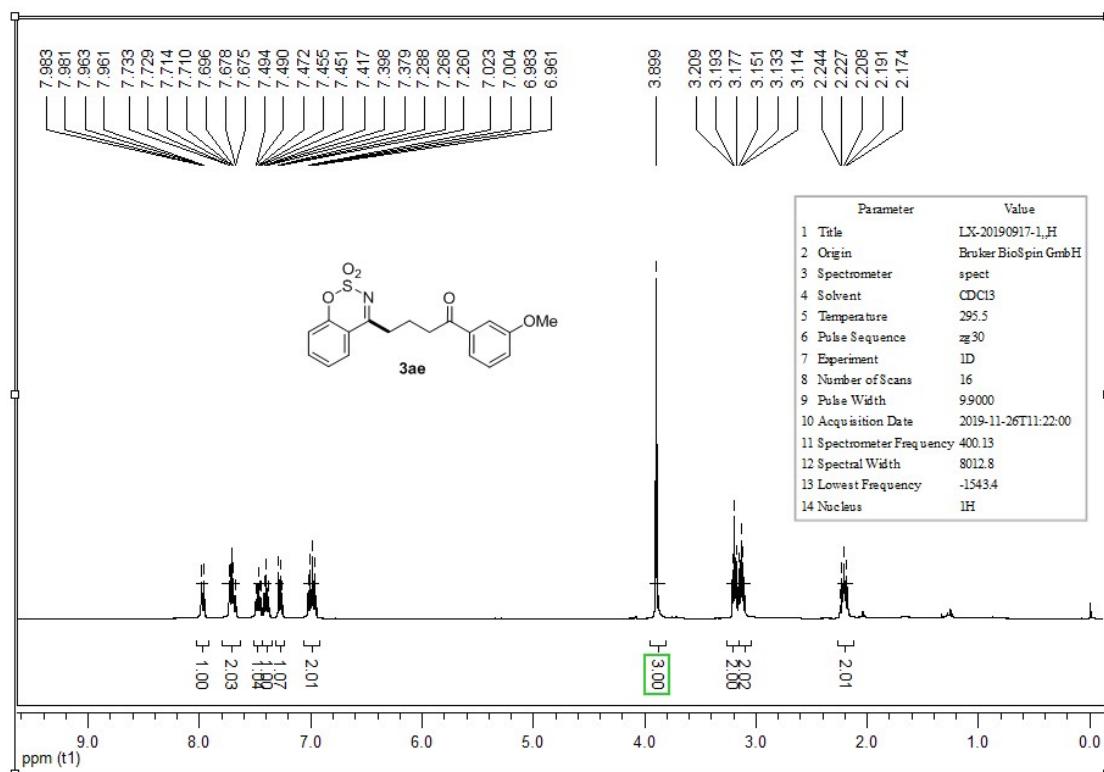


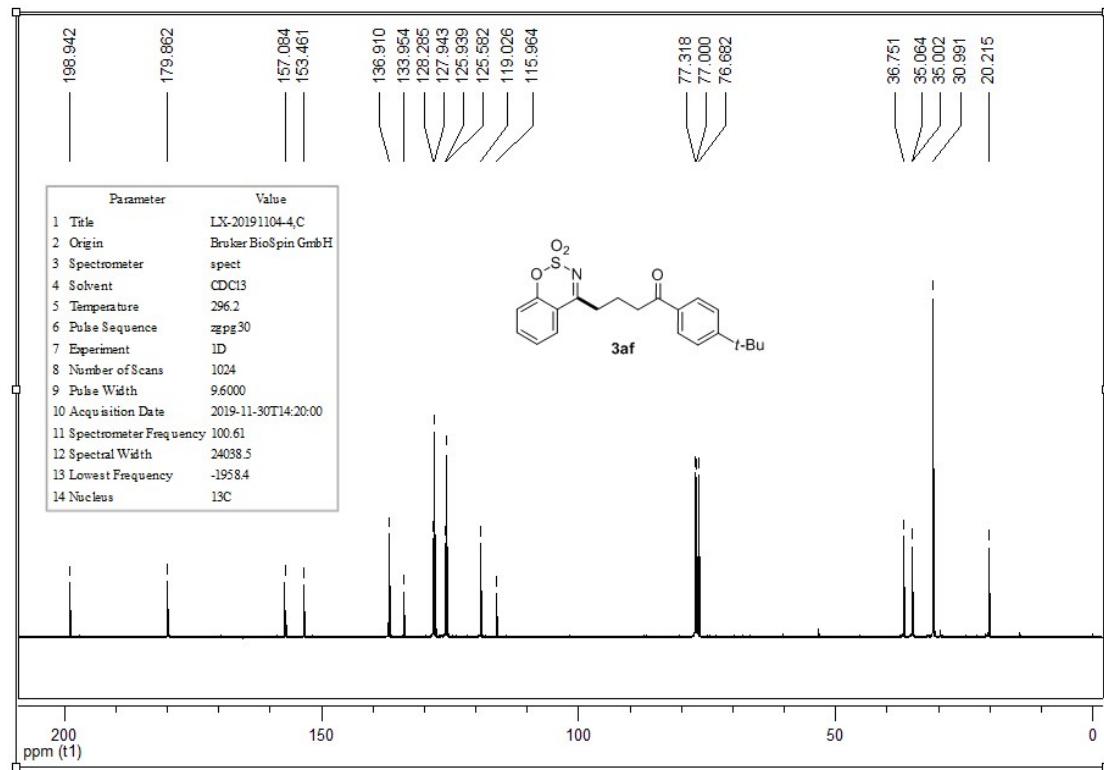
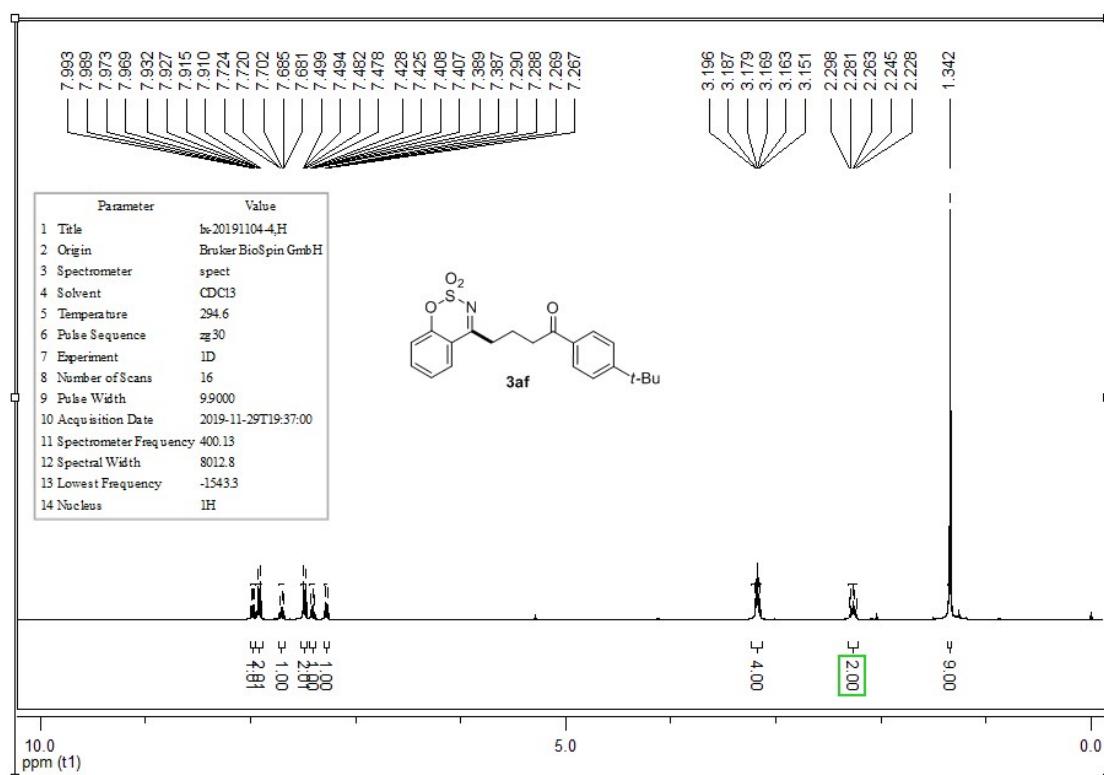


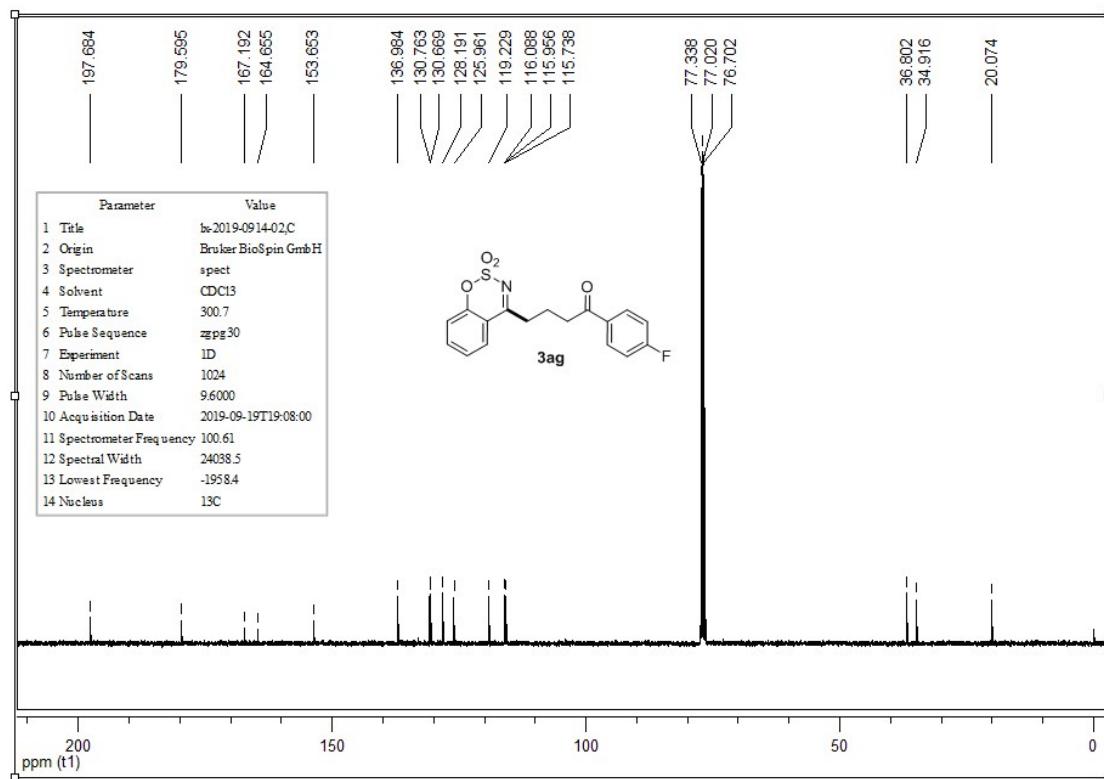
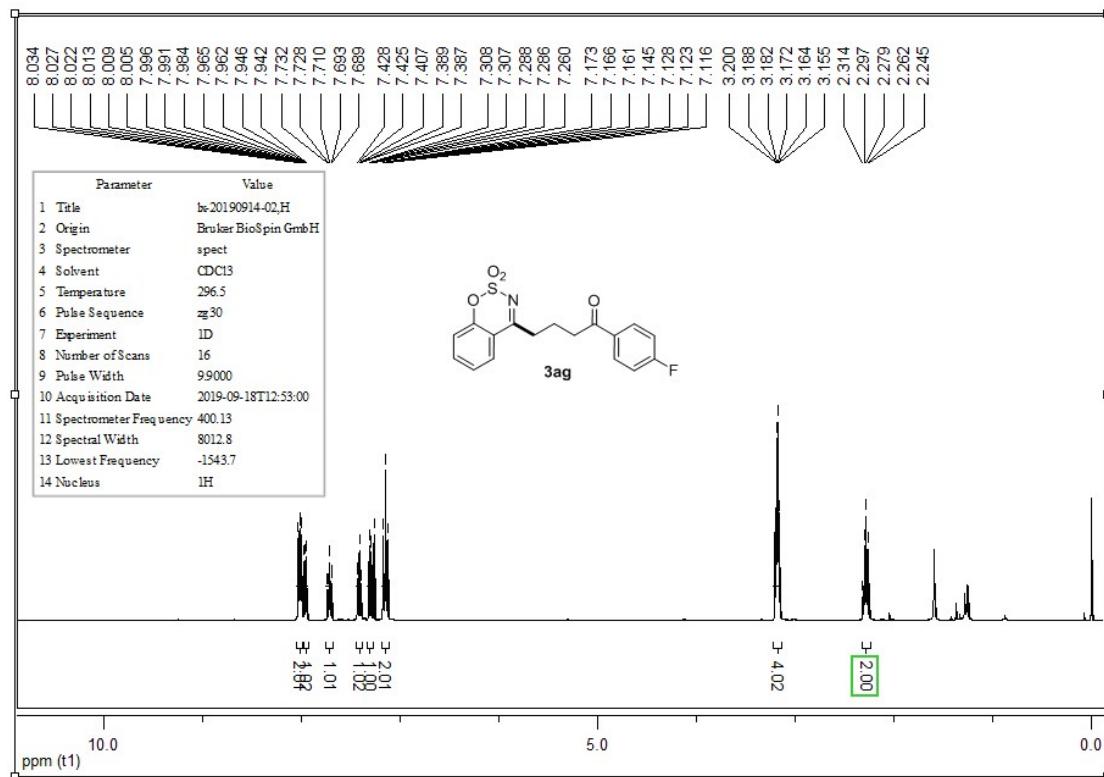


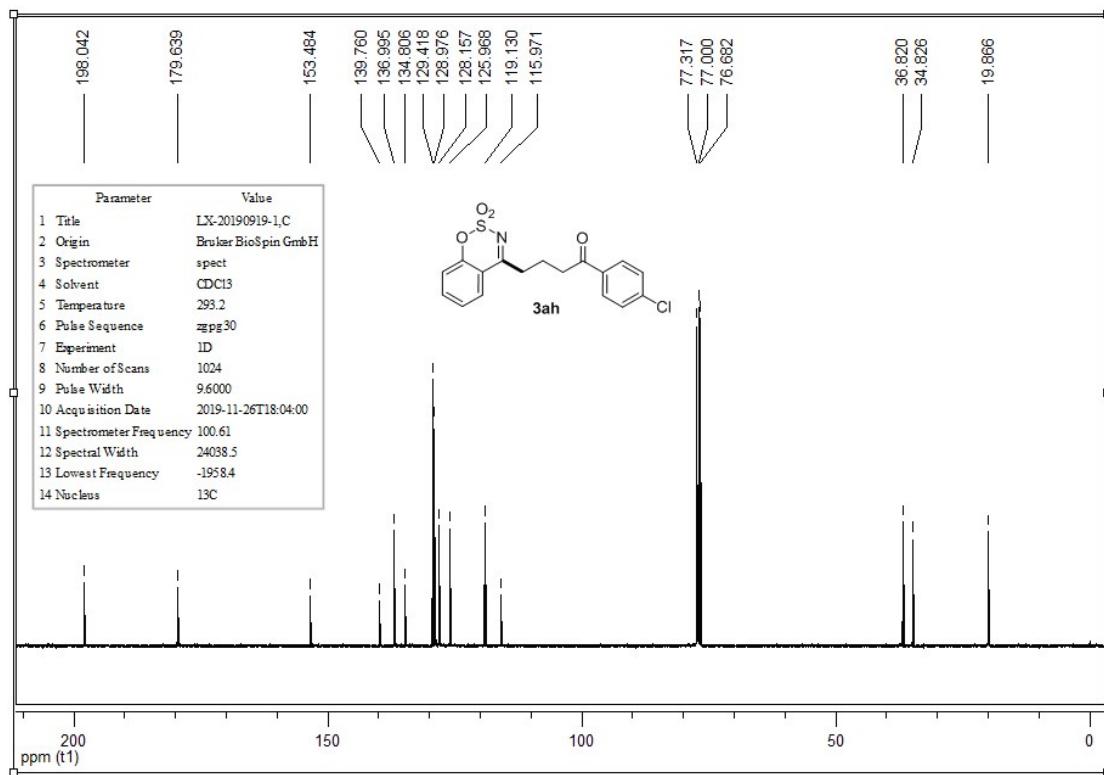
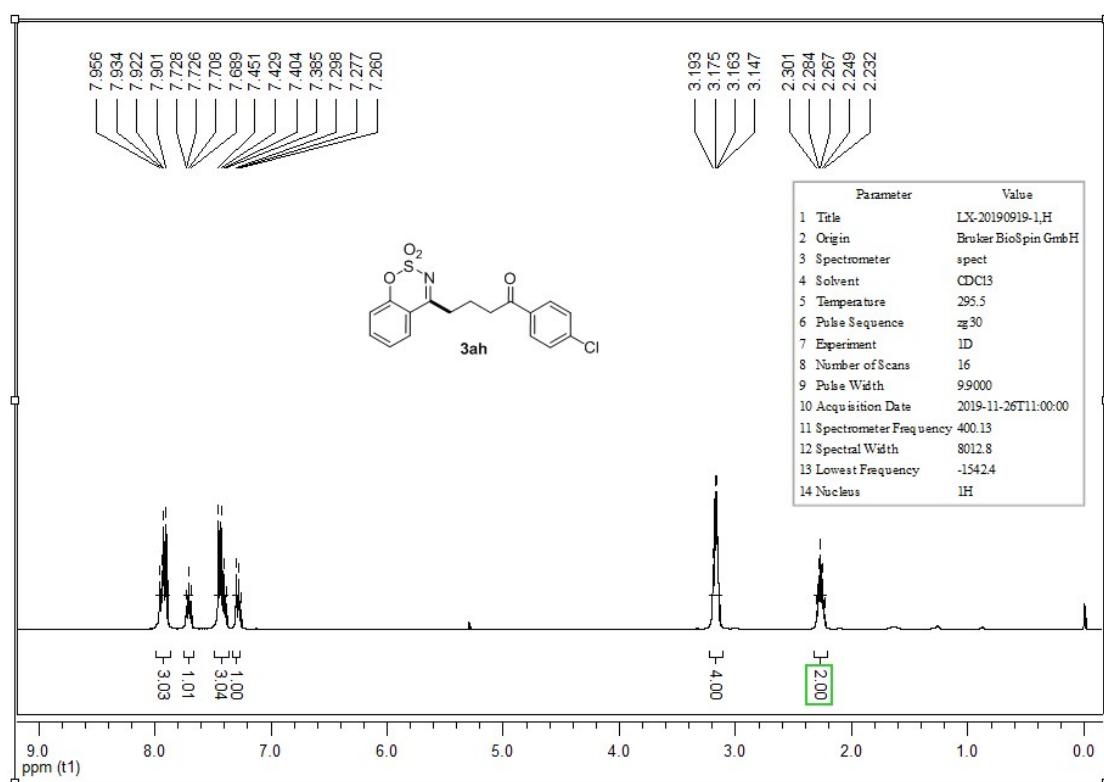


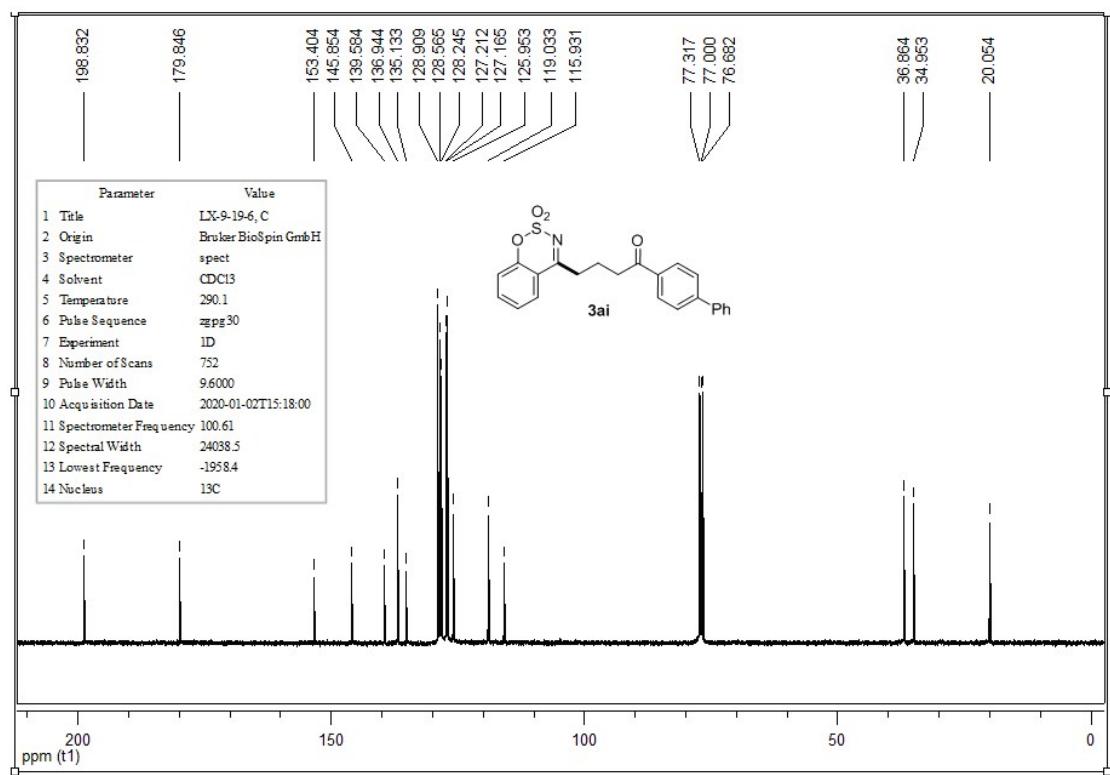
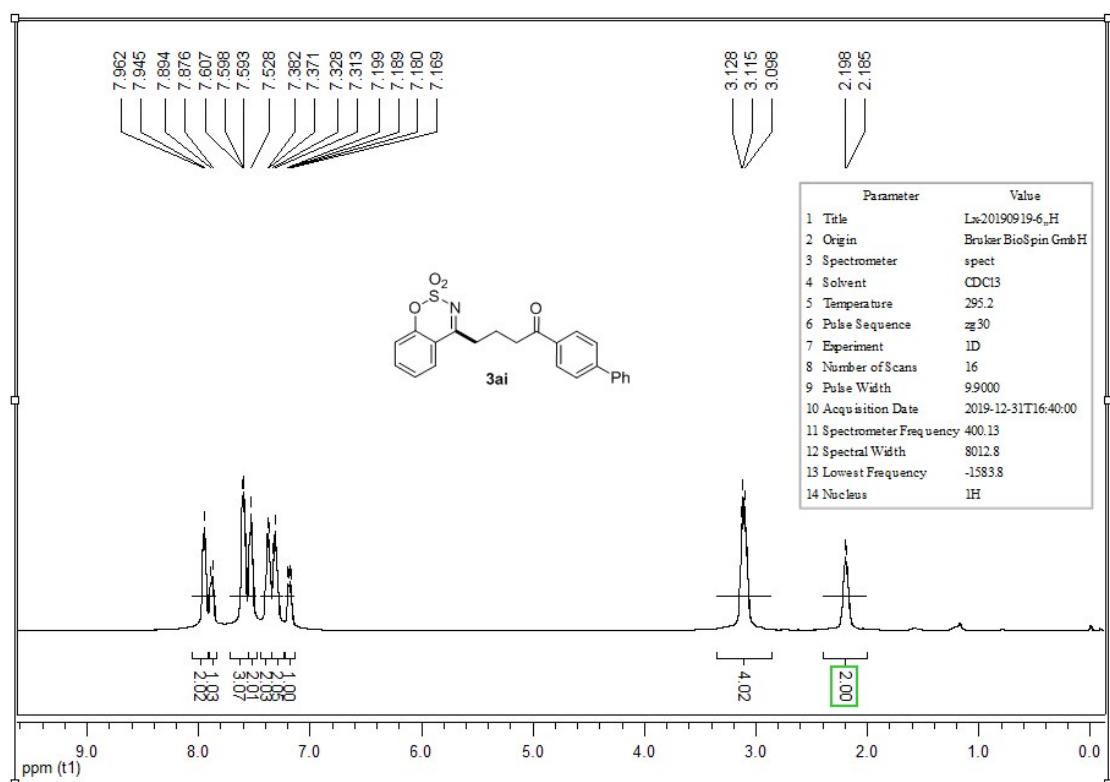


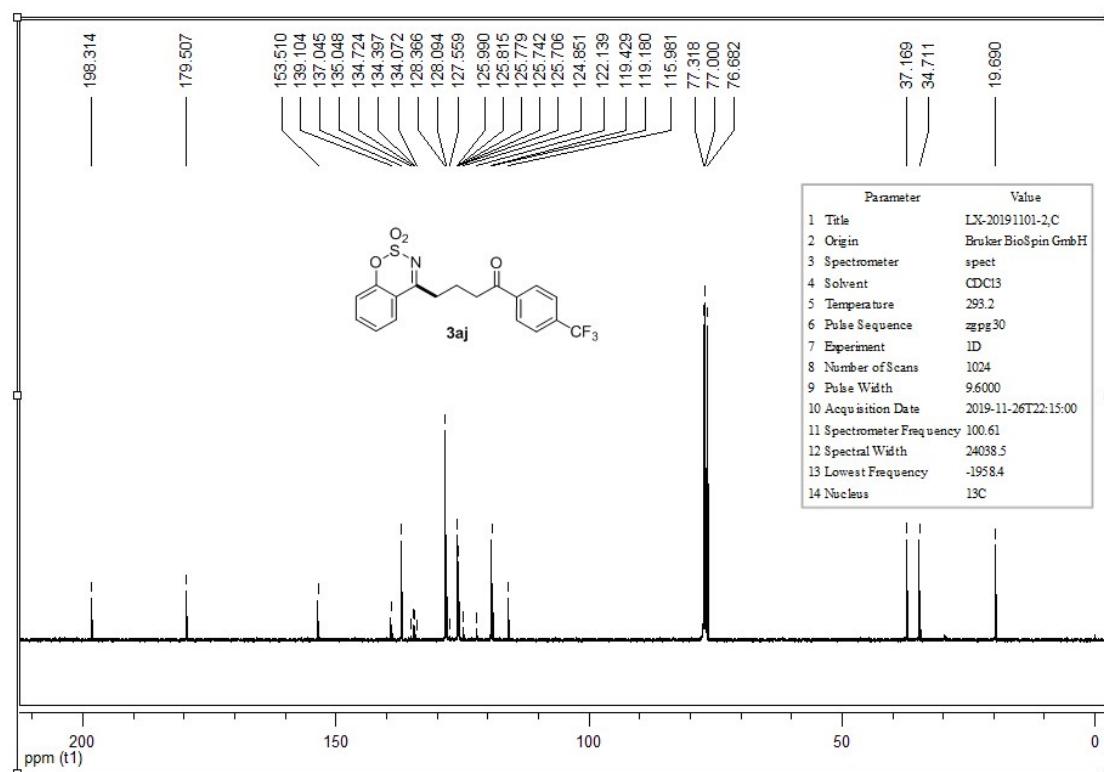
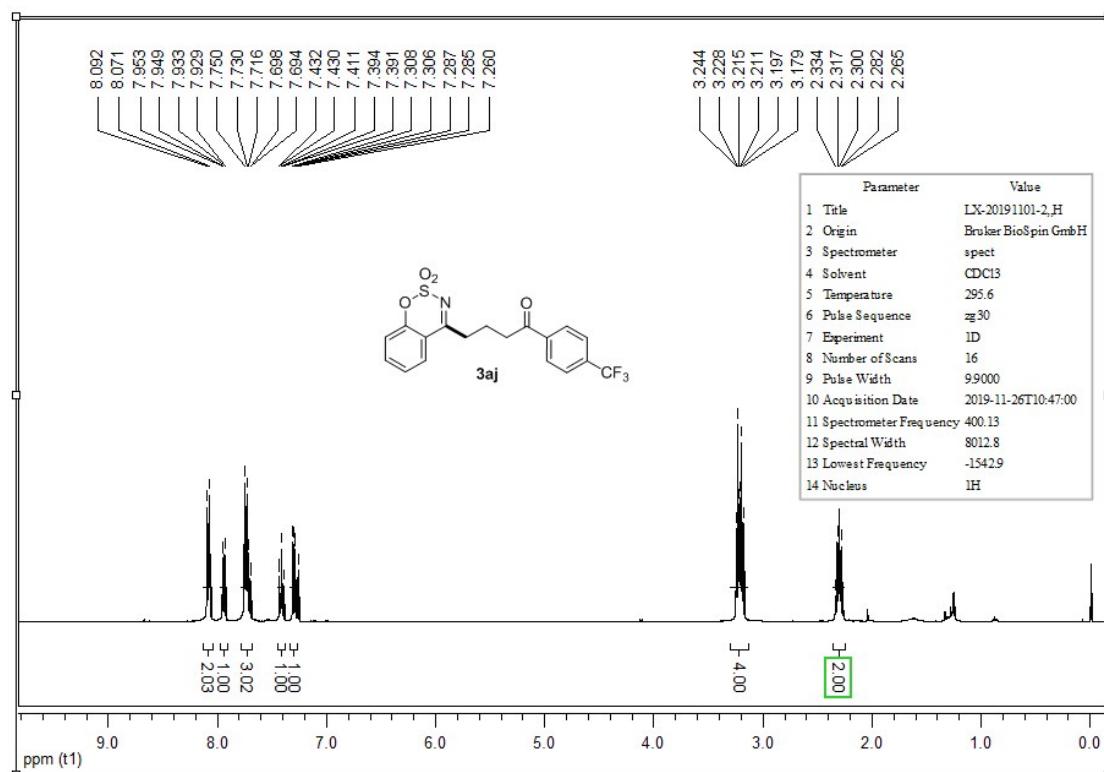


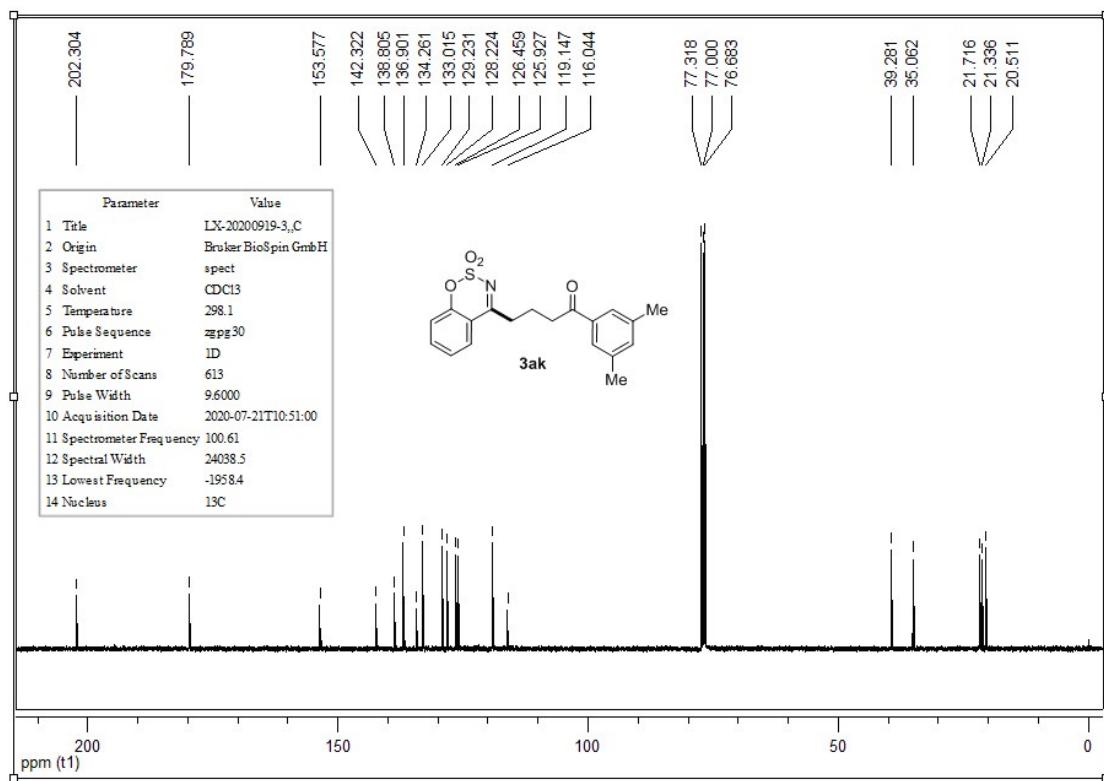
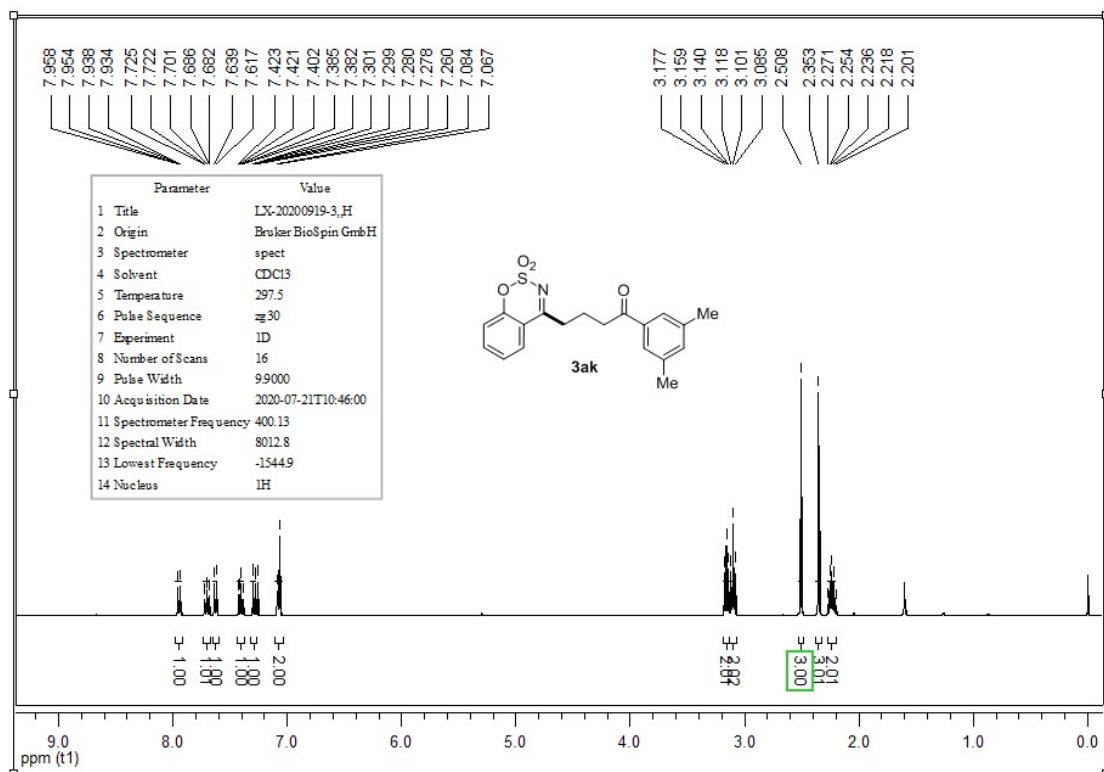


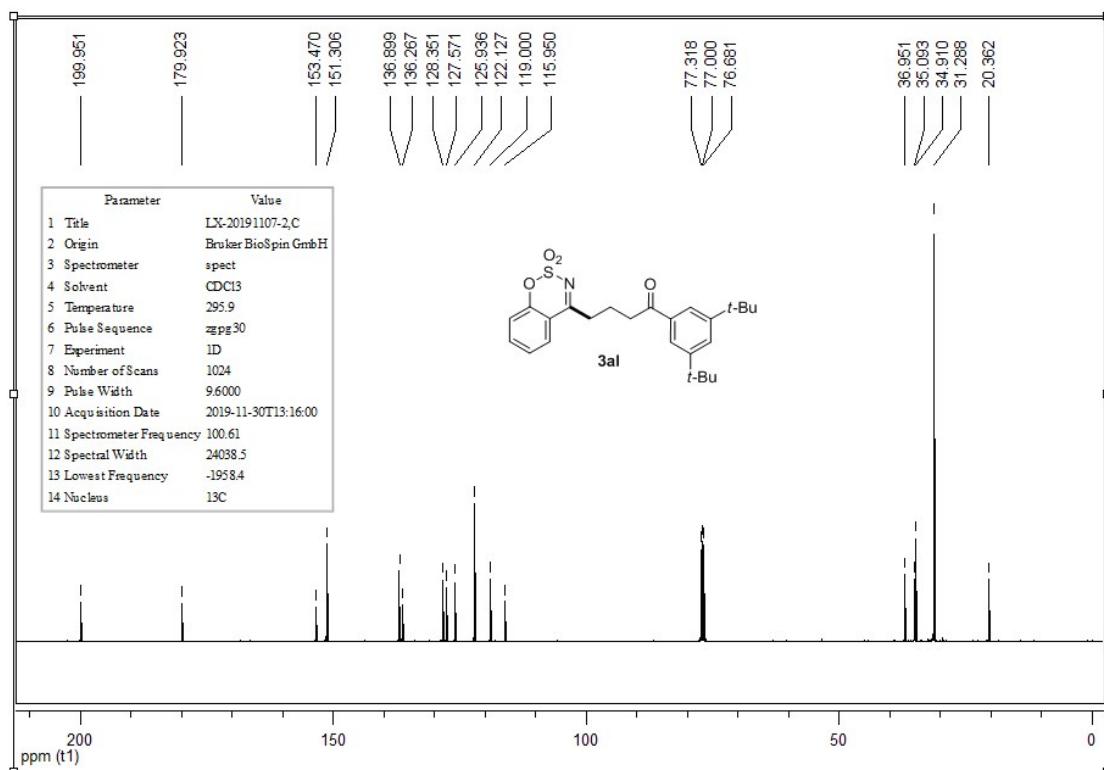
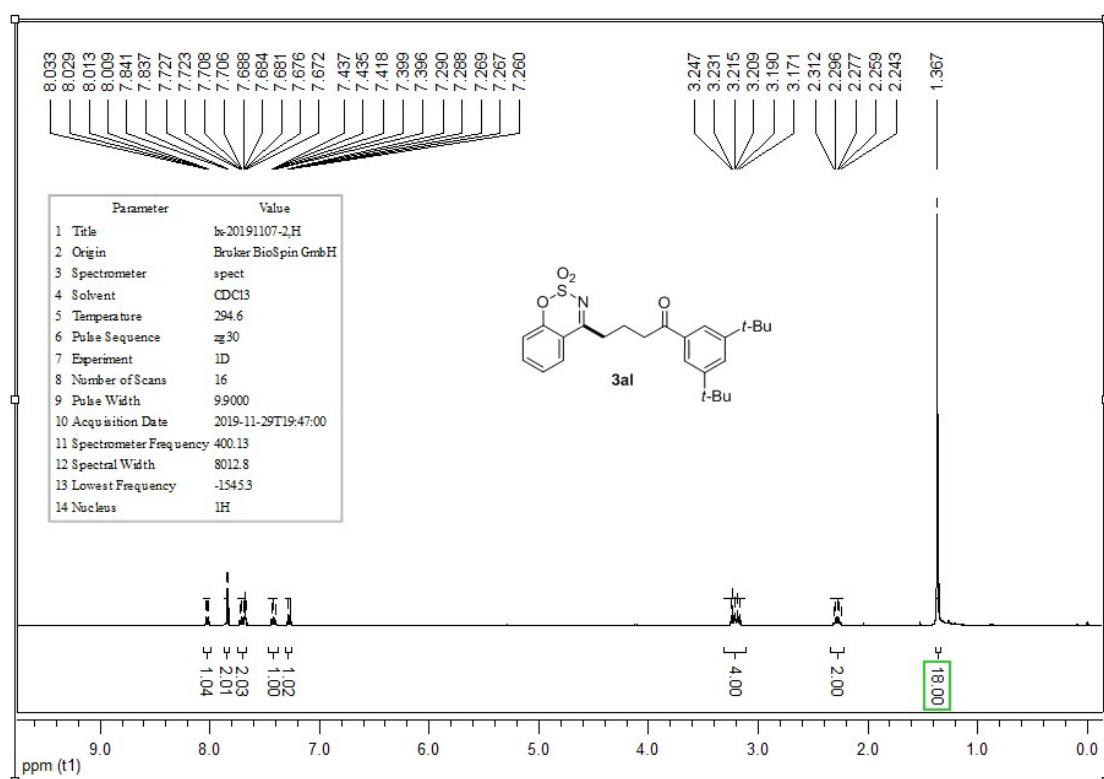


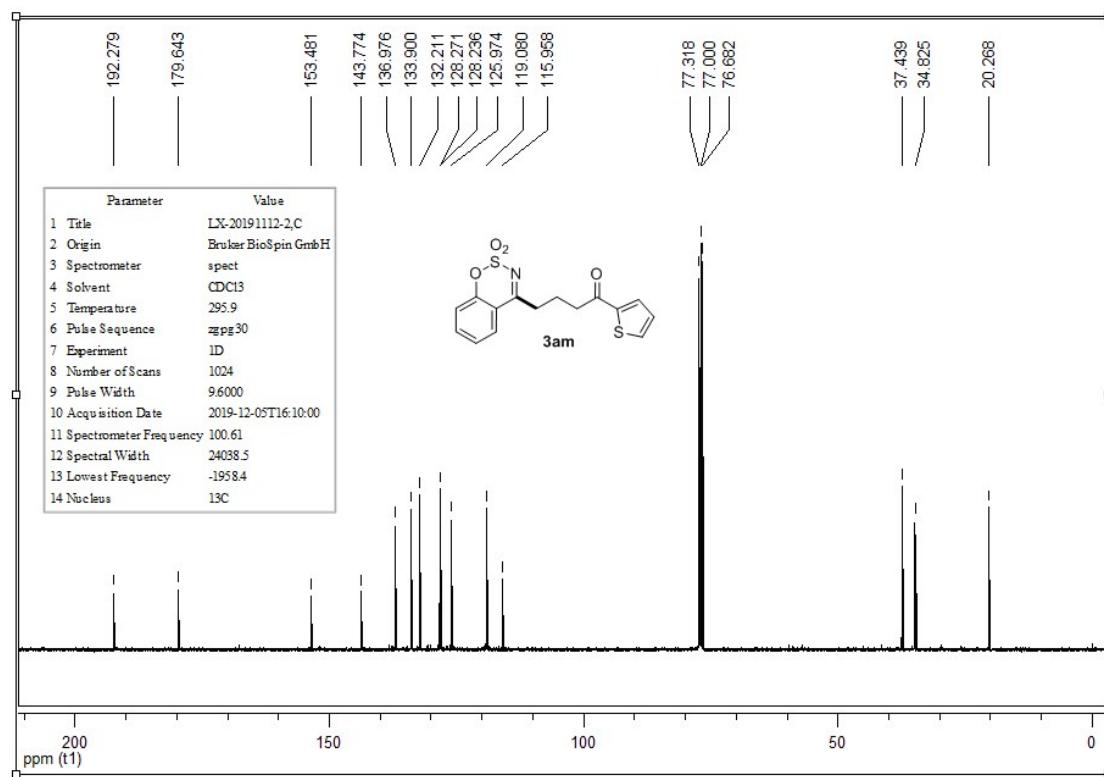
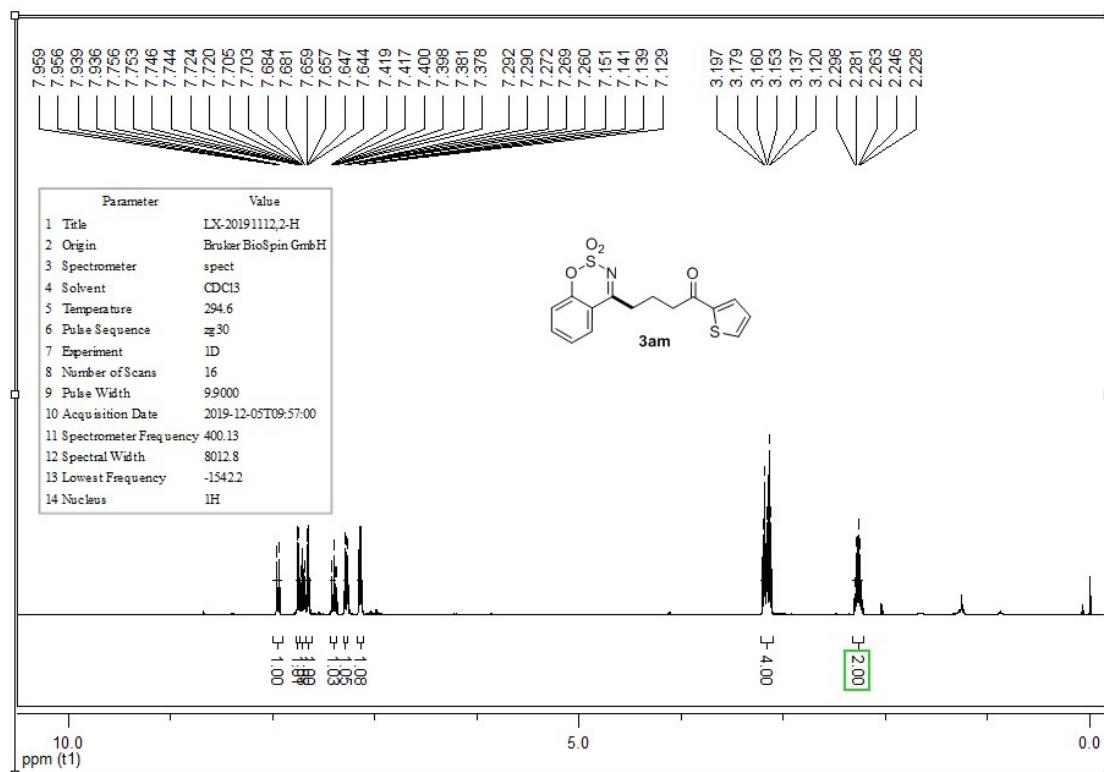


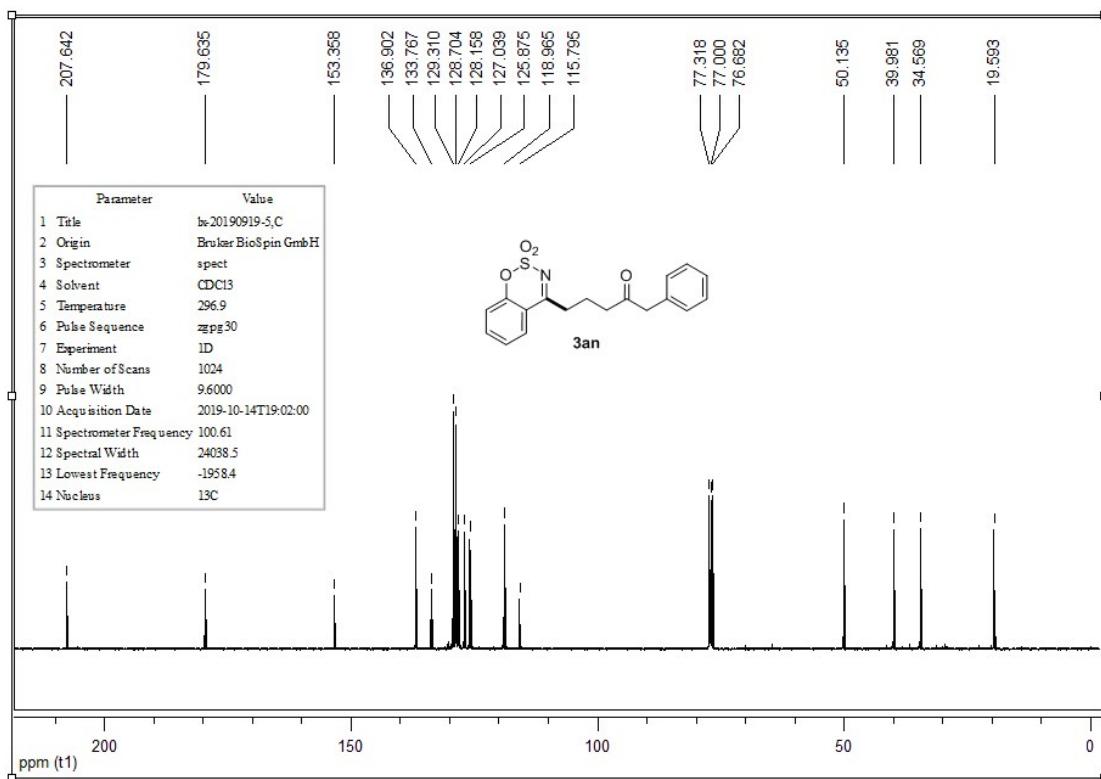
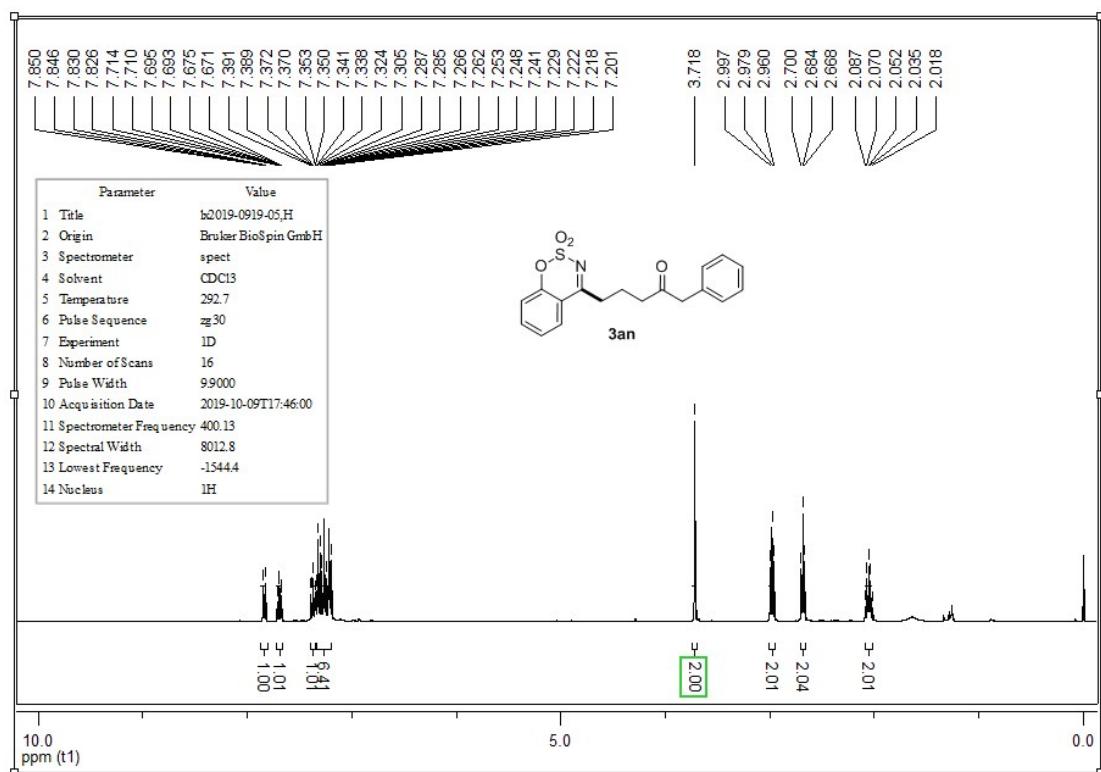


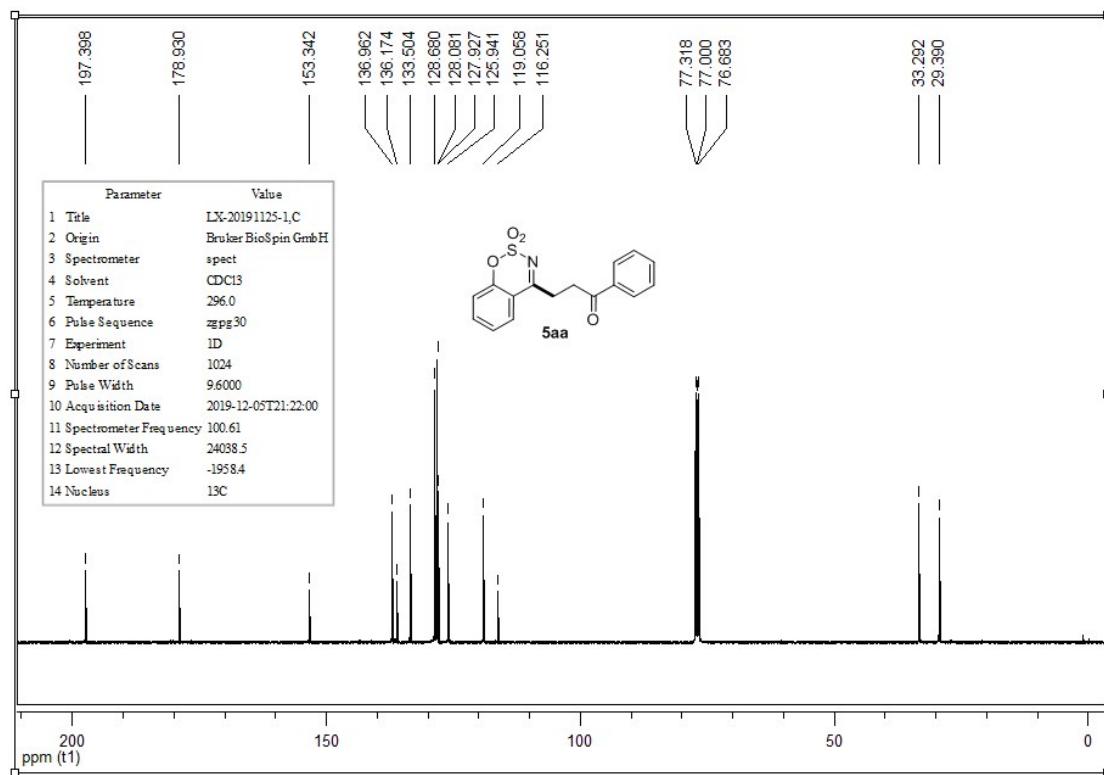
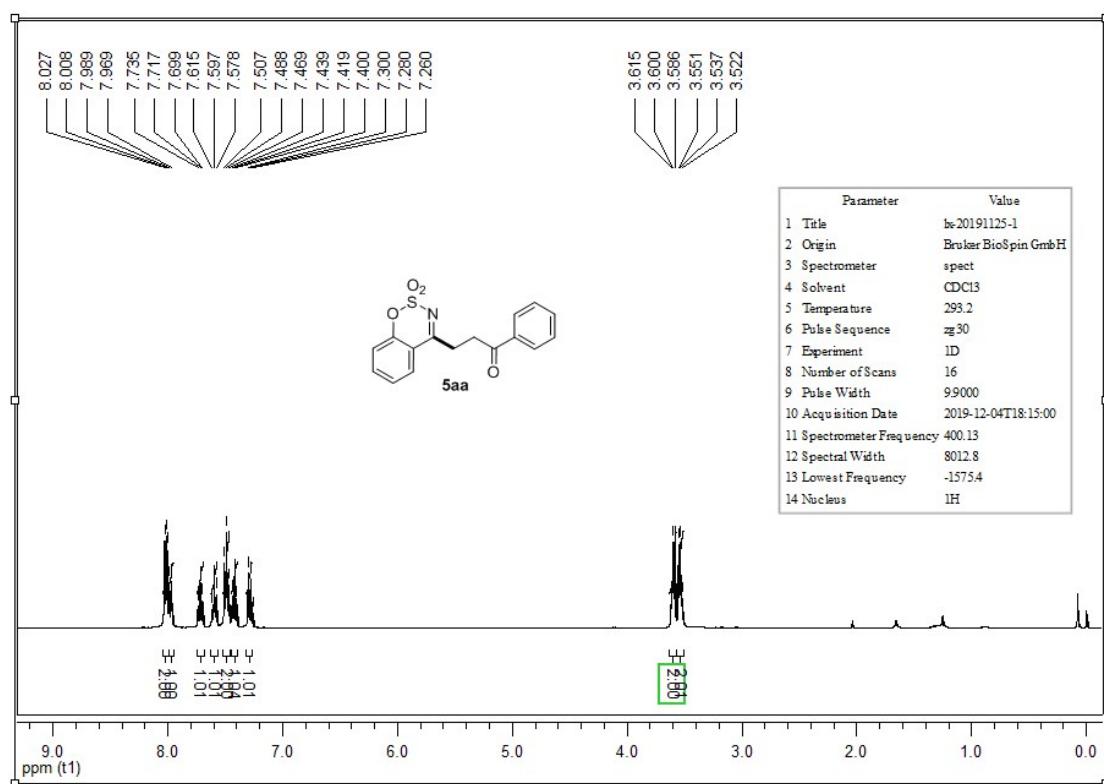


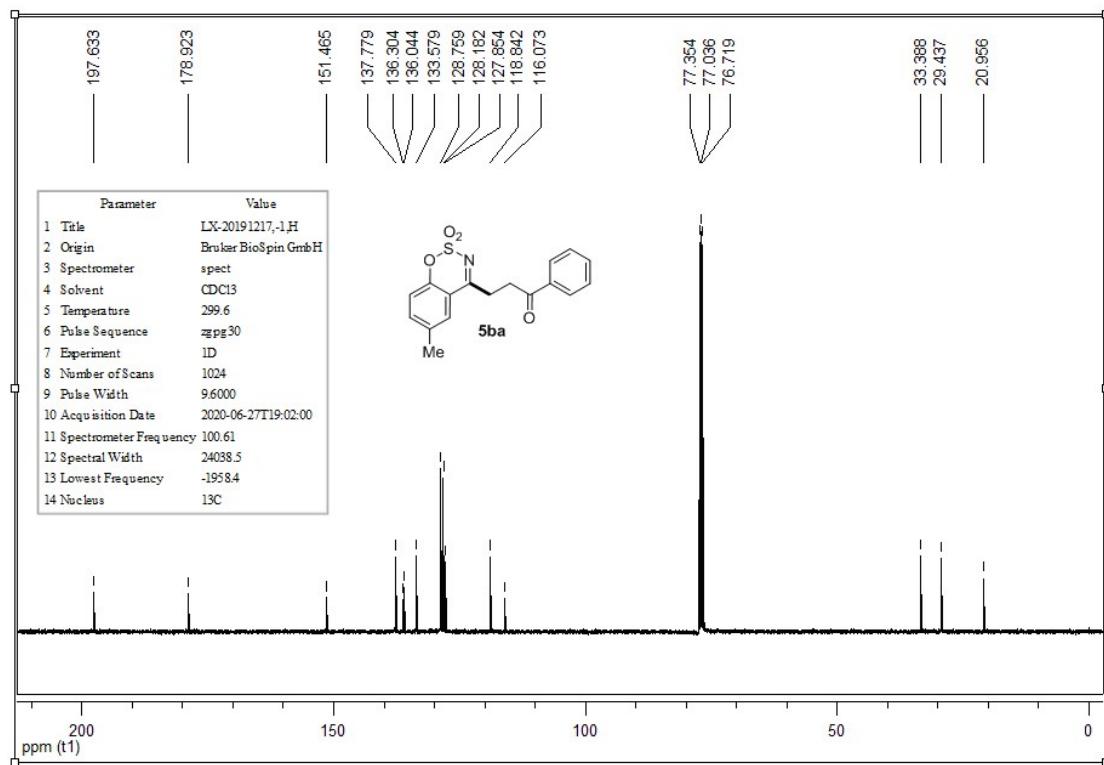
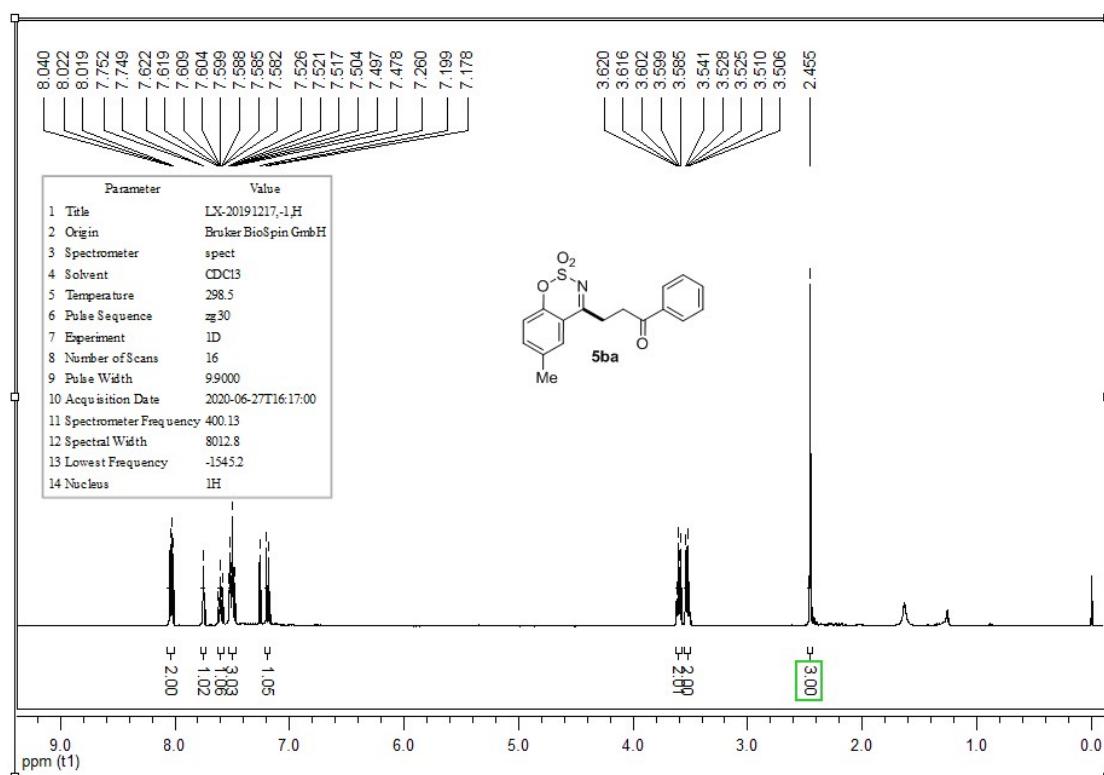


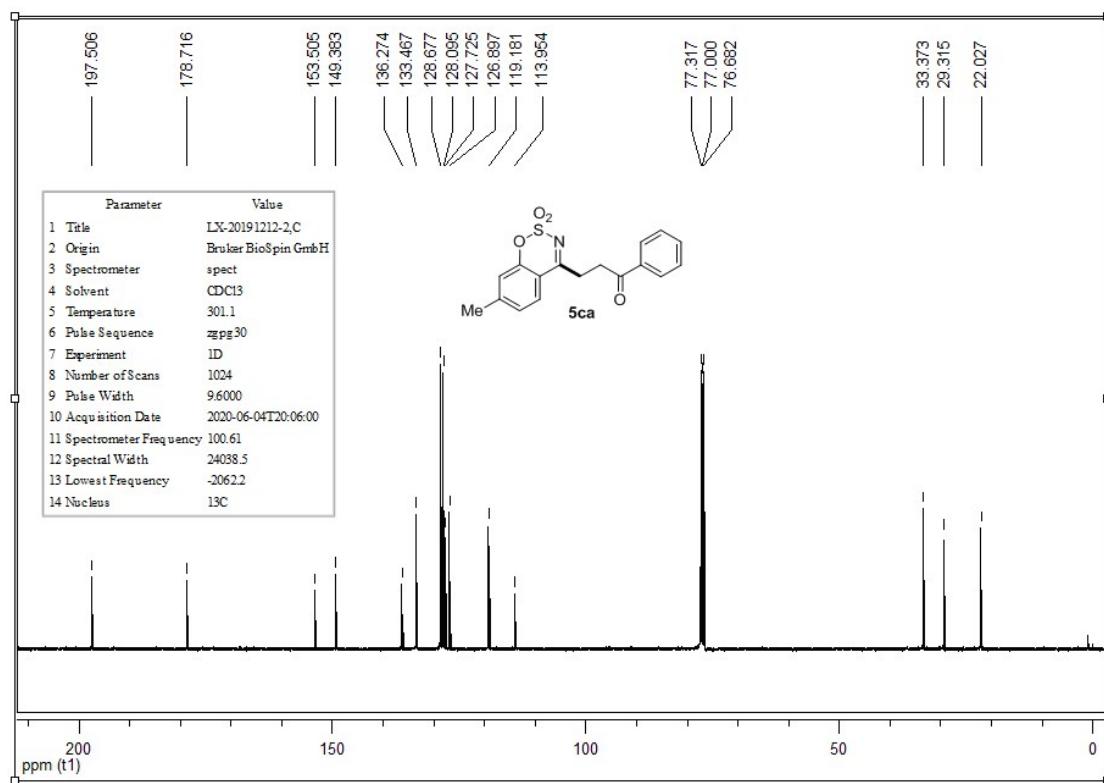
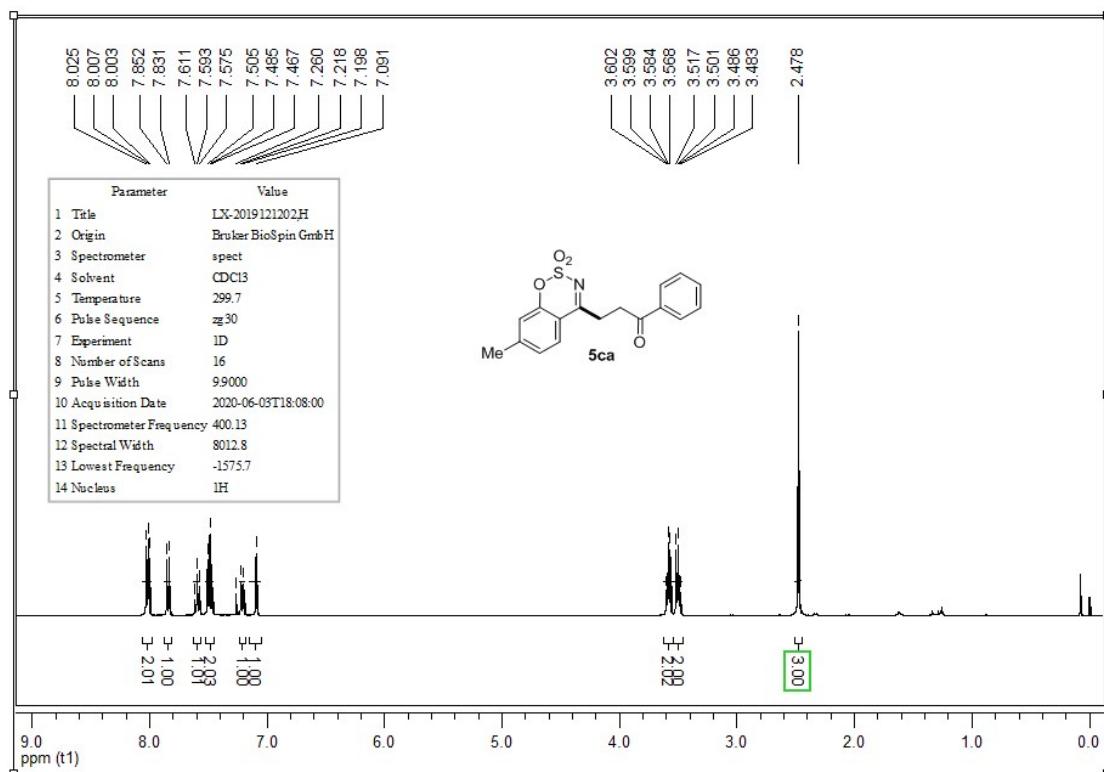


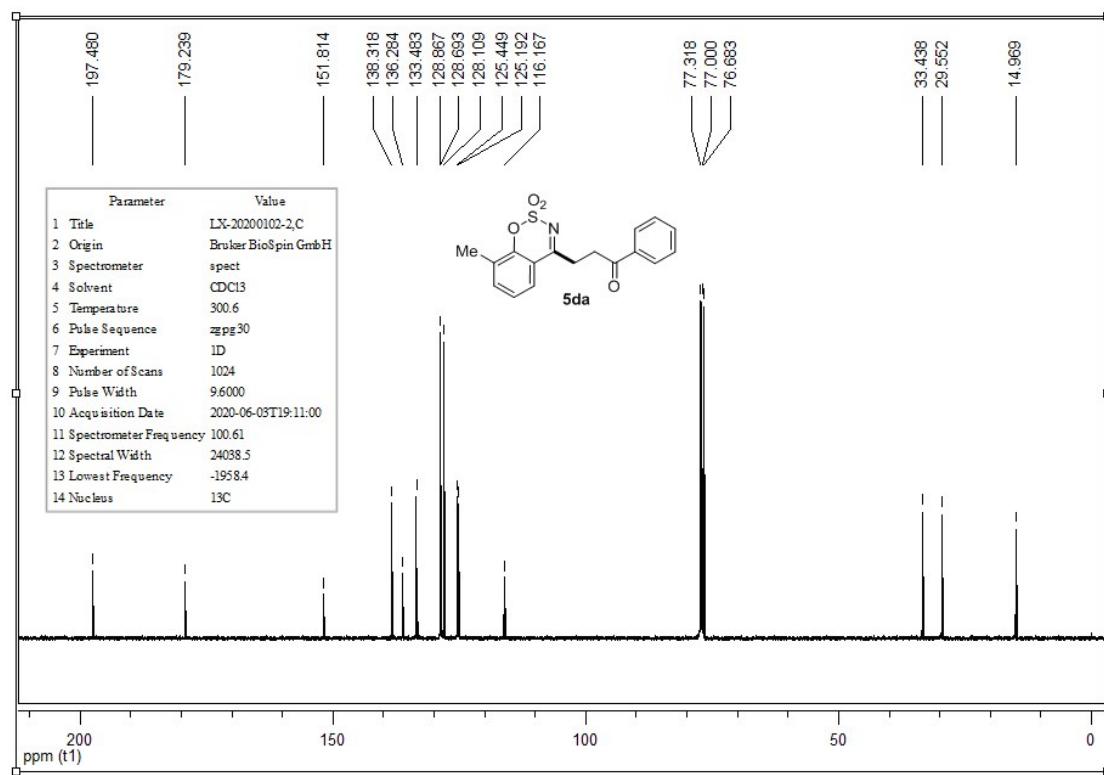
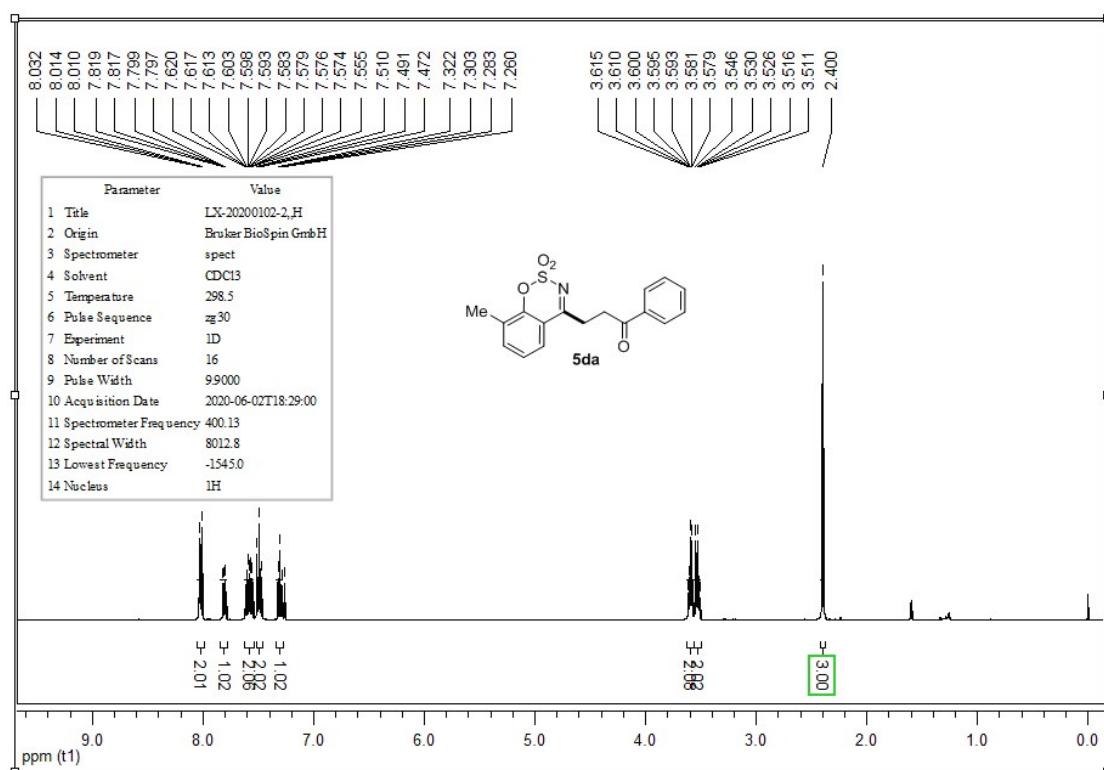


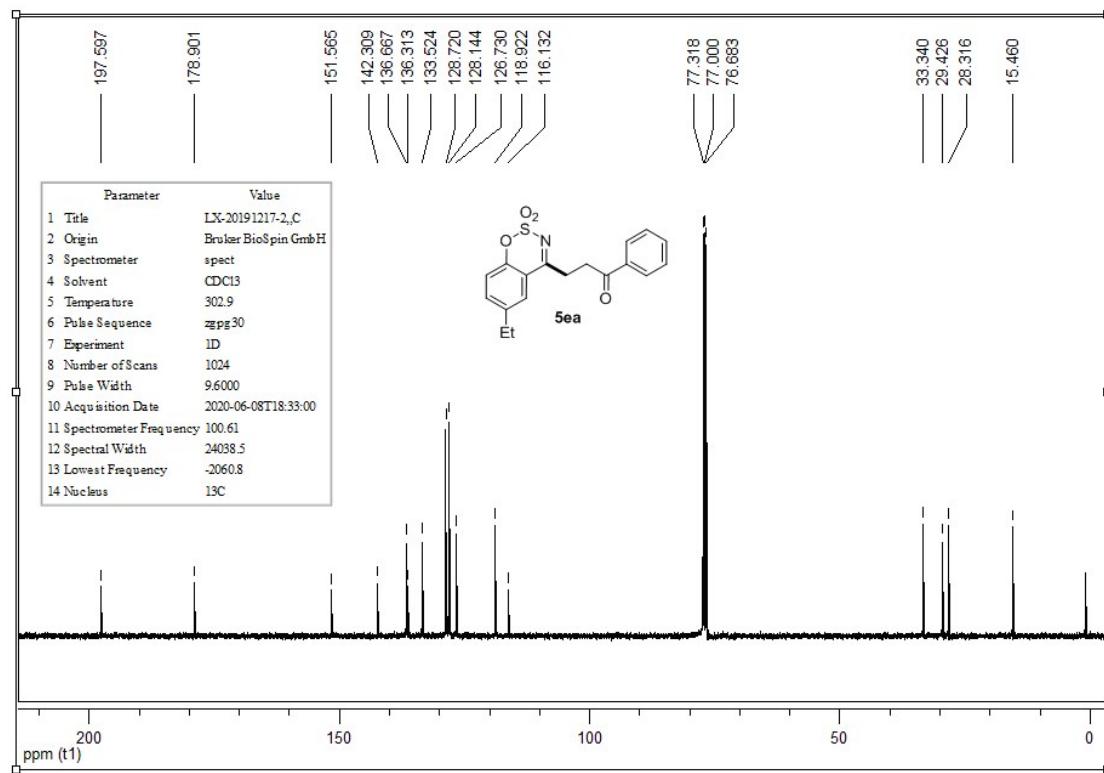
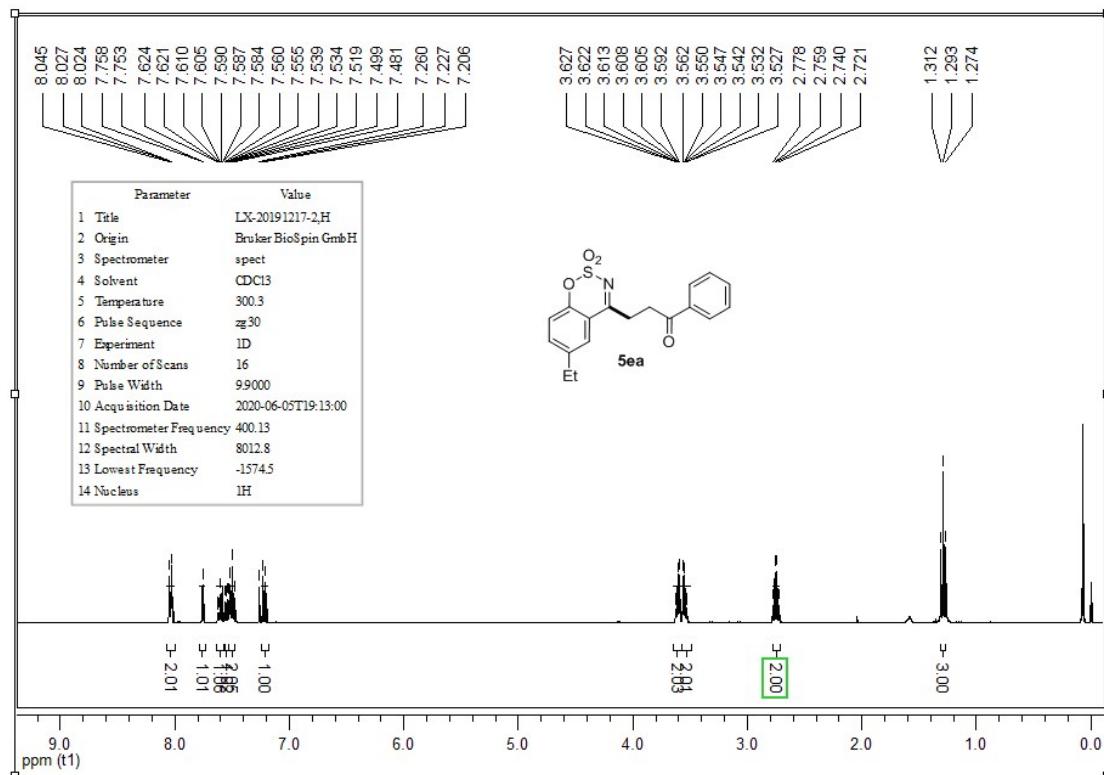


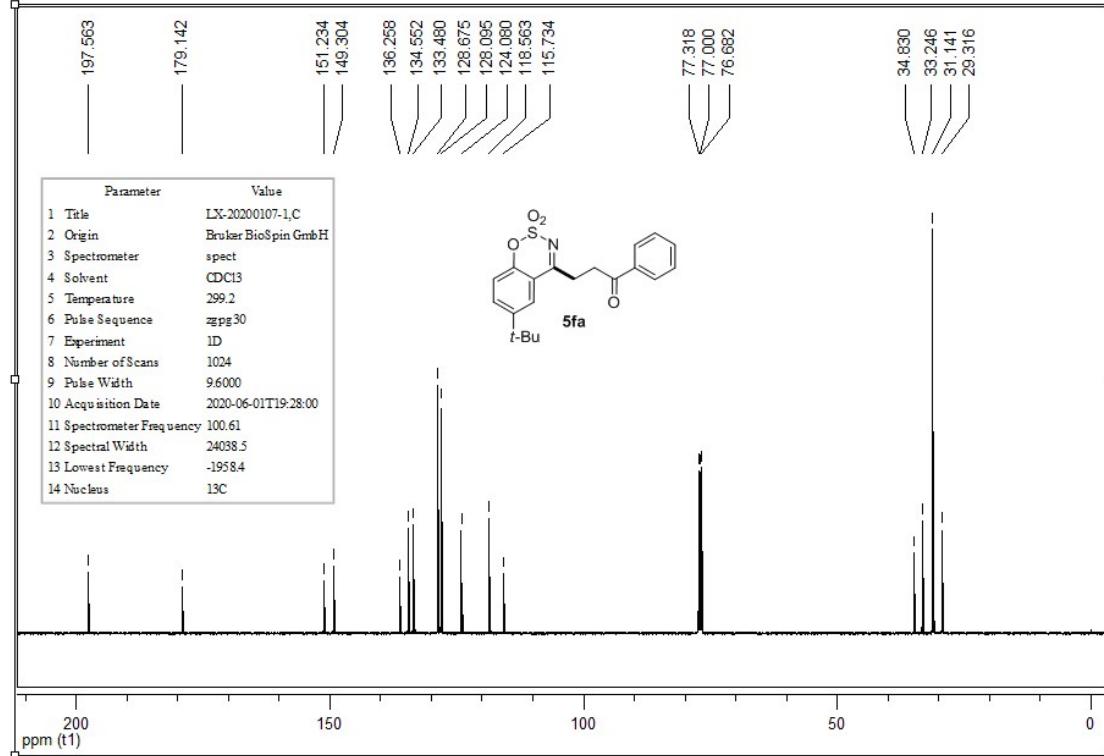
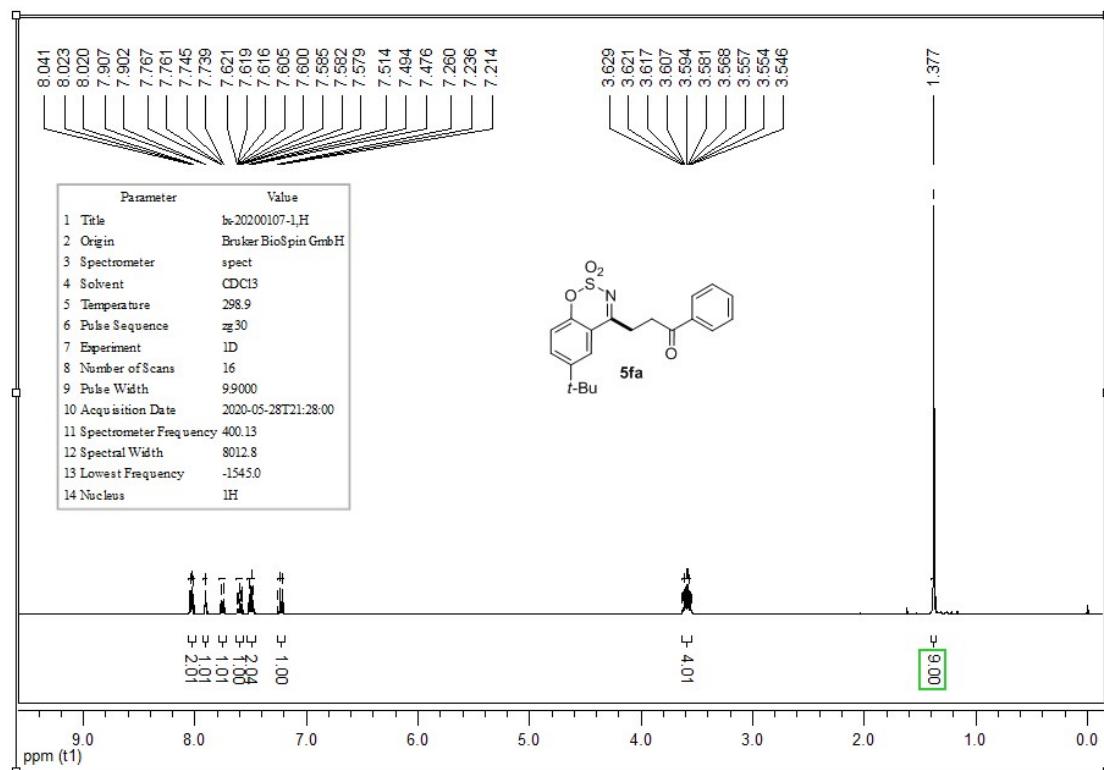


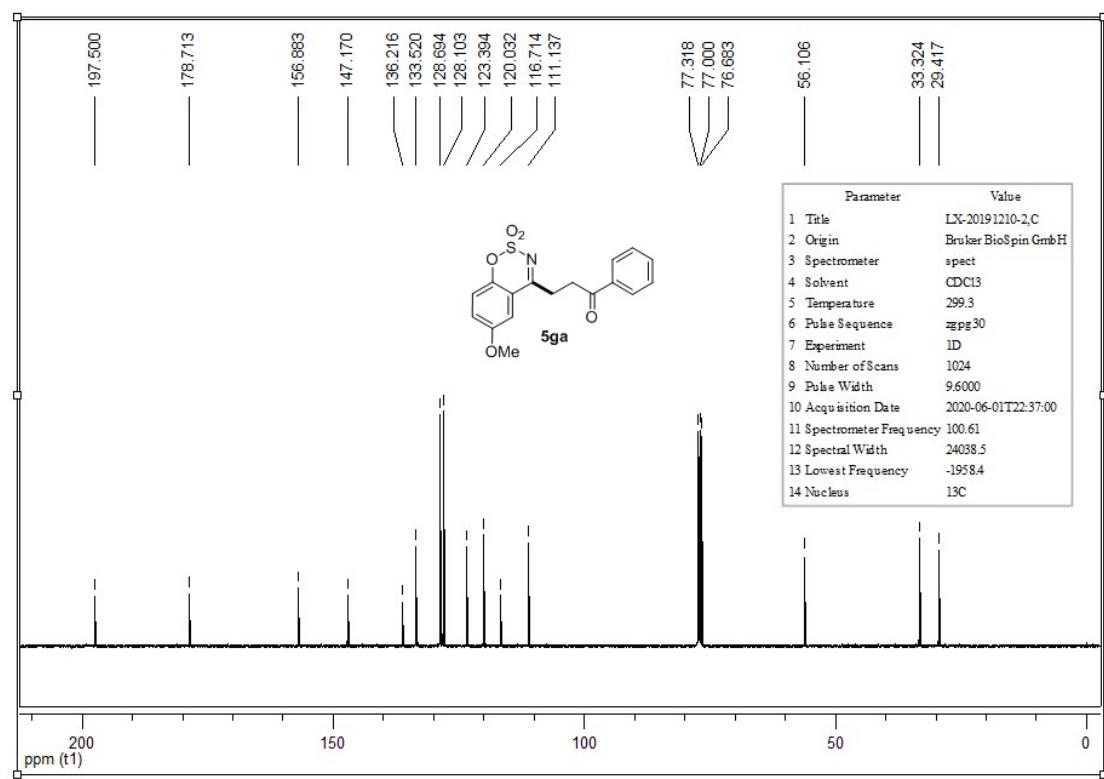
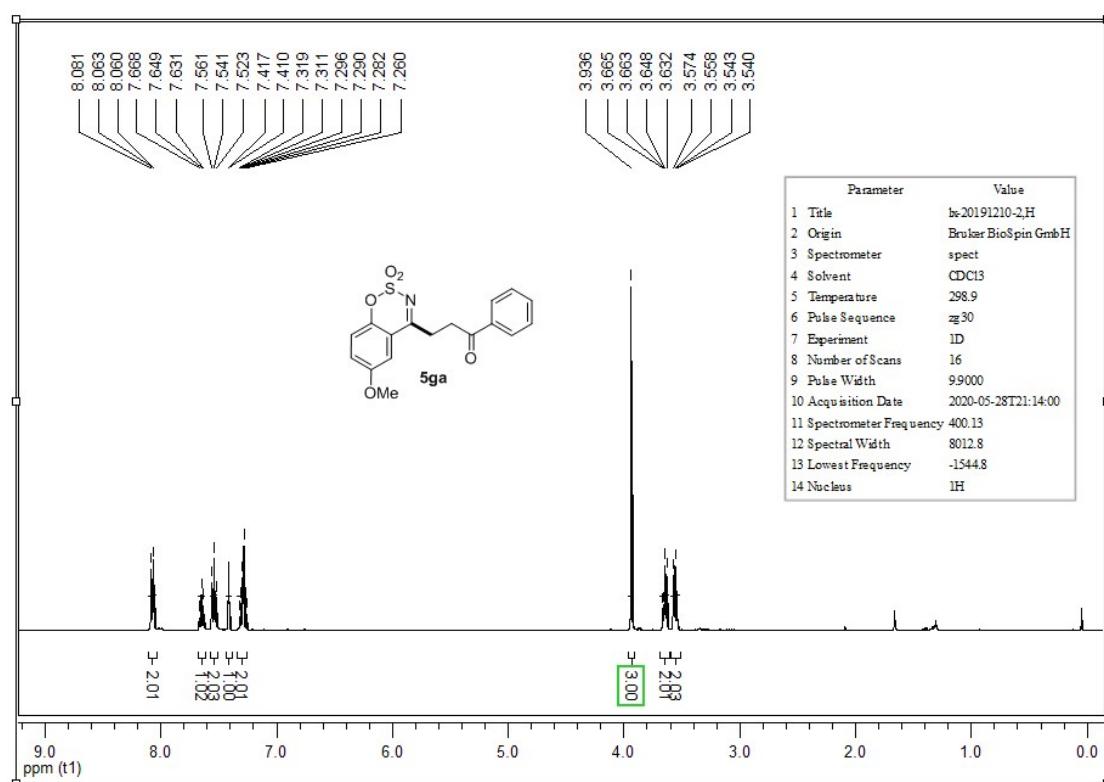


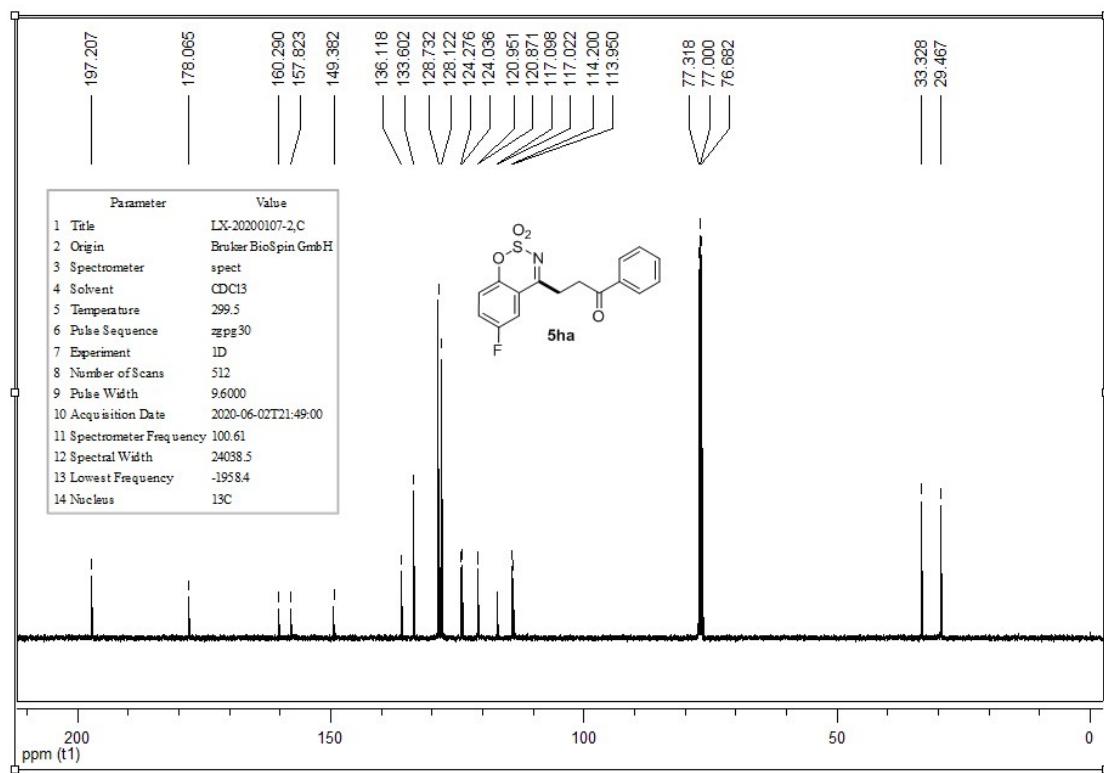
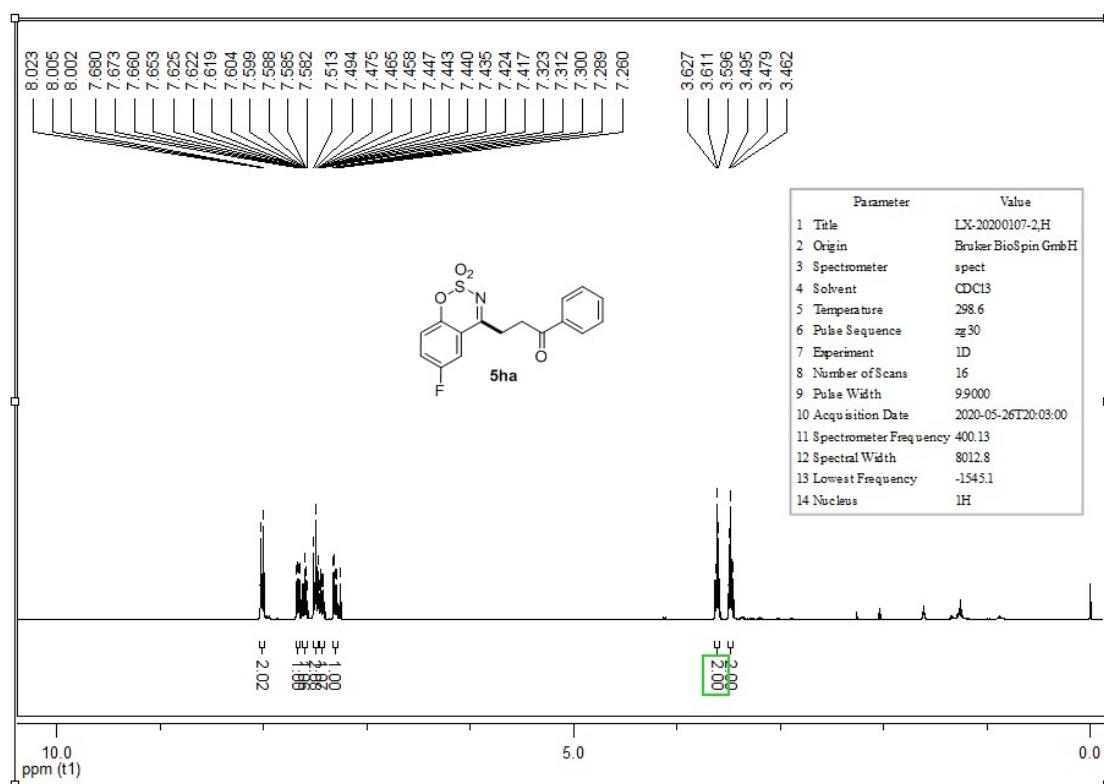


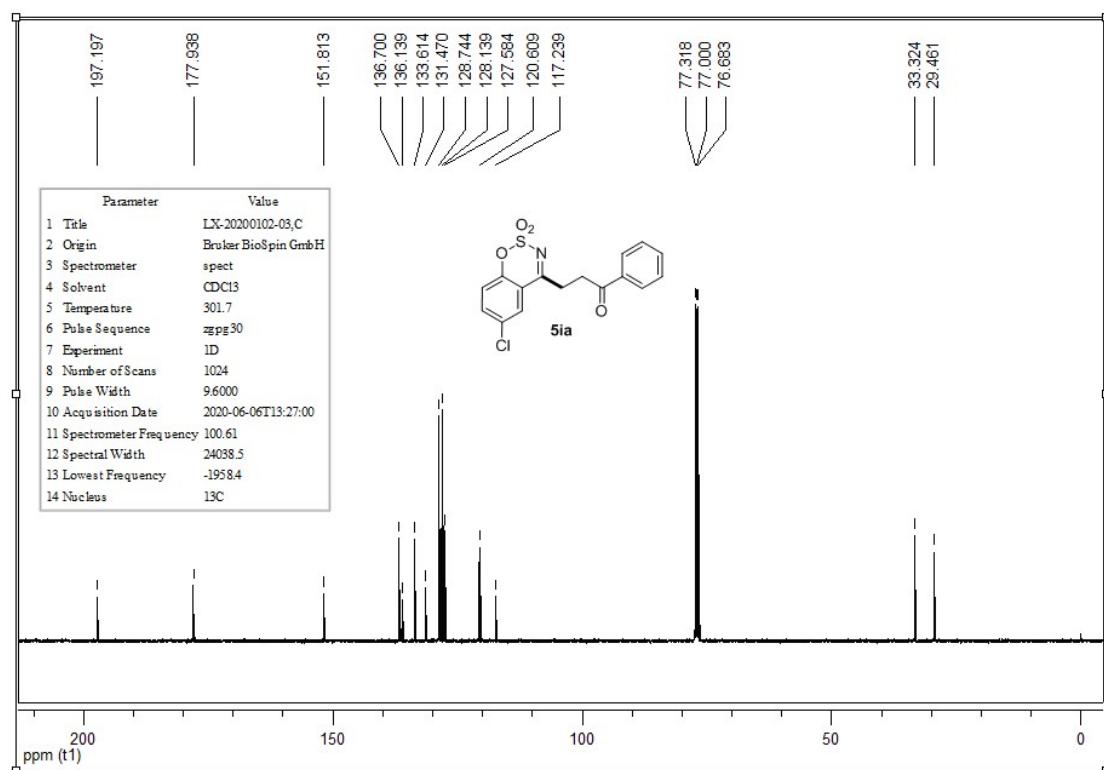
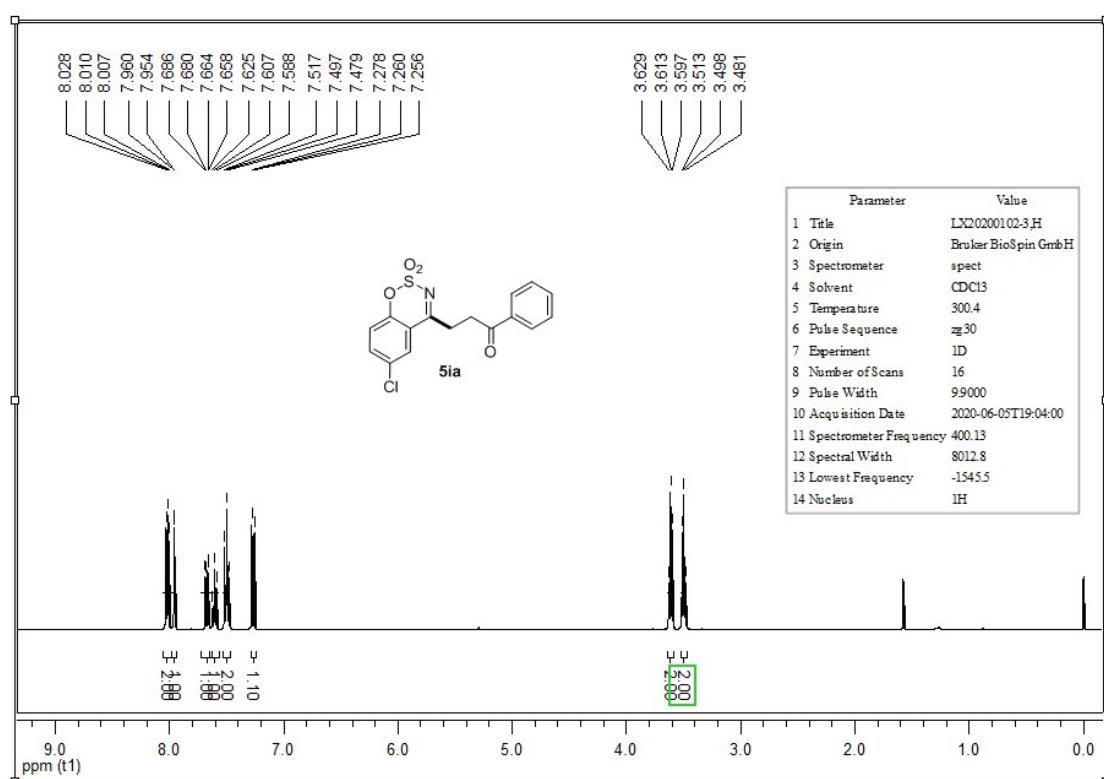


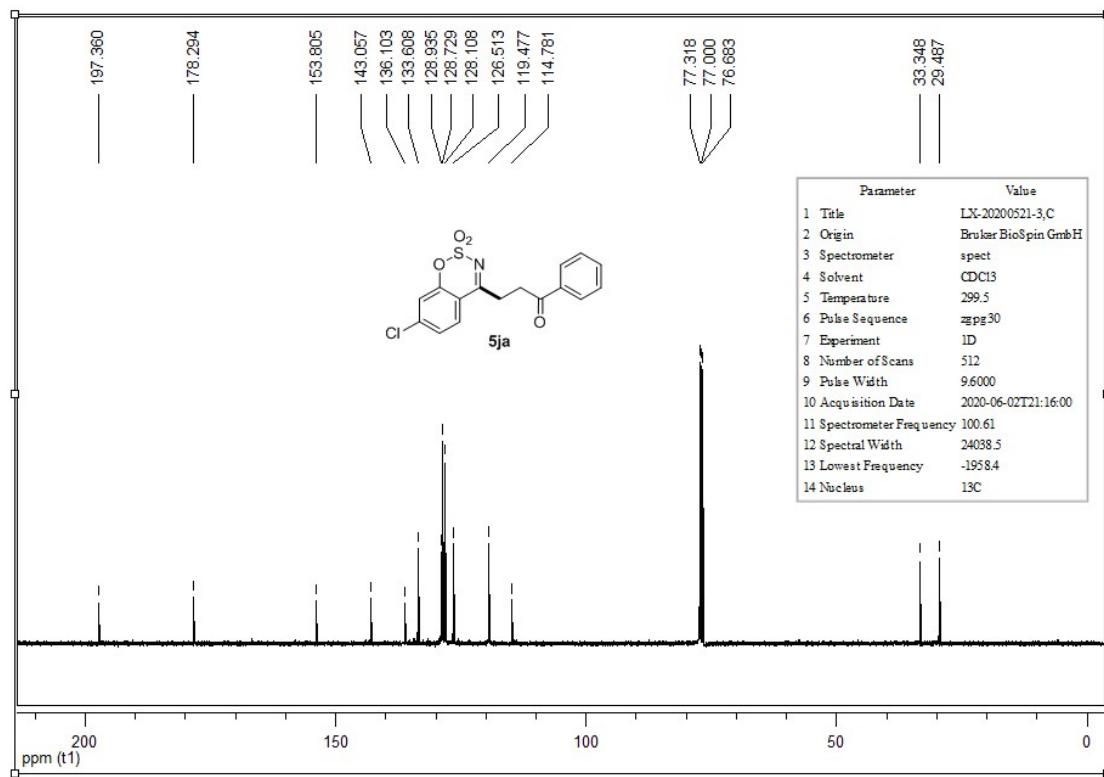
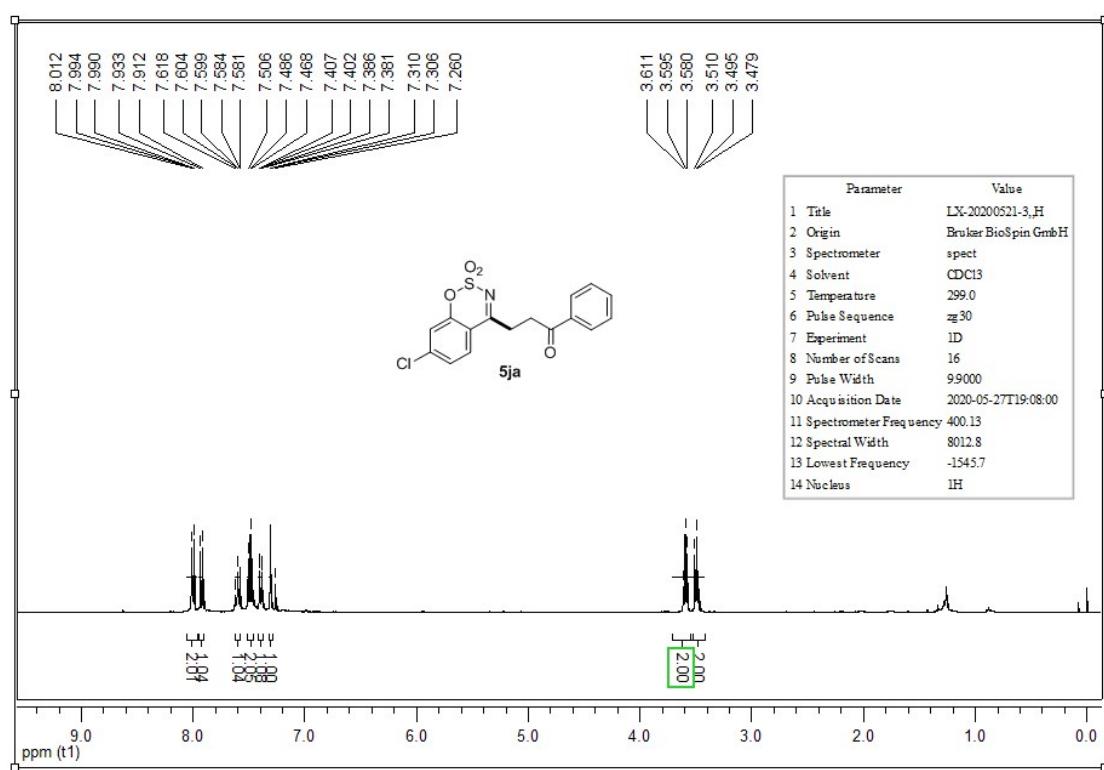


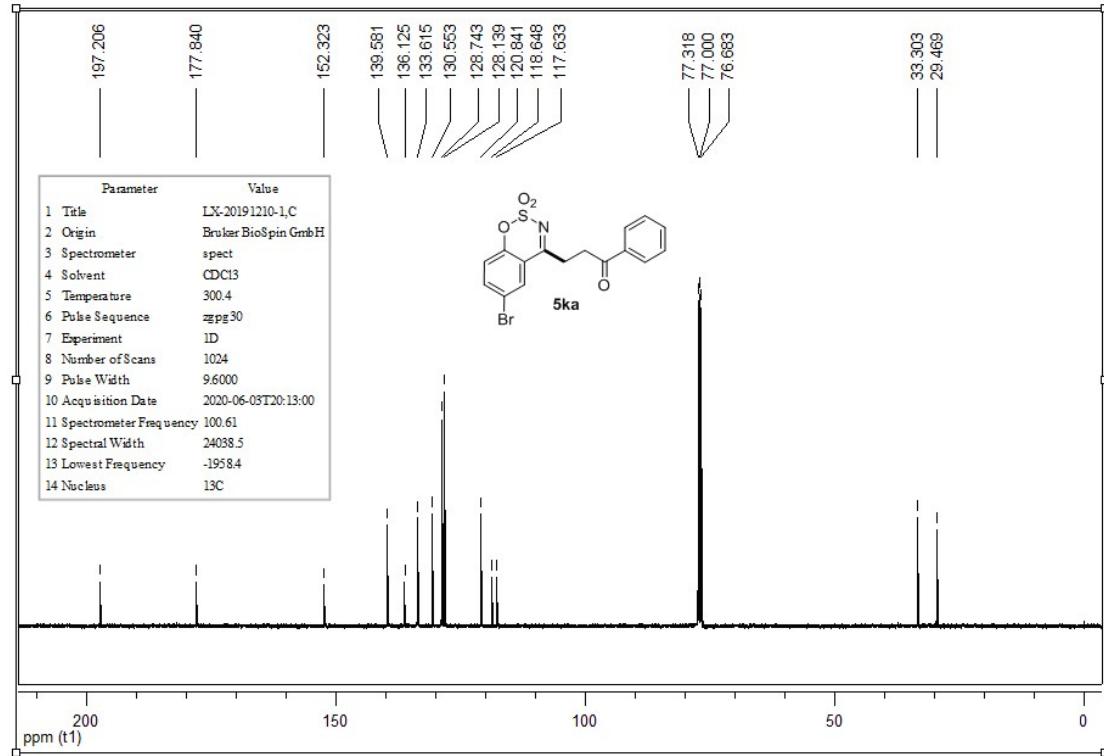
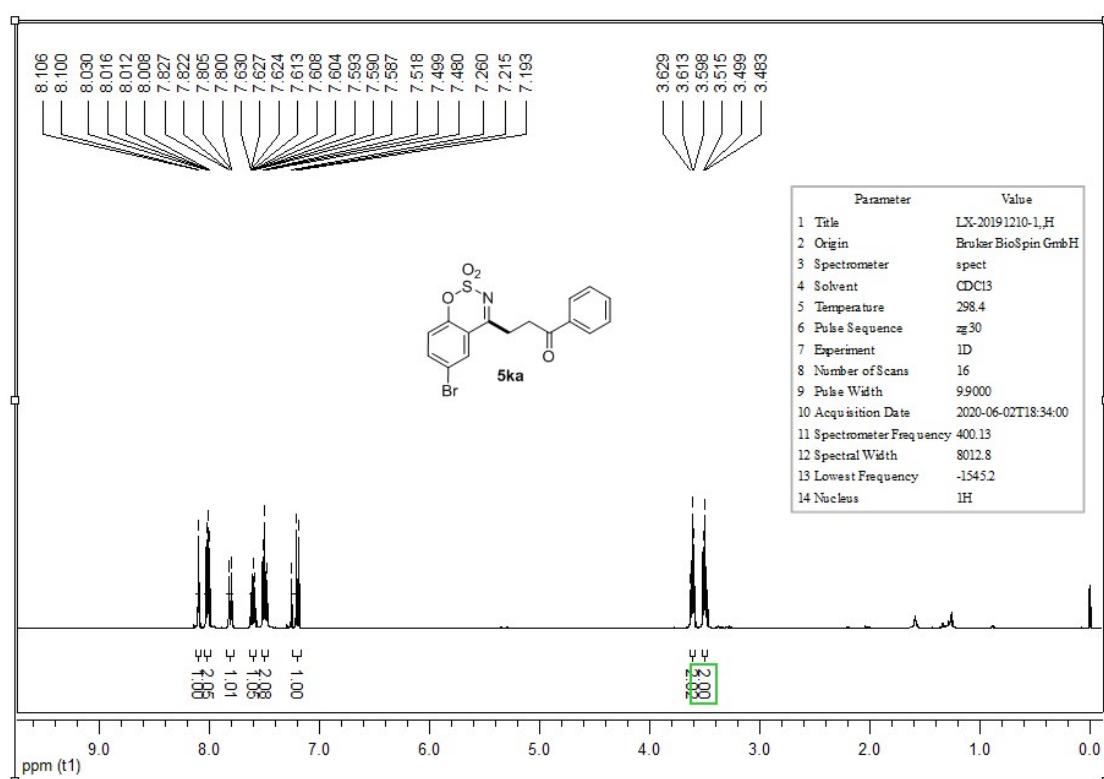


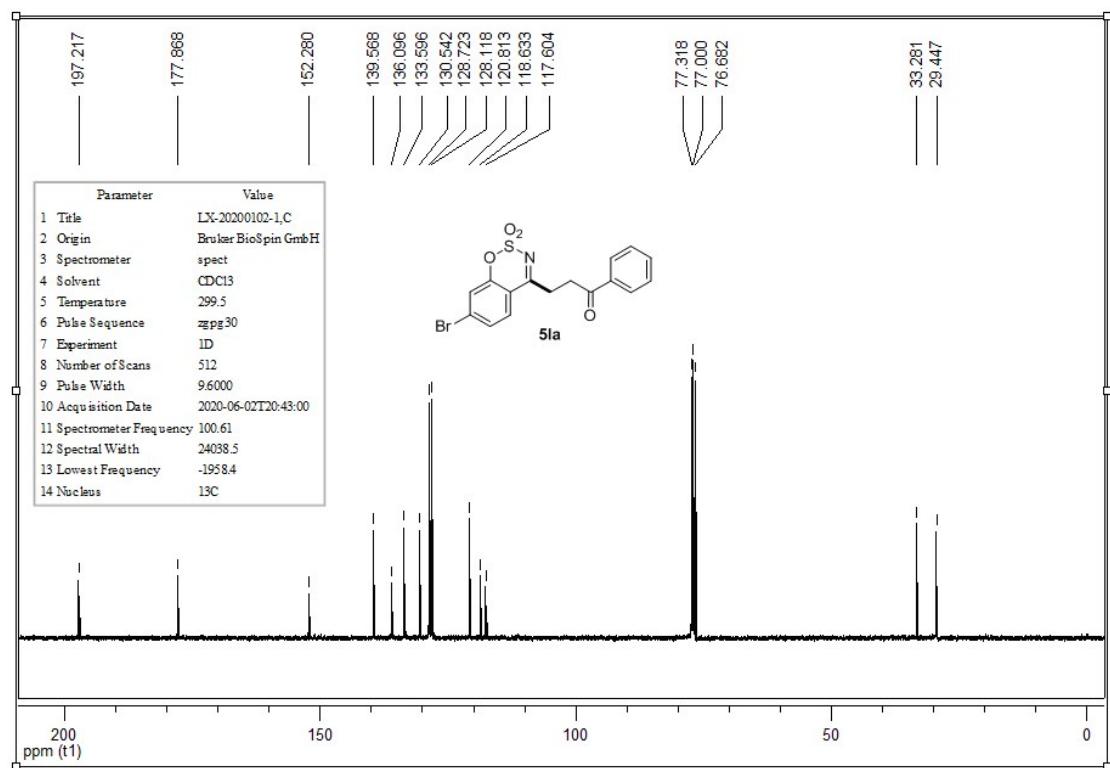
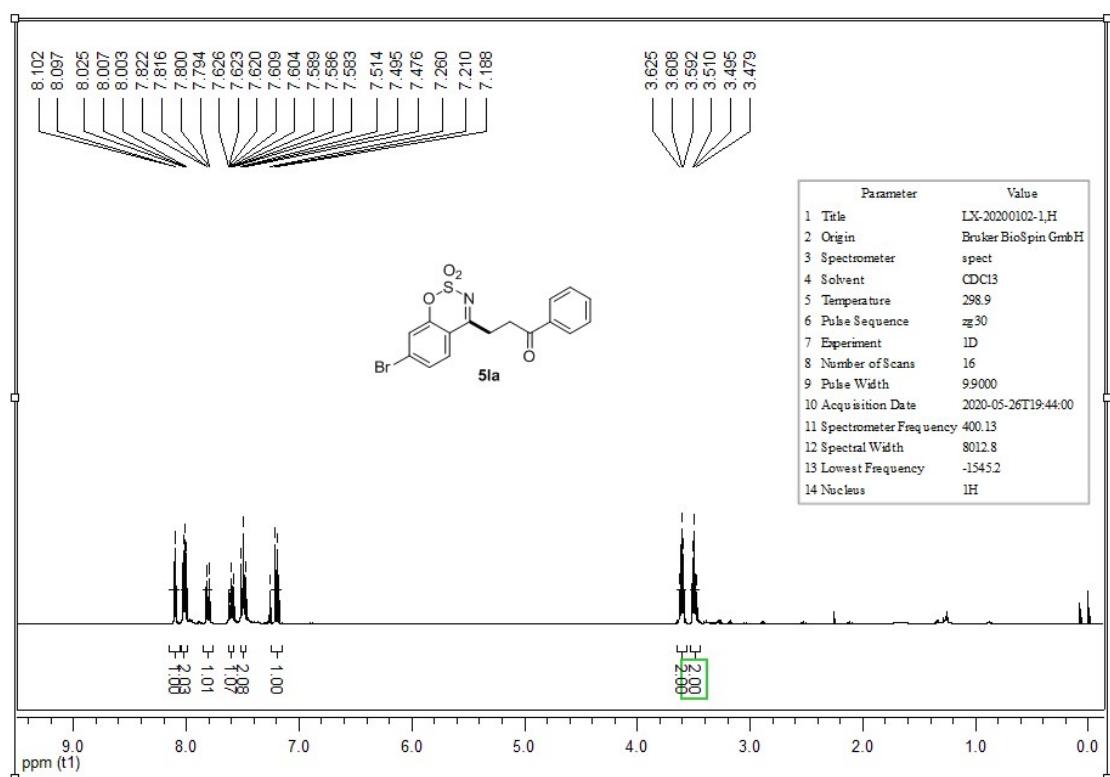


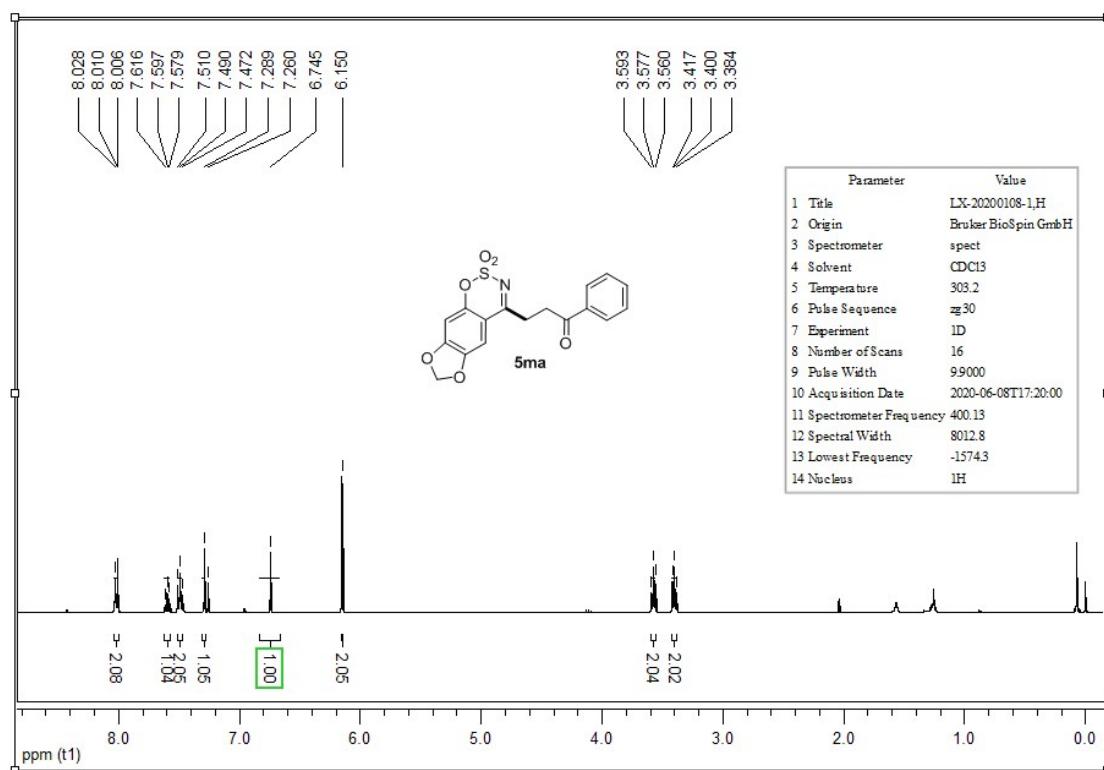


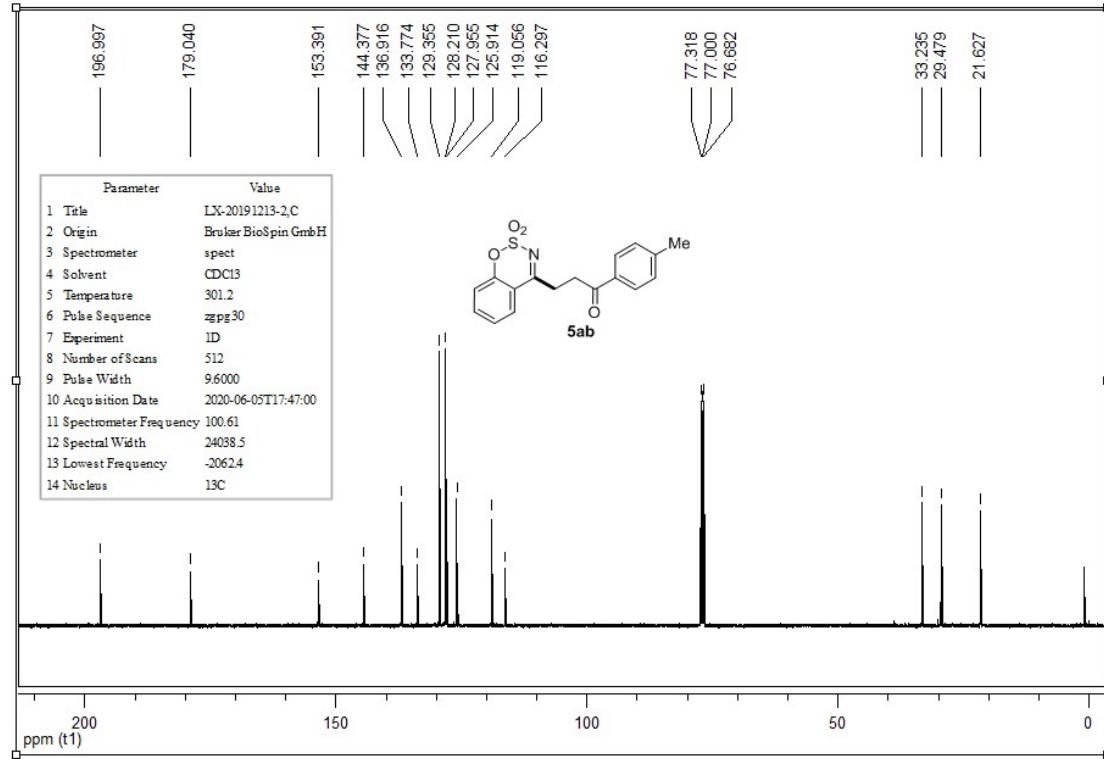
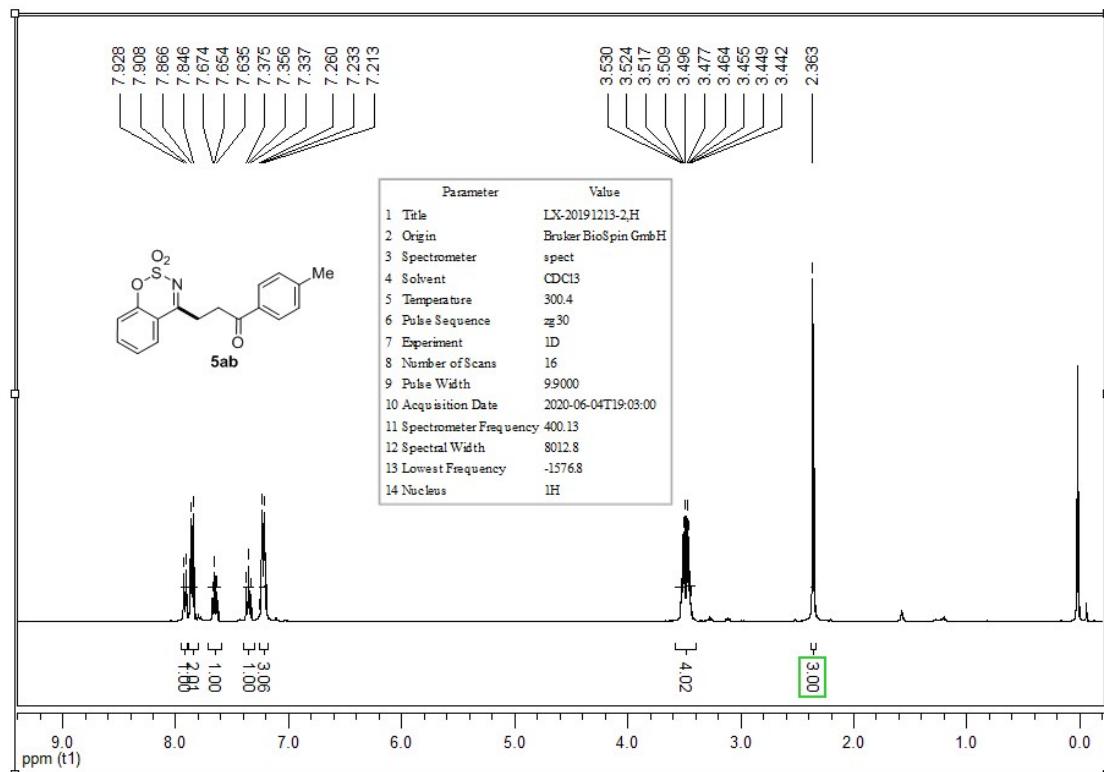


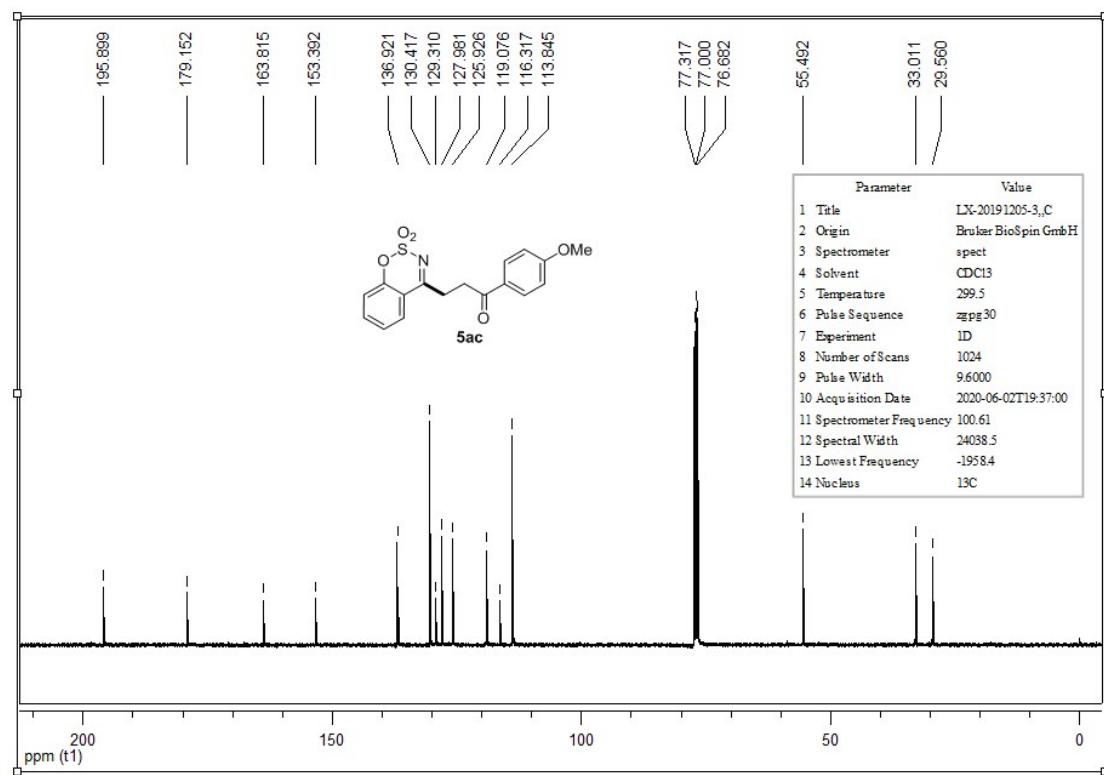
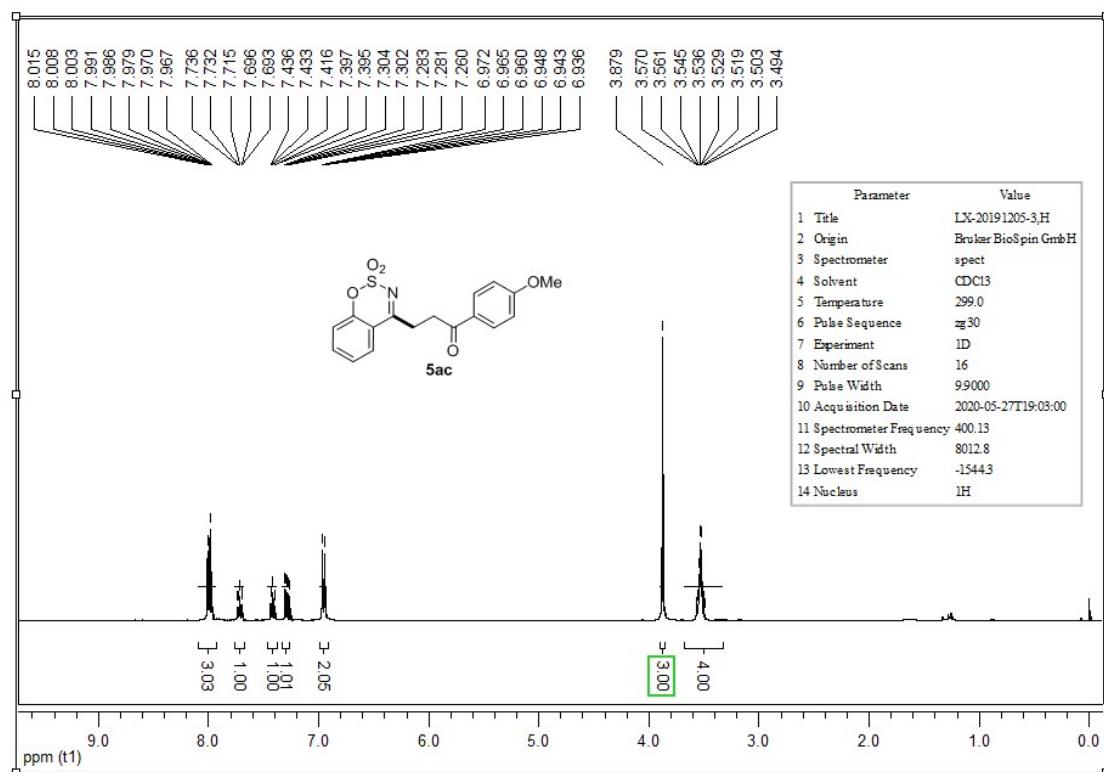


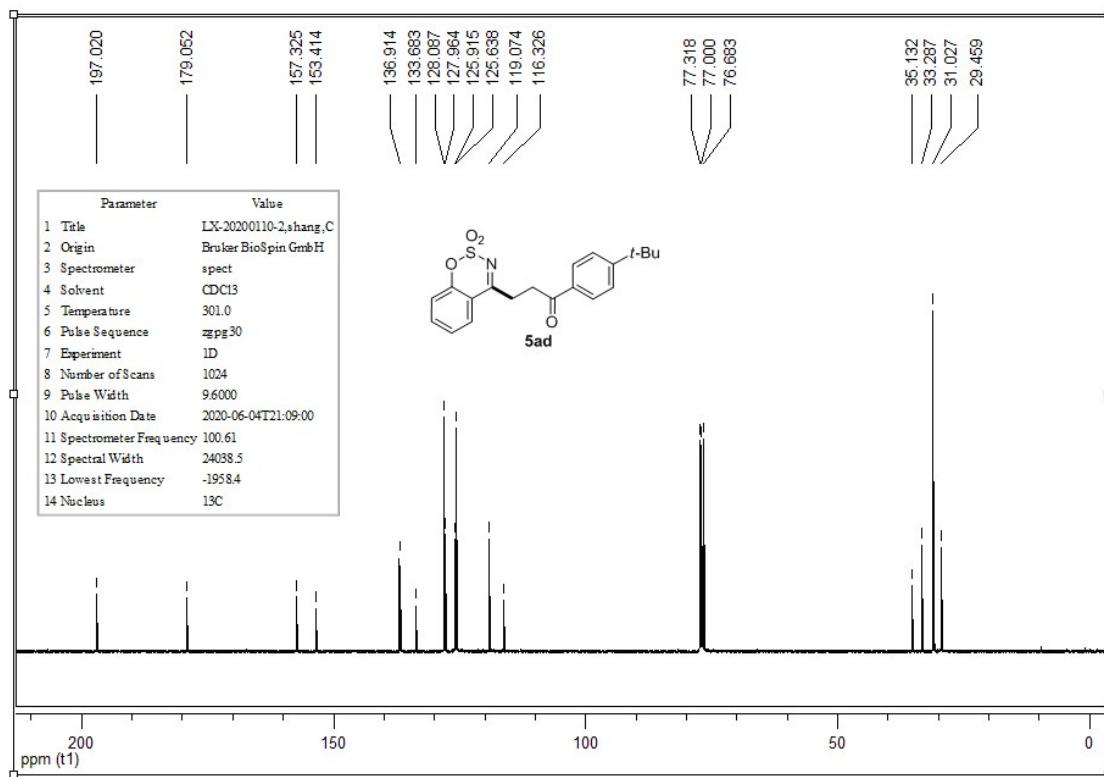
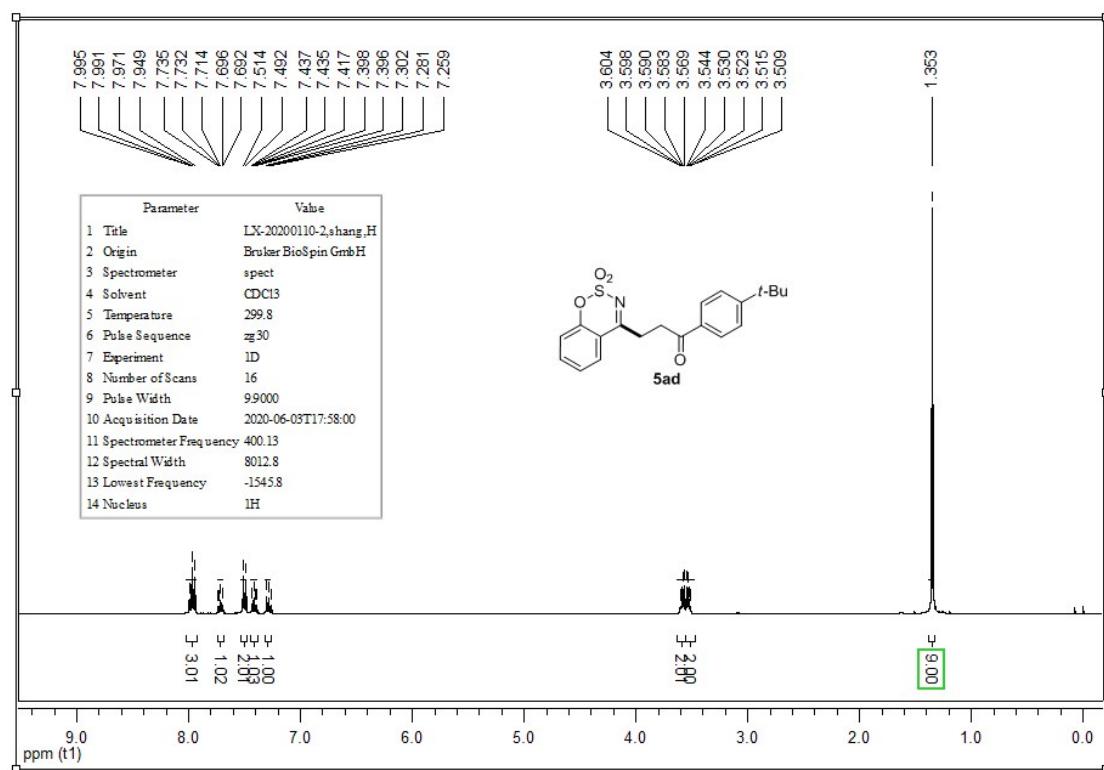


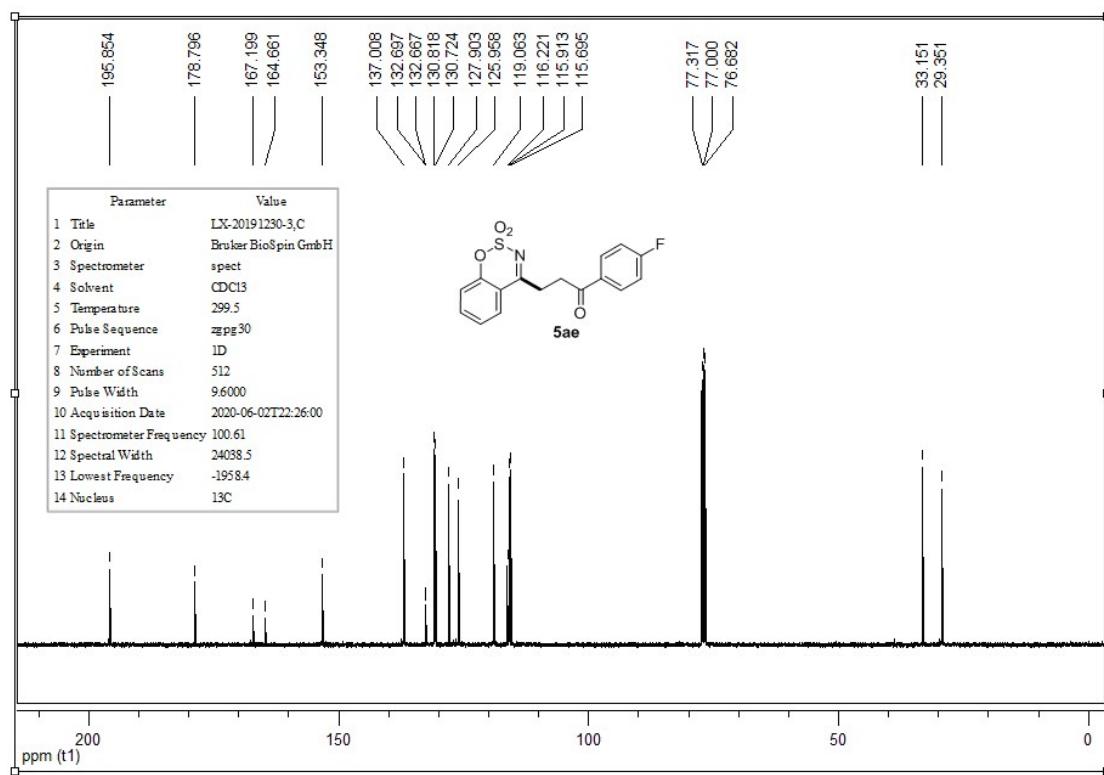
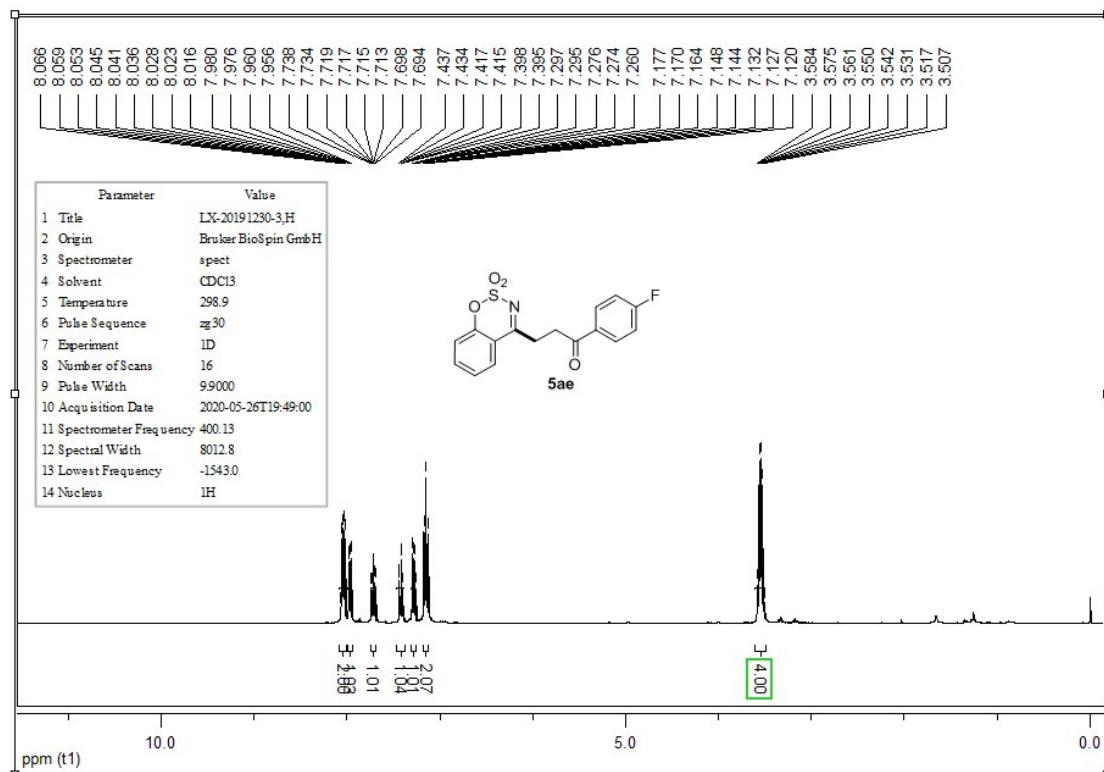


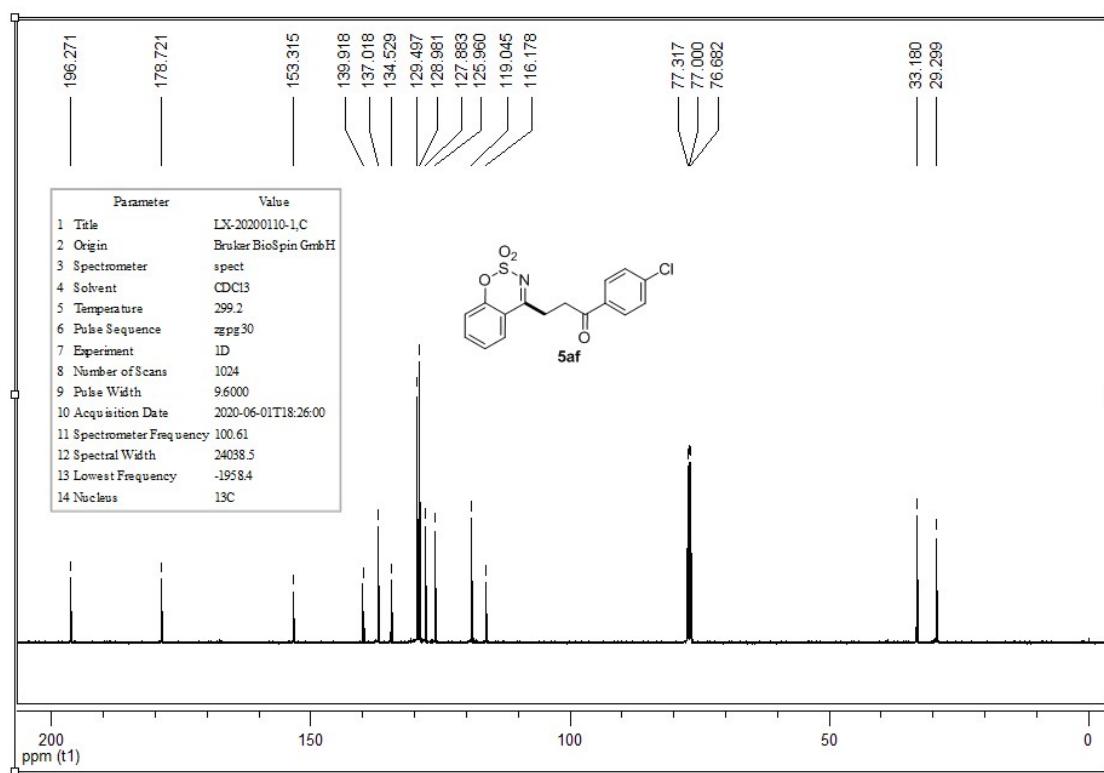
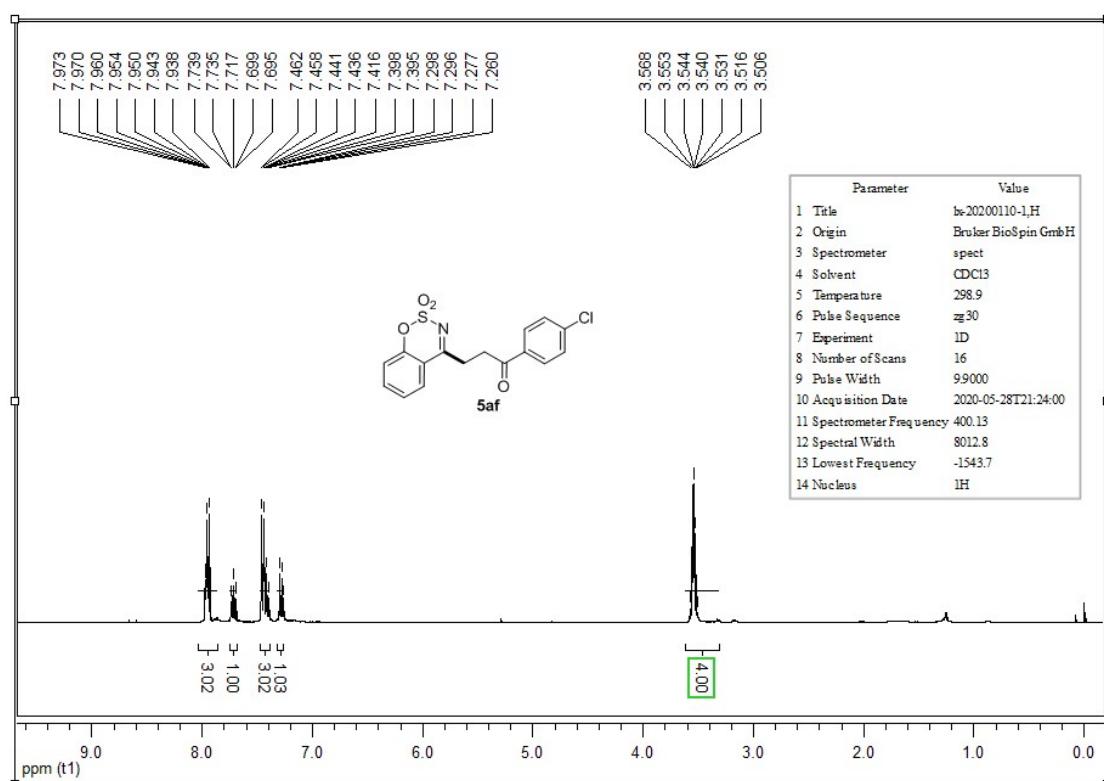


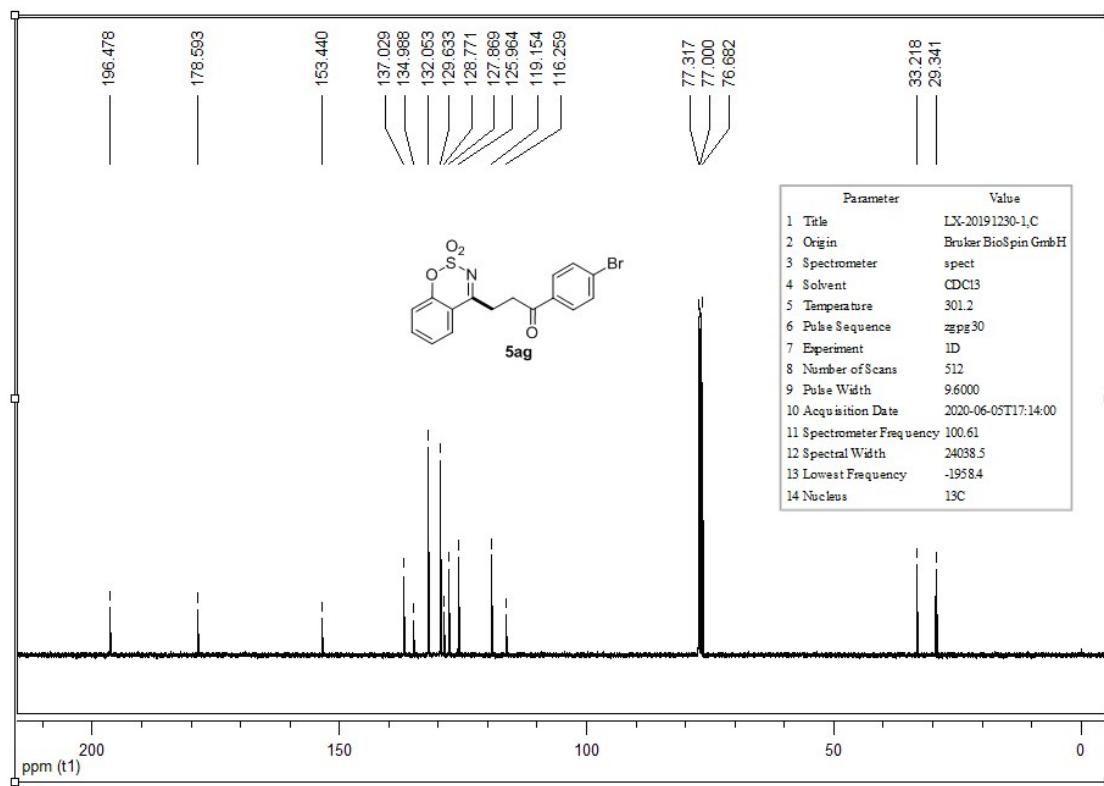
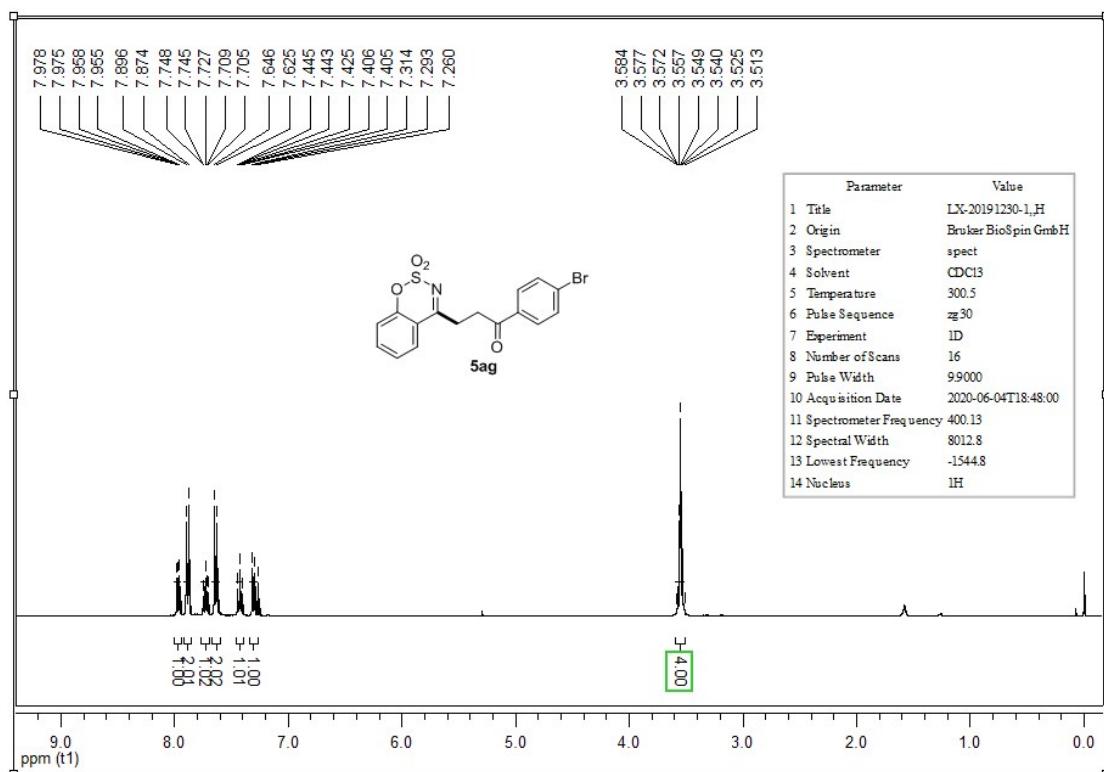


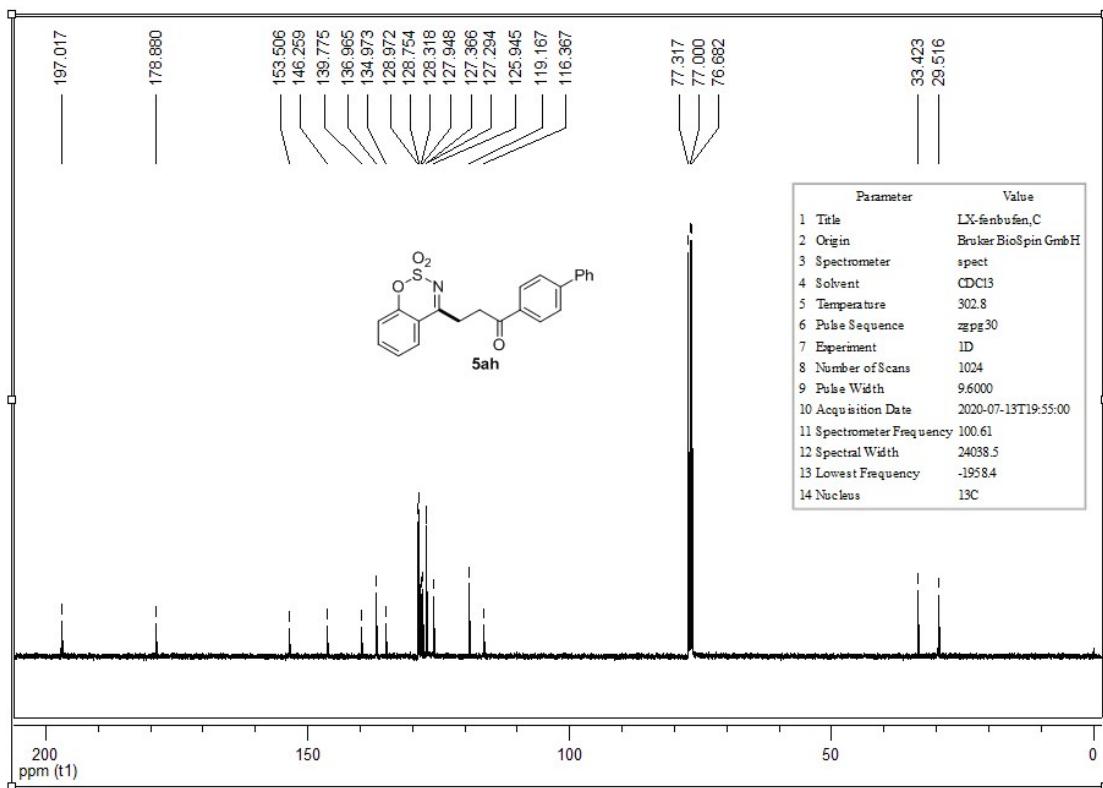
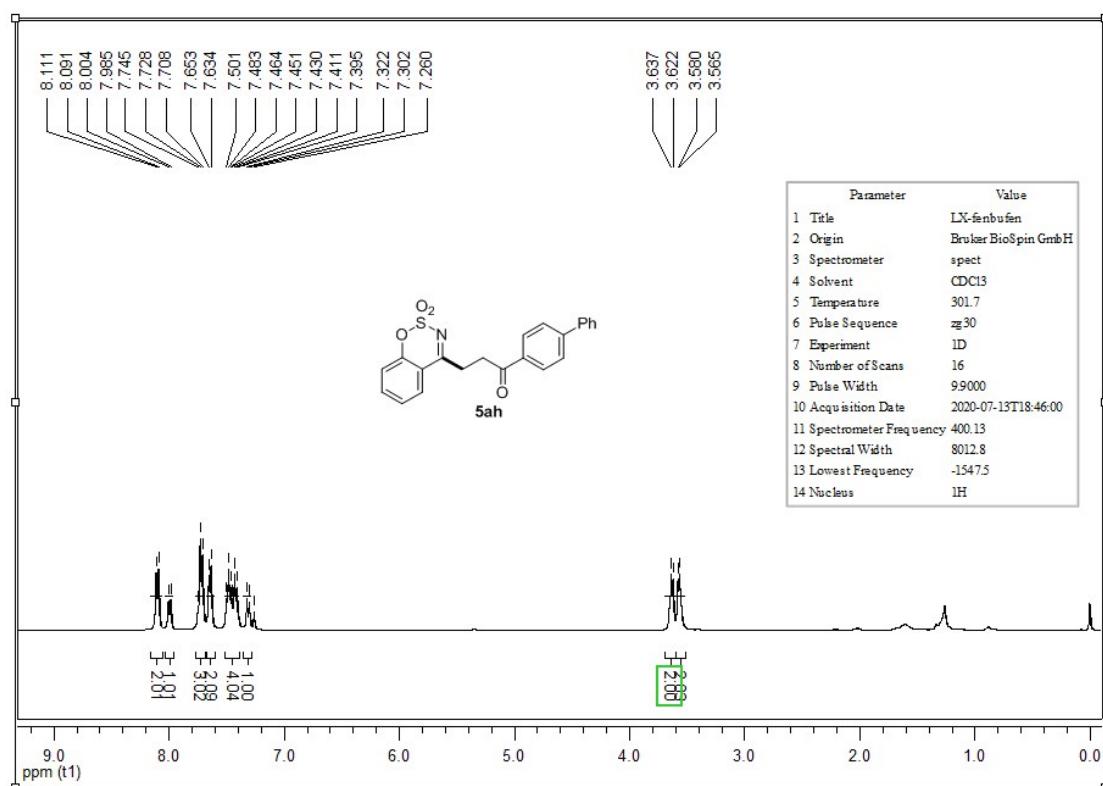


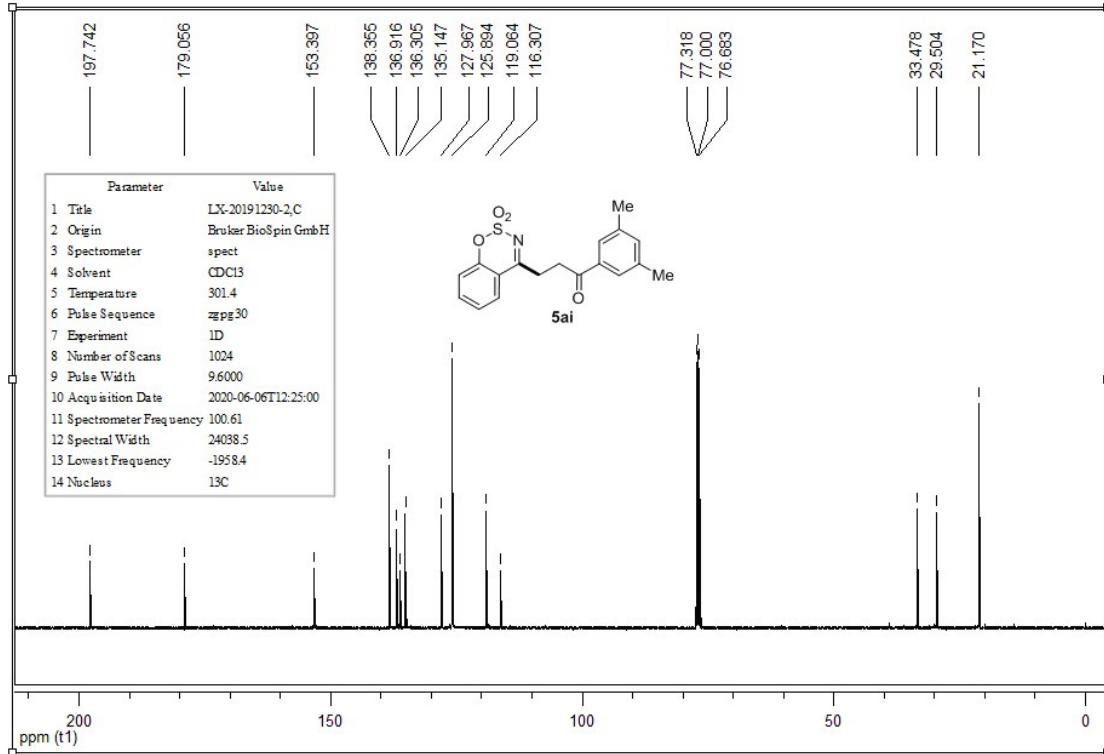
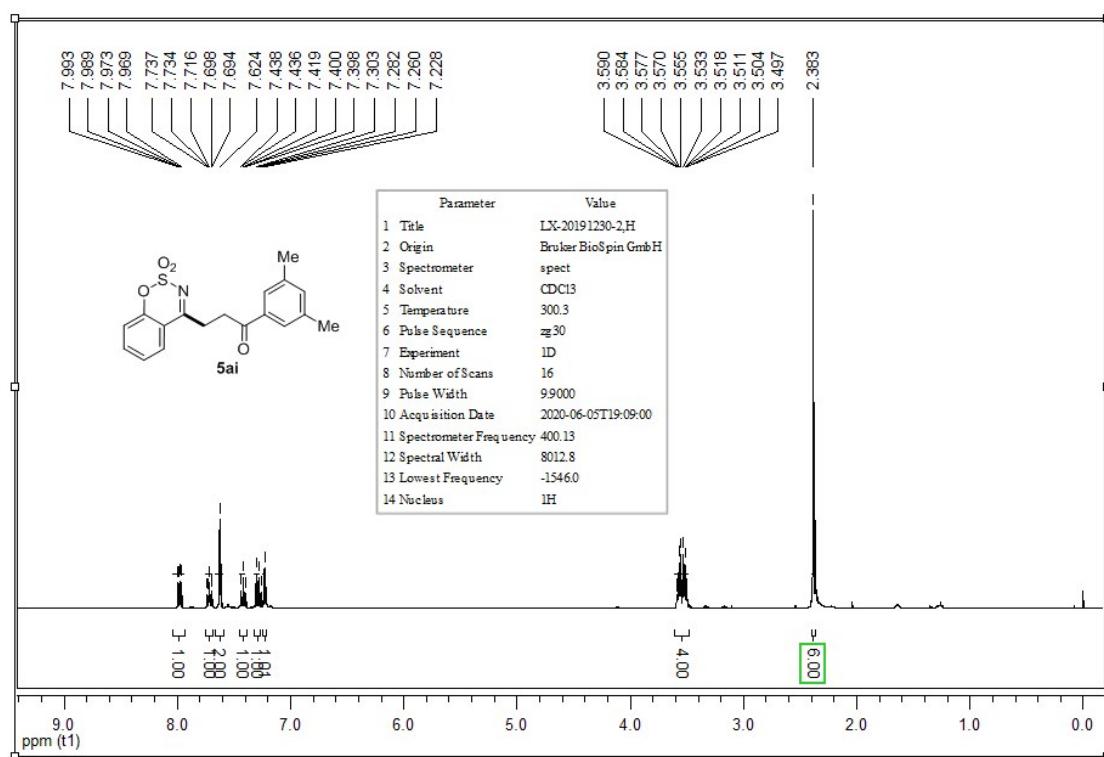


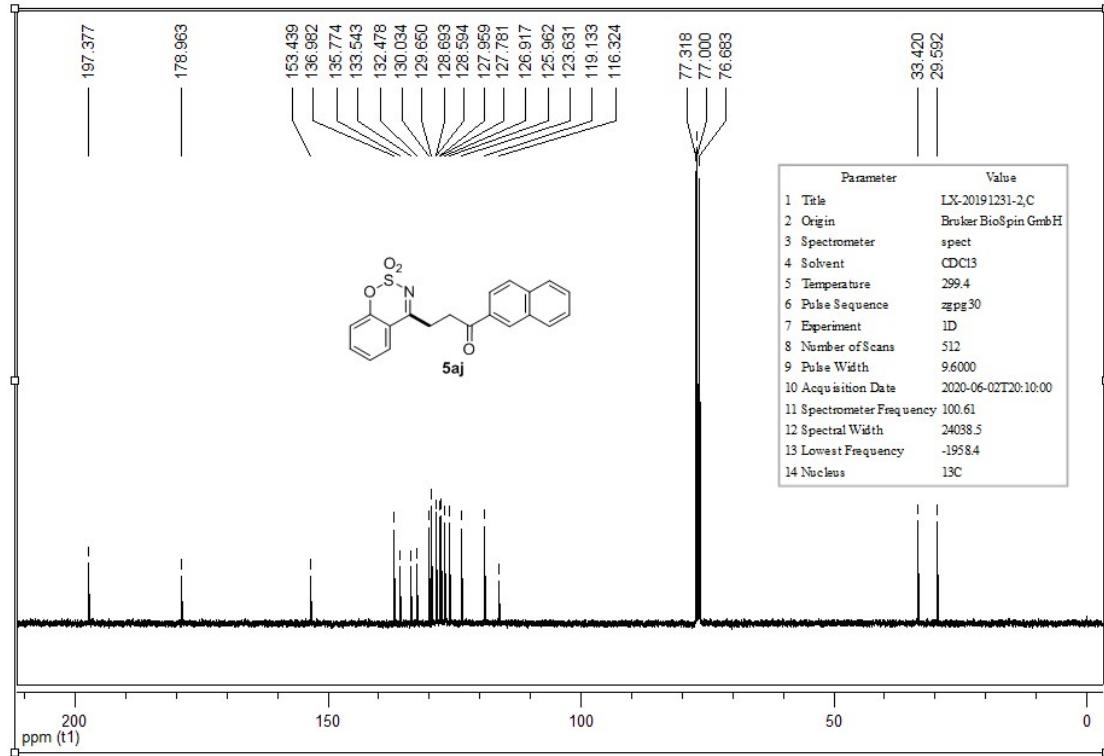
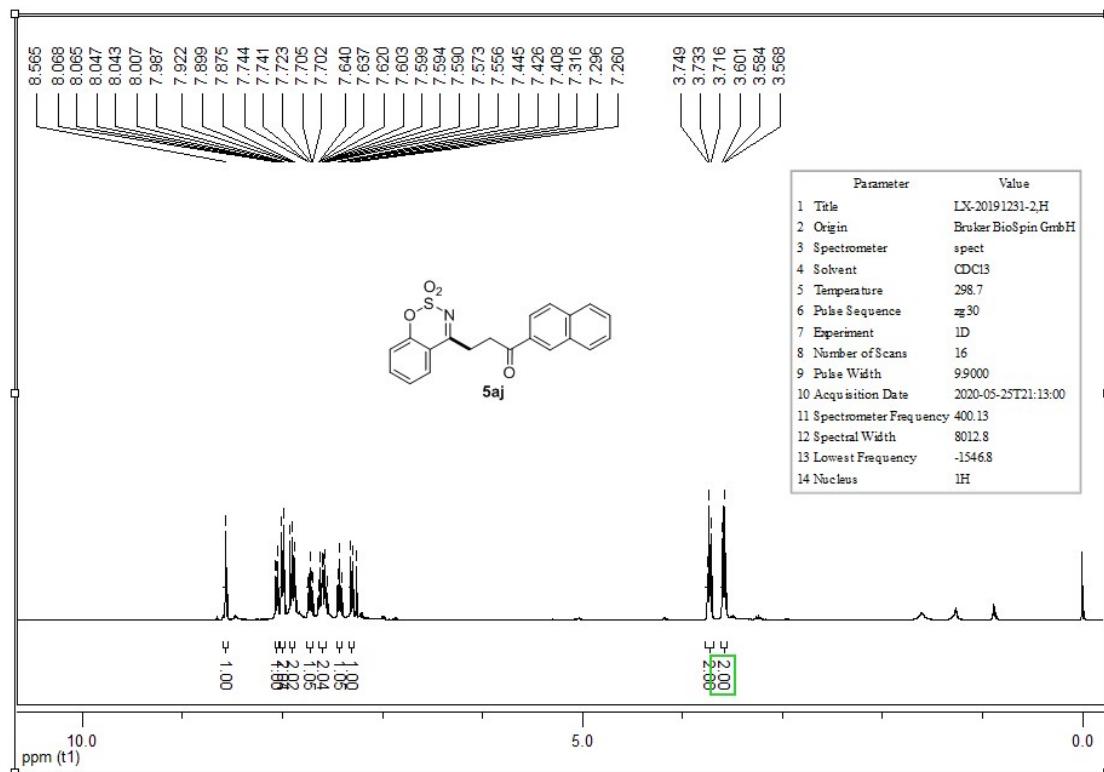


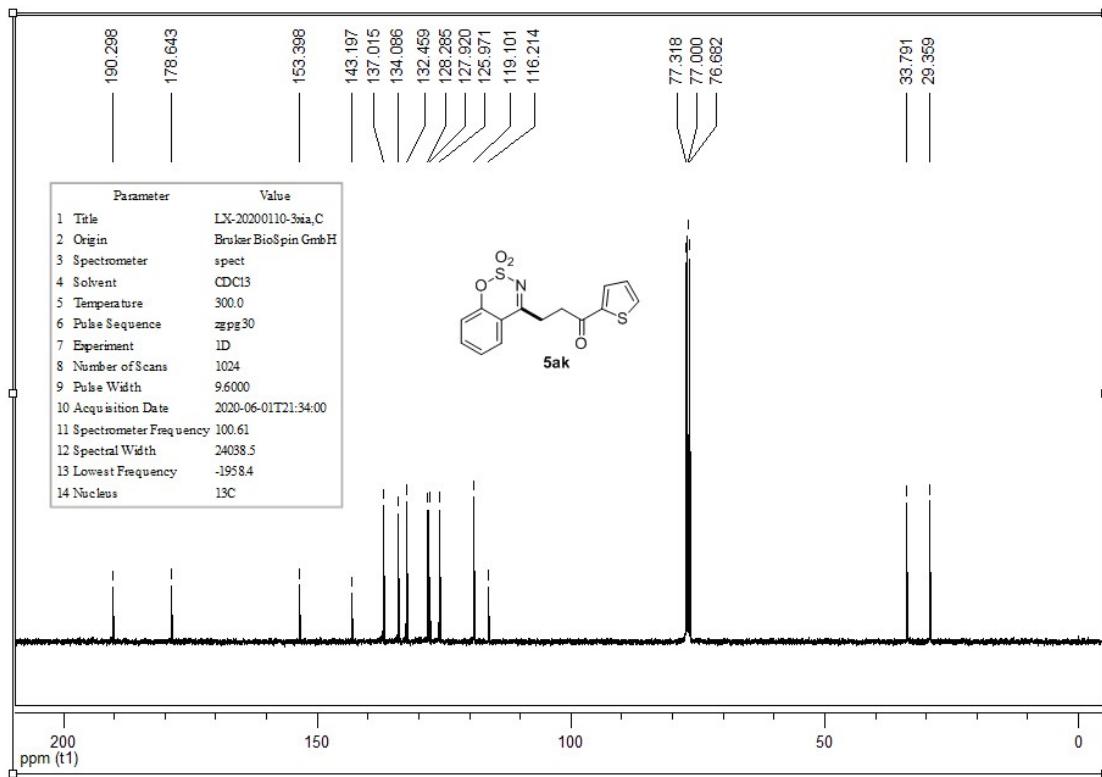
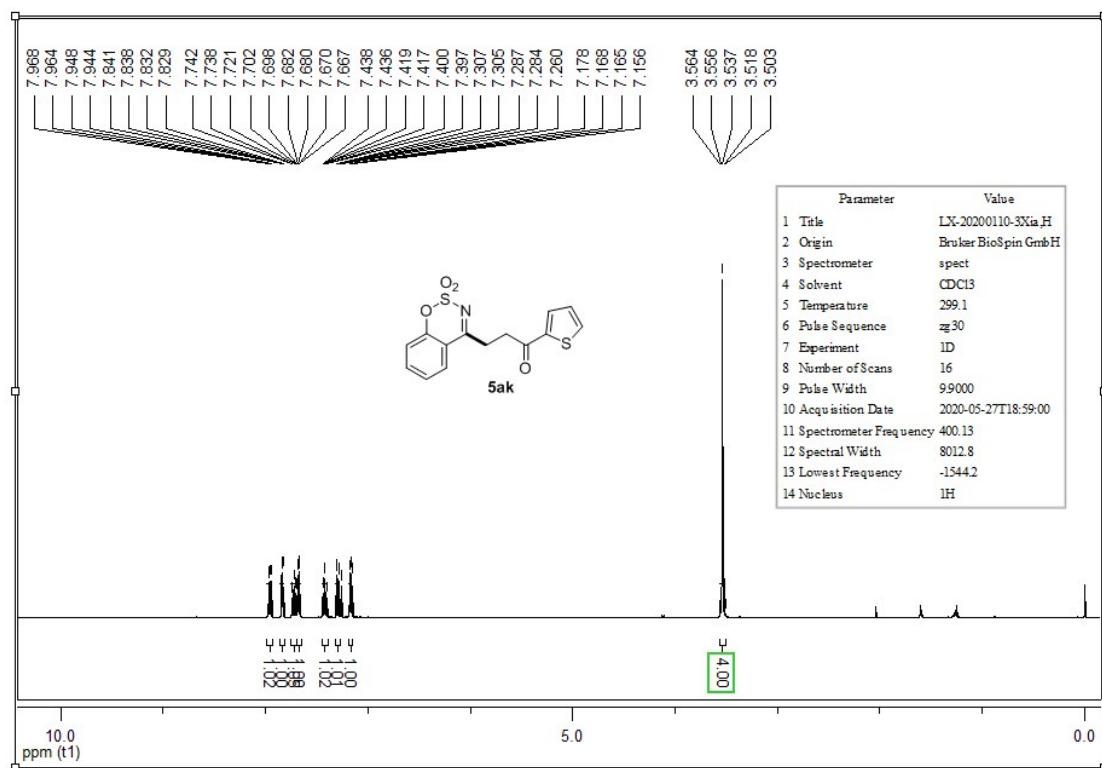


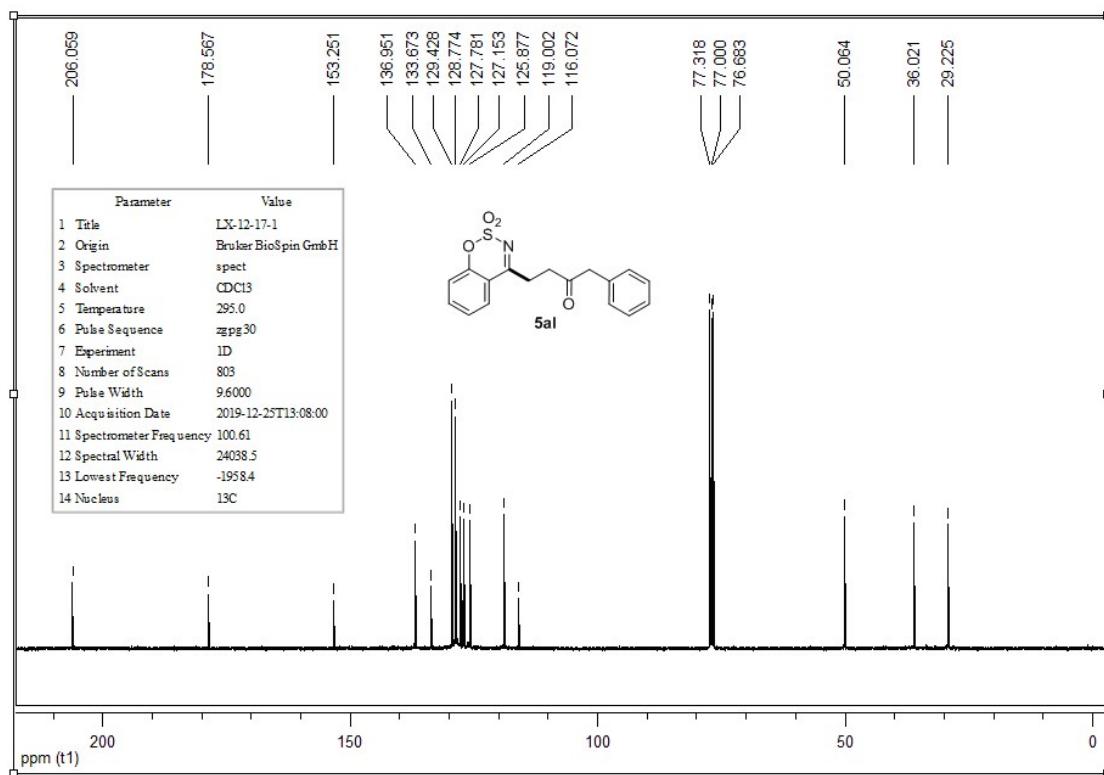
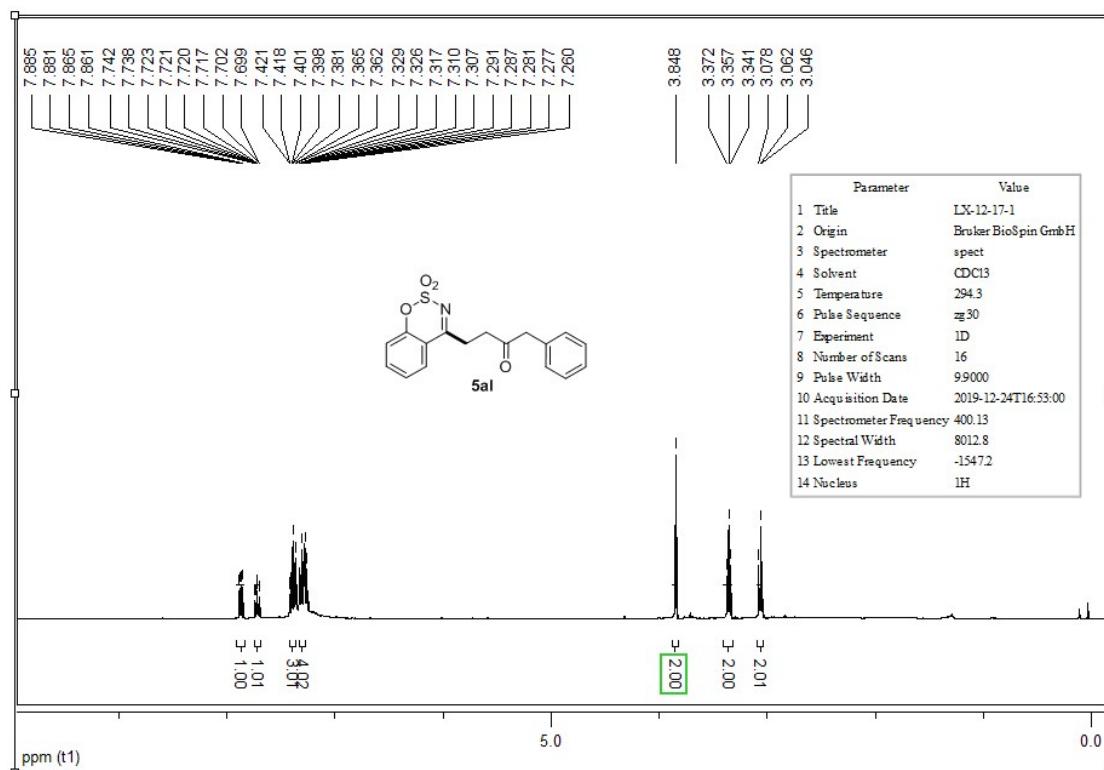


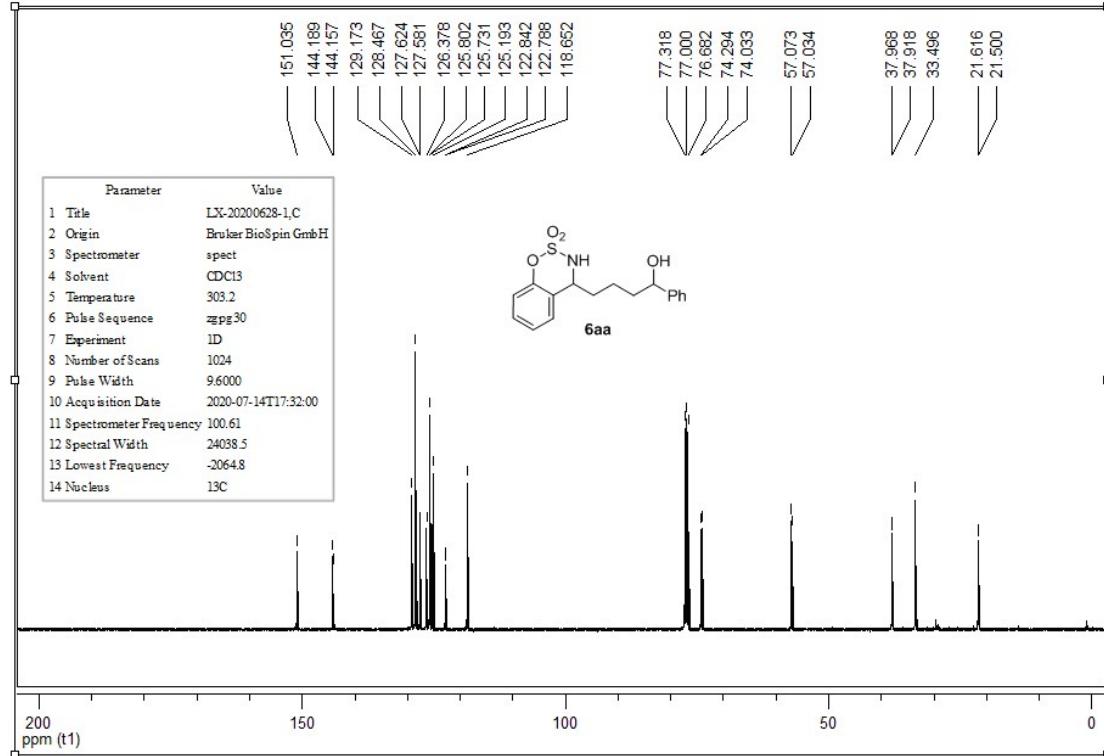
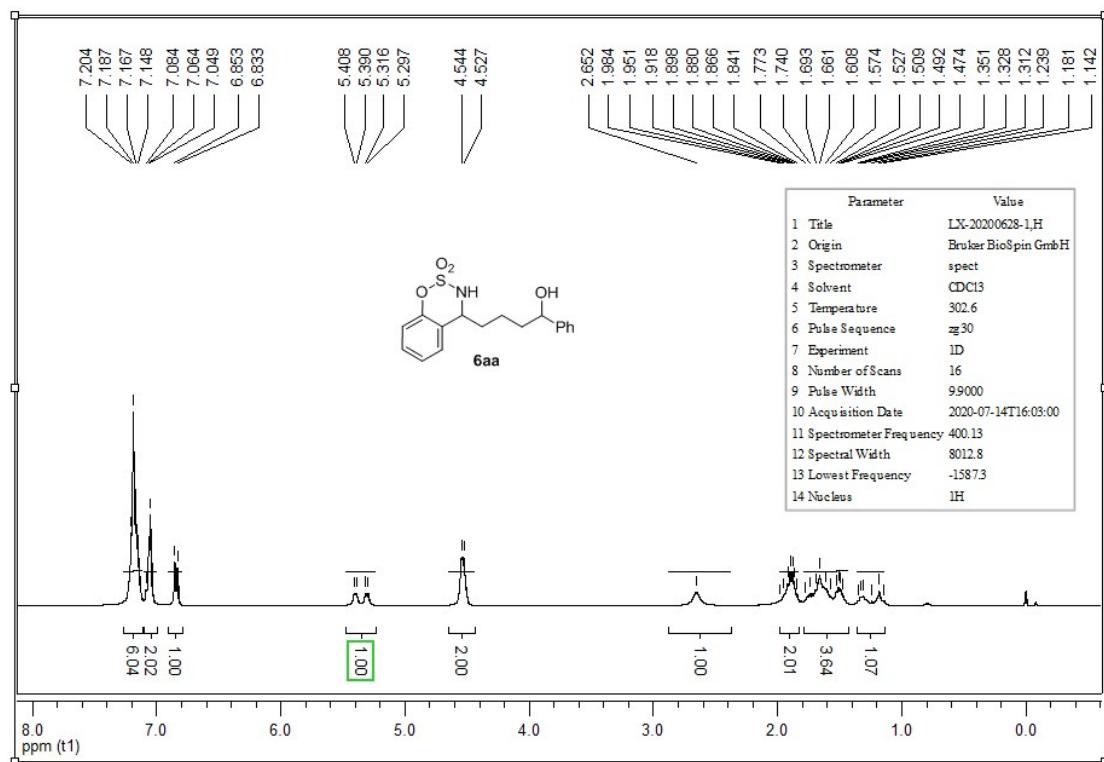


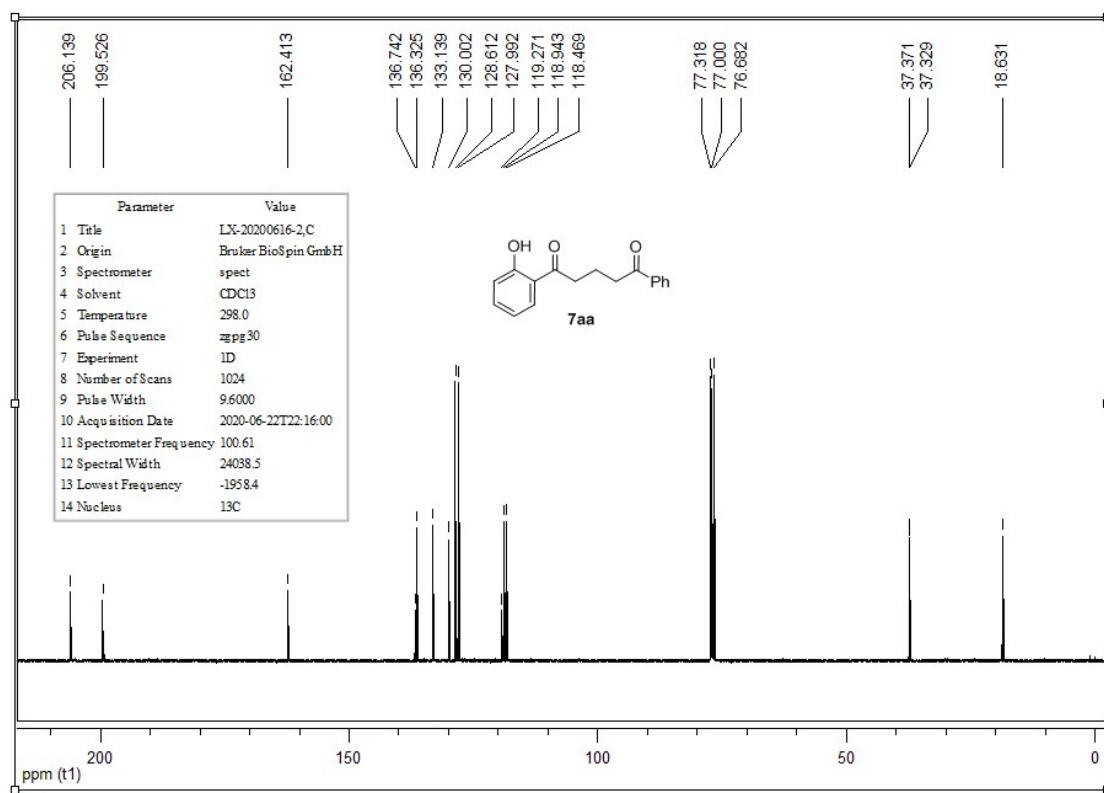
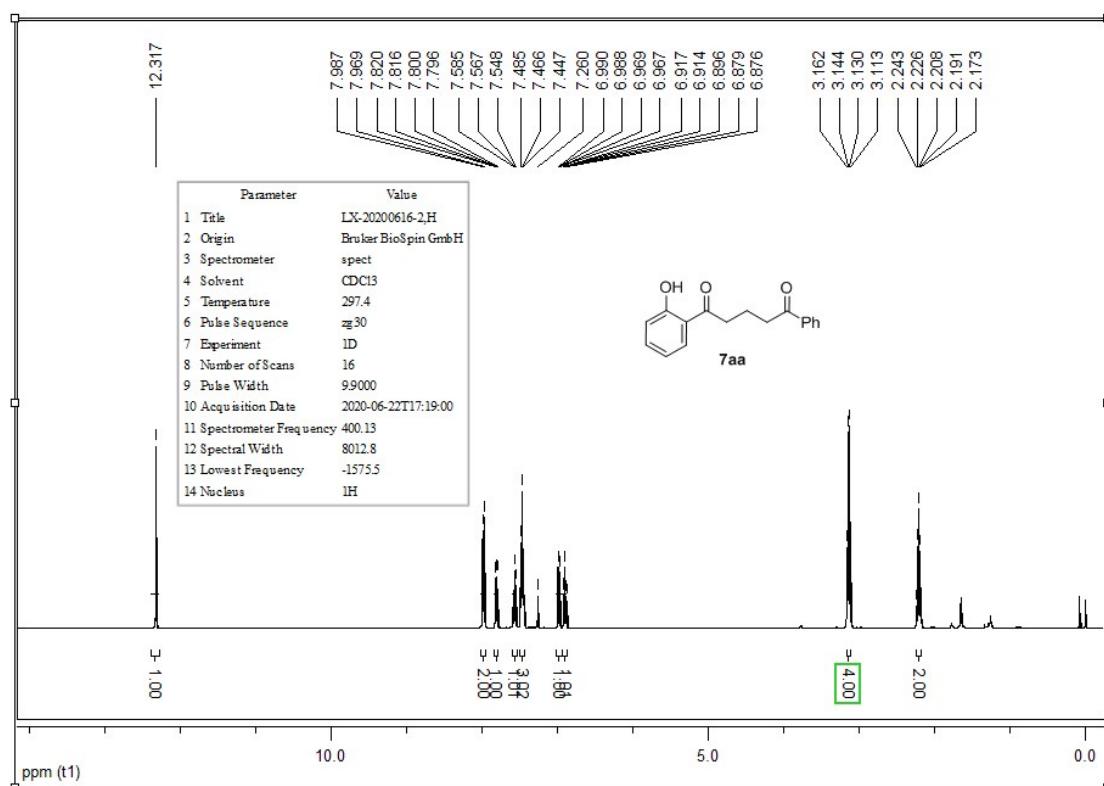


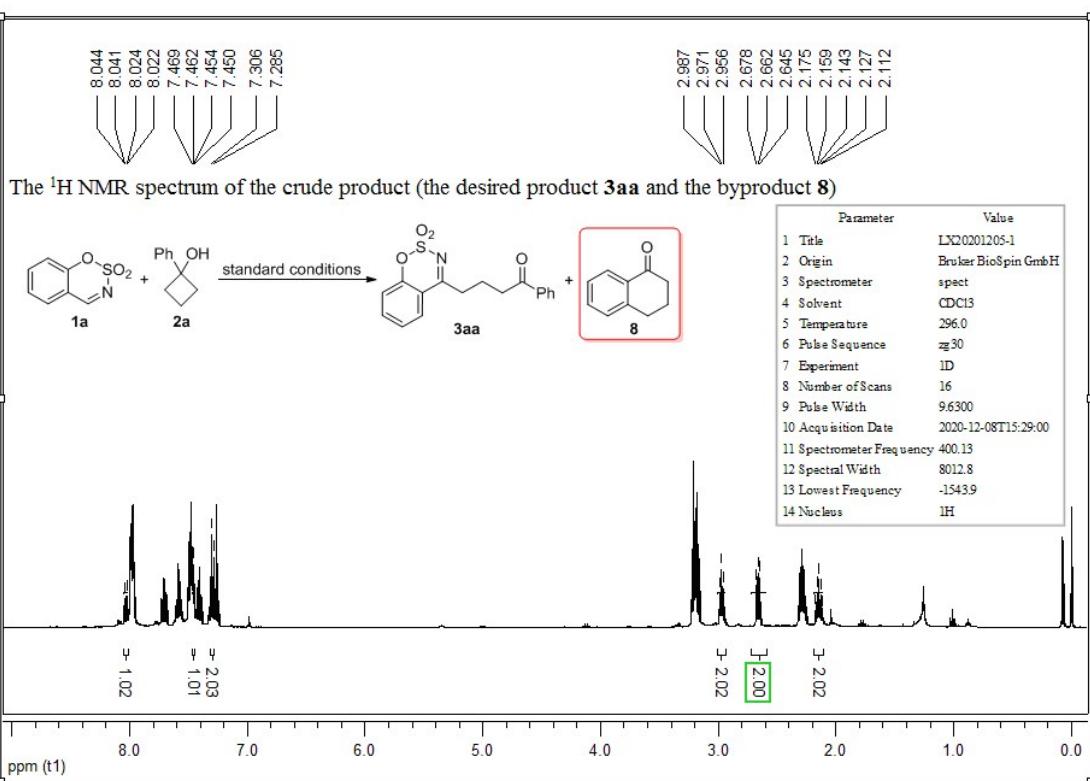
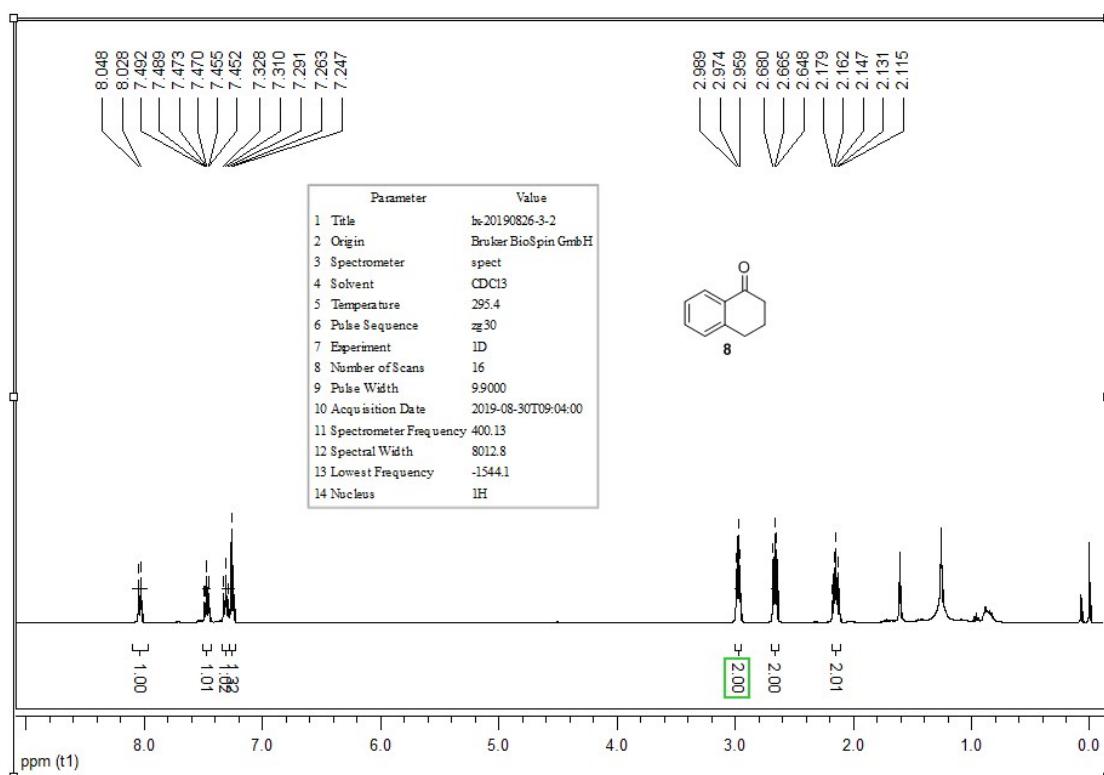


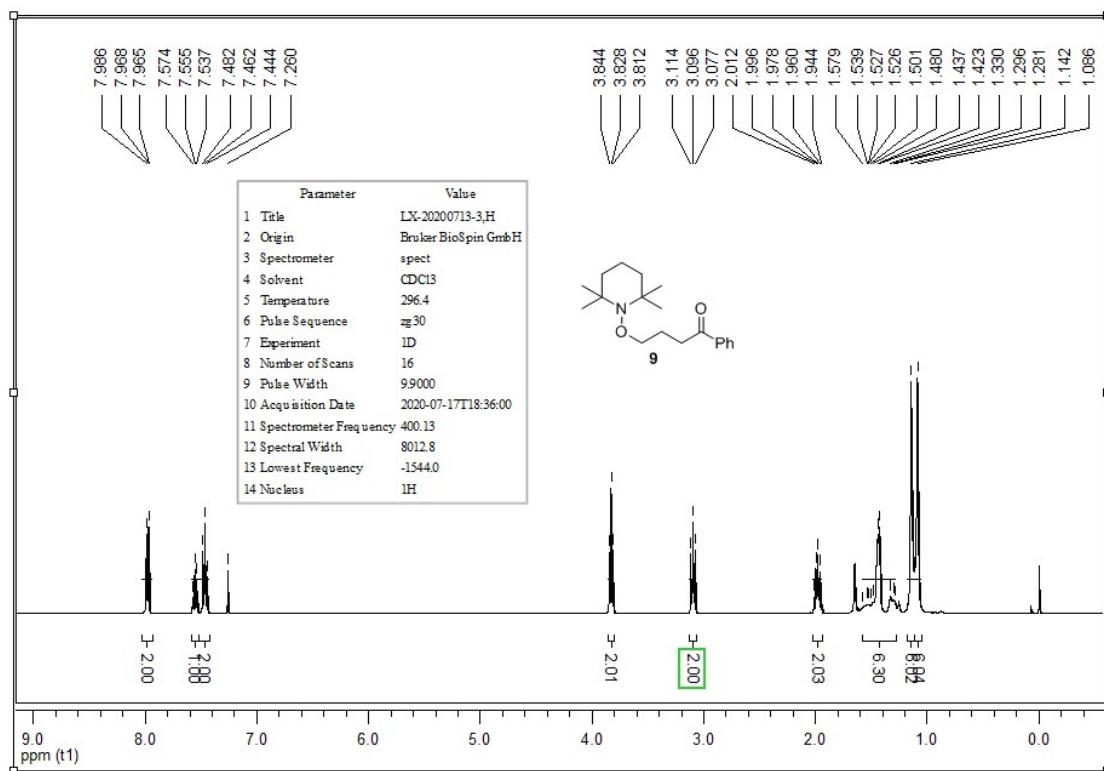
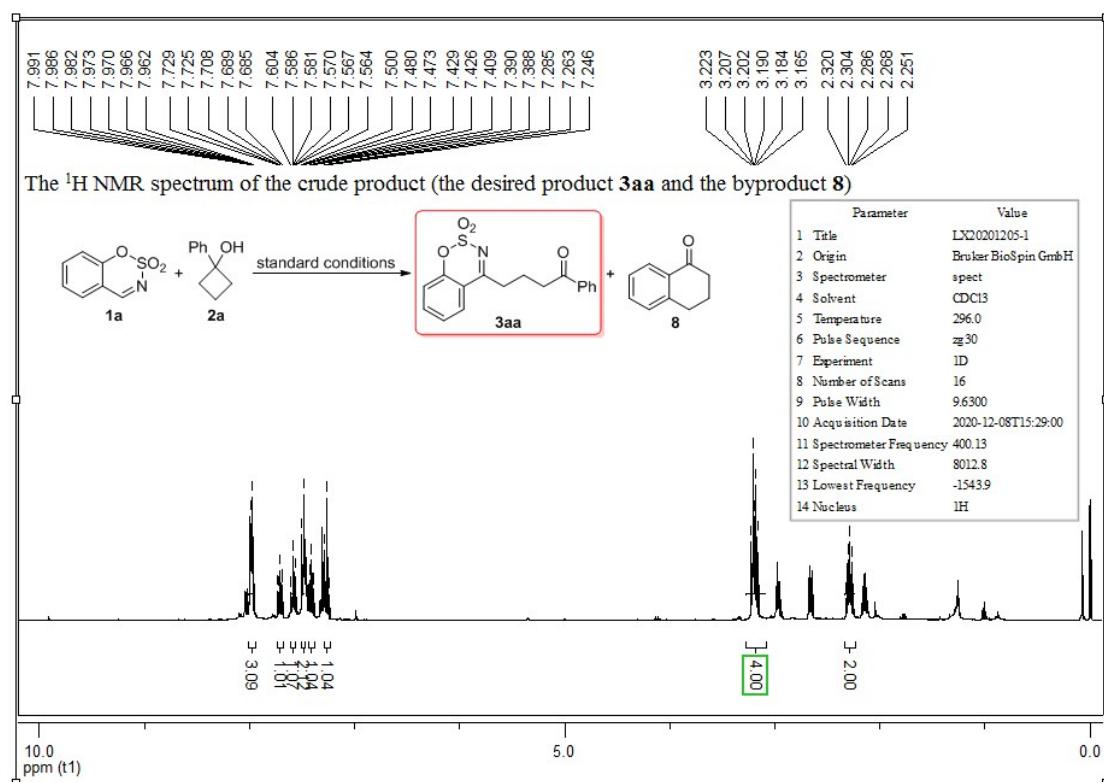


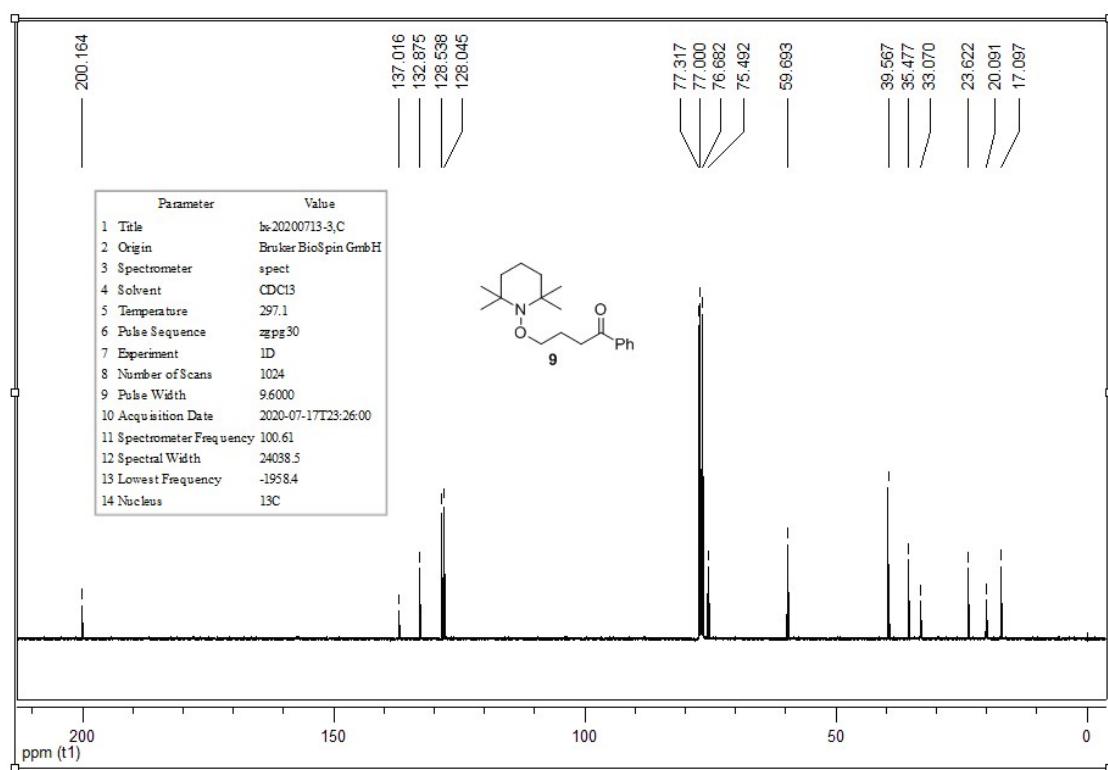












Crystallographic data for the product 3aa and 5aa:

(1) CCDC 1961173 contains the supplementary crystallographic data for the product **3aa**. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

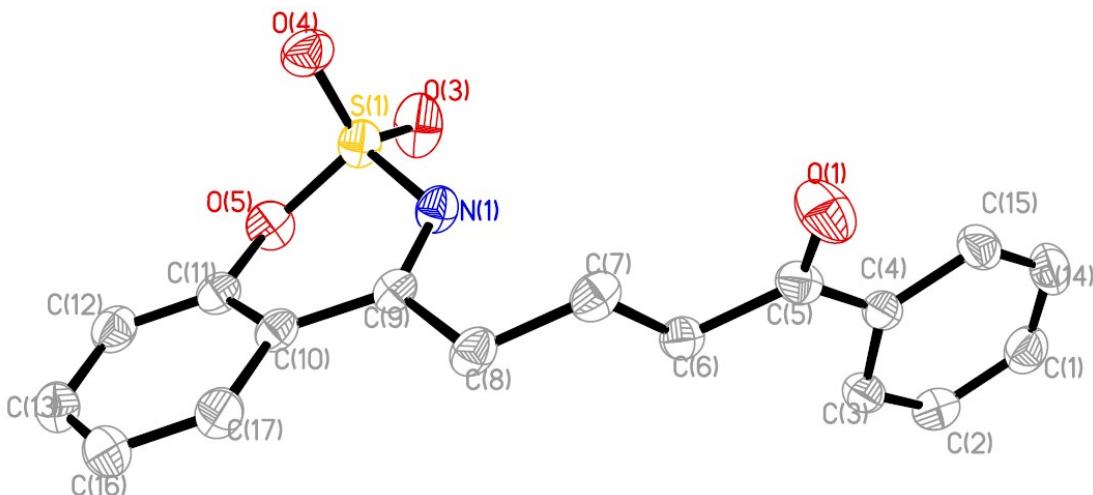


Table 1. Crystal data and structure refinement for 1.

Empirical formula	C ₁₇ H ₁₅ N O ₄ S	
Formula weight	329.36	
Temperature	298(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 5.8457(8) Å	alpha = 102.410(4) deg.
	b = 8.1317(9) Å	beta = 91.559(5) deg.
	c = 16.630(2) Å	gamma = 99.457(5) deg..
Volume	759.95(16) Å ³	
Z, Calculated density	2, 1.439 Mg/m ³	
Absorption coefficient	0.233 mm ⁻¹	
F(000)	344	
Crystal size	0.23 x 0.07 x 0.06 mm	
Theta range for data collection	3.13 to 27.52 deg.	
Limiting indices	-7<=h<=7, -10<=k<=10, -21<=l<=21	
Reflections collected / unique	14193 / 3424 [R(int) = 0.0266]	
Completeness to theta = 25.03	97.9 %	
Max. and min. transmission	0.9868 and 0.9462	
Absorption correction	None	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3424 / 0 / 208
Goodness-of-fit on F^2	1.050
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0383$, $wR_2 = 0.0969$
R indices (all data)	$R_1 = 0.0544$, $wR_2 = 0.1063$
Largest diff. peak and hole	0.192 and -0.350 e. \AA^{-3}

(2) CCDC 1972915 contains the supplementary crystallographic data for the product **5aa**. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

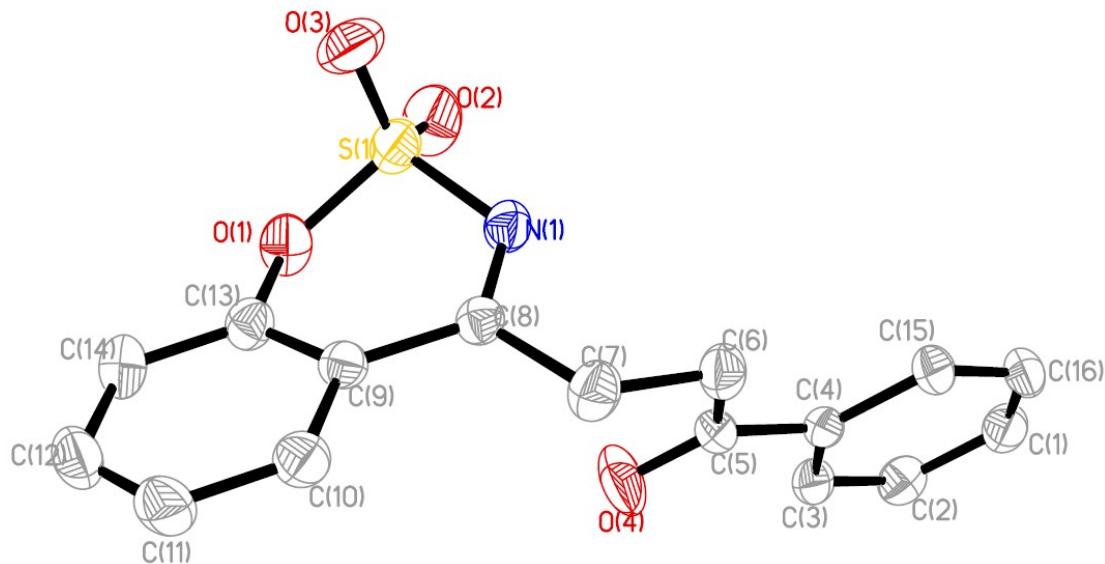


Table 1. Crystal data and structure refinement for 1.

Empirical formula	C ₁₆ H ₁₃ N O ₄ S	
Formula weight	315.33	
Temperature	297(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Monoclinic, P2(1)/n	
Unit cell dimensions	a = 16.556(2) Å b = 5.6598(7) Å c = 17.572(2) Å	alpha = 90 deg. beta = 115.833(4) deg. gamma = 90 deg.
Volume	1482.0(3) Å ³	
Z, Calculated density	4, 1.413 Mg/m ³	
Absorption coefficient	0.236 mm ⁻¹	
F(000)	656	

Crystal size	0.32 x 0.23 x 0.08 mm
Theta range for data collection	3.49 to 25.00 deg.
Limiting indices	-19<=h<=19, -6<=k<=6, -20<=l<=20
Reflections collected / unique	20112 / 2597 [R(int) = 0.1068]
Completeness to theta = 25.03	99.7 %
Absorption correction	None
Max. and min. transmission	0.9814 and 0.9284
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2597 / 0 / 199
Goodness-of-fit on F ²	1.072
Final R indices [I>2sigma(I)]	R1 = 0.0588, wR2 = 0.1191
R indices (all data)	R1 = 0.1062, wR2 = 0.1332
Largest diff. peak and hole	0.214 and -0.283 e.A ⁻³