Supporting Information

Synthesis of 3-acylindoles by oxidative rearrangement of 2-aminochalcone using a hypervalent iodine reagent and cyclization sequence

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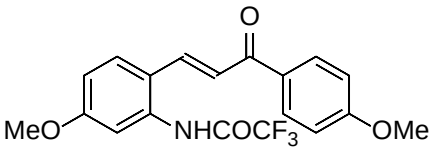
Experimental Section

General. Column chromatography and TLC were performed on Merck Silica gel 60 (230–400 mesh) and Merck Silica gel F254 plates (0.25 mm), respectively. The melting point was measured using the Stuart® melting point apparatus SMP3 with an AC input of 100 V. ^{1}H and ^{13}C NMR spectra were recorded on the JEOL JMN-400 spectrometer in $CDCl_3$ or DMSO-d_6 with tetramethylsilane as an internal standard. Data are reported as follows: chemical shift in ppm (δ), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, m = multiplet), and coupling constant (Hz). High-resolution mass spectra were obtained on the SHIMAZU IRAffinity-1 instrument with ionization voltages of 70 eV.

Materials: Unless otherwise noted, all reagents, including $PhI(OCOCH_3)_2$ (PIDA), PhI(OH)OTs and $PhI(OCOCF_3)_2$, and solvents were purchased from commercial suppliers and used without further purification.

General procedure for synthesis of chalcone

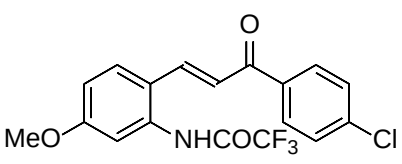
To the solution of aldehyde (1 equiv.) in toluene (0.2 M) was added ylide **(**1.2 equiv.) at 80 °C, and then stirred at same temperature. After cooling to room temperature, the resulting mixture was concentrated in vacuo. The residue was purified by SiO_2 column chromatography (eluent: Hexane/AcOEt) to give the desired chalcone.



(*E*)-2,2,2-Trifluoro-*N*-(5-methoxy-2-(3-(4-methoxyphenyl)-3-oxoprop-1-en-1-yl)phenyl)acetamide (1a)

According to the general procedure, aldehyde (443 mg, 1.60 mmol) in toluene (8.0 mL) was added ylide (722 mg, 1.76 mmol) at 80 °C, then stirred at same temperature in 15 h. The resulting mixture was concentrated in vacuo. The residue was purified by SiO_2 column chromatography (Hexane/AcOEt = 1/1) to give **1a** (537 mg, 89%) as ocher solid.

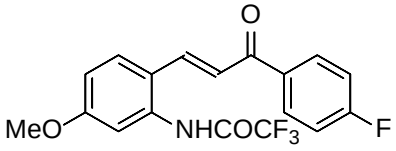
mp 170-171 °C ^{1}H-NMR ($CDCl_3$) δ : 3.87 (3H, s, OMe), 3.88 (3H, s, OMe), 6.88 (1H, dd, *J* = 2.4, 8.8 Hz), 6.95 (2H, d, *J* = 8.8 Hz), 7.40 (1H, d, *J* = 15.6 Hz), 7.45 (1H, d, *J* = 2.4 Hz), 7.65 (1H, d, *J* = 8.8 Hz), 7.84 (1H, d, *J* = 16.0 Hz), 7.96 (2H, d, *J* = 8.8 Hz), 8.54 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 55.7, 55.9, 112.9, 114.2, 114.7, 116.3 (q, *J* = 287.3 Hz), 121.3, 123.6, 129.2, 130.7, 131.1, 136.1, 137.6, 155.9 (q, *J* = 37.1 Hz), 161.5, 163.4, 187.4; HRFABMS: calcd for $C_{19}H_{17}NO_4F_3$ $[M+H]^+$: 380.1110, found 380.1086.



(*E*)-*N*-(2-(3-(4-Chlorophenyl)-3-oxoprop-1-en-1-yl)-5-methoxyphenyl)-2,2,2-trifluoroacetamide (1b)

According to the general procedure, the reaction of aldehyde (395 mg, 1.60 mmol) with ylide (730 mg, 1.76 mmol) in toluene (8.0 mL) gave **1b** (580 mg, 93%) as ocher solid. Reaction time: 15 h. Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1.

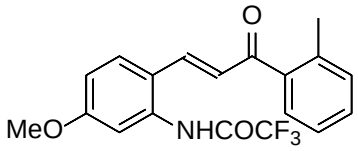
mp 173-174 °C; ^{1}H-NMR ($CDCl_3$) δ: 3.84 (3H, s, OMe), 7.01 (1H, d, *J* = 2.8 Hz), 7.06 (1H, dd, *J* = 2.4, 8.8 Hz), 7.63 (2H, d, *J* = 8.4 Hz), 7.71 (1H, d, *J* = 15.2 Hz), 7.83 (1H, d, *J* = 16.0 Hz), 8.16 (2H, d, *J* = 8.4 Hz), 8.19 (1H, d, *J* = 8.8 Hz), 11.42 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 55.9, 112.9, 114.7, 116.2 (q, *J* = 286.5 Hz), 120.9, 123.2, 129.1, 129.3, 130.6, 136.4, 136.5, 138.3, 139.0, 156.0 (q, *J* = 37.0 Hz), 161.8, 188.2; HRFABMS: calcd for $C_{18}H_{14}NO_3F_3Cl$ $[M+H]^+$ 384.0614, found 384.0625.



(*E*)-2,2,2-Trifluoro-*N*-(2-(3-(4-fluorophenyl)-3-oxoprop-1-en-1-yl)-5-methoxyphenyl)acetamide (1c)

According to the general procedure, the reaction of aldehyde (49.4 mg, 0.20 mmol) with ylide (87.6 mg, 0.22 mmol) in toluene (1.0 mL) gave **1c** (66.1 mg, 90%) as ocher solid. Reaction time: 2 h. Eluent of SiO_2 column chromatography: CH_2Cl_2.

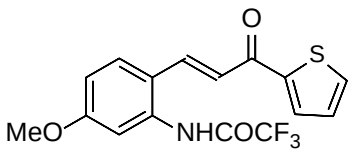
mp 168-169 °C; ^{1}H-NMR ($CDCl_3$) δ: 3.87 (3H, s, OMe), 6.89 (1H, dd, *J* = 2.4, 8.4 Hz), 7.15 (2H, t, *J* = 7.2 Hz), 7.37 (1H, d, *J* = 15.2 Hz), 7.41 (1H, d, *J* = 2.4 Hz), 7.67 (1H, d, *J* = 8.8 Hz), 7.85 (1H, d, *J* = 15.6 Hz), 7.99 (2H, dd, *J* = 5.6, 8.4 Hz), 8.44 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 55.7, 110.0, 114.3, 115.7, 115.8 (q, *J* = 286.5 Hz), 115.9 (d, *J* = 4.1 Hz), 120.9, 121.7, 128.9, 131.0 (d, *J* = 9.1 Hz), 134.1 (d, *J* = 2.5 Hz), 135.1, 138.3, 155.7 (q, *J* = 37.0 Hz), 162.1, 165.7 (d, *J* = 254.6 Hz), 188.3; HRFABMS: calcd for $C_{18}H_{14}NO_3F_4$ $[M+H]^+$ 368.0910, found 368.0958.



(*E*)-2,2,2-Trifluoro-*N*-(5-methoxy-2-(3-oxo-3-(*o*-tolyl)prop-1-en-1-yl)phenyl)acetamide (1d)

To the solution of aldehyde (98.8 mg, 0.40 mmol) and phosphonium salts (190 mg, 0.40 mmol) in THF (4.0 mL) was added DBU (89 μL, 0.6 mmol) at 40 °C. Then stirred at same temperature in 20 h. The reaction was quenched with NH_4Cl aq. The organic layer was extracted with AcOEt, washed with brine, dried over with Na_2SO_4 and concentrated in vacuo. The residue was purified by SiO_2 column chromatography (Hexane/AcOEt = 4/1) to give **1d** (111.4 mg, 77%) as ocher solid.

mp 130-131 °C; ^{1}H-NMR ($CDCl_3$) δ : 2.43 (3H, s, Me), 3.86 (3H, s, OMe), 6.89 (1H, dd, *J* = 2.8, 8.8 Hz), 7.04 (1H, d, *J* = 15.6 Hz), 7.24-7.28 (2H, m), 7.32 (1H, d, *J* = 2.4 Hz), 7.38 (1H, dt, *J* = 1.6, 8.0 Hz), 7.48 (1H, d, *J* = 8.8 Hz), 7.51 (1H, d, *J* = 15.6 Hz), 7.65 (1H, d, *J* = 8.8 Hz), 8.15 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 19.9, 55.5, 110.9, 114.4, 115.7 (q, *J* = 286.5 Hz), 121.7, 125.3, 126.0, 127.9, 128.5, 130.6, 131.2, 134.9, 136.8, 138.3, 139.8, 156.0 (q, *J* = 37.9 Hz), 161.9, 196.4; HRFABMS: calcd for $C_{19}H_{16}NO_3F_3$ $[M]^+$ 363.1082, found 363.1091.

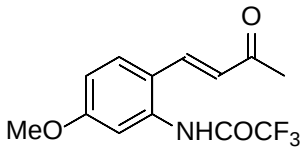


(*E*)-2,2,2-Trifluoro-*N*-(5-methoxy-2-(3-oxo-3-(thiophen-2-yl)prop-1-en-1-yl)phenyl)acetamide (1e)

According to general procedure, the reaction of aldehyde (49.4 mg, 0.20 mmol) with ylide (87.6 mg, 0.22 mmol) in toluene (1.0 mL) gave **1e** (66.1 mg, 90%) as yellow solid. Reaction time: 2 h. Eluent of SiO_2 column chromatography: CH_2Cl_2.

mp 213-214 °C, ^{1}H-NMR ($CDCl_3$) δ : 3.87 (3H, s, Me), 6.89 (1H, dd, *J* = 2.4, 8.8 Hz), 7.17 (1H, t, *J* = 4.0 Hz), 7.26 (1H, d, *J* = 7.6 Hz), 7.45 (1H, d, *J* = 2.4 Hz), 7.65-7.69 (2H, m), 7.80 (1H, d, *J* = 4.0 Hz), 7.89 (1H, d, *J* = 7.2

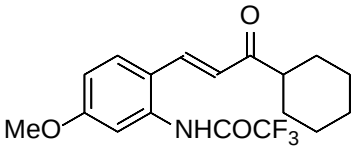
Hz), 8.57 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ : 55.7, 112.7, 114.5, 116.0 (q, J = 288.4 Hz), 121.0, 123.0, 128.9, 129.0, 133.6, 135.5, 136.1, 137.3, 145.6, 155.8 (q, J = 38.0 Hz), 161.5, 181.5; HRFABMS: calcd for $C_{16}H_{13}NO_3F_3S$ $[M+H]^+$ 356.0568, found 356.0548.



(*E*)-2,2,2-Trifluoro-*N*-(5-methoxy-2-(3-oxobut-1-en-1-yl)phenyl)acetamide (1f)

According to the procedure for **1d**, the reaction of aldehyde (74.1 mg, 0.30 mmol) with (2-oxopropyl)triphenylphosphonium bromide (107 mg, 0.30 mmol) and DBU (67 μL, 0.45 mmol) in THF (3.0 mL) gave **1f** (66.2 mg, 77%) as colorless oil. Reaction time: 1 h. Eluent of SiO_2 column chromatography: Hexane/AcOEt = 4/1.

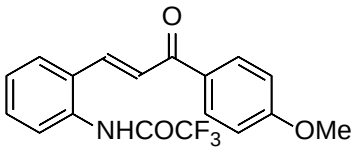
mp 122-123 °C; ^{1}H-NMR ($CDCl_3$) δ : 2.33 (3H, s, Me), 3.86 (3H, s, OMe), 6.61 (1H, d, J = 16.0 Hz), 6.88 (1H, dd, J = 2.8, 8.8 Hz), 7.28 (1H, d, J = 2.4 Hz), 7.52 (1H, d, J = 7.6 Hz), 7.57 (1H, d, J = 8.8 Hz), 8.30 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 27.7, 55.6, 110.8, 114.4, 115.8 (q, J = 284.9 Hz), 121.2, 126.6, 128.6, 134.7, 137.1, 156.1 (q, J = 37.9 Hz), 161.8, 198.5; HRFABMS: calcd for $C_{13}H_{13}NO_3F_3$ $[M+H]^+$ 288.0848, found 288.0821.



(*E*)-*N*-(2-(3-Cyclohexyl-3-oxoprop-1-en-1-yl)-5-methoxyphenyl)-2,2,2-trifluoroacetamide (1g)

To the solution of aldehyde (24.7 mg, 0.10 mmol) and phosphonium salts[1] (93.4 mg, 0.20 mmol) in THF (1.0 mL) and DMF (1.0 mL) was added DBU (22 μL, 0.15 mmol) at 65 °C. Then stirred at same temperature in 25 h and quenched with NH_4Cl aq. The organic layer was extracted with AcOEt, washed with brine, dried over with Na_2SO_4 and concentrated in vacuo. The residue was purified by SiO_2 column chromatography (CH_2Cl_2) to give **1g** (22.4 mg, 63%) as white solid.

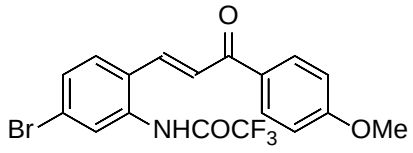
mp 137-138 °C; ^{1}H-NMR ($CDCl_3$) δ : 1.21-1.44 (5H, m), 1.60-1.90 (5H, m), 2.55 (1H, tt, J = 3.6, 7.6 Hz), 3.86 (3H, s, OMe), 6.72 (1H, d, J = 15.6 Hz), 6.86 (1H, dd, J = 2.8, 8.8 Hz), 7.42 (1H, d, J = 3.2 Hz), 7.58 (1H, d, J = 8.8 Hz), 7.62 (1H, d, J = 15.6 Hz), 8.16 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 25.6, 25.7, 28.4, 49.9, 55.6, 110.3, 114.3, 115.9 (q, J = 287.3 Hz), 121.1, 124.2, 128.5, 135.1, 136.1, 155.9 (q, J = 37.9 Hz), 161.7, 202.9; HRFABMS: calcd for $C_{18}H_{20}NO_3F_3$ $[M]^+$ 355.1395, found 355.1351.



(*E*)-2,2,2-Trifluoro-*N*-(2-(3-(4-methoxyphenyl)-3-oxoprop-1-en-1-yl)phenyl)acetamide (1h)

According to the general procedure, the reaction of aldehyde (65.1 mg, 0.30 mmol) with ylide (131 mg, 0.33 mmol) in toluene (1.5 mL) gave **1h** (91.1 mg, 87%) as pale yellow solid. Reaction time: 2 h. Eluent of SiO_2 column chromatography: CH_2Cl_2.

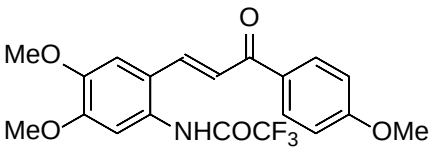
mp 203-204 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.87 (3H, s, OMe), 7.10 (1H, d, J = 8.4 Hz), 7.41 (1H, d, J = 7.6 Hz), 7.47 (1H, d, J = 7.6 Hz), 7.53 (1H, d, J = 7.6 Hz), 7.72 (1H, d, J = 15.6 Hz), 7.94 (1H, d, J = 15.6 Hz), 8.16-8.21 (3H, m), 11.4 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ :55.6, 114.1, 116.1 (q, J = 288.4 Hz), 123.7, 127.6, 127.8, 128.0, 130.2, 130.9, 130.9, 131.0, 134.2, 137.4, 155.8 (d, J = 37.1 Hz), 163.4, 187.3 ; HRFABMS: calcd for $C_{18}H_{15}NO_3F_3$ $[M+H]^+$ 350.1004, found 350.0963.



(*E*)-*N*-(5-Bromo-2-(3-(4-methoxyphenyl)-3-oxoprop-1-en-1-yl)phenyl)-2,2,2-trifluoroacetamide (1i)

According to the general procedure, the reaction of aldehyde (355 mg, 1.20 mmol) with ylide (541 mg, 1.32 mmol) in toluene (6 mL) gave **1i** (456 mg, 89%) as pale yellow solid. Reaction time: 12 h. Eluent of SiO_2 column chromatography: Hexane/AcOEt = 1/1.

mp 202-203 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.86 (3H, s, OMe), 7.08 (1H, d, *J* = 8.8 Hz), 7.62-7.71 (3H, m), 7.99 (2H, d, *J* = 15.6 Hz), 8.16 (2H, d, *J* = 8.8 Hz), 11.5 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 55.6, 114.1, 115.9 (q, *J* = 287.6 Hz), 123.2, 124.3, 129.3, 130.15, 130.23, 130.4, 130.9, 131.1, 135.5, 136.3, 155.9 (q, *J* = 36.3 Hz), 163.4, 187.2; HRFABMS: calcd for $C_{18}H_{14}NO_3F_3Br$ $[M+H]^+$ 428.0109, found 428.0102.



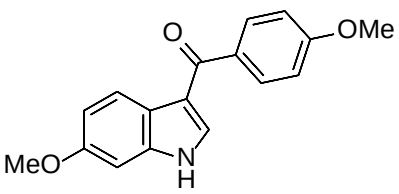
(*E*)-*N*-(4,5-Dimethoxy-2-(3-(4-methoxyphenyl)-3-oxoprop-1-en-1-yl)phenyl)-2,2,2-trifluoroacetamide (1j)

According to the general procedure, the reaction of aldehyde (443 mg, 1.6 mmol) with ylide (722 mg, 1.76 mmol) in toluene (8 mL) gave **1j** (622 mg, 95%) as pale yellow solid. Reaction time: 16 h. Eluent of SiO_2 column chromatography: Hexane/AcOEt = 1/1.

mp 210-212 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.82 (3H, s, OMe), 3.87 (3H, s, OMe), 3.93 (3H, s, OMe), 7.00 (1H, s), 7.10 (2H, d, *J* = 8.8 Hz), 7.65 (1H, s), 7.67 (1H, d, *J* = 15.2 Hz), 7.87 (1H, d, *J* = 15.2 Hz), 8.17 (2H, d, *J* = 8.8 Hz), 11.27 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ 55.5, 55.9, 56.1, 109.2, 110.9, 114.0, 116.1 (q, *J* = 288.4 Hz), 121.3, 123.3, 128.4, 130.5, 130.9, 137.6, 148.4, 151.1, 155.9 (q, *J* = 36.3 Hz), 163.2, 187.3; HRFABMS: calcd for $C_{20}H_{19}F_3NO_5$ $[M+H]^+$ 410.1215, found 410.1218.

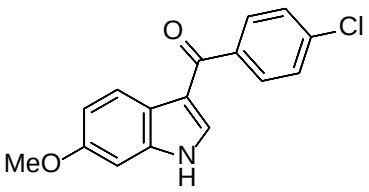
General procedure for synthesis of 3-acylindole

To the solution of chalcone (0.1 mmol) in $CH(OMe)_3$ (1 ml) was added BF_3•Et_2O (0.25 mmol) at room temperature. The resulting mixture was added $PhI(OAc)_2$ (0.15 mmol), then stirred at same temperature for 1 h. THF and 30% aqueous K_2CO_3 were then added, and the mixture was stirred at 60 °C for an appropriate time.. The organic layer was extracted with CH_2Cl_2, washed with brine, dried over with Na_2SO_4 and concentrated *in vacuo*. The residue was purified by SiO_2 column chromatography (eluent: hexane/AcOEt) to give the desired 3-acylindole.



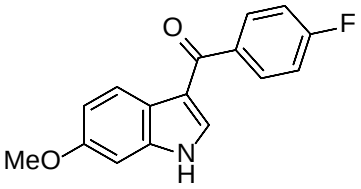
(6-Methoxy-1*H*-indol-3-yl)(4-methoxyphenyl)methanone (3a)

99%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; white solid; mp 216-217 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.80 (3H, s, OMe), 3.85 (3H, s, OMe), 6.86 (1H, dd, *J* = 1.6, 8.8 Hz), 7.00 (1H, s), 7.06 (2H, d, *J* = 8.8 Hz), 7.79 (2H, d, *J* = 8.8 Hz), 7.80 (1H, s), 8.07 (1H, d, *J* = 8.8 Hz), 11.79 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ : 55.4, 55.6, 95.3, 111.7, 113.8, 115.3, 120.6, 122.3, 130.7, 133.1, 134.1, 137.7, 156.7, 161.9, 188.8; HRFABMS: calcd for $C_{17}H_{16}NO_3$ $[M+H]^+$ 282.1130, found 282.1118.



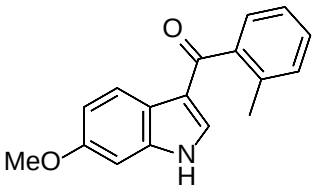
(4-Chlorophenyl)(6-methoxy-1*H*-indol-3-yl)methanone (3b)

99%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 1/1; yellow solid; mp 251-253 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.80 (3H, s, OMe), 6.90 (1H, dd, *J* = 2.0, 8.8 Hz), 7.00 (1H, d, *J* = 2.0 Hz), 7.59 (2H, d, *J* = 7.6 Hz), 7.79 (2H, d, *J* = 8.4 Hz), 7.84 (1H, d, *J* = 2.8 Hz), 8.09 (1H, d, *J* = 8.8 Hz), 11.93 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ : 55.4, 95.5, 112.0, 115.1, 120.3, 122.3, 128.6, 130.4, 135.3, 136.0, 137.9, 139.3, 156.9, 188.7; HRFABMS: calcd for $C_{16}H_{13}NO_2Cl$ $[M+H]^+$ 286.0635, found 286.0620.



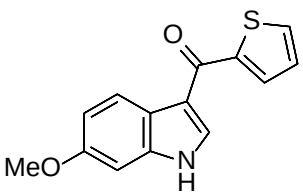
(4-Fluorophenyl)(6-methoxy-1*H*-indol-3-yl)methanone (3c)

91%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; ocher solid; mp 241-243 °C; ^{1}H-NMR (DMSO d_6) δ : 3.80 (3H, s, OMe), 6.88 (1H, dd, *J* = 1.6, 8.8 Hz), 7.00 (1H, d, *J* = 2.0 Hz), 7.35 (2H, t, *J* = 8.8 Hz), 7.82-7.87 (3H, m), 8.09 (1H, d, *J* = 8.8 Hz), 11.91 (1H, s, NH) ; ^{13}C-NMR (125.8 MHz, DMSO-d_6) 55.2, 95.2, 111.7, 114.9, 115.3 (d, *J* = 21.6 Hz), 120.2, 122.1, 130.9 (d, *J* = 9.1 Hz), 134.8, 137.0 (d, *J* = 3.0 Hz), 137.6, 156.6, 163.7 (d, *J* = 248.4 Hz), 188.3; HRFABMS: calcd for $C_{16}H_{13}NO_2F$ $[M+H]^+$ 270.0930, found 270.0970.



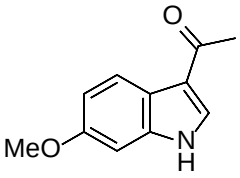
(6-Methoxy-1*H*-indol-3-yl)(*o*-tolyl)methanone (3d)

84%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; yellow solid; mp 216-217 °C; ^{1}H-NMR (DMSO- d_6) δ : 2.26 (3H, s, Me), 3.79 (3H, s, OMe), 6.87 (1H, dd, *J* = 2.0, 8.8 Hz), 6.99 (1H, d, *J* = 1.6 Hz), 7.28-7.32 (2H, m), 7.36-7.41 (2H, m), 7.46 (1H, d, *J* = 2.4 Hz), 8.03 (1H, d, *J* = 8.8 Hz), 11.82 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ : 19.4, 55.4, 95.6, 112.0, 116.9, 119.8, 122.1, 125.4, 127.4, 129.3, 130.7, 135.0, 135.6, 138.0, 141.1, 156.8, 192.1; HRFABMS: calcd for $C_{17}H_{16}NO_2$ $[M+H]^+$ 266.1181, found 266.1139.



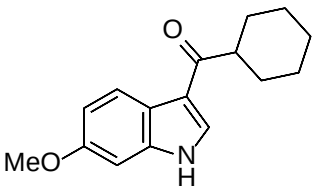
(6-Methoxy-1*H*-indol-3-yl)(thiophen-2-yl)methanone (3e)

94%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; ocher solid; mp 229-231 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.80 (3H, s, OMe), 6.87 (1H, dd, *J* = 2.4, 8.8 Hz), 7.00 (1H, d, *J* = 2.4 Hz), 7.25 (1H, dd, *J* = 4.0, 4.8 Hz), 7.91-7.93 (2H, m), 8.07 (1H, d, *J* = 8.8 Hz), 8.22 (1H, d, *J* = 2.8 Hz), 11.92 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ: 45.0, 95.2, 111.6, 114.6, 120.2, 122.0, 128.2, 131.1, 132.1, 133.1, 137.4, 145.1, 156.6, 180.5; HRFABMS: calcd for $C_{14}H_{12}NO_2S$ $[M+H]^+$ 258.0589, found 258.0598.



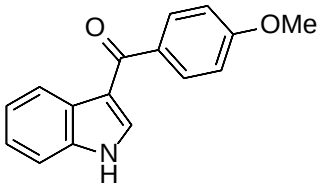
1-(6-Methoxy-1*H*-indol-3-yl)ethan-1-one (3f)[2]

91%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 1/1; ocher solid; ^{1}H-NMR (DMSO-d_6) δ : 2.41 (3H, s, Me), 3.77 (3H, s, OMe), 6.80 (1H, d, *J* = 8.8 Hz), 6.93 (1H, s), 8.01 (1H, d, *J* = 8.4 Hz), 8.16 (1H, s), 11.71 (1H, s, NH).



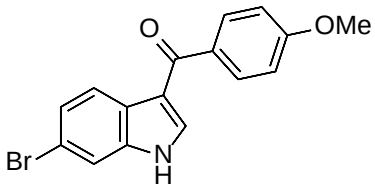
Cyclohexyl(6-methoxy-1*H*-indol-3-yl)methanone (3g)

89%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; beige solid; mp 161-163 °C; ^{1}H-NMR ($CDCl_3$) δ : 1.25-1.42 (3H, m), 1.58-1.75 (3H, m), 1.84-1.93 (4H, m), 3.03 (1H, tt, *J* = 3.2, 12.0 Hz), 3.80 (3H, s, OMe), 6.86 (1H, d, *J* = 2.4 Hz), 6.93 (1H, dd, *J* = 2.4, 8.8 Hz), 7.79 (1H, d, *J* = 2.8 Hz), 8.27 (1H, d, *J* = 8.8 Hz), 9.06 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ: 25.3, 25.7, 29.7, 46.0, 55.2, 95.0, 111.4, 115.2, 119.7, 122.1, 132.4, 137.7, 156.2, 198.7; HRFABMS: calcd for $C_{16}H_{20}NO_2$ $[M+H]^+$ 258.1494, found 258.1447.



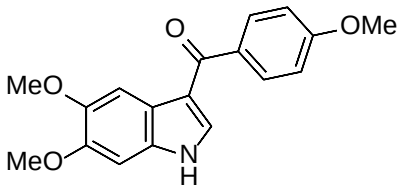
(1*H*-Indol-3-yl)(4-methoxyphenyl)methanone (3h)[3]

81%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; beige solid; ^{1}H-NMR (DMSO-d_6) δ : 3.34 (3H, s), 7.06 (2H, d, *J* = 8.8 Hz), 7.20-7.26 (2H, m), 7.50 (1H, d, *J* = 7.2 Hz), 7.80 (2H, d, *J* = 8.8 Hz), 7.93 (1H, d, *J* = 3.2 Hz), 8.22 (1H, d, *J* = 7.2 Hz), 12.00 (1H, s, NH).



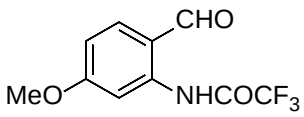
(6-Bromo-1*H*-indol-3-yl)(4-methoxyphenyl)methanone (3i)

51%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; beige solid; mp 208-209 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.85 (3H, s, OMe), 7.07 (2H, d, *J* = 8.8 Hz), 7.36 (1H, d, *J* = 8.4 Hz), 7.71 (1H, s), 7.81 (2H, d, *J* = 8.8 Hz), 8.00 (1H, s), 8.15 (1H, d, *J* = 8.4 Hz), 12.1 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ: 55.4, 113.7, 114.8, 115.0, 115.5, 123.2, 124.6, 125.5, 130.7, 132.5, 135.6, 137.5, 161.9, 188.6; HRFABMS: calcd for $C_{16}H_{12}NO_2Br$ $[M+H]^+$ 330.0130, found 330.0102.



(5,6-Dimethoxy-1*H*-indol-3-yl)(4-methoxyphenyl)methanone (3j)

74%; Eluent of SiO_2 column chromatography: Hexane/AcOEt = 2/1; beige solid; mp 216-217 °C; ^{1}H-NMR ($CDCl_3$) δ : 3.90 (3H, s, OMe), 3.92 (3H, s, OMe), 3.95 (3H, s, OMe), 6.90 (1H, s), 7.01 (1H, d, *J* = 8.8 Hz), 7.05 (1H, s), 8.01 (1H, d, *J* = 8.8 Hz), 9.54 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ: 55.4, 113.7, 114.8, 115.0, 115.5, 123.2, 124.6, 125.5, 130.1, 132.5, 135.6, 137.5, 161.9, 188.6; HRFABMS: calcd for $C_{18}H_{18}NO_4$ $[M+H]^+$ 312.1236, found 312.1247.

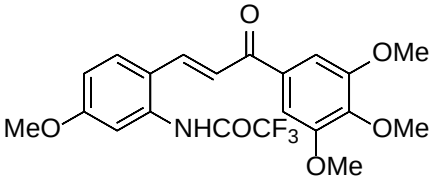


2,2,2-Trifluoro-*N*-(2-formyl-5-methoxyphenyl)acetamide (5)

To the solutioin of 4-methoxy-2-nitrobenzaldehyde (300 mg, 1.57 mmol) in EtOH (5 mL) and water (5 mL) was added Fe (292 mg, 4.71 mmol) and NH_4Cl (466 mg, 7.85 mmol) then stirred at 60 °C for 1 h. Fe was removed by filtration and the solvent was extracted with CH_2Cl_2. The organic layer was concentrated in vacuo, and then added CH_2Cl_2 (8 mL). Trifluoroacetic anhydride (TFAA) was slowly added to the solution at 0 °C. After 12 min, the reaction was quenched with sat. $NaHCO_3$ solution. The organic layer was extracted with CH_2Cl_2, washed with brine, dried over Na_2SO_4 and concentrated in vacuo. The

residue was purified by SiO_2 column chromatography (Hexane/AcOEt = 4/1) to give **5** (308 mg, 72%) as yellow solid.

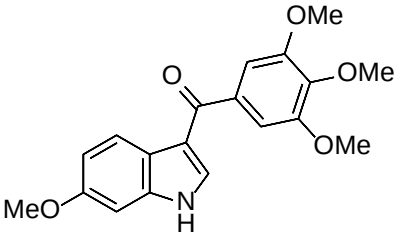
^{1}H-NMR ($CDCl_3$) δ: 3.93 (3H, s, OMe), 6.85 (1H, dd, *J* = 2.4, 8.4 Hz), 7.65 (1H, d, *J* = 8.4 Hz), 8.25 (1H, d, *J* = 2.0 Hz), 9.80 (1H, s), 12.49 (1H, s, NH); ^{13}C-NMR ($CDCl_3$) δ: 56.0, 105.2, 111.5, 115.4 (q, *J* = 287.3 Hz), 116.5, 137.9, 140.6, 156.0 (q, *J* = 37.9 Hz), 165.9, 193.7; HRFABMS: calcd for $C_{10}H_9NO_3F_3$ $[M+H]^+$ 248.0535, found 248.0525.



(*E*)-2,2,2-Trifluoro-*N*-(5-methoxy-2-(3-oxo-3-(3,4,5-trimethoxyphenyl) prop-1-en-1-yl)phenyl)acetamide (7)

According to the procedure for **1a**, the reaction of aldehyde **5** (45.3 mg, 0.18 mmol) with ylide (94.7 mg, 0.20 mmol) in toluene (1.0 mL) gave **7** (78.5 mg, 98%) as ocher solid. Reaction time: 22 h. Eluent of SiO_2 column chromatography: CH_2Cl_2.

mp 167-168 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.75 (3H, s, OMe), 3.83 (3H, s, OMe), 3.89 (3H, s, OMe), 7.00 (1H, d, *J* = 2.4 Hz), 7.06 (1H, dd, *J* = 2.0, 8.8 Hz) 7.40 (2H, s), 7.68 (1H, d, *J* = 15.6 Hz), 7.82 (1H, d, *J* = 15.2 Hz), 8.18 (1H, d, *J* = 8.8 Hz), 11.40 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ : 55.7, 56.2, 60.2, 106.1, 112.7, 114.5, 116.1 (d, *J* = 287.6 Hz), 121.0, 123.3, 129.2, 133.0, 136.1, 138.1, 142.0, 153.0, 155.8 (q, *J* = 37.2 Hz), 161.5, 187.9; HRFABMS: calcd for $C_{21}H_{21}NO_6F_3$ $[M+H]^+$ 440.1321, found 440.1293.



(6-Methoxy-1*H*-indol-3-yl)(3,4,5-trimethoxyphenyl)methanone (SCB01A)

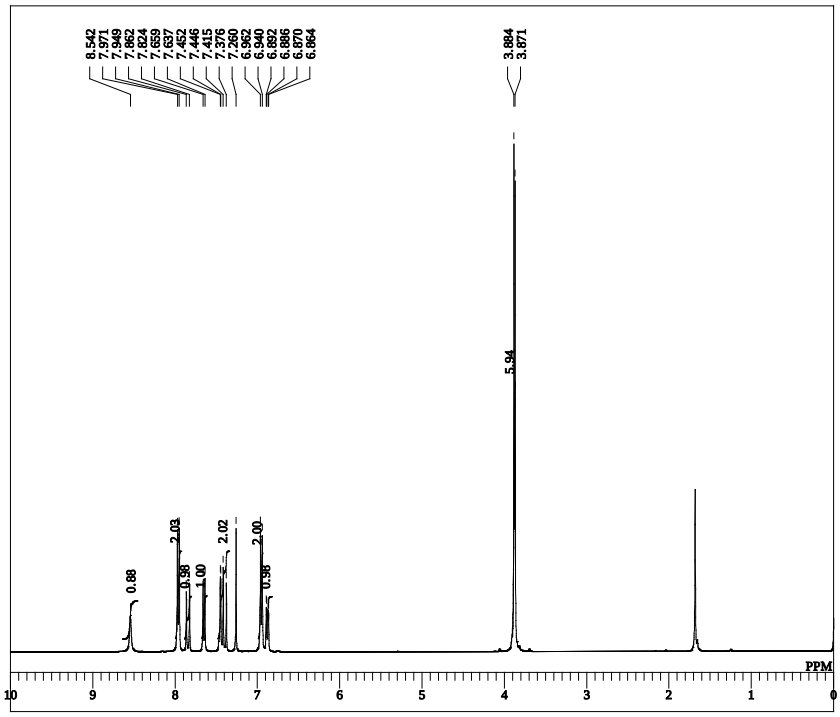
To the solution of **7** (44.0 mg, 0.10 mmol) in $CH(OMe)_3$ (2.0 mL) was added BF_3•Et_2O (64 μL, 0.51 mmol) at room temperature. The resulting mixture was added $PhI(OAc)_2$ (64.4 mg, 0.20 mmol), then stirred at same temperature for 1 h. The resulting mixture was added THF (4.0 mL) and 30% K_2CO_3 (1.0 mL) then stirred at 60 °C in 5 h. The organic layer was extracted with CH_2Cl_2, washed with brine, dried over with Na_2SO_4 and concentrated in vacuo. The residue was purified by SiO_2 column chromatography (Hexane/AcOEt = 1/1) to give **SCB01A** (32.4 mg, 95%) as pale yellow solid.

mp 187-188 °C; ^{1}H-NMR (DMSO-d_6) δ : 3.76 (3H, s, OMe), 3.80 (3H, s, OMe), 3.86 (6H, s, OMe), 6.88 (1H, dd, *J* = 2.0, 8.8 Hz), 7.00 (1H, d, *J* = 2.0 Hz), 7.09 (2H, s), 7.97 (1H, d, *J* = 2.4 Hz), 8.11 (1H, d, *J* = 8.8 Hz), 11.84 (1H, s, NH); ^{13}C-NMR (DMSO-d_6) δ : 55.2, 55.9, 60.1, 95.1, 106.0, 111.6, 114.9, 120.4, 122.1, 134.7, 135.8, 137.6, 140.0, 152.6, 156.5, 188.8; HRFABMS: calcd for $C_{19}H_{20}NO_5$ $[M+H]^+$ 342.1341, found 342.1327.

References

1. L. Luqing, R. Ciro and M. Clement, *J. Am. Chem. Soc.*, 2016, **138**, 10344.
2. De Luca, Laura; Barreca, Maria Letizia; Ferro, Stefania; Iraci, Nunzio; Michiels, Martine; Christ, Frauke; Debyser, Zeger; Witvrouw, Myriam and Chimirri, Alba, *Bioorg. Med. Chem. Lett.*, 2008, **18**, 2891.
3. D. Tuluma, C. Amarnath and S. Amitabha, *Tetrahedron Lett.*, 2014, **55**, 719.

1a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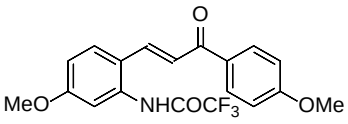
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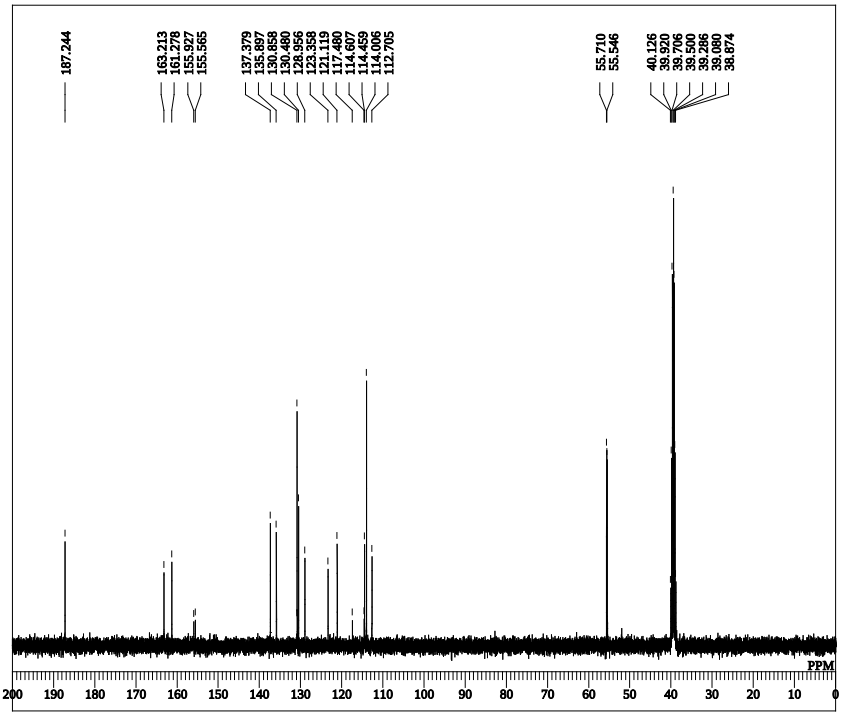
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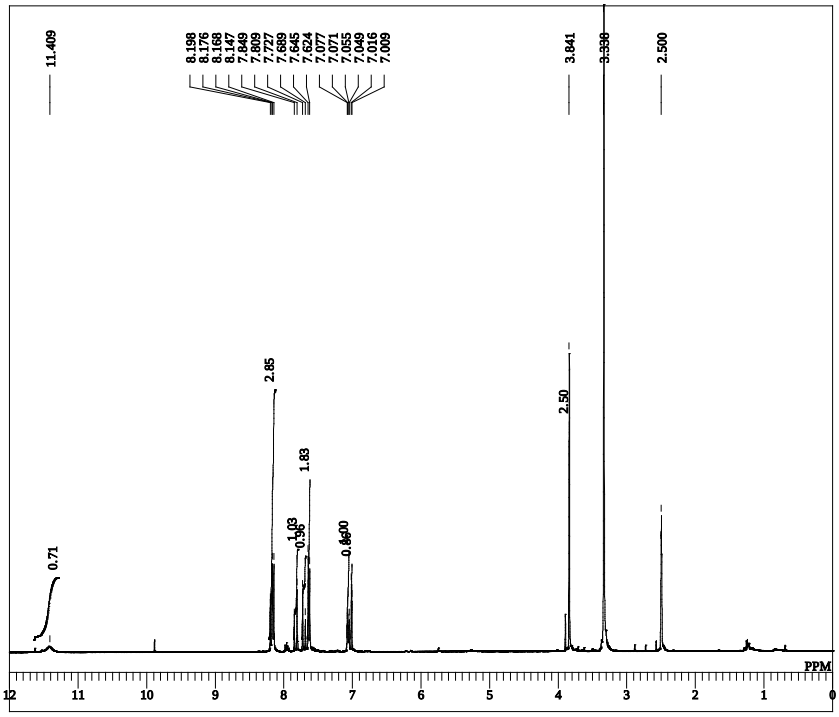
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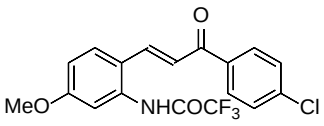
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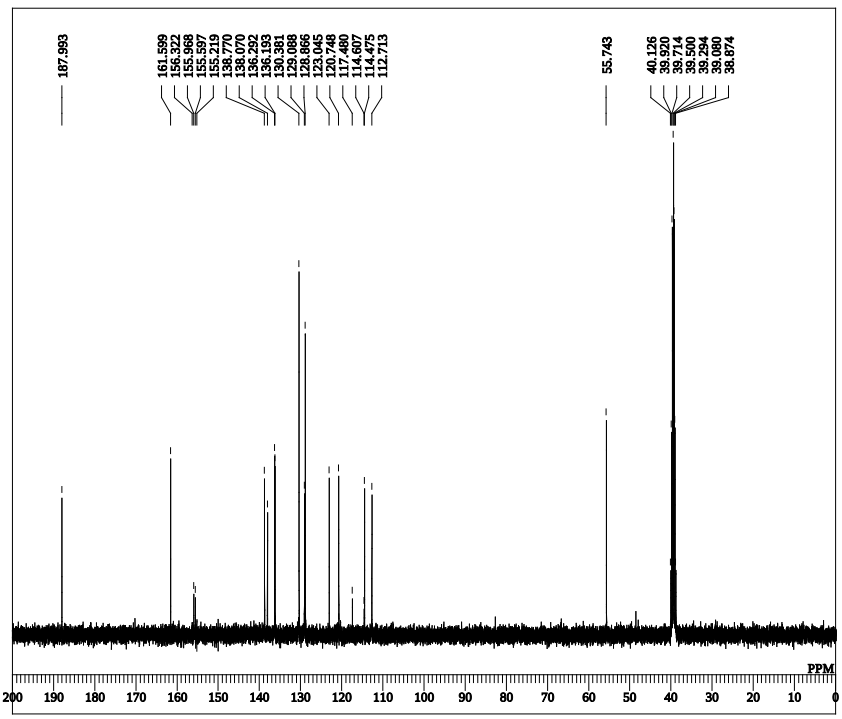
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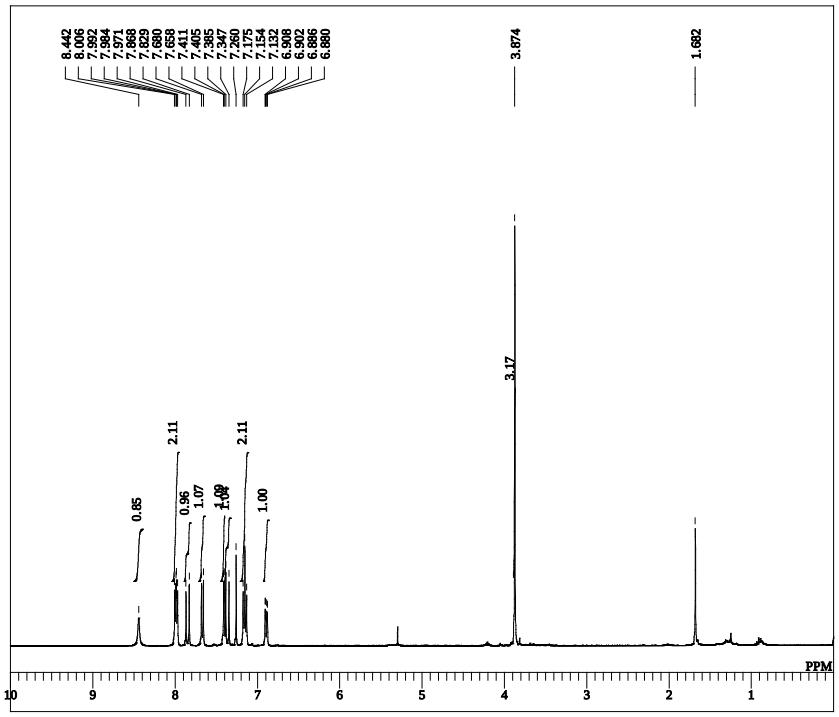
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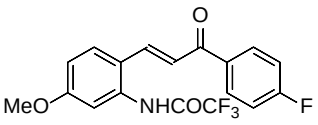
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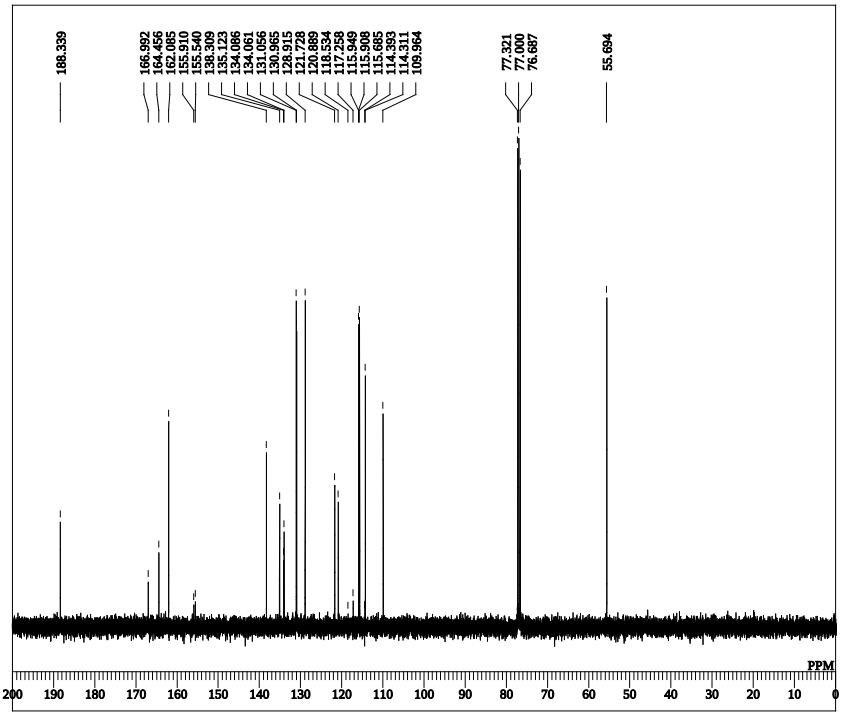
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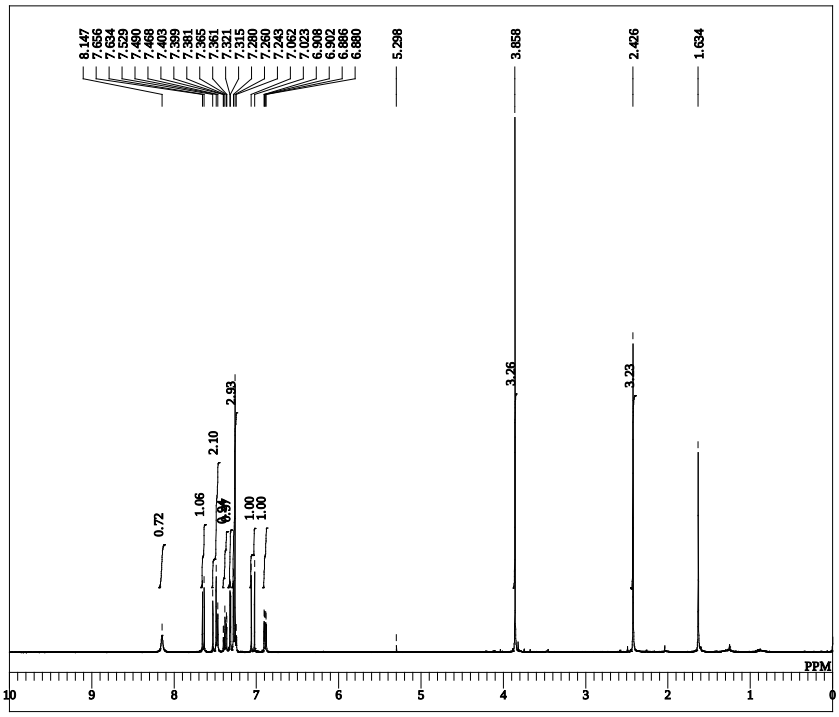
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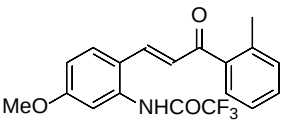
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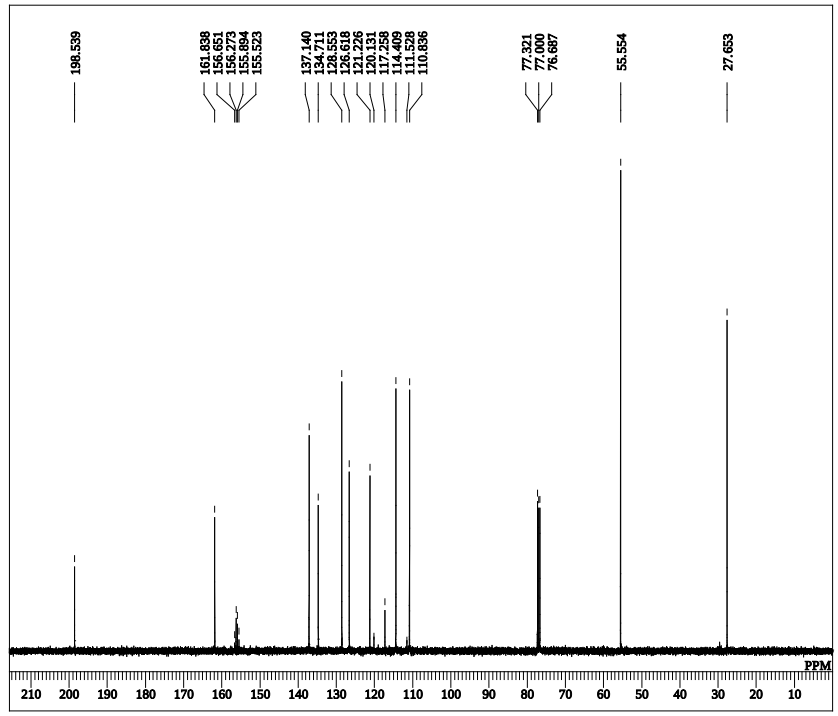
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111.528
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55.554
27.653
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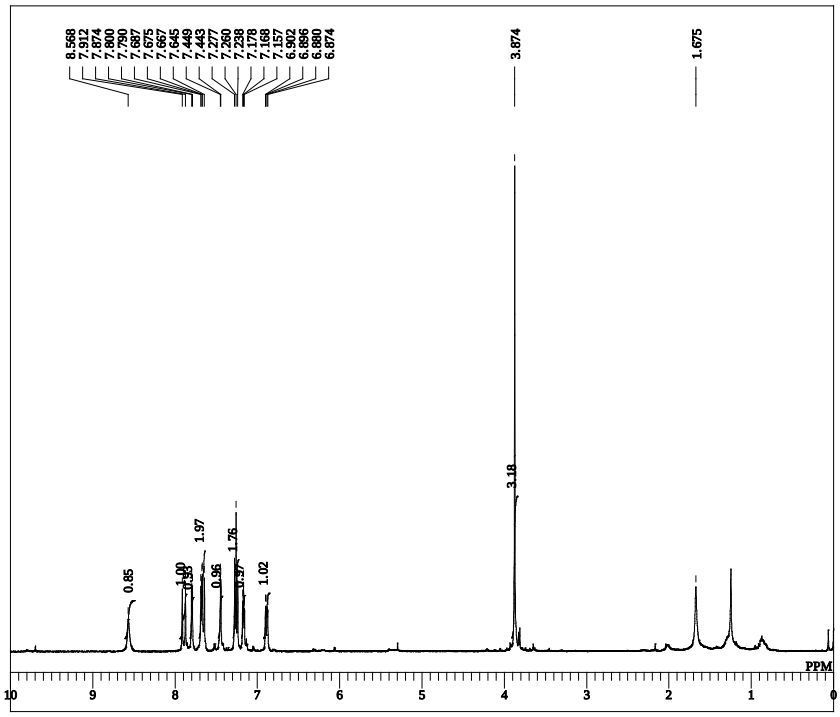
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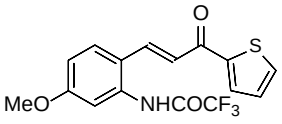
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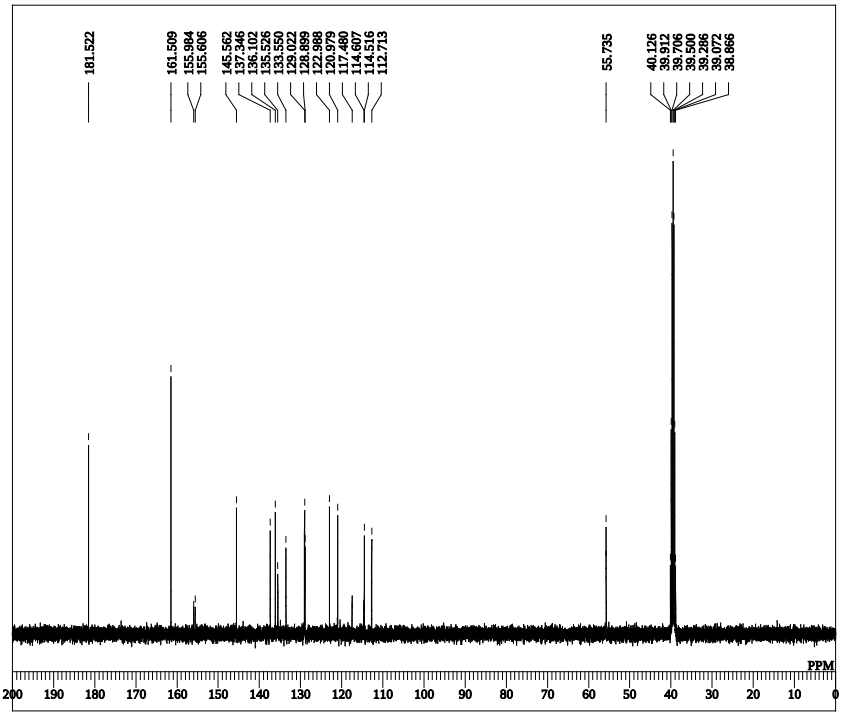
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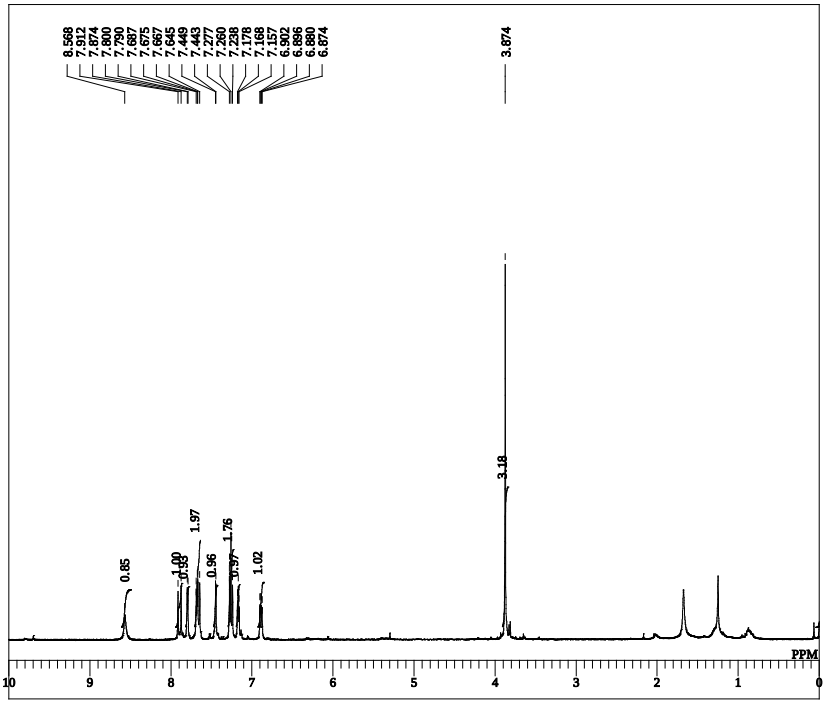
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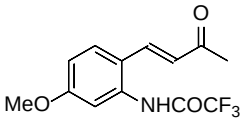
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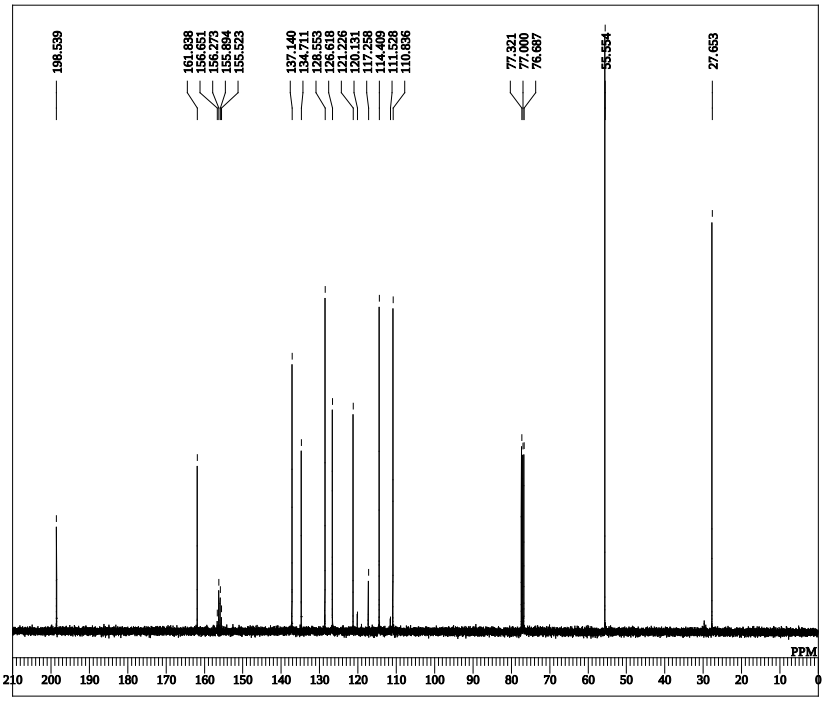
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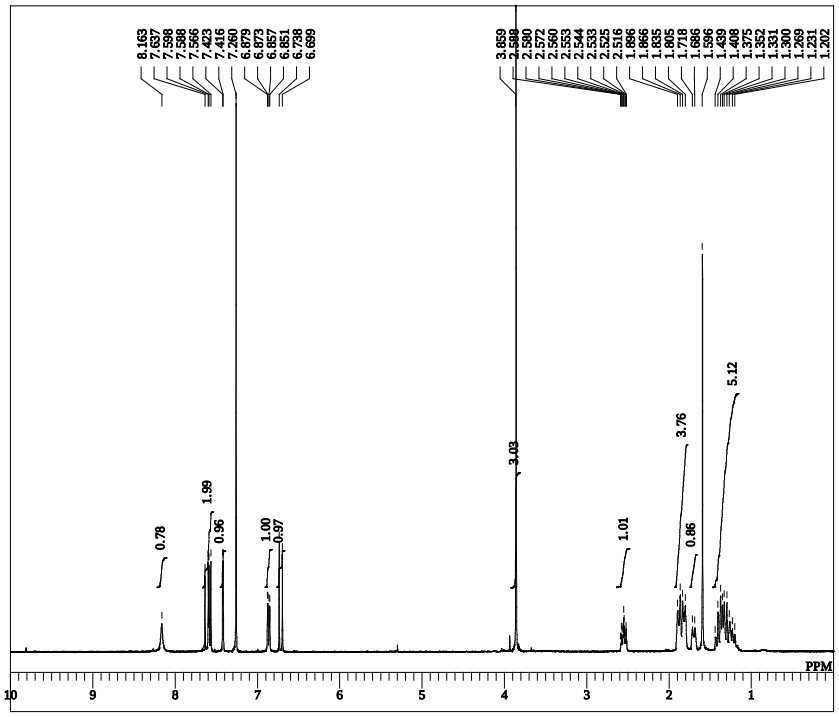
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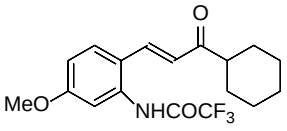
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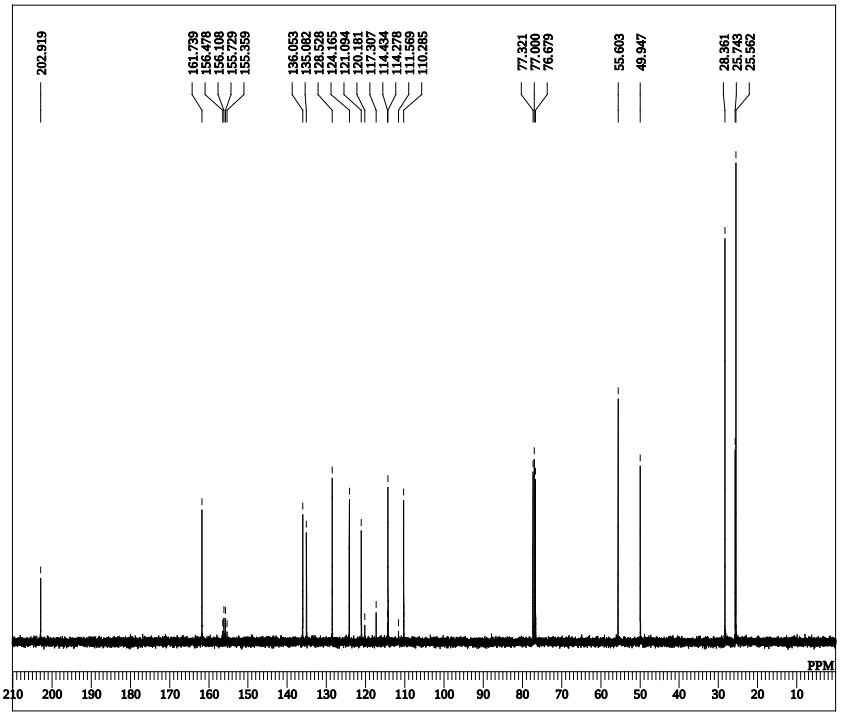
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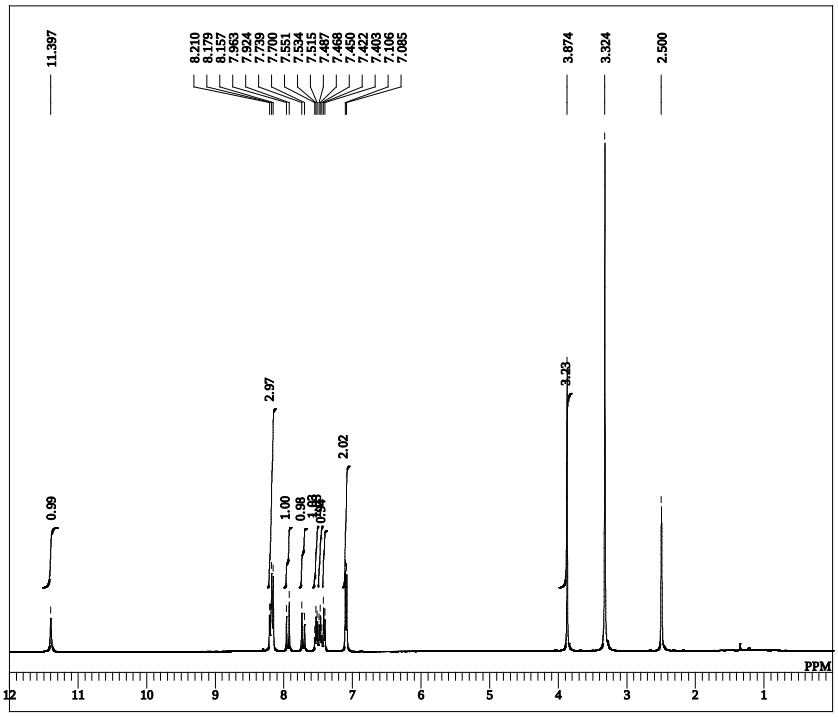
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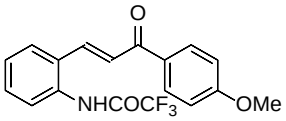
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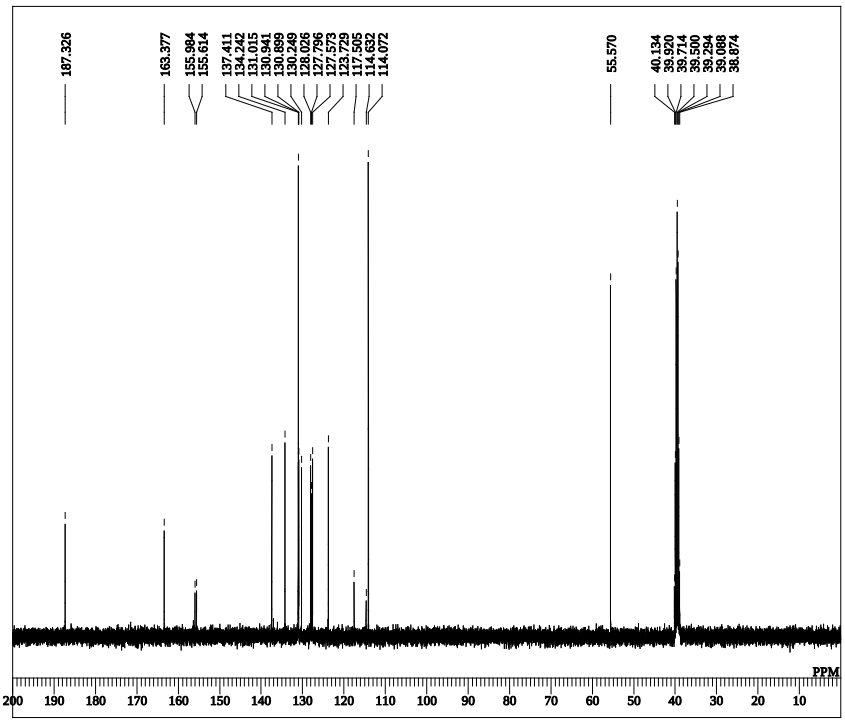
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PPM
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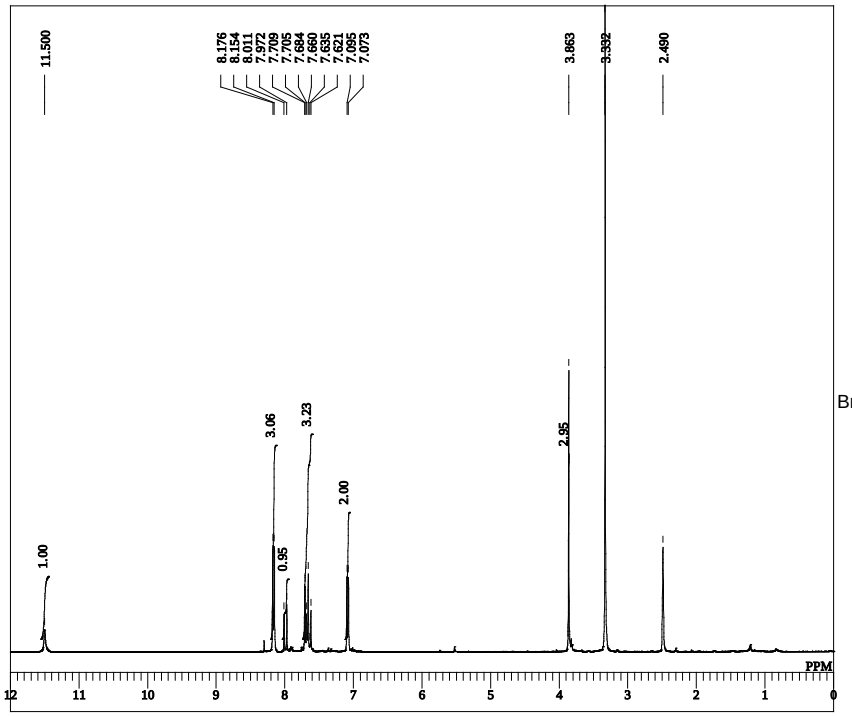
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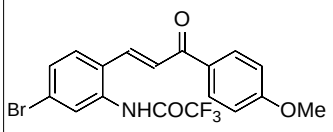
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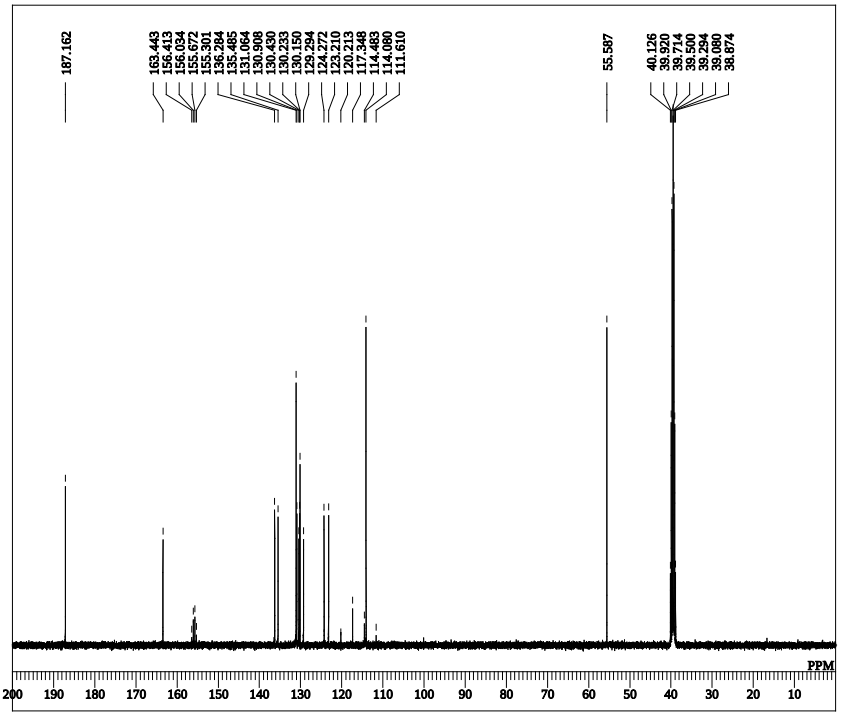
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39.500
39.294
39.080
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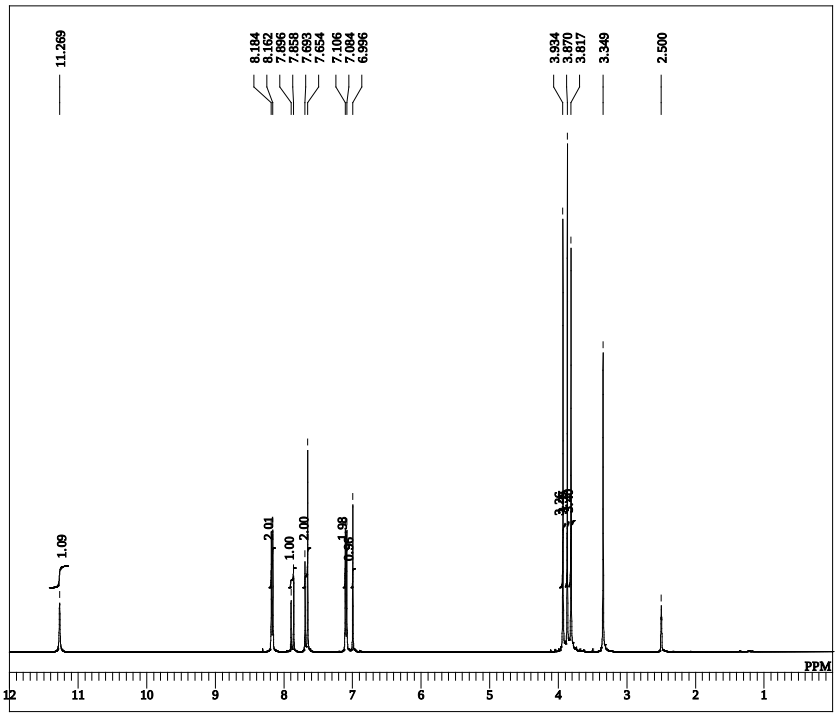
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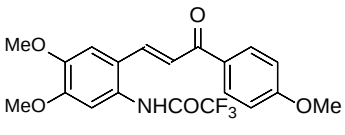
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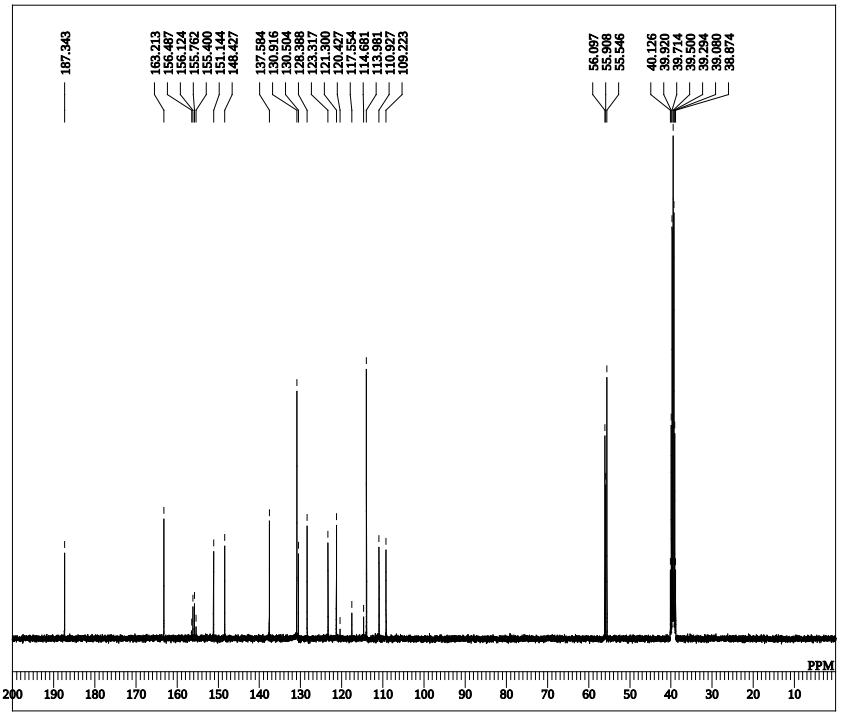
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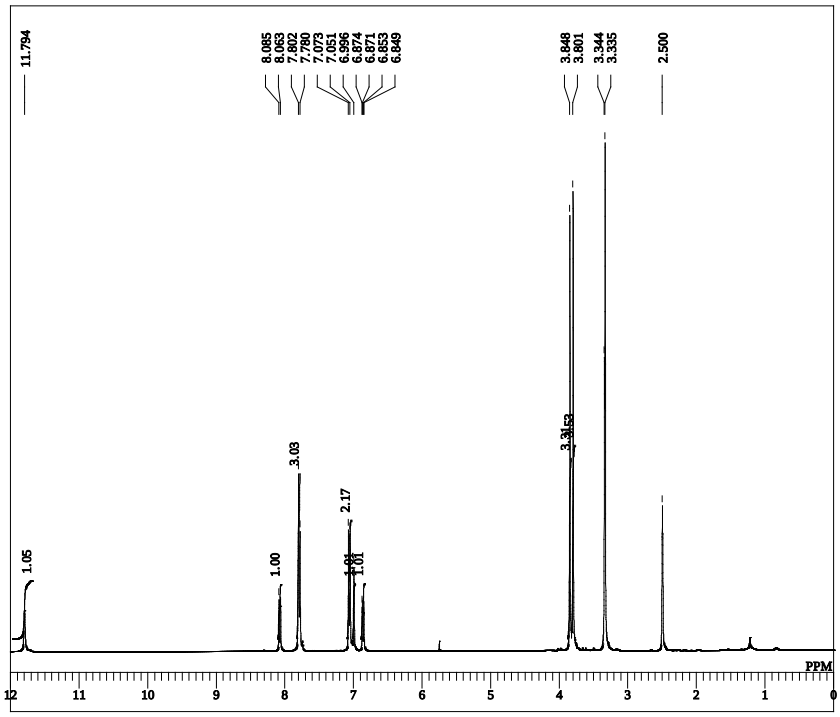
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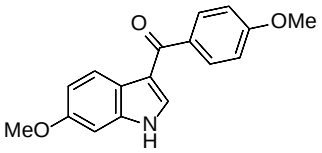
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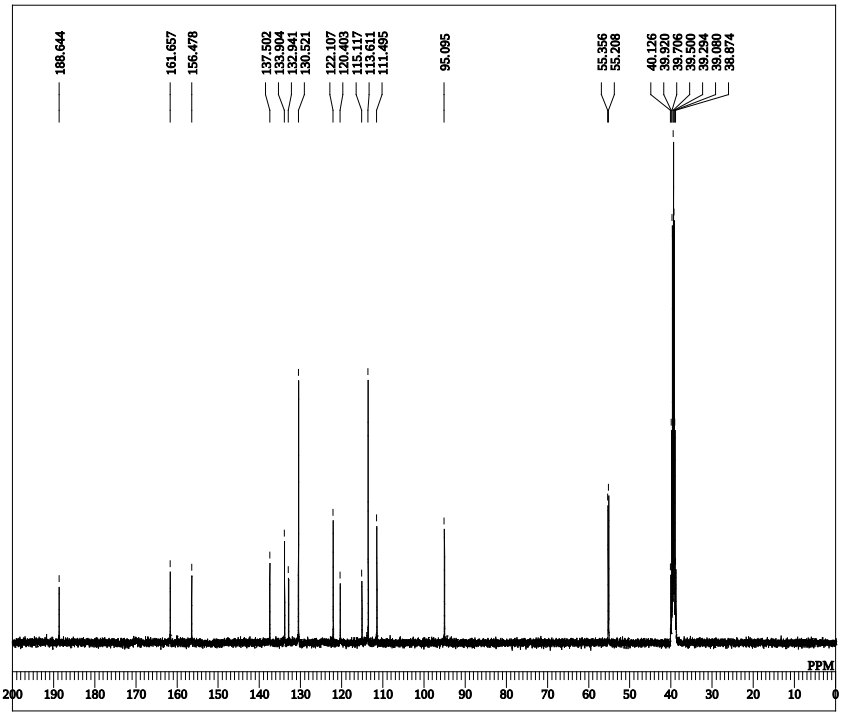
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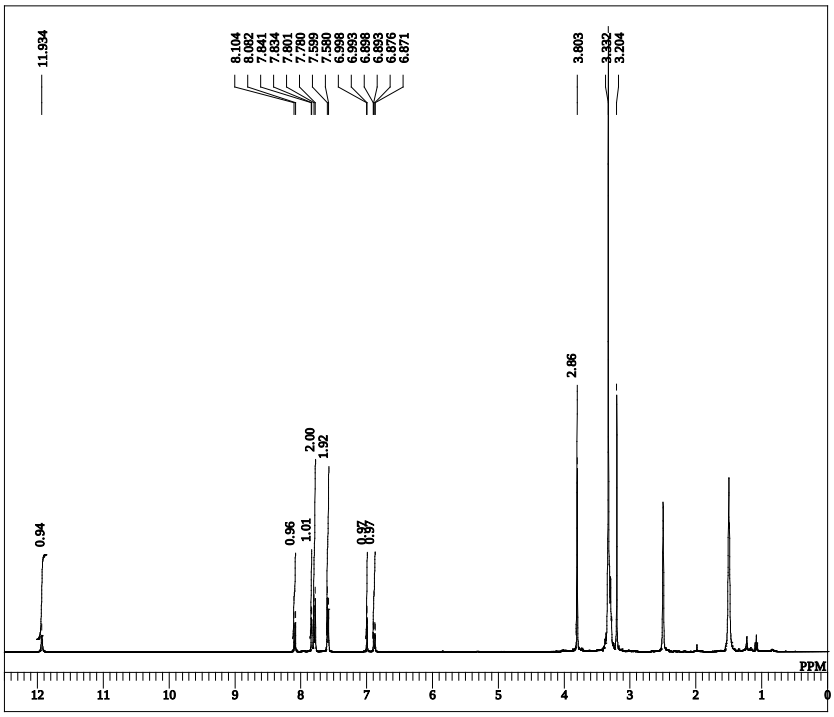
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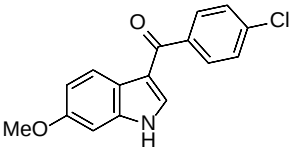
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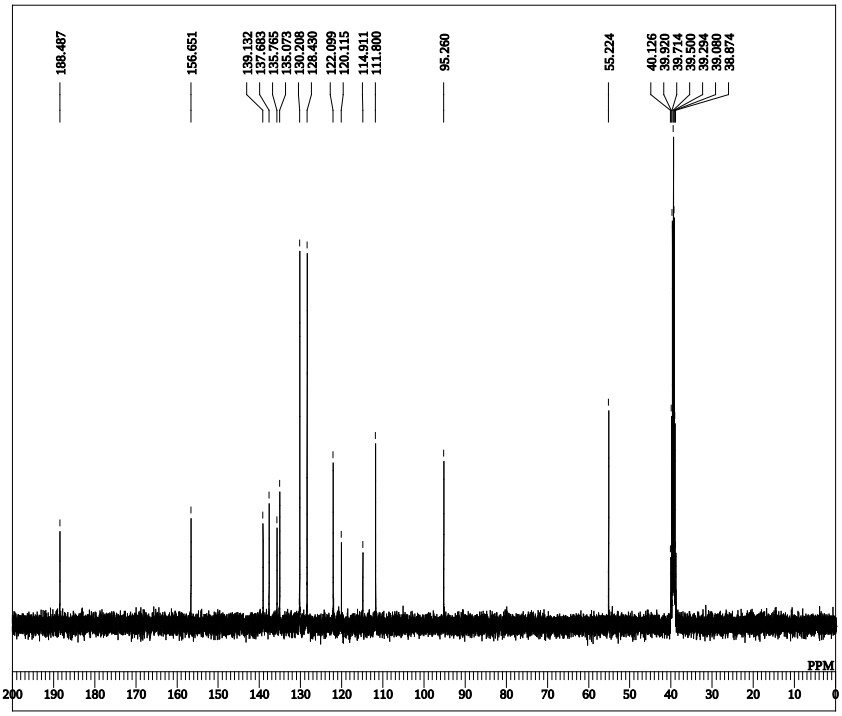
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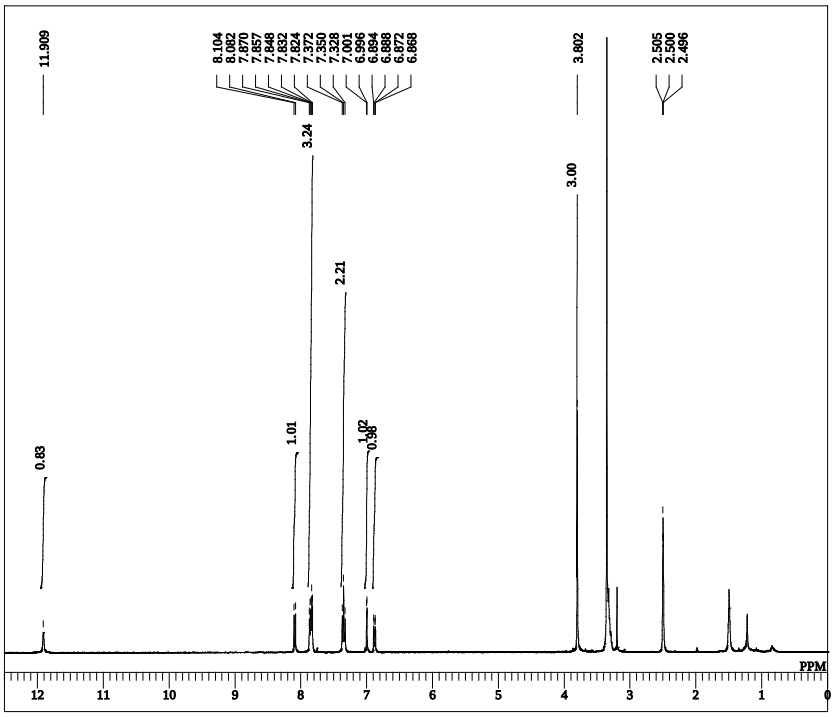
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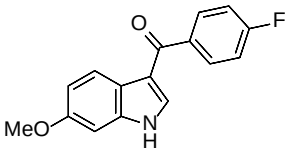
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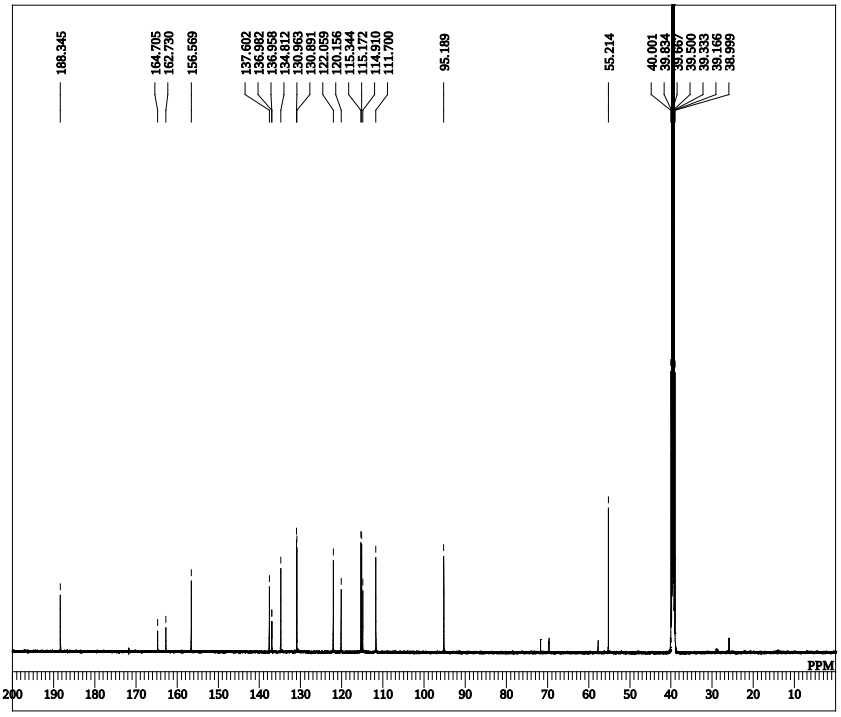
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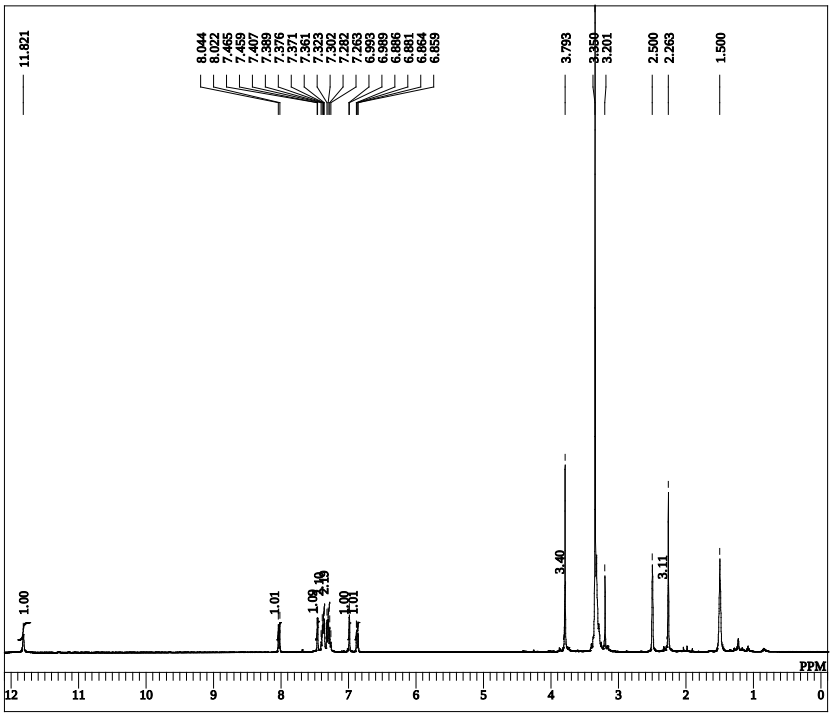
3d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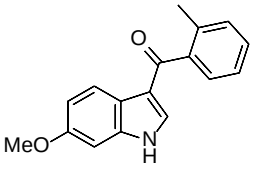
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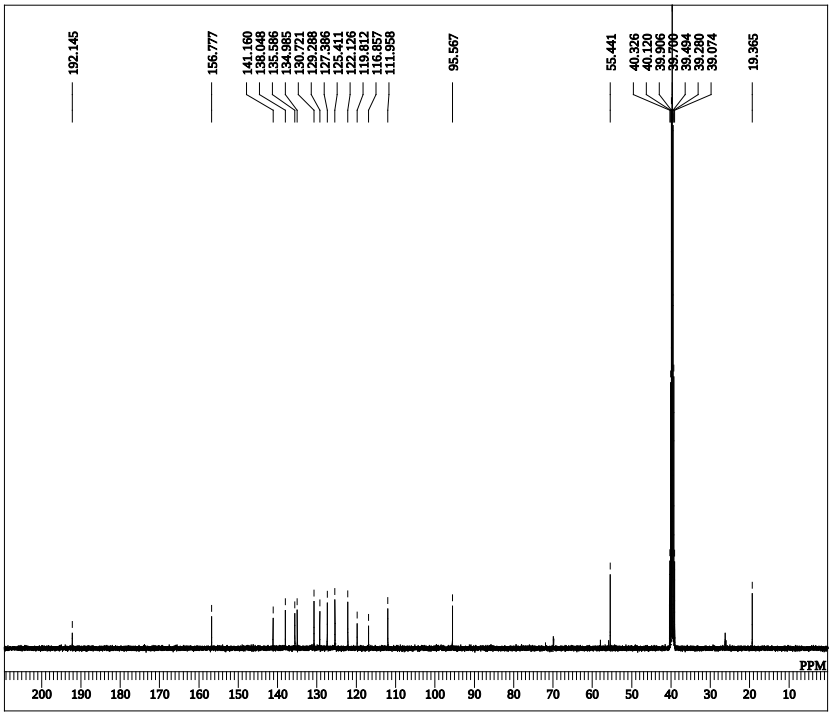
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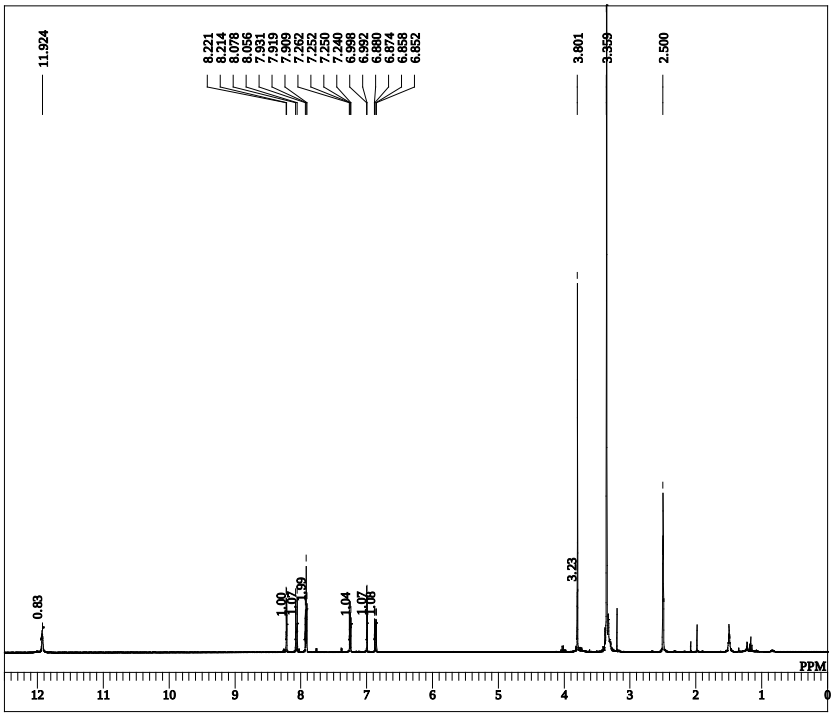
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1.07
1.39
1.04
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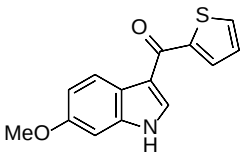
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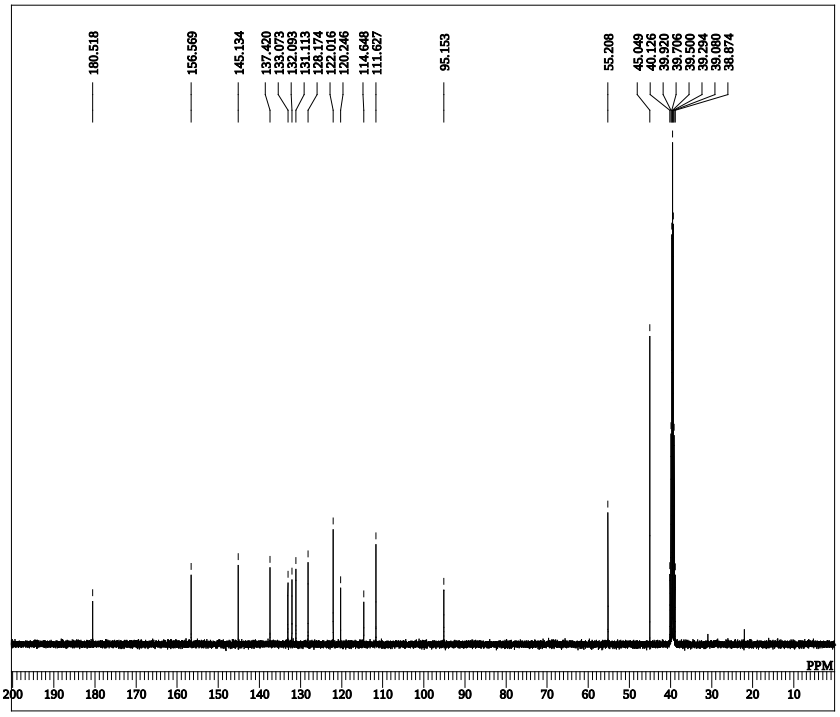
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128.714
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120.246
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95.153
55.208
45.049
40.126
39.920
39.706
39.500
39.294
39.080
38.874
PPM
200
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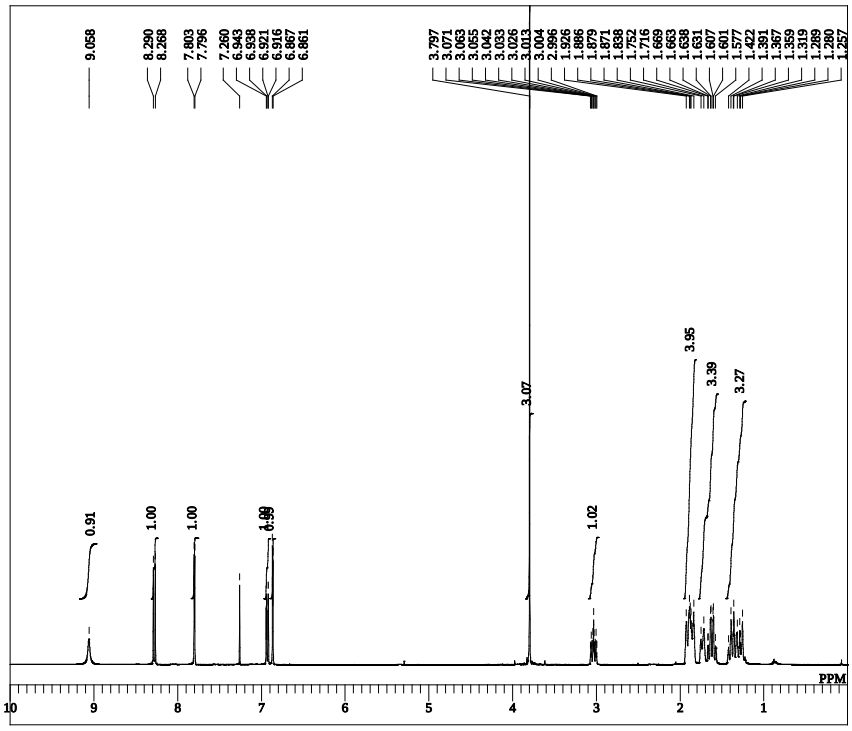
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6.861
3.797
3.071
3.063
3.055
3.042
3.033
3.026
3.013
3.004
2.996
1.926
1.886
1.879
1.871
1.838
1.752
1.716
1.669
1.663
1.638
1.631
1.607
1.601
1.577
1.422
1.391
1.367
1.359
1.319
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1.257
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3.39
3.27
3.07
1.02
0.91
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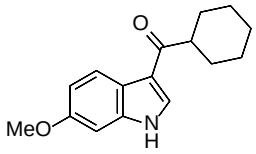
1.00
PPM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MeO
N
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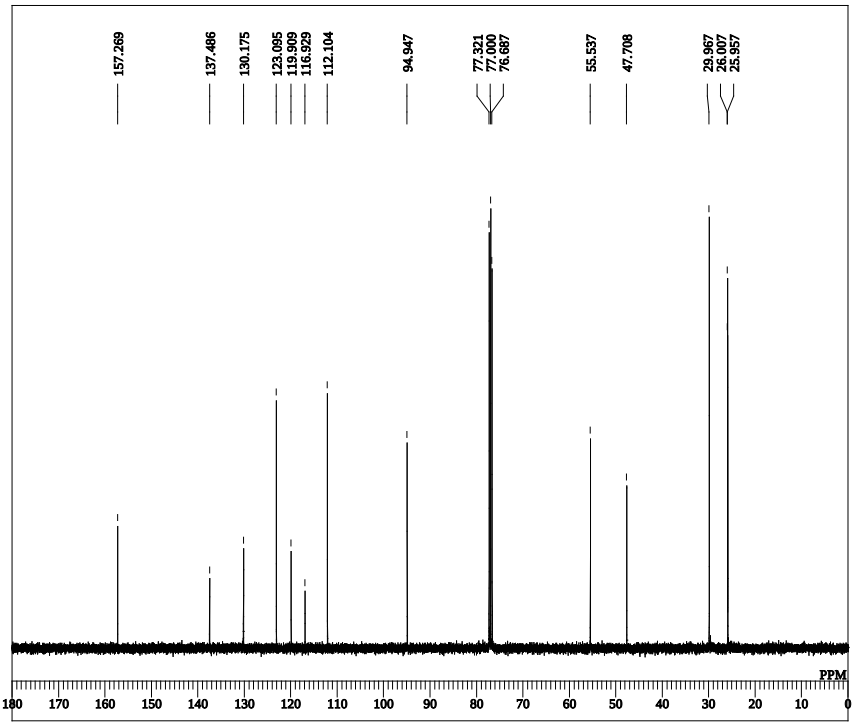
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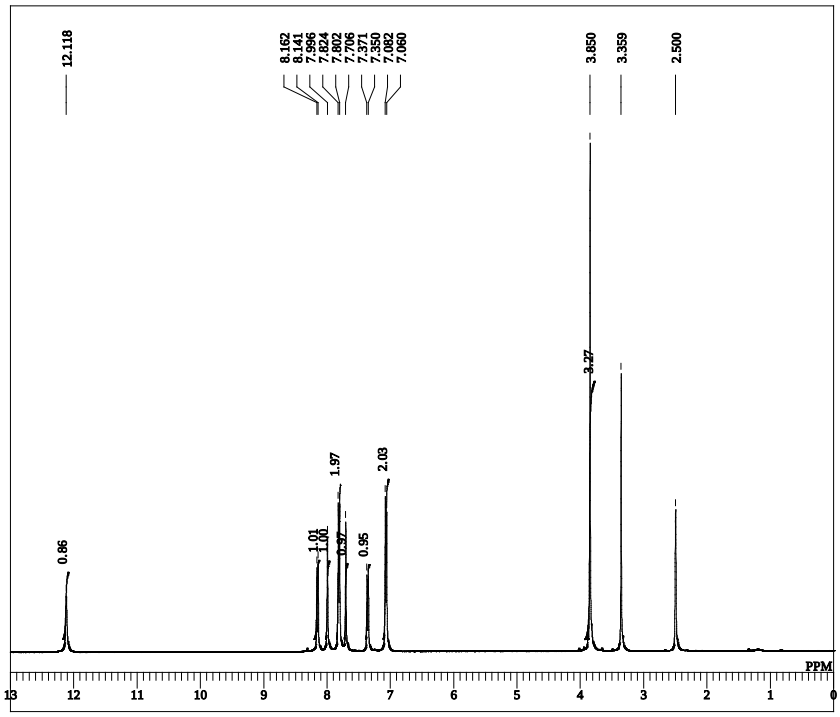
29.967
26.007
25.957
PPM

3i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8.141
7.996
7.824
7.802
7.706
7.371
7.350
7.082
7.060
3.850
3.359
2.500
3.27
2.03
1.97
1.01
1.00
0.97
0.95
0.86
PPM
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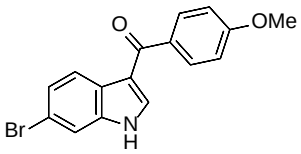
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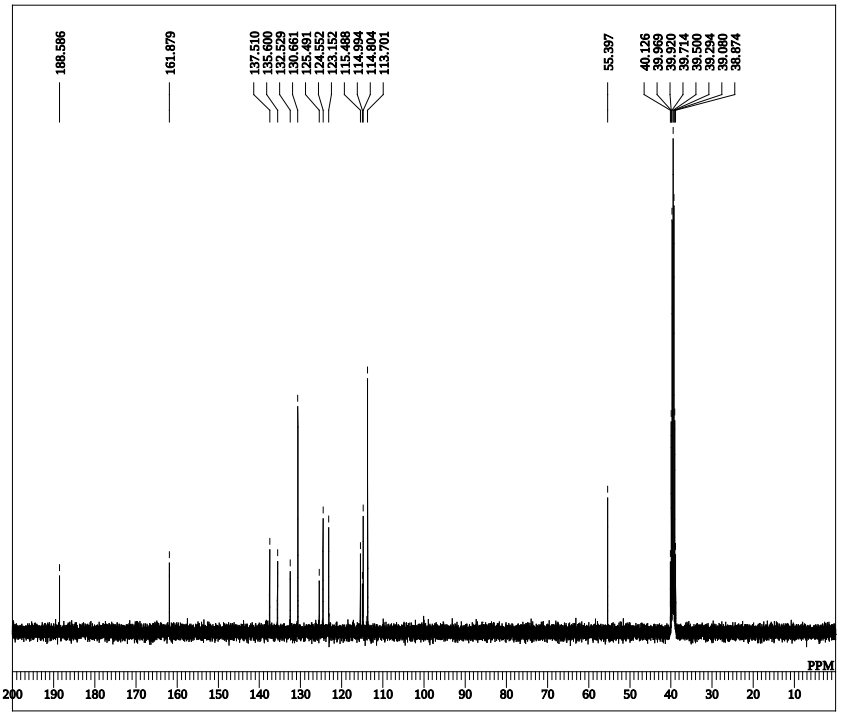
OMe
Br
N
H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113.701
55.397
40.126
39.969
39.920
39.714
39.500
39.294
39.080
38.874
PPM
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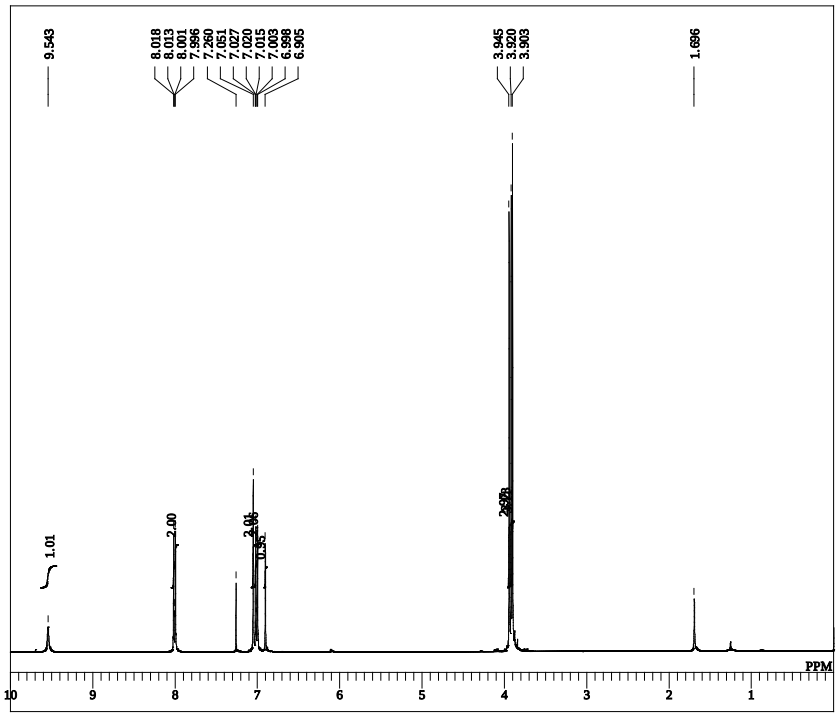
3j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8.013
8.001
7.996
7.260
7.051
7.027
7.020
7.015
7.003
6.998
6.905
3.945
3.920
3.903
1.696
1.01
2.00
2.01
2.06
10.83
29.93
PPM
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8
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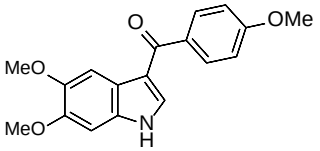
2
1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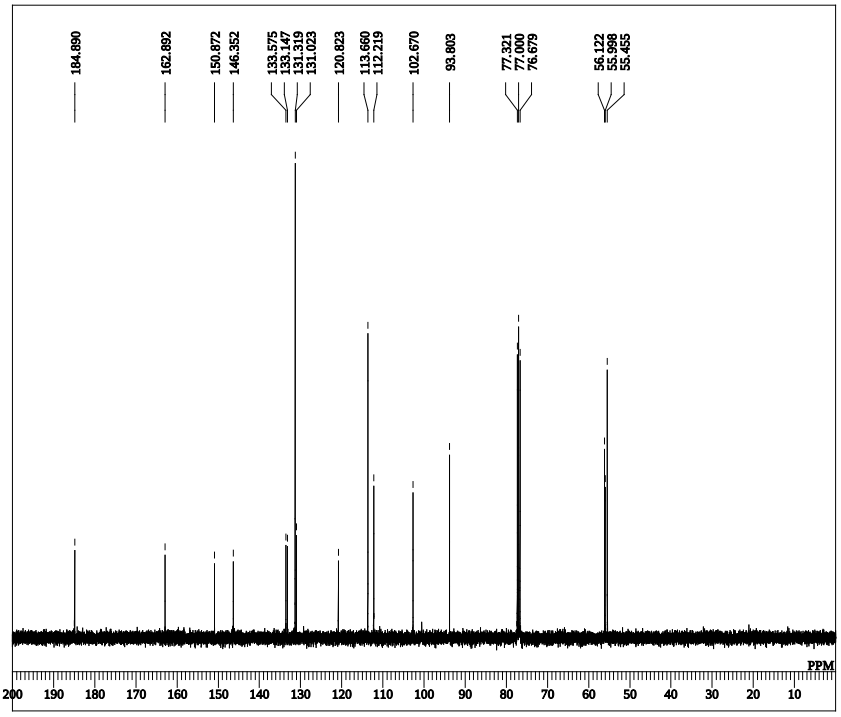
OMe
MeO
MeO
N
H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131.023
120.823
113.660
112.219
102.670
93.803
77.321
77.000
76.679
56.122
55.598
55.455
PPM
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150
140
130
120
110
100
90
80
70
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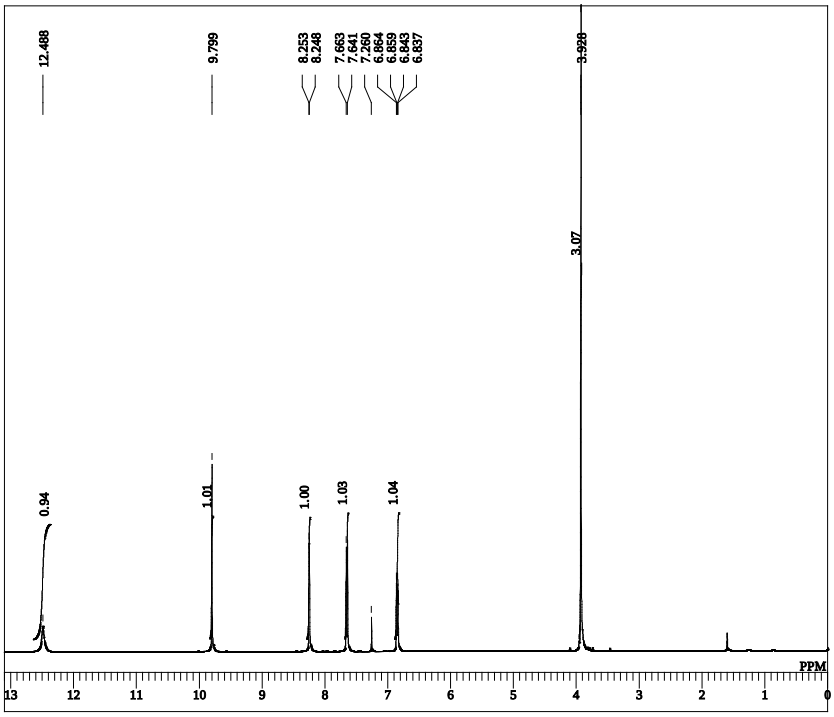
50
40
30
20
10

5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6.864
6.859
6.843
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3.07
0.94
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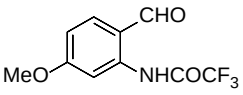
1.00
1.03
1.04
PPM
13 12 11 10 9 8 7 6 5 4 3 2 1 0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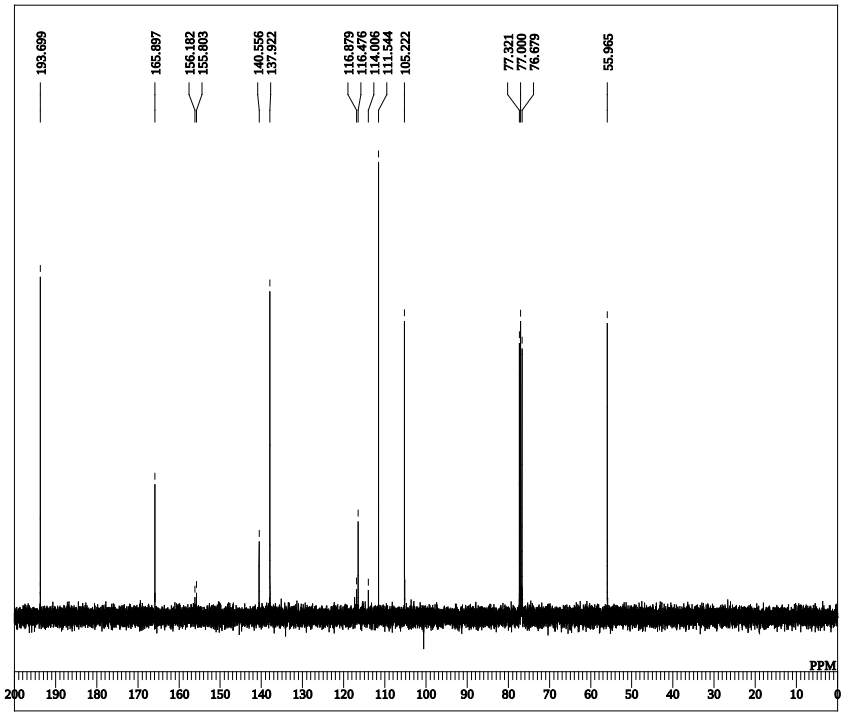
MeO
NHCOCF3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140.556
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77.000
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PPM
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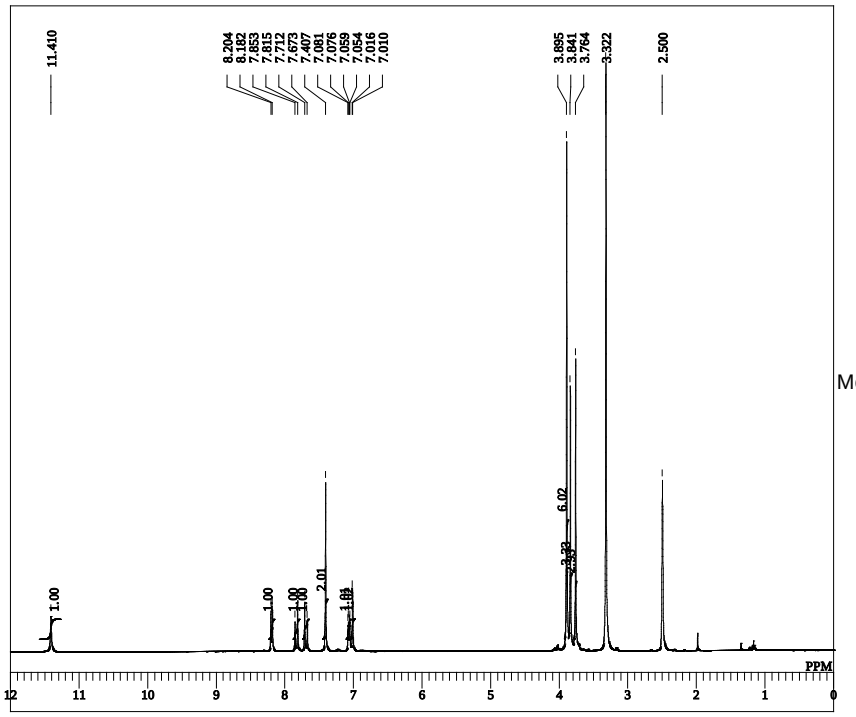
7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7.815
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7.673
7.407
7.081
7.076
7.059
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7.016
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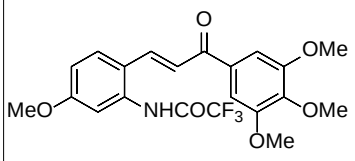
2.500
6.02
2.01
1.00
PPM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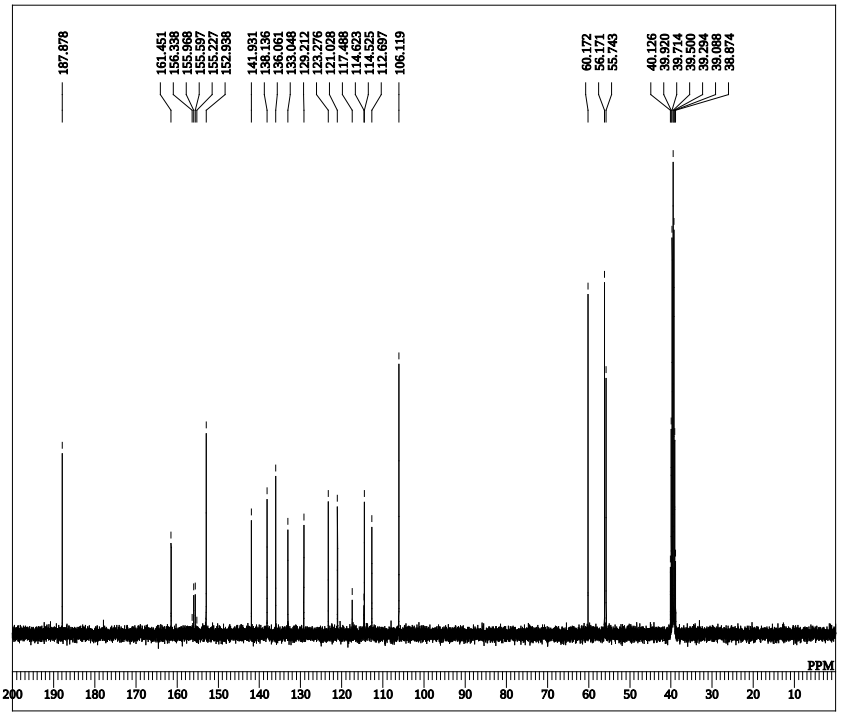
OMe
MeO
NHCOCF3
OMe
OMe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152.938
141.931
138.136
136.061
133.048
129.712
123.276
121.028
117.488
114.623
114.525
112.697
106.119
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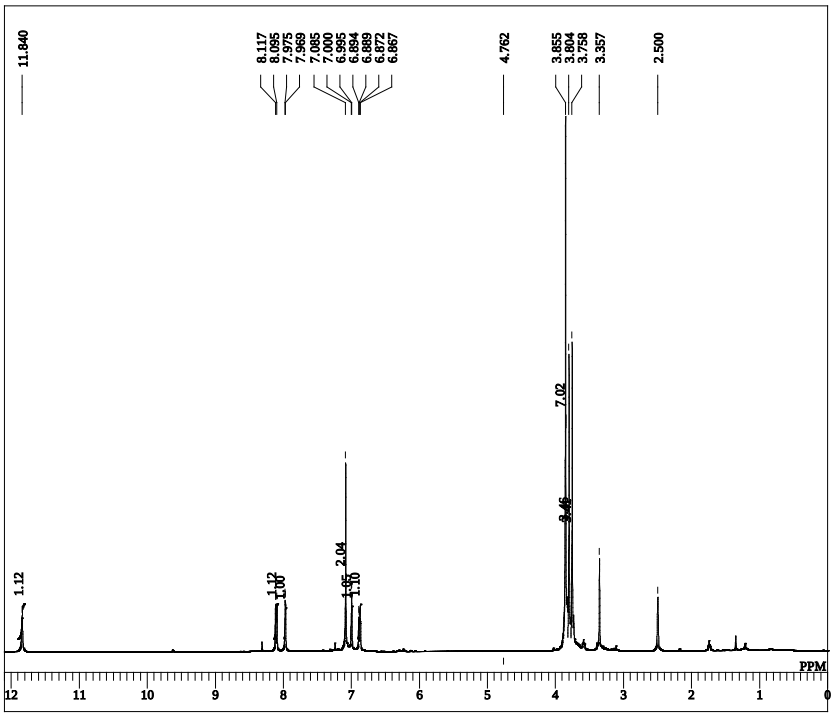
39.920
39.714
39.500
39.294
39.088
38.874
PPM

SCB01A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6.995
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6.889
6.877
6.867
4.762
3.855
3.804
3.758
3.357
2.500
7.02
2.04
1.12
1.07
1.00
1.05
1.10
PPM
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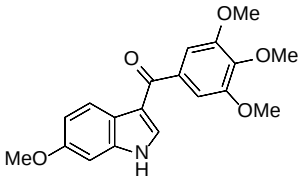
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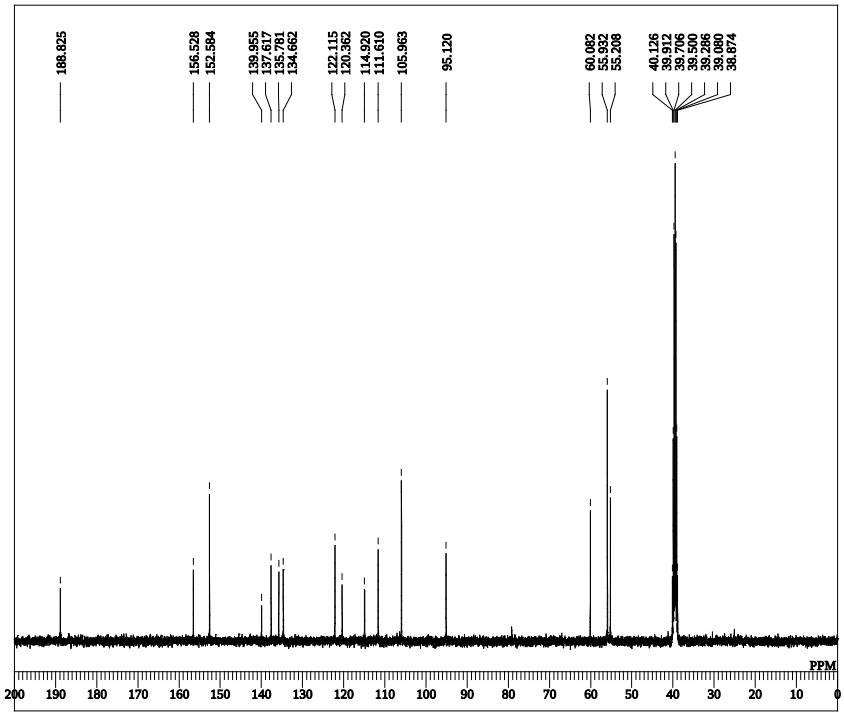
O
OMe
OMe
MeO
N
H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105.963
95.120
60.082
55.932
55.208
40.126
39.912
39.706
39.500
39.286
39.080
38.874
PPM
200
190
180
170
160
150
140
130
120
110
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50
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0