78Electronic supporting information for:

Temperature switchable Brønsted acid-promoted selective syntheses of spiro-indolenines and quinolines

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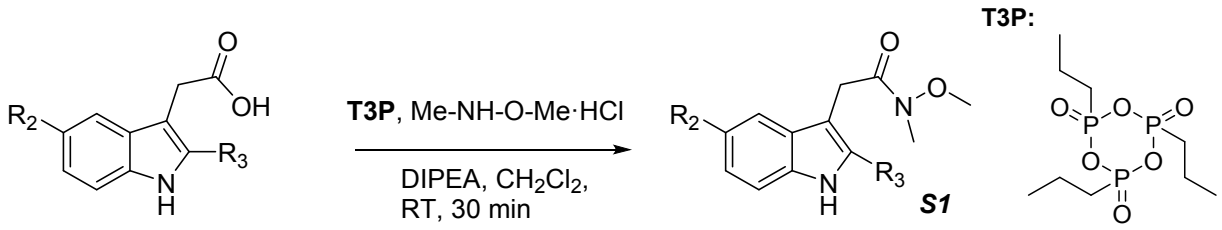
General Information

General Information NMR spectra were recorded on a 300 MHz instrument using $CDCl_3$ as solvent unless and otherwise stated (s = singlet, d = doublet, t = triplet, m = multiplet). The ^{1}H and ^{13}C chemical shifts are reported in parts per million relative to tetramethylsilane as an internal standard. For the Mass spectrometry, ion source temperature was 150-250°C, as required. High-resolution EImass spectra were performed with a resolution of 10,000. For chromatography, analytical TLC plates and 70-230 mesh silica gel were used. All the solvents and chemicals were purchased and used as available. The microwave irradiation experiments were carried out in a dedicated CEM-Discover monomode microwave apparatus, operating at a frequency of 2.45 GHz with continuous irradiation power from 0 to 300 W and utilization of the standard absorbance level of 100 W. The reactions were carried out in 10 mL glass tubes, sealed with Teflon septum and placed in the microwave cavity. The reactions were irradiated at the required set temperature for the stipulated time and then cooled to ambient temperature with air jet cooling.

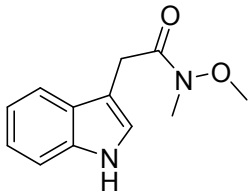
General procedure A for synthesis of Indole-3-ynones (S1)

First step: Weinreb amide formation



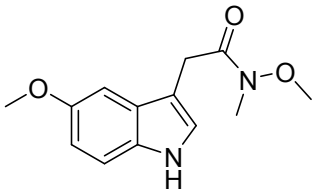
The procedure is adapted from: M. J. James, J. D. Cuthbertson, P. O'Brien, R. J. K. Taylor and W. P. Unsworth, *Angew. Chem. Int. Ed.*, 2015, **54**, 7640–7643

To a stirred solution of carboxylic acid (1.00 mmol), MeNHOMe·HCl (107 mg, 1.10 mmol) and DIPEA (0.52 mL, 3.00 mmol) in CH_2Cl_2 (2.5 mL) was added T3P (50% in EtOAc; 955 mg, 1.5 mmol). The solution was stirred for 1 h at RT. The reaction mixture was poured into water (20 mL) and acidified using aq. 10% HCl (5 mL). The organics were collected and the aqueous was extracted with EtOAc (3 × 30 mL). The organics were combined, washed with aq. 2M NaOH (20 mL), brine (20 mL), dried over Na_2SO_4 and concentrated *in vacuo* to afford the Weinreb amide product.



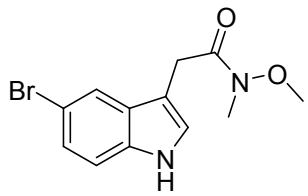
2-(1H-indol-3-yl)-N-methoxy-N-methylacetamide (S1a)

Pale brown solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.51 (s, 1H), 7.63 (d, J = 7.2 Hz, 1H), 7.26 – 7.21 (m, 1H), 7.17 – 7.06 (m, 2H), 6.95 (s, 1H), 3.89 (s, 2H), 3.62 (s, 3H), 3.20 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.02, 136.11, 127.35, 123.42, 121.78, 119.32, 118.68, 111.31, 108.48, 61.35, 32.30, 28.95. Spectroscopic data matched those reported in the literature[1]



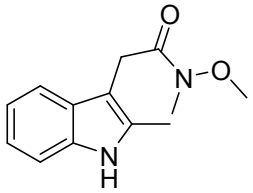
N-methoxy-2-(5-methoxy-1H-indol-3-yl)-N-methylacetamide (S1b)

Pale brown solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.17 (d, J = 8.8 Hz, 1H), 7.10 (d, J = 2.4 Hz, 1H), 7.03 (s, 1H), 6.82 (dd, J = 8.8, 2.4 Hz, 1H), 3.87 (s, 2H), 3.85 (s, 3H), 3.65 (s, 3H), 3.21 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.96, 154.01, 131.27, 127.78, 124.04, 112.18, 111.96, 108.52, 100.61, 77.48, 77.06, 76.63, 61.38, 55.87, 32.30. Spectroscopic data matched those reported in the literature[2]



2-(5-bromo-1H-indol-3-yl)-N-methoxy-N-methylacetamide (S1c)

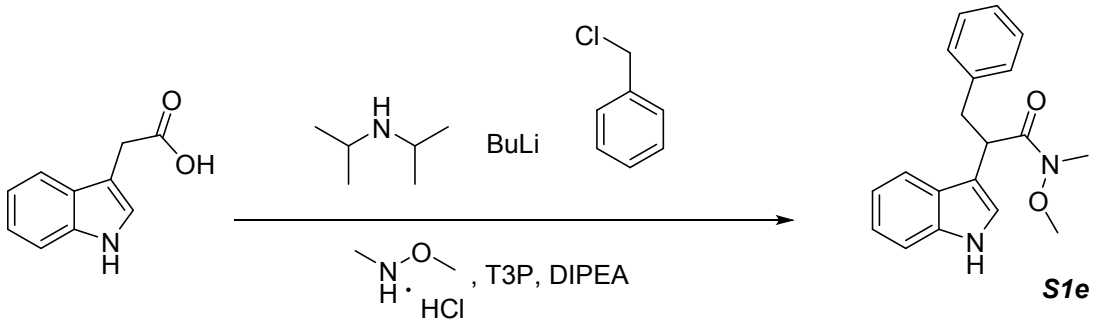
Pale brown solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (s, 1H), 7.70 (s, 1H), 7.17 (dd, J = 8.6, 1.5 Hz, 1H), 7.04 (d, J = 8.6 Hz, 1H), 6.91 (s, 1H), 3.83 (s, 2H), 3.69 (s, 3H), 3.23 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.71, 134.78, 129.12, 124.83, 124.59, 121.18, 112.80, 112.62, 108.09, 61.43, 32.37, 28.60. Spectroscopic data matched those reported in the literature[1]



N-methoxy-N-methyl-2-(2-methyl-1H-indol-3-yl)acetamide (S1d)

Pale brown solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23 (s, 1H), 7.56 – 7.51 (m, 1H), 7.09 – 7.00 (m, 3H), 3.80 (s, 2H), 3.56 (s, 3H), 3.15 (s, 3H), 2.19 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.11, 135.26, 133.11, 128.66, 120.82, 119.19, 118.13, 110.48, 104.39, 61.23, 61.12, 32.36, 28.76, 11.63. Spectroscopic data matched those reported in the literature[1]

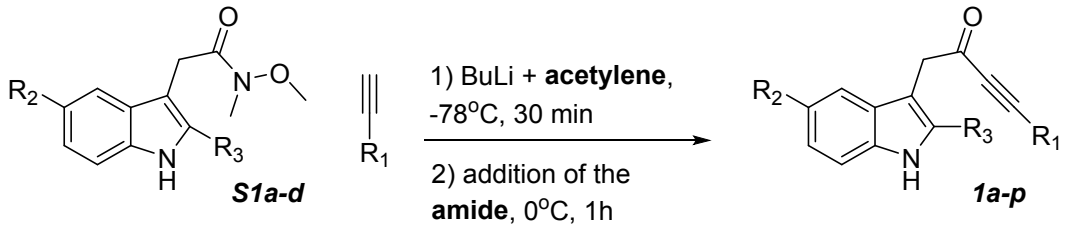
Synthetic procedure for the synthesis of S1e



The procedure is adapted from: M. J. James, J. D. Cuthbertson, P. O'Brien, R. J. K. Taylor and W. P. Unsworth, *Angew. Chem. Int. Ed.*, 2015, **54**, 7640–7643.

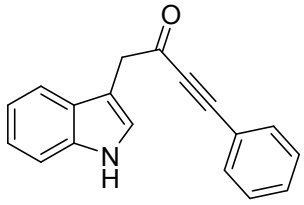
To solution of diisopropylamine (6.44 ml, 45.6 mmol) in THF (23 ml) at a 0 °C under argon was added butyllithium (18.24 ml, 45.6 mmol) dropwise. The solution was stirred for 15 min before 2-(1H-indol-3-yl)acetic acid (2 g, 11.42 mmol) in THF (12 ml) was added dropwise to the solution, which was stirred at 0 °C for 2 h. (chloromethyl)benzene (2.90 ml, 25.2 mmol) was then added dropwise to the mixture, which was allowed to warm to RT. The resulting mixture was stirred for 16 h. The reaction mixture was quenched with water (40 mL) and extracted with EtOAc (3 × 40 mL). The organics were combined, dried over $MgSO_4$ and concentrated *in vacuo*. The crude material was dissolved in DCM (43 ml), to which N,O-dimethylhydroxylamine hydrochloride (1,225 g, 12,56 mmol), DIPEA (5.96 ml, 34.2 mmol) and T3P (10.20 ml, 17.14 mmol) were added successively. The mixture was stirred for 1h at RT The reaction mixture was poured into water (60 mL) and the organics were collected. The aqueous was extracted with EtOAc (2 × 60 mL). The organics were combined, washed with aq. 10% HCl (2 × 40 mL), aq. 2M NaOH (40 mL) and brine (40 mL) then dried over $MgSO_4$ and concentrated *in vacuo*. The crude material was purified by column chromatography to afford the title compound as a pale brown solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.73 (d, J = 7.7 Hz, 1H), 7.35 (d, J = 7.9 Hz, 1H), 7.26 – 7.21 (m, 4H), 7.20 – 7.09 (m, 4H), 4.73 – 4.60 (m, 1H), 3.50 (dd, J = 13.3, 9.7 Hz, 1H), 3.26 (s, 3H), 3.15 – 3.07 (m, 4H). Spectroscopic data matched those reported in the literature[1]

Second step: Weinreb ketone synthesis



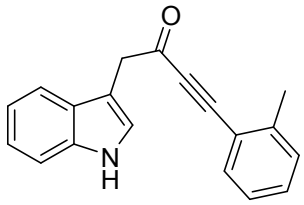
The procedure is adapted from: M. J. James, J. D. Cuthbertson, P. O'Brien, R. J. K. Taylor and W. P. Unsworth, *Angew. Chem. Int. Ed.*, 2015, **54**, 7640–7643

To a solution of terminal alkyne (3.0 mmol) in THF (3 mL) at −78 °C under nitrogen was added n-BuLi (1.0 mL, 2.5 mmol, 2.5 M in hexane). The resulting solution was stirred at −78 °C for 30 min, then a solution of Weinreb amide (1.0 mmol) in THF (9 mL) was quickly added to the solution. The mixture was stirred at −78 °C for 5 min then warmed to 0 °C and stirred for another 1 h. The reaction was then re-cooled to −78 °C and quenched with sat. aq. NH_4Cl (30 mL), allowed to warm to RT, diluted with water (70 mL), extracted with ethyl acetate (3 × 100 mL), dried over $MgSO_4$ and concentrated *in vacuo*. Purification by flash column chromatography (20-40% of ethyl acetate in heptane) afforded the desired ynone.



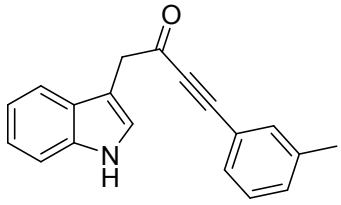
1-(1H-indol-3-yl)-4-phenylbut-3-yn-2-one (1a)

Light-brown solid; R_f=0.45 (Heptane/Ethyl acetate 1:1); Yield 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.66 (dd, *J* = 7.9, 0.5 Hz, 1H), 7.41 – 7.33 (m, 2H), 7.32 – 7.27 (m, 1H), 7.24 – 7.13 (m, 2H), 4.08 (s, *J* = 0.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 185.66, 136.17, 133.08, 130.64, 128.49, 127.46, 123.72, 122.30, 119.91, 119.84, 118.92, 111.31, 107.61, 92.08, 87.99, 42.00; HRMS (ESI) m/z: calculated for $C_{18}H_{13}NO^+$ $[M + H]^+$: 260.1069, found: 260.1074. Spectroscopic data matched those reported in the literature[1]



1-(1H-indol-3-yl)-4-(o-tolyl)but-3-yn-2-one (1b)

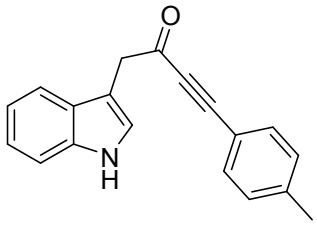
Brown solid; R_f=0.46 (Heptane/Ethyl acetate 1:1); Yield 67%; mp 109-111 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23 (s, 1H), 7.63 (d, *J* = 7.8 Hz, 1H), 7.37 – 7.19 (m, 4H), 7.18 – 7.07 (m, 4H), 4.08 (s, 2H), 2.21 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.95, 142.22, 136.19, 133.62, 130.70, 129.65, 127.45, 125.72, 123.76, 122.29, 119.82, 119.69, 118.83, 111.30, 107.63, 91.73, 91.19, 42.05, 20.26; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1220.



1-(1H-indol-3-yl)-4-(m-tolyl)but-3-yn-2-one (1c)

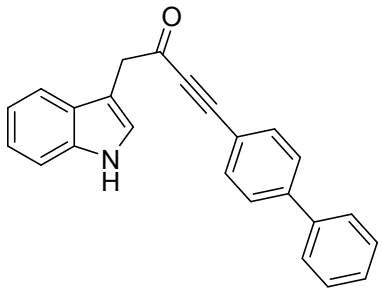
Brown solid; R_f=0.46 (Heptane/Ethyl acetate 1:1); Yield 78%; mp 98-99 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (s, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.24 – 7.13 (m, 6H), 7.10 (s, 1H), 4.07 (s, 2H), 2.28 (s,

3H).[13]C NMR (101 MHz, $CDCl_3$) δ 185.81, 138.35, 136.23, 133.68, 131.63, 130.22, 128.41, 127.54, 123.79, 122.31, 119.86, 119.72, 118.99, 111.36, 107.70, 92.63, 87.83, 42.03, 21.13; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1222, found:



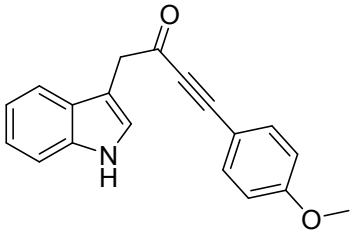
1-(1H-indol-3-yl)-4-(p-tolyl)but-3-yn-2-one (1d)

Brown solid; R_f=0.46 (Heptane/Ethyl acetate 1:1); Yield 70%; mp 86-88 °C; [1]H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.24 – 7.08 (m, 7H), 4.07 (s, 2H), 2.33 (s, 3H). [13]C NMR (101 MHz, $CDCl_3$) δ 185.76, 141.39, 136.17, 133.11, 129.30, 127.48, 123.71, 122.25, 119.79, 118.94, 116.79, 111.30, 107.71, 92.82, 87.90, 41.97, 21.69; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1229



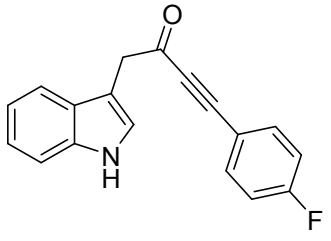
4-([1,1'-biphenyl]-4-yl)-1-(1H-indol-3-yl)but-3-yn-2-one (1e)

Brown solid; R_f=0.46 (Heptane/Ethyl acetate 1:1); Yield 17%; [1]H NMR (300 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.69 (d, *J* = 7.7 Hz, 1H), 7.57 – 7.51 (m, 4H), 7.47 – 7.37 (m, 6H), 7.24 – 7.15 (m, 4H), 4.10 (s, 2H). [13]C NMR (101 MHz, $CDCl_3$) δ 185.66, 143.44, 139.76, 136.19, 134.25, 133.59, 128.98, 128.95, 128.14, 127.51, 127.15, 127.09, 123.72, 122.33, 119.88, 118.97, 118.63, 111.31, 107.73, 92.22, 88.67, 41.99, 29.70 HRMS (ESI) m/z: calculated for $C_{24}H_{17}NO^+$ $[M + H]^+$: 336.1382, found: 336.1386



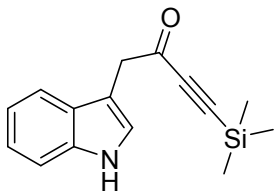
1-(1H-indol-3-yl)-4-(4-methoxyphenyl)but-3-yn-2-one (1f)

Light-brown solid; R_f=0.37 (Heptane/Ethyl acetate 1:1); Yield 70%; [1]H NMR (300 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.67 (d, *J* = 7.7 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.30 – 7.12 (m, 5H), 6.83 – 6.76 (m, 2H), 4.06 (s, 2H), 3.79 (s, 3H); [13]C NMR (101 MHz, $CDCl_3$) δ 185.75, 161.59, 136.18, 135.14, 127.53, 123.67, 122.25, 119.79, 118.98, 114.24, 111.70, 111.29, 107.93, 93.37, 87.98, 55.38, 41.88 HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 289.1103, found: 289.1106. Spectroscopic data matched those reported in the literature[1]



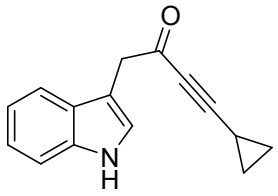
4-(4-fluorophenyl)-1-(1H-indol-3-yl)but-3-yn-2-one (1g)

Dark-orange solid; R_f=0.43 (Heptane/Ethyl acetate 1:1); Yield 62%; [1]H NMR (300 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.33 – 7.12 (m, 5H), 7.02 – 6.93 (m, 2H), 4.07 (s, 2H). [13]C NMR (75 MHz, $CDCl_3$) δ 185.61, 163.92 (d, *J* = 253.7 Hz), 136.21, 135.39 (d, *J* = 8.9 Hz), 127.50, 123.76, 122.36, 119.89, 118.94, 116.19, 116.04 (d, *J* = 22.3 Hz), 115.89, 111.38, 107.63, 91.13, 87.90, 41.95 HRMS (ESI) m/z: calculated for $C_{18}H_{12}FNO^+$ $[M + H]^+$: 278.0975, found: 278.0979 Spectroscopic data matched those reported in the literature[3]



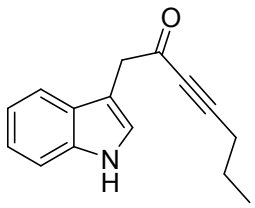
1-(1H-indol-3-yl)-4-(trimethylsilyl)but-3-yn-2-one (1h)

Brown solid; R_f=0.40 (Heptane/Ethyl acetate 1:1); Yield 61%; mp 91-93 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.59 (d, *J* = 7.3 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.23 – 7.10 (m, 2H), 3.99 (s, 1H), 0.14 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.18, 137.02, 128.26, 124.60, 123.15, 120.64, 119.84, 112.15, 108.17, 102.95, 100.22, 42.75 HRMS (ESI) m/z: calculated for $C_{15}H_{17}NOSi^+$ $[M + H]^+$: 256.1152, found: 256.1154 Spectroscopic data matched those reported in the literature[1]



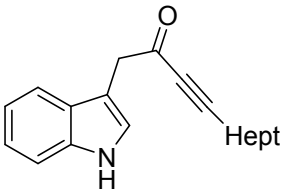
4-cyclopropyl-1-(1H-indol-3-yl)but-3-yn-2-one (1i)

Brown solid; R_f=0.45 (Heptane/Ethyl acetate 1:1); Yield 92%; mp 63-65 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.57 (d, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.23 – 7.16 (m, 1H), 7.16 – 7.08 (m, 2H), 3.92 (s, 2H), 1.32 – 1.23 (m, 1H), 0.90 – 0.82 (m, 2H), 0.72 – 0.65 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.92, 136.38, 127.67, 123.85, 122.47, 119.93, 119.13, 111.53, 108.11, 100.67, 42.09, 10.01 HRMS (ESI) m/z: calculated for $C_{15}H_{13}NO^+$ $[M + H]^+$: 224.1069, found: 224.1075. Spectroscopic data matched those reported in the literature[3]



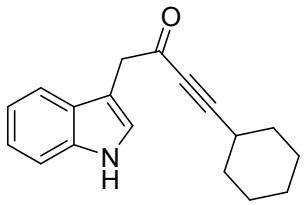
1-(1H-indol-3-yl)hept-3-yn-2-one (1j)

Light-brown solid; R_f=0.47 (Heptane/Ethyl acetate 1:1); Yield 64%; mp 62-64 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.24 (s, 1H), 7.57 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 8.2 Hz, 1H), 7.23 – 7.06 (m, 2H), 7.05 (s, 1H), 3.95 (s, 2H), 2.22 (t, *J* = 7.0 Hz, 2H), 1.55 – 1.38 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.06, 136.13, 127.32, 123.71, 122.11, 119.60, 118.77, 111.32, 107.42, 95.73, 81.07, 42.09, 21.06, 20.88, 13.33 HRMS (ESI) m/z: calculated for $C_{15}H_{15}NO^+$ $[M + H]^+$: 226.1226, found: 226.1232



1-(1H-indol-3-yl)undec-3-yn-2-one (1k)

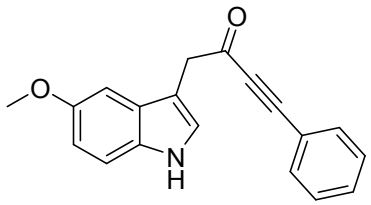
Brown sticky oil; R_f=0.49 (Heptane/Ethyl acetate 1:1); Yield 72%; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.58 (d, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.23 – 7.08 (m, 1H), 3.96 (s, 1H), 2.25 (t, *J* = 7.1 Hz, 1H), 1.49 – 1.38 (m, 1H), 1.31 – 1.16 (m, 3H), 0.88 (t, *J* = 6.8 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.94, 136.14, 127.39, 123.63, 122.22, 119.70, 118.87, 111.28, 107.67, 95.89, 80.98, 42.10, 31.63, 28.73, 28.69, 27.57, 22.63, 19.01, 14.12 HRMS (ESI) m/z: calculated for $C_{19}H_{23}NO^+$ $[M + H]^+$: 282.1853, found: 282.1862.



4-cyclohexyl-1-(1H-indol-3-yl)but-3-yn-2-one (1l)

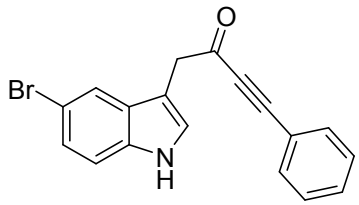
Orange solid; R_f=0.53 (Heptane/Ethyl acetate 1:1); Yield 30%; mp 109-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.35 (d, *J* = 8.1 Hz, 1H), 7.22 – 7.17 (m, 1H), 7.15 – 7.10 (m, 2H), 3.96 (s, 2H), 2.50 – 2.40 (m, 1H), 1.75 – 1.65 (m, 2H), 1.63 – 1.52 (m, 2H), 1.50 – 1.42 (m, 1H), 1.41 – 1.31 (m, 2H), 1.30 –

1.20 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 186.06, 136.12, 127.45, 123.53, 122.21, 119.69, 118.94, 111.17, 107.93, 99.33, 80.85, 42.09, 31.36, 29.04, 25.59, 24.46; HRMS (ESI) m/z: calculated for $C_{18}H_{19}NO^+$ $[M + H]^+$: 266.1539, found: 266.1539.



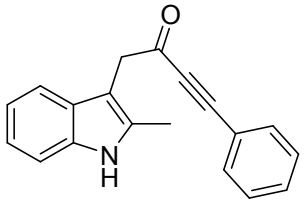
1-(5-methoxy-1H-indol-3-yl)-4-phenylbut-3-yn-2-one (1m)

Brown solid; R_f=0.37 (Heptane/Ethyl acetate 1:1); Yield 23%; mp 63-65 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.43 – 7.35 (m, 3H), 7.33 – 7.28 (m, 2H), 7.26 – 7.25 (m, 1H), 7.18 (d, *J* = 2.4 Hz, 1H), 7.08 (d, *J* = 2.4 Hz, 1H), 6.88 (dd, *J* = 8.8, 2.4 Hz, 1H), 4.05 (s, 2H), 3.83 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 185.61, 154.35, 133.06, 131.30, 130.66, 128.52, 127.89, 124.45, 119.94, 112.70, 112.07, 107.35, 100.61, 92.04, 88.01, 55.88, 42.13; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO_2^+$ $[M + H]^+$: 290.1175, found: 290.1179.



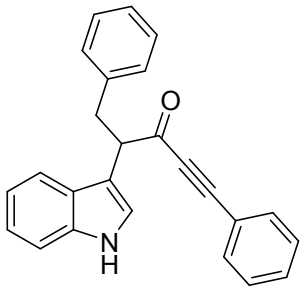
1-(5-bromo-1H-indol-3-yl)-4-phenylbut-3-yn-2-one (1n)

Beige solid; R_f=0.37 (Heptane/Ethyl acetate 1:1); Yield 44%; mp 118-120 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (s, 1H), 7.79 (d, *J* = 1.7 Hz, 1H), 7.43 – 7.38 (m, 3H), 7.34 – 7.24 (m, 3H), 7.20 (d, *J* = 8.6 Hz, 1H), 7.14 (d, *J* = 2.4 Hz, 1H), 4.03 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 185.17, 134.80, 133.14, 130.82, 129.18, 128.58, 125.18, 124.99, 121.57, 119.70, 113.15, 112.84, 107.24, 92.47, 87.91, 41.85; HRMS (ESI) m/z: calculated for $C_{18}H_{19}BrNO^+$ $[M + H]^+$: 338.0175, found: 338.0177; 340.0158.



1-(2-methyl-1H-indol-3-yl)-4-phenylbut-3-yn-2-one (1o)

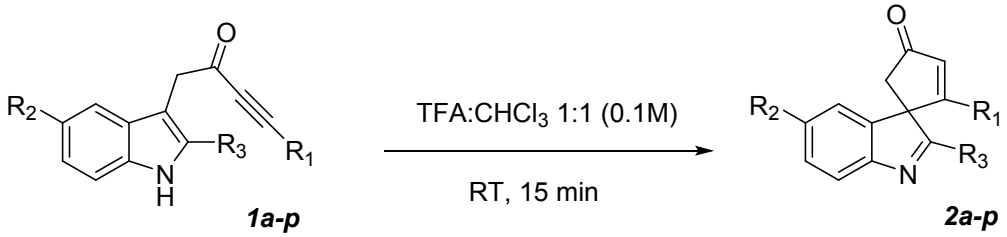
Light-orange solid; R_f=0.42 (Heptane/Ethyl acetate 1:1); Yield 89%; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.59 – 7.54 (m, 1H), 7.37 – 7.31 (m, 1H), 7.28 – 7.20 (m, 2H), 7.14 – 7.08 (m, 1H), 3.96 (s, 1H), 2.35 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.56, 135.36, 133.71, 133.13, 130.71, 128.71, 128.56, 121.36, 119.94, 119.71, 118.14, 110.61, 103.31, 91.90, 88.18, 41.28, 11.81; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1220. Spectroscopic data matched those reported in the literature[3]



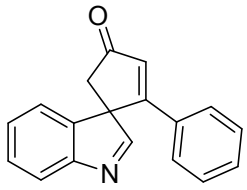
4-(1H-indol-3-yl)-1,5-diphenylpent-1-yn-3-one (1p)

Yellow solid; mp 121-123 °C; ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.16 (s, 1H), 7.75 – 7.69 (m, 1H), 7.41 – 7.33 (m, 4H), 7.32 – 7.25 (m, 2H), 7.25 – 7.12 (m, 8H), 4.52 – 4.40 (m, 1H), 3.66 (dd, *J* = 14.0, 8.1 Hz, 1H), 3.26 (dd, *J* = 14.0, 6.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 186.98, 139.42, 136.29, 132.97, 130.51, 129.01, 128.45, 128.33, 126.74, 126.27, 123.01, 122.39, 120.03, 119.92, 119.24, 112.33, 111.33, 92.06, 87.80, 53.93, 37.04; HRMS (ESI) m/z: calculated for $C_{25}H_{19}NO^+$ $[M + H]^+$: 349.1467, found: 349.1466.

General procedure B for synthesis of spiro-indolenines (2a-p)

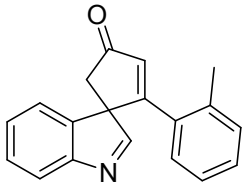


Corresponding ynone **1a-p** (0.3 mmol) was dissolved in $CHCl_3$ (1.5 ml) to afford a concentration of 0.2M. The solution was degassed and flushed with nitrogen followed by addition of trifluoracetic acid (1.5 ml) to afford concentration of 0.1 M. The solution was stirred at room temperature for 15 min (2.5h for **2o** and 1h for 2p). Then the reaction was quenched with sat. sol. of $NaHCO_3$ (50 ml) and extracted with ethyl acetate (50 ml). Organic layer was washed with brine twice until pH=5.5, dried over Na_2SO_4 and concentrated *in vacuo*. Purification when needed was performed by column chromatography (20-30% of ethyl acetate in heptane) affording spiro-indolenines **2a-p**.



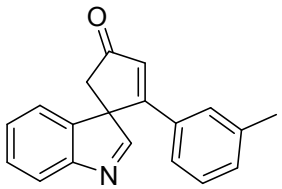
2-phenylspiro[cyclopentane-1,3'-indol]-2-en-4-one (2a)

Brown solid; R_f=0.25 (Heptane/Ethyl acetate 1:1); Yield 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.47 – 7.42 (m, 1H), 7.32 – 7.24 (m, 3H), 7.18 (t, *J* = 7.7 Hz, 2H), 6.97 (d, *J* = 7.5 Hz, 2H), 6.84 (s, 1H), 3.05 (d, *J* = 18.7 Hz, 1H), 2.68 (d, *J* = 18.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.35, 174.08, 172.00, 154.90, 140.86, 132.44, 131.36, 130.81, 129.11, 128.93, 127.75, 126.80, 122.16, 121.55, 65.98, 42.40; HRMS (ESI) m/z: calculated for $C_{18}H_{13}NO^+$ $[M + H]^+$: 260.1069, found: 260.1071. Spectroscopic data matched those reported in the literature[1]



2-(o-tolyl)spiro[cyclopentane-1,3'-indol]-2-en-4-one (2b)

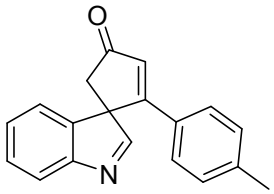
Brown solid; R_f=0.25 (Heptane/Ethyl acetate 1:1); Yield 99%; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (s, 1H), 7.57 (d, *J* = 8.5 Hz, 1H), 7.40 – 7.28 (m, 3H), 7.12 – 7.08 (m, 2H), 6.90 – 6.82 (m, 1H), 6.49 (d, *J* = 7.8 Hz, 1H), 6.47 (s, 1H), 3.09 (d, *J* = 18.7 Hz, 1H), 2.77 (d, *J* = 18.7 Hz, 1H), 2.31 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.07, 174.04, 172.38, 155.38, 139.22, 135.16, 134.65, 132.45, 130.85, 129.40, 129.10, 127.24, 125.49, 125.38, 121.78, 121.73, 68.47, 41.13, 20.87; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1224.



2-(m-tolyl)spiro[cyclopentane-1,3'-indol]-2-en-4-one (2c)

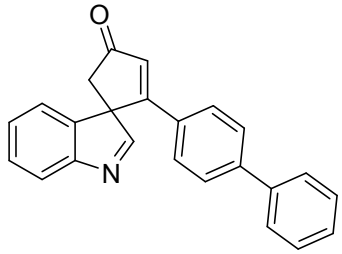
Brown solid; R_f=0.25 (Heptane/Ethyl acetate 1:1); Yield 99%; mp 110-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.76 (d, *J* = 7.8 Hz, 1H), 7.46 – 7.41 (m, 1H), 7.30 – 7.22 (m, 1H), 7.13 – 7.02 (m, 2H), 6.84 – 6.80 (m, 2H), 6.70 (d, *J* = 7.8 Hz, 1H), 3.03 (d, *J* = 18.7 Hz, 1H), 2.66 (d, *J* = 18.7 Hz, 1H), 2.18 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.42, 174.18, 172.24, 154.92, 140.95, 138.58, 132.41, 132.17, 130.66, 129.05, 128.80, 127.70,

127.53, 123.80, 122.03, 121.55, 66.00, 42.32, 21.25; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ [M + H]$^+$: 274.1226, found: 274.1226.



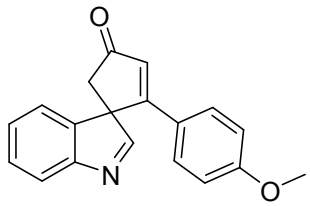
2-(p-tolyl)spiro[cyclopentane-1,3'-indol]-2-en-4-one (2d)

Brown solid; R_f=0.25 (Heptane/Ethyl acetate 1:1); Yield 99%; mp 127-129 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.76 (d, *J* = 7.8 Hz, 1H), 7.46 – 7.41 (m, 1H), 7.29 – 7.21 (m, 2H), 6.98 (d, *J* = 8.3 Hz, 2H), 6.87 (d, *J* = 8.3 Hz, 2H), 6.82 (s, 1H), 3.02 (d, *J* = 18.6 Hz, 1H), 2.65 (d, *J* = 18.6 Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.37, 174.36, 171.83, 154.83, 142.13, 141.12, 129.85, 129.66, 129.60, 129.01, 127.70, 126.84, 122.10, 121.53, 65.92, 42.36, 21.35; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ [M + H]$^+$: 274.1226, found: 274.1237.



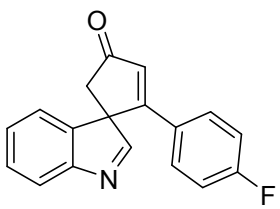
2-([1,1'-biphenyl]-4-yl)spiro[cyclopentane-1,3'-indol]-2-en-4-one (2e)

Brown solid; R_f=0.23 (Heptane/Ethyl acetate 1:1); Yield 95%; mp 95-97 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (s, 1H), 7.79 (d, *J* = 7.8 Hz, 2H), 7.50 – 7.42 (m, 4H), 7.42 – 7.35 (m, 4H), 7.32 – 7.27 (m, 2H), 7.07 – 7.02 (m, 2H), 6.89 (s, 1H), 3.06 (d, *J* = 18.7 Hz, 1H), 2.69 (d, *J* = 18.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.32, 174.26, 171.33, 154.81, 144.11, 140.99, 139.47, 131.16, 130.42, 129.16, 128.90, 128.14, 127.84, 127.49, 127.40, 126.94, 122.20, 121.60, 65.92, 42.42; HRMS (ESI) m/z: calculated for $C_{24}H_{17}NO^+$ [M + H]$^+$: 336.1382, found: 336.1379.



2-(4-methoxyphenyl)spiro[cyclopentane-1,3'-indol]-2-en-4-one (2f)

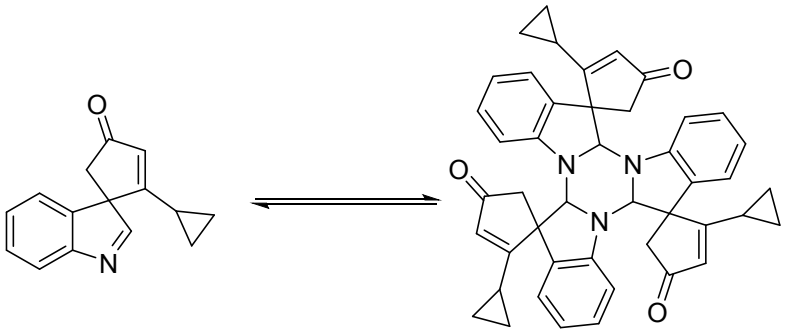
Brown solid; R_f=0.21 (Heptane/Ethyl acetate 1:1); Yield 41%; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.48 – 7.41 (m, 1H), 7.32 – 7.21 (m, 1H), 6.98 – 6.91 (m, 1H), 6.77 (s, 1H), 6.72 – 6.66 (m, 1H), 3.73 (s, *J* = 7.2 Hz, 1H), 3.02 (d, *J* = 18.6 Hz, 1H), 2.64 (d, *J* = 18.6 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.27, 174.65, 171.15, 162.15, 154.73, 141.35, 129.02, 128.85, 128.53, 127.76, 124.81, 122.15, 121.56, 114.35, 65.86, 55.31, 42.39 HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO_2^+$ [M + H]$^+$: 290.1175, found: 290.1175. Spectroscopic data matched those reported in the literature[1]



2-(4-fluorophenyl)spiro[cyclopentane-1,3'-indol]-2-en-4-one (2g)

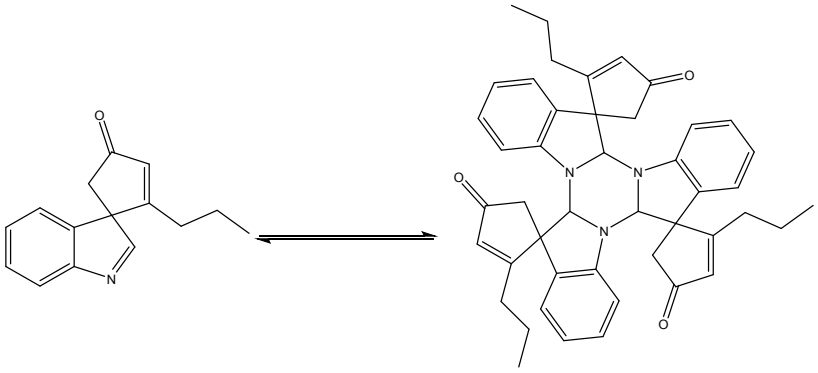
Brown solid; R_f=0.23 (Heptane/Ethyl acetate 1:1); Yield 99%; mp 77-79 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.48 – 7.42 (m, 1H), 7.33 – 7.21 (m, 3H), 6.99 – 6.93 (m, 2H), 6.89 – 6.84 (m, 2H), 6.79 (s, 1H), 3.04 (d, *J* = 18.7 Hz, 1H), 2.68 (d, *J* = 18.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.12, 173.99, 170.62, 164.35 (d, *J* = 254.0 Hz), 154.77, 140.63, 130.59 (d, *J* = 1.4 Hz), 129.27, 129.11, 129.02, 128.61 (d, *J* = 3.4 Hz), 127.89, 122.23, 121.57, 116.17 (d, *J* = 21.9 Hz), 65.93, 42.33; HRMS (ESI) m/z: calculated for $C_{18}H_{12}FNO^+$ [M + H]$^+$: 278.0975, found: 278.0977.

2-cyclopropylspiro[cyclopentane-1,3'-indol]-2-en-4-one (2i)

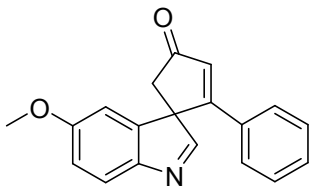


Brown solid; mixture of monomer and trimer (see ref. 1 and 3, supporting information); R_f=0.20 (Heptane/Ethyl acetate 1:1); Yield 69%; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (s), 7.72 (d, *J* = 7.7 Hz), 7.48 – 7.39 (m), 7.32 (ddd, *J* = 15.6, 11.1, 4.0 Hz), 7.19 – 7.12 (m), 7.05 – 6.97 (m), 6.86 (d, *J* = 6.7 Hz), 6.71 (d, *J* = 7.2 Hz), 6.45 (d, *J* = 7.8 Hz), 5.87 (s), 5.65 (s), 5.13 (d, *J* = 9.5 Hz), 4.39 (d, *J* = 9.5 Hz), 3.12 (d, *J* = 18.7 Hz), 2.93 (d, *J* = 18.7 Hz), 2.66 (d, *J* = 18.7 Hz), 2.34 (d, *J* = 18.8 Hz), 1.66 – 1.56 (m), 1.35 – 1.19 (m), 1.08 – 0.94 (m), 0.93 – 0.81 (m), 0.77 – 0.68 (m), 0.63 – 0.54 (m). ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.93, 205.45, 189.43, 184.63, 184.50, 173.45, 155.85, 149.34, 139.67, 136.71, 133.37, 129.20, 127.56, 124.32, 122.04, 121.72, 119.46, 109.51, 68.15, 60.00, 40.48, 13.52, 13.07, 10.65 HRMS (ESI) m/z: calculated for $C_{15}H_{13}NO^+$ $[M + H]^+$: 224.1069, found: 224.1065.

2-propylspiro[cyclopentane-1,3'-indol]-2-en-4-one (2j)



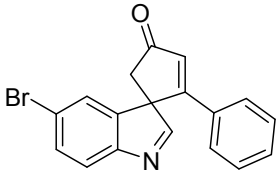
Brown solid; mixture of monomer and trimer (see ref. 1 and 3, supporting information); R_f=0.28 (Heptane/Ethyl acetate 1:1); Yield 55%; ^{1}H NMR (400 MHz, CDCl3) δ 8.33 – 8.28 (m), 8.14 (s), 7.97 (s), 7.81 (d, *J* = 2.9 Hz), 7.71 (d, *J* = 7.7 Hz), 7.46 – 7.41 (m), 7.35 – 7.28 (m), 7.25 – 7.19 (m), 6.98 (d, *J* = 4.3 Hz), 6.87 (d, *J* = 7.8 Hz), 6.30 (t, *J* = 1.5 Hz), 5.04 (d, *J* = 2.3 Hz), 4.82 (d, *J* = 3.0 Hz), 4.23 (d, *J* = 2.9 Hz), 4.16 (d, *J* = 2.2 Hz), 3.20 (s), 2.93 (d, *J* = 18.8 Hz), 2.66 (d, *J* = 18.8 Hz), 2.46 (t, *J* = 7.1 Hz), 2.39 (t, *J* = 7.1 Hz), 2.17 (s), 1.71 – 1.58 (m), 1.49 – 1.38 (m), 1.34 – 1.24 (m), 1.04 (t, *J* = 7.4 Hz), 0.99 (t, *J* = 7.4 Hz), 0.88 (t, *J* = 6.9 Hz), 0.80 (t, *J* = 7.3 Hz). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.73, 186.70, 185.69, 185.22, 180.16, 176.57, 173.29, 155.58, 142.34, 139.39, 136.73, 135.59, 131.08, 129.06, 129.00, 128.76, 127.39, 124.53, 124.45, 124.33, 124.26, 123.59, 122.97, 122.83, 122.53, 121.91, 121.71, 121.61, 119.58, 111.62, 110.04, 109.92, 102.56, 67.77, 49.53, 40.38, 31.88, 30.70, 29.70, 29.02, 22.69, 21.24, 21.08, 20.97, 20.60, 14.12, 13.59, 13.56, 13.53; HRMS (ESI) m/z: calculated for $C_{15}H_{15}NO^+$ $[M + H]^+$: 226.1226, found: 226.1234.



5'-methoxy-2-phenylspiro[cyclopentane-1,3'-indol]-2-en-4-one (2m)

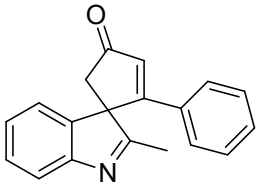
R_f=0.16 (Heptane/Ethyl acetate 1:1); Yield 52%; mp 134-136 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.35 – 7.29 (m,

1H), 7.24 – 7.17 (m, 2H), 7.03 – 6.99 (m, 1H), 6.94 (dd, *J* = 8.5, 2.5 Hz, 1H), 6.84 (s, 1H), 6.76 (d, *J* = 2.5 Hz, 1H), 3.77 (s, 3H), 3.05 (d, *J* = 18.7 Hz, 1H), 2.66 (d, *J* = 18.7 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.50, 172.08, 171.84, 159.79, 148.42, 142.67, 132.38, 131.42, 130.62, 128.97, 126.91, 122.64, 114.08, 107.60, 65.97, 42.85; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO_2^+$ [M + H]$^+$: 290.1175, found: 290.1177.



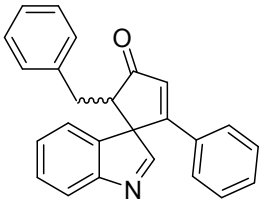
5'-bromo-2-phenylspiro[cyclopentane-1,3'-indol]-2-en-4-one (2n)

Brown solid; R_f=0.22 (Heptane/Ethyl acetate 1:1); Yield 98%; mp 134-136 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.63 (d, *J* = 8.2 Hz, 1H), 7.57 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.38 (d, *J* = 1.7 Hz, 1H), 7.36 – 7.32 (m, 1H), 7.25 – 7.20 (m, 2H), 7.00 – 6.97 (m, 2H), 6.85 (s, 1H), 3.04 (d, *J* = 18.7 Hz, 1H), 2.66 (d, *J* = 18.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.57, 174.45, 171.07, 153.77, 143.04, 132.35, 132.15, 131.62, 130.98, 129.10, 126.77, 125.03, 123.45, 121.68, 66.13, 42.20; HRMS (ESI) m/z: calculated for $C_{18}H_{12}BrNO^+$ [M + H]$^+$: 338.0175, found: 338.0178; 340.0160.



2'-methyl-2-phenylspiro[cyclopentane-1,3'-indol]-2-en-4-one (2o)

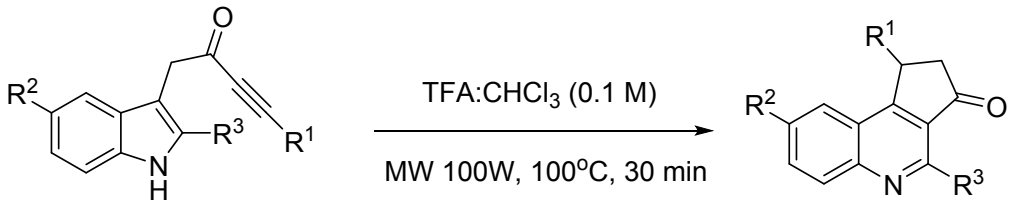
Brown solid; R_f=0.19 (Heptane/Ethyl acetate 1:1); Yield 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.8 Hz, 1H), 7.43 – 7.38 (m, 1H), 7.31 (t, *J* = 7.4 Hz, 1H), 7.23 – 7.17 (m, 4H), 7.00 – 6.96 (m, 2H), 6.88 (s, 1H), 2.83 (d, *J* = 18.8 Hz, 1H), 2.73 (d, *J* = 18.8 Hz, 1H), 2.21 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.69, 182.94, 172.80, 154.64, 141.76, 132.18, 131.40, 130.95, 129.05, 129.02, 126.90, 126.62, 121.68, 120.86, 66.67, 45.15, 15.77; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ [M + H]$^+$: 274.1226, found: 274.1222. Spectroscopic data matched those reported in the literature[1]



5-benzyl-2-phenylspiro[cyclopentane-1,3'-indol]-2-en-4-one (2p)

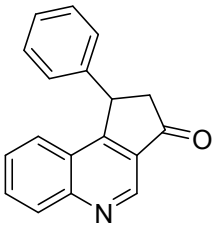
Light-brown solid; Isolated as an inseparatable mixture of diastereomers (approx. 5:4 ratio A/B according ^{1}H NMR); R_f=0.39 (Heptane/Ethyl acetate 1:1); Yield 94%; mp 78-81 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (s), 7.97 (s), 7.60 – 7.52 (m), 7.47 (t, *J* = 7.6 Hz), 7.35 – 7.27 (m), 7.26 – 7.20 (m), 7.19 – 7.11 (m), 7.07 – 6.98 (m), 6.91 (dd, *J* = 7.9, 1.9 Hz), 6.88 – 6.82 (m), 6.47 (d, *J* = 7.3 Hz), 3.56 (dd, *J* = 10.3, 4.4 Hz), 3.48 (dd, *J* = 8.8, 5.7 Hz), 3.29 – 3.17 (m), 2.53 (dd, *J* = 14.9, 8.8 Hz), 2.10 (dd, *J* = 14.5, 10.3 Hz). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.08, 205.06, 173.39, 173.01, 171.40, 170.72, 156.38, 155.38, 140.06, 137.69, 137.66, 137.60, 132.71, 132.51, 131.16, 131.09, 130.39, 129.35, 129.18, 128.89, 128.84, 128.75, 128.40, 128.22, 127.98, 127.96, 127.38, 126.73, 126.27, 126.17, 123.27, 122.47, 121.99, 121.81, 57.95, 52.45, 32.55, 32.00; HRMS (ESI) m/z: calculated for $C_{25}H_{19}NO^+$ [M + H]$^+$: 350.1539, found: 350.1542.

General procedure C for the synthesis of quinolines (3a-o)



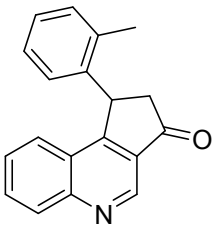
1a-o ***3a-o***

Corresponding ynone **1a-o** (0.3 mmol) was dissolved in $CHCl_3$ (1.5 ml) to afford concentration of 0.2M. The solution was degassed and flushed with nitrogen followed by addition of trifluoracetic acid (1.5 ml) at rt to afford concentration of 0.1 M. Then the reaction mixture was irradiated under MW at 100°C with maximum power of 100W for 30 min. Then the reaction was quenched with sat. sol. of $NaHCO_3$ (50 ml) and extracted with ethyl acetate (50 ml). Organic layer was washed with brine twice until pH=5.5, dried under Na_2SO_4 and concentrated *in vacuo*. Purification when needed was performed by column chromatography (20-30% of ethyl acetate in heptane) affording quinolines **3a-o**.



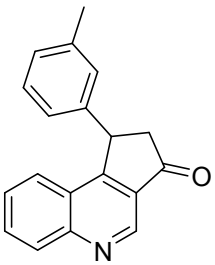
1-phenyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3a)

Brown solid; R_f=0.28 (Heptane/Ethyl acetate 1:1); Yield 99%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.28 (s, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 7.79 (t, *J* = 7.7 Hz, 1H), 7.70 (d, *J* = 8.2 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.32 – 7.22 (m, 3H), 7.10 (d, *J* = 7.1 Hz, 2H), 5.02 (dd, *J* = 8.0, 2.6 Hz, 1H), 3.38 (dd, *J* = 19.2, 8.0 Hz, 1H), 2.75 (dd, *J* = 19.2, 2.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.12, 165.36, 150.88, 145.52, 142.54, 132.24, 130.52, 130.17, 129.30, 127.67, 127.40, 127.32, 125.58, 125.31, 47.63, 43.84; HRMS (ESI) m/z: calculated for $C_{18}H_{13}NO^+$ $[M + H]^+$: 260.1069, found: 260.1076. Spectroscopic data matched those reported in the literature[4]



1-(o-tolyl)-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3b)

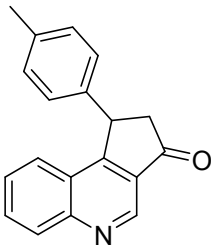
Brown solid; R_f=0.29 (Heptane/Ethyl acetate 1:1); Yield 35%; mp 97-99 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.30 (s, 1H), 8.24 (d, *J* = 8.5 Hz, 1H), 7.86 – 7.76 (m, 1H), 7.56 – 7.41 (m, 2H), 7.35 – 7.27 (m, 1H), 7.21 – 7.11 (m, 1H), 7.01 – 6.89 (m, 1H), 6.46 – 6.35 (m, 1H), 5.27 – 5.17 (m, 1H), 3.42 (dd, *J* = 19.1, 8.0 Hz, 1H), 2.70 – 2.57 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.17, 166.06, 150.78, 145.61, 141.01, 135.01, 132.33, 130.76, 130.56, 127.73, 127.27, 126.99, 126.35, 125.66, 125.40, 46.44, 39.69, 20.02; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1229.



1-(m-tolyl)-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3c)

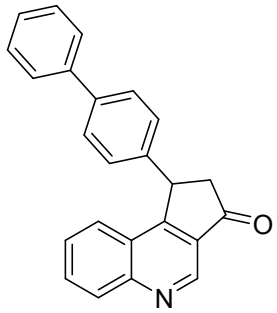
Brown solid; R_f=0.29 (Heptane/Ethyl acetate 1:1); Yield 98%; mp 93-95 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.29 (s, 1H), 8.22 (d, *J* = 8.4 Hz, 1H), 7.82 – 7.77 (m, 1H), 7.72 (dd, *J* = 8.3, 0.7 Hz, 1H), 7.49 – 7.38 (m, 2H), 7.21 – 7.14 (m, 1H), 7.08 – 7.02 (m, 1H), 6.92 – 6.87 (m, 2H), 4.96 (dd, *J* = 8.0, 2.6 Hz, 1H), 3.36 (dd, *J* = 19.3, 8.0 Hz, 1H), 2.74 (dd, *J* = 19.3, 2.8 Hz, 1H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, CDCl3) δ

204.34, 165.61, 150.77, 145.48, 142.46, 139.08, 132.27, 130.39, 130.15, 129.12, 128.18, 127.90, 127.68, 125.64, 125.39, 124.41, 47.68, 43.83, 21.39; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1232.



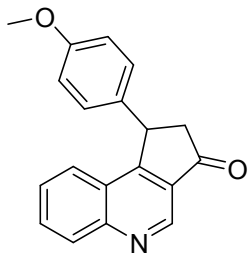
1-(p-tolyl)-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3d)

Brown solid; R_f=0.29 (Heptane/Ethyl acetate 1:1); Yield 97%; mp 83-85 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.28 (s, 1H), 8.22 (d, *J* = 8.5 Hz, 1H), 7.82 – 7.76 (m, 1H), 7.72 (d, *J* = 8.3 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.9 Hz, 2H), 6.97 (d, *J* = 8.0 Hz, 2H), 4.97 (dd, *J* = 8.0, 2.7 Hz, 1H), 3.36 (dd, *J* = 19.3, 8.0 Hz, 1H), 2.72 (dd, *J* = 19.3, 2.8 Hz, 1H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.30, 165.81, 150.59, 145.38, 139.46, 137.11, 132.32, 130.27, 130.14, 129.96, 127.72, 127.18, 125.67, 125.39, 47.73, 43.52, 21.03; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1219.



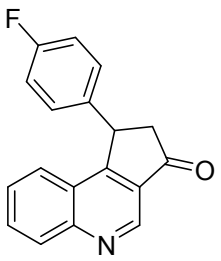
1-([1,1'-biphenyl]-4-yl)-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3e)

Brown solid; R_f=0.23 (Heptane/Ethyl acetate 1:1); Yield 90%; mp 116-118 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.32 (s, 1H), 8.24 (d, *J* = 8.4 Hz, 1H), 7.82 (t, *J* = 7.7 Hz, 1H), 7.76 (d, *J* = 8.2 Hz, 1H), 7.56 – 7.47 (m, 5H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.36 – 7.30 (m, 1H), 7.17 (d, *J* = 8.1 Hz, 2H), 5.08 (dd, *J* = 8.0, 2.6 Hz, 1H), 3.43 (dd, *J* = 19.3, 8.0 Hz, 1H), 2.80 (dd, *J* = 19.2, 2.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.13, 165.29, 150.96, 145.62, 141.54, 140.32, 140.25, 132.31, 130.62, 130.22, 128.82, 127.97, 127.76, 127.49, 126.96, 125.64, 125.36, 47.64, 43.52; HRMS (ESI) m/z: calculated for $C_{24}H_{17}NO^+$ $[M + H]^+$: 336.1383, found: 336.1385.



2-(4-methoxyphenyl)spiro[cyclopentane-1,3'-indol]-2-en-4-one (3f)

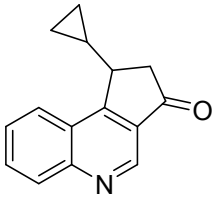
Brown solid; R_f=0.24 (Heptane/Ethyl acetate 1:1); Yield 67%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.27 (s, 1H), 8.20 (d, *J* = 8.4 Hz, 1H), 7.80 (t, *J* = 7.6 Hz, 1H), 7.72 (d, *J* = 8.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.01 (d, *J* = 8.5 Hz, 2H), 6.82 (d, *J* = 8.5 Hz, 2H), 4.98 (dd, *J* = 7.9, 2.0 Hz, 1H), 3.76 (s, 3H), 3.37 (dd, *J* = 19.2, 7.9 Hz, 1H), 2.72 (dd, *J* = 19.2, 1.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.41, 165.66, 158.75, 150.89, 145.51, 134.59, 132.22, 130.50, 130.08, 128.36, 127.64, 125.68, 125.37, 114.65, 55.25, 47.80, 43.12; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO_2^+$ $[M + H]^+$: 290.1175, found: 290.1183. Spectroscopic data matched those reported in the literature[4]



1-(4-fluorophenyl)-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3g)

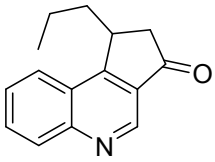
Brown solid; R_f=0.22 (Heptane/Ethyl acetate 1:1); Yield 99%; mp 116-118 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.26 (s, 1H), 8.22 (d, *J* = 8.5 Hz, 1H), 7.85 – 7.76 (m, 1H), 7.65 (d, *J* = 8.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.10 – 7.02 (m, 2H), 7.03 – 6.94 (m, 2H), 5.01 (dd, *J* = 8.1, 2.8 Hz, 1H), 3.38 (dd, *J* = 19.3, 8.1 Hz, 1H), 2.70

(dd, J = 19.2, 2.6 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.84, 165.12, 161.90 (d, J = 246.6 Hz), 150.78, 145.45, 138.28 (d, J = 3.4 Hz), 132.42, 130.49, 130.46, 130.13, 128.87 (d, J = 8.1 Hz, 127.82, 125.44, 125.17, 116.27 (d, J = 21.6 Hz), 47.59, 43.08, 29.70; HRMS (ESI) m/z: calculated for $C_{18}H_{12}FNO^+$ [M + H]$^+$: 278.0975, found: 278.0969.



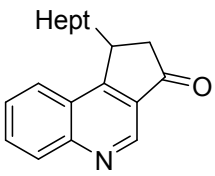
1-cyclopropyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3i)

Beige solid; R_f=0.38 (Heptane/Ethyl acetate 1:1); Yield 52%; mp 123-125 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.19 (s, 1H), 8.36 (dd, J = 8.3, 0.9 Hz, 1H), 8.24 (d, J = 8.4 Hz, 1H), 7.93 – 7.86 (m, 1H), 7.74 – 7.67 (m, 1H), 3.54 – 3.47 (m, 1H), 3.01 (dd, J = 19.0, 7.5 Hz, 1H), 2.70 (dd, J = 19.0, 2.1 Hz, 1H), 1.14 – 1.05 (m, 1H), 0.88 – 0.80 (m, 1H), 0.74 – 0.67 (m, 1H), 0.67 – 0.59 (m, 1H), 0.42 – 0.34 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.49, 166.88, 150.76, 145.73, 132.23, 130.65, 129.39, 127.52, 125.98, 125.84, 43.76, 41.63, 17.21, 7.60, 4.15; HRMS (ESI) m/z: calculated for $C_{15}H_{13}NO^+$ [M + H]$^+$: 224.1069, found: 224.1075.



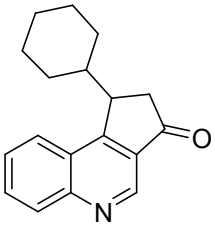
1-propyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3j)

Brown solid; R_f=0.33 (Heptane/Ethyl acetate 1:1); Yield 45%; mp 82-85 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.18 (s, 1H), 8.24 (d, J = 8.5 Hz, 1H), 8.12 (d, J = 8.2 Hz, 1H), 7.89 (t, J = 7.7 Hz, 1H), 7.70 (t, J = 7.6 Hz, 1H), 3.98 – 3.86 (m, 1H), 2.98 (dd, J = 18.9, 7.3 Hz, 1H), 2.62 (d, J = 19.0 Hz, 1H), 2.19 – 2.09 (m, 1H), 1.59 – 1.34 (m, 3H), 0.98 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.75, 167.78, 150.54, 145.74, 132.23, 130.83, 129.43, 127.57, 125.30, 124.64, 43.38, 38.36, 37.43, 20.76, 14.02; HRMS (ESI) m/z: calculated for $C_{15}H_{15}NO^+$ [M + H]$^+$: 226.1226, found: 226.1219.



1-heptyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3k)

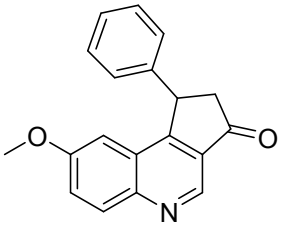
Brown sticky oil; R_f=0.20 (Heptane/Ethyl acetate 1:1); Yield 37%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.18 (s, 1H), 8.25 (d, J = 8.4 Hz, 1H), 8.12 (dd, J = 8.2, 0.7 Hz, 1H), 7.92 – 7.86 (m, 1H), 7.73 – 7.67 (m, 1H), 3.97 – 3.87 (m, 1H), 2.98 (dd, J = 19.0, 7.3 Hz, 1H), 2.62 (dd, J = 19.0, 1.5 Hz, 1H), 2.21 – 2.09 (m, 1H), 1.62 – 1.51 (m, 2H), 1.35 – 1.19 (m, 14H), 0.87 (t, J = 6.9 Hz, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.79, 167.82, 150.51, 145.73, 132.24, 130.80, 129.45, 127.59, 125.31, 124.64, 43.39, 37.64, 36.19, 31.74, 29.51, 29.10, 27.50, 22.59, 14.06; HRMS (ESI) m/z: calculated for $C_{19}H_{23}NO^+$ [M + H]$^+$: 282.1852, found: 282.1863.



1-cyclohexyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3l)

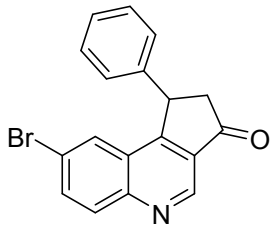
Brown solid; R_f=0.23 (Heptane/Ethyl acetate 1:1); Yield 47%; mp 103-105 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.17 (s, 1H), 8.25 (d, J = 8.4 Hz, 1H), 8.17 (dd, J = 8.3, 0.9 Hz, 1H), 7.92 – 7.86 (m, 1H), 7.70 (m, 1H), 3.96 (m, 1H), 2.77 (d, J = 4.6 Hz, 2H), 2.28 – 2.19 (m, 1H), 1.93 – 1.82 (m, 2H), 1.66 – 1.54 (m, 2H), 1.35 – 1.30 (m, 2H), 1.08 – 0.96 (m, 2H), 0.89 – 0.75 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.93, 166.54, 150.49,

145.47, 132.24, 130.80, 130.03, 127.52, 125.49, 124.75, 43.32, 41.81, 39.57, 32.95, 29.70, 26.68, 26.12, 25.85, 25.68; HRMS (ESI) m/z: calculated for $C_{18}H_{19}NO^+$ $[M + H]^+$: 266.1539, found: 266.1540.



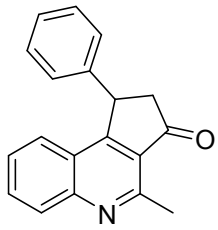
8-methoxy-1-phenyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3m)

Brown solid; R_f=0.24 (Heptane/Ethyl acetate 1:1); Yield 68%; mp 111-113 °C; ^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.15 (s, 1H), 8.10 (d, *J* = 9.2 Hz, 1H), 7.41 (dd, *J* = 9.2, 2.8 Hz, 1H), 7.38 – 7.28 (m, 1H), 7.33 – 7.23 (m, 2H), 7.19 – 7.10 (m, 2H), 6.86 (d, *J* = 2.8 Hz, 1H), 4.93 (dd, *J* = 8.0, 3.1 Hz, 1H), 3.61 (s, 3H), 3.40 (dd, *J* = 19.3, 8.0 Hz, 1H), 2.78 (dd, *J* = 19.3, 3.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.46, 163.85, 158.21, 146.81, 142.88, 142.43, 131.75, 130.23, 129.30, 127.46, 126.41, 124.45, 103.43, 55.39, 47.60, 44.03; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO_2^+$ $[M + H]^+$: 290.1175, found: 290.1179.



8-bromo-1-phenyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3n)

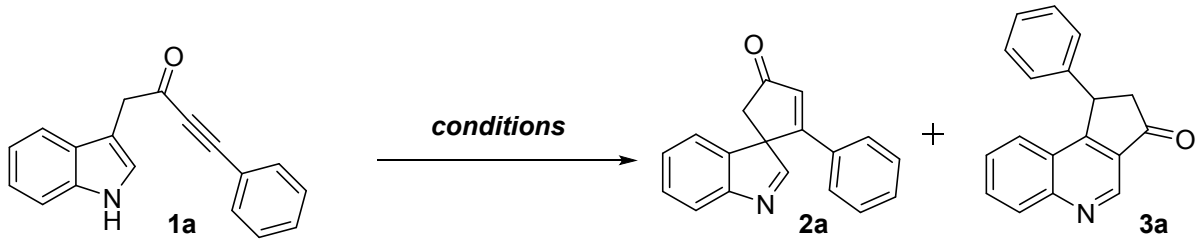
Brown solid; R_f=0.34 (Heptane/Ethyl acetate 1:1); Yield 99%; mp 153-155 °C; ^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.26 (s, 1H), 8.05 (d, *J* = 9.5 Hz, 1H), 7.86 – 7.79 (m, 2H), 7.37 – 7.23 (m, 3H), 7.09 (dd, *J* = 7.8, 1.5 Hz, 2H), 4.95 (dd, *J* = 8.0, 2.7 Hz, 1H), 3.38 (dd, *J* = 19.4, 8.0 Hz, 1H), 2.76 (dd, *J* = 19.4, 2.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.87, 164.35, 149.36, 145.83, 145.73, 141.85, 135.63, 130.65, 129.49, 127.75, 127.27, 126.61, 121.91, 47.63, 43.82; HRMS (ESI) m/z: calculated for $C_{18}H_{12}BrNO^+$ $[M + H]^+$: 338.0175, found: 338.0180; 340.0165.



4-methyl-1-phenyl-1,2-dihydro-3H-cyclopenta[c]quinolin-3-one (3o)

Beige solid; R_f=0.31 (Heptane/Ethyl acetate 1:1); Yield 99%; mp 131-133; ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.09 (d, *J* = 8.3 Hz, 1H), 7.80 – 7.68 (m, 1H), 7.65 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.43 – 7.30 (m, 1H), 7.37 – 7.19 (m, 3H), 7.14 – 7.04 (m, 2H), 4.95 (dd, *J* = 8.1, 2.8 Hz, 1H), 3.35 (dd, *J* = 19.1, 8.2 Hz, 1H), 3.04 (s, 3H), 2.72 (dd, *J* = 19.2, 2.8 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.76, 166.39, 157.11, 149.94, 142.91, 132.29, 129.25, 128.83, 127.30, 126.53, 125.36, 124.41, 47.85, 43.11, 22.70; HRMS (ESI) m/z: calculated for $C_{19}H_{15}NO^+$ $[M + H]^+$: 274.1226, found: 274.1225.

Full optimization table



№	Acid	Equiv. (conc.)	Temp.	Time	Solvent[1]	MW[2]	Conversion[3] (%)	Yield of **2a**[3] (%)	Yield of **3a**[3] (%)
1	TFA	1	RT	3h	$CHCl_3$	-	40	34	0
2	TFA	1	RT	18h	$CHCl_3$	-	98	5	3
3	TFA	2	RT	18h	$CHCl_3$	-	100	43	51
4	TFA	2	60°C	3h	$CHCl_3$	-	100	2	15
5	PTSA	1	RT	3h	$CHCl_3$	-	76	33	42
6	PTSA	1	60°C	3h	$CHCl_3$	-	100	0	62
7	TFA	2	60°C	18h	$CHCl_3$	-	100	0	52
8	TFA	2	60°C	18h	MeCN	-	100	0	28
9	TFA	2	60°C	18h	THF	-	33	0	0
10	TFA	2	100°C	30 min	Toluene	+	70	0	40
11	TFA	2	100°C	1h	Toluene	+	98	0	50
12	TFA	2	100°C	30 min	$CHCl_3$	+	84	9	37
13	TFA	2	100°C	1h	$CHCl_3$	+	97	4	57
14	TFA	2	100°C	1.5h	$CHCl_3$	+	97	4	57
15	TFA	2	60°C	18h	$CHCl_3$ (0.2M)	-	100	0	23
16	TFA	4	60°C	18h	$CHCl_3$	-	100	0	34
17	TFA	4	60°C	18h	$CHCl_3$ (0.2M)	-	100	0	34
18	TFA	2	60°C	18h	$CHCl_3$	-	100	0	49
19	TFA+H_2O	2+2	60°C	18h	$CHCl_3$	-	100	0	50
20	AcOH	2.5	60°C	18h	$CHCl_3$	-	7	0	0
21	CF_3SO_3H	1.5	RT	1h	$CHCl_3$	-	100	0	45
22	CF_3SO_3H	1.5	60°C	30 min	$CHCl_3$	-	decomposition		
23	HBF_4	1.5	RT	1h	$CHCl_3$	-	100	30	0
24	PTSA	2	60°C	20h	$CHCl_3$	-	100	0	53
25	PTSA	2	RT	3h	$CHCl_3$	-	93	30	44
26	PTSA	2	60°C	3h	$CHCl_3$	-	100	0	39
27	PTSA	2	RT	18h	$CHCl_3$	-	100	0	45
28	PTSA	1.5	RT	18h	$CHCl_3$	-	100	0	46
29	TFA	4	RT	3h	$CHCl_3$	-	100	35	15
30	TFA	4	60°C	3h	$CHCl_3$	-	100	0	75
31	TFA	4	RT	18h	$CHCl_3$	-	100	42	55
32	TFA	4	60°C	18h	$CHCl_3$	-	100	0	73
33	TFA	2	60°C	18h	$CHCl_3$	-	100	0	43
34	TFA	4	60°C	18h	$CHCl_3$	-	100	0	61
35	TFA	6	60°C	18h	$CHCl_3$	-	100	0	60
36	TFA	6	60°C	1.5h	$CHCl_3$	-	100	31	60
37	TFA	8	60°C	1.5h	$CHCl_3$	-	100	20	47
38	TFA	10	60°C	1.5h	$CHCl_3$	-	100	23	73
39	TFA	10	50°C	3h	$CHCl_3$	-	100	12	67

40	TFA+$CHCl_3$ 1:1	(0.1M)	50°C	15 min	-	-	100	70	30
41	CF_3SO_3H+$CHCl_3$ 1:1	(0.1M)	RT	15 min	-	-	87	36	0
42	**TFA+$CHCl_3$ 1:1**	**(0.1M)**	**RT**	**15 min**	**-**	**-**	**100**	**100**	**0**
43	TFA+$CHCl_3$ 1:1	(0.1M)	60°C	30 min	-	-	100	38	62
44	TFA+$CHCl_3$ 1:1	(0.1M)	60°C	45 min	-	-	100	25	75
45	TFA+$CHCl_3$ 1:1	(0.1M)	60°C	1h	-	-	100	16	84
46	**TFA+$CHCl_3$ 1:1**	**(0.1M)**	**100°C**	**30 min**	**-**	+	**100**	**0**	**100**

[1] Concentration is 0.1M unless another mentioned
[2] Microwave irradiation with max. power 100W
[3] Calculation based on NMR using CH_2Br_2 as an internal standart

References

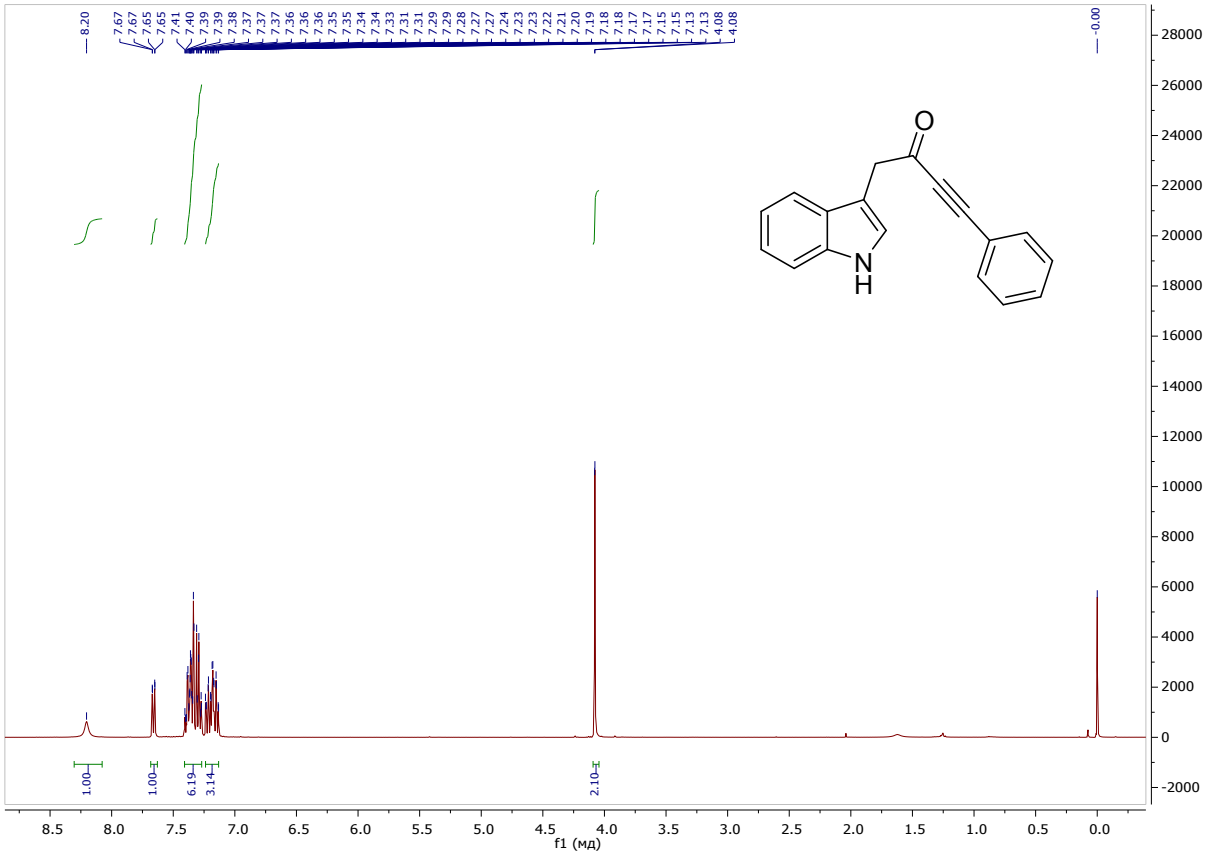
1 M. J. James, J. D. Cuthbertson, P. O'Brien, R. J. K. Taylor and W. P. Unsworth, *Angew. Chem. Int. Ed.*, 2015, **54**, 7640–7643.

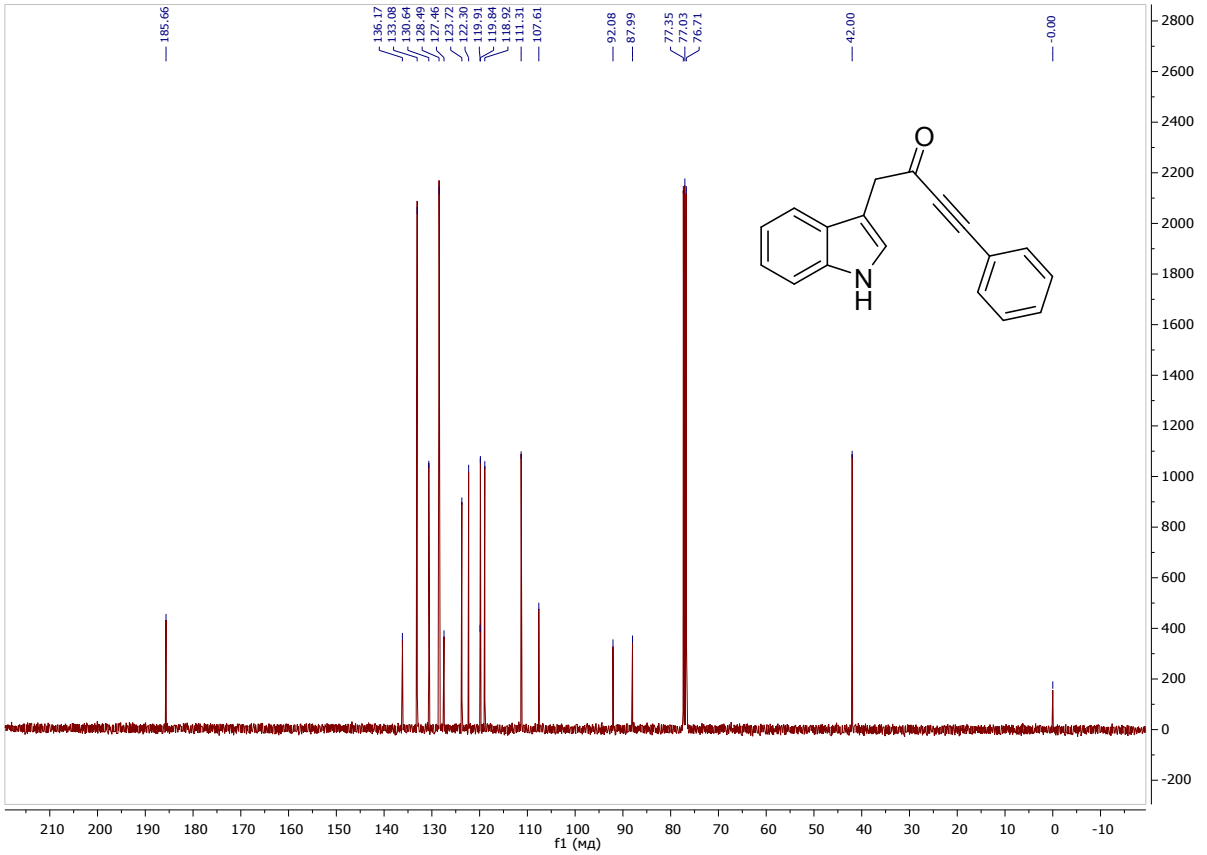
2 P. Wanga; J. Z. Zhaoa, H. F. Lia, Lianga X.M., Y. L. Zhanga and C.S. Da, *Tetrahedron Letters* 2017, **58**, 129-133.

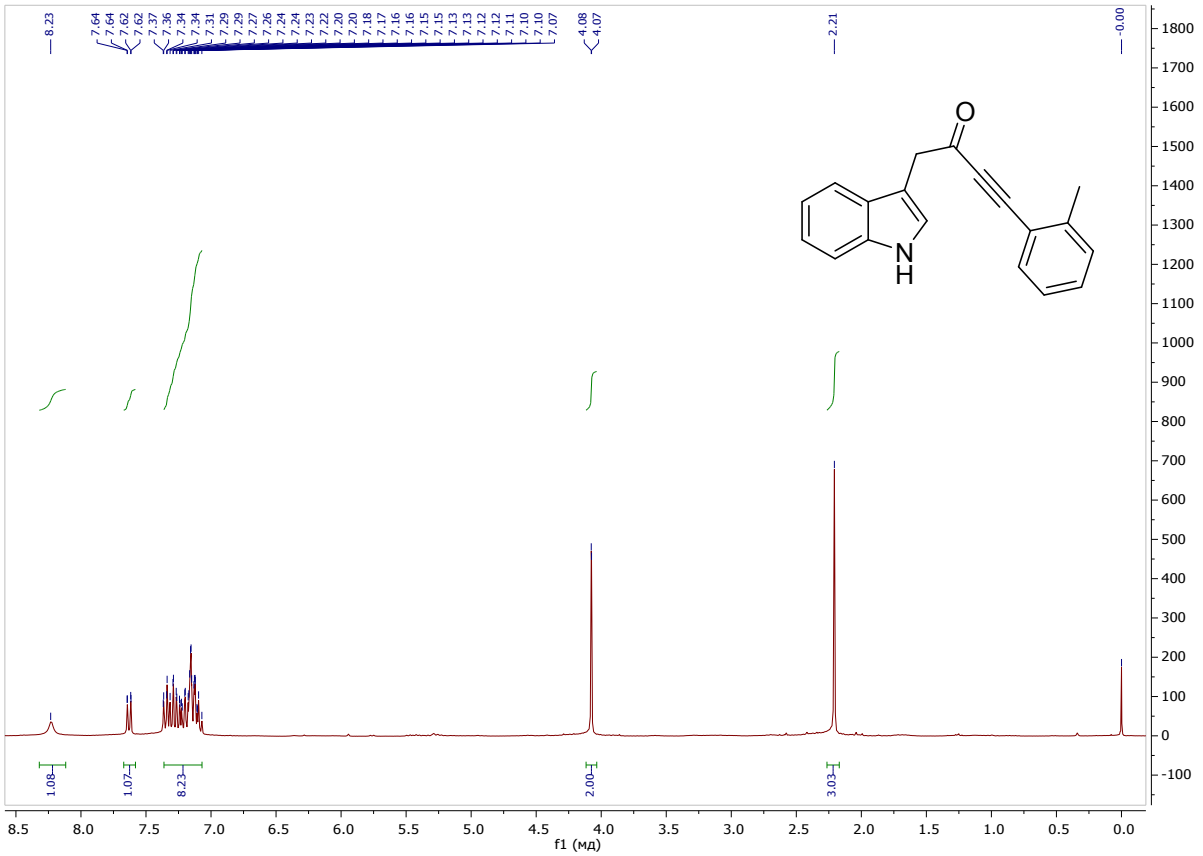
3 M. J. James, R. E. Clubley, K. Y. Palate, T. J. Procter, A. C. Wyton, P. O'Brien, R. J. K. Taylor and W. P. Unsworth, *Org. Lett.*, 2015, **17**, 4372–4375.

4 J. T. R. Liddon, M. J. James, A. K. Clarke, P. O'Brien, R. J. K. Taylor and W. P. Unsworth, *Chem. Eur. J.*, 2016, **22**, 8777–8780.

^{1}H and ^{13}C NMR spectra



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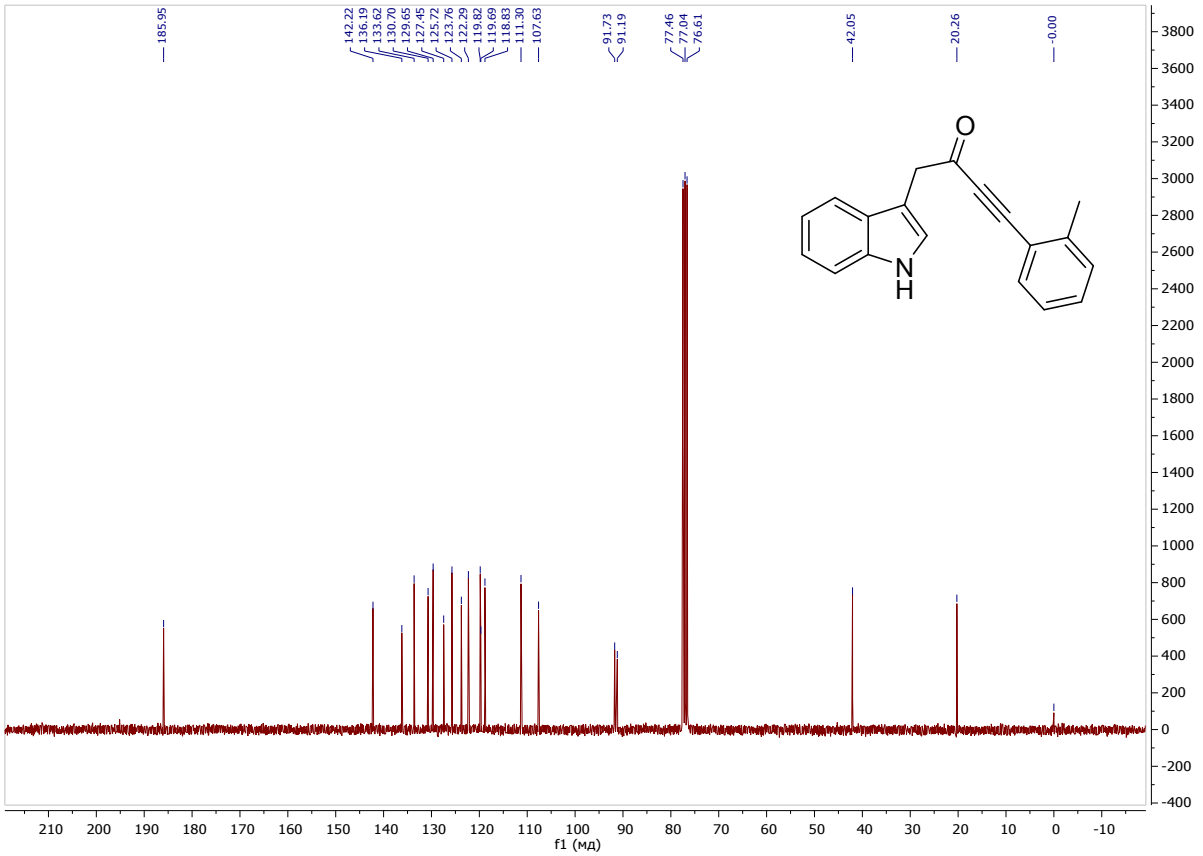
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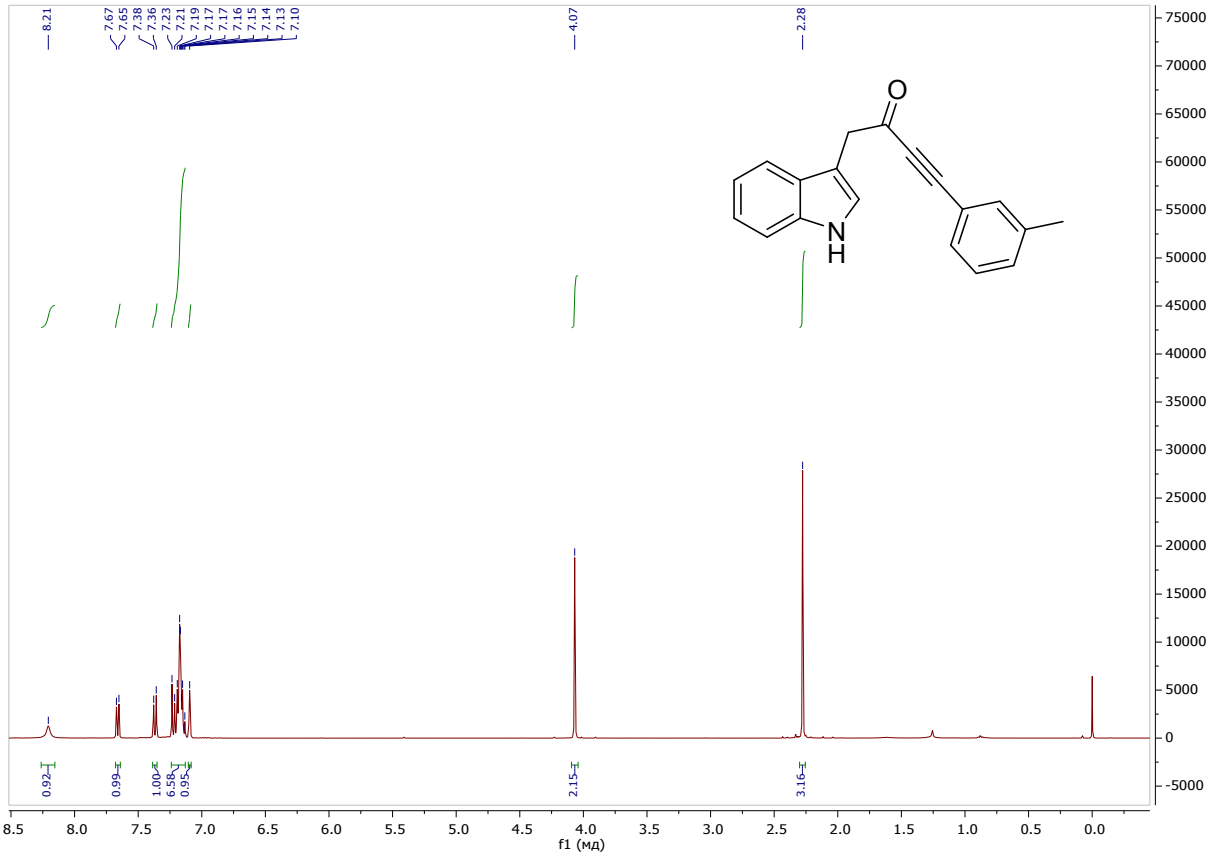
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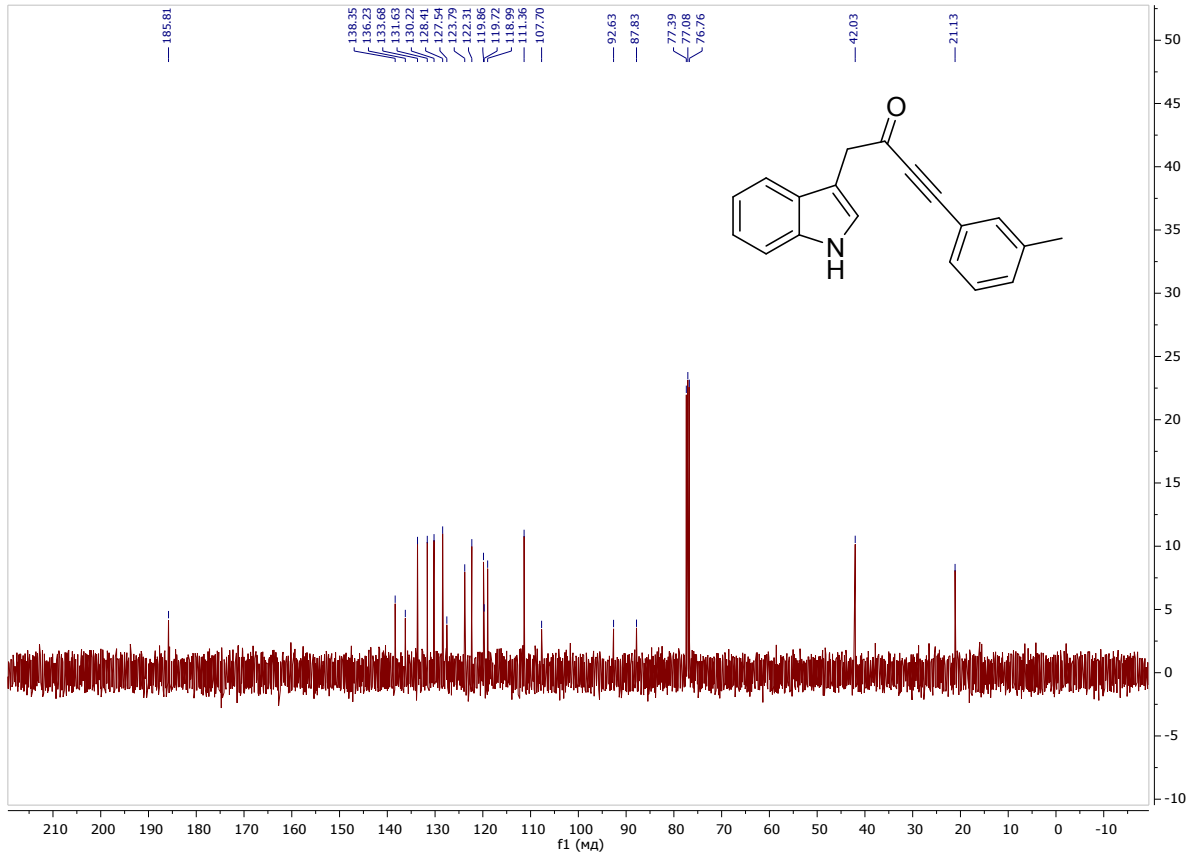
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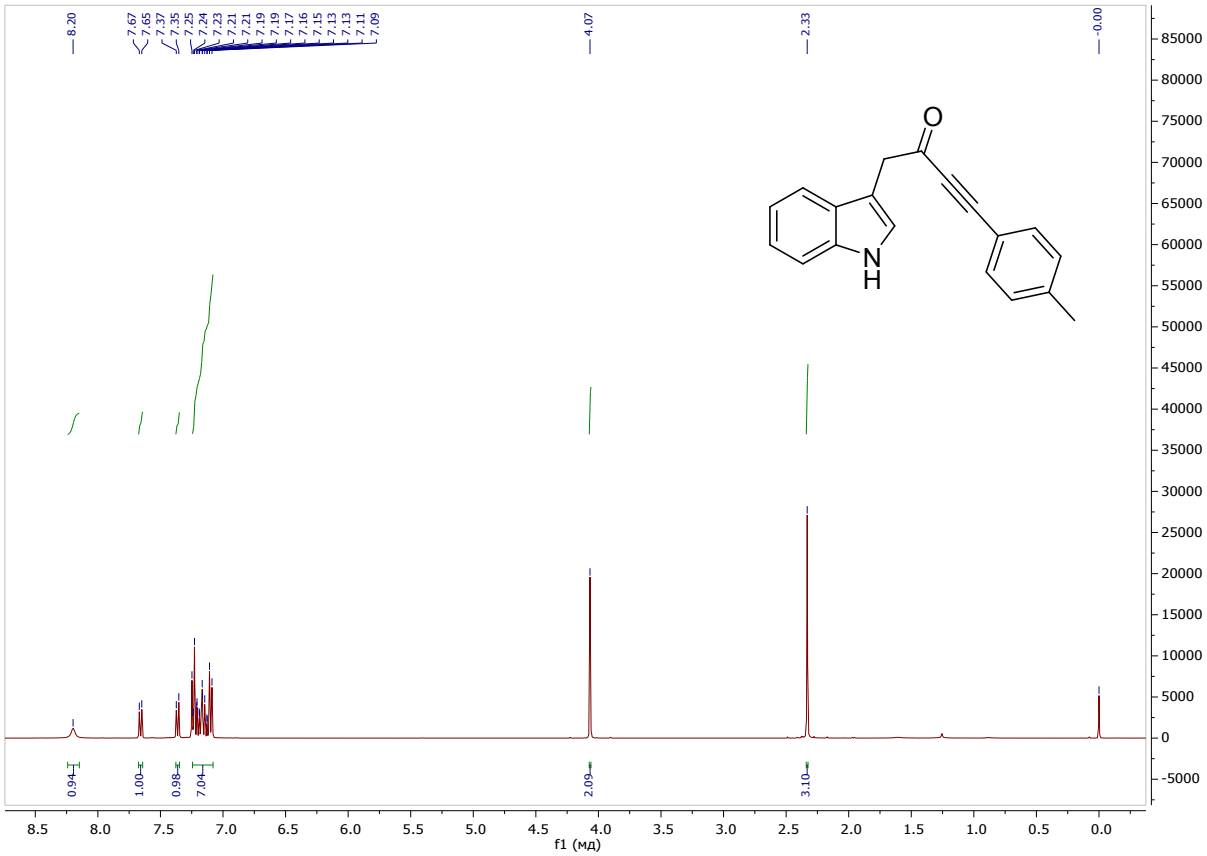
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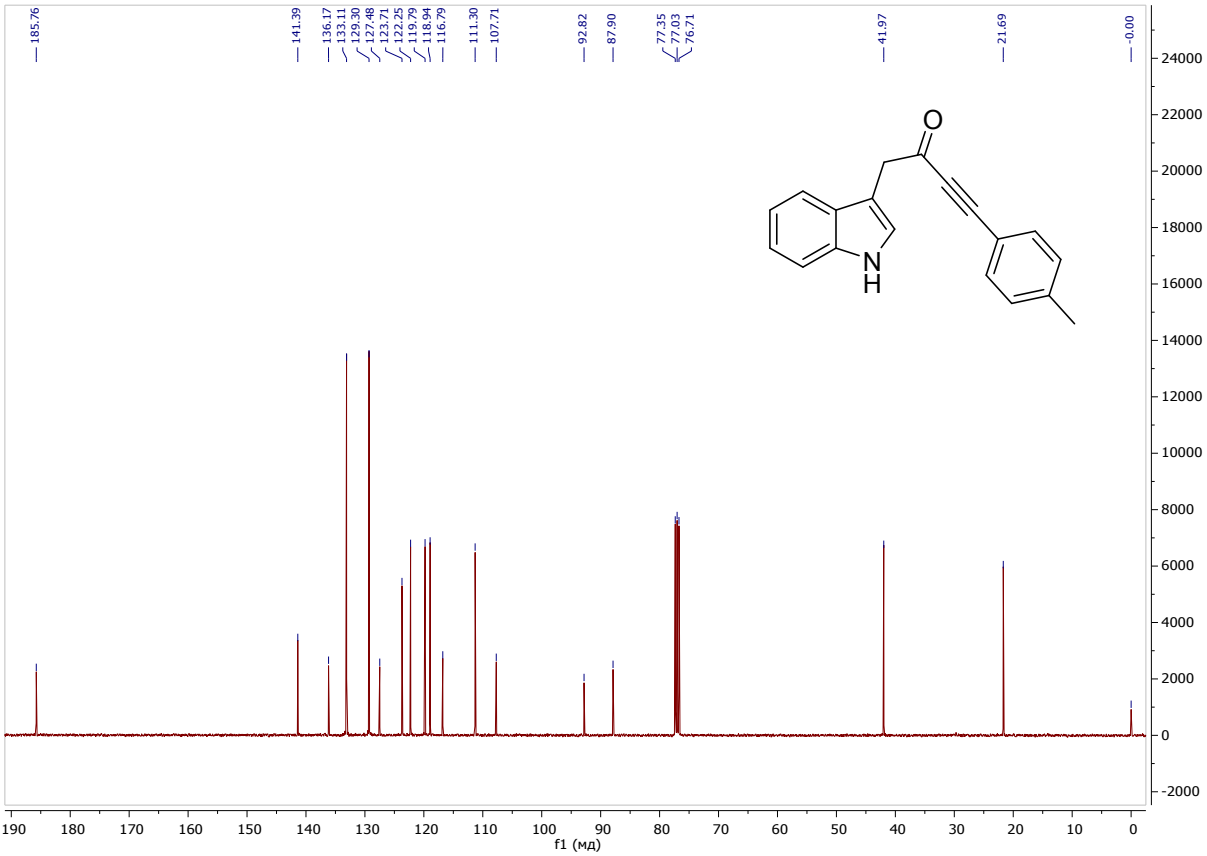
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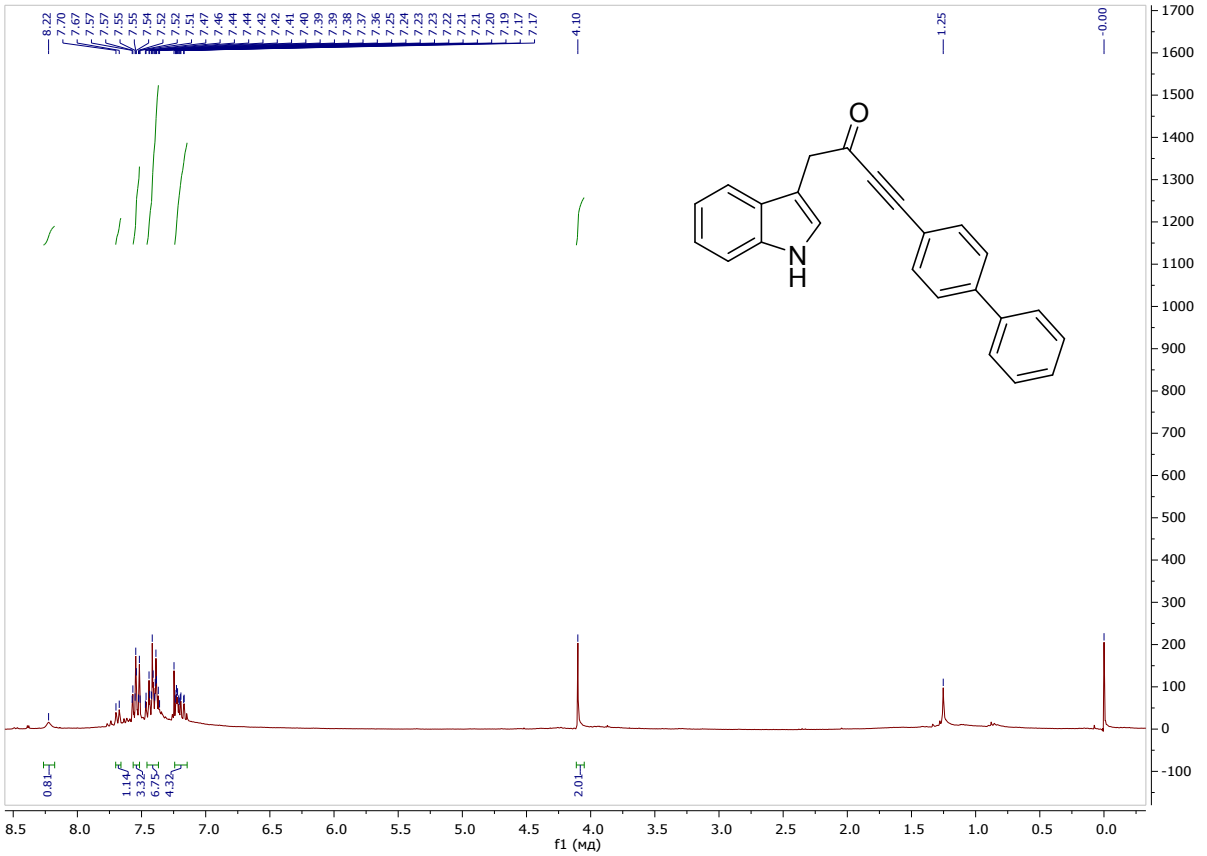
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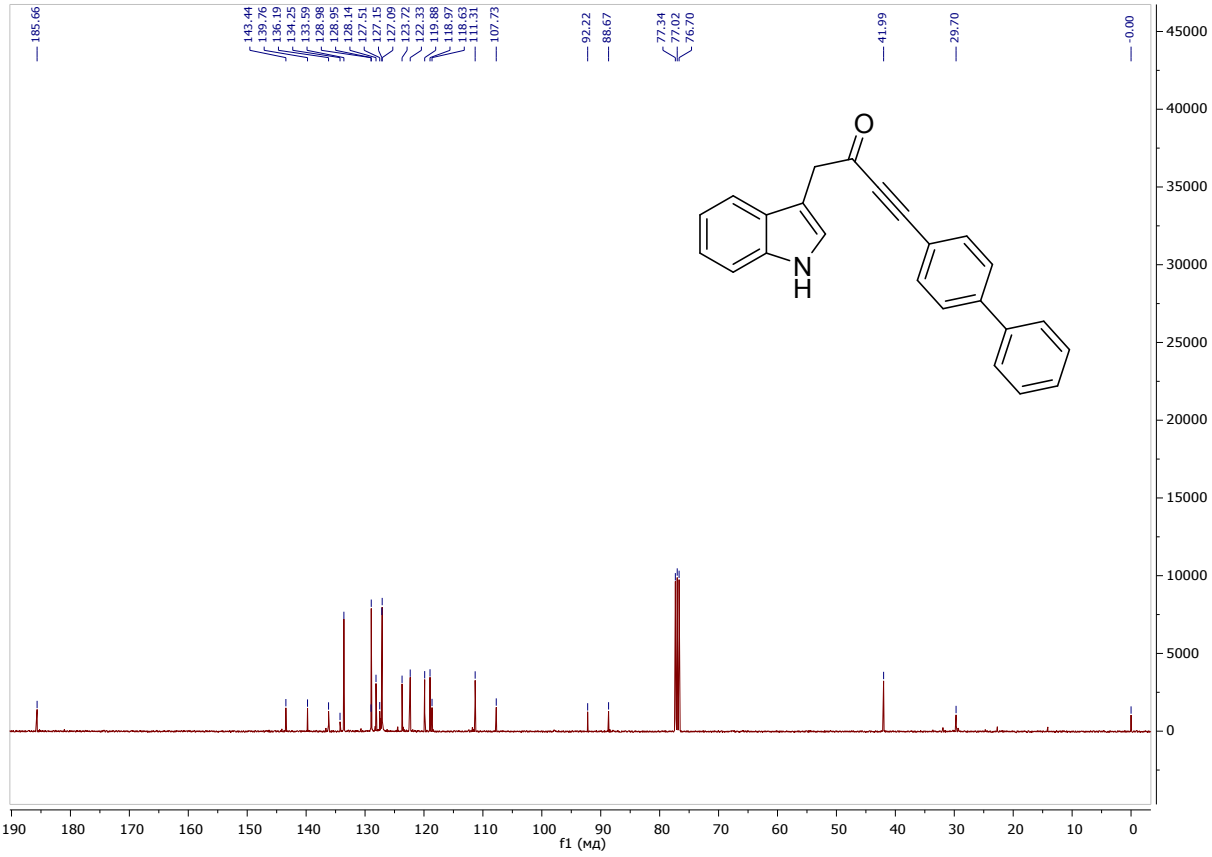
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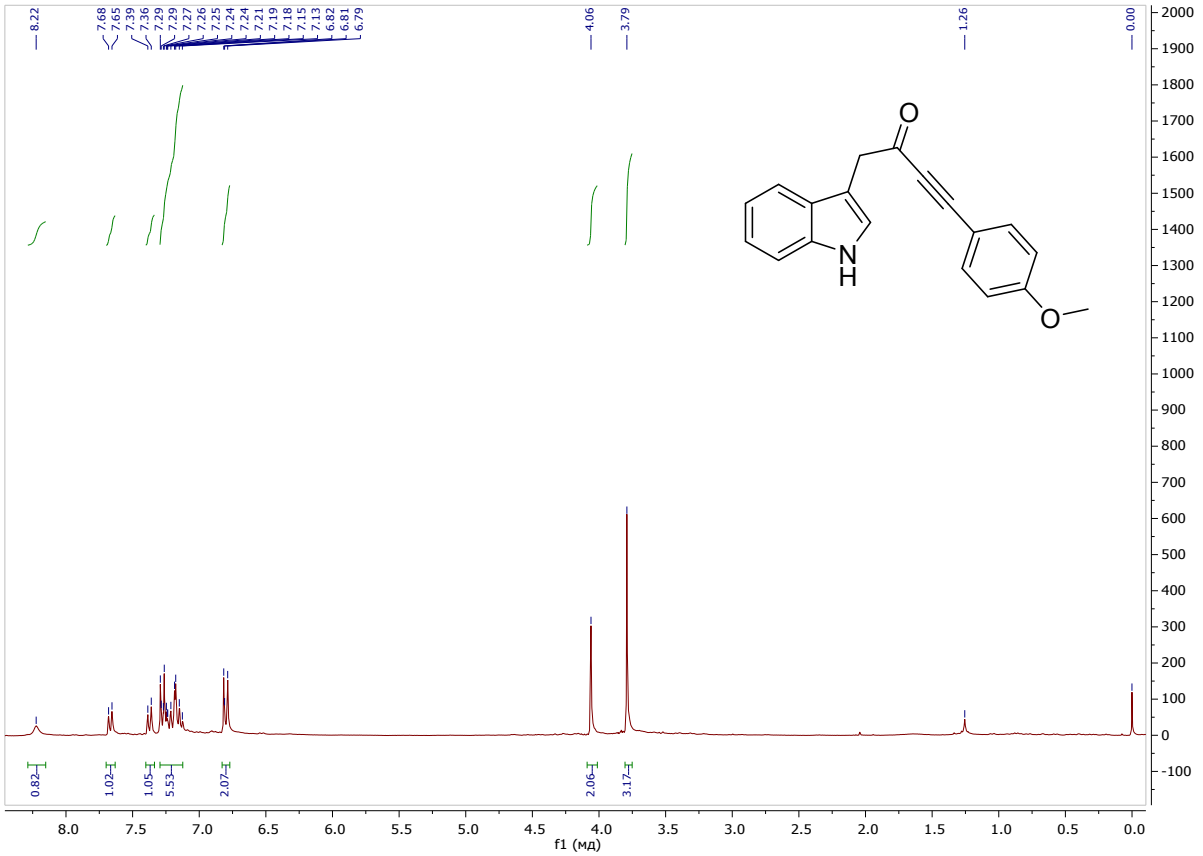
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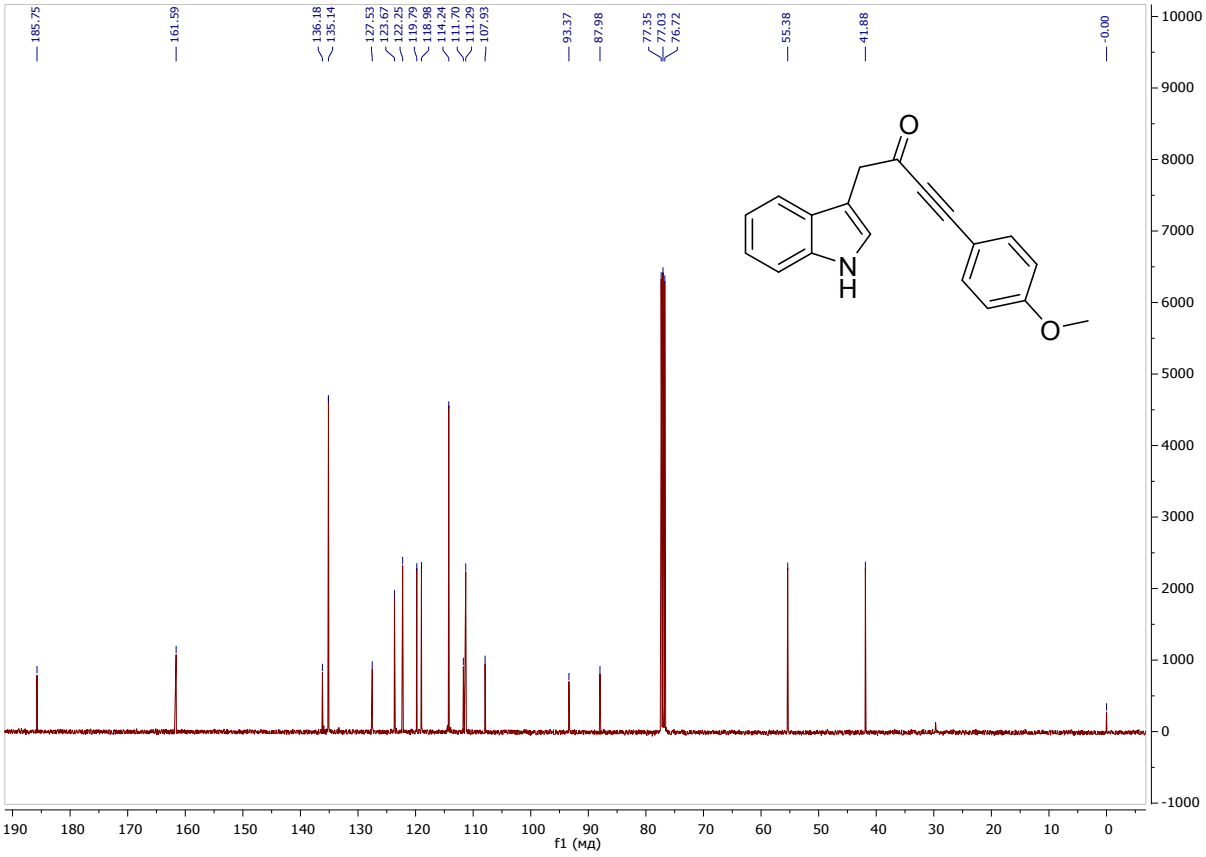
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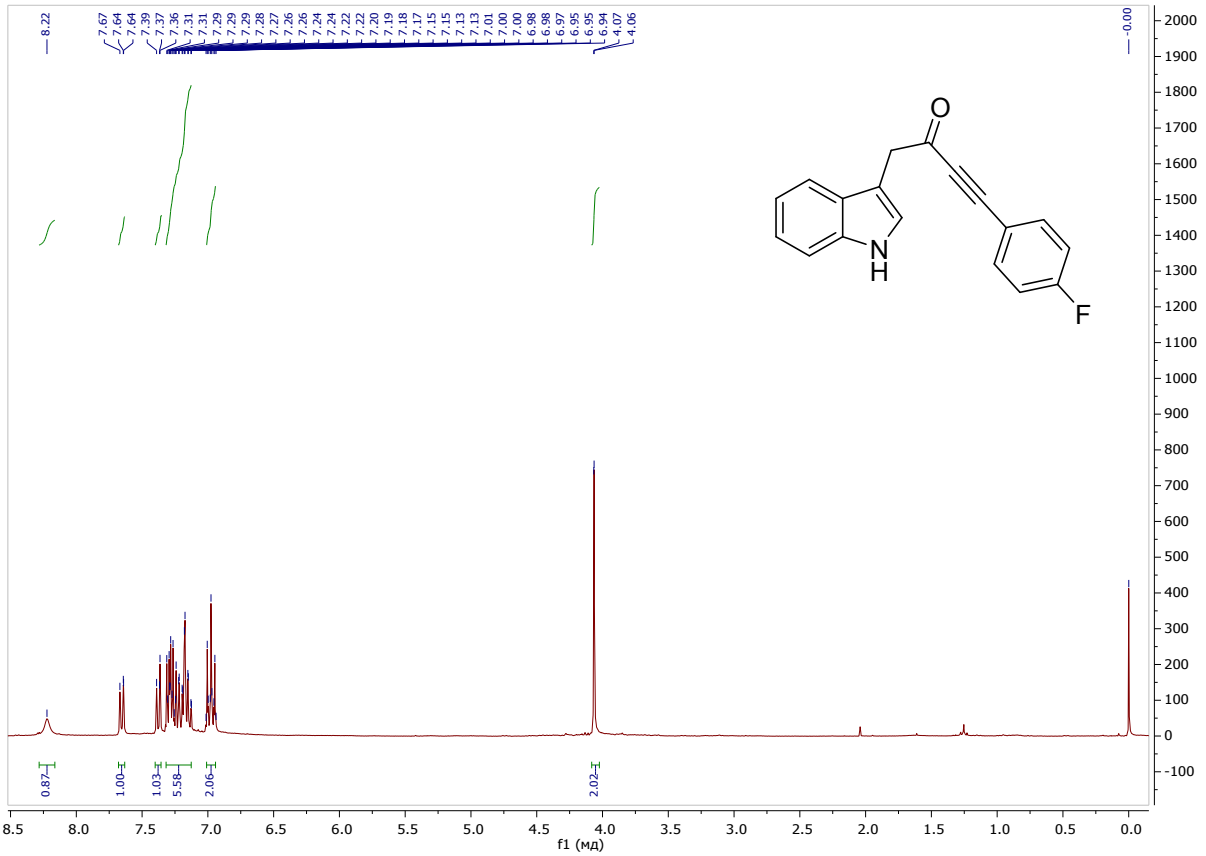
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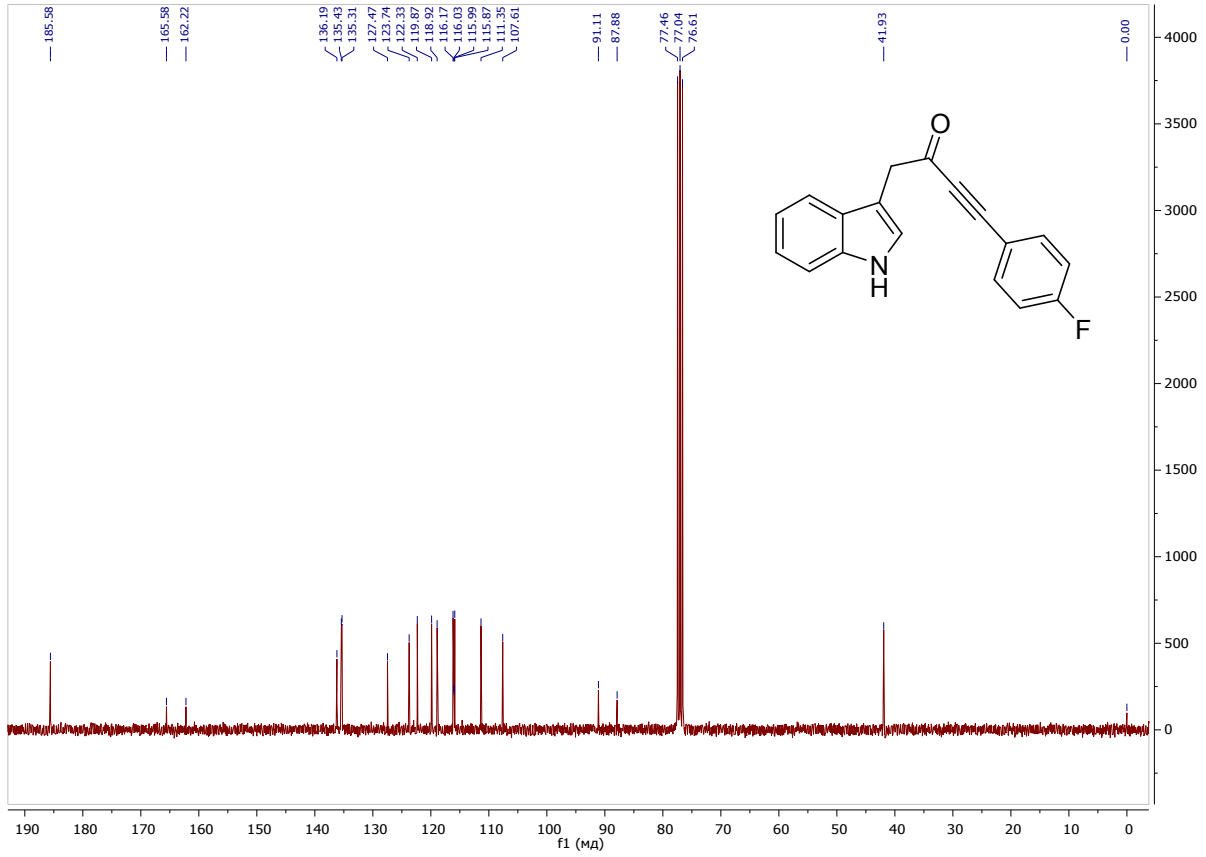
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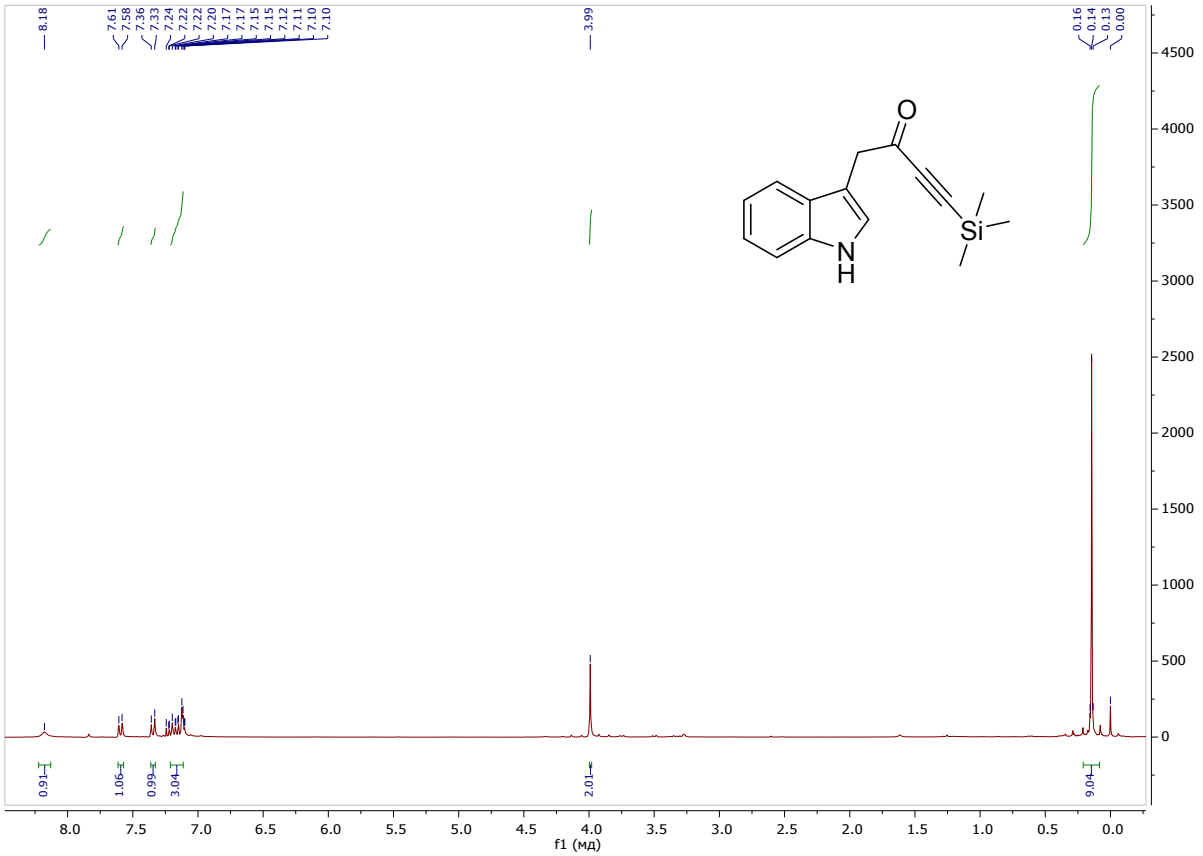
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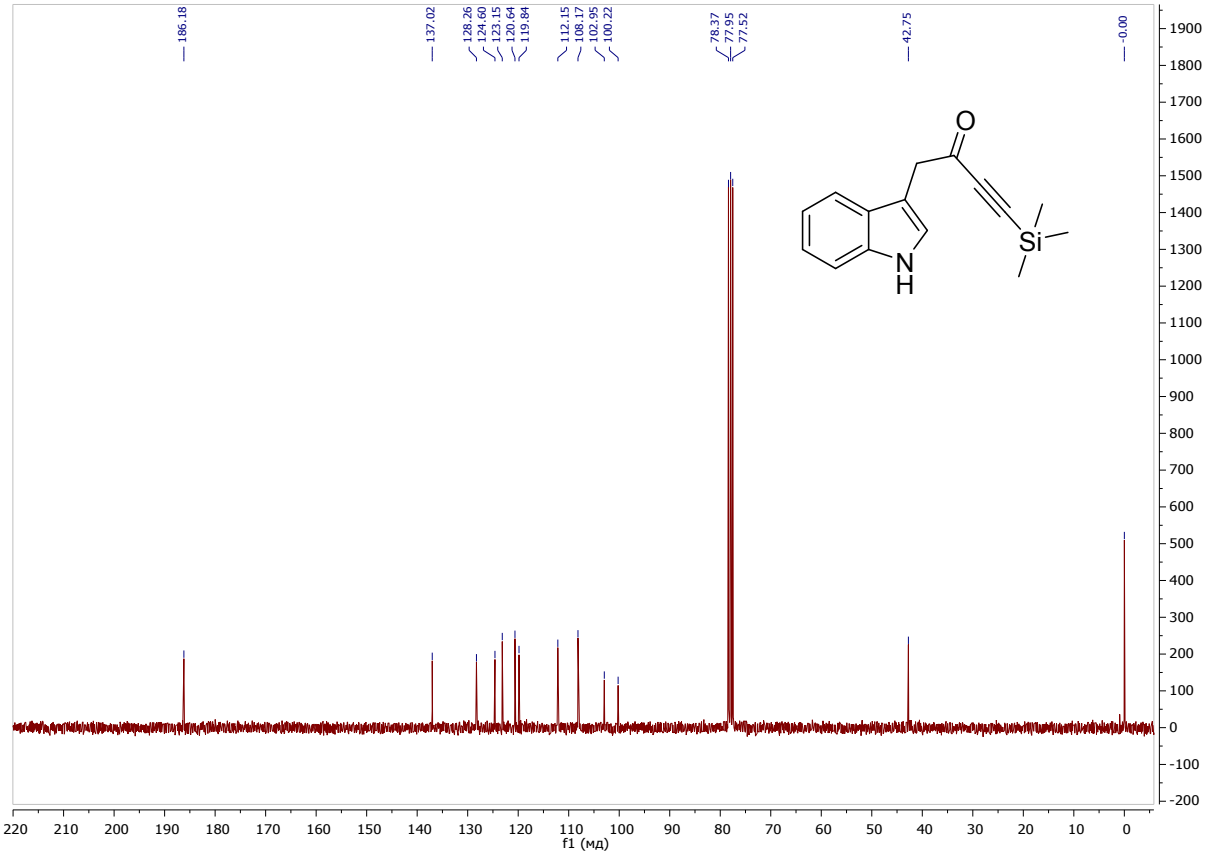
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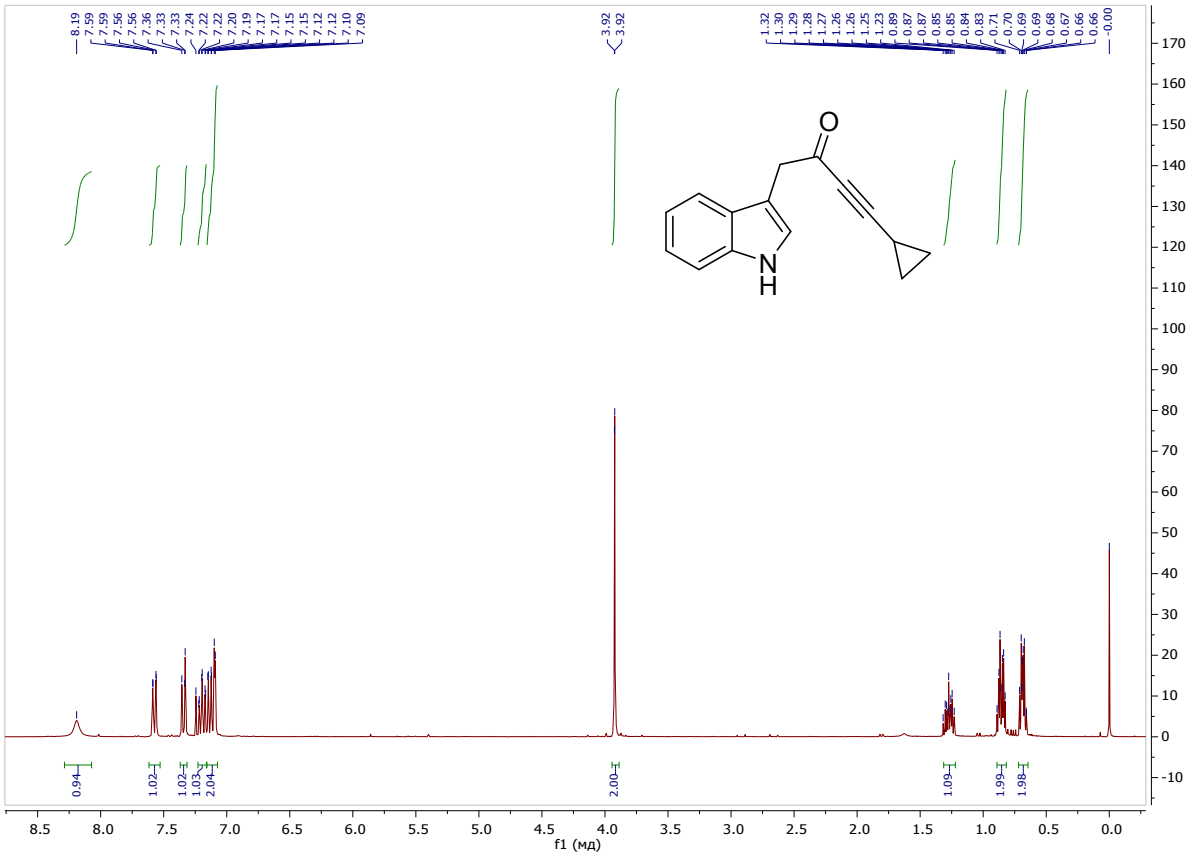
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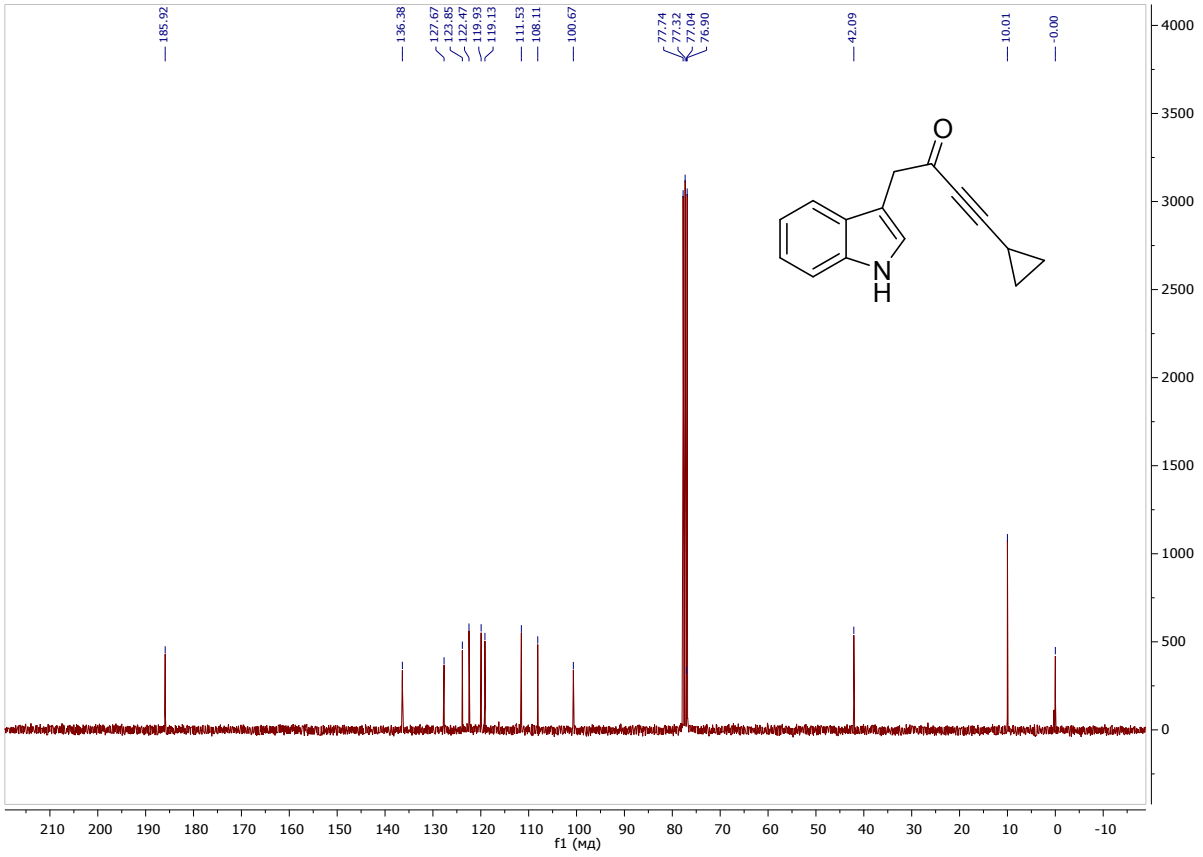
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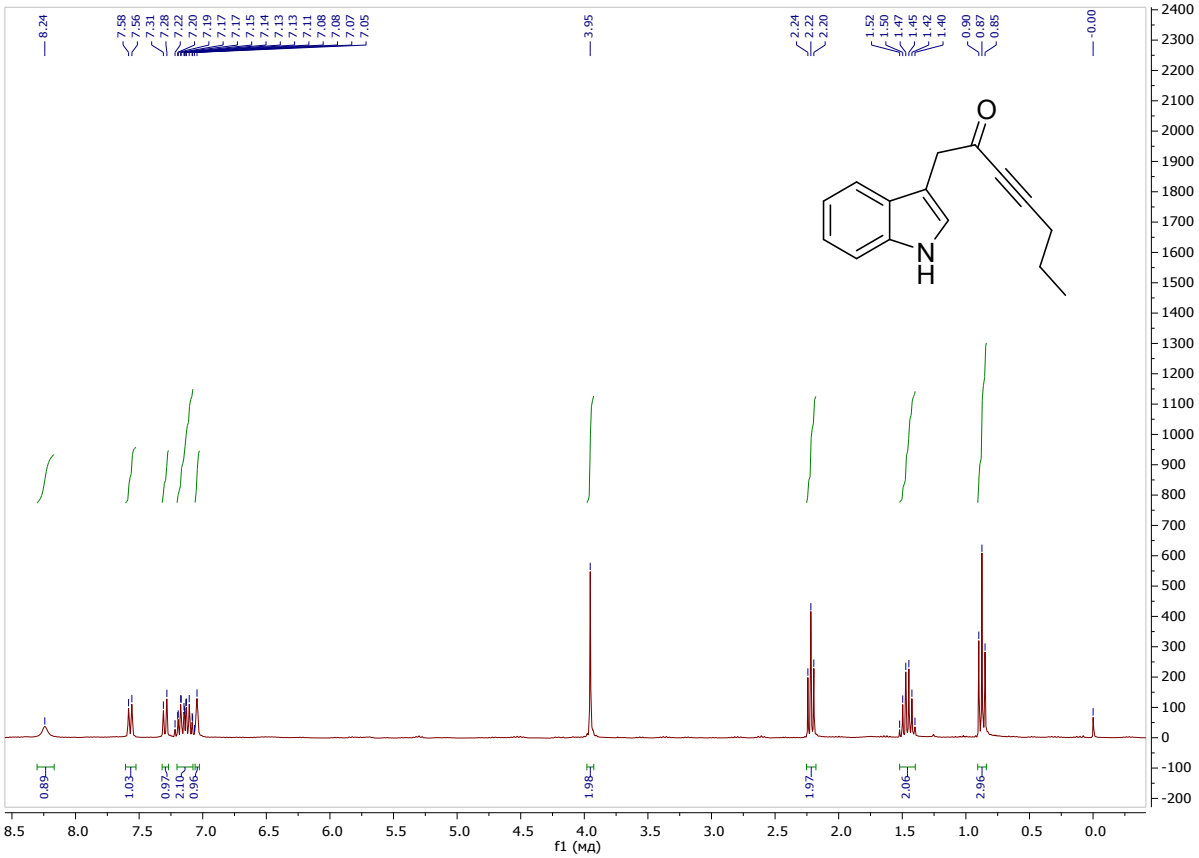
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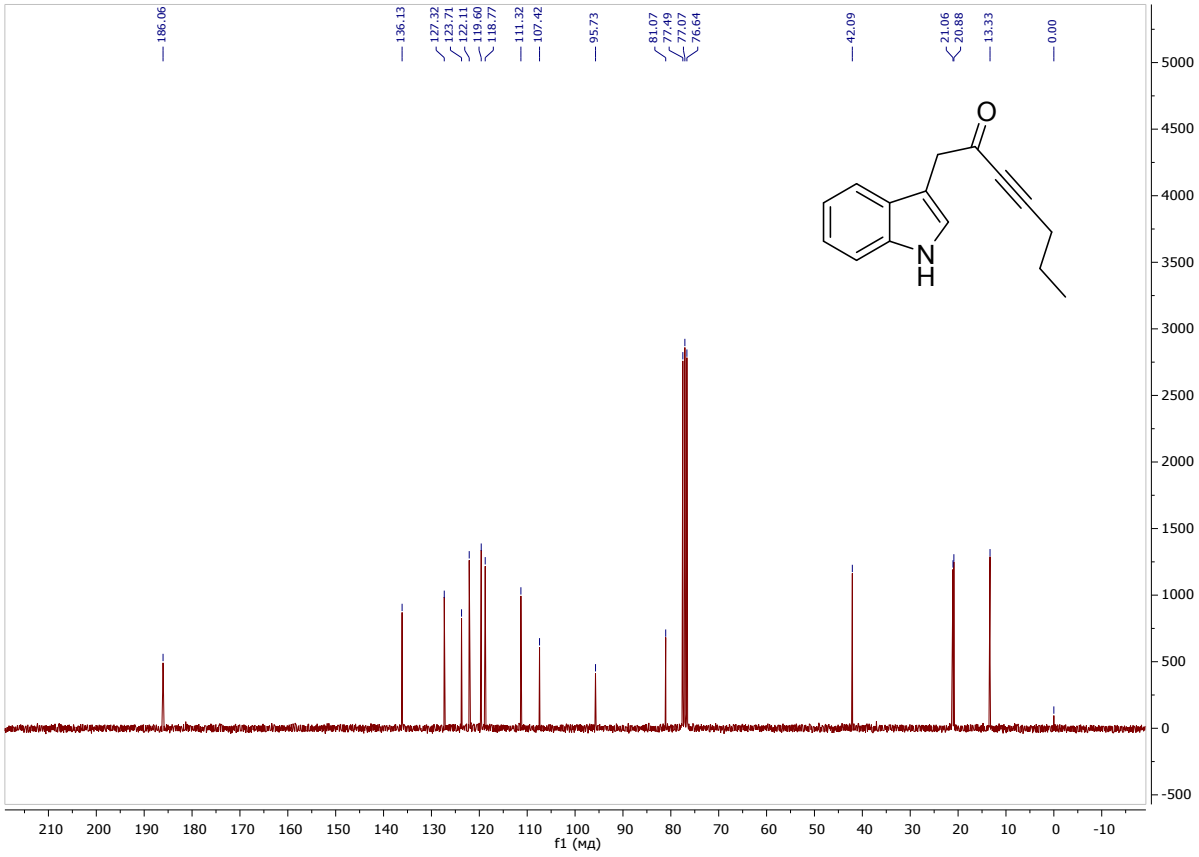
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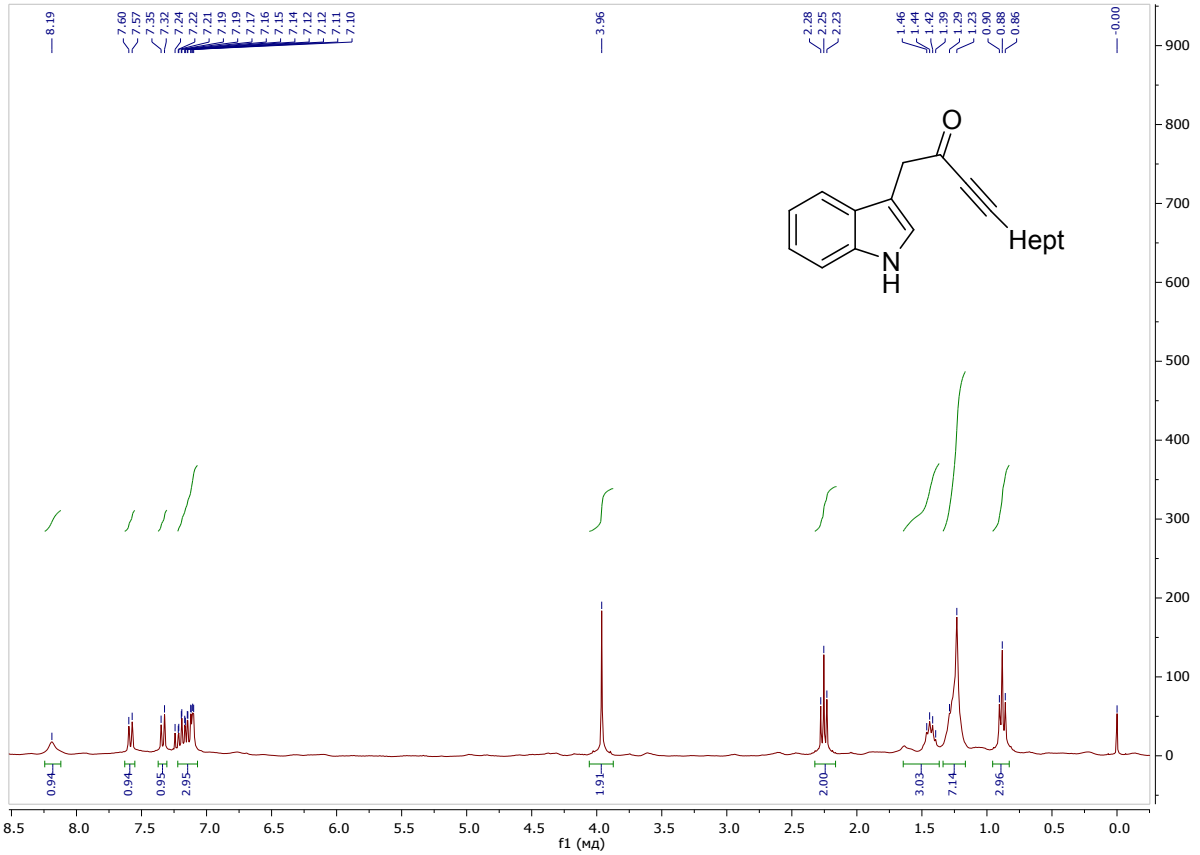
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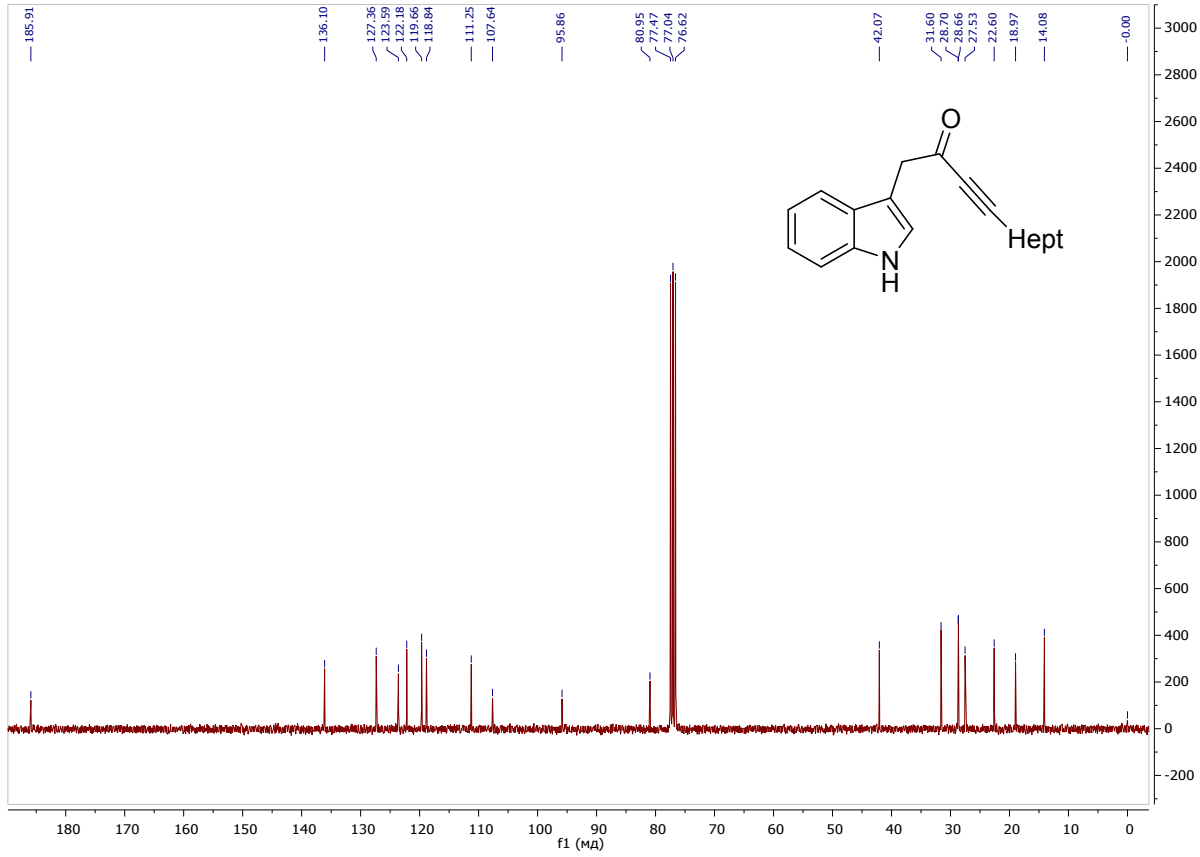
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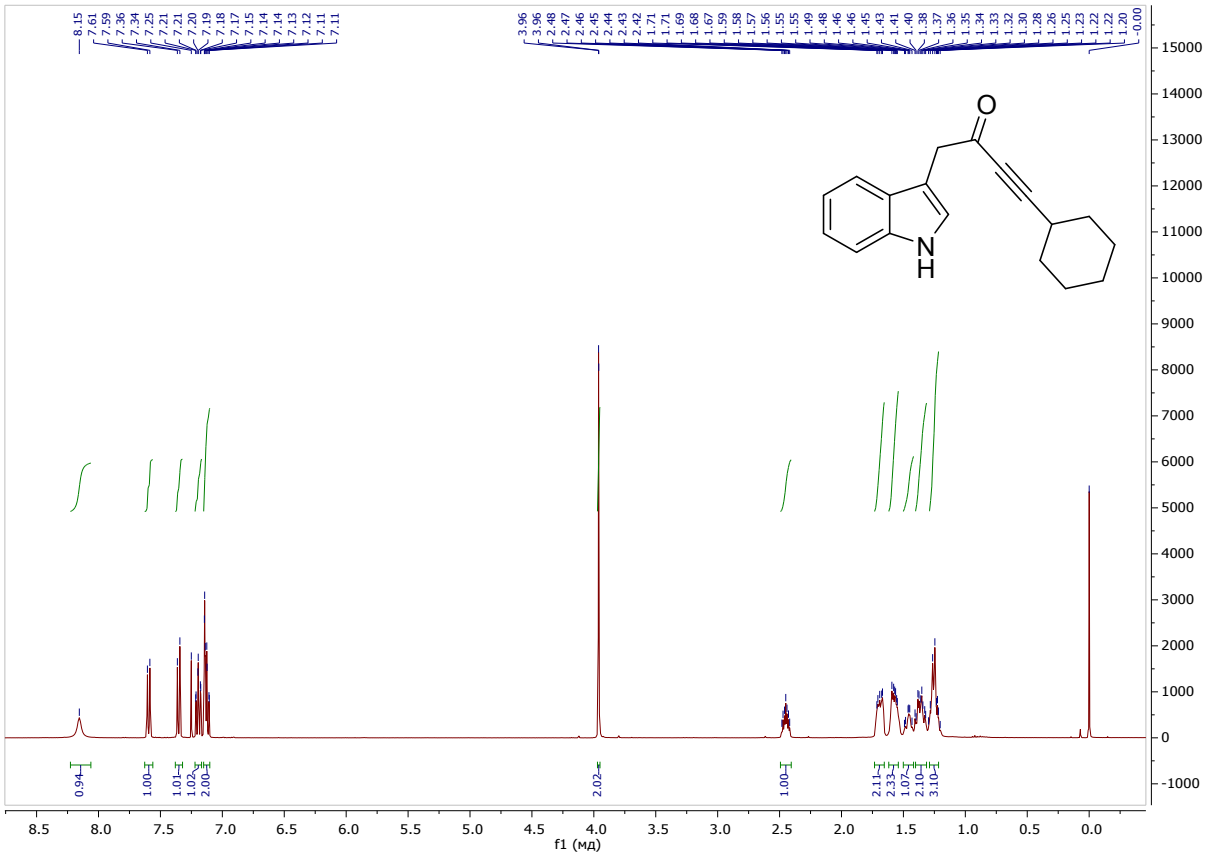
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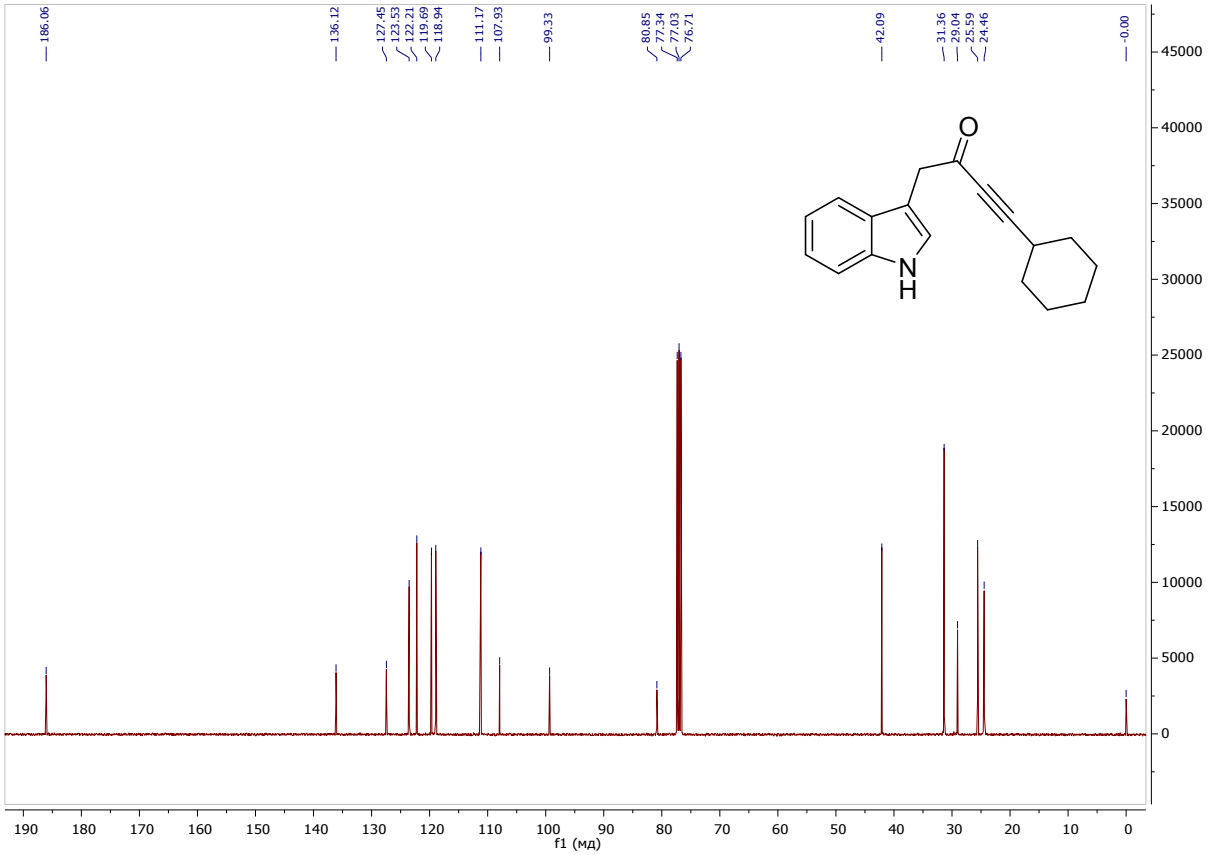
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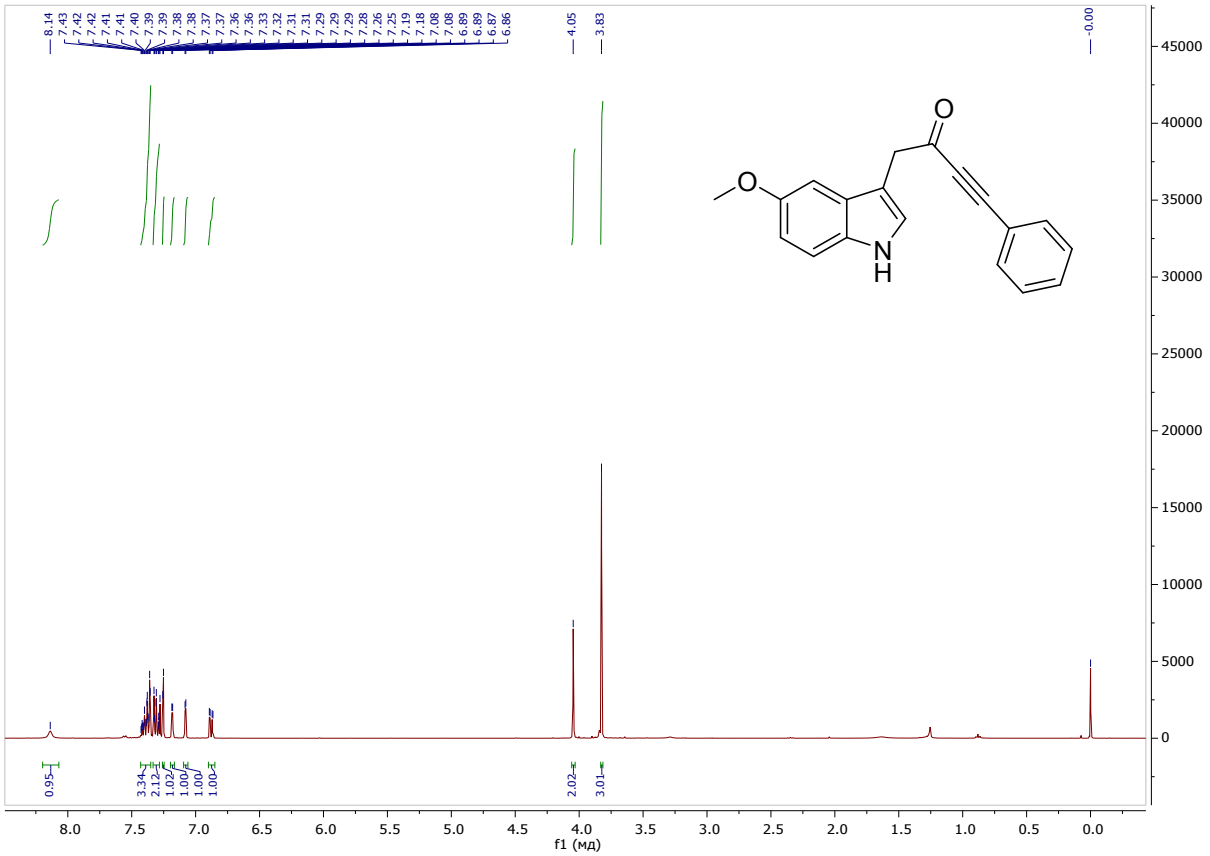
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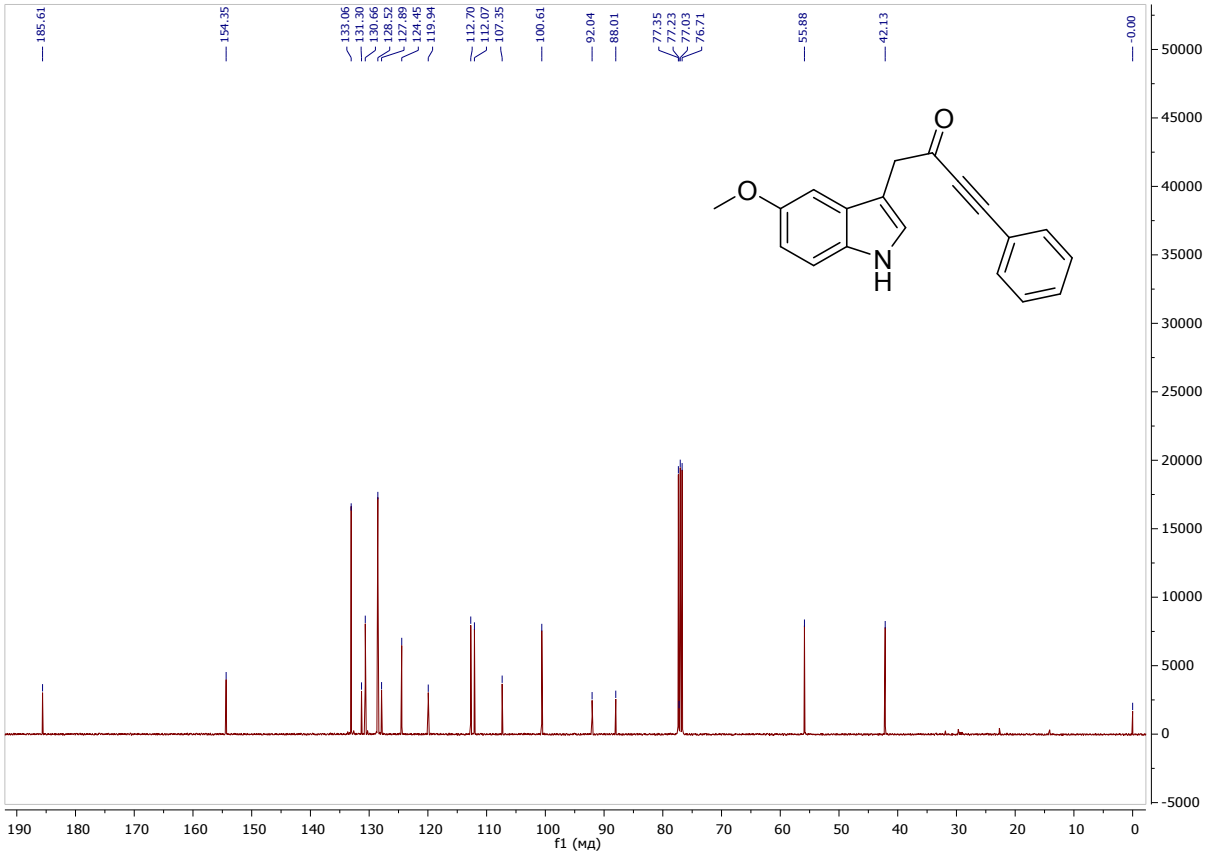
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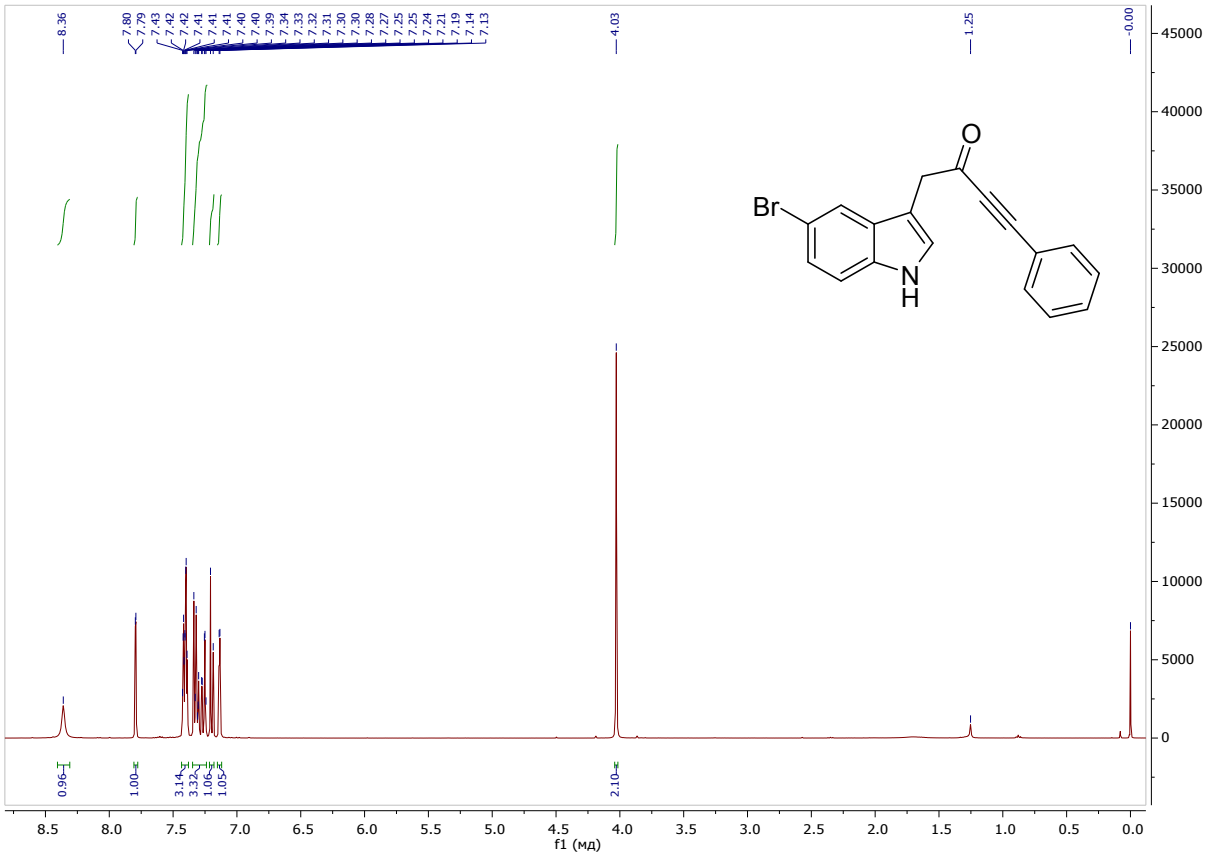
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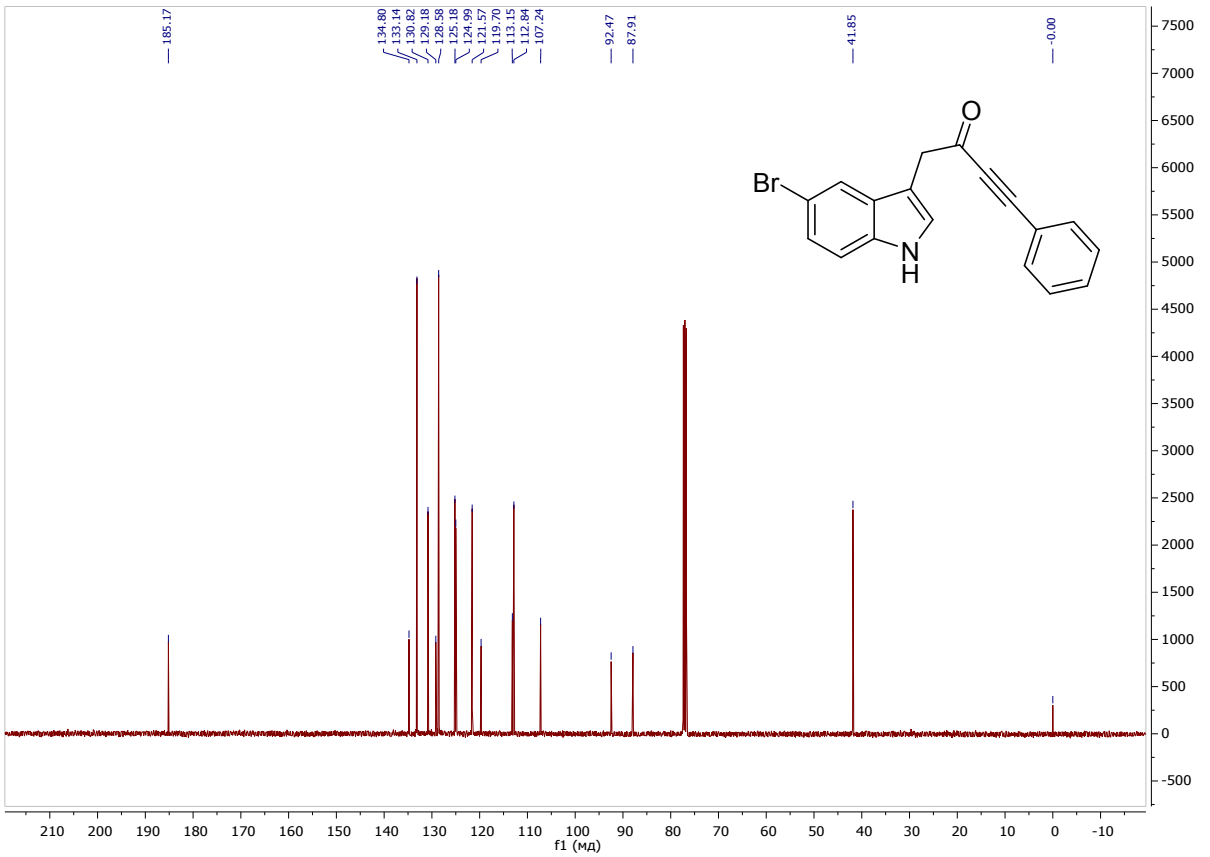
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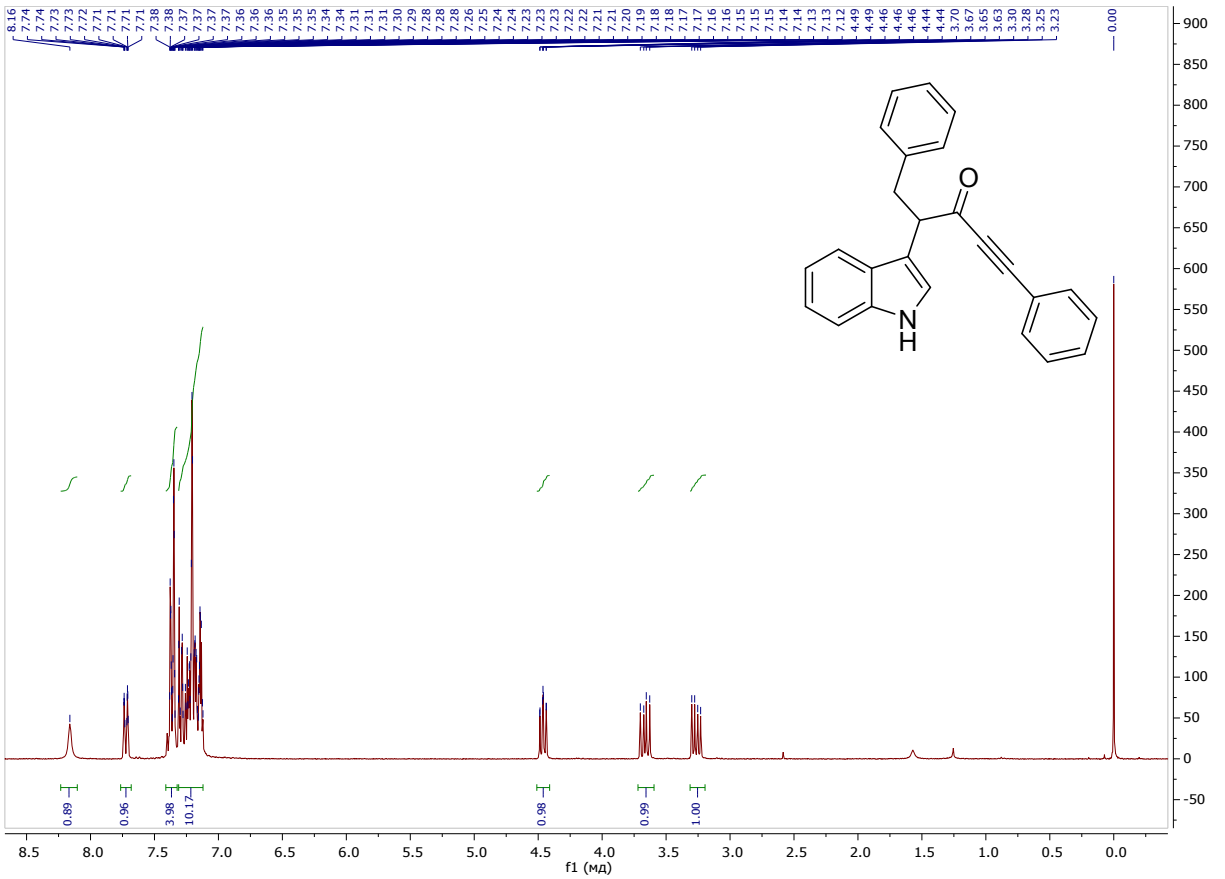
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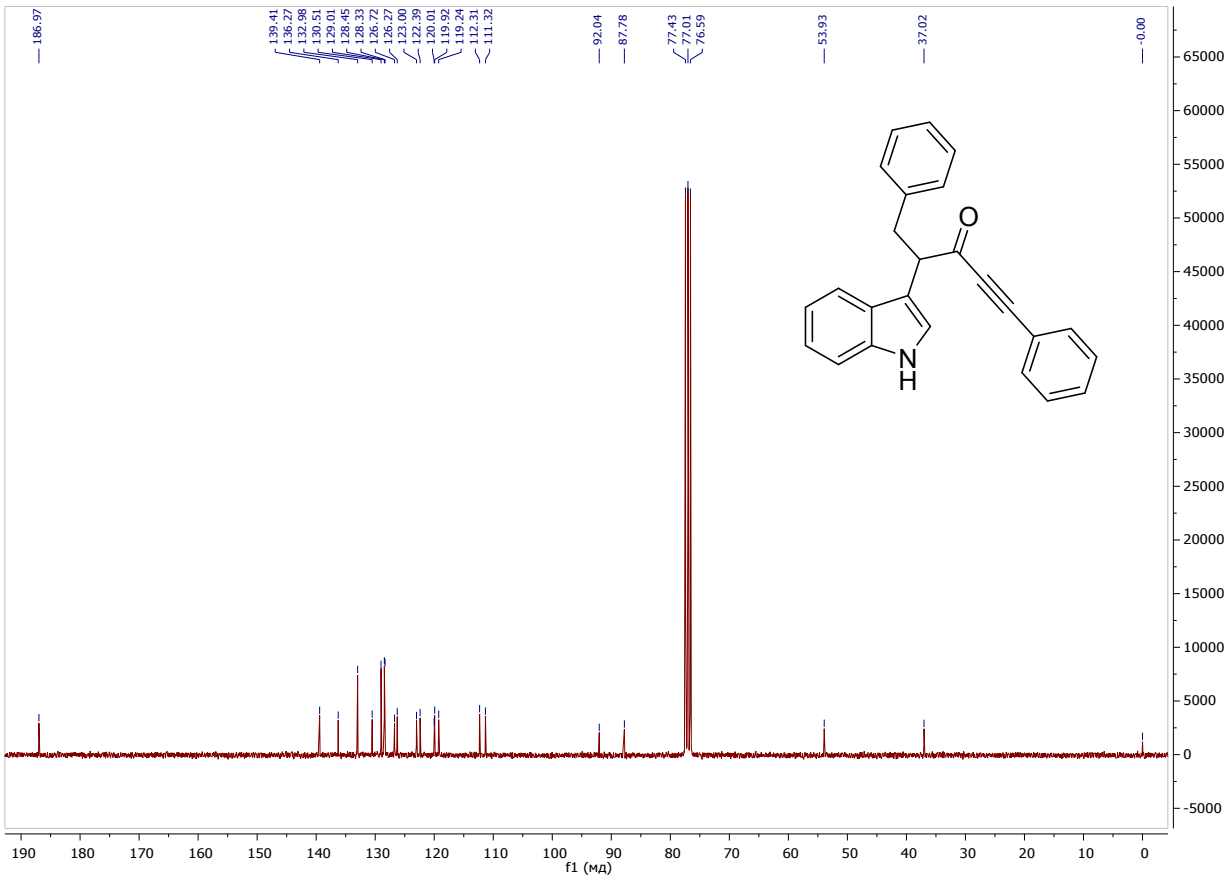
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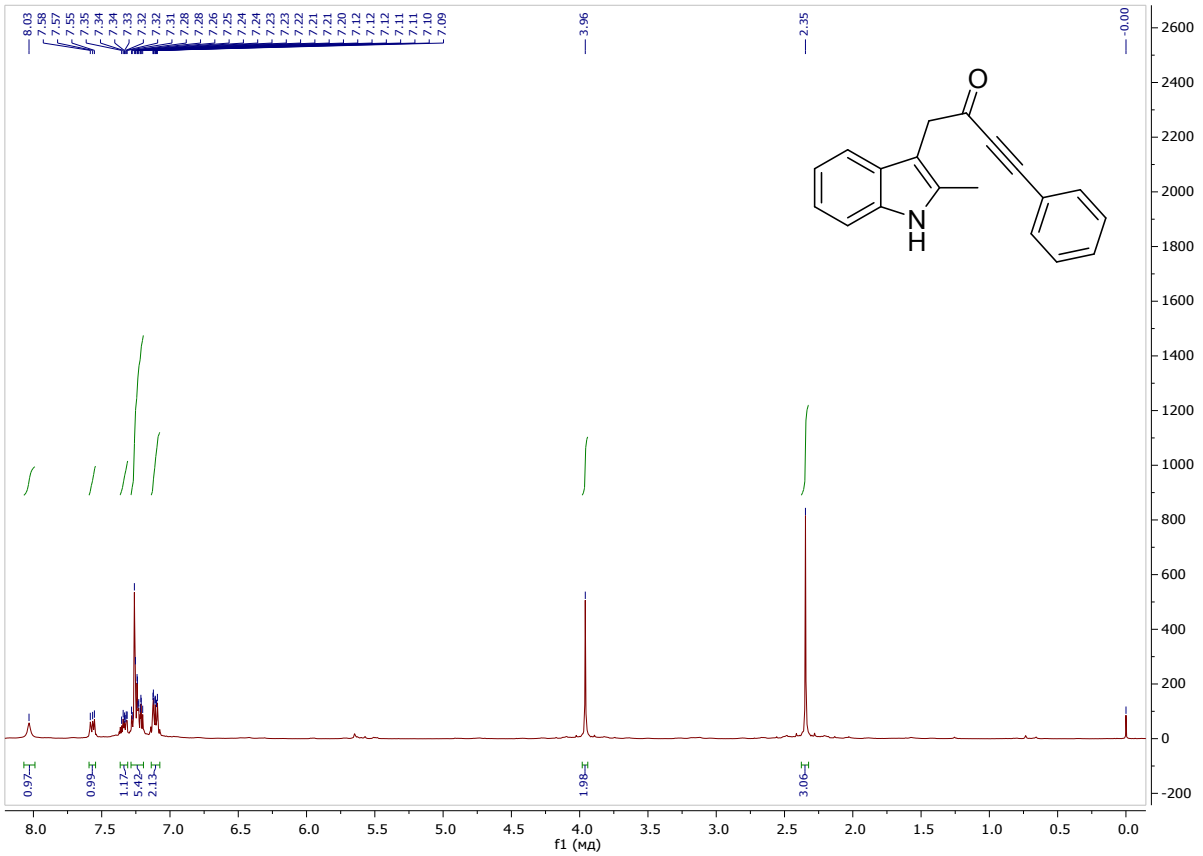
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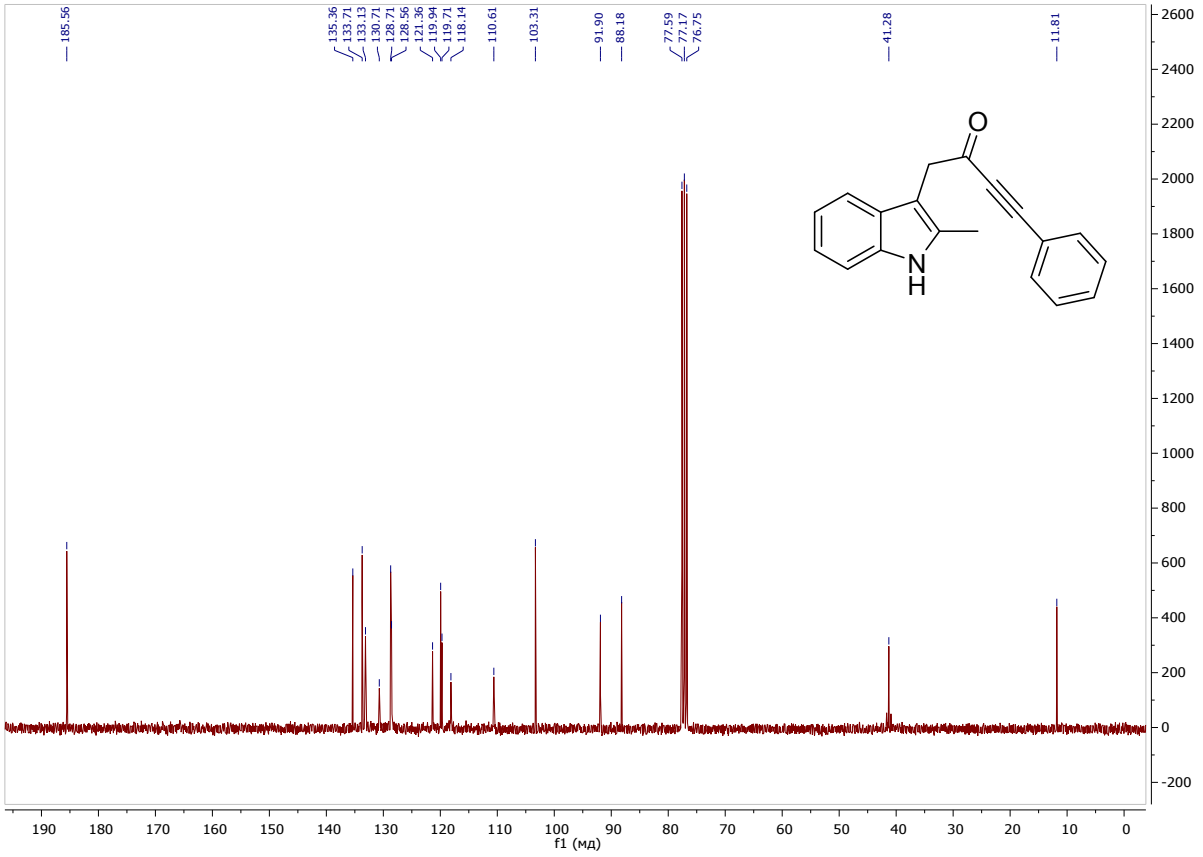
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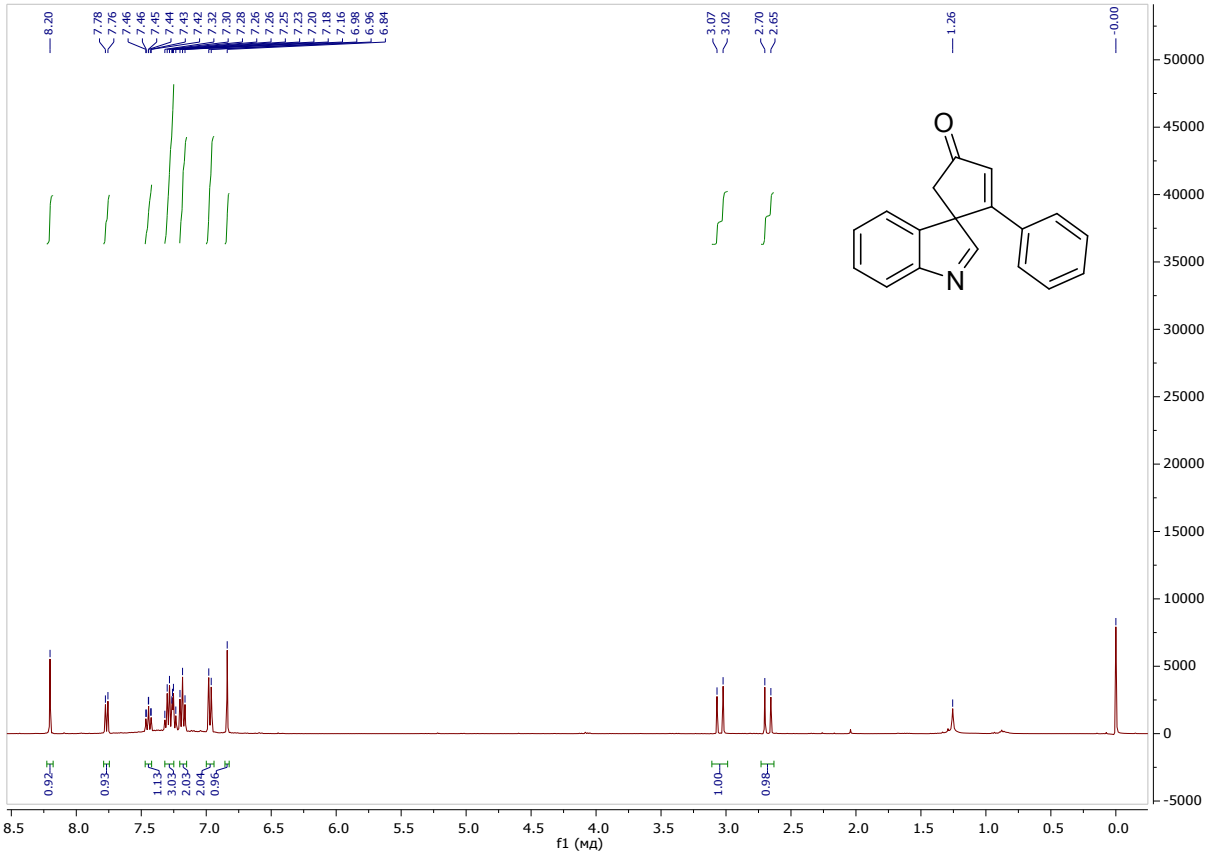
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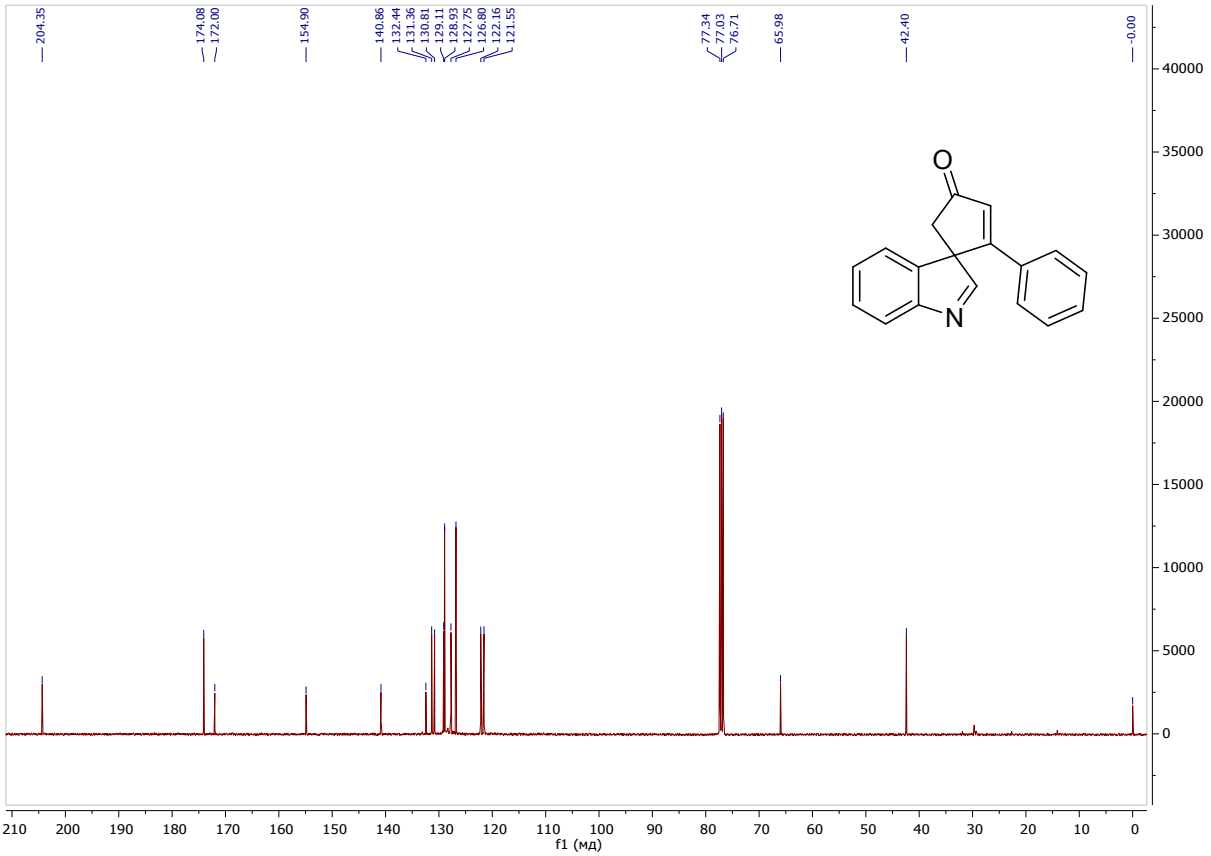
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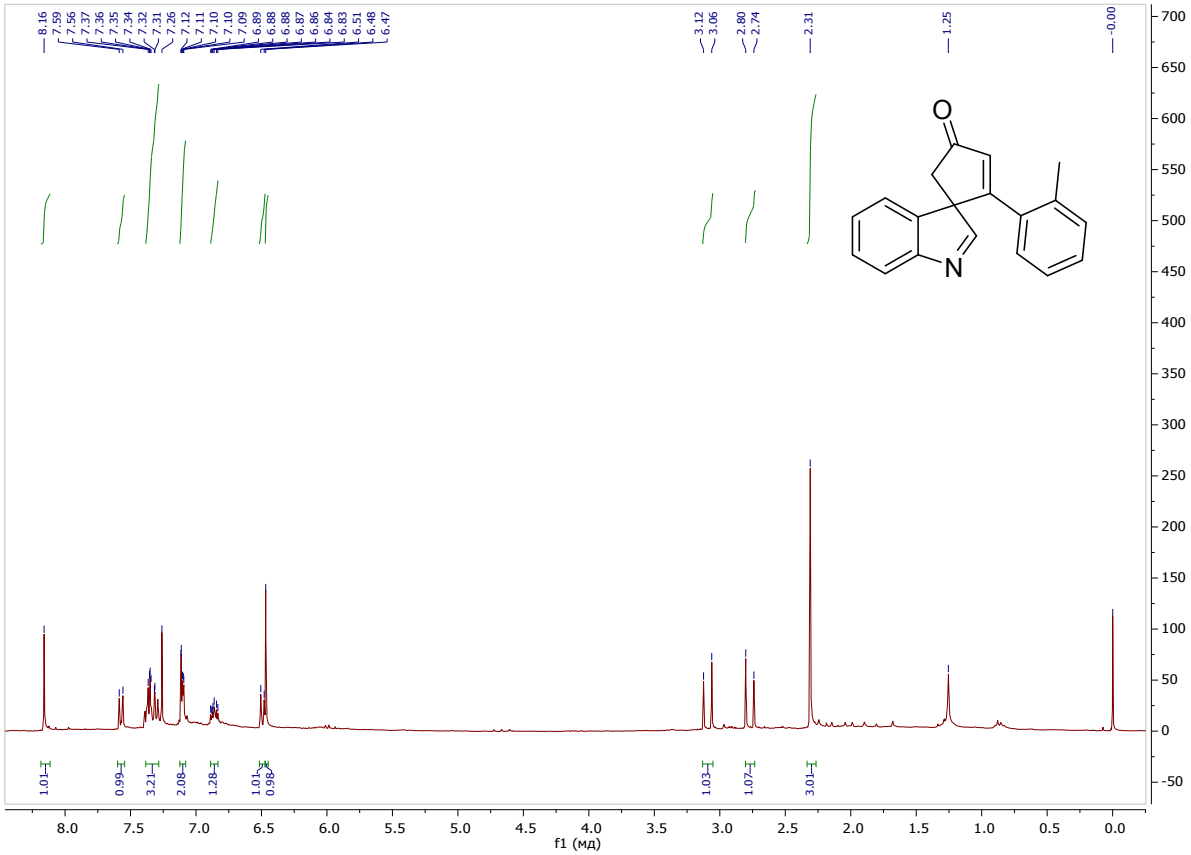
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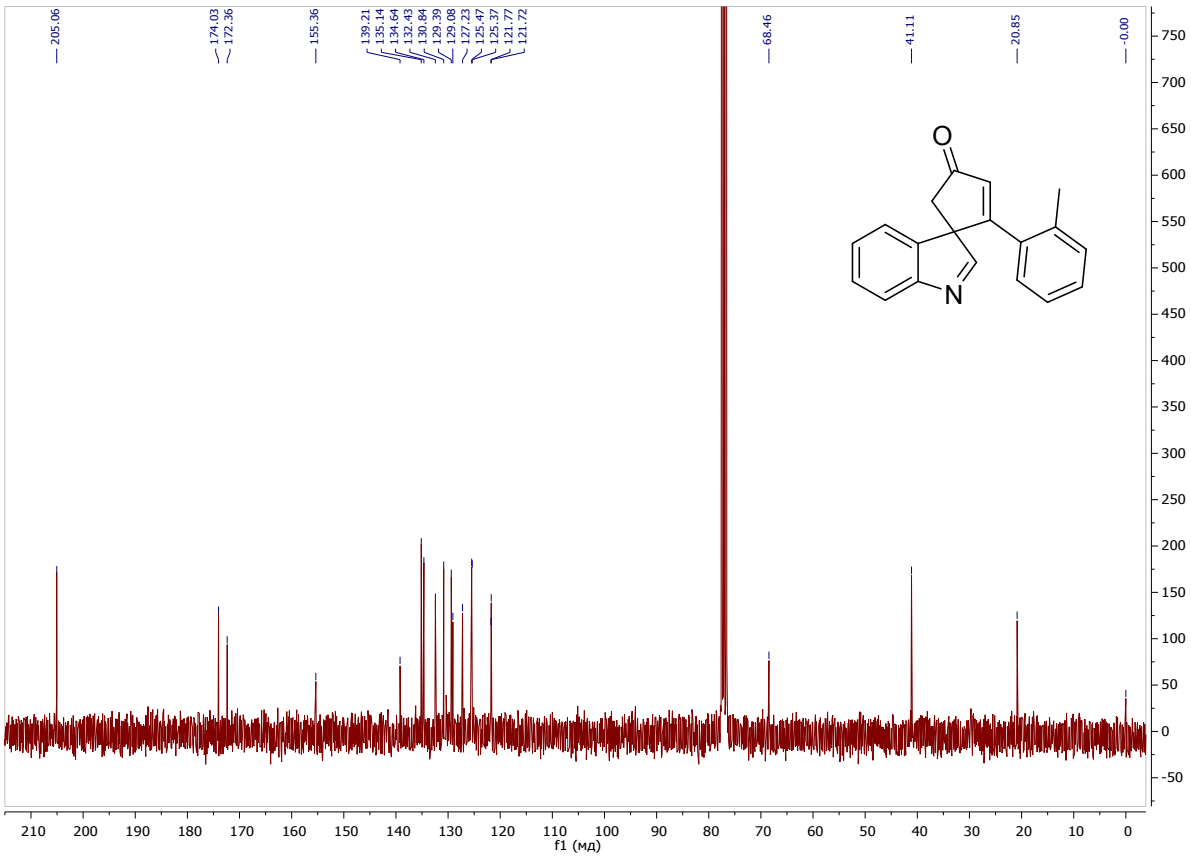
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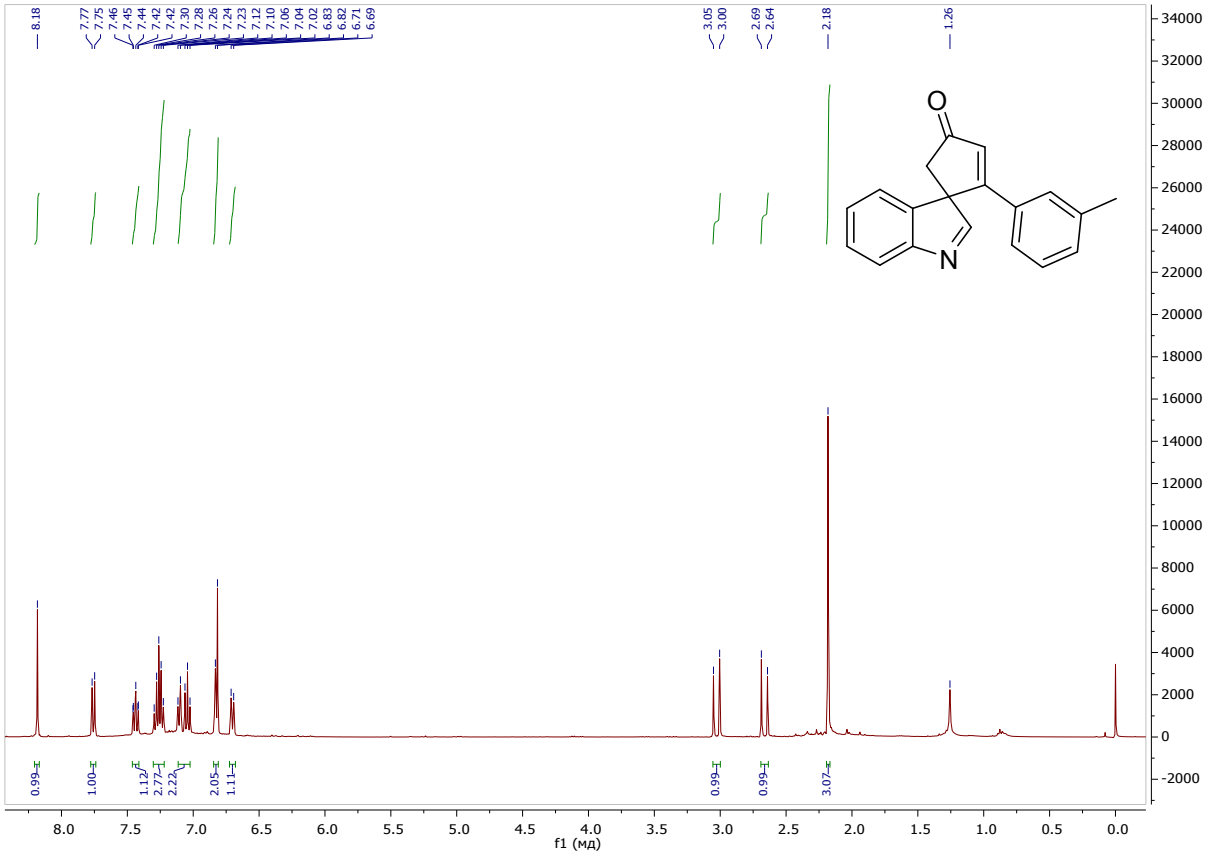
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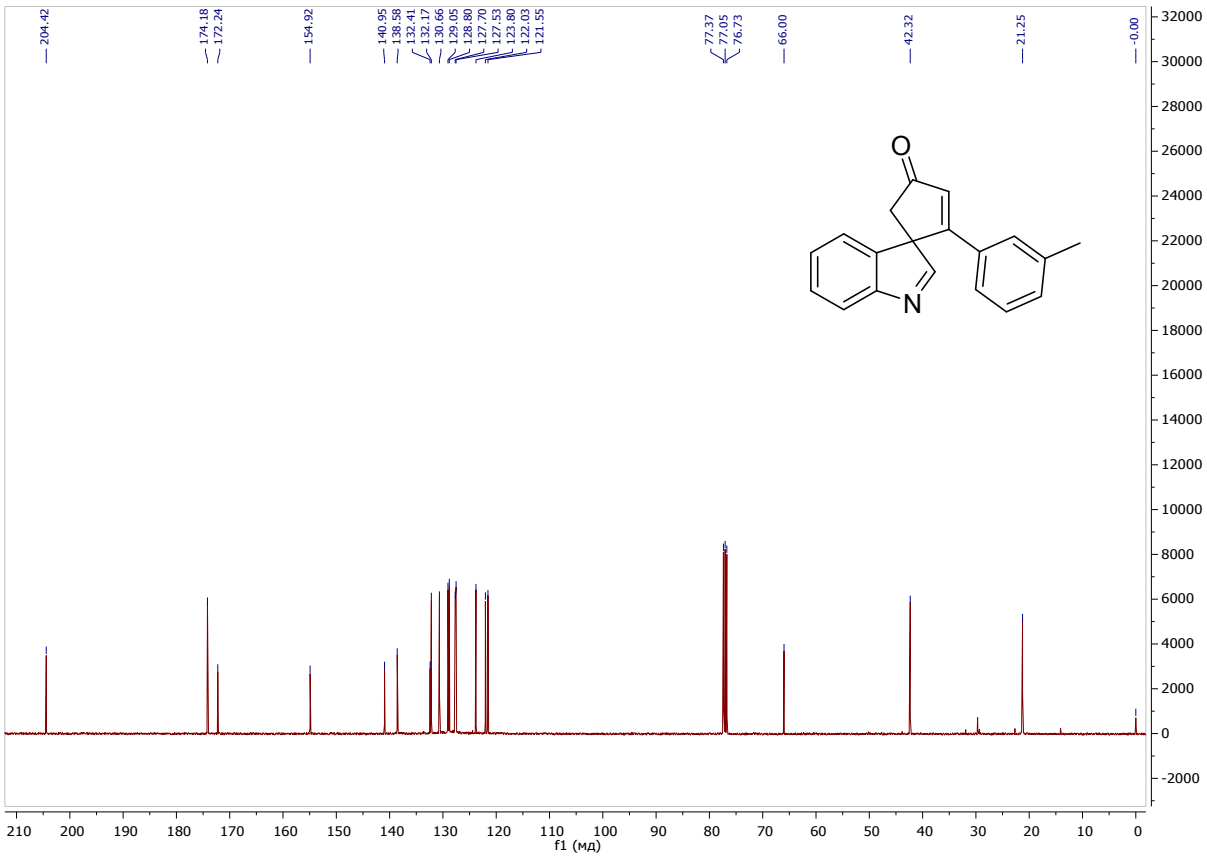
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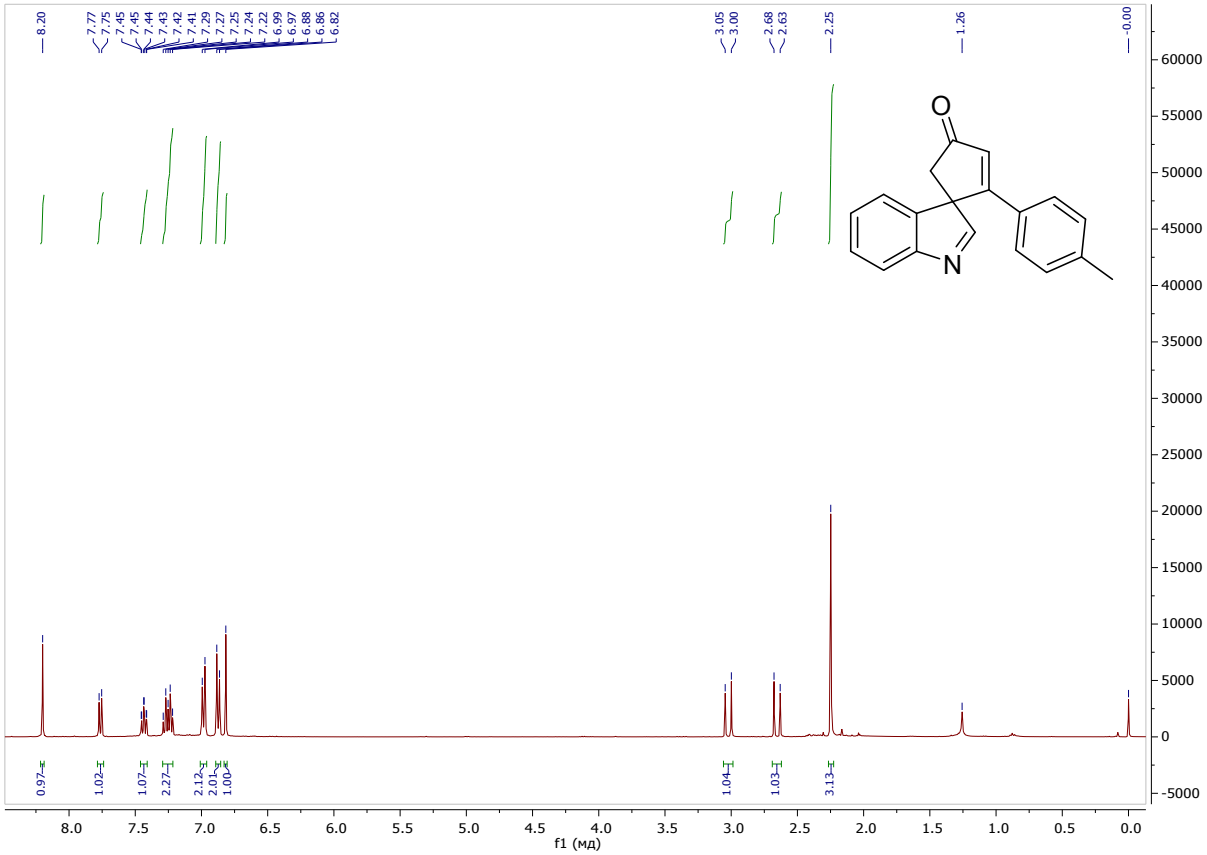
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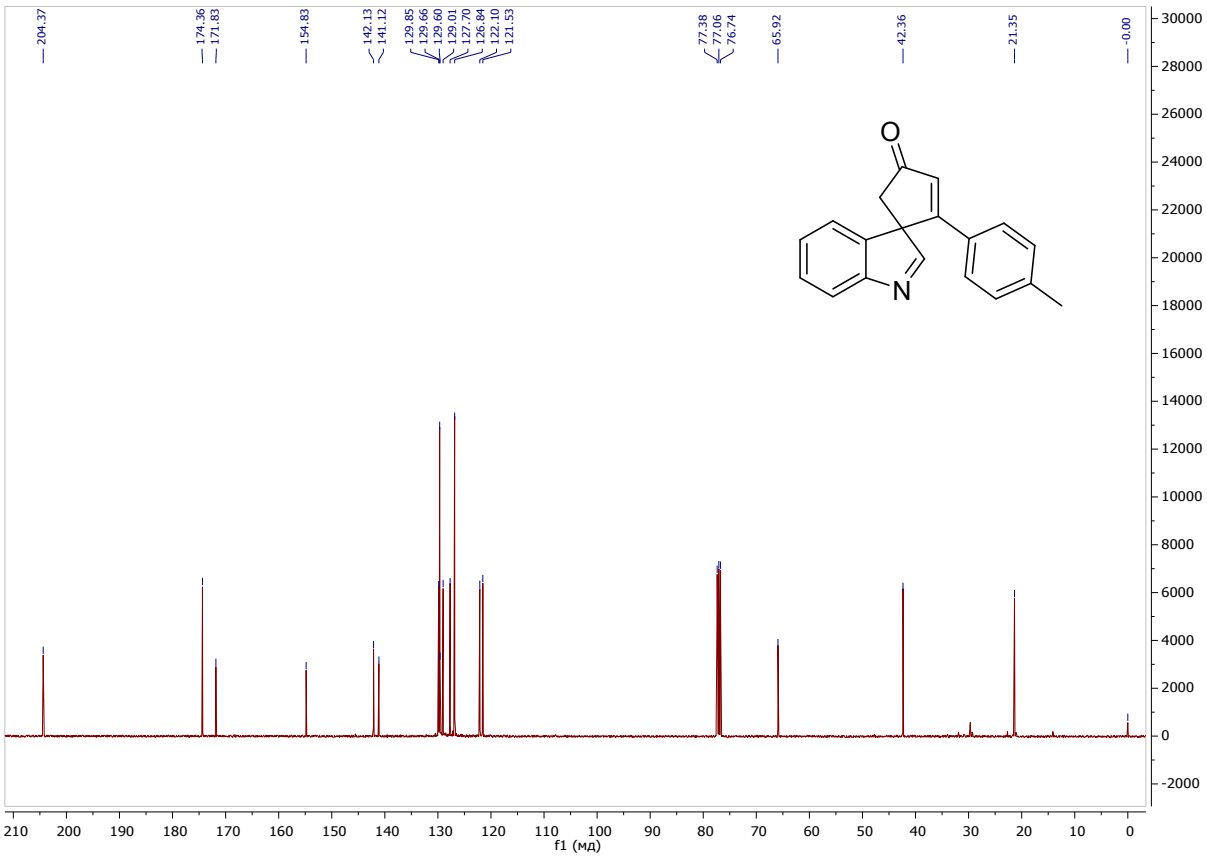
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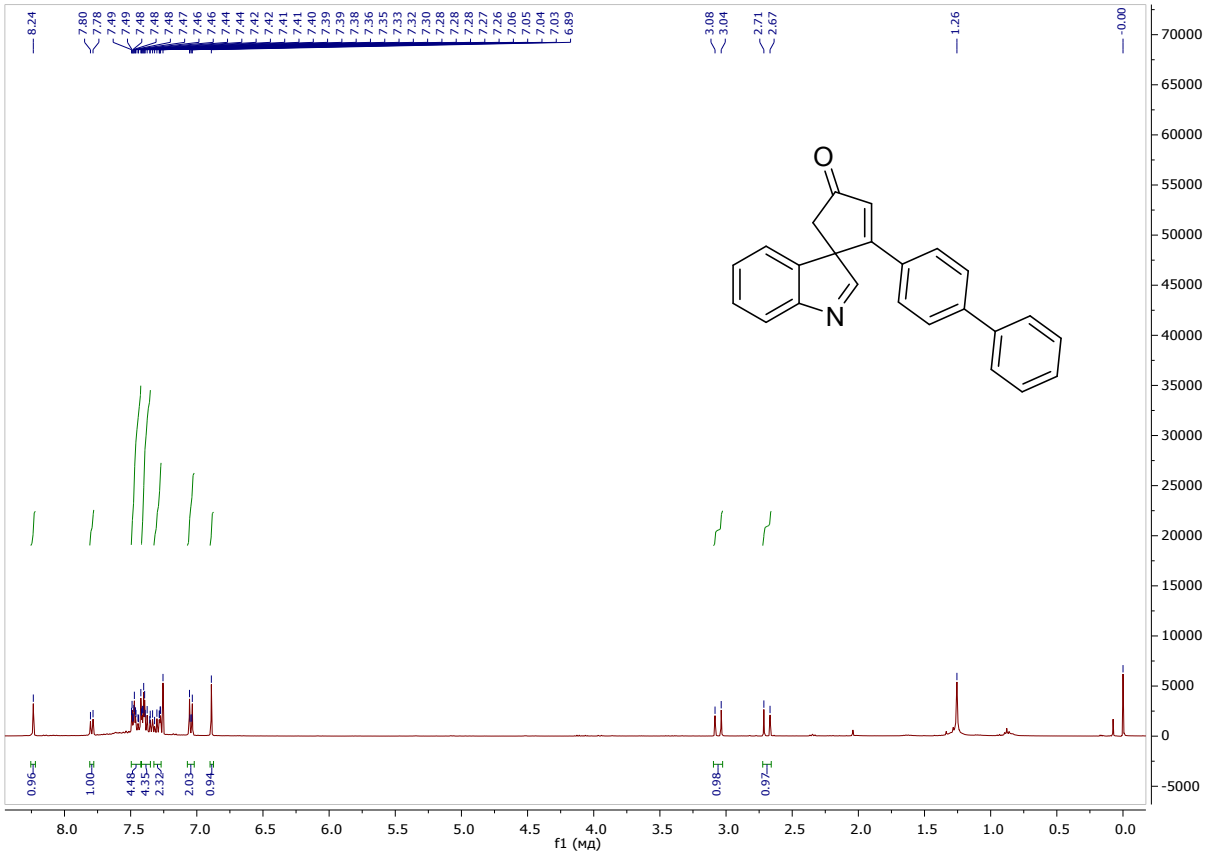
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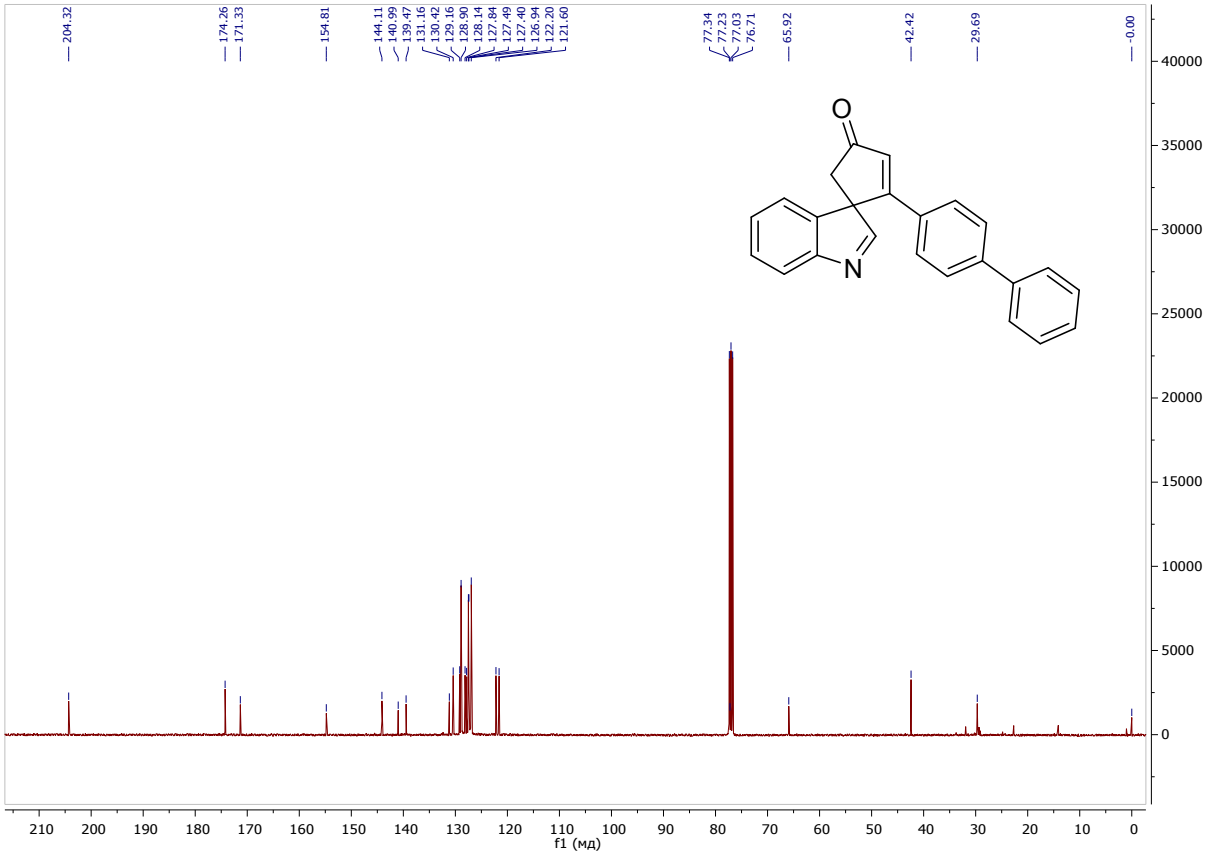
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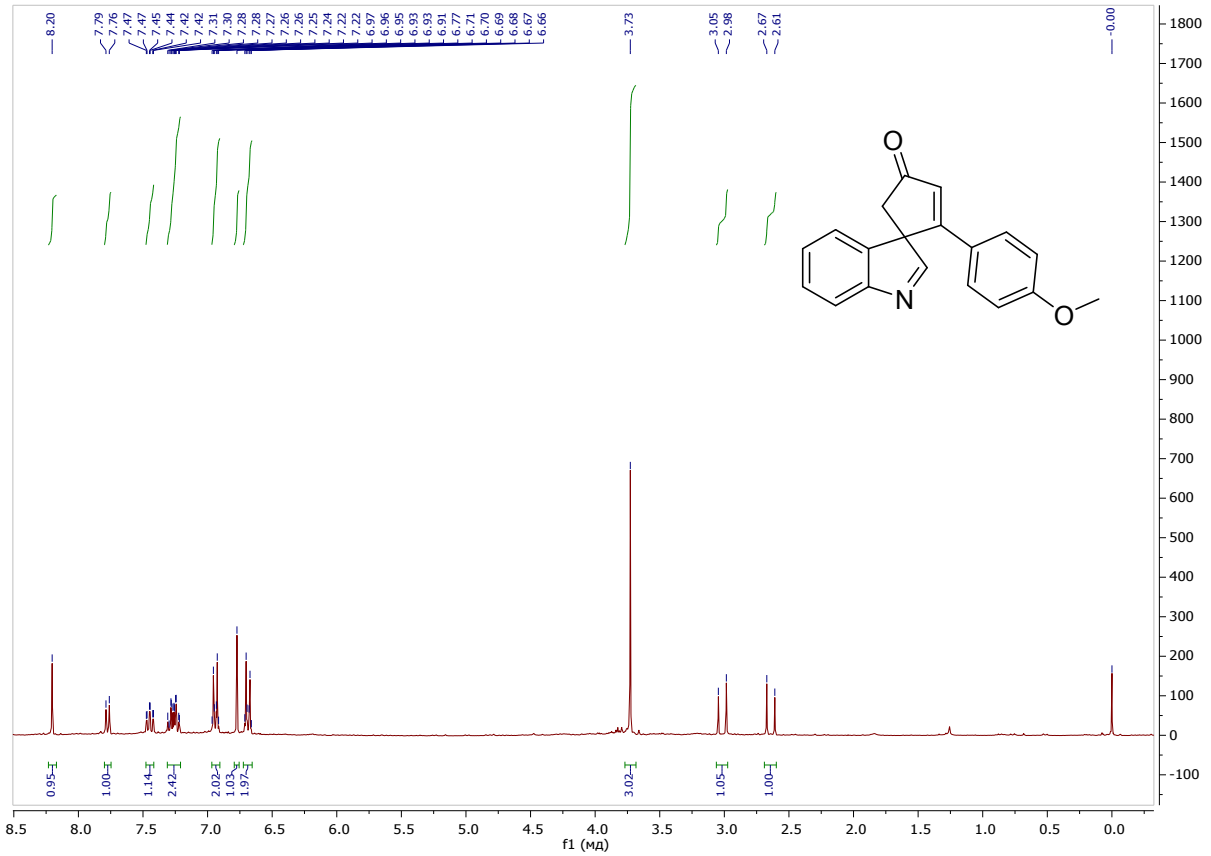
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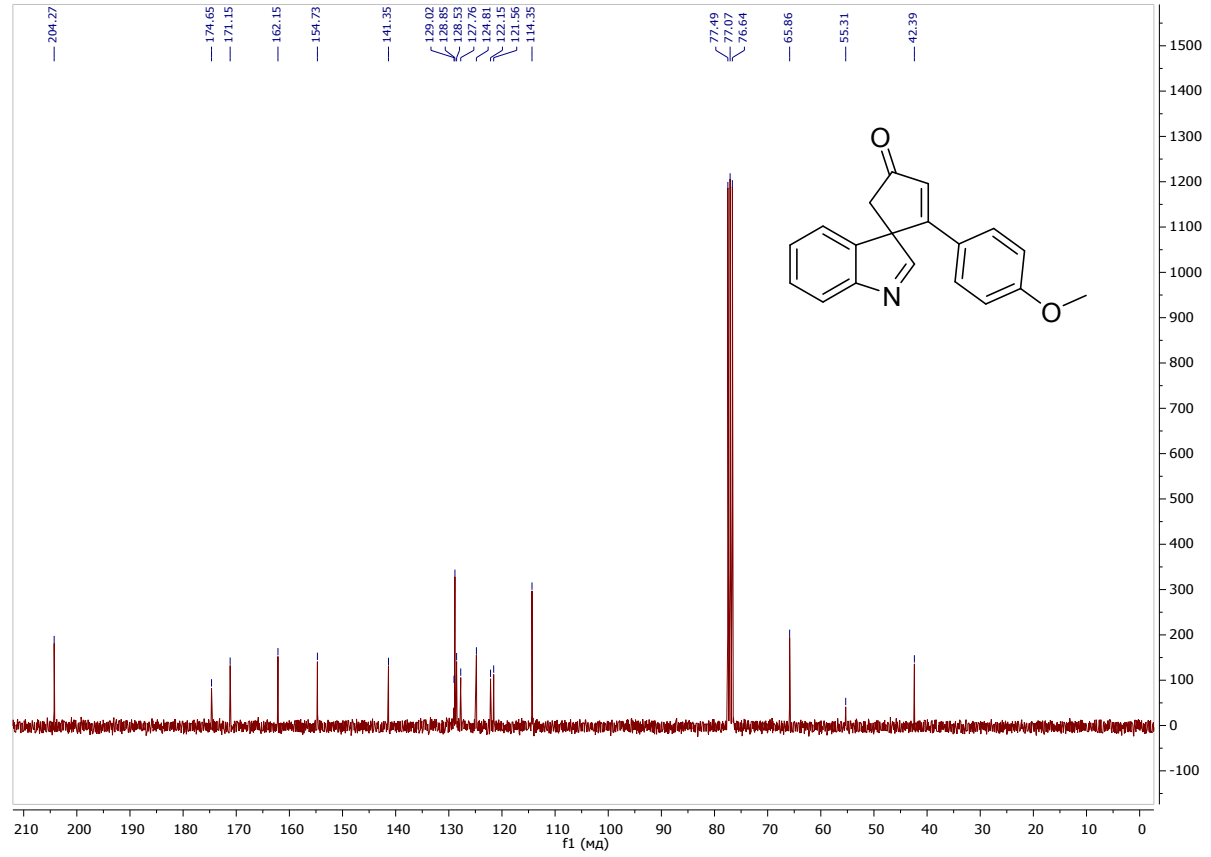
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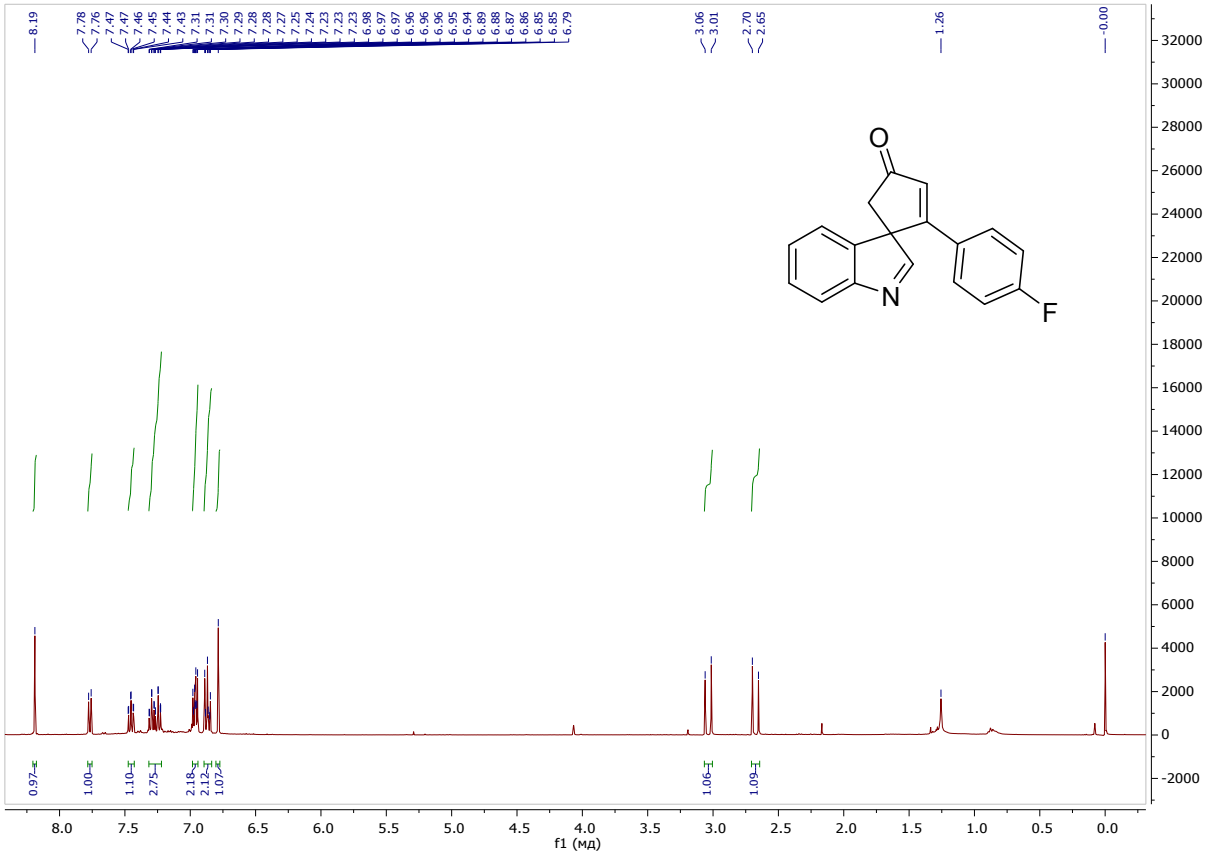
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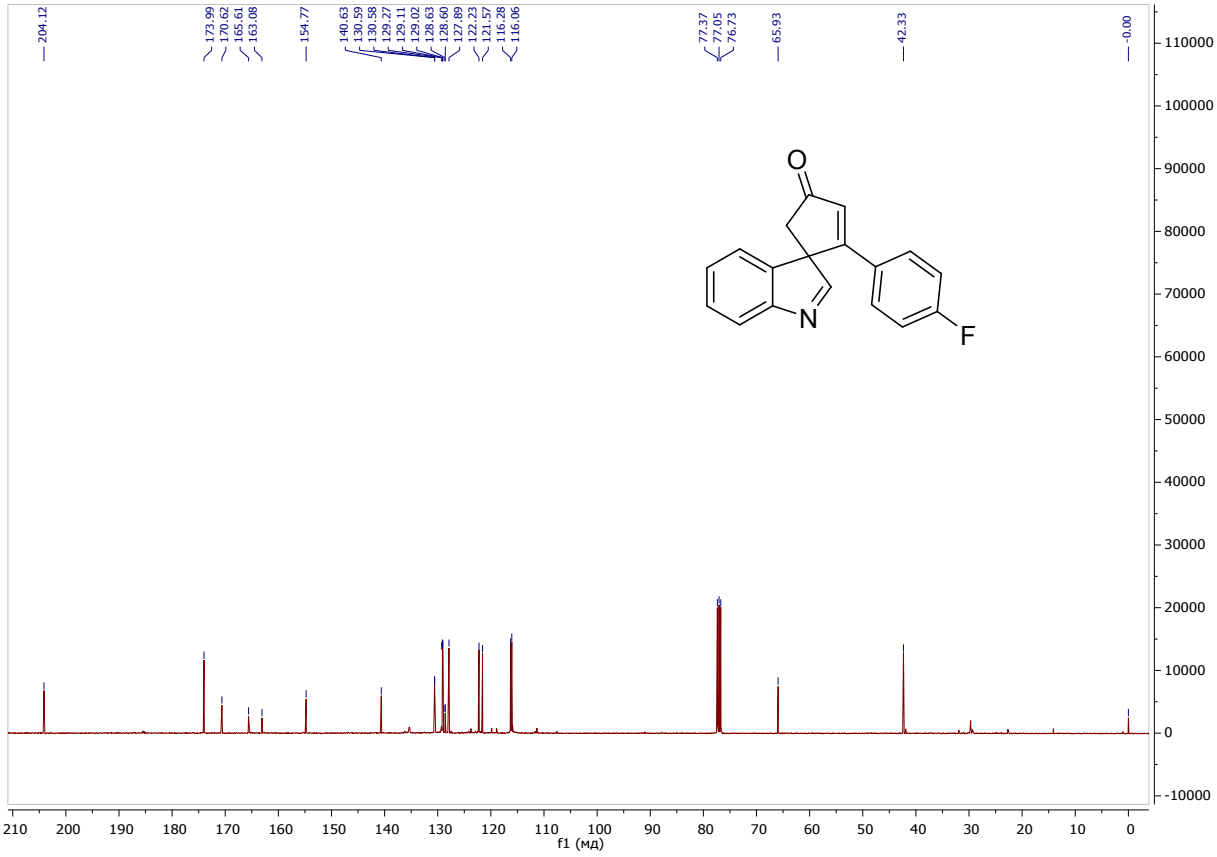
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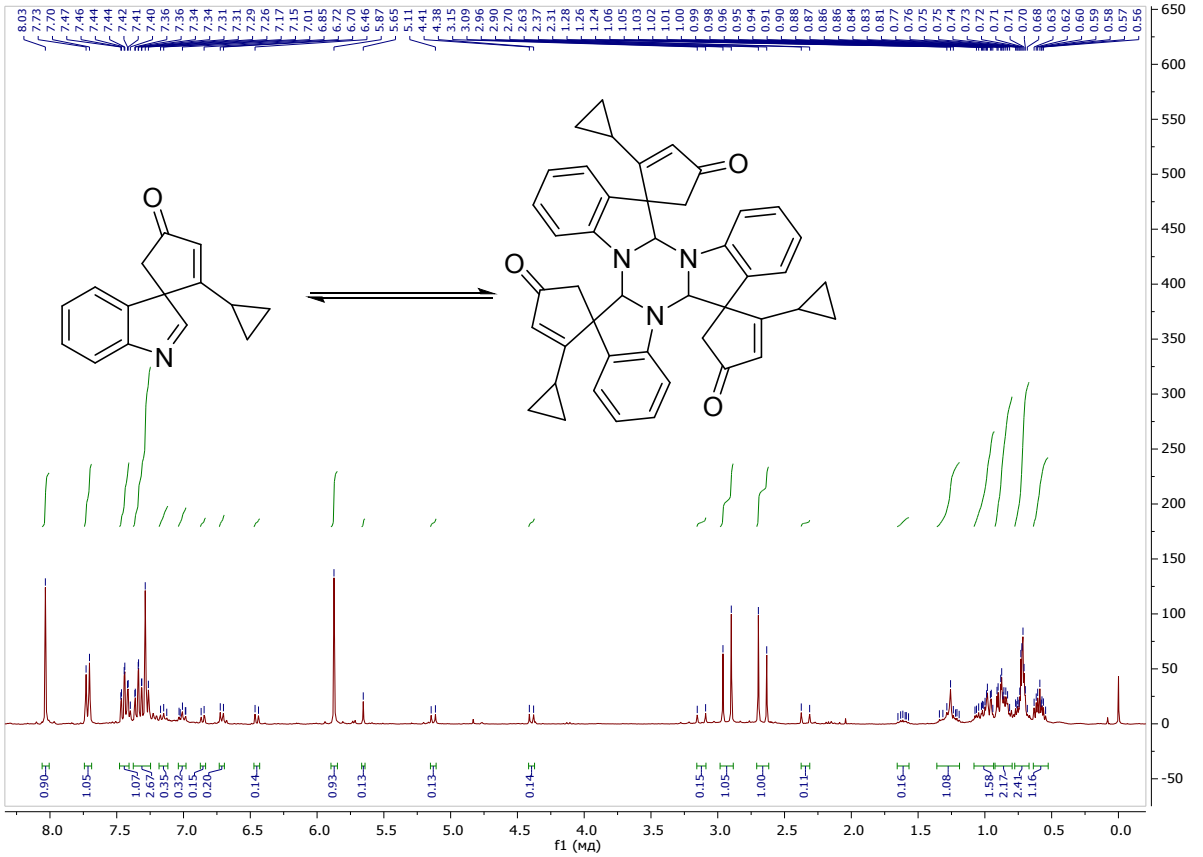
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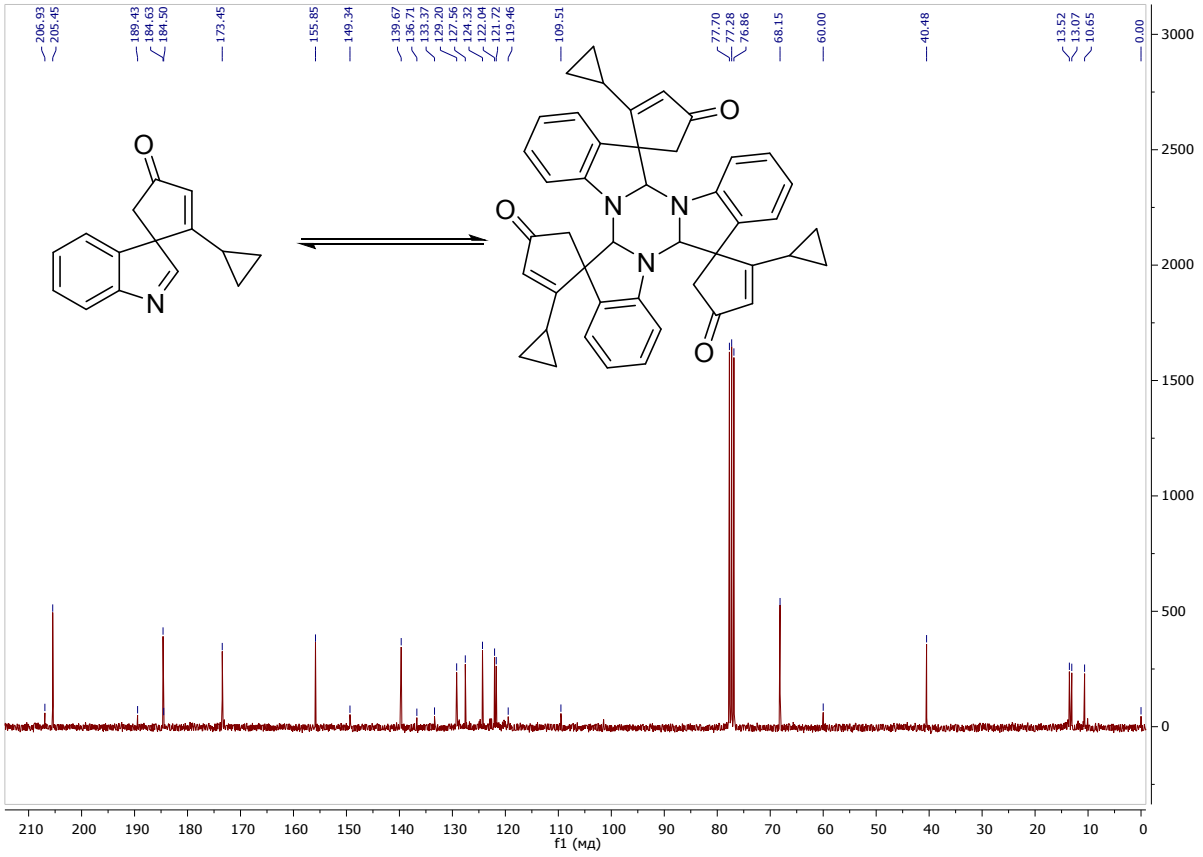
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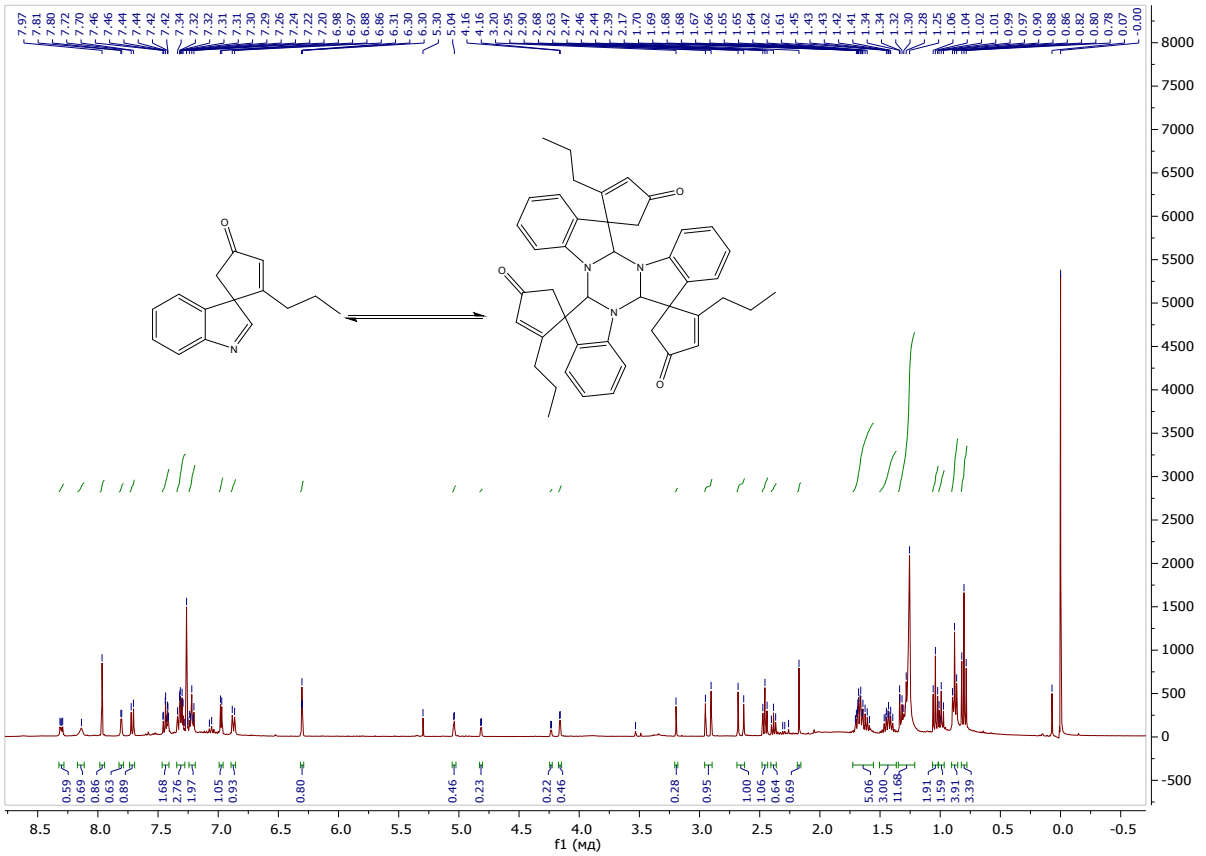
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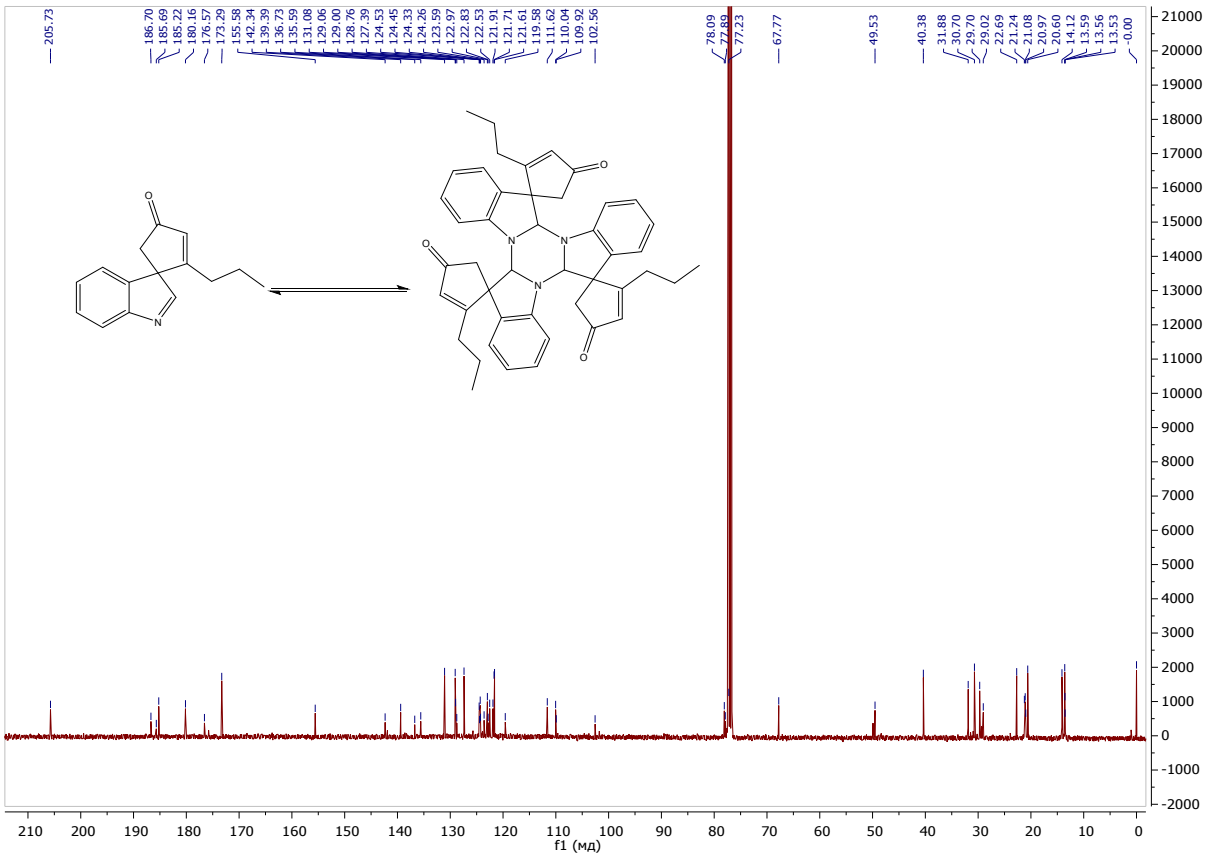
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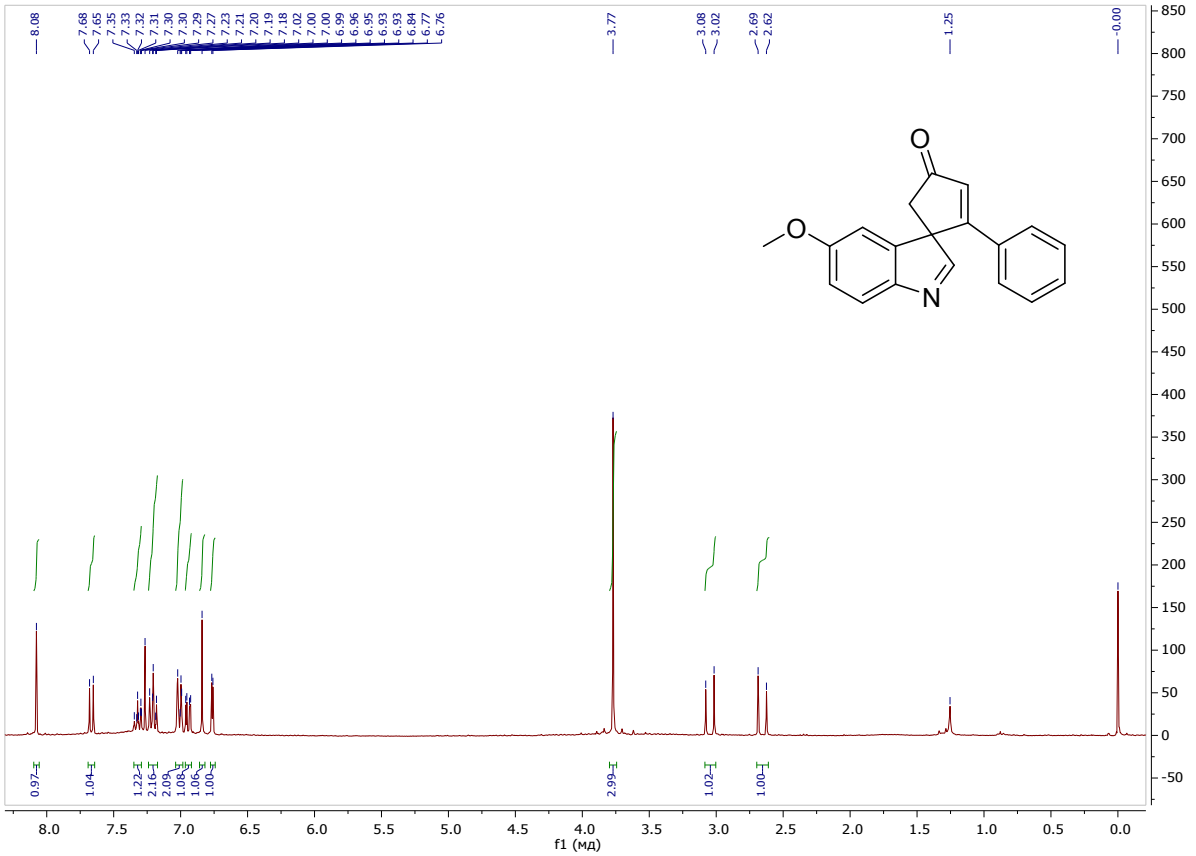
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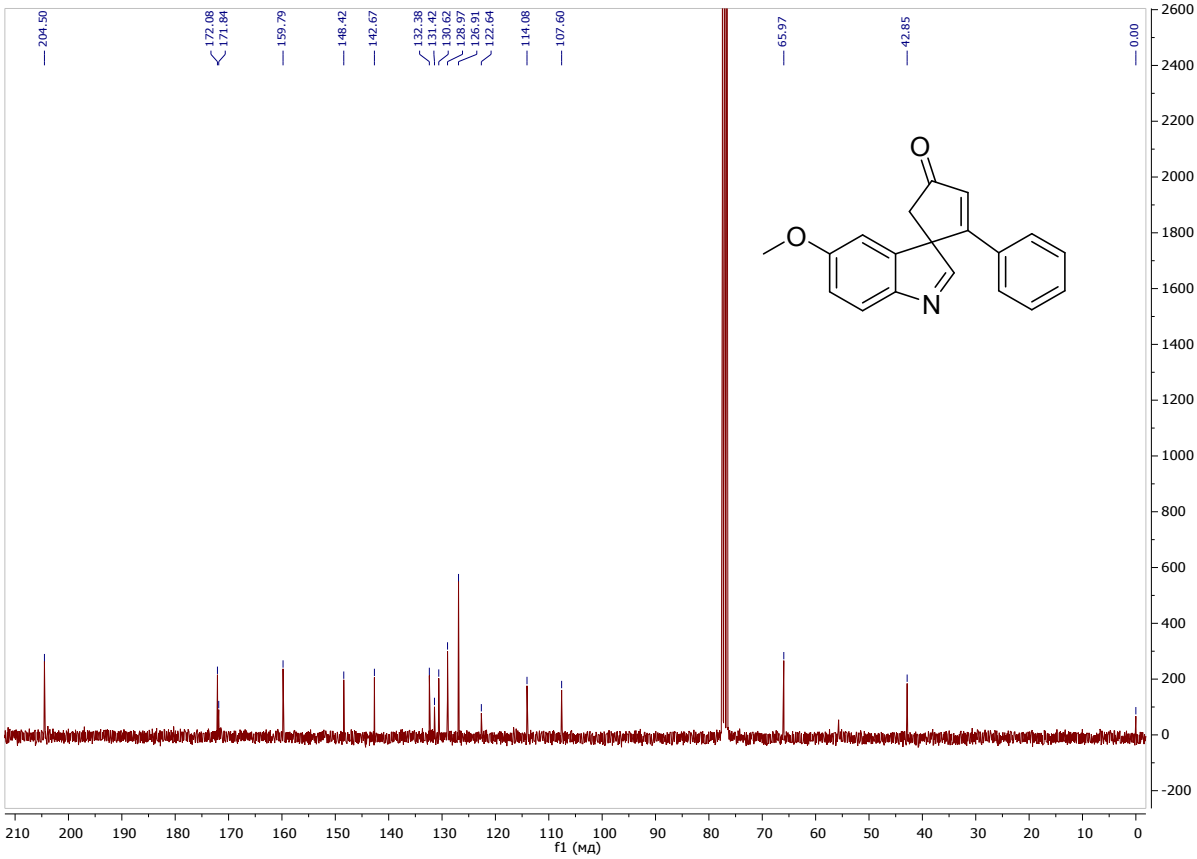
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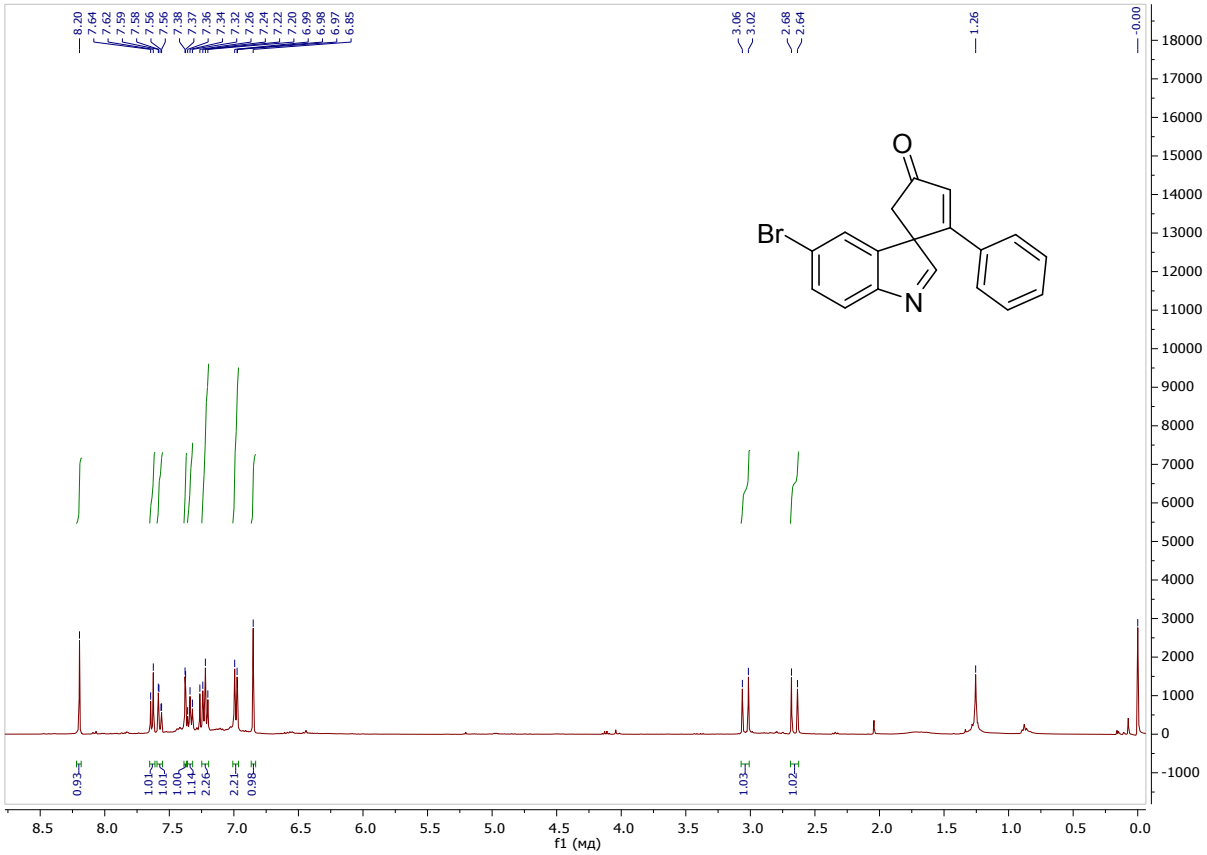
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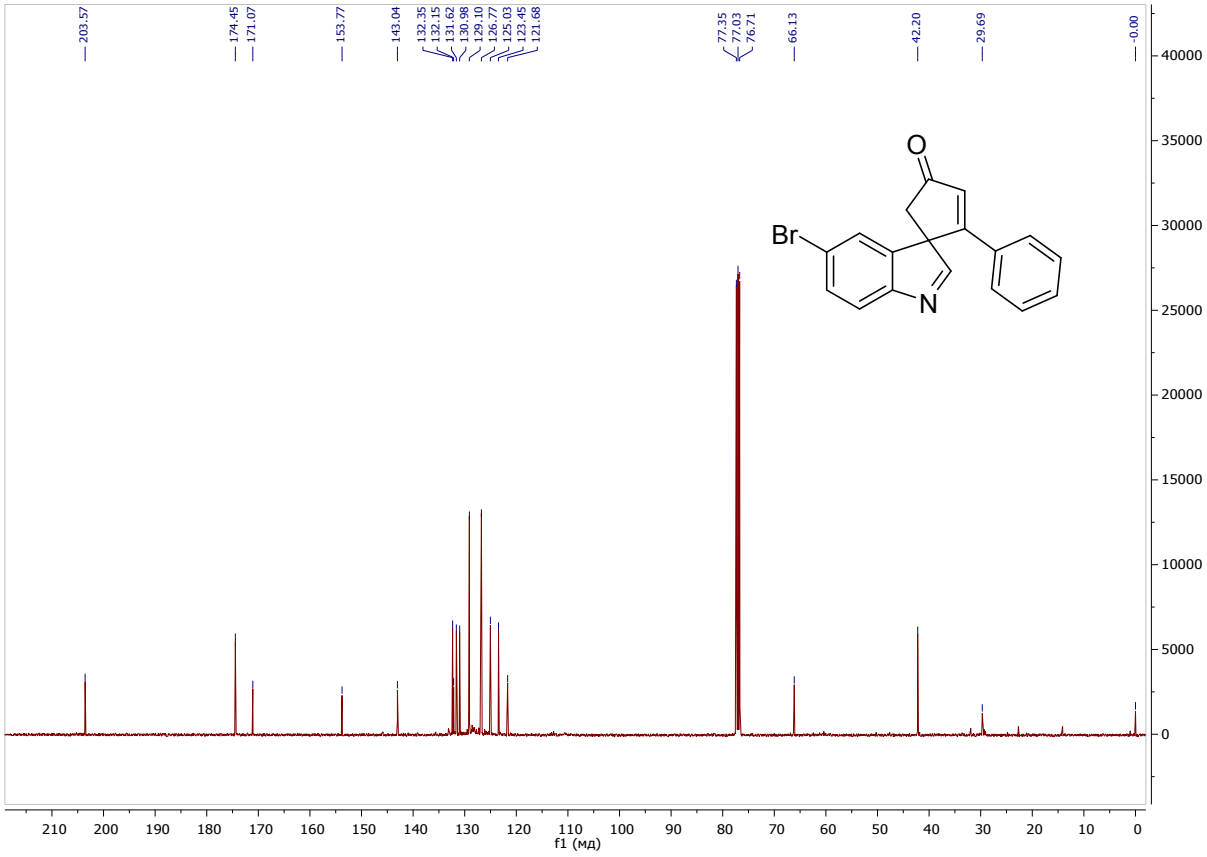
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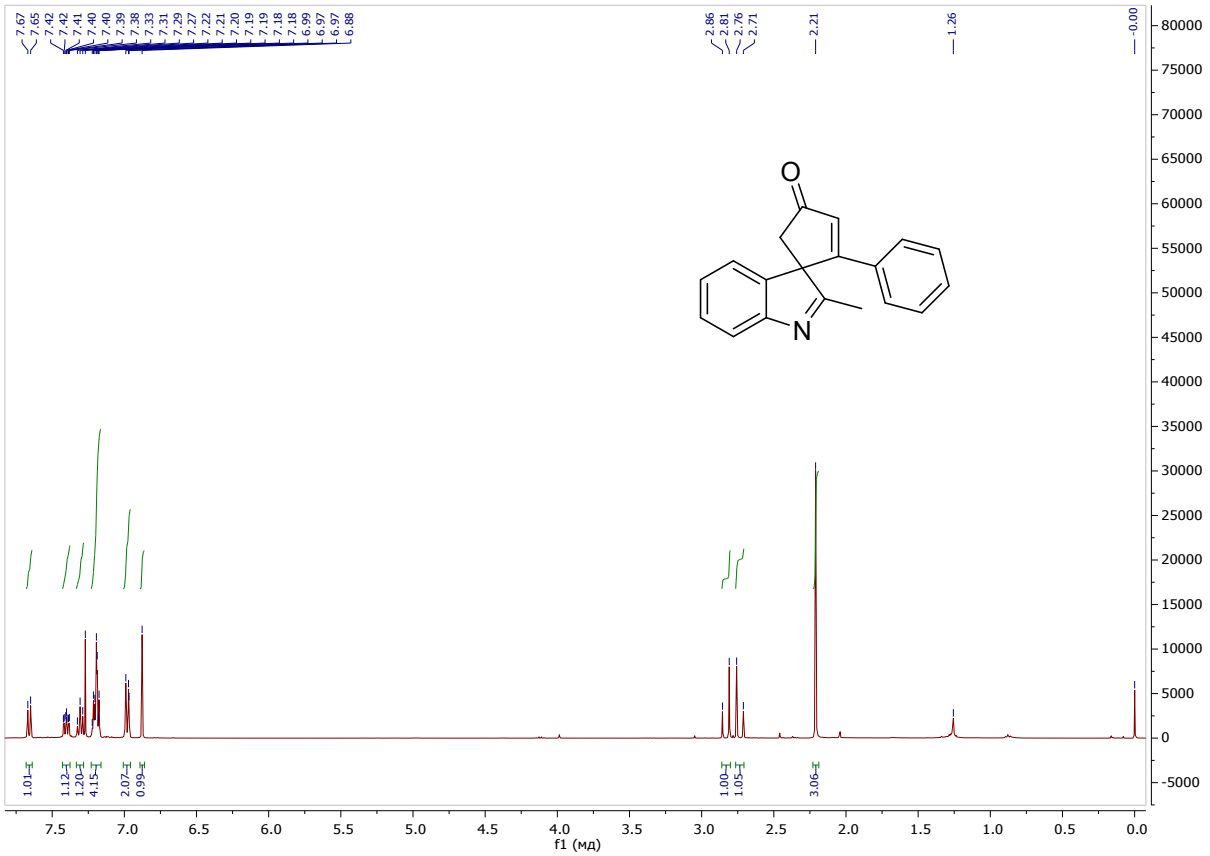
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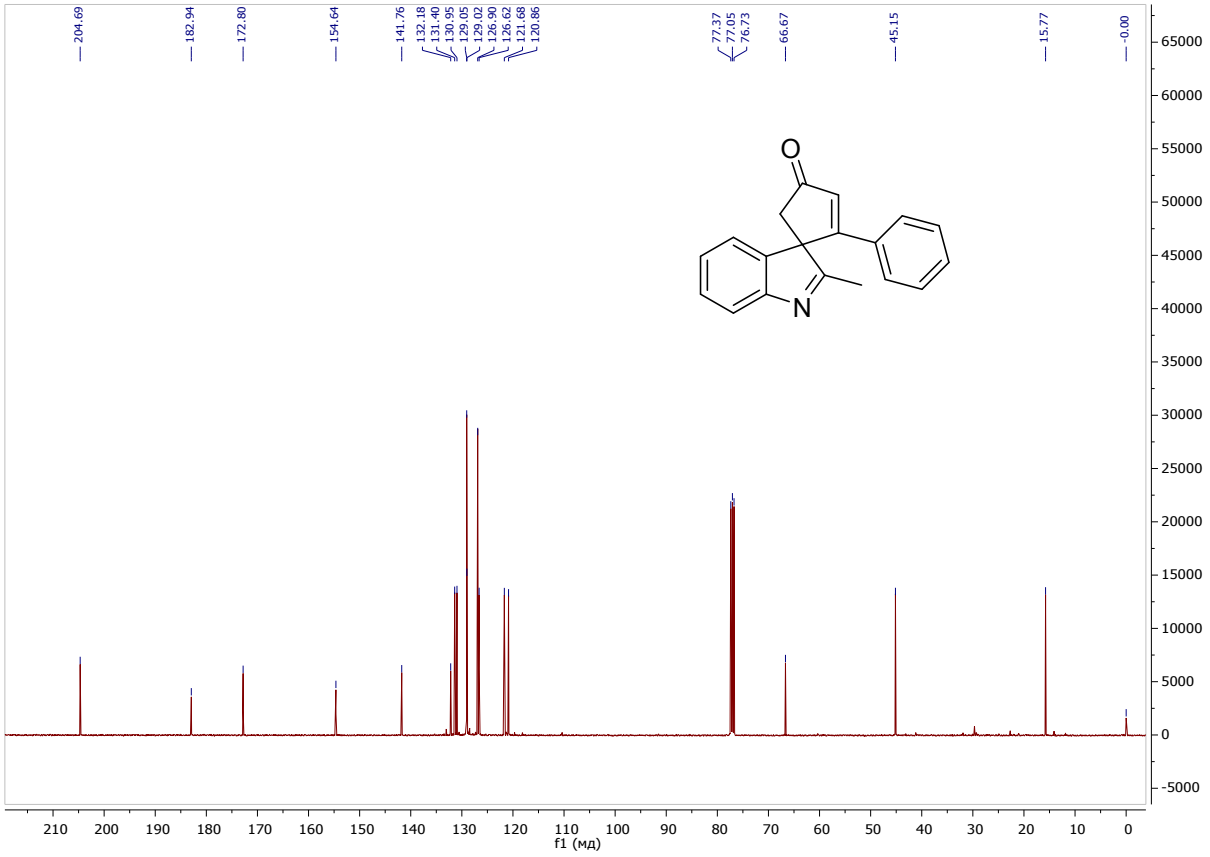
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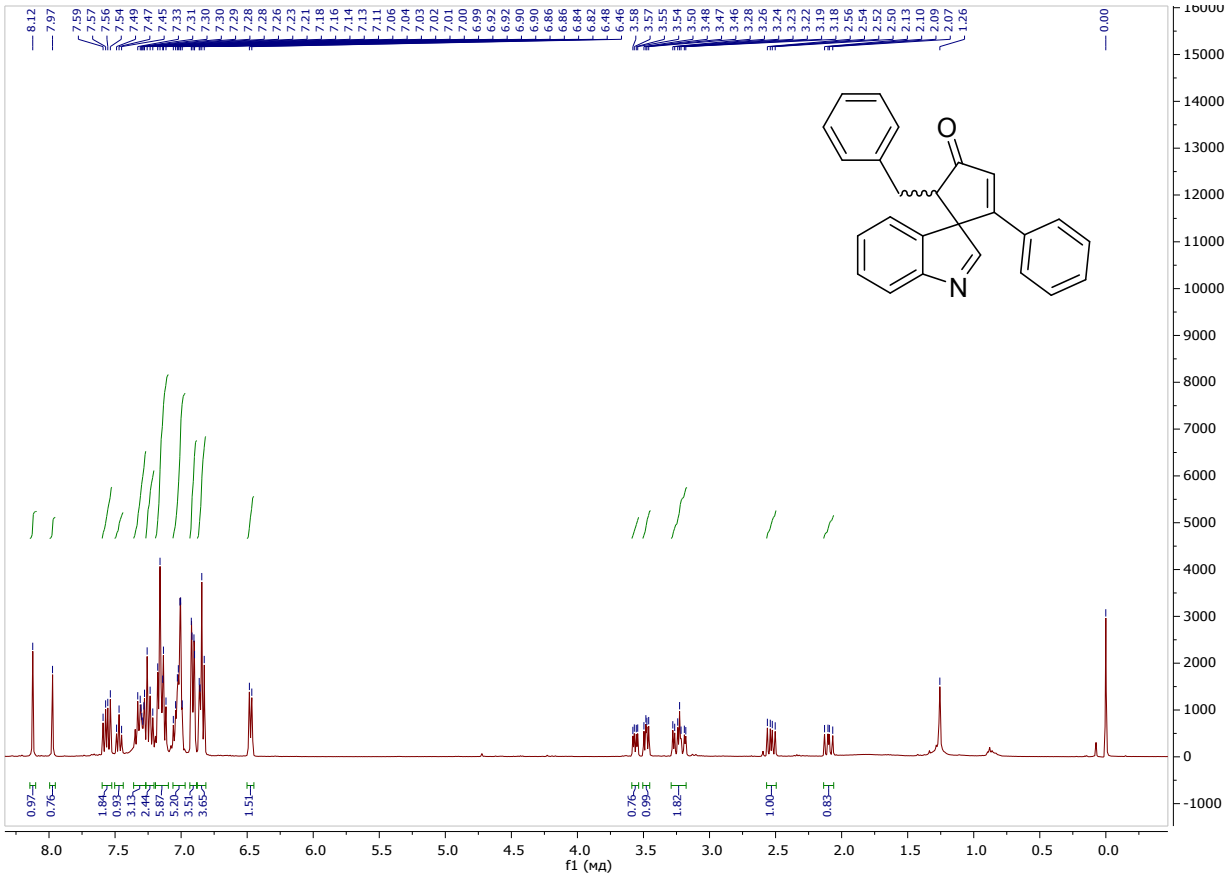
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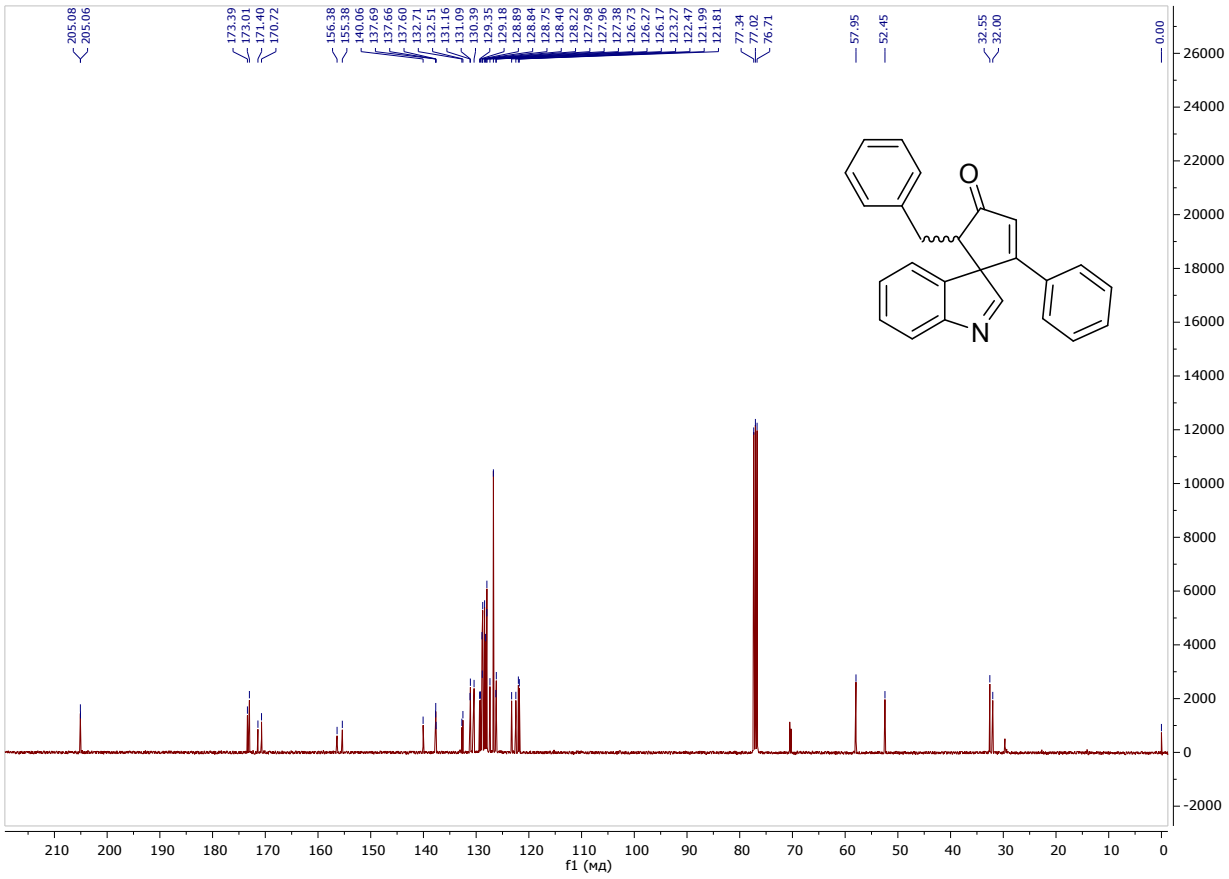
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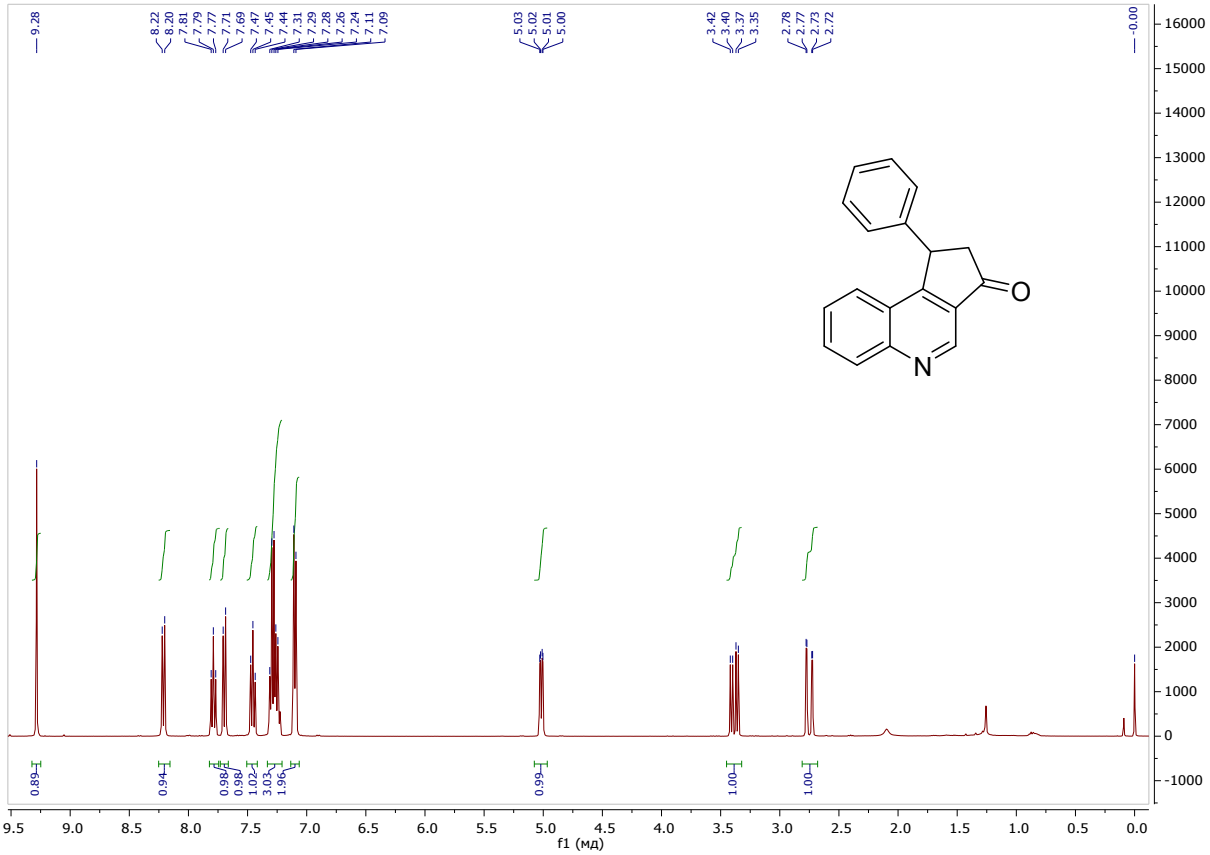
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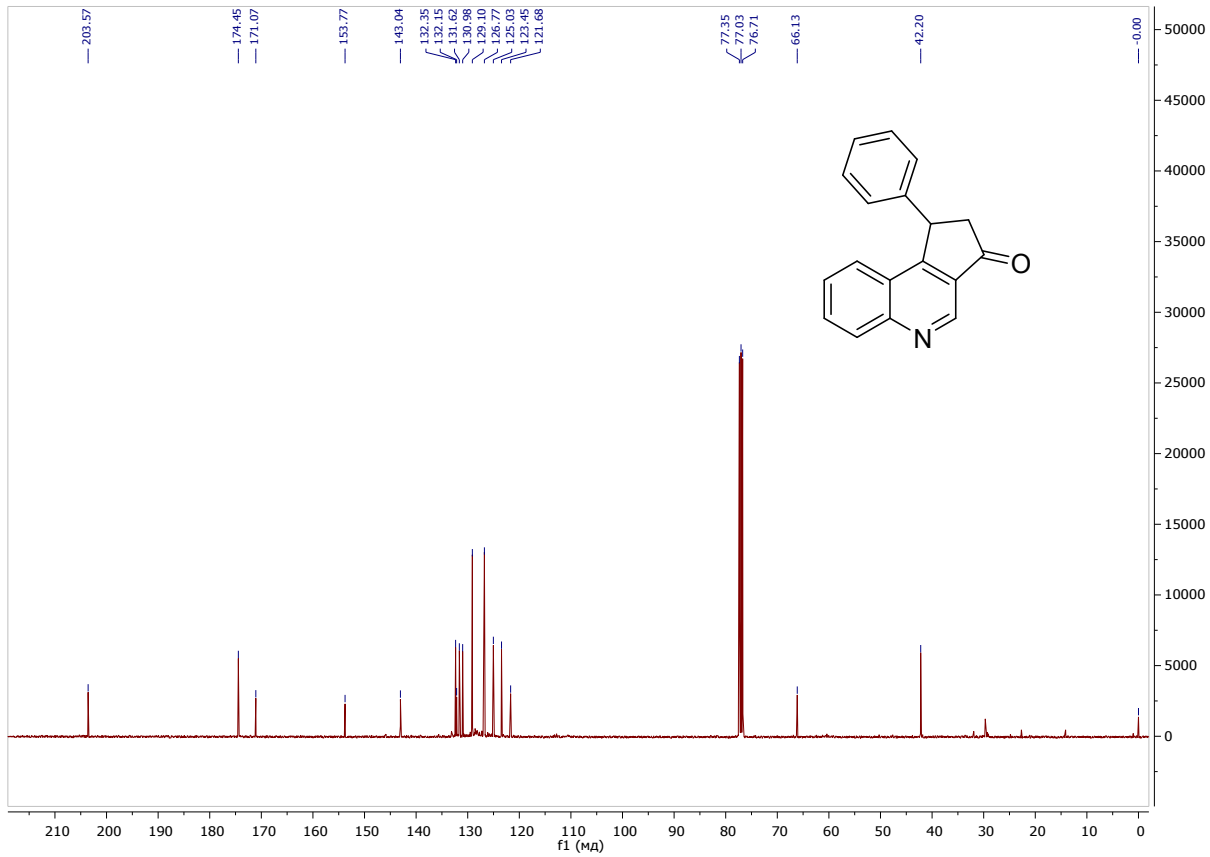
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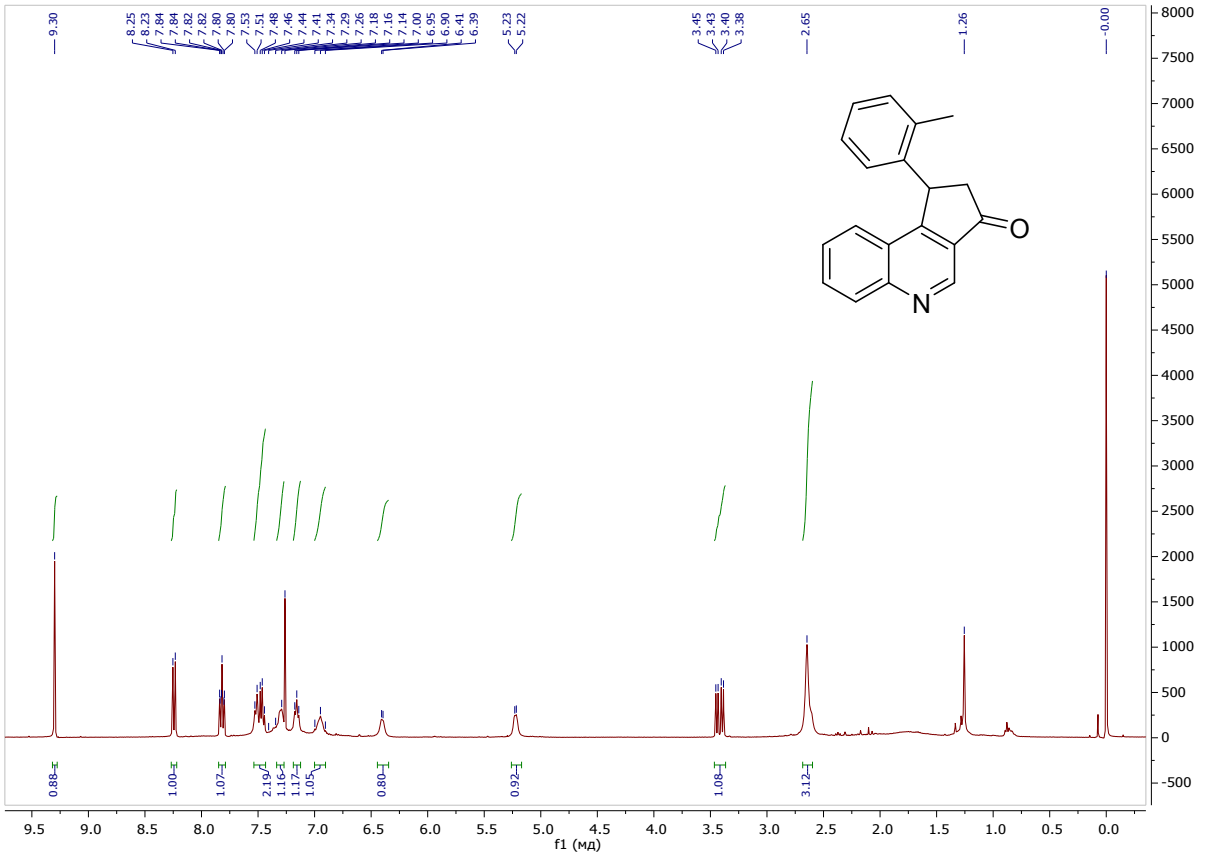
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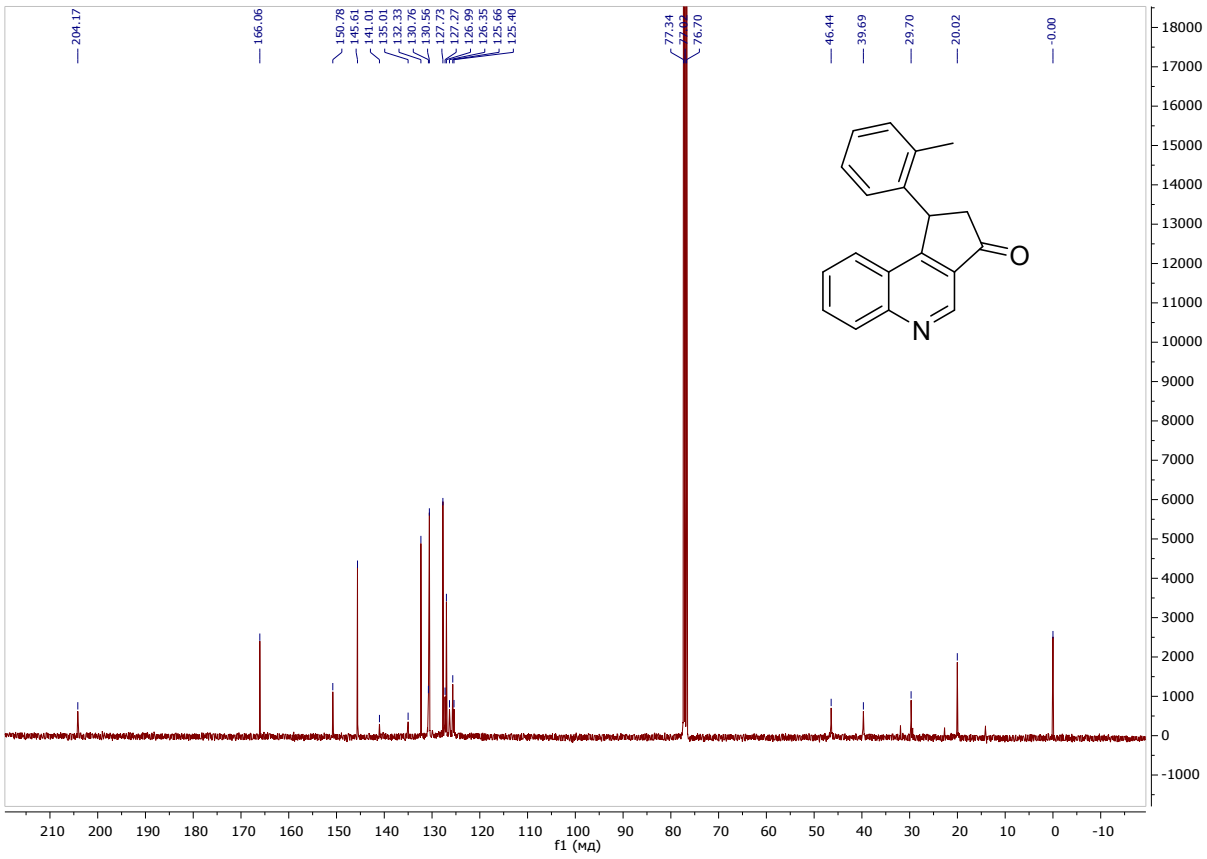
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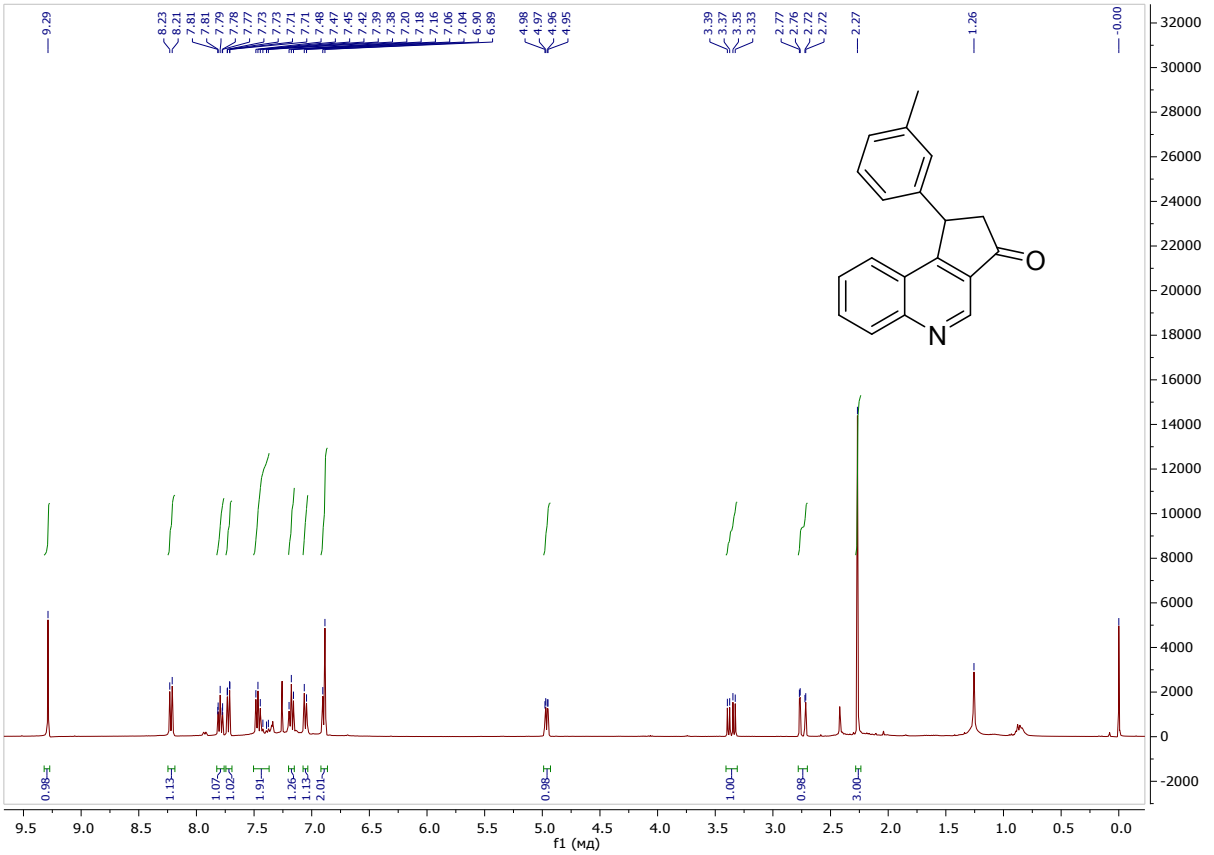
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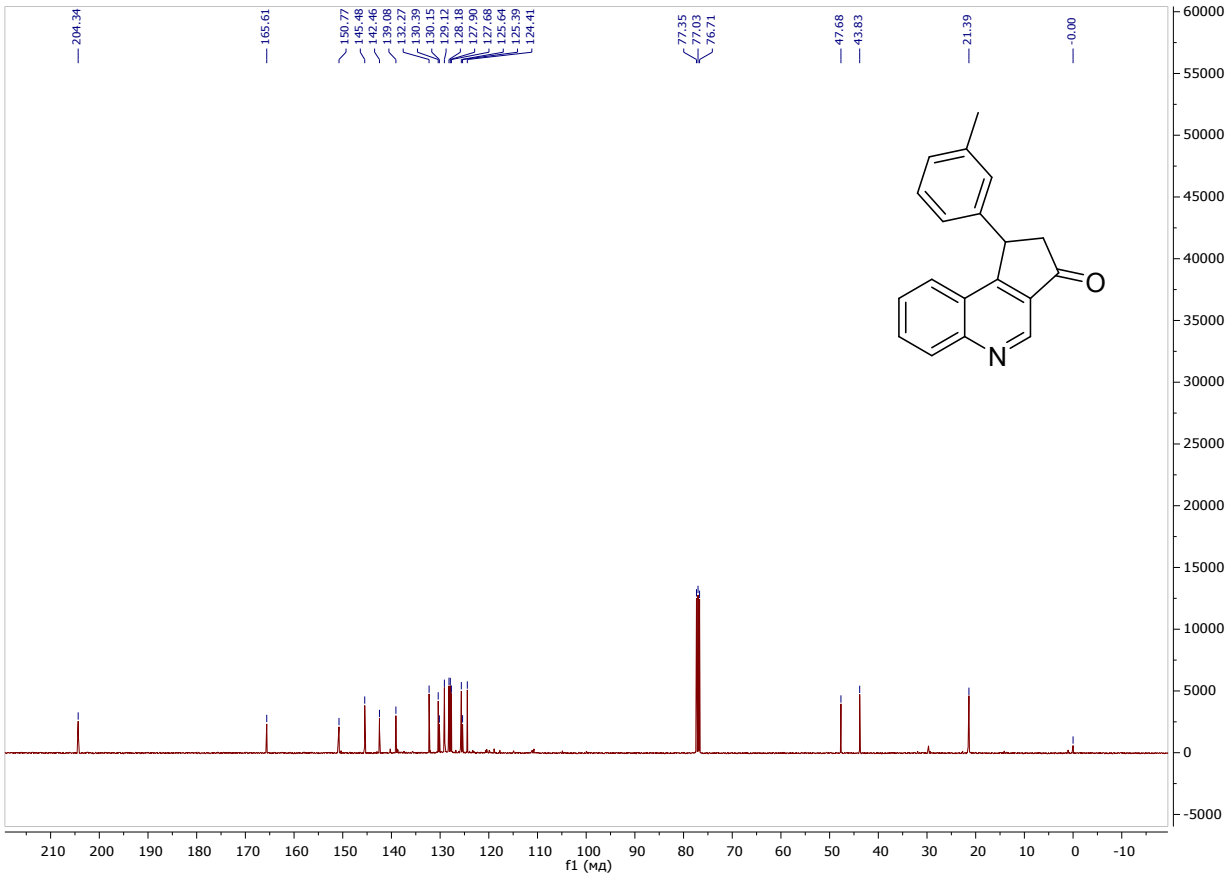
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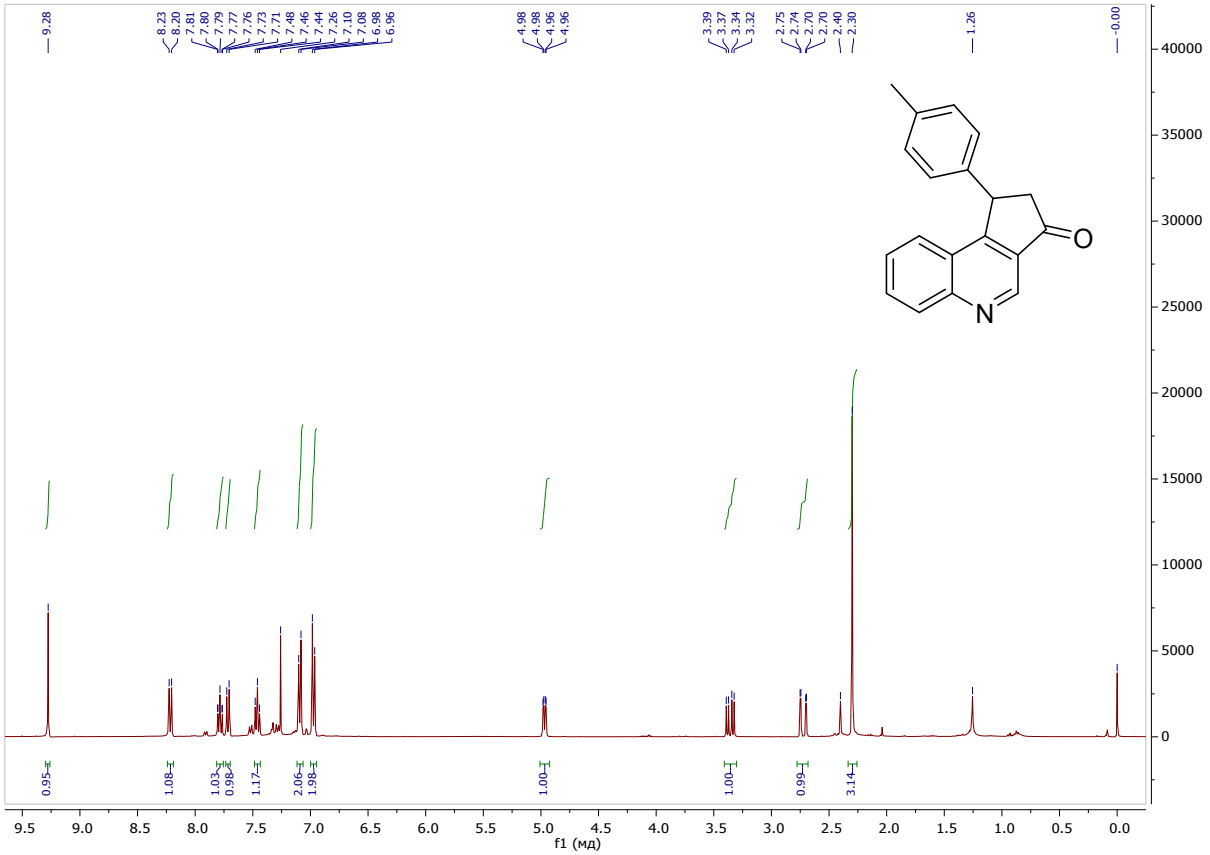
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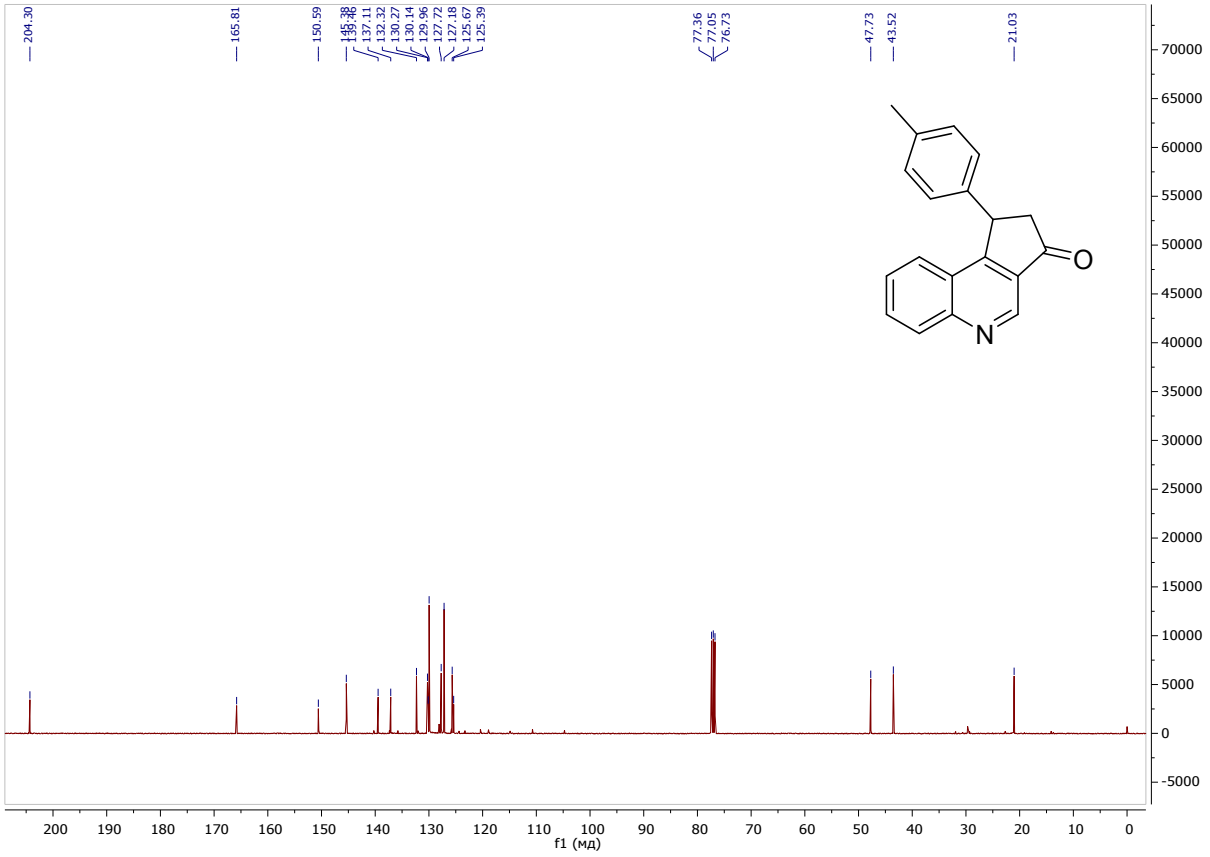
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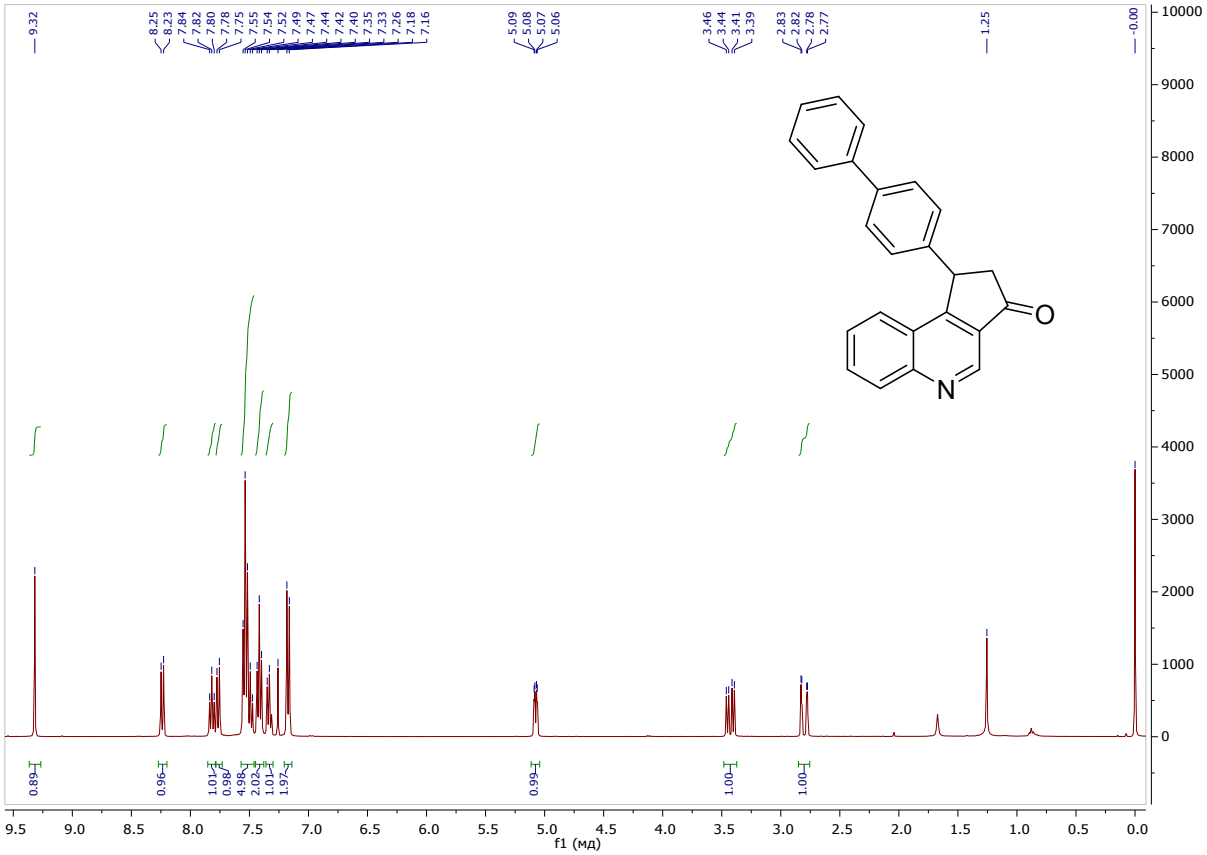
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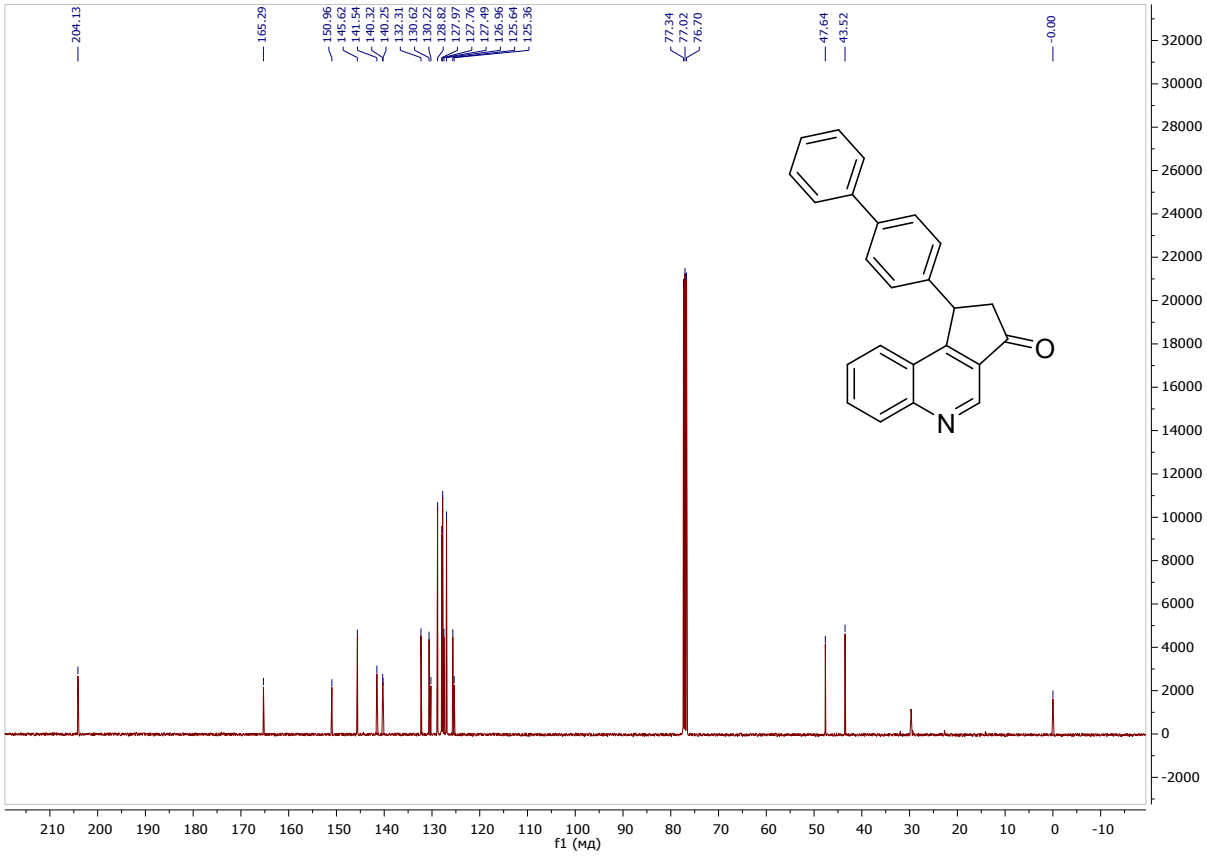
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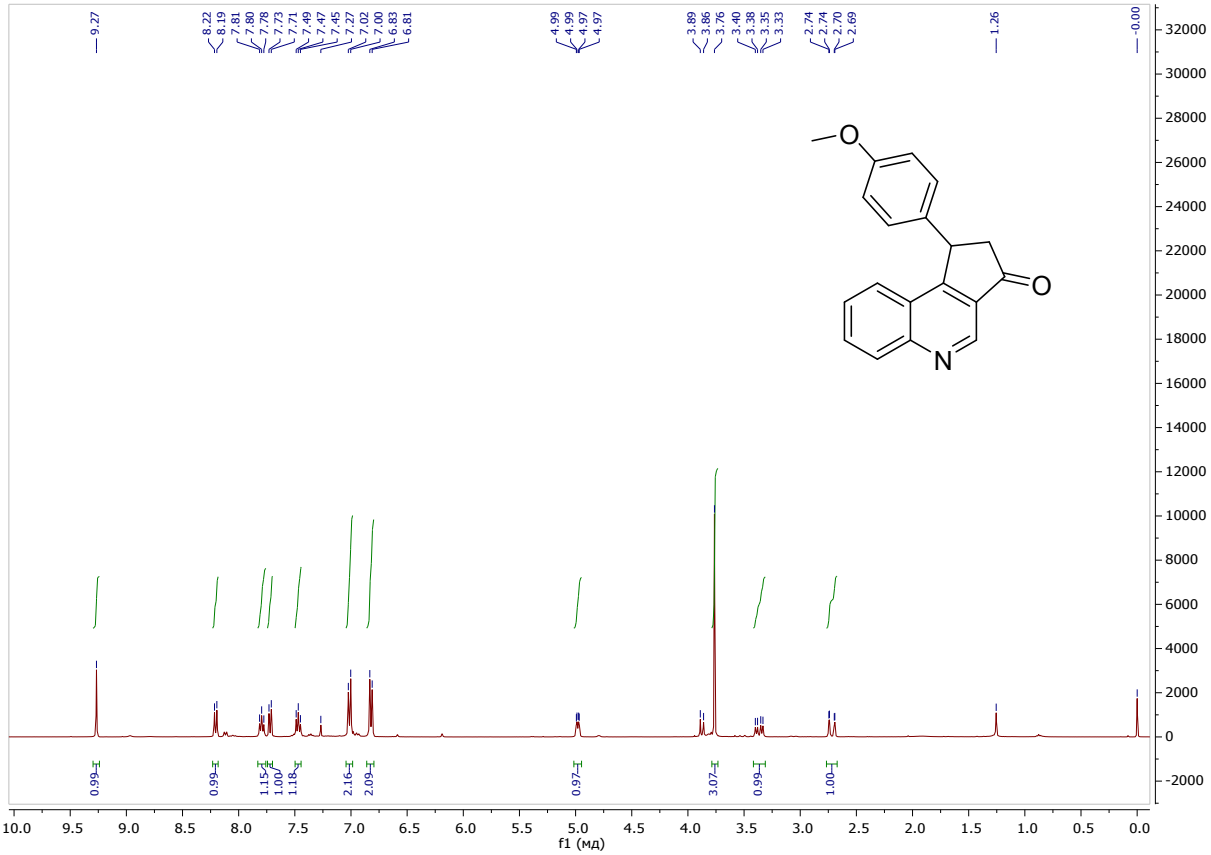
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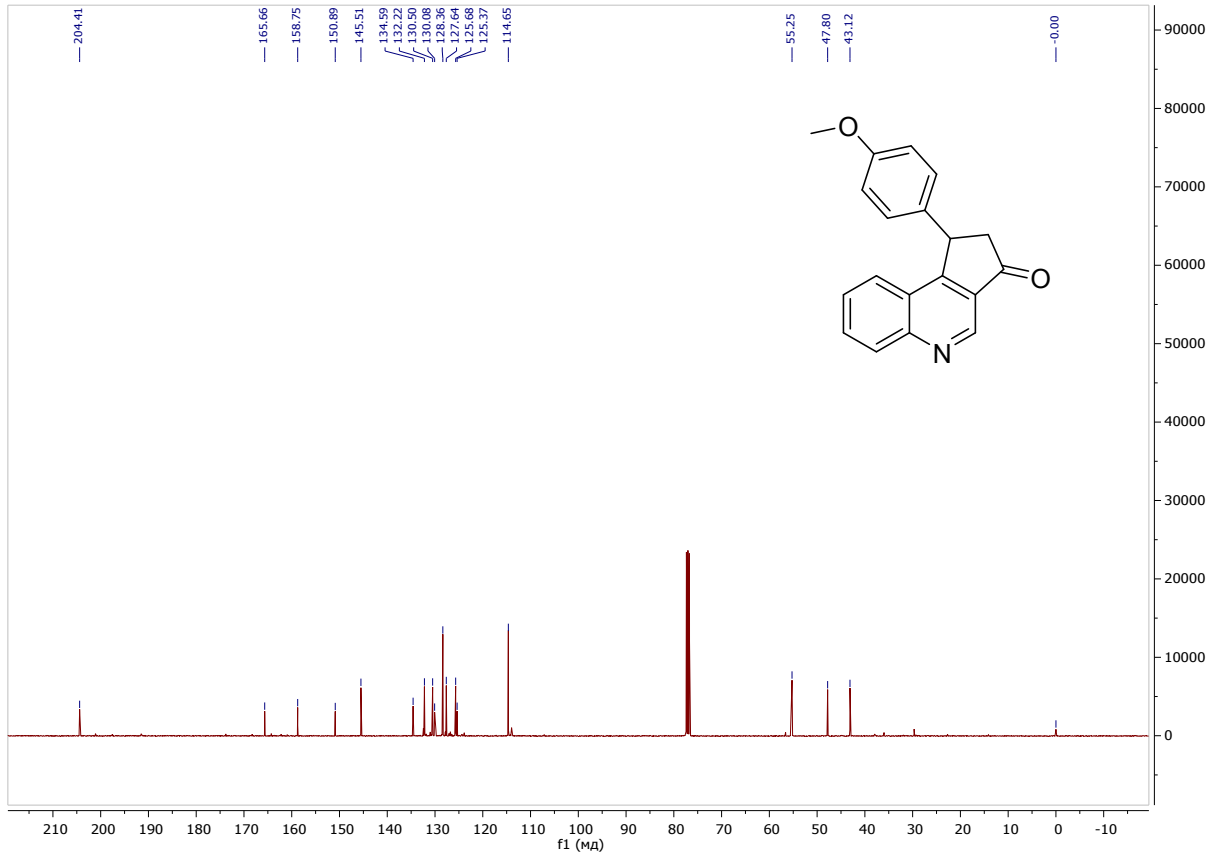
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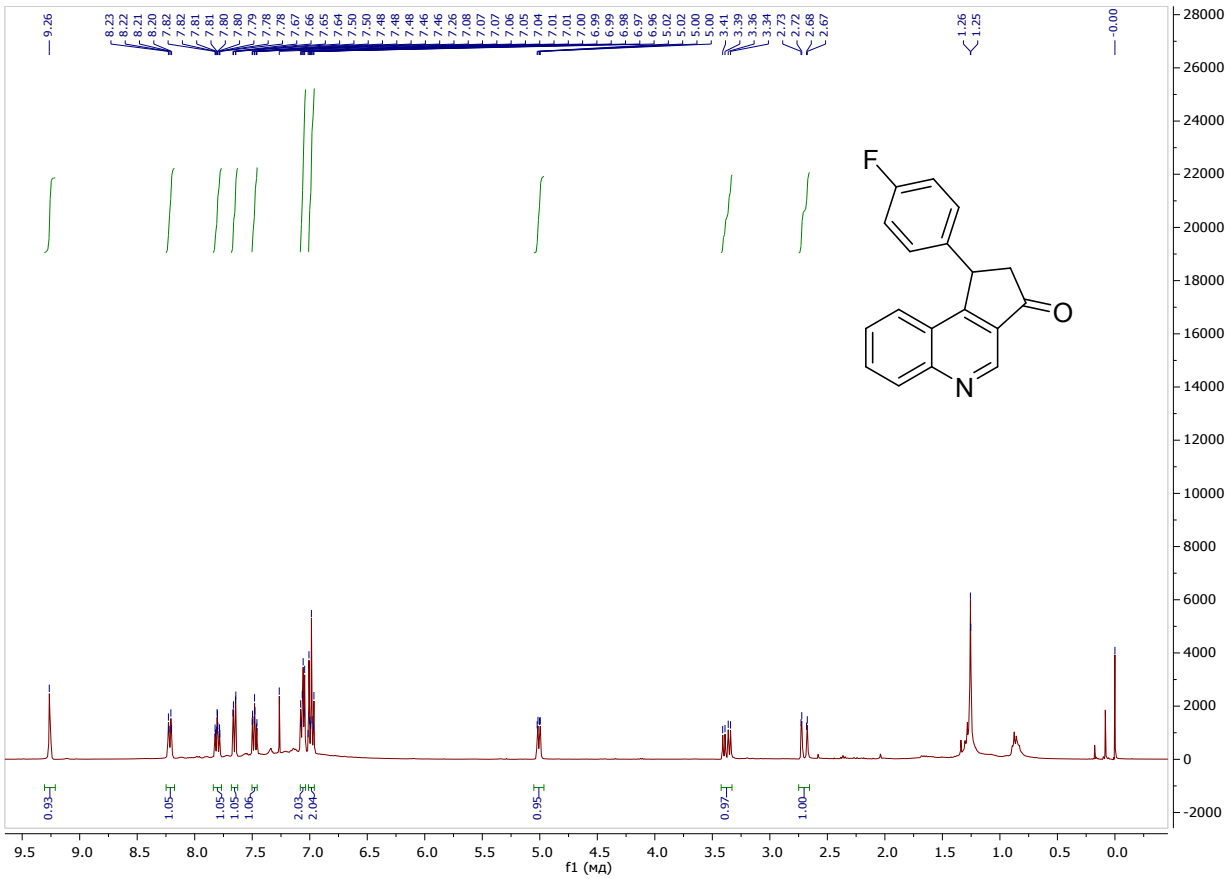
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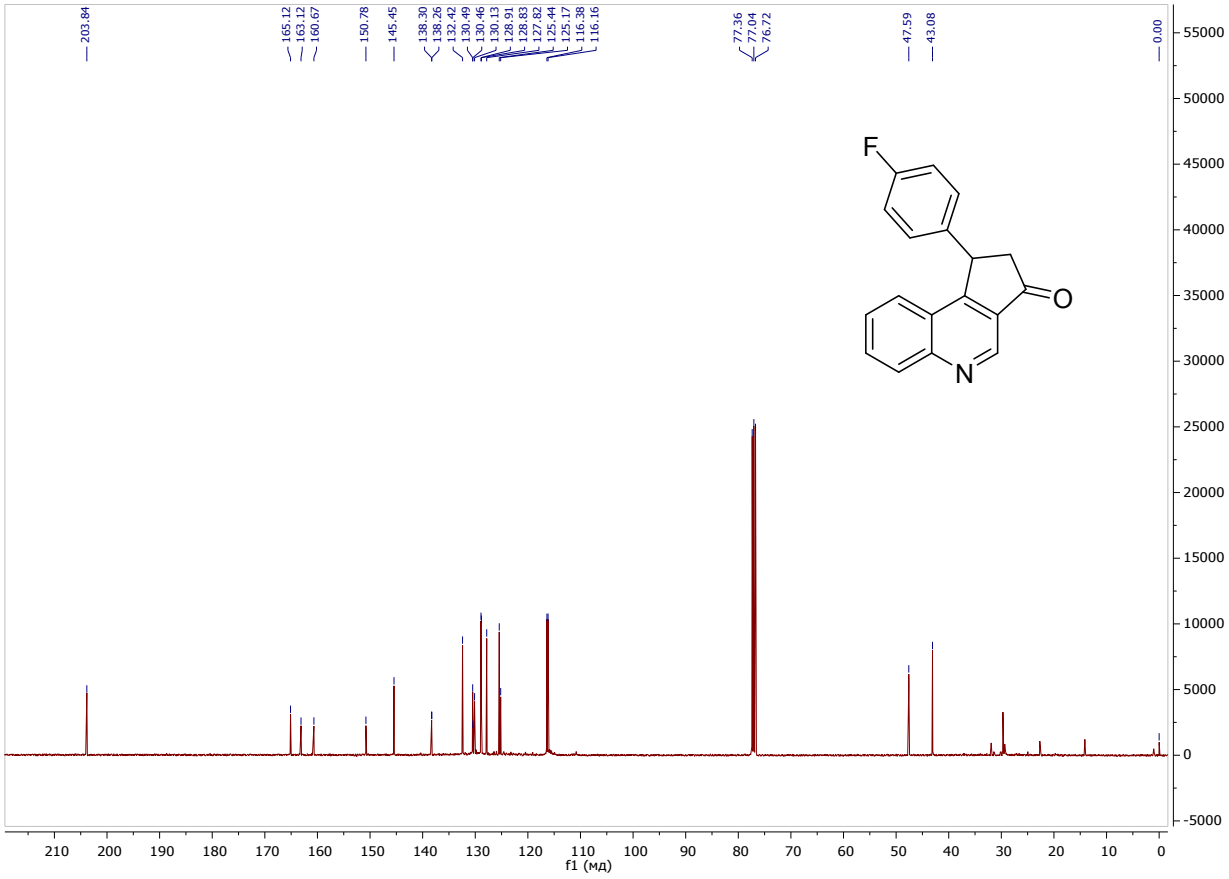
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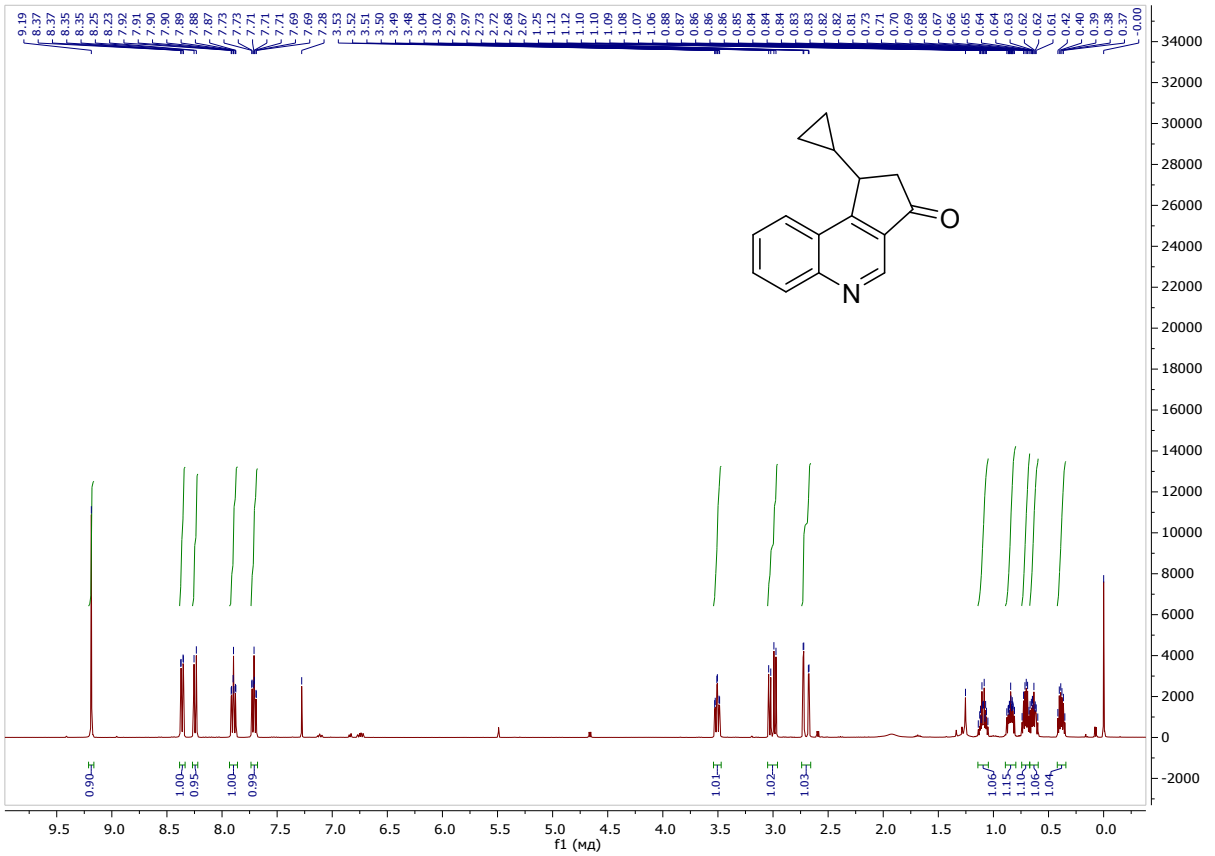
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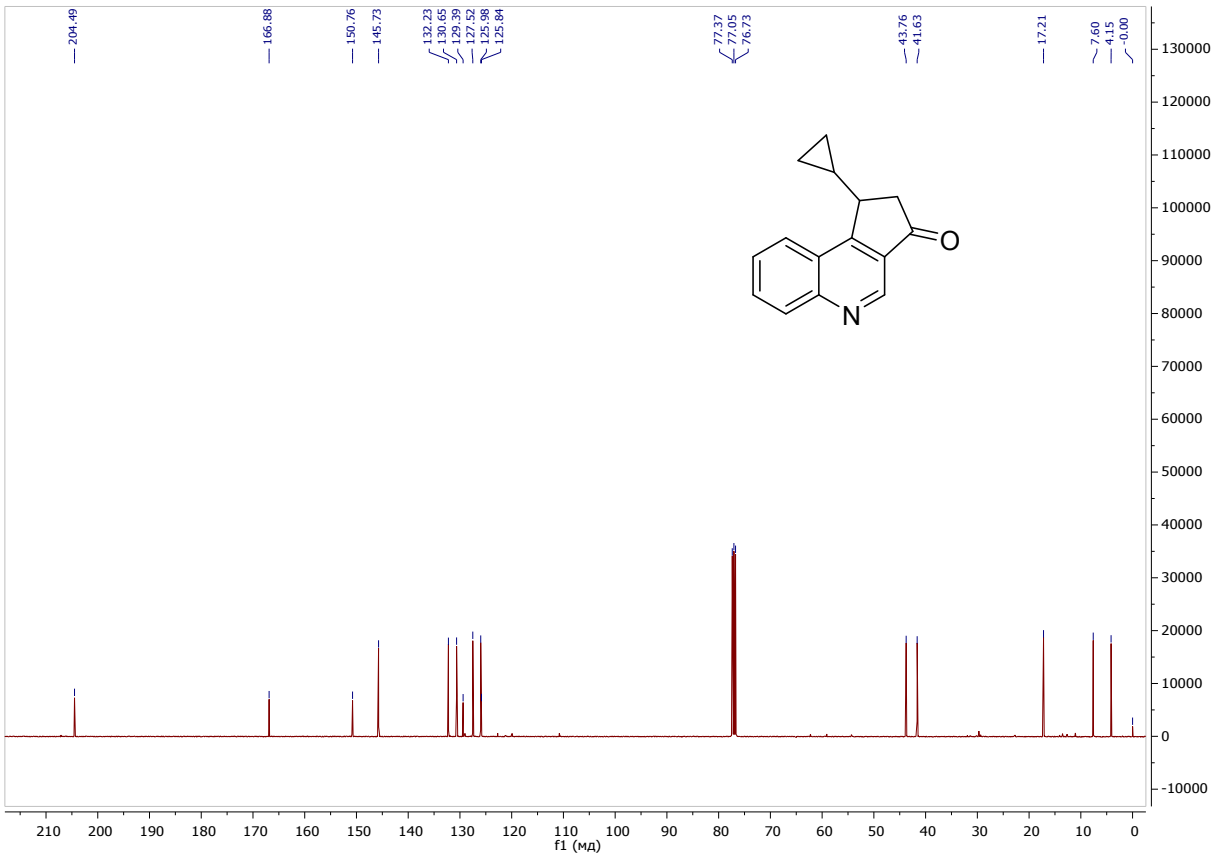
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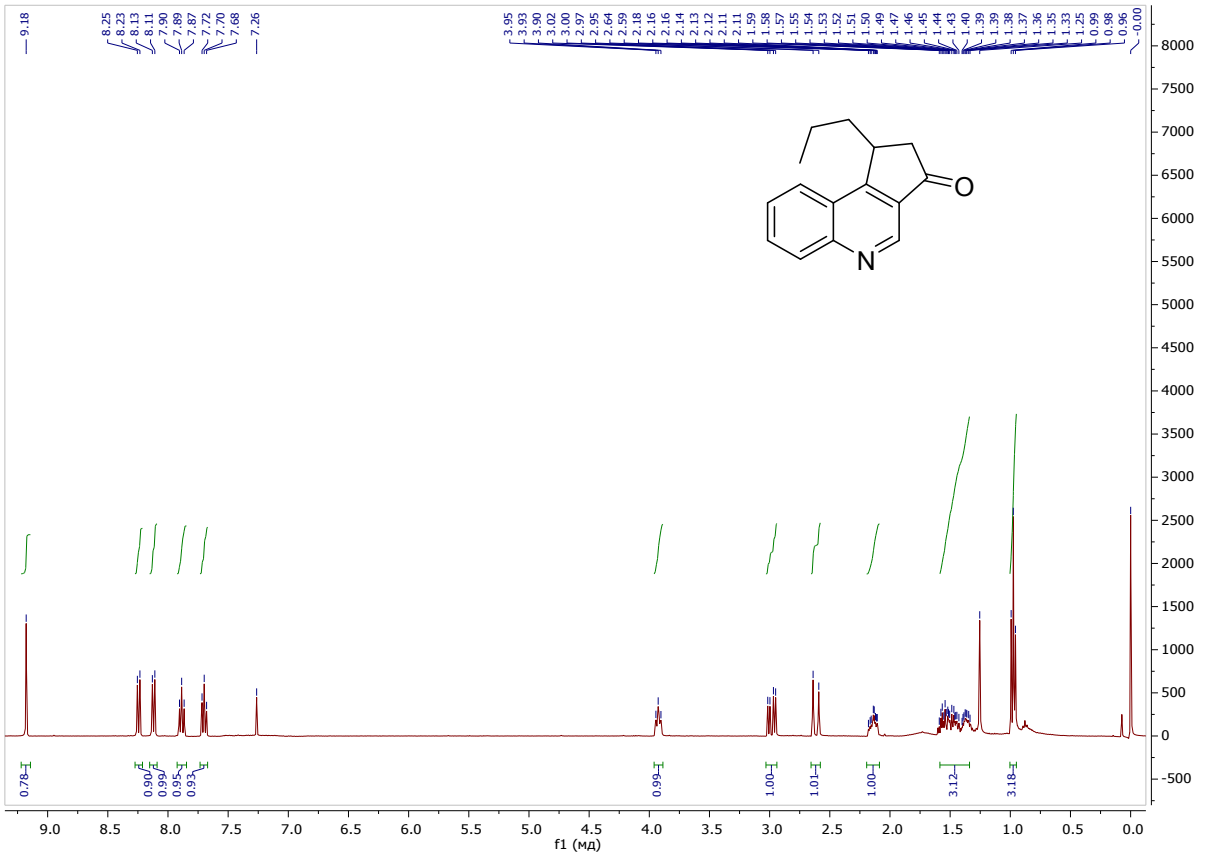
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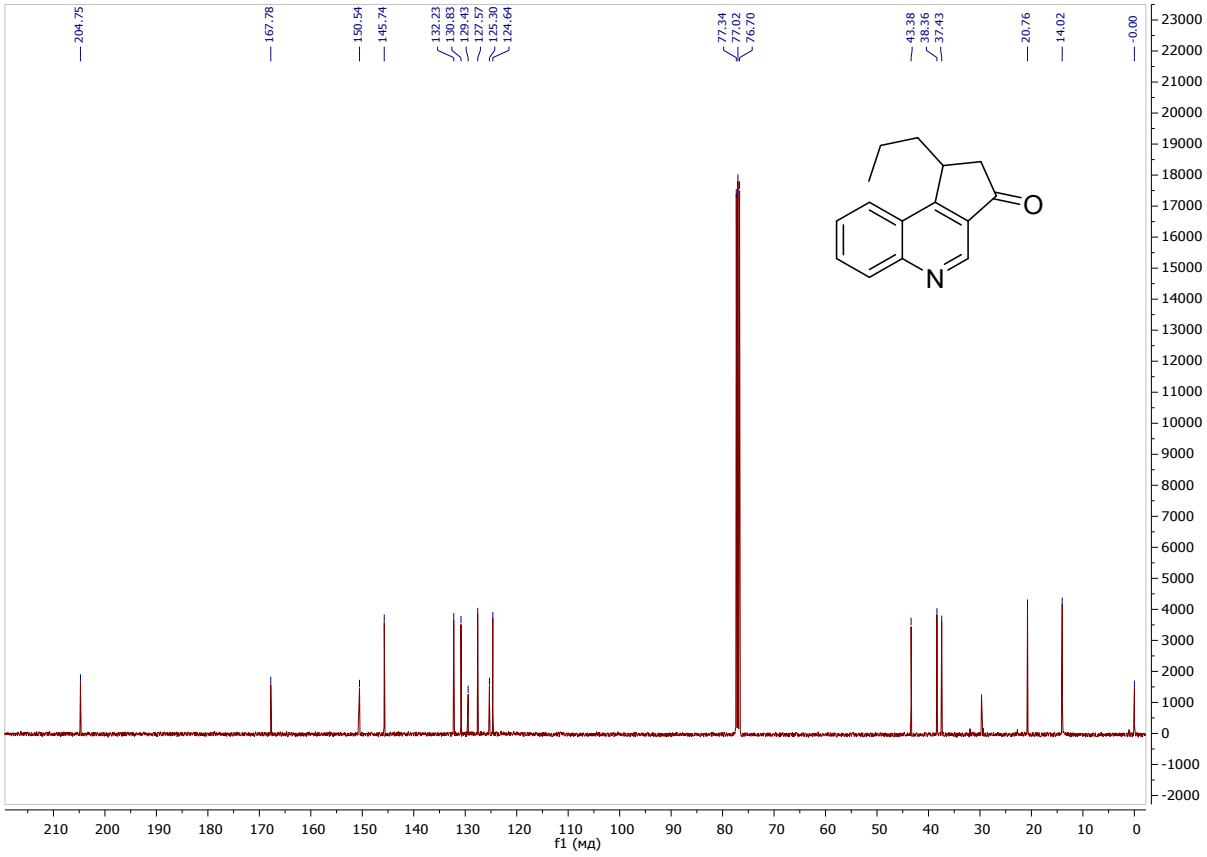
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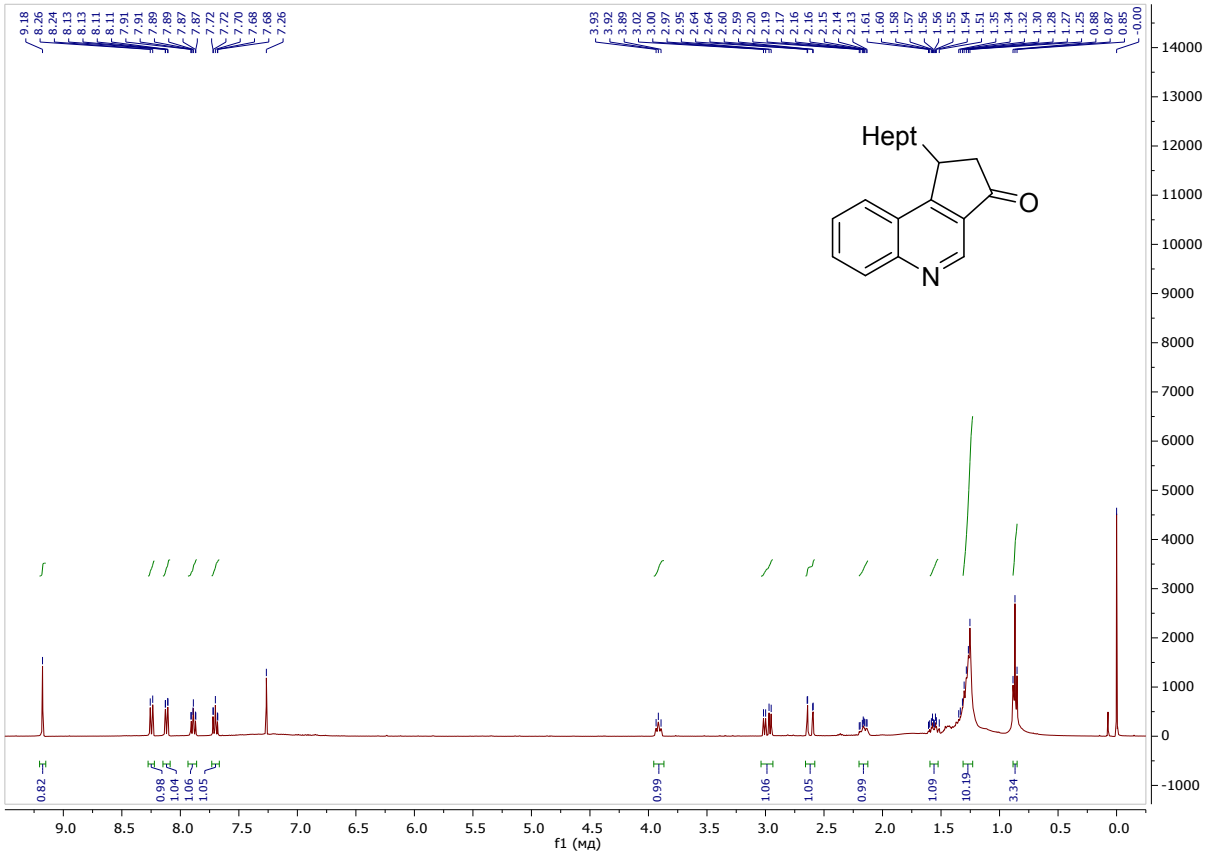
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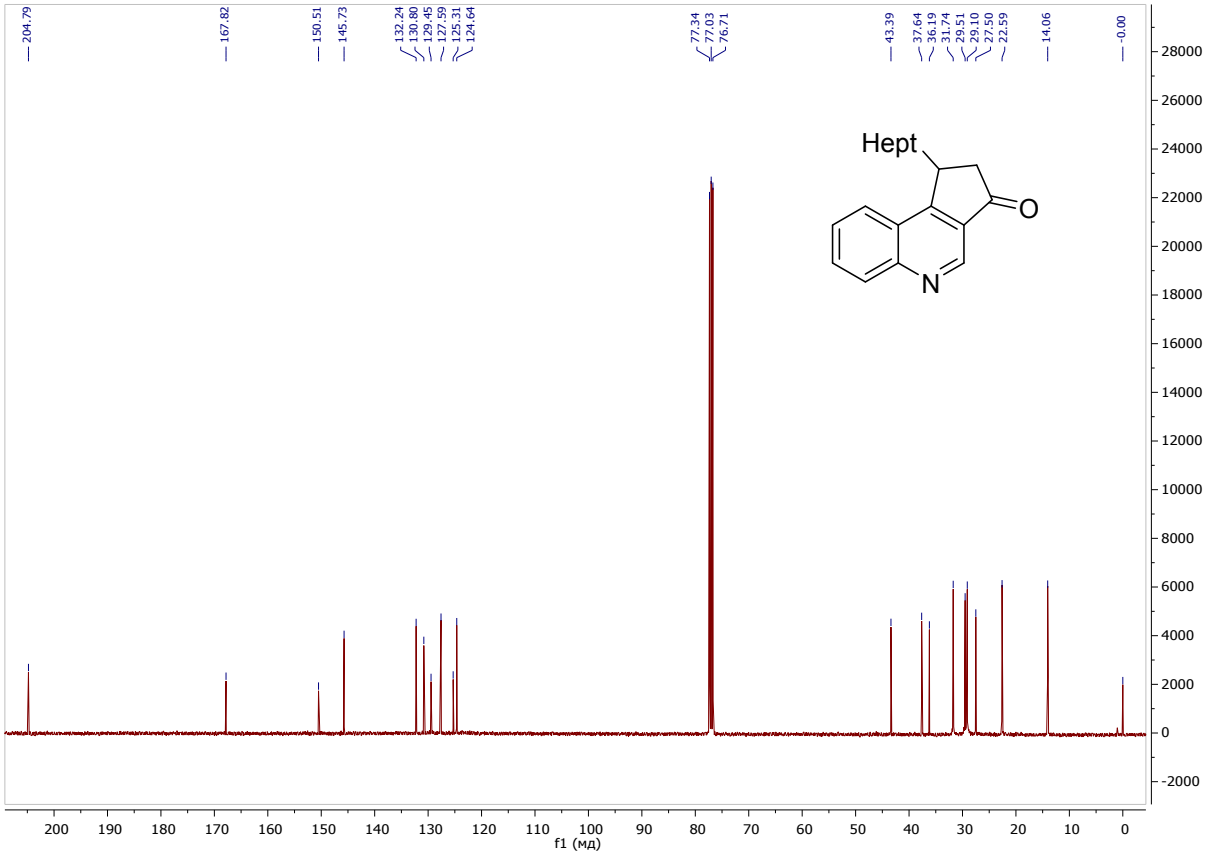
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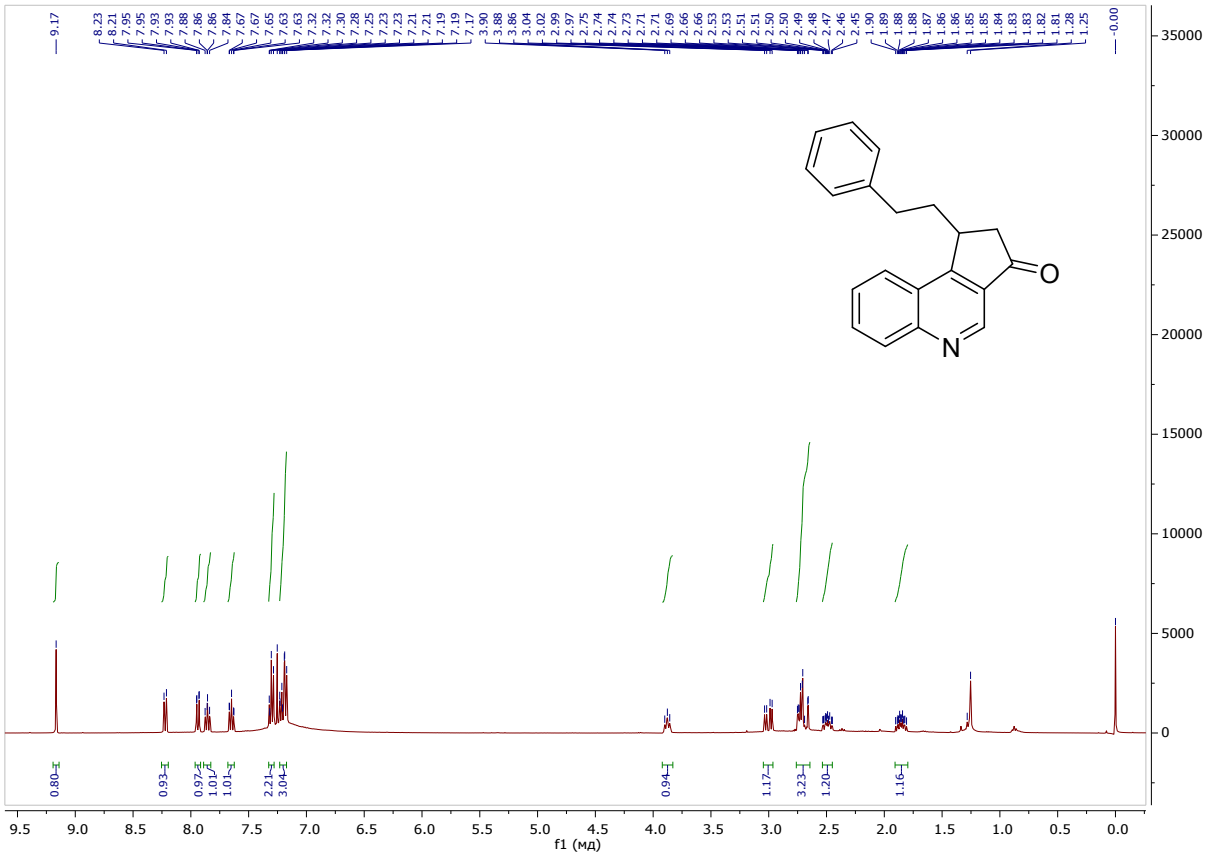
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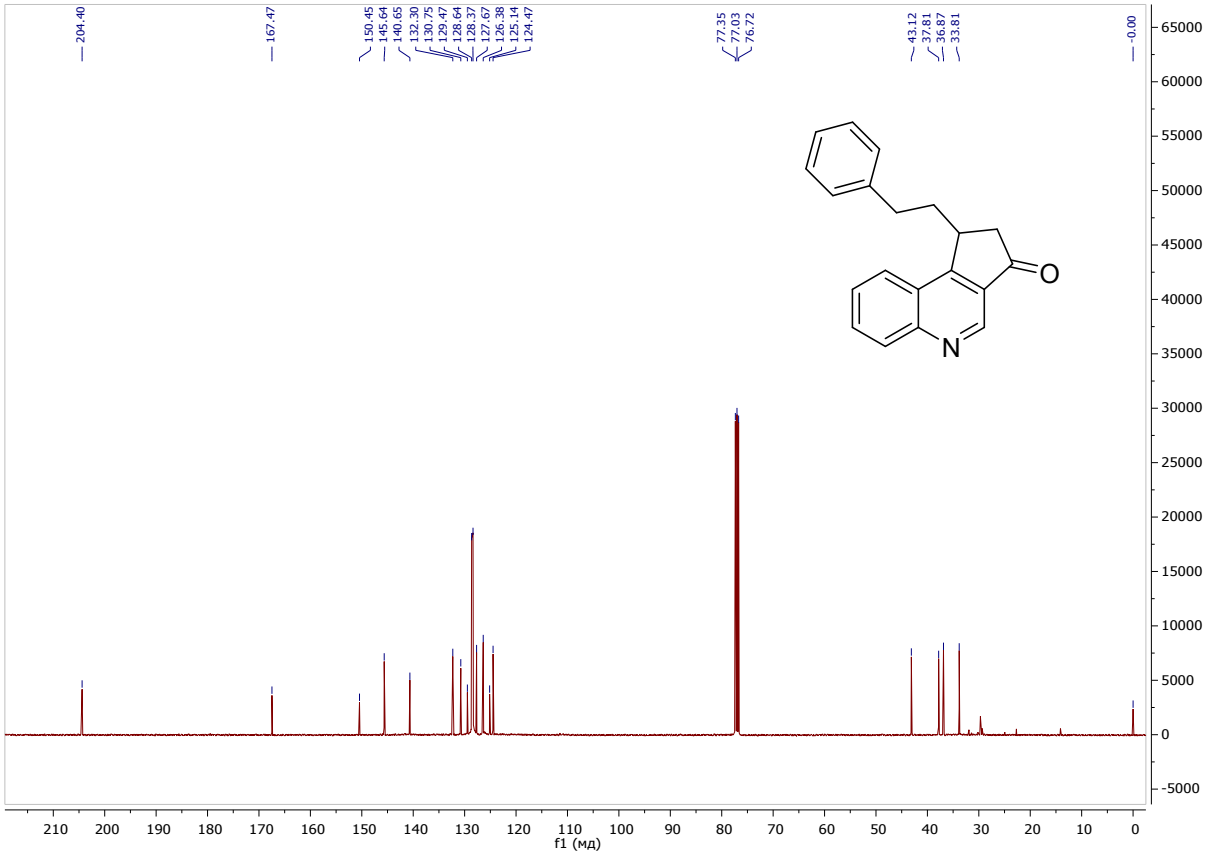
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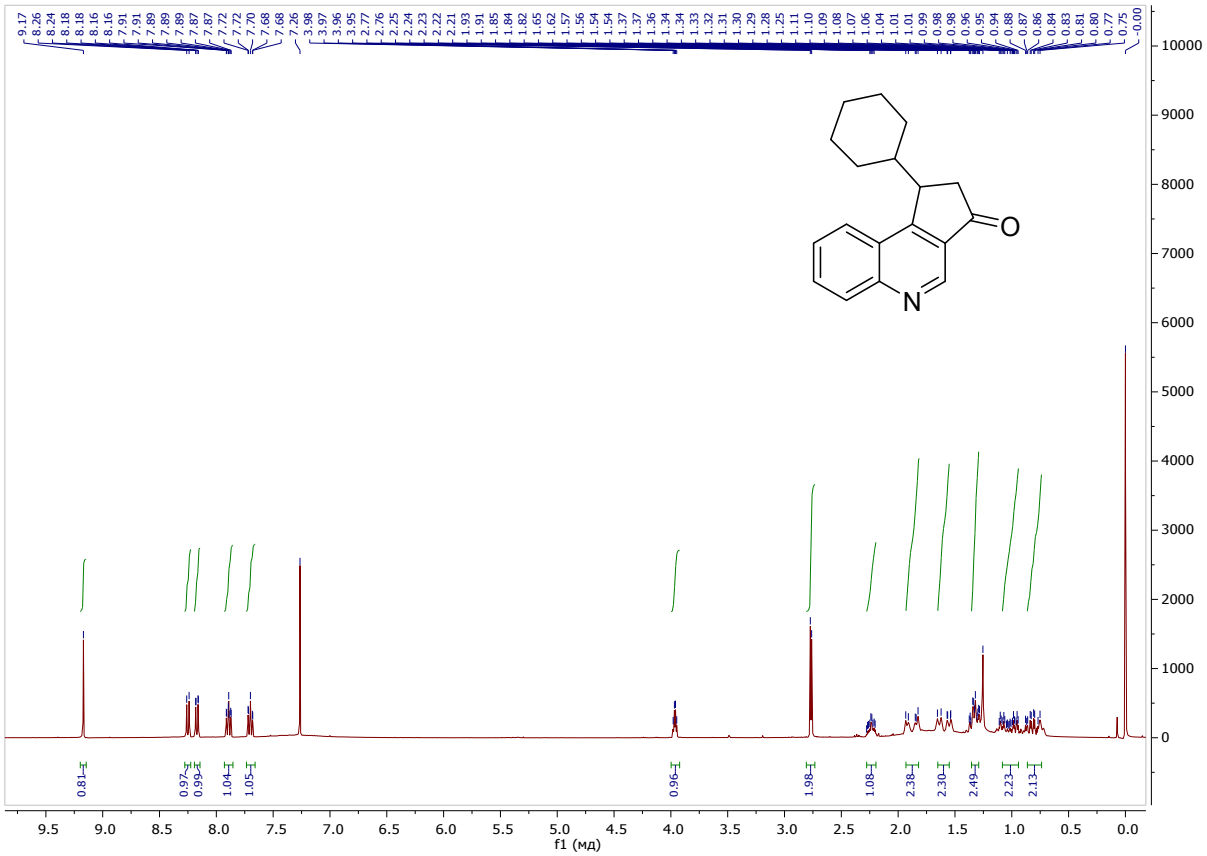
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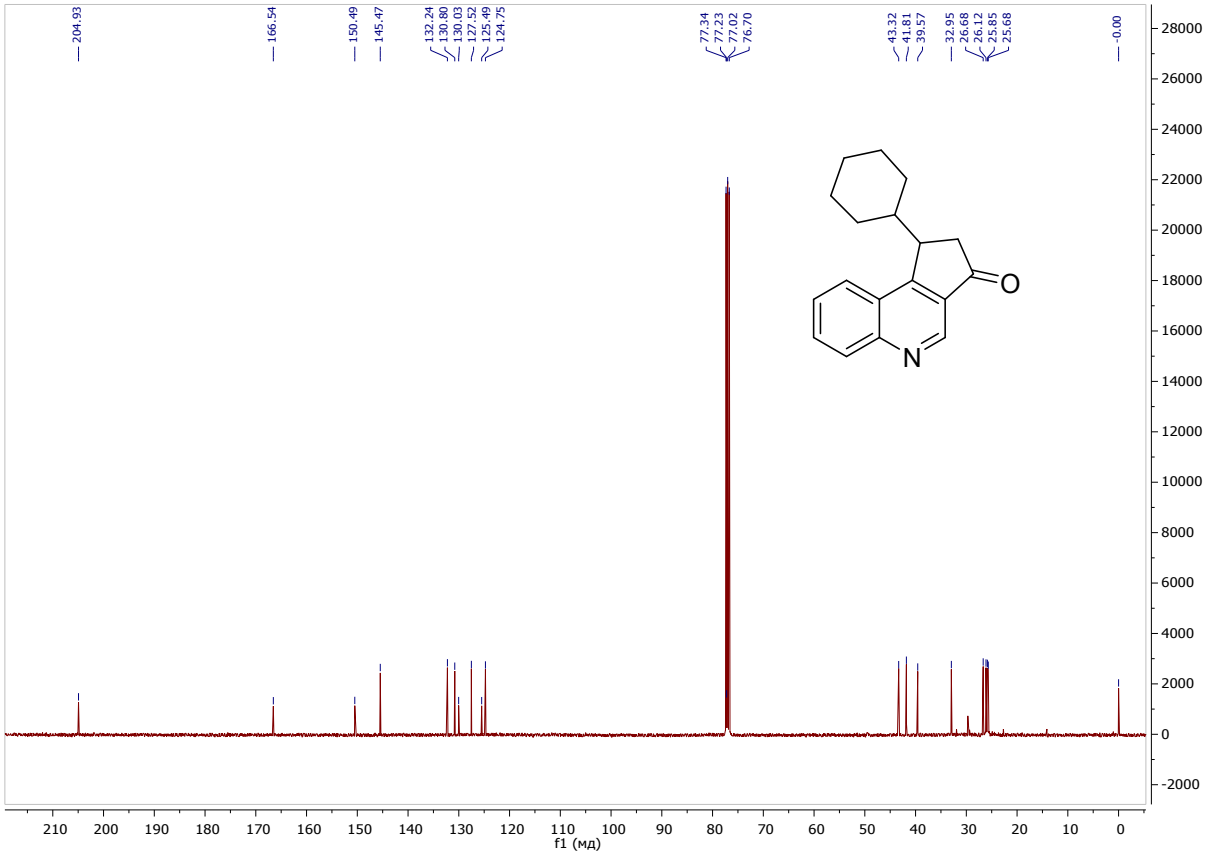
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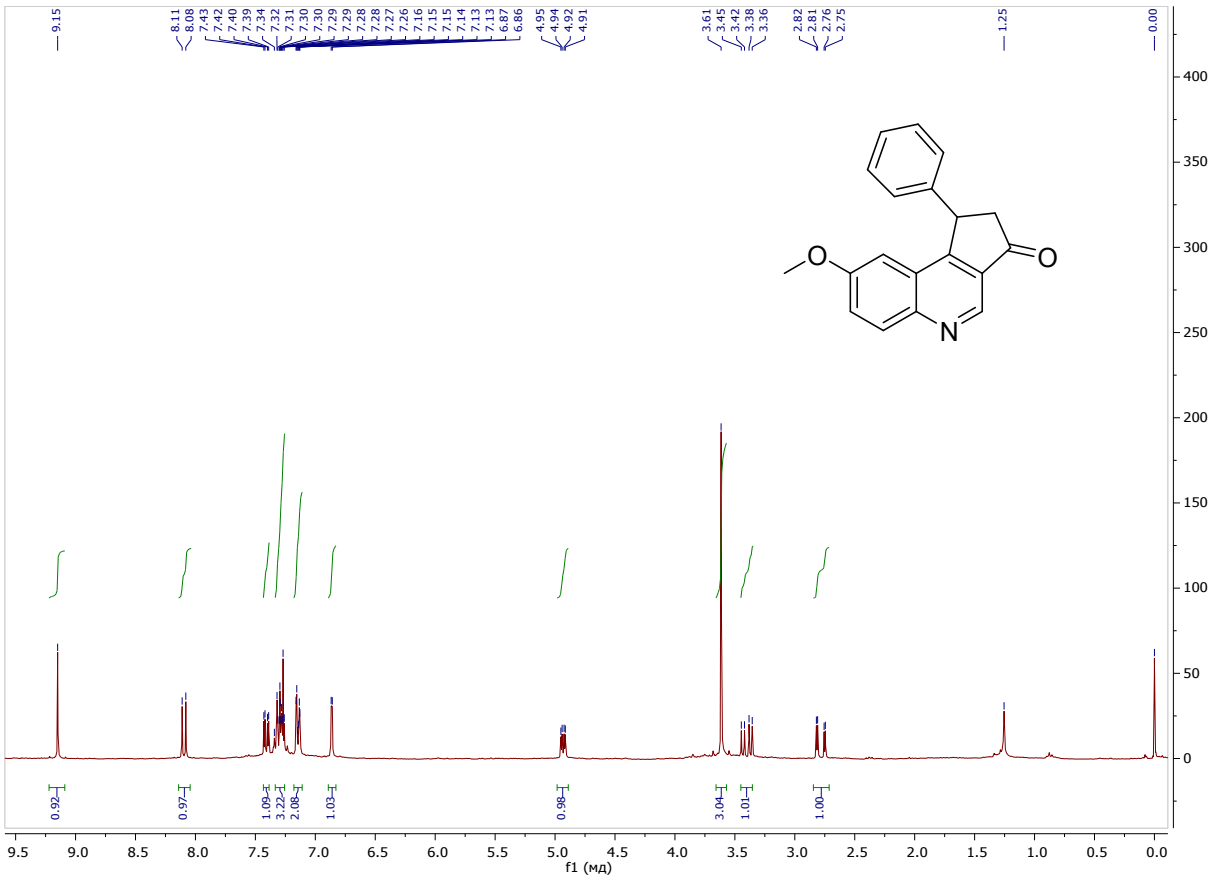
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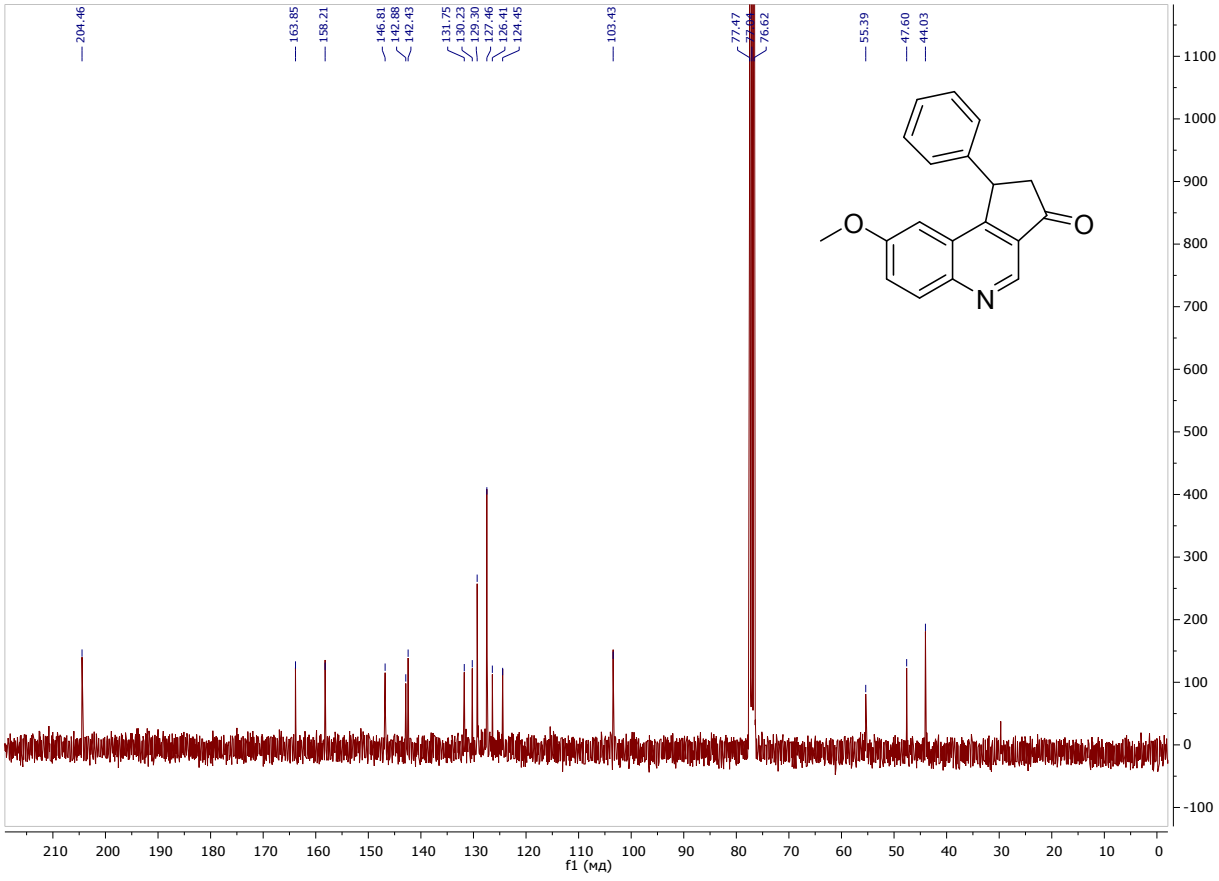
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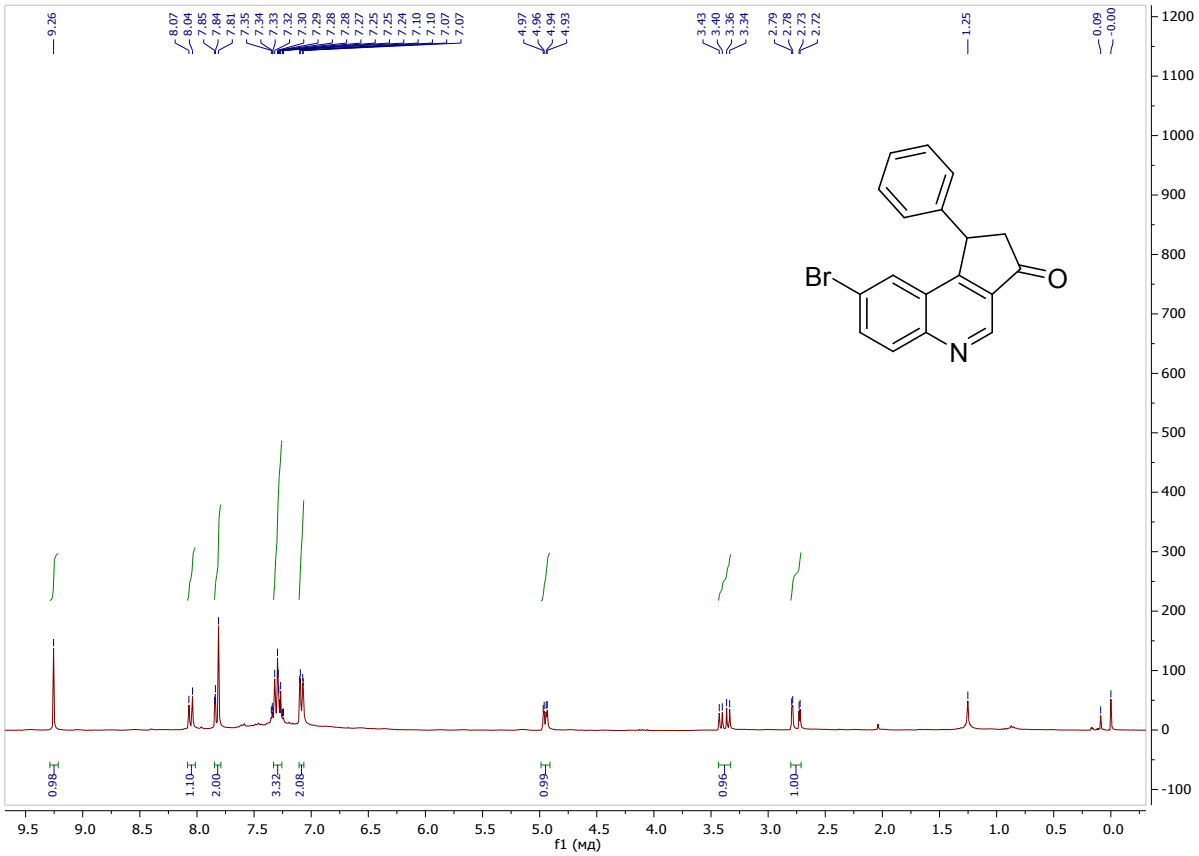
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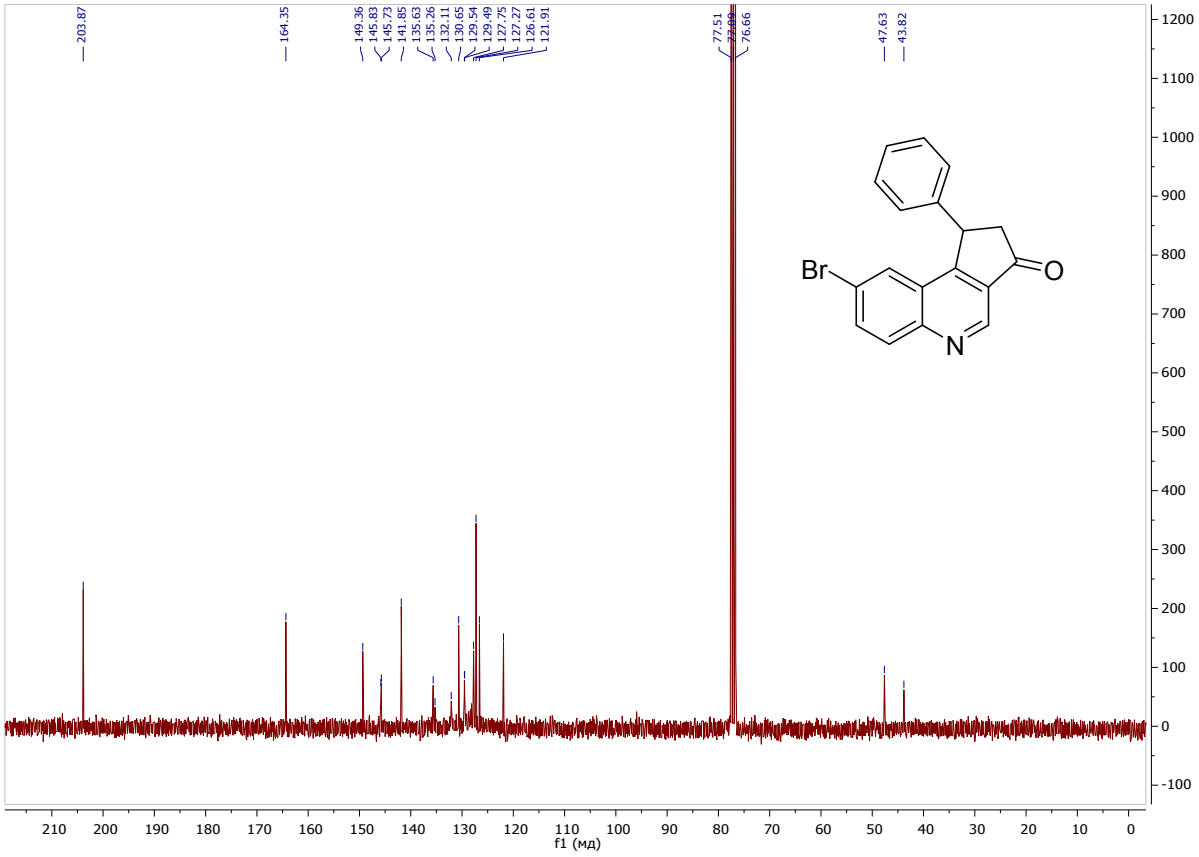
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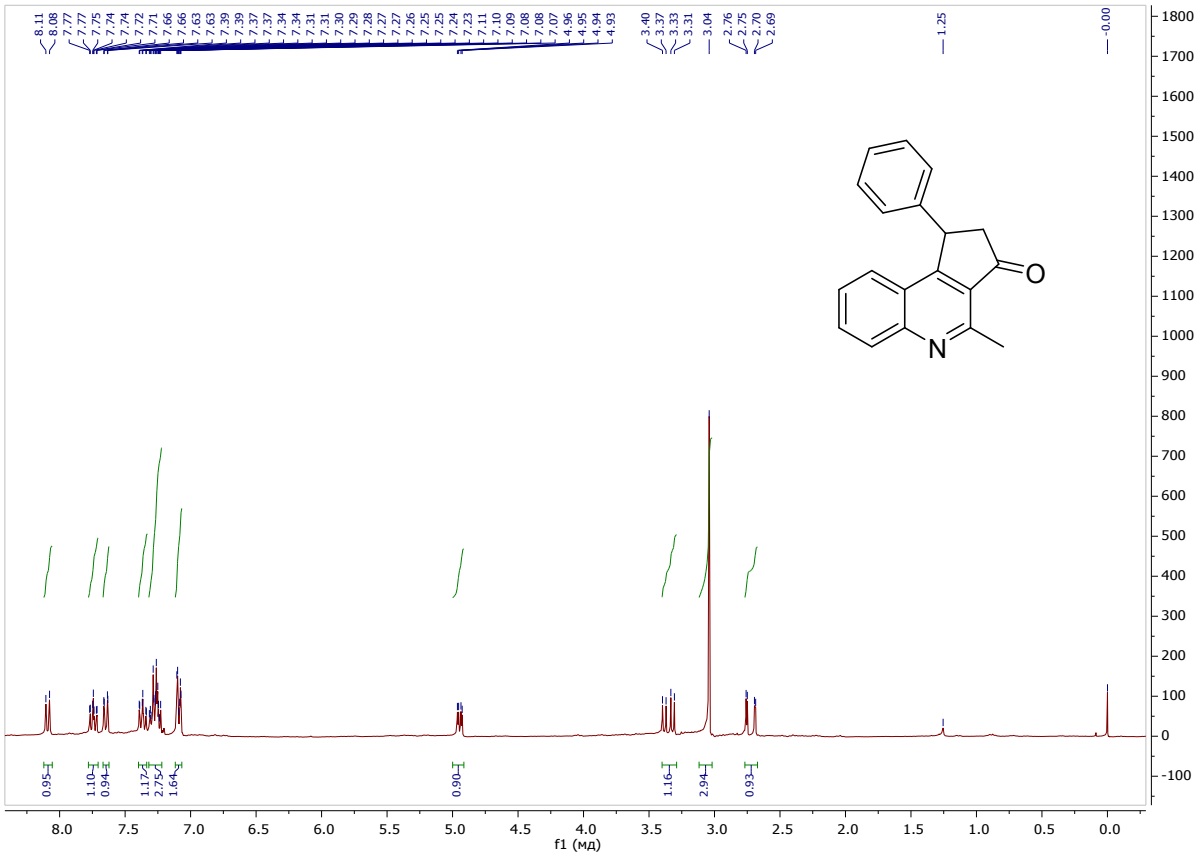
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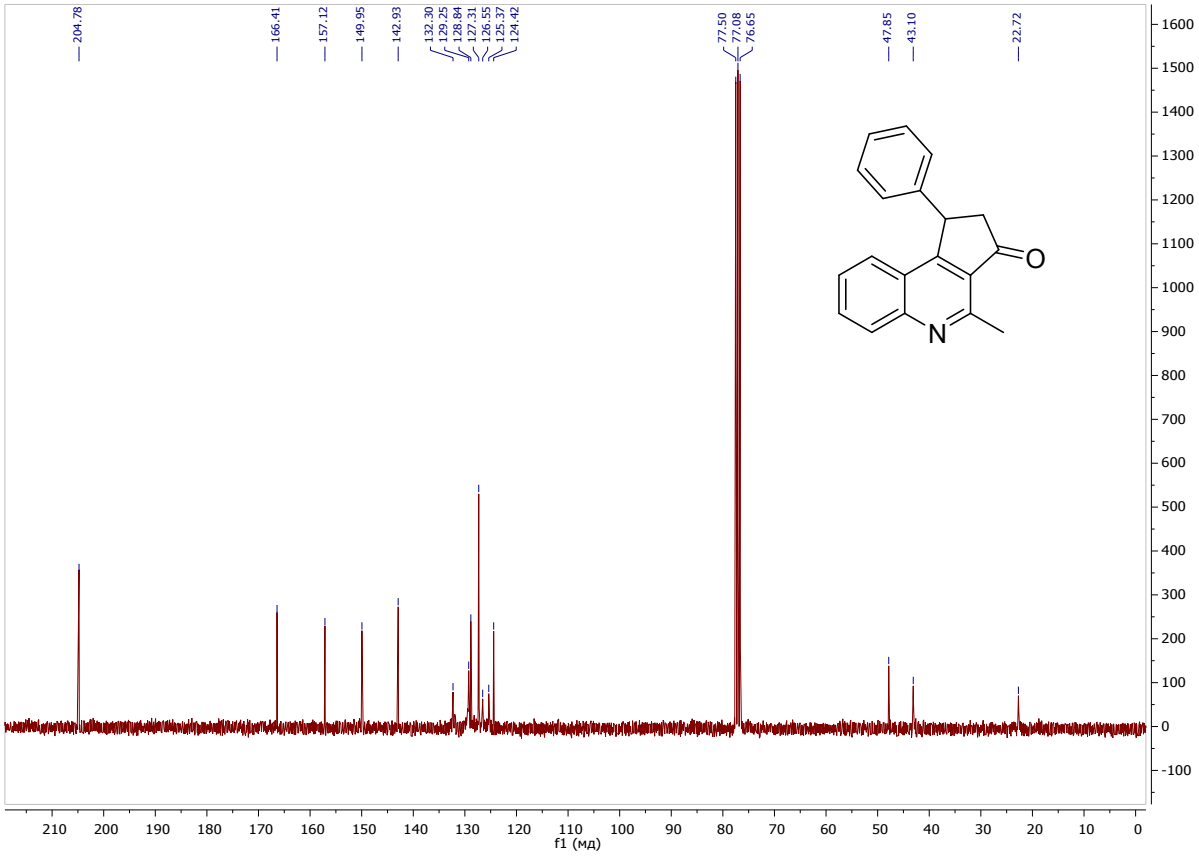
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