Supporting Information

Solvent Controlled Three-Component Reaction of Diazo Compounds for the Synthesis of Hydrazone Compounds under Brønsted Acid Catalysis

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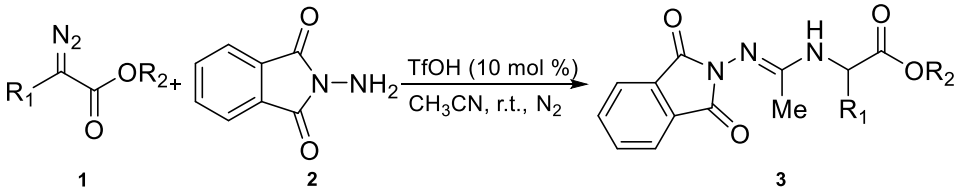
1. General Information and Materials

All ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) and ^{19}F NMR (376 MHz) spectra were recorded on Brucker spectrometers in DMSO-d_6. Chemical shifts (δ) for NMR were quoted in ppm relative to the solvent peak (2.50 ppm for ^{1}H and 40.00 ppm for ^{13}C in DMSO-d_6). Chemical shifts are reported in parts per million as follows: chemical shift, multiplicity (s =singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Coupling constants J are recorded in Hz. High-resolution mass spectra (HRMS) were reported from the Thermo Orbitrap Elite or Bruker Daltonics APEXII 47e FT-ICR instrument with an ESI source. Single crystal X-ray diffraction data (**3n** and **4k**) was recorded on Bruker-AXS SMART APEX II single crystal X-ray diffractometer.

Unless otherwise noted, all reactions were carried out under nitrogen in a flamedried or oven-dried flask containing magnetic stir bar. Diazo compounds **1a–t** were prepared according to literature reported procedure[1]. *N*-aminophthalimide (**2**) was purchased from Bidepharm.com and were used directly without further purification. The other materials obtained from commercial suppliers were used directly without further purification. Reactions were monitored by thin layer chromatography (TLC) using pre-coated silica gel plates (GF254). Flash column chromatography was performed on silica gel (particle size 200-300 mesh ASTM) and eluted with petroleum ether/ethylacetate. Solvents for the column chromatography were distilled before used.

2. General Experimental Procedures

2.1 General procedure to synthesize 3:

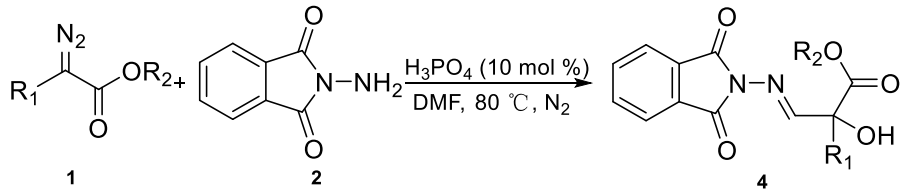


1 2 3

α-Diazo ester **1** (0.26 mmol), *N*-aminophthalimide **2** (32.4 mg, 0.20 mmol) and

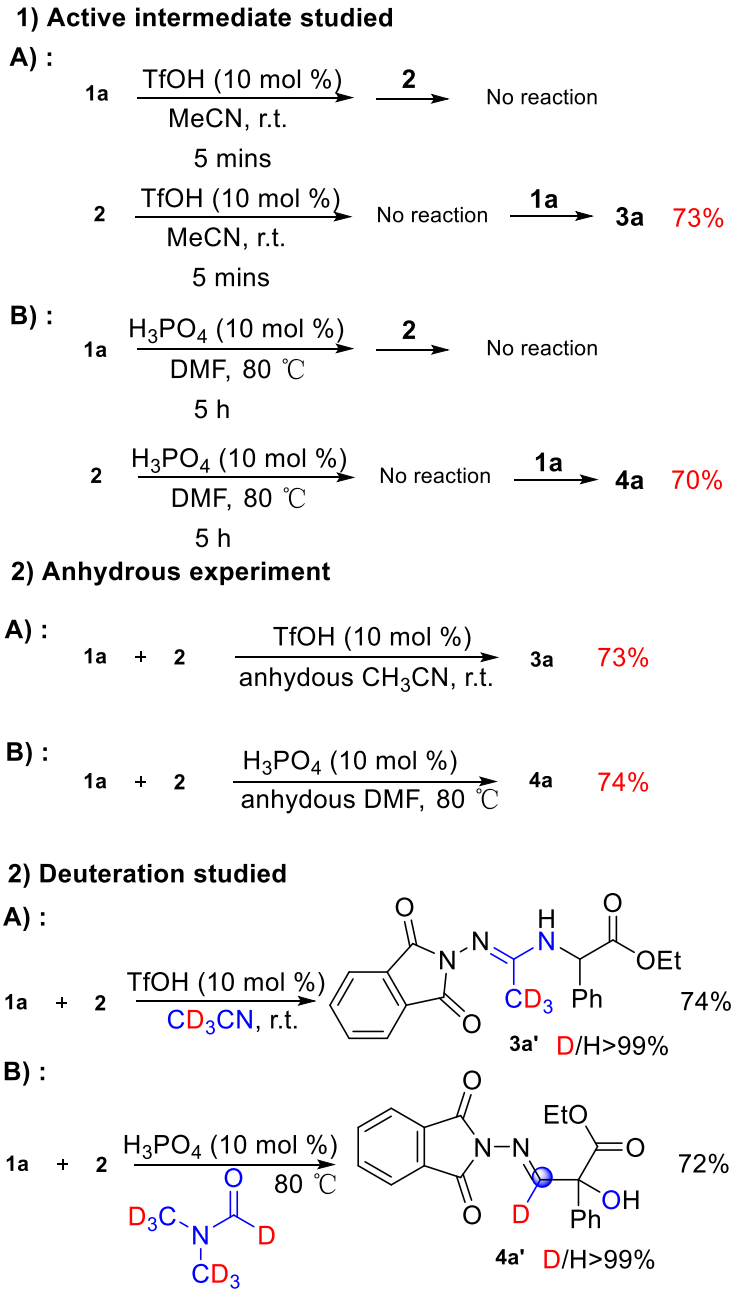
CH_3CN (1 mL) were added into a 10 mL glass tube. Then a solution of CF_3SO_3H (3.0 mg, 0.02 mmol, 10 mol %) dissolved in CH_3CN (1 mL) was introduced into the reaction mixture. The resulting mixture was continually stirred under nitrogen atmosphere at room temperature for 5 mins. The reaction solution was quenched with saturated aq. NaCl and extracted with CH_2Cl_2 (5 mL×3). The combined organic phase was dried over anhydrous Na_2SO_4, filtrated and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford the pure product **3**.

2.2 General procedure to synthesize 4:



α-Diazo ester **1** (0.40 mmol), *N*-aminophthalimide **2** (32.4 mg, 0.20 mmol) and DMF (1 mL) were added into a 10 mL glass tube. Then a solution of H_3PO_4 (3.0 mg, 0.02 mmol, 10 mol %) dissolved in DMF (1 mL) was introduced into the reaction mixture. The resulting mixture was continually stirred under nitrogen atmosphere at 80 ℃ for 5 hours. The reaction solution was quenched with saturated aq. NaCl and extracted with CH_2Cl_2 (5 mL×3). The combined organic phase was dried over anhydrous Na_2SO_4, filtrated and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford the pure product **4**.

3. Mechanistic Studies



Scheme 4. Mechanistic studies

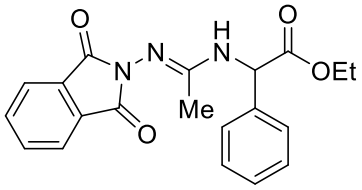
4. References

(1) (a) M. Hu, J. Rong, W. Miao, C. Ni, Y. Han and J. Hu, Copper-mediated trifluoromethylthiolation of alpha-diazoesters, *Org Lett*, 2014, **16**, 2030-2033; (b) J. X. Guo, T. Zhou, B. Xu, S. F. Zhu and Q. L. Zhou, Enantioselective synthesis of alpha-

alkenyl alpha-amino acids via N-H insertion reactions, *Chem Sci*, 2016, **7**, 1104-1108.

5. Characterization Data of Compounds

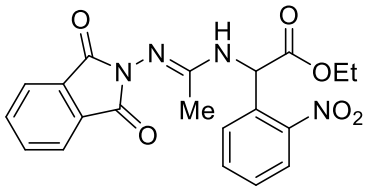
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-phenylacetate (3a)



Yellow oil, yield: 73%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.99 (d, J = 6.7 Hz, 1H), 7.81 (d, J = 8.7 Hz, 4H), 7.57 – 7.47 (m, 2H), 7.46 – 7.37 (m, 3H), 5.48 (d, J = 6.6 Hz, 1H), 4.22 – 4.07 (m, 2H), 1.85 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.9, 167.0, 165.6, 136.5, 134.6, 131.0, 129.2, 128.8, 128.3, 123.1, 61.3, 58.9, 16.2, 14.4; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}N_3O_4^+$: 366.1448; found: 366.1450.

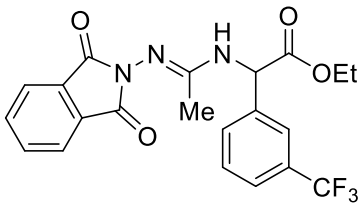
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(2-nitrophenyl)acetate (3b)



Yellow oil, yield: 63%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.27 (d, J = 7.5 Hz, 1H), 8.08 (d, J = 8.1 Hz, 1H), 7.85 – 7.76 (m, 5H), 7.72 (d, J = 7.8 Hz, 1H), 7.63 (d, J = 7.4 Hz, 1H), 6.35 (d, J = 7.5 Hz, 1H), 4.16 (q, J = 7.1 Hz, 2H), 1.88 (s, 3H), 1.18 (t, J = 7.1 Hz, 3H).; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 169.3, 166.8, 165.4, 148.8, 134.6, 134.3, 131.8, 131.0, 130.1, 129.9, 125.3, 123.1, 62.0, 54.5, 16.3, 14.3; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}N_4O_6^+$: 411.1299; found: 411.1305.

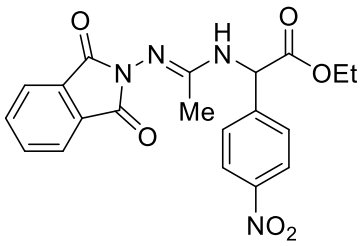
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(3-(trifluoromethyl)phenyl)acetate (3c)



Yellow oil, yield: 59%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.00 (d, J = 6.8 Hz, 1H), 7.80 (s, 4H), 7.63 (d, J = 8.5 Hz, 2H), 7.45 (d, J = 8.4 Hz, 2H), 5.50 (d, J = 6.8 Hz, 1H), 4.14 (dd, J = 12.9, 7.1 Hz, 2H), 1.85 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.5, 166.9, 165.5, 136.0, 134.7, 134.5, 132.0, 131.0, 130.5, 123.2, 123.1, 122.0, 61.4, 58.1, 16.2, 14.3; ^{19}F NMR (376 MHz, DMSO-d_6): δ = -61.1; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{19}F_3N_3O_4^+$: 434.1322; found: 434.1330.

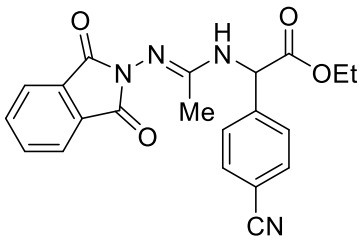
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(4-nitrophenyl)acetate (3d)



Yellow oil, yield: 82%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.28 (d, J = 8.8 Hz, 2H), 8.21 (d, J = 7.0 Hz, 1H), 7.78 (d, J = 10.4 Hz, 6H), 5.73 (d, J = 7.0 Hz, 1H), 4.16 (dd, J = 12.5, 7.1 Hz, 2H), 1.88 (s, 3H), 1.18 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 167.0, 166.9, 165.5, 147.8, 144.0, 134.6, 131.0, 129.7, 124.1, 123.2, 61.9, 58.1, 16.3, 14.3; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}N_4O_6^+$: 411.1299; found: 411.1304.

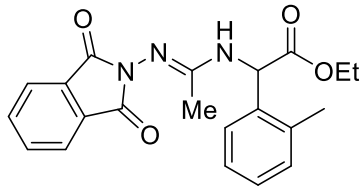
ethyl (*E*)-2-(4-cyanophenyl)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)acetate (3e)



Yellow oil, yield: 70%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.13 (d, J = 7.0 Hz, 1H), 7.92 – 7.88 (m, 2H), 7.79 (s, 4H), 7.70 (d, J = 8.2 Hz, 2H), 5.66 (d, J = 7.0 Hz, 1H), 4.15 (dd, J = 12.1, 7.1 Hz, 2H), 1.87 (s, 3H), 1.18 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.0, 166.9, 165.5, 142.0, 134.6, 133.0, 131.0, 129.3, 123.1, 118.9, 111.6, 61.7, 58.3, 16.2, 14.3; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{19}N_4O_4^+$: 391.1401; found: 391.1404.

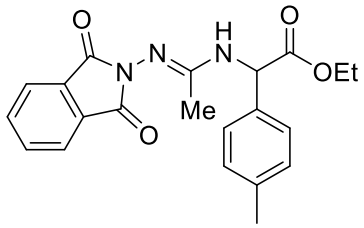
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(*o*-tolyl)acetate (3f)



Yellow oil, yield: 70%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.92 (d, J = 7.1 Hz, 1H), 7.80 (s, 4H), 7.32 – 7.23 (m, 4H), 5.76 (d, J = 7.2 Hz, 1H), 4.15 (dd, J = 9.1, 7.1 Hz, 2H), 2.41 (s, 3H), 1.83 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.2, 166.9, 165.7, 137.2, 135.4, 134.6, 131.1, 131.0, 128.7, 127.4, 126.7, 123.1, 61.3, 55.2, 19.4, 16.2, 14.4; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}N_3O_4^+$: 380.1605; found: 380.1611.

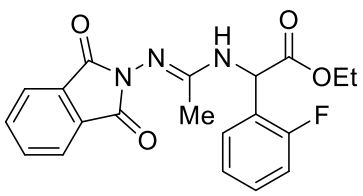
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(*p*-tolyl)acetate (3g)



Yellow oil, yield: 62%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.92 (d, J = 6.6 Hz, 1H), 7.80 (s, 4H), 7.36 (d, J = 8.0 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 5.41 (d, J = 6.6 Hz, 1H), 4.11 (dd, J = 16.4, 7.1 Hz, 2H), 2.32 (s, 3H), 1.84 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.0, 167.0, 165.6, 138.2, 134.6, 133.5, 131.0, 129.7, 128.2, 123.1, 61.2, 58.6, 21.1, 16.2, 14.4; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}N_3O_4^+$: 380.1605; found: 380.1608.

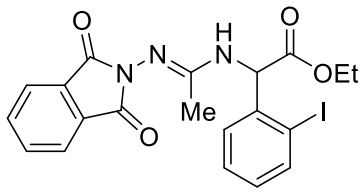
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(2-fluorophenyl)acetate (3h)



Yellow oil, yield: 71%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.04 (d, J = 7.1 Hz, 1H), 7.80 (s, 4H), 7.51 (dd, J = 8.3, 6.8 Hz, 1H), 7.45 (dd, J = 6.3, 1.8 Hz, 1H), 7.28 (t, J = 8.1 Hz, 2H), 5.82 (d, J = 7.1 Hz, 1H), 4.15 (t, J = 7.1 Hz, 2H), 1.85 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.1, 166.9, 165.6, 134.6, 131.0, 130.9, 129.9, 129.8, 125.2, 125.2, 124.0, 123.1, 116.2, 116.0, 61.5, 51.9, 16.2, 14.3; ^{19}F NMR (376 MHz, DMSO-d_6): δ = -117.4 – -117.5 (m); HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{20}H_{19}FN_3O_4^+$: 384.1354; found: 384.1358.

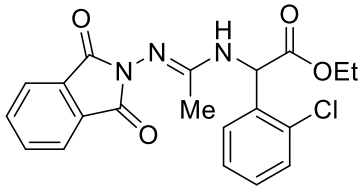
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(2-iodophenyl)acetate (3i)



Yellow oil, yield: 74%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.97 – 7.92 (m, 2H), 7.80 (s, 4H), 7.51 – 7.42 (m, 2H), 7.14 (td, J = 7.8, 1.8 Hz, 1H), 5.74 (d, J = 6.6 Hz, 1H), 4.15 (dd, J = 9.2, 7.1 Hz, 2H), 1.84 (s, 3H), 1.19 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.5, 166.8, 165.6, 140.18, 139.5, 134.6, 131.1, 130.8, 129.2, 128.7, 123.1, 101.9, 62.9, 61.5, 16.2, 14.3; HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{20}H_{19}IN_3O_4^+$: 492.0415; found: 492.0407.

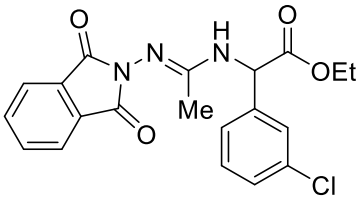
ethyl (*E*)-2-(2-chlorophenyl)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)acetate (3j)



Yellow oil, yield: 70%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.01 (d, J = 7.0 Hz, 1H), 7.80 (s, 4H), 7.57 – 7.49 (m, 2H), 7.44 – 7.38 (m, 2H), 5.95 (d, J = 7.0 Hz, 1H), 4.15 (dtd, J = 8.6, 4.9, 4.7, 3.7 Hz, 2H), 1.85 (s, 3H), 1.18 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.2, 166.9, 165.5, 134.5, 134.5, 133.7, 131.0, 130.5, 130.1, 129.6, 128.0, 123.1, 61.5, 55.5, 16.2, 14.3; HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{20}H_{19}F_3ClN_3O_4^+$: 400.1059; found: 400.1068.

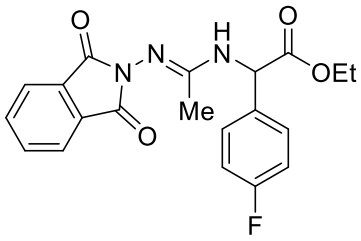
ethyl (*E*)-2-(3-chlorophenyl)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)acetate (3k)



Yellow oil, yield: 65%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.98 (d, J = 6.7 Hz, 1H), 7.80 (s, 4H), 7.49 – 7.39 (m, 4H), 5.46 (d, J = 6.6 Hz, 1H), 4.22 – 3.98 (m, 2H), 1.85 (s, 3H), 0.77 (d, J = 4.7 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.4, , 167.0, 165.6, 138.9, 134.6, 133.7, 131.0, 128.8, 128.1, 127.2, 123.1, 61.6, 58.2, 16.2, 14.3; HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{20}H_{19}ClN_3O_4^+$: 400.1059; found: 400.1063.

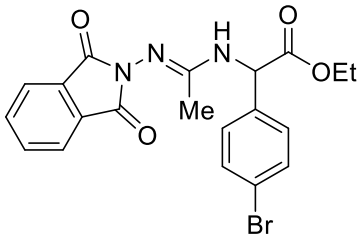
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-(4-fluorophenyl)acetate (3l)



Yellow oil, yield: 68%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.00 (d, J = 6.6 Hz, 1H), 7.80 (s, 4H), 7.54 (dd, J = 8.6, 5.5 Hz, 2H), 7.26 (t, J = 8.8 Hz, 2H), 5.49 (d, J = 6.6 Hz, 1H), 4.13 (qd, J = 10.7, 7.1 Hz, 2H), 1.85 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.8, 166.9, 165.6, 134.6, 132.8, 131.0, 130.5, 130.5, 123.1, 116.0, 115.9, 61.4, 58.1, 16.2, 14.3; ^{19}F NMR (376 MHz, DMSO-d_6): δ = -113.65 – -113.87 (m); HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}FN_3O4^+$: 384.1354; found: 384.1367.

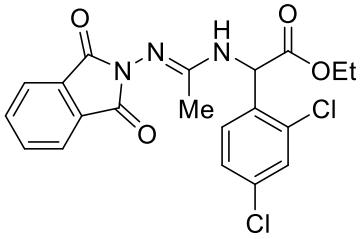
ethyl (*E*)-2-(4-bromophenyl)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)acetate (3m)



Yellow oil, yield: 57%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.12 (d, J = 6.7 Hz, 1H), 7.87 – 7.67 (m, 8H), 5.67 (d, J = 6.8 Hz, 1H), 4.16 (dd, J = 10.3, 7.2 Hz, 2H), 1.87 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.3, 166.9, 165.5, 137.9, 134.5, 132.5, 131.0, 130.2, 125.5, 124.7, 123.1, 61.6, 58.1, 16.2, 14.2; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}BrN_3O4^+$: 444.0553; found: 444.0557.

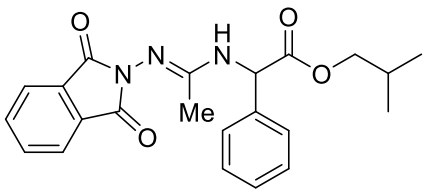
ethyl (*E*)-2-(2,4-dichlorophenyl)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)acetate (3n)



Yellow oil, yield: 71%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.11 (d, J = 7.2 Hz, 1H), 7.79 (s, 4H), 7.71 (d, J = 0.7 Hz, 1H), 7.53 (d, J = 1.5 Hz, 2H), 5.92 (d, J = 7.0 Hz, 1H), 4.17 (dt, J = 7.2, 2.9 Hz, 2H), 1.85 (s, 3H), 1.18 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 169.8, 166.8, 165.5, 134.7, 134.6, 134.2, 133.9, 131.0, 131.0, 129.6, 128.2, 123.1, 61.8, 55.1, 16.3, 14.3; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}Cl_2N_3O_4^+$: 434.0669; found: 434.0676.

isobutyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-phenylacetate (3o)

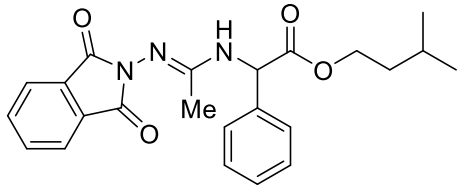


Yellow oil, yield: 57%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.99 (d, J = 6.8 Hz, 1H), 7.80 (s, 4H), 7.51 (dd, J = 8.1, 1.3 Hz, 2H), 7.45 – 7.38 (m, 3H), 5.52 (d, J = 6.8 Hz, 1H), 3.87 (dd, J = 6.5, 1.3 Hz, 2H), 1.86 (s, 3H), 0.79 (dd, J = 6.7, 5.4 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.9, 167.0, 165.6, 136.6, 134.5, 131.0, 129.1, 128.8 128.3, 123.1, 70.9, 58.8, 27.6, 19.1, 19.1, 16.1; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{24}N_3O_4^+$:

394.1761; found: 394.1768.

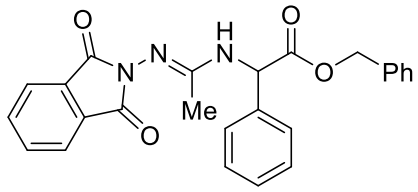
isopentyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-phenylacetate (3p)



Yellow oil, yield: 52%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.96 (d, J = 6.7 Hz, 1H), 7.80 (s, 4H), 7.51 – 7.47 (m, 2H), 7.39 (ddd, J = 12.4, 8.3, 5.5 Hz, 3H), 5.47 (d, J = 6.6 Hz, 1H), 4.11 (ddd, J = 17.5, 10.9, 4.4 Hz, 2H), 1.85 (s, 3H), 1.54 (dt, J = 13.3, 6.6 Hz, 1H), 1.43 (q, J = 6.8 Hz, 2H), 0.78 (dd, J = 6.6, 2.0 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.9, 166.9, 165.5, 136.4, 134.5, 131.0, 129.1, 128.8, 128.3, 123.1, 63.6, 58.9, 37.1, 24.7 22.5, 16.1; HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{23}H_{26}N_3O_4^+$: 408.1918; found: 408.1924.

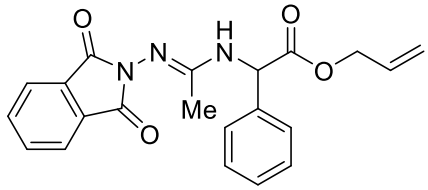
benzyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-phenylacetate (3q)



Yellow oil, yield: 49%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.04 (d, J = 6.5 Hz, 1H), 7.85 – 7.78 (m, 4H), 7.51 (dd, J = 7.8, 1.6 Hz, 2H), 7.44 – 7.38 (m, 3H), 7.30 – 7.24 (m, 5H), 5.57 (d, J = 6.5 Hz, 1H), 5.18 (dd, J = 56.4, 12.6 Hz, 2H), 1.88 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.9, 167.1, 165.6, 136.4, 136.2, 134.6, 131.0, 129.1, 128.9, 128.7, 128.3, 128.1, 123.1, 66.5, 59.0, 40.6, 16.2; HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{25}H_{22}N_3O_4^+$: 428.1605; found: 428.1607.

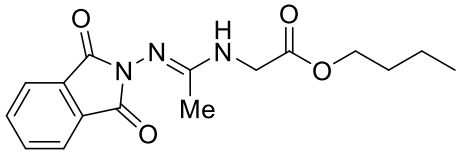
allyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido)-2-phenylacetate (3r)



Yellow oil, yield: 60%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.01 (d, J = 6.7 Hz, 1H), 7.80 (s, 4H), 7.51 (dd, J = 8.1, 1.4 Hz, 2H), 7.45 – 7.39 (m, 3H), 5.88 (ddd, J = 21.2, 10.6, 5.3 Hz, 1H), 5.54 (d, J = 6.7 Hz, 1H), 5.15 (ddd, J = 13.1, 10.5, 8.5 Hz, 2H), 4.61 (ddt, J = 5.5, 2.8, 1.6 Hz, 2H), 1.86 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.6, 167.0, 165.6, 136.3, 134.5, 132.8, 131.0, 129.1, 128.4, 123.1, 118.0, 65.5, 58.8, 39.6, 16.2; HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{21}H_{20}N_3O_4^+$: 378.1448; found: 378.1459.

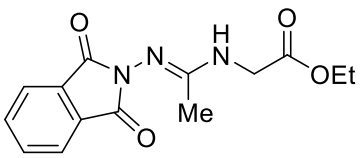
butyl (*E*)-(1-((1,3-dioxoisoindolin-2-yl)imino)ethyl)glycinate (3s)



Yellow oil, yield: 56%

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.79 (s, 4H), 7.69 (s, 1H), 4.09 (t, J = 6.6 Hz, 2H), 4.00 (d, J = 5.9 Hz, 2H), 1.79 (s, 3H), 1.62 – 1.55 (m, 2H), 1.35 (dq, J = 14.7, 7.4 Hz, 3H), 0.88 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.1, 167.2, 165.7, 134.5, 131.0, 123.1, 64.8, 43.2, 30.6, 19.0, 16.4, 14.0; HRMS (ESI): *m/z* [M+H]$^+$ calcd for $C_{16}H_{20}N_3O_4^+$: 318.1448; found: 318.1453.

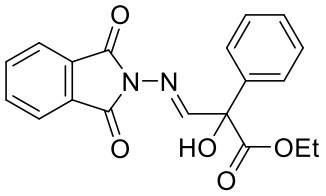
ethyl (*E*)-(1-((1,3-dioxoisoindolin-2-yl)imino)ethyl)glycinate (3t)



White oil, yield: 69%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.79 (d, J = 5.3 Hz, 4H), 7.67 (s, 1H), 4.13 (q, J = 7.1 Hz, 2H), 3.99 (d, J = 5.8 Hz, 2H), 1.79 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.0, 167.3, 165.7, 134.5, 131.0, 123.1, 60.8, 55.3, 43.3, 16.4, 14.5; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{14}H_{16}N_3O_4^+$: 290.1135; found: 290.1137.

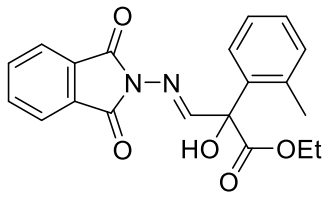
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-phenylpropanoate (4a)



Yellow oil, yield: 70%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.03 (s, 1H), 7.93 – 7.86 (m, 4H), 7.57 – 7.53 (m, 2H), 7.44 – 7.34 (m, 3H), 6.90 (s, 1H), 4.25 – 4.14 (m, 2H), 1.20 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.2, 164.8, 162.3, 139.6, 135.4, 130.3, 128.6, 128.6, 126.7, 123.9, 79.4, 62.0, 14.3; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{19}H_{16}N_2NaO_5^+$: 375.0951; found: 375.0952.

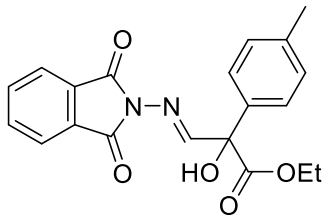
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-(*o*-tolyl)propanoate (4b)



White oil, yield: 76%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.04 (s, 1H), 7.95 – 7.86 (m, 4H), 7.47 (d, J = 7.6 Hz, 1H), 7.24 (dt, J = 13.7, 5.0 Hz, 3H), 6.76 (s, 1H), 4.28 – 4.15 (m, 2H), 2.31 (s, 3H), 1.20 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.4, 164.8, 162.5, 138.4, 137.1, 135.4, 132.1, 130.3, 128.5, 127.4, 125.8, 123.9, 80.3, 61.9, 20.4, 14.3; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{20}H_{18}N_2NaO_5^+$:389.1108; found: 389.1115.

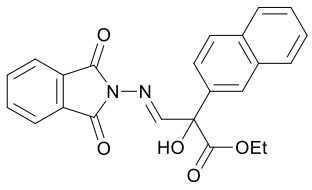
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-(*p*-tolyl)propanoate (4c)



White oil, yield: 75%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.00 (s, 1H), 7.94 – 7.87 (m, 4H), 7.42 (d, J = 8.2 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 6.78 (s, 1H), 4.18 (dd, J = 10.4, 7.1 Hz, 2H), 2.31 (s, 3H), 1.20 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.2, 164.8, 162.5, 137.9, 136.7, 135.4, 130.3, 129.2, 126.5, 123.9, 79.2, 61.9, 21.0, 14.3; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{20}H_{18}NaN_2O_5^+$: 389.1108; found: 389.1093.

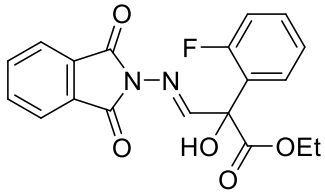
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-(*p*-tolyl)propanoate (4d)



White oil, yield: 57%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.21 (s, 1H), 8.30 – 8.25 (m, 1H), 7.93 (tdd, J = 5.2, 4.4, 2.5 Hz, 6H), 7.72 – 7.69 (m, 1H), 7.52 (dt, J = 7.1, 2.9 Hz, 3H), 7.10 (s, 1H), 4.22 – 4.08 (m, 2H), 1.09 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.7, 164.8, 162.4, 136.2, 135.5, 134.4, 131.0, 130.4, 129.7, 129.1, 126.5, 126.2, 125.8, 125.5, 125.2, 124.0, 80.6, 61.9, 14.3; HRMS (ESI): *m/z* $[M+K]^+$ calcd for $C_{23}H_{18}KN_2O_5^+$: 441.0847; found: 441.0832.

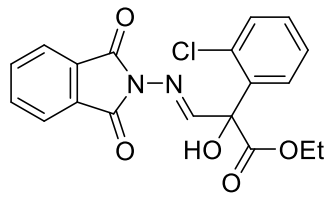
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-(2-fluorophenyl)-2-hydroxypropanoate (4e)



White oil, yield: 63%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.13 (d, J = 1.4 Hz, 1H), 7.92 – 7.85 (m, 4H), 7.65 (td, J = 7.7, 1.6 Hz, 1H), 7.46 – 7.40 (m, 1H), 7.29 – 7.20 (m, 2H), 7.12 (s, 1H), 4.21 (qd, J = 7.1, 4.0 Hz, 2H), 1.19 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.5, 164.8, 160.2, 135.4, 130.9, 130.9, 130.2, 128.4, 128.4, 127.5, 127.4, 124.7, 124.6, 123.9, 116.0, 115.8, 77.4, 62.0, 14.2; ^{19}F NMR (376 MHz, DMSO-d_6): δ = -112.5 – -112.7 (m); HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{19}H_{15}FN_2NaO_5^+$: 393.0857; found: 393.0865.

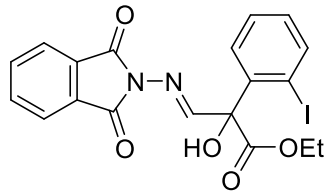
ethyl (*E*)-2-(2-chlorophenyl)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxypropanoate (4f)



White oil, yield: 80%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.30 (s, 1H), 7.90 (t, J = 5.6 Hz, 4H), 7.78 (d, J = 7.2 Hz, 1H), 7.48 – 7.39 (m, 3H), 7.18 (s, H), 4.21 (dd, J = 7.0, 4.4 Hz, 2), 1.19 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.2, 164.8, 160.2, 137.9, 135.4, 132.1, 130.4, 130.3, 130.2, 128.9, 127.4, 123.9, 78.8, 62.0, 14.2; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{19}H_{15}ClN_2NaO_5^+$: 409.0562; found: 409.0572.

ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-(2-iodophenyl)propanoate (4g)

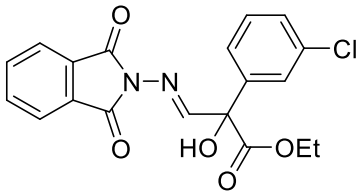


White oil, yield: 49%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.39 (s, 1H), 7.94 – 7.86 (m, 5H), 7.69 (dd, J = 7.8, 1.3 Hz, 1H), 7.46 (t, J = 7.6 Hz, 1H), 7.15 – 7.06 (m, 2H), 4.26 – 4.15 (m, 2H), 1.23 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.0, 164.8, 160.5, 142.6, 141.2, 135.5, 130.2, 129.4, 128.1, 124.0, 97.2, 81.2, 62.0, 14.2; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{19}H_{15}IN_2NaO_5^+$: 500.9918; found: 500.9917.

ethyl (*E*)-2-(3-chlorophenyl)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-

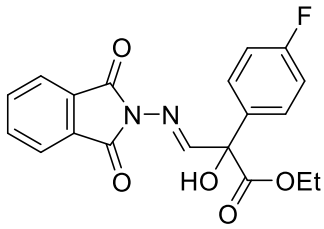
hydroxypropanoate (4h)



White oil, yield: 57%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.99 (s, 1H), 7.90 (dq, J = 8.9, 4.3 Hz, 4H), 7.58 (s, 1H), 7.46 (dt, J = 14.1, 7.1 Hz, 3H), 7.15 (s, 1H), 4.20 (dd, J = 13.8, 6.9 Hz, 2H), 1.20 (d, J = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.6, 164.8, 161.5, 141.9, 135.4, 135.4, 133.4, 130.5, 130.3, 128.6, 125.6, 124.0, 79.1, 62.2, 14.3; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{19}H_{15}ClN_2NaO_5^+$: 409.0562; found: 409.0571.

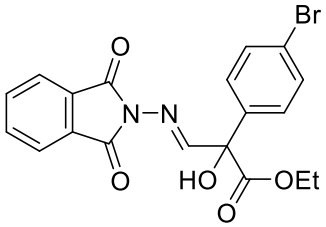
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-(4-fluorophenyl)-2-hydroxypropanoate (4i)



White oil, yield: 59%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.99 (s, 1H), 7.93 – 7.86 (m, 4H), 7.58 (dd, J = 8.7, 5.5 Hz, 2H), 7.25 (t, J = 8.9 Hz, 2H), 7.02 (s, 1H), 4.19 (qd, J = 7.2, 3.6 Hz, 2H), 1.21 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 164.8, 162.0, 135.7, 135.4, 130.3, 129.0, 128.9, 123.9, 115.5, 115.3, 79.0, 62.1, 14.3; ^{19}F NMR (376 MHz, DMSO-d_6): δ = -114.3; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{19}H_{15}FN_2NaO_5^+$: 393.0857; found: 393.0865.

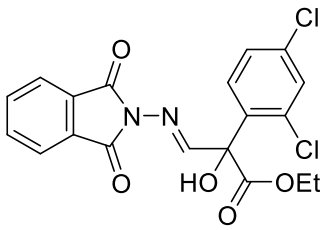
ethyl (*E*)-2-(4-bromophenyl)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxypropanoate (4j)



White oil, yield: 53%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.98 (s, 1H), 7.92 – 7.86 (m, 4H), 7.62 (d, J = 8.5 Hz, 2H), 7.49 (d, J = 8.6 Hz, 2H), 7.08 (s, 1H), 4.25 – 4.15 (m, 2H), 1.21 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.7, 164.8, 161.6, 138.9, 135.4, 131.5, 130.3, 129.0, 123.9, 122.0, 79.1, 62.1, 14.3; HRMS (ESI): *m/z* $[M+Na]^+$ calcd for $C_{19}H_{15}BrN_2NaO_5^+$: 453.0057; found: 453.0050.

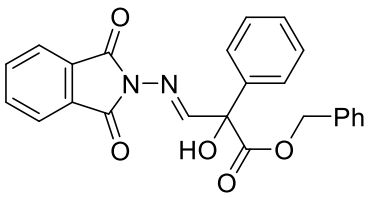
ethyl (*E*)-2-(2,4-dichlorophenyl)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxypropanoate (4k)



White oil, yield: 72%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.29 (s, 1H), 7.92 – 7.87 (m, 4H), 7.79 (d, J = 8.5 Hz, 1H), 7.63 (d, J = 2.1 Hz, 1H), 7.52 (dd, J = 8.5, 2.2 Hz, 1H), 7.39 (s, 1H), 4.21 (dd, J = 7.1, 5.2 Hz, 2H), 1.19 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 169.9, 164.8, 159.5, 137.2, 135.5, 134.0, 130.3, 130.2, 129.8, 127.5, 125.2, 124.0, 78.5, 62.1, 14.2; HRMS (ESI): *m/z* $[M+NH_4]^+$ calcd for $C_{19}H_{18}Cl_2N_3O_5^+$: 438.0618; found: 438.0610.

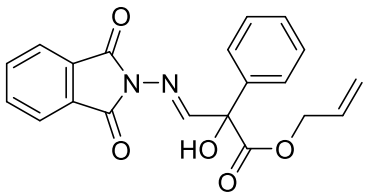
benzyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-phenylpropanoate (4l)



White oil, yield: 42%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.07 (d, J = 0.6 Hz, 1H), 7.95 – 7.87 (m, 4H), 7.55 (d, J = 7.5 Hz, 2H), 7.41 – 7.31 (m, 8H), 7.04 (s, 1H), 5.22 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.1, 164.8, 162.2, 139.5, 136.5, 135.5, 130.3, 128.7, 128.7, 128.4, 128.0, 126.7, 124.0, 79.6, 67.2; HRMS (ESI): *m/z* [M+Na]$^+$ calcd for $C_{24}H_{18}N_2NaO_5^+$: 437.1108; found: 437.1131.

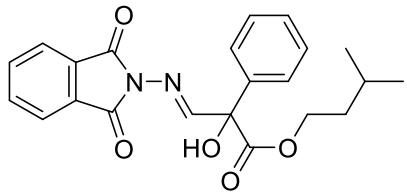
allyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-phenylpropanoate (4m)



White oil, yield: 60%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.07 (s, 1H), 7.95 – 7.88 (m, 4H), 7.60 – 7.55 (m, 2H), 7.45 – 7.36 (m, 3H), 7.01 (s, 1H), 5.96 – 5.86 (m, 1H), 5.27 – 5.15 (m, 2H), 4.68 (ddd, J = 5.1, 2.8, 1.4 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.9, 164.8, 162.1, 139.6, 135.4, 132.5, 130.3, 129.2, 128.7, 126.7, 124.0, 118.1, 79.5, 66.1; HRMS (ESI): *m/z* [M+Na]$^+$ calcd for $C_{20}H_{16}N_2NaO_5^+$: 387.0951; found: 387.0961.

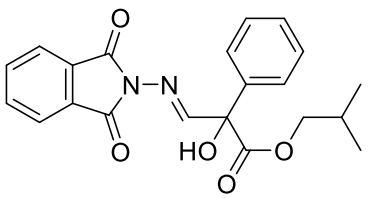
isopentyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-phenylpropanoate (4n)



White oil, yield: 57%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.03 (s, 1H), 8.15 – 8.04 (m, 1H), 7.90 (d, J = 5.2 Hz, 2H), 7.83 (s, 2H), 7.55 (d, J = 7.1 Hz, 2H), 7.38 (dd, J = 17.3, 7.2 Hz, 2H), 6.90 (s, 1H), 4.16 (d, J = 6.2 Hz, 2H), 1.55 (dd, J = 13.2, 6.6 Hz, 1H), 1.46 (dd, J = 13.2, 6.6 Hz, 2H), 0.81 (d, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.2, 164.8, 162.4, 136.7, 135.5, 130.3, 128.6, 126.7, 125.2, 124.0, 79.4, 64.3, 37.1, 24.8, 22.6; HRMS (ESI): *m/z* [M+Na]$^+$ calcd for $C_{22}H_{22}N_2NaO_5^+$: 417.1421; found: 417.1434.

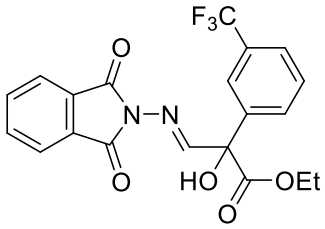
isobutyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-phenylpropanoate (4o)



White oil, yield: 64%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.06 (s, 1H), 7.92 – 7.82 (m, 4H), 7.57 (d, J = 7.2 Hz, 2H), 7.44 – 7.34 (m, 3H), 6.91 (s, 1H), 3.97 – 3.88 (m, 2H), 1.87 (dt, J = 13.1, 6.5 Hz, 1H), 0.81 (d, J = 6.5 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 171.1, 164.8, 162.3, 139.7, 135.4, 134.7, 130.3, 128.6, 126.6, 123.9, 79.4, 71.5, 27.6, 19.1; HRMS (ESI): *m/z* [M+Na]$^+$ calcd for $C_{21}H_{20}N_2NaO_5^+$: 409.0562; found: 409.0572.

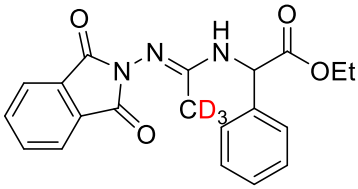
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-(3-(trifluoromethyl)phenyl)propanoate (4q)



White oil, yield: 30%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.01 (s, 1H), 8.09 (dd, J = 23.3, 3.0 Hz, 1H), 7.88 (dd, J = 15.5, 5.7 Hz, 5H), 7.75 (d, J = 7.5 Hz, 1H), 7.69 (d, J = 7.8 Hz, 1H), 7.32 (s, 1H), 4.27 – 4.17 (m, 2H), 1.20 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.6, 164.7, 161.5, 140.7, 136.7, 135.4, 131.2, 130.3, 129.7, 129.2, 125.4, 125.4, 125.2, 124.0, 123.3, 79.1, 62.3, 14.2; ^{19}F NMR (376 MHz, DMSO-d_6): δ = -60.8 – -61.3 (m); HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{16}F_3N_2O_5^+$: 421.1006; found: 421.1024.

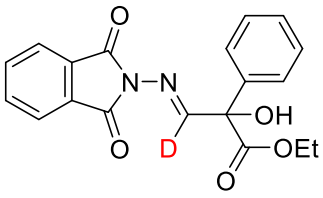
ethyl (*E*)-2-(*N*'-(1,3-dioxoisoindolin-2-yl)acetimidamido-2,2,2-*d*$_3$)-2-phenylacetate (3a')



Yellow oil, yield: 74%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.97 (d, J = 6.7 Hz, 1H), 7.80 (s, 4H), 7.49 (dd, J = 8.1, 1.3 Hz, 2H), 7.41 (ddt, J = 9.7, 5.6, 2.2 Hz, 3H), 5.48 (d, J = 6.7 Hz, 1H), 4.13 (ddd, J = 25.8, 10.8, 7.1 Hz, 2H), 1.17 (t, J = 7.1 Hz, 3H).

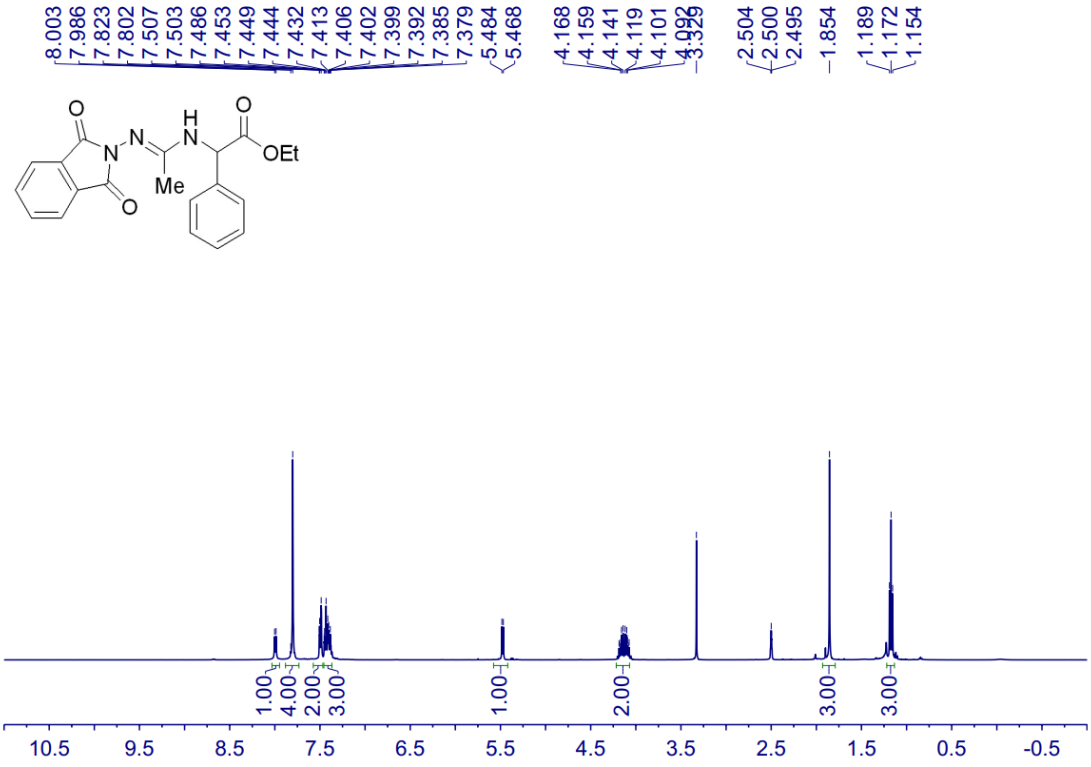
ethyl (*E*)-3-((1,3-dioxoisoindolin-2-yl)imino)-2-hydroxy-2-phenylpropanoate-3-*d* (4a')



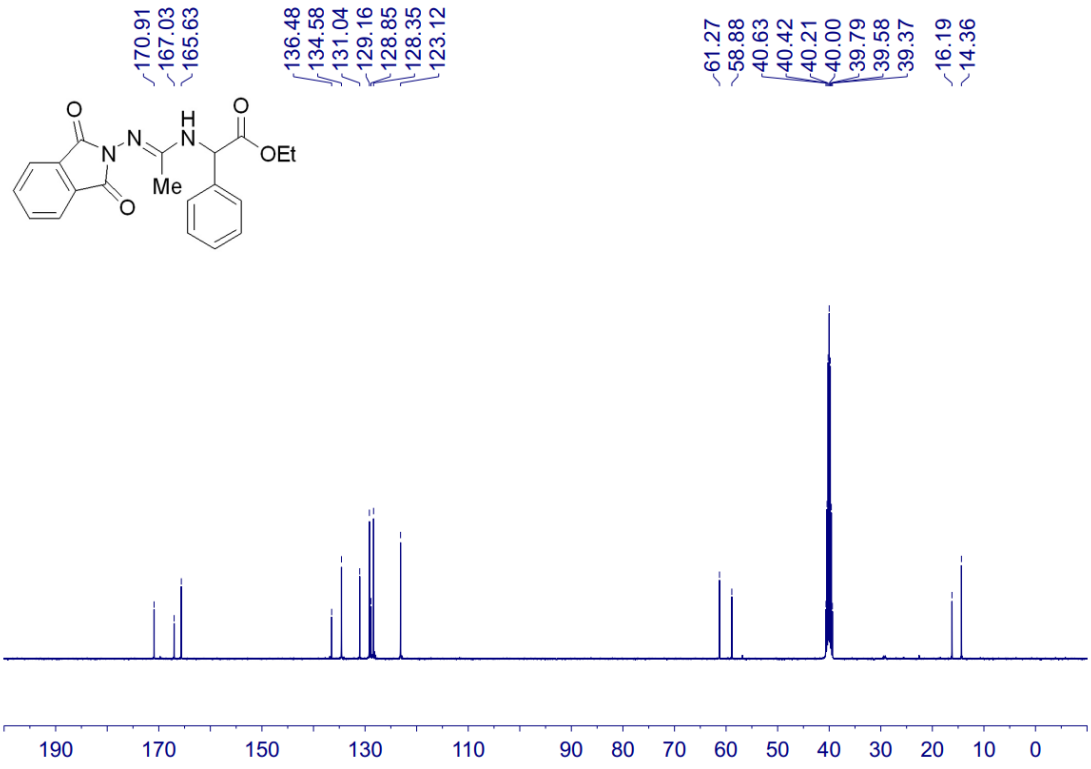
Yellow oil, yield: 72%

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.89 (qd, J = 6.5, 4.3 Hz, 4H), 7.58 – 7.54 (m, 2H), 7.42 (dd, J = 10.0, 4.8 Hz, 2H), 7.38 – 7.34 (m, 1H), 6.93 (s, 1H), 4.25 – 4.15 (m, 2H), 1.20 (t, J = 7.1 Hz, 3H).

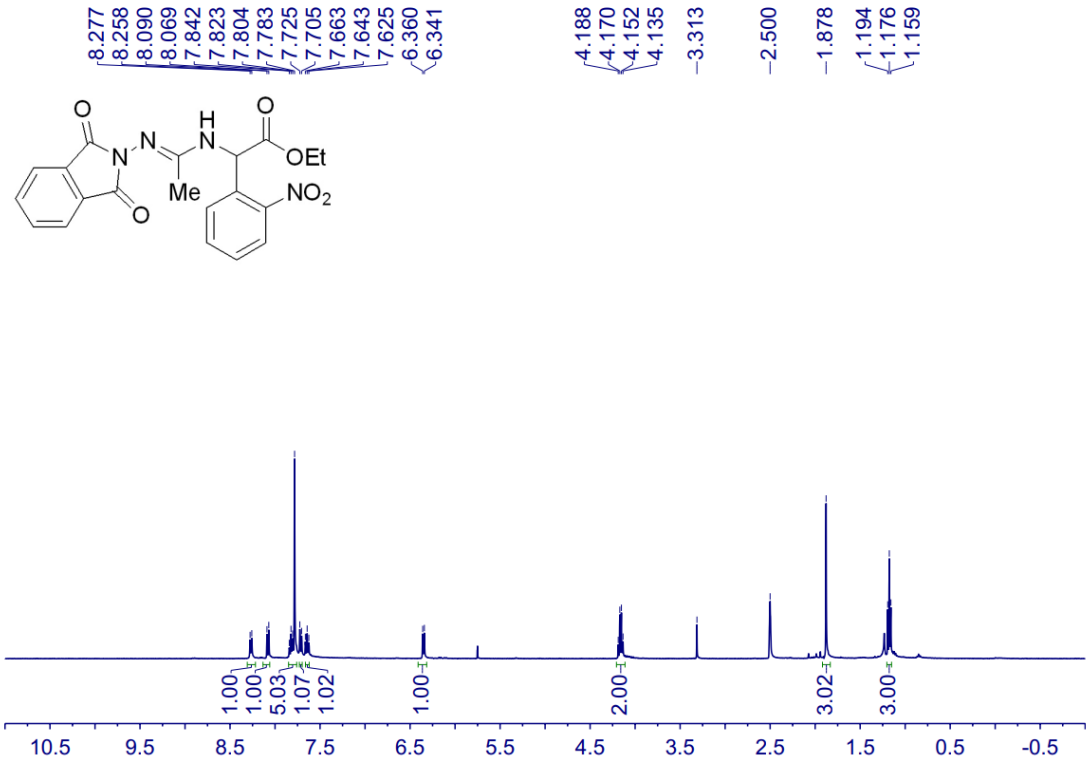
6. NMR Spectra of Compounds



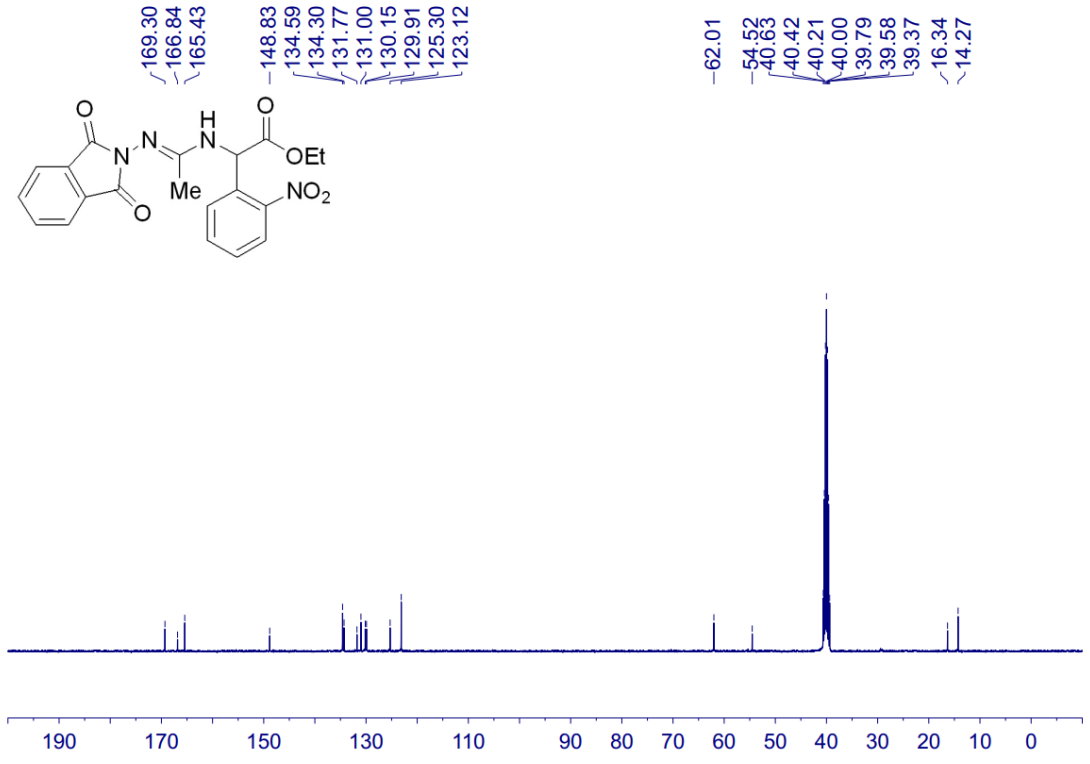
^{1}H NMR Spectrum of Compound **3a** (400 MHz, DMSO-d_6).



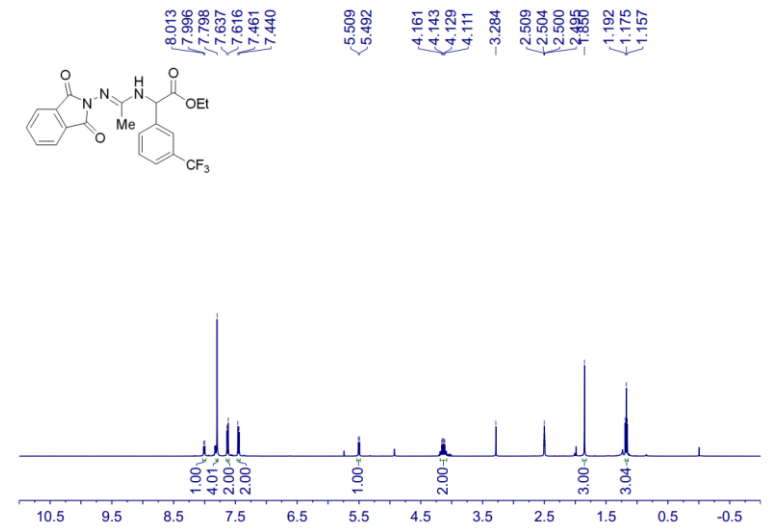
^{13}C NMR Spectrum of Compound **3a** (100 MHz, DMSO-d_6).



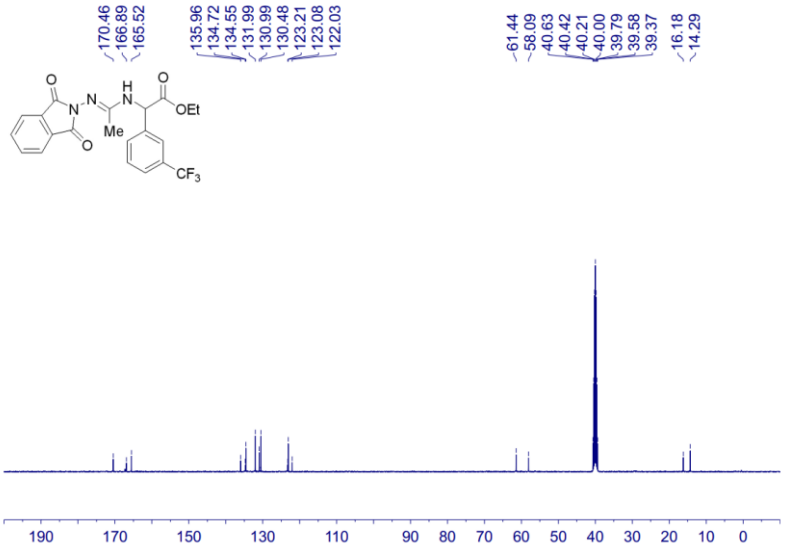
^{1}H NMR Spectrum of Compound **3b** (400 MHz, DMSO-d_6)



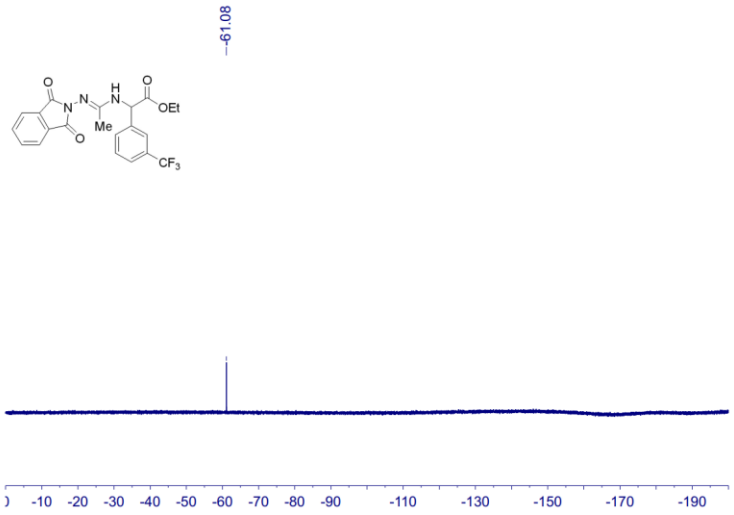
^{13}C NMR Spectrum of Compound **3b** (100 MHz, DMSO-d_6)



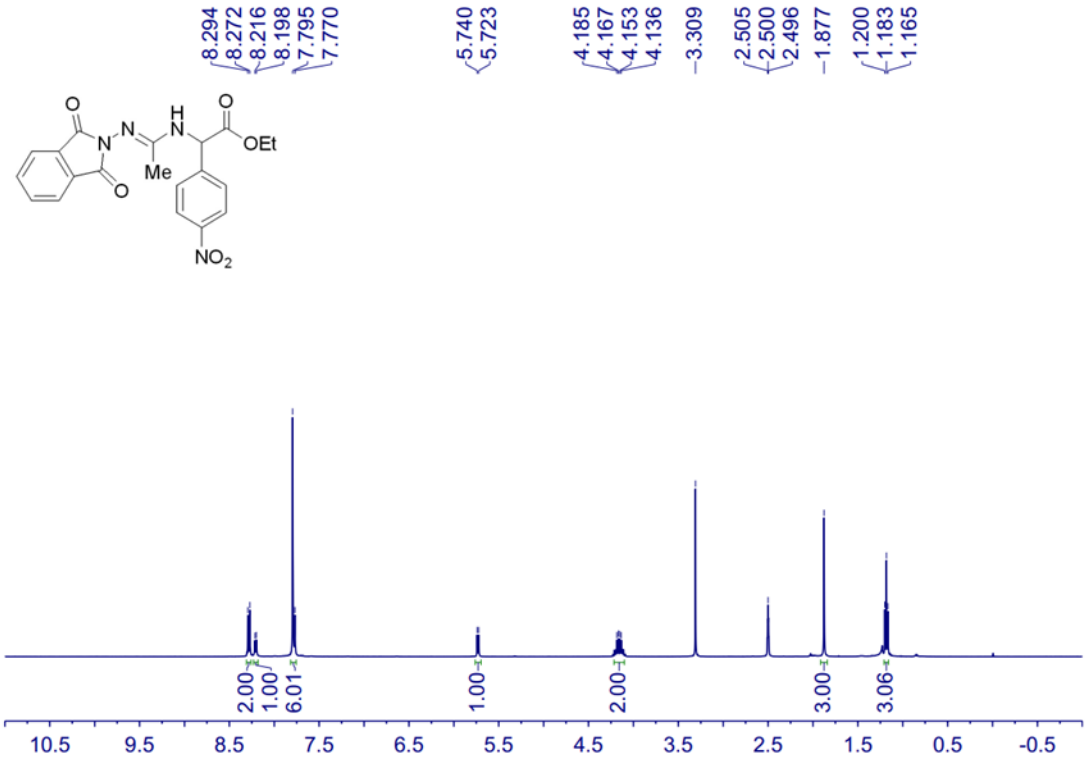
^{1}H NMR Spectrum of Compound **3c** (400 MHz, DMSO-d_6)



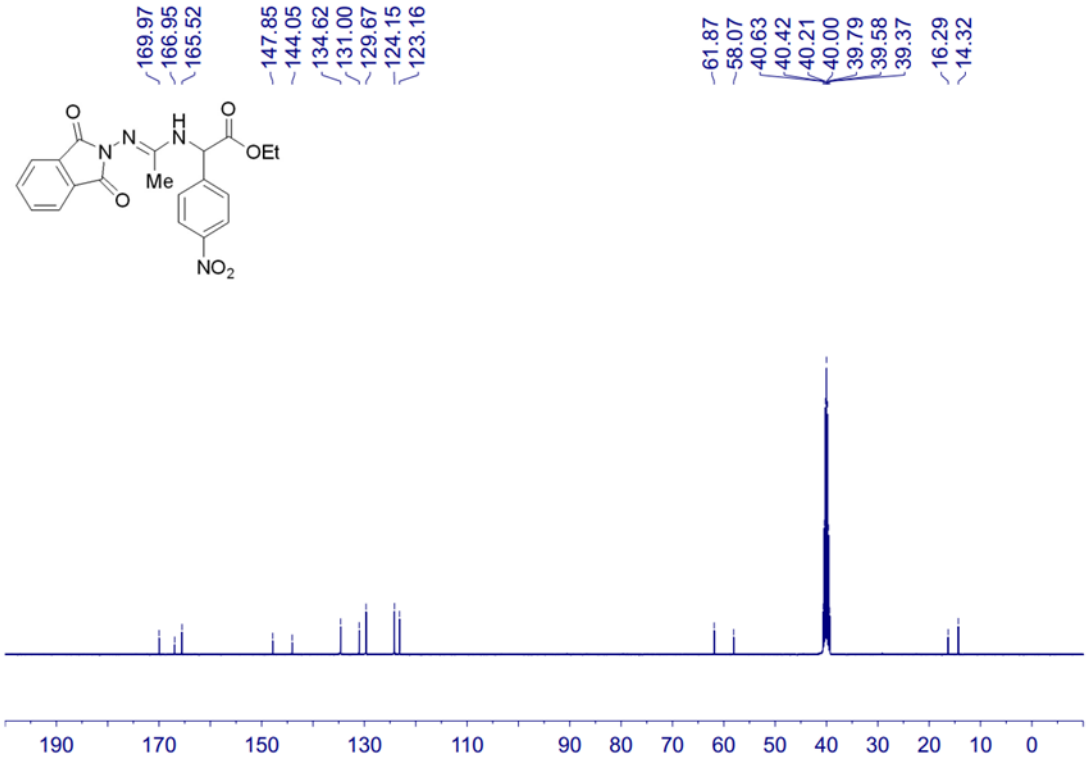
^{13}C NMR Spectrum of Compound **3c** (100 MHz, DMSO-d_6)



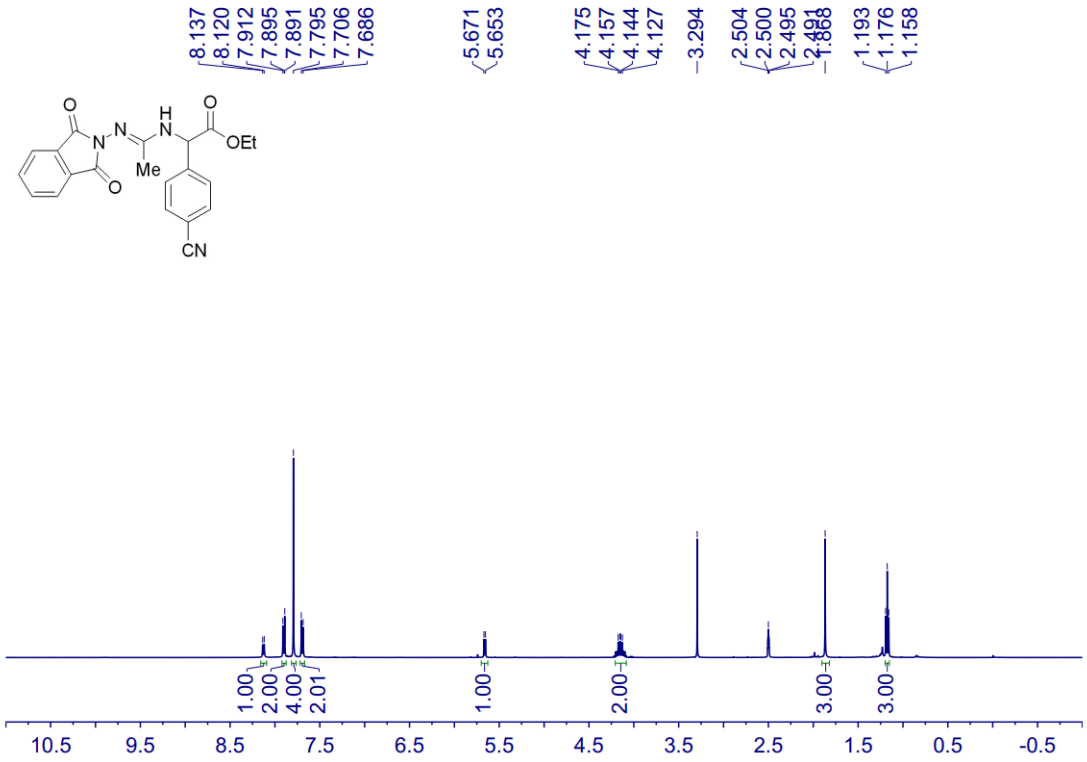
^{19}F NMR Spectrum of Compound **3c** (376 MHz, DMSO-d_6)



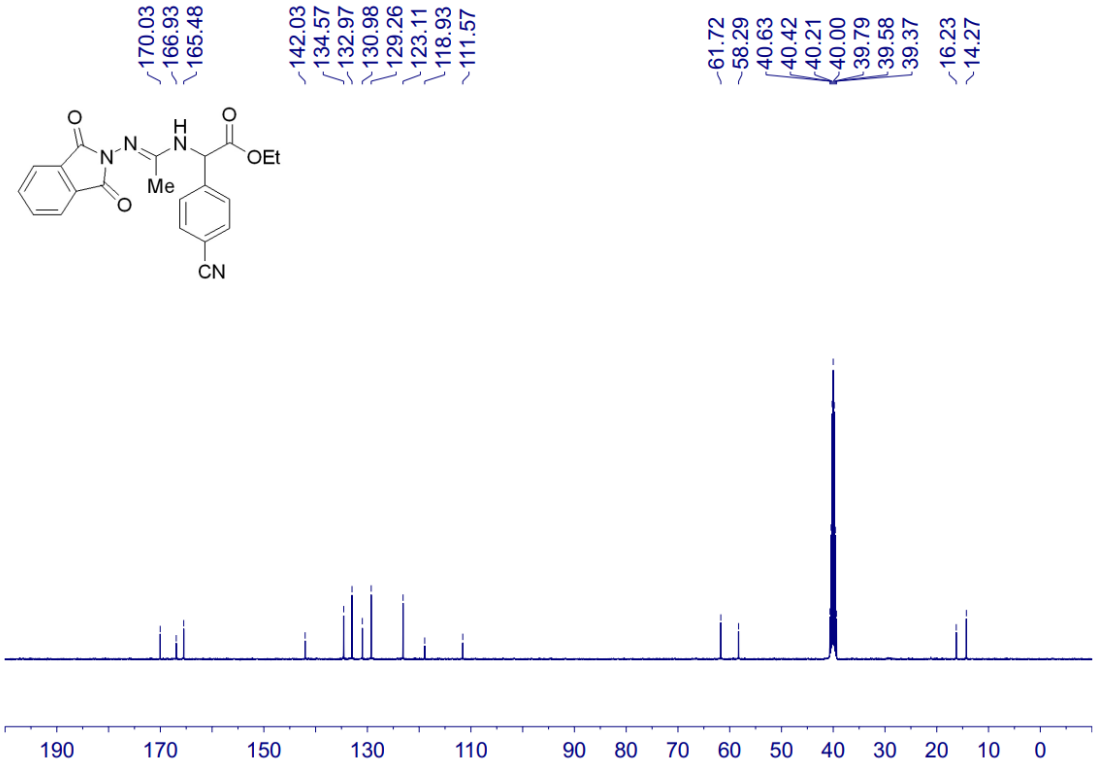
^{1}H NMR Spectrum of Compound **3d** (400 MHz, DMSO-d_6)



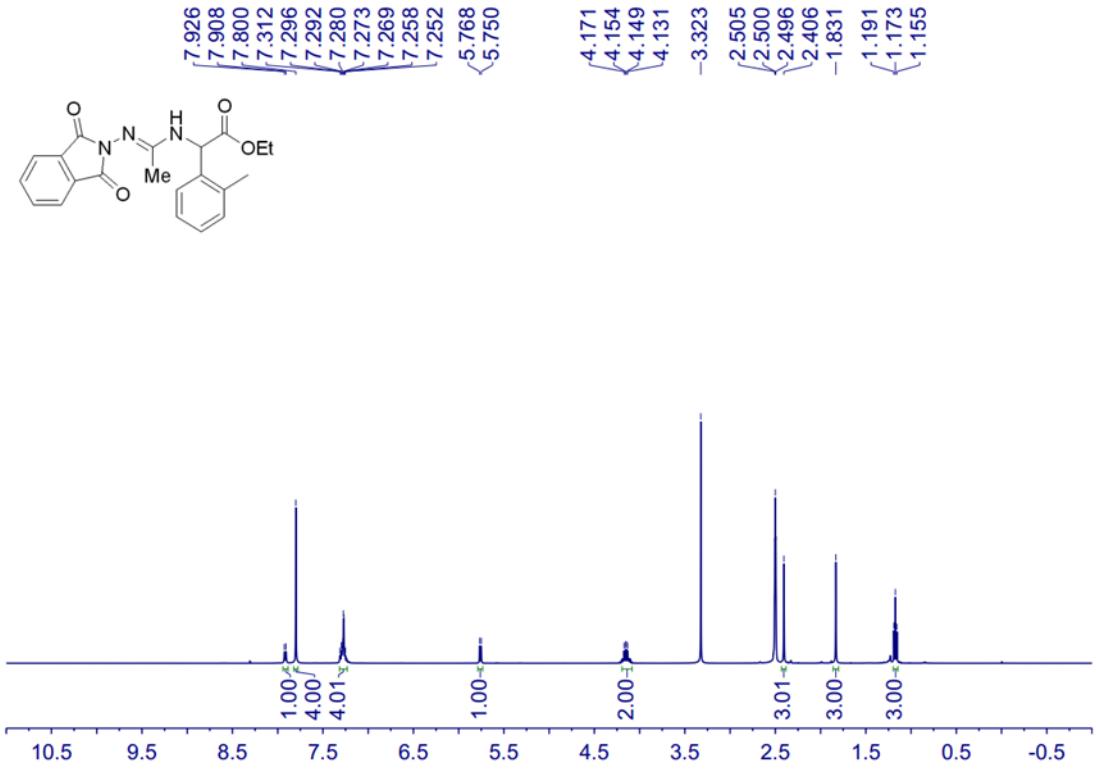
^{13}C NMR Spectrum of Compound **3d** (100 MHz, DMSO-d_6)



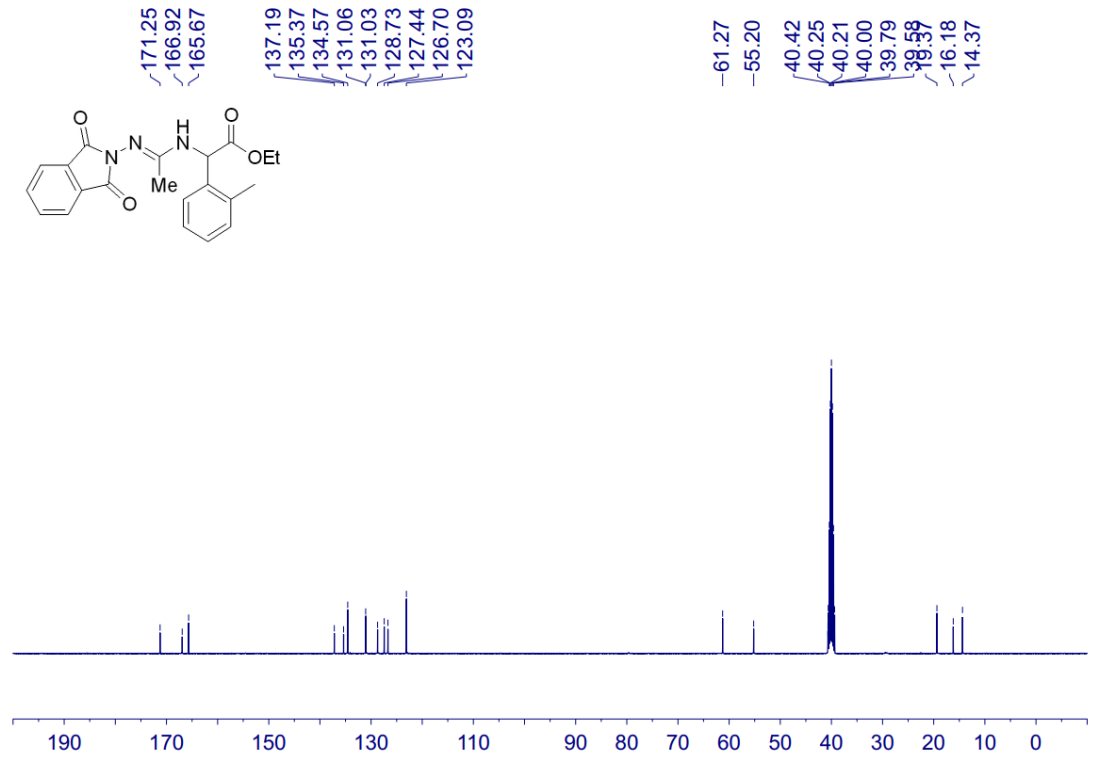
^{1}H NMR Spectrum of Compound **3e** (400 MHz, DMSO-d_6)



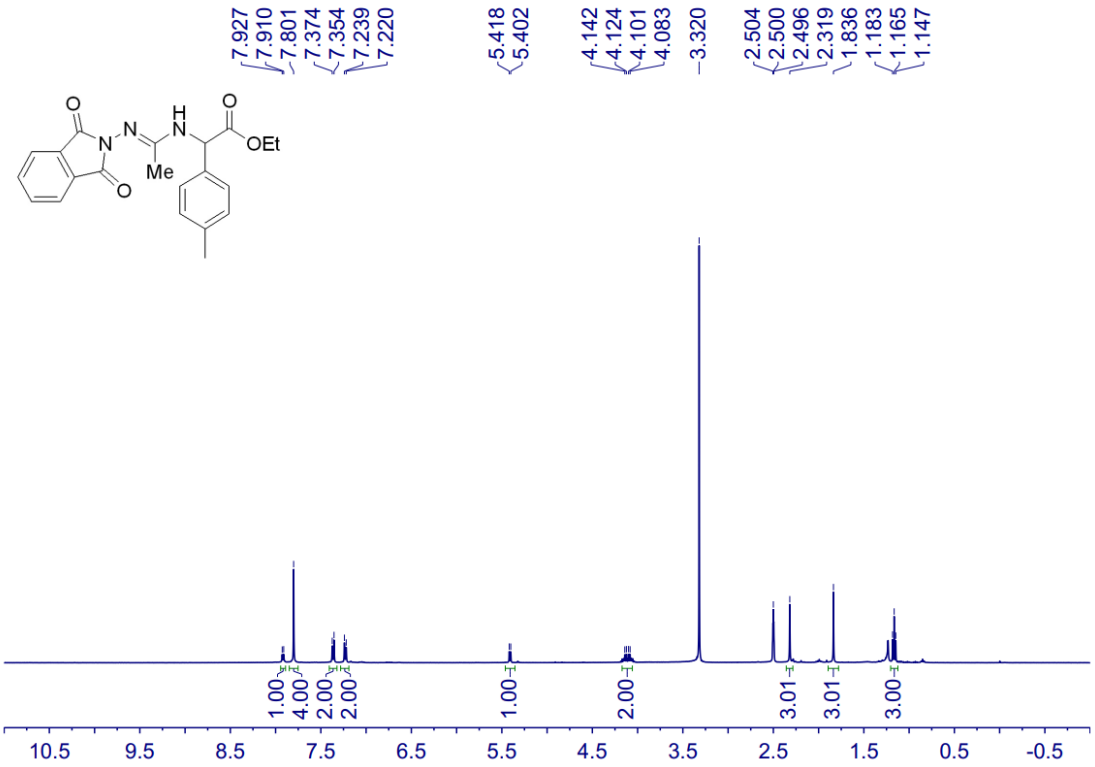
^{13}C NMR Spectrum of Compound **3e** (100 MHz, DMSO-d_6)



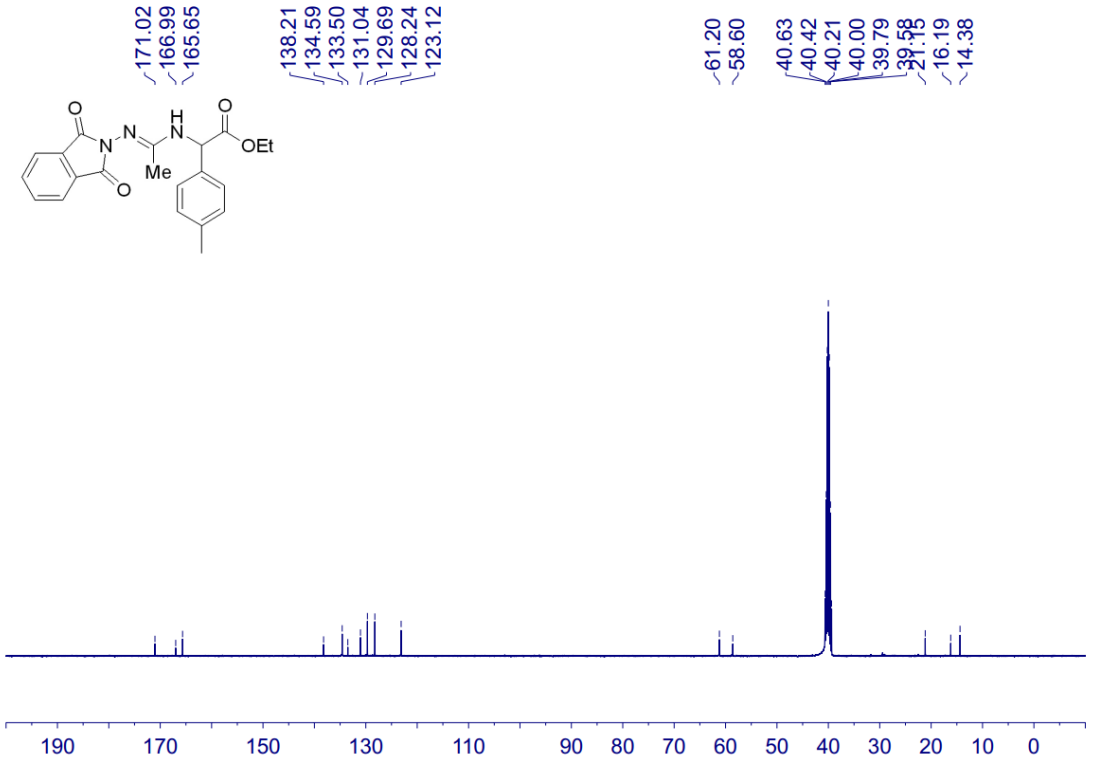
^{1}H NMR Spectrum of Compound **3f** (400 MHz, DMSO-d_6)



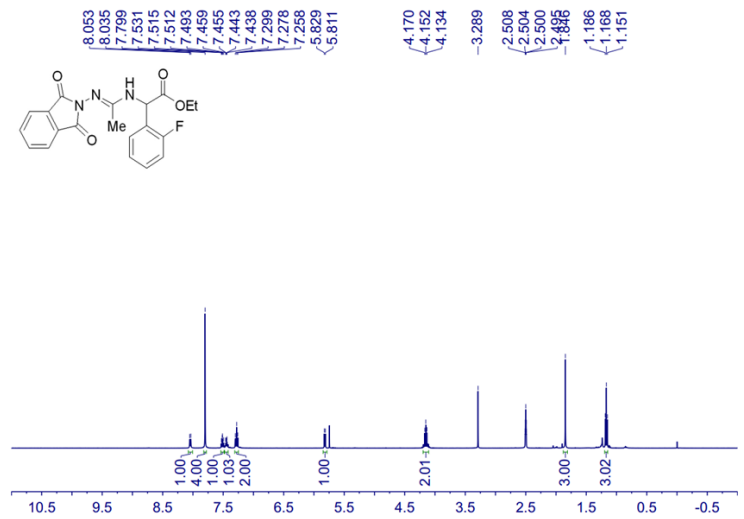
^{13}C NMR Spectrum of Compound **3f** (100 MHz, DMSO-d_6)



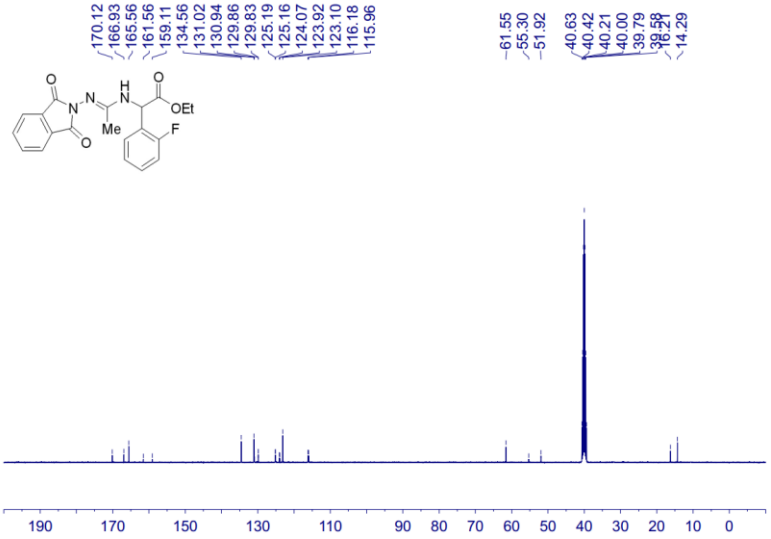
^{1}H NMR Spectrum of Compound **3g** (400 MHz, DMSO-d_6)



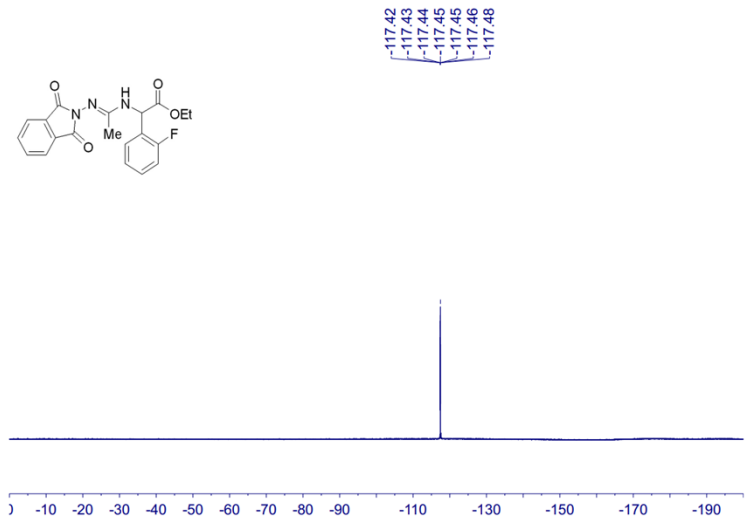
^{13}C NMR Spectrum of Compound **3g** (100 MHz, DMSO-d_6)



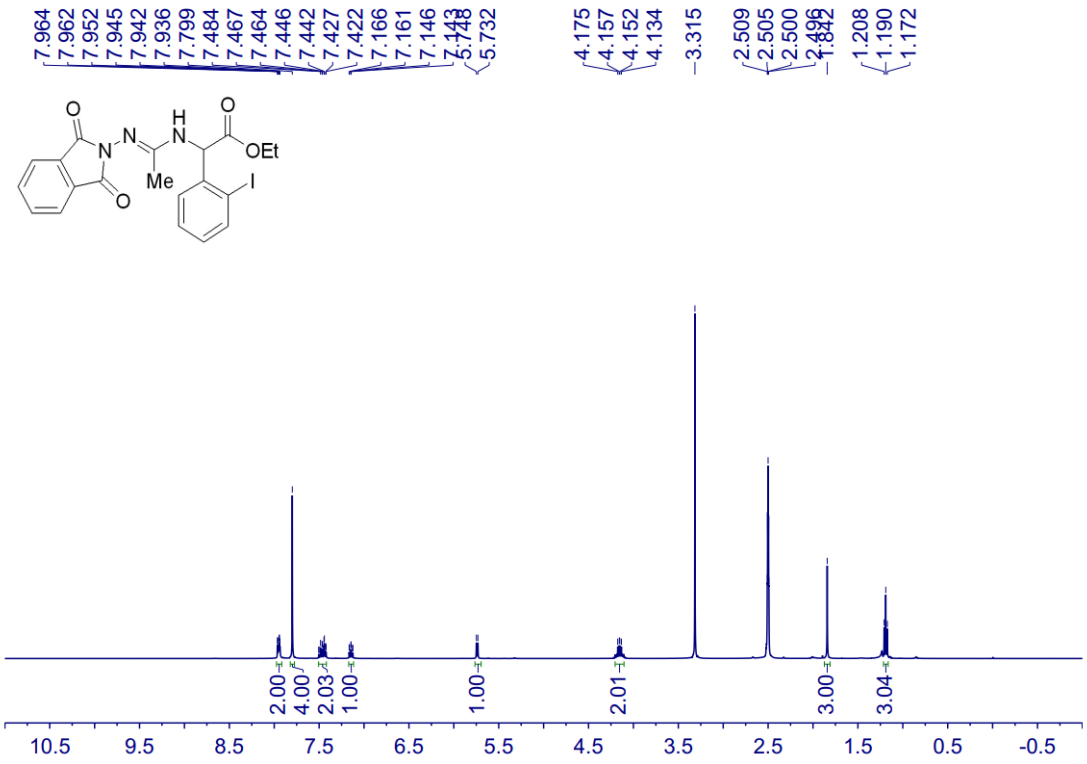
^{1}H NMR Spectrum of Compound **3h** (400 MHz, DMSO-d_6)



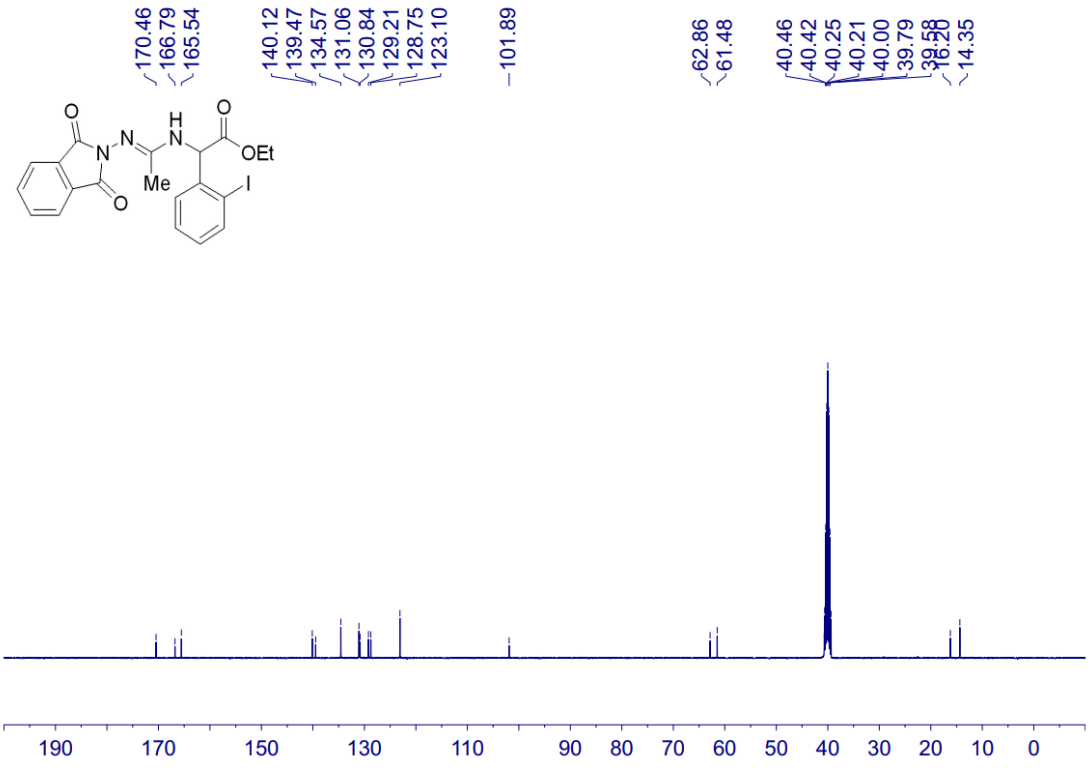
^{13}C NMR Spectrum of Compound **3h** (100 MHz, DMSO-d_6)



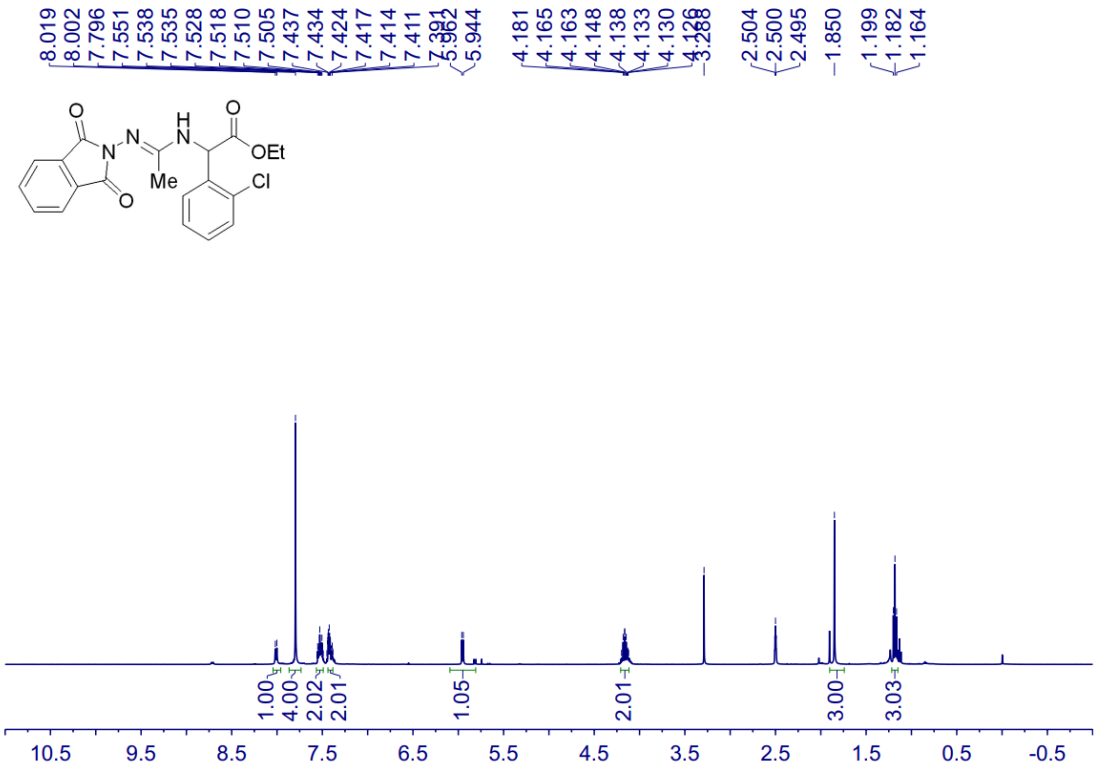
^{19}F NMR Spectrum of Compound **3h** (376 MHz, DMSO-d_6)



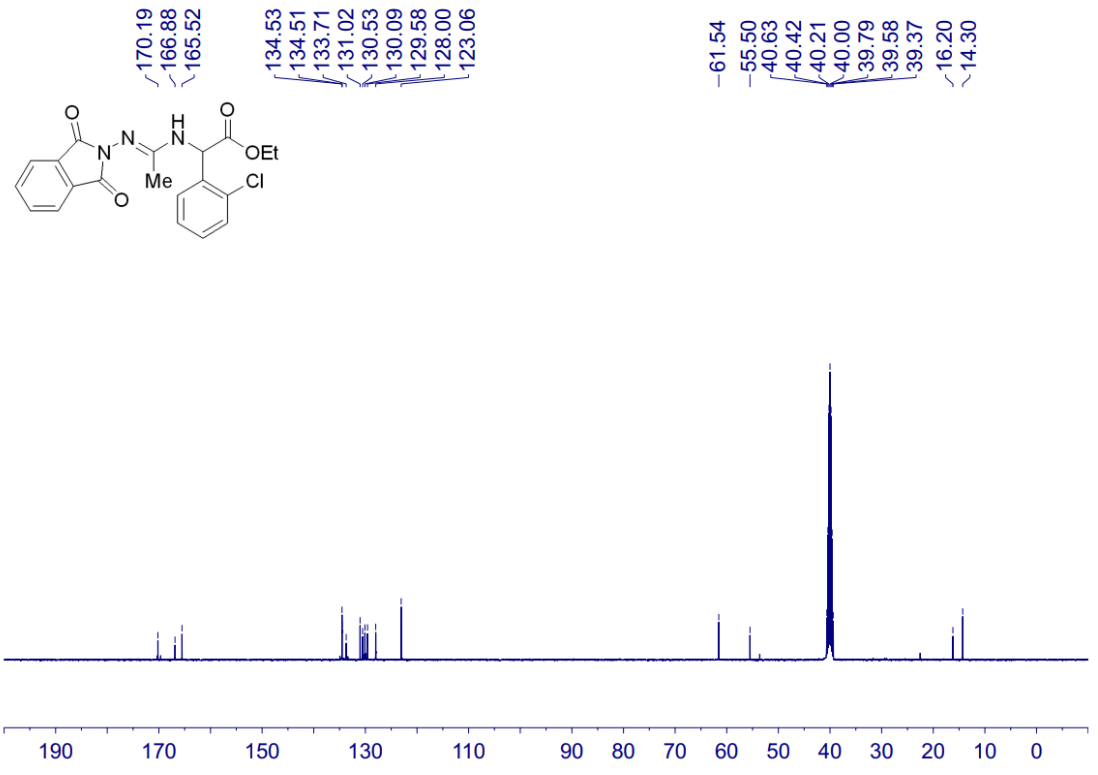
^{1}H NMR Spectrum of Compound **3i** (400 MHz, $CDCl_3$)



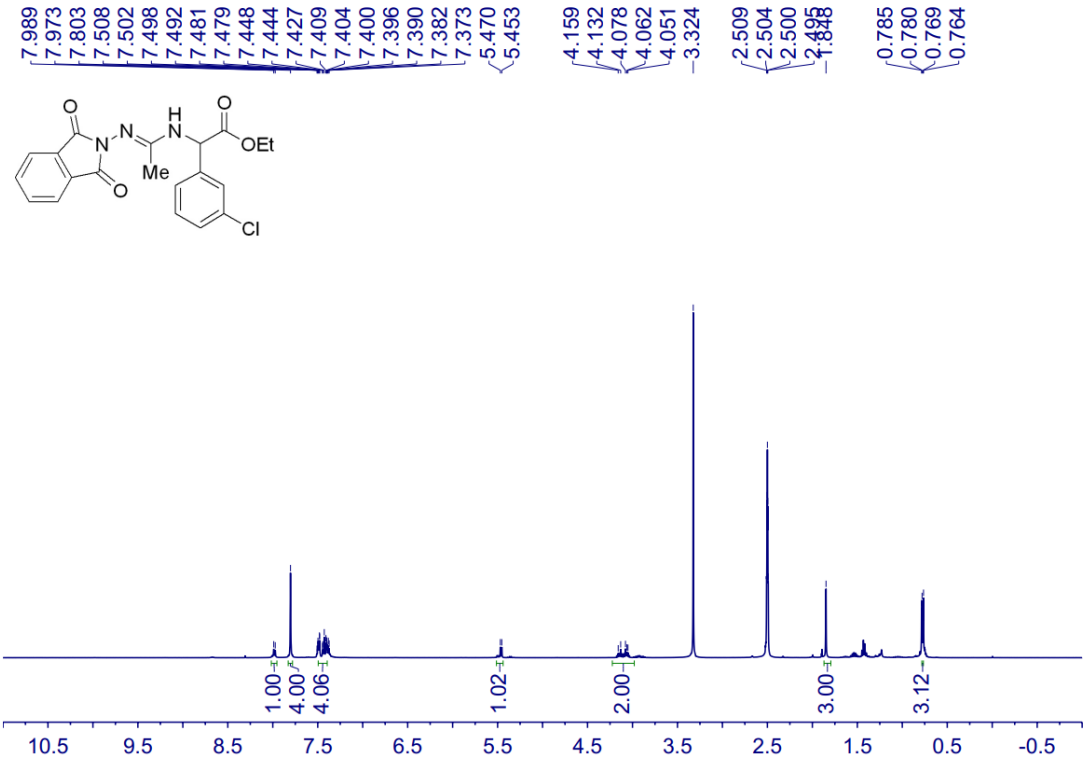
^{13}C NMR Spectrum of Compound **3i** (100 MHz, DMSO-d_6)



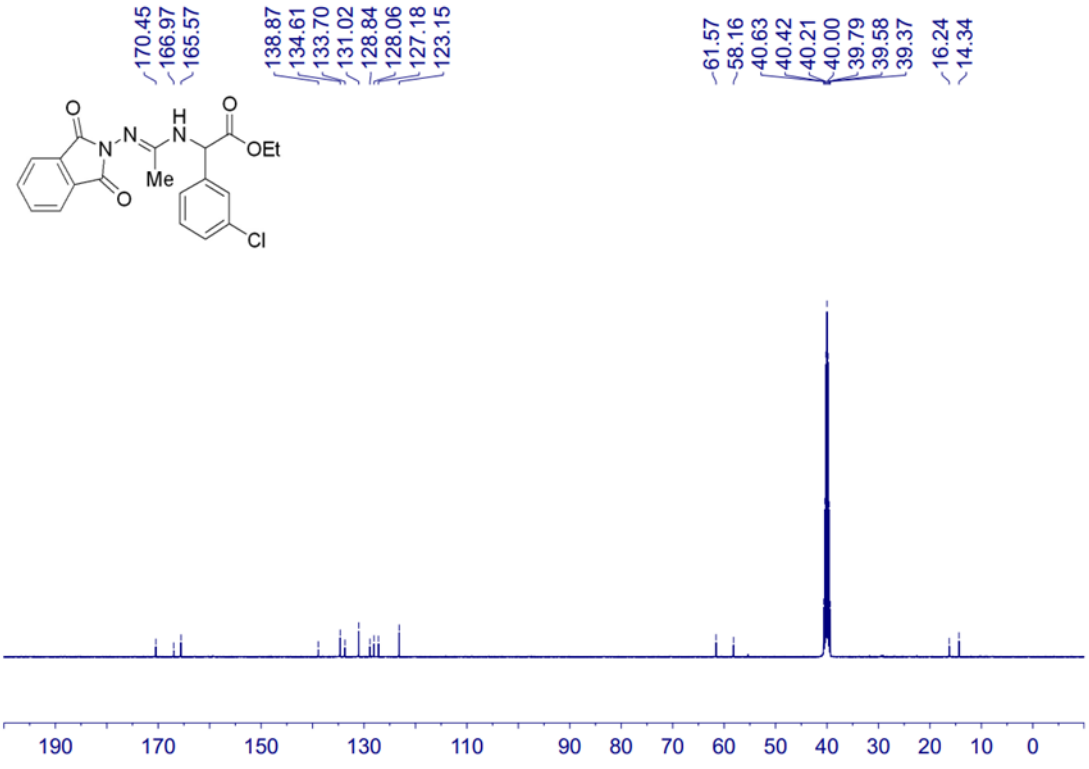
^{1}H NMR Spectrum of Compound **3j** (400 MHz, DMSO-d_6)



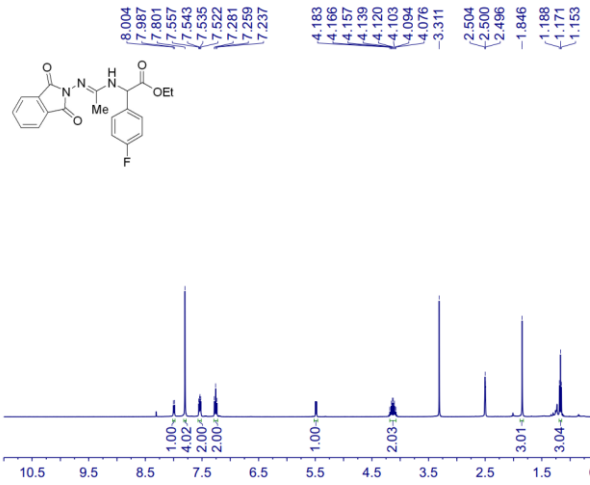
^{13}C NMR Spectrum of Compound **3j** (100 MHz, DMSO-d_6)



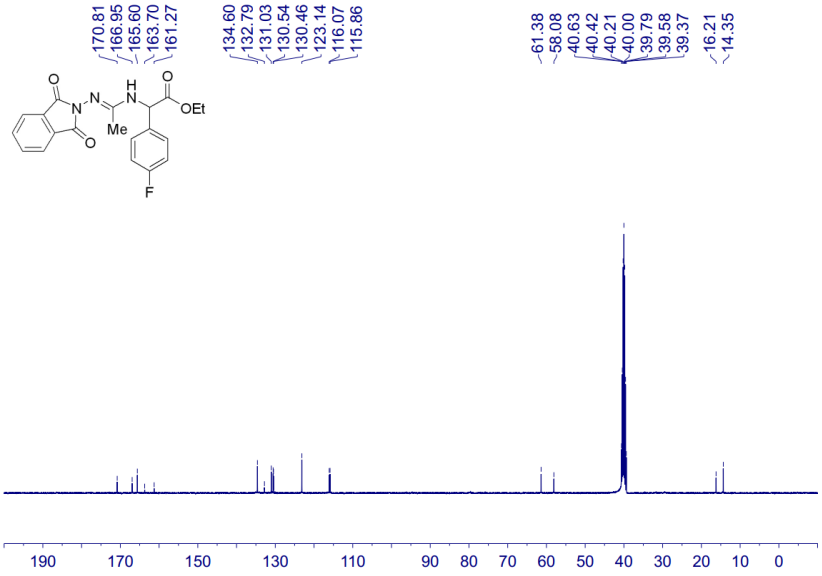
^{1}H NMR Spectrum of Compound **3k** (400 MHz, DMSO-d_6)



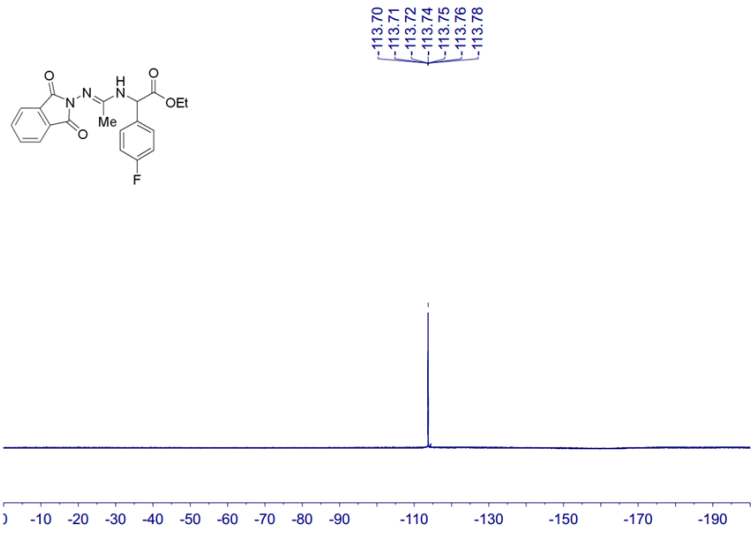
^{13}C NMR Spectrum of Compound **3k** (100 MHz, DMSO-d_6)



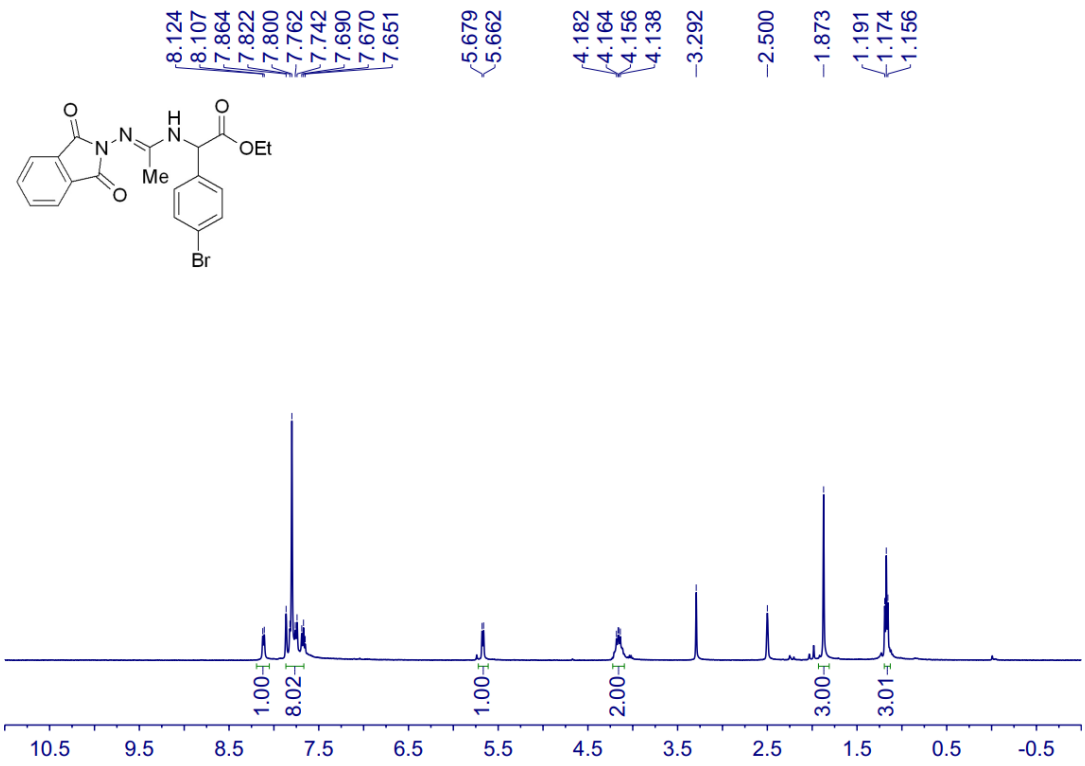
^{1}H NMR Spectrum of Compound **3l** (400 MHz, DMSO-d_6)



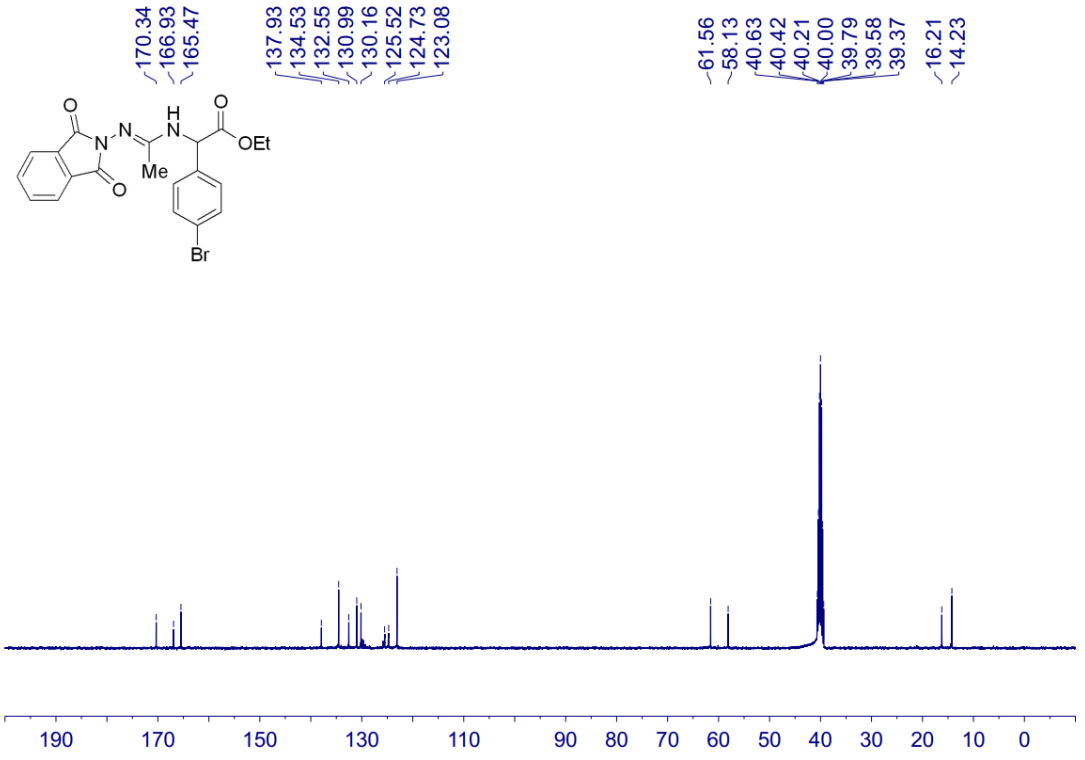
^{13}C NMR Spectrum of Compound **3l** (100 MHz, DMSO-d_6)



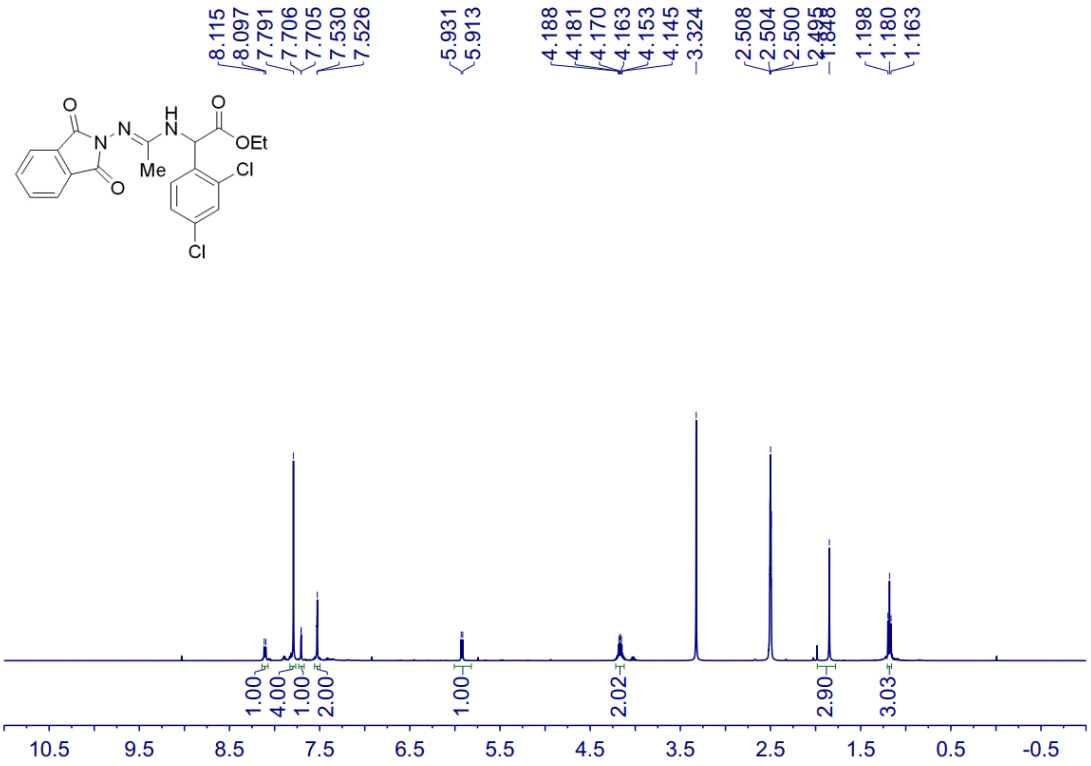
^{19}F NMR Spectrum of Compound **3l** (376 MHz, DMSO-d_6)



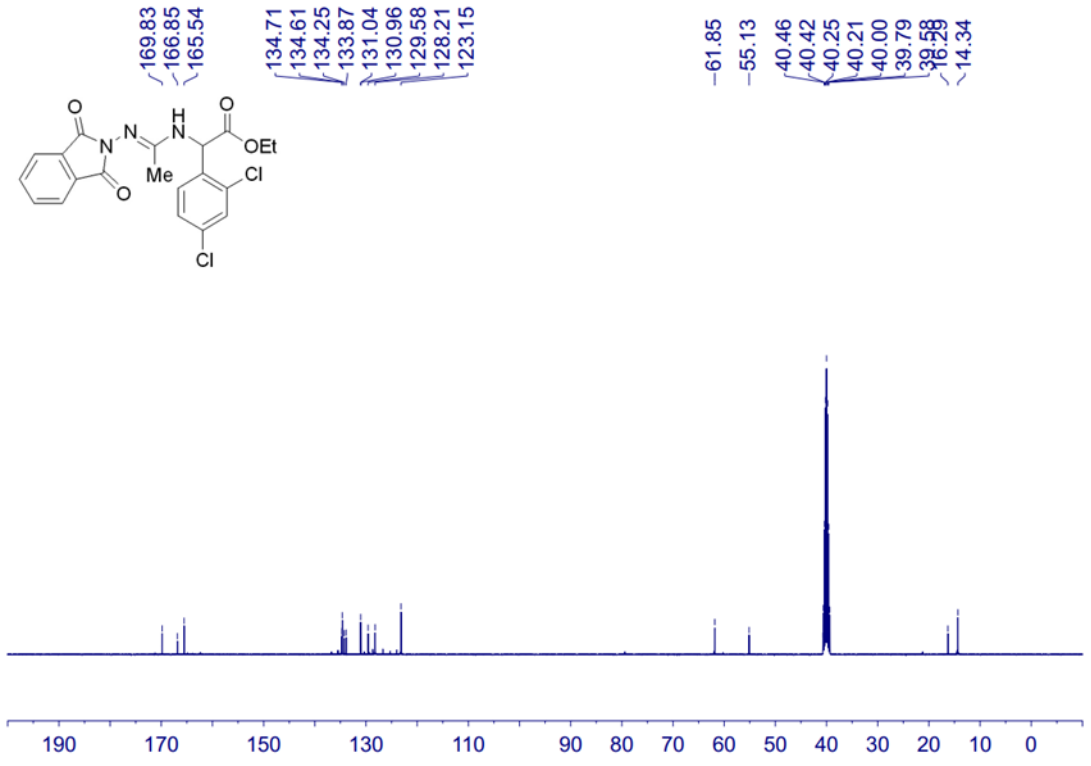
^{1}H NMR Spectrum of Compound **3m** (400 MHz, DMSO-d_6)



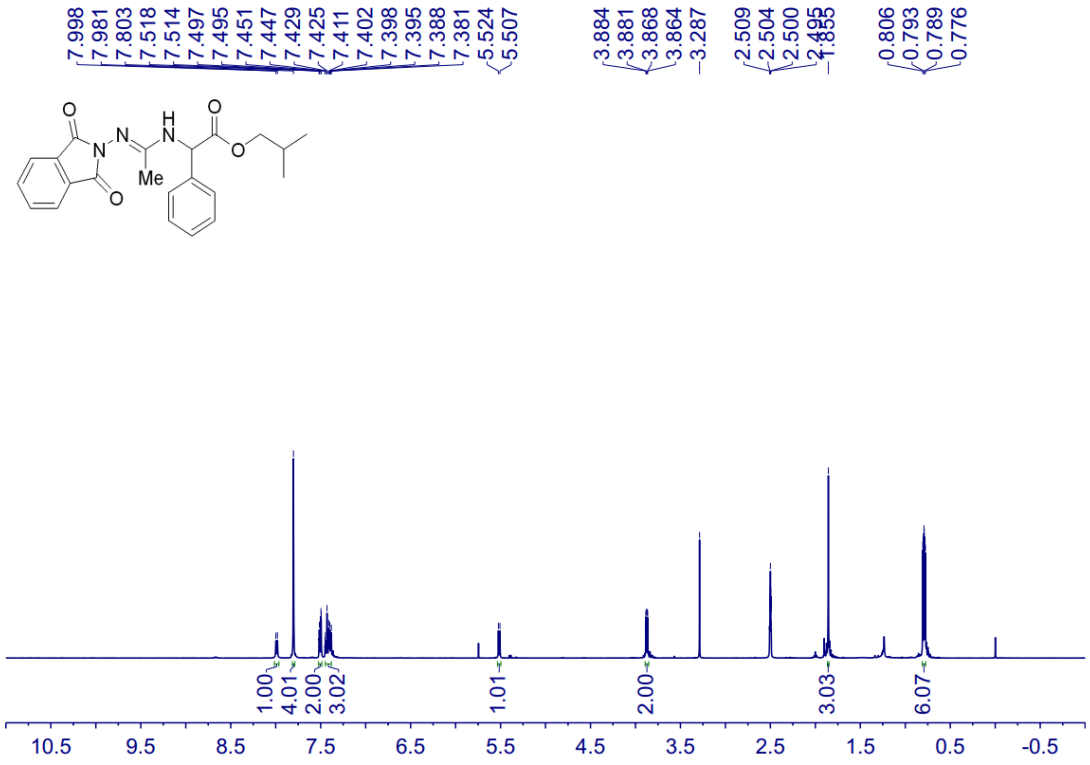
^{13}C NMR Spectrum of Compound **3m** (100 MHz, DMSO-d_6)



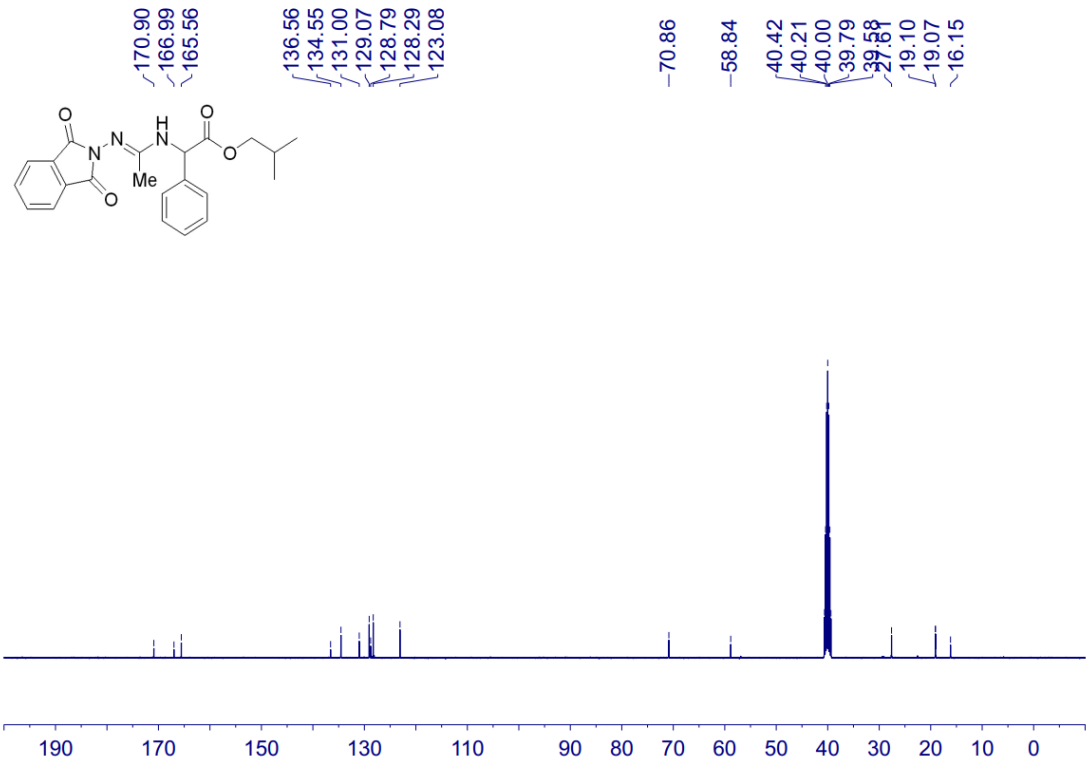
^{1}H NMR Spectrum of Compound **3n** (400 MHz, DMSO-d_6)



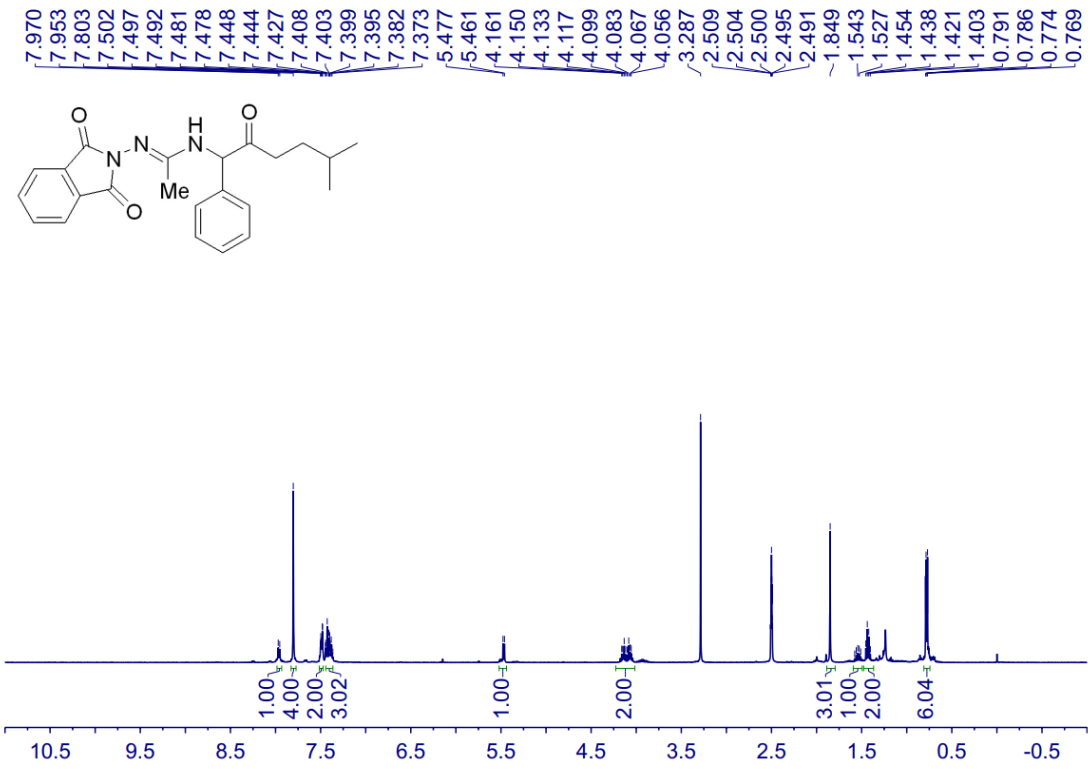
^{13}C NMR Spectrum of Compound **3n** (100 MHz, DMSO-d_6)



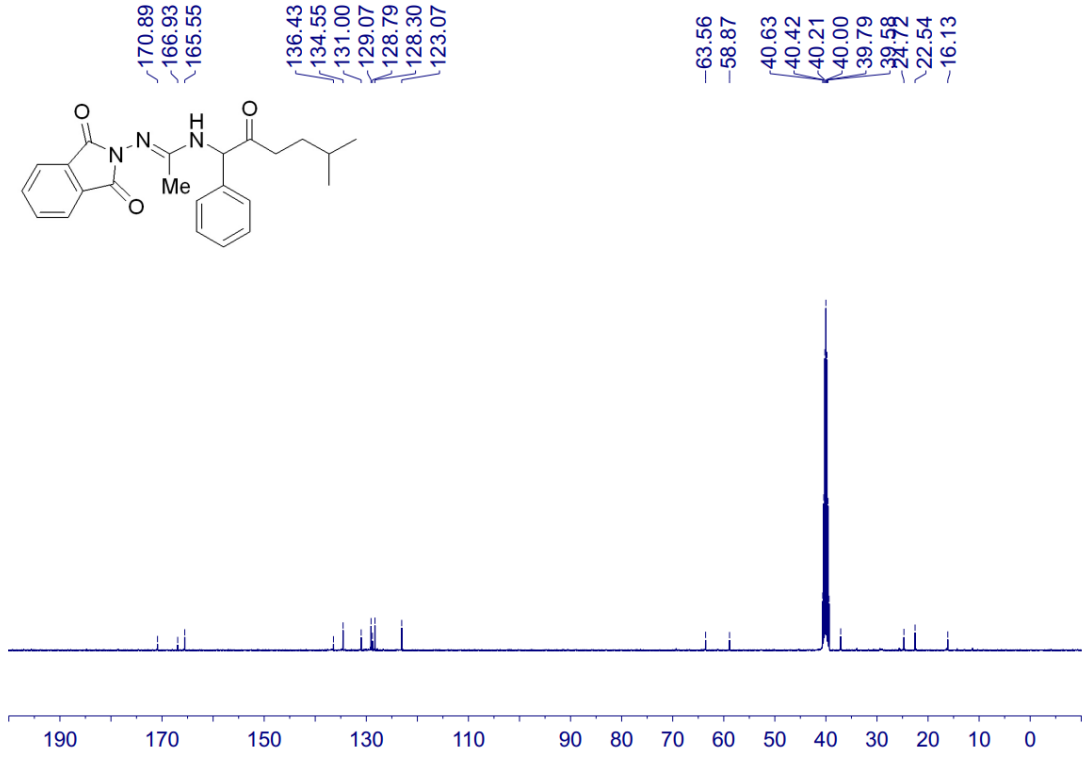
^{1}H NMR Spectrum of Compound **3o** (400 MHz, DMSO-d_6)



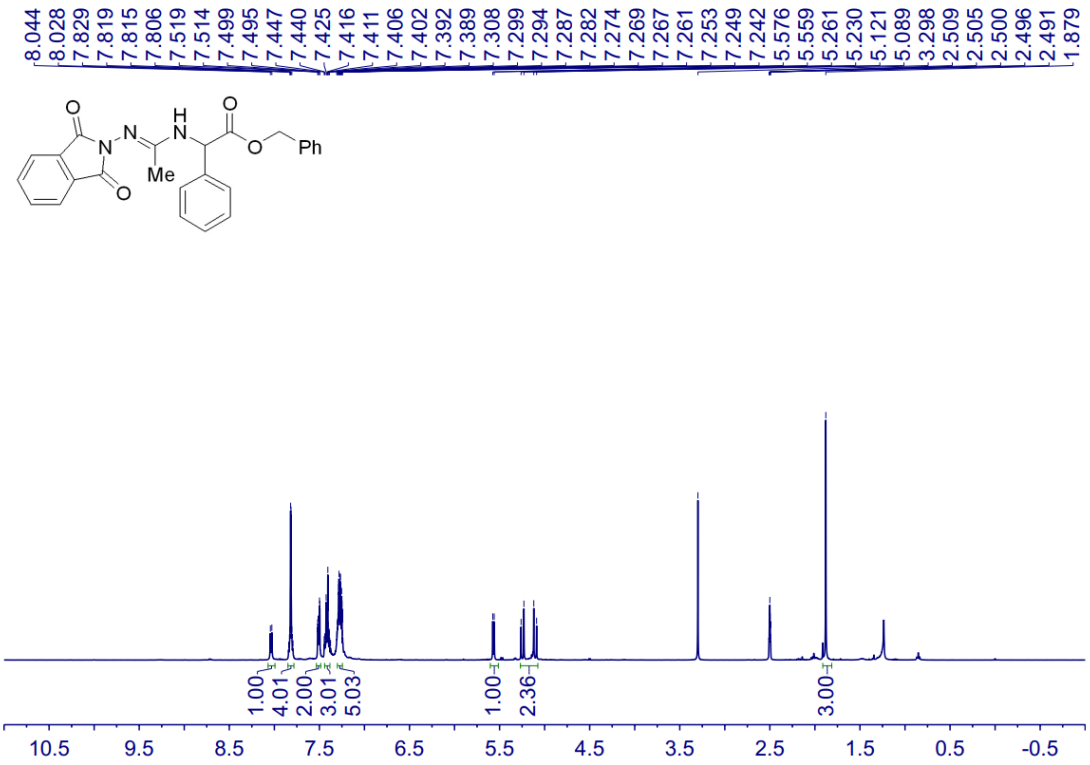
^{13}C NMR Spectrum of Compound **3o** (100 MHz, DMSO-d_6)



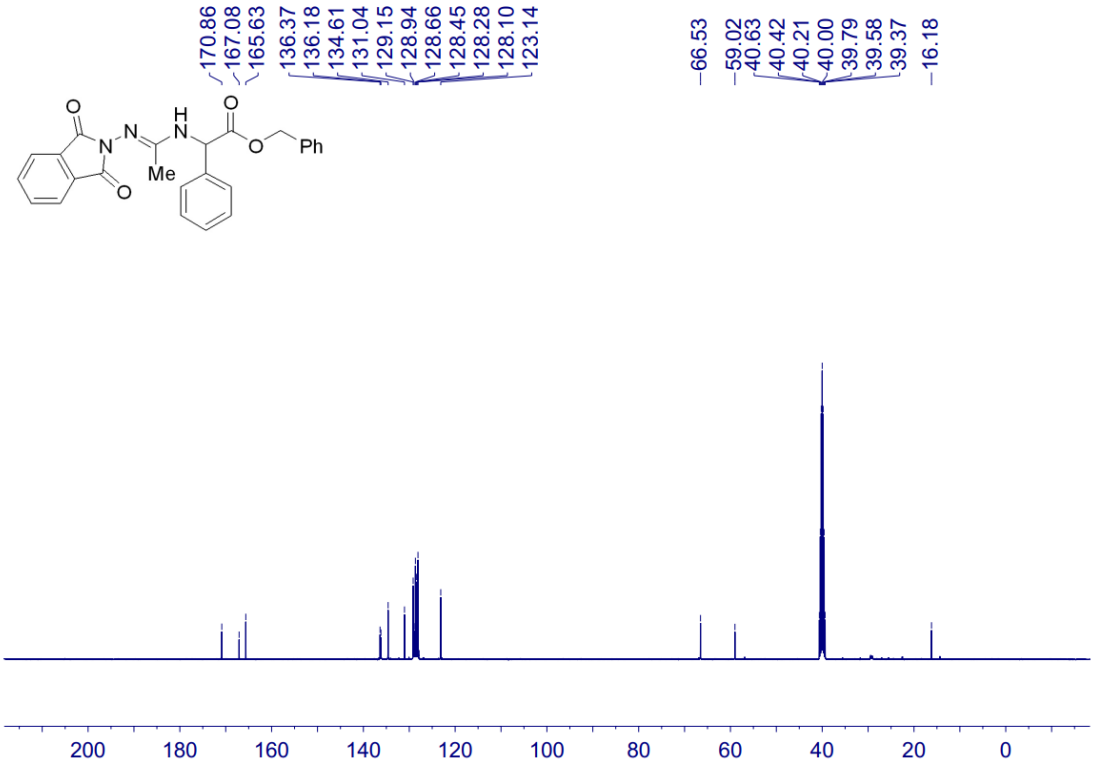
^{1}H NMR Spectrum of Compound **3p** (400 MHz, DMSO-d_6)



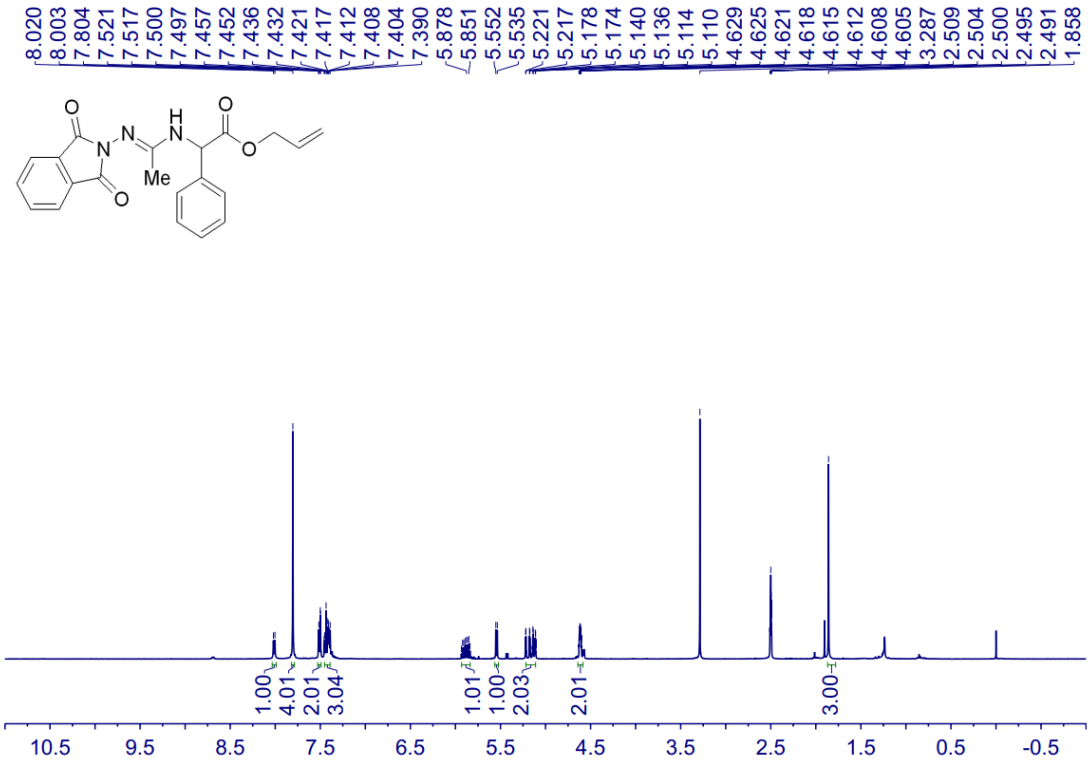
^{13}C NMR Spectrum of Compound **3p** (100 MHz, DMSO-d_6)



^{1}H NMR Spectrum of Compound **3q** (400 MHz, DMSO-d_6)



^{13}C NMR Spectrum of Compound **3q** (100 MHz, DMSO-d_6)



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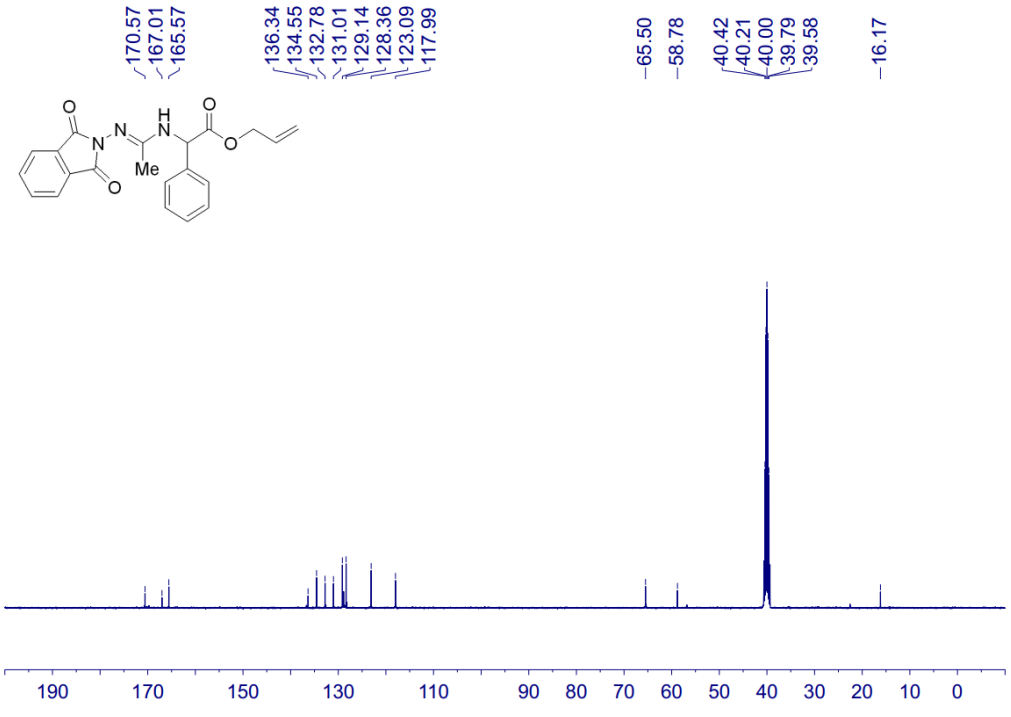
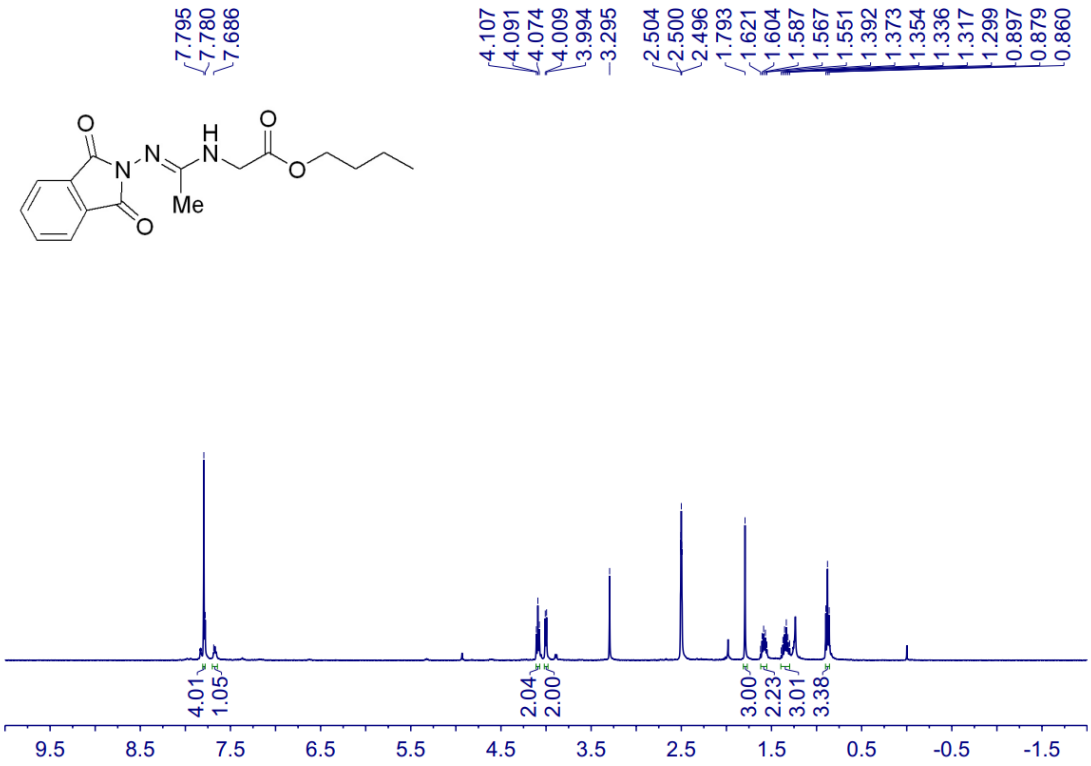
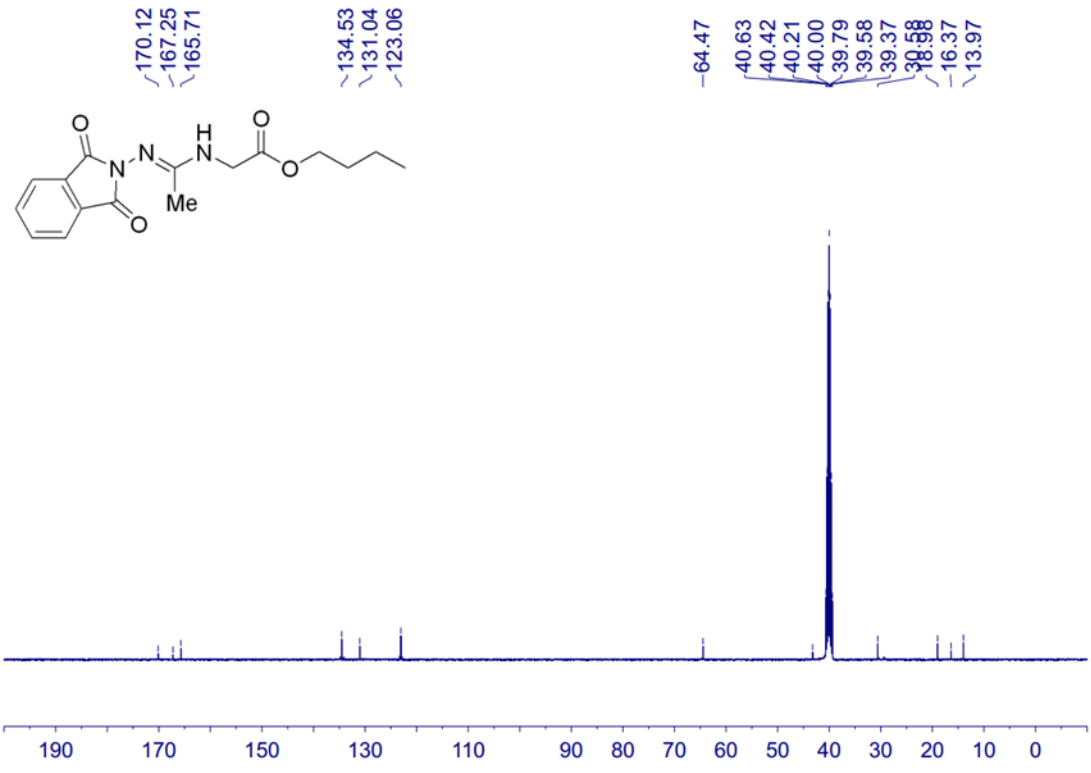


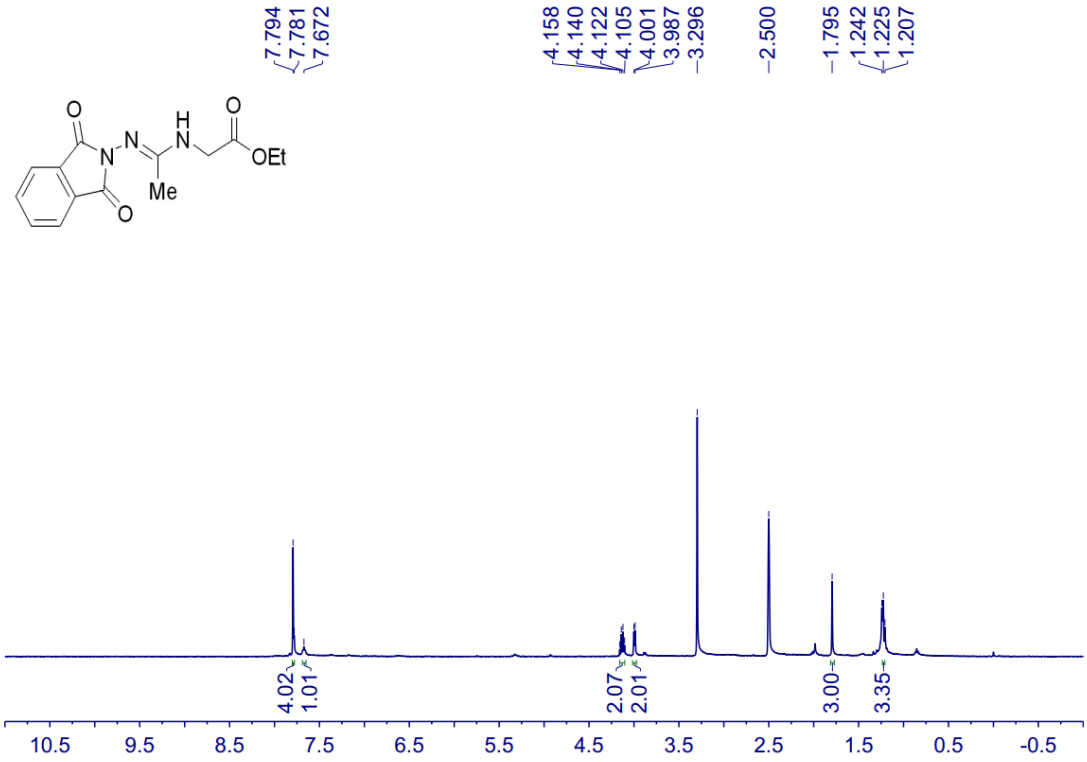
^{13}C NMR Spectrum of Compound **3r** (100 MHz, DMSO-d_6)



^{1}H NMR Spectrum of Compound **3s** (400 MHz, DMSO-d_6)



^{13}C NMR Spectrum of Compound **3s** (100 MHz, DMSO-d_6)



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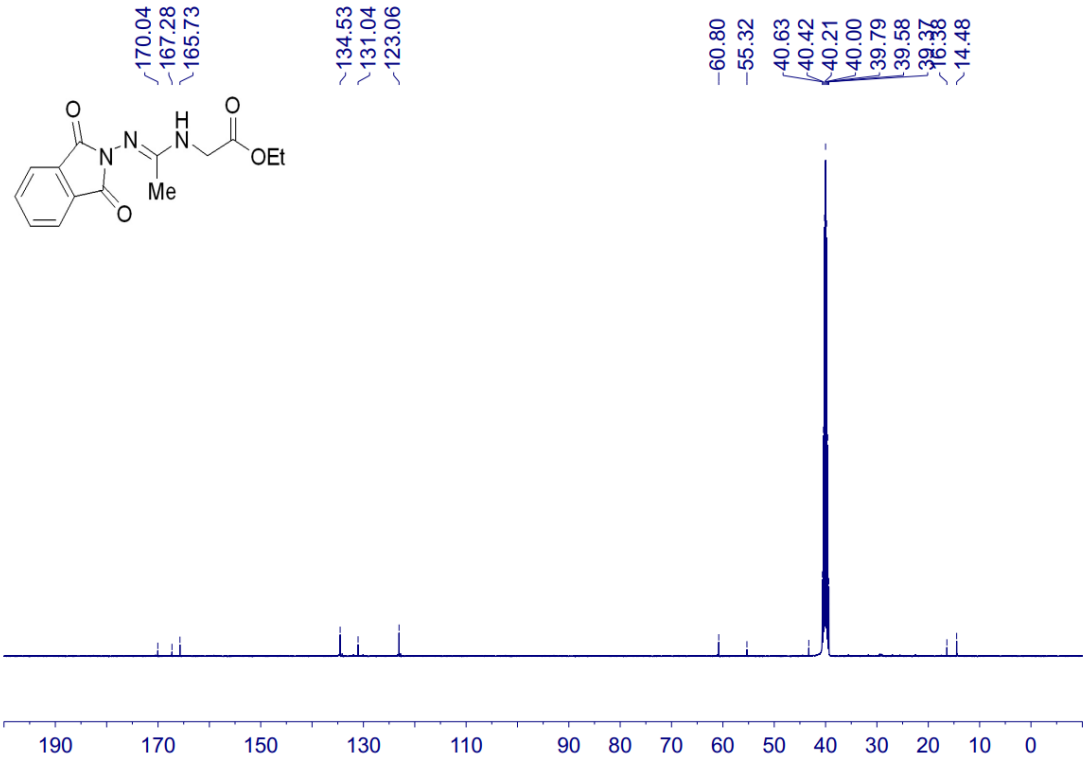
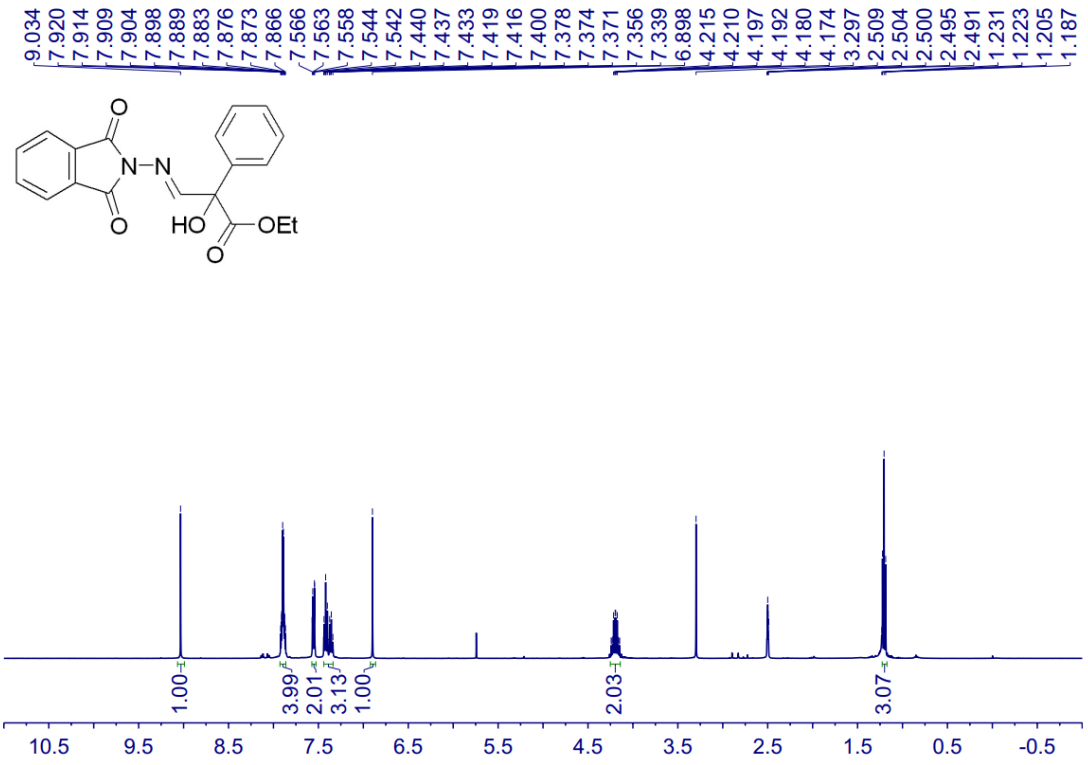


^{13}C NMR Spectrum of Compound **3t** (100 MHz, DMSO-d_6)



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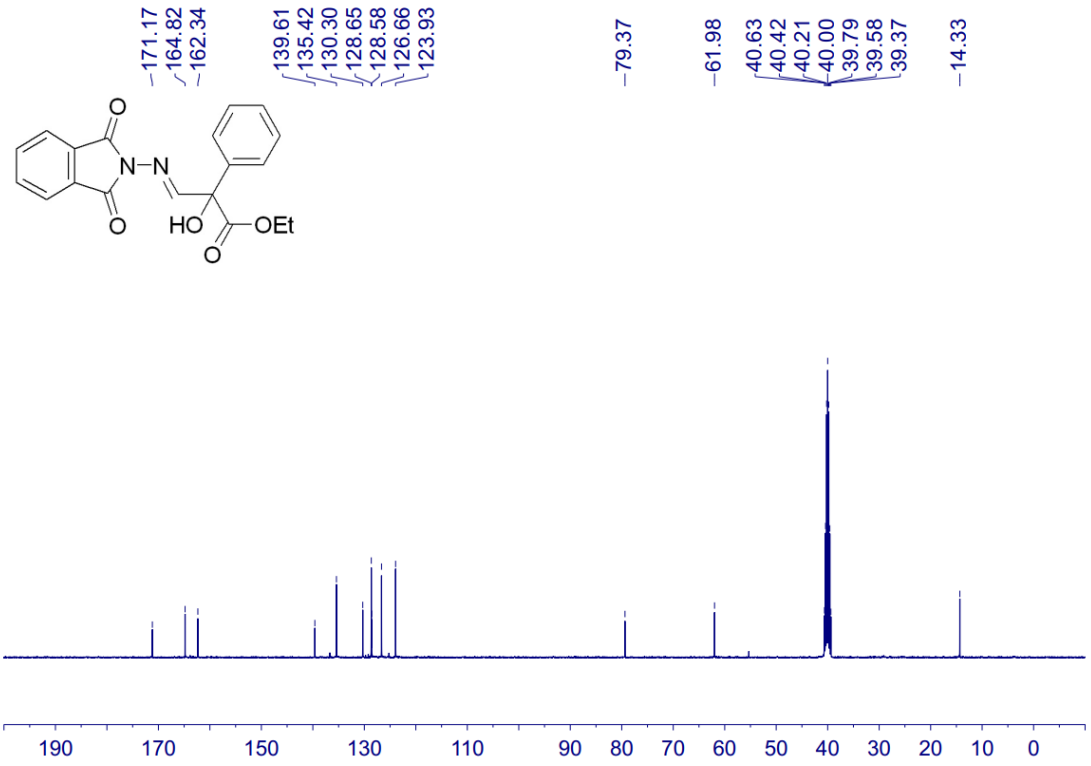
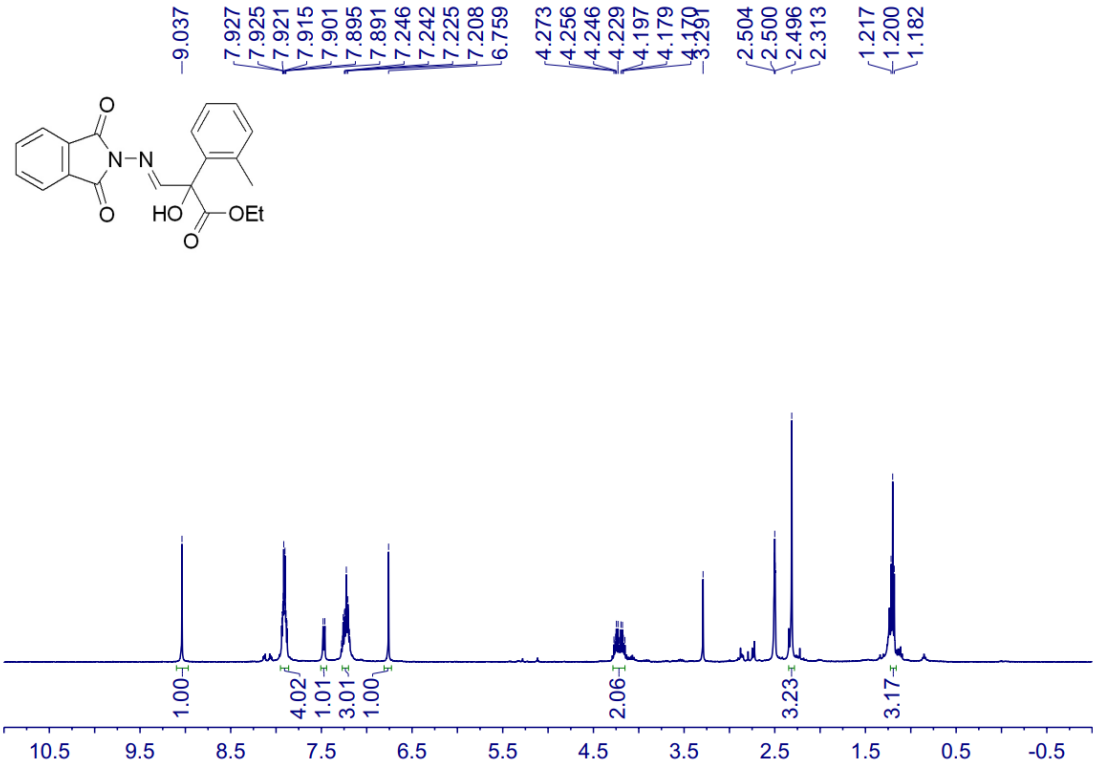
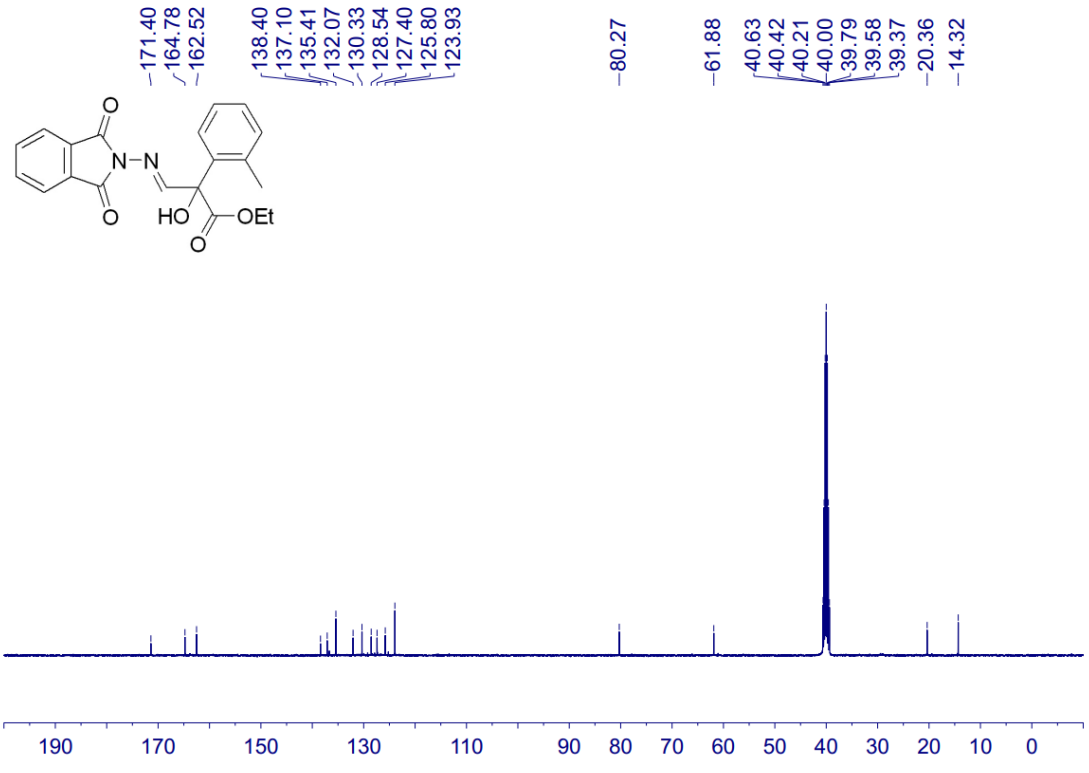


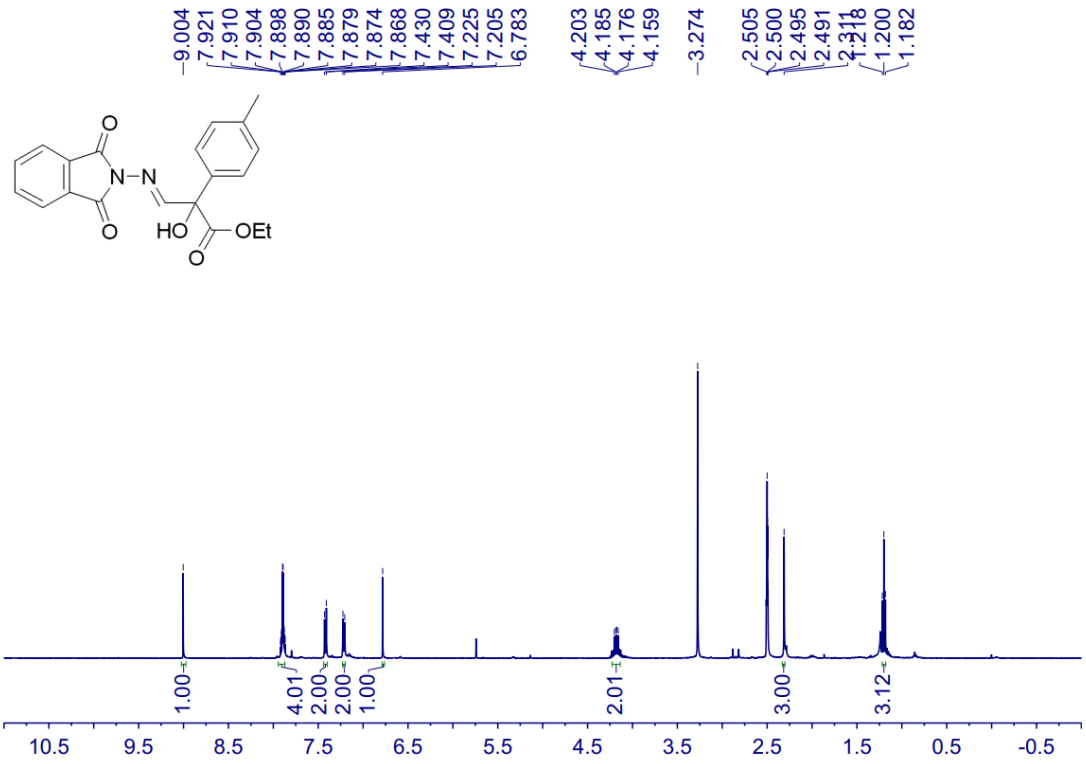
^{13}C NMR Spectrum of Compound **4a** (100 MHz, DMSO-d_6)



^{1}H NMR Spectrum of Compound **4b** (400 MHz, DMSO-d_6)



^{13}C NMR Spectrum of Compound **4b** (100 MHz, DMSO-d_6)



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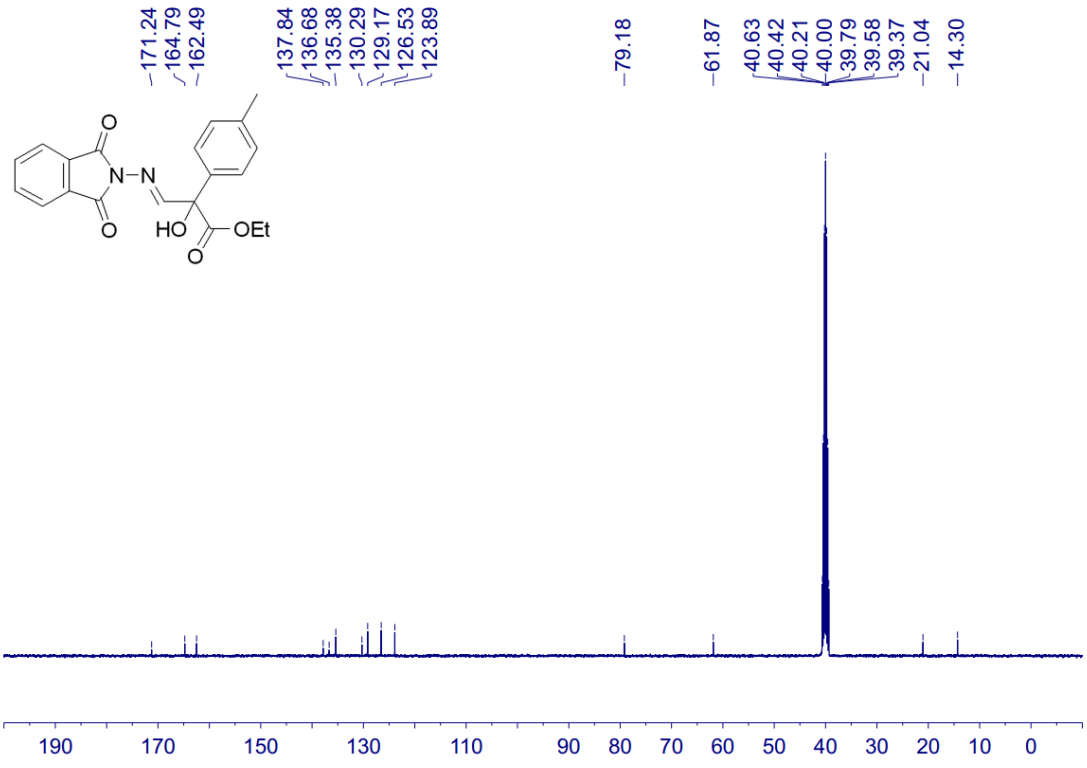

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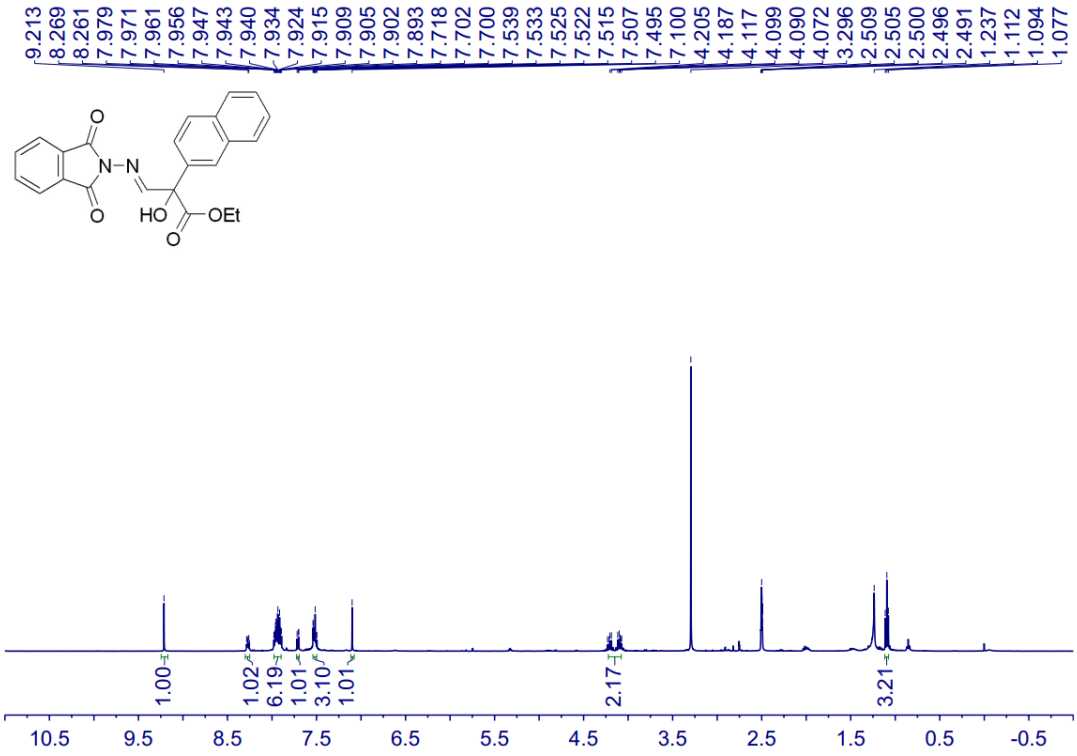

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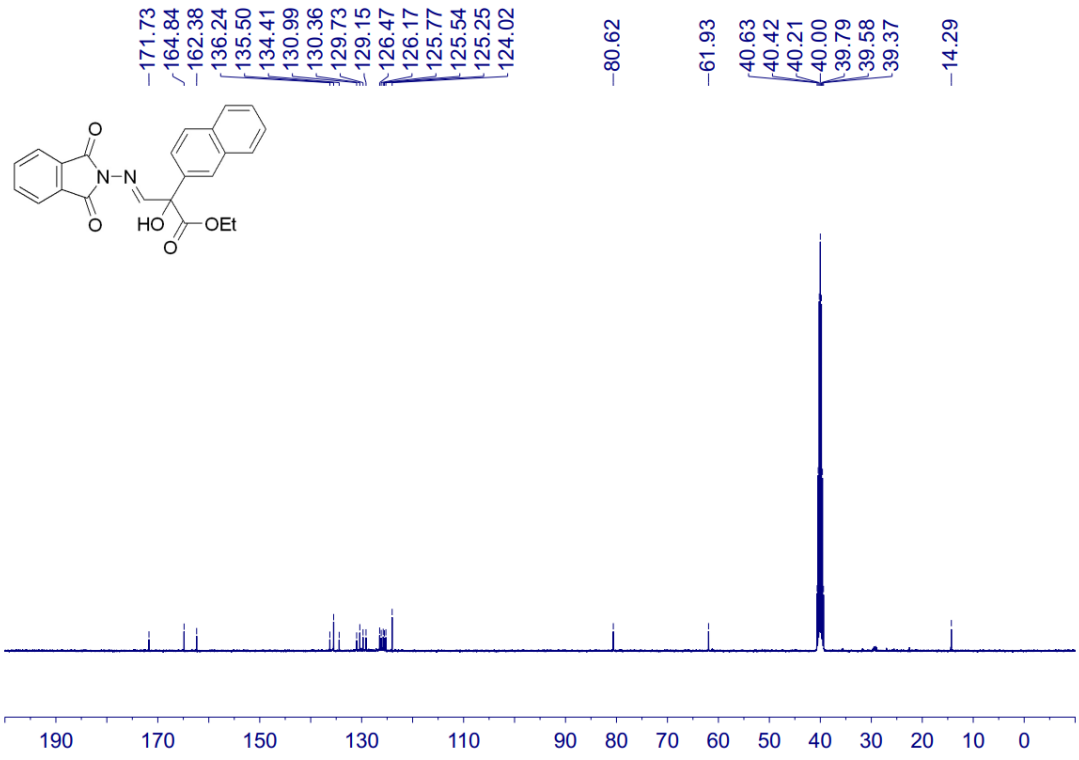

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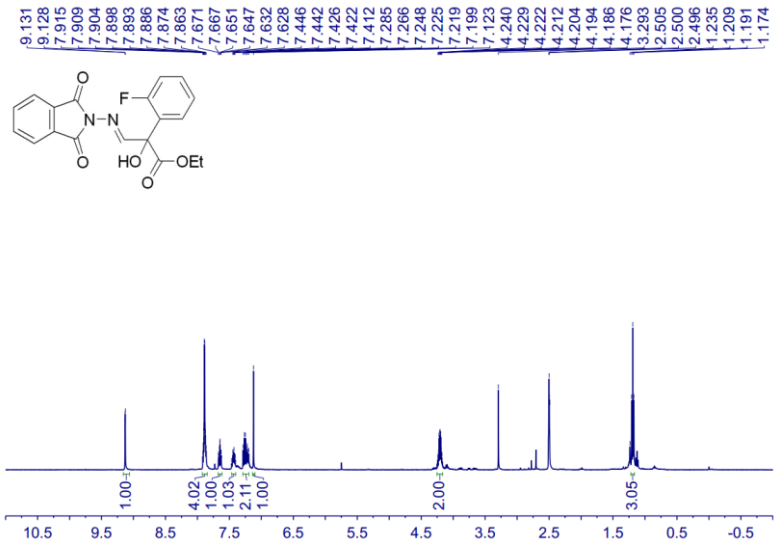
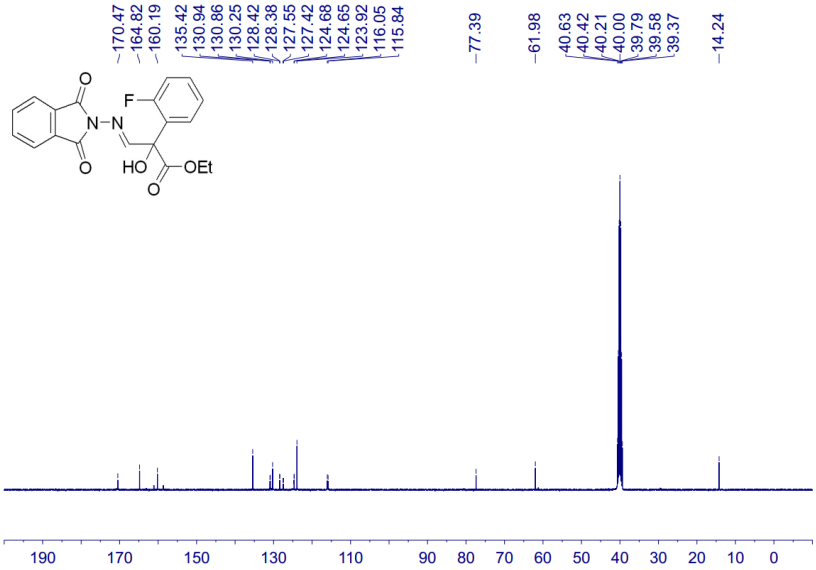
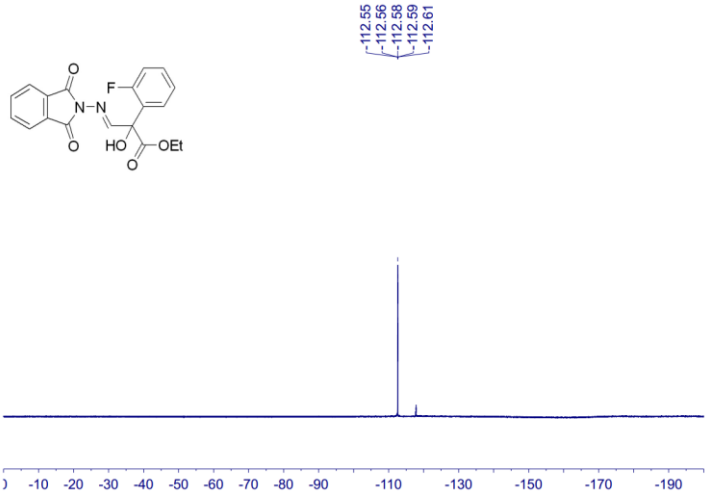


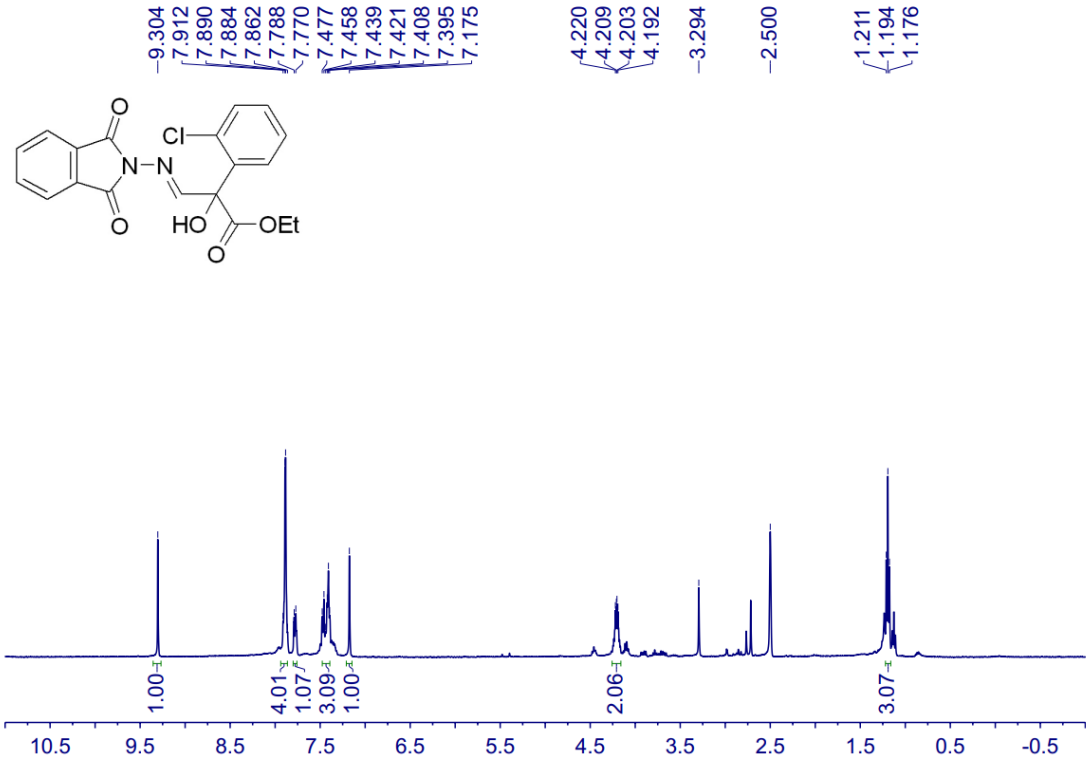
^{1}H NMR Spectrum of Compound **4e** (400 MHz, DMSO-d_6)



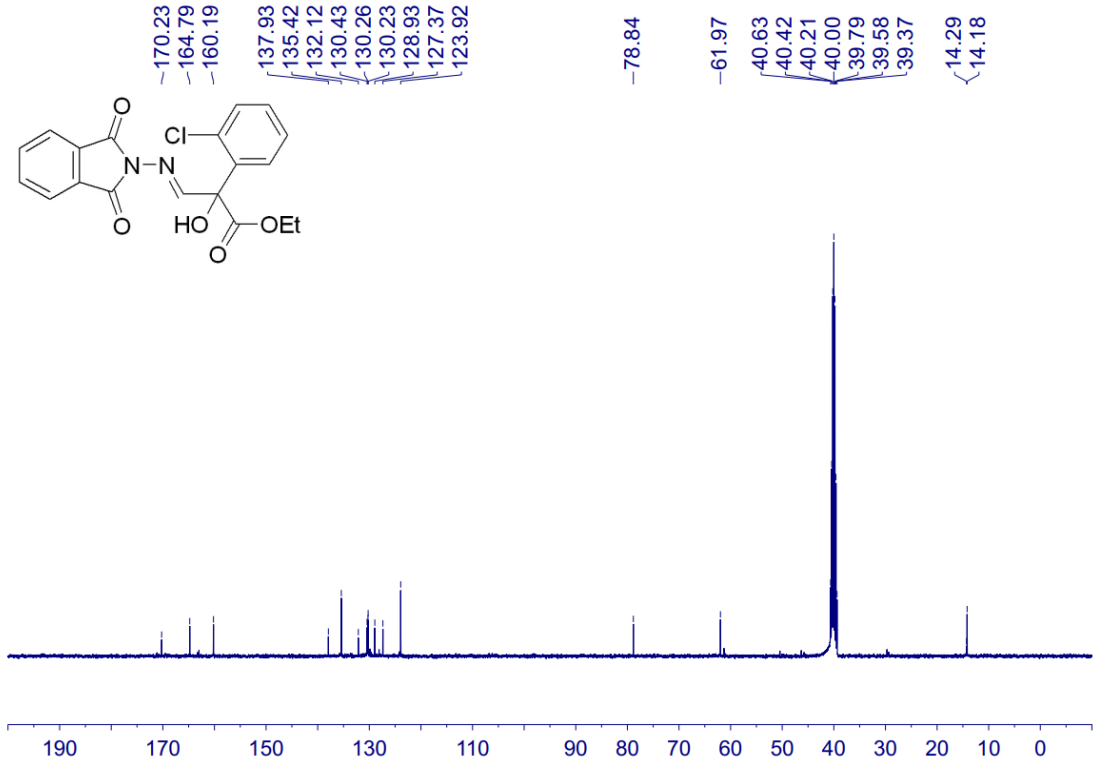
^{13}C NMR Spectrum of Compound **4e** (100 MHz, DMSO-d_6)



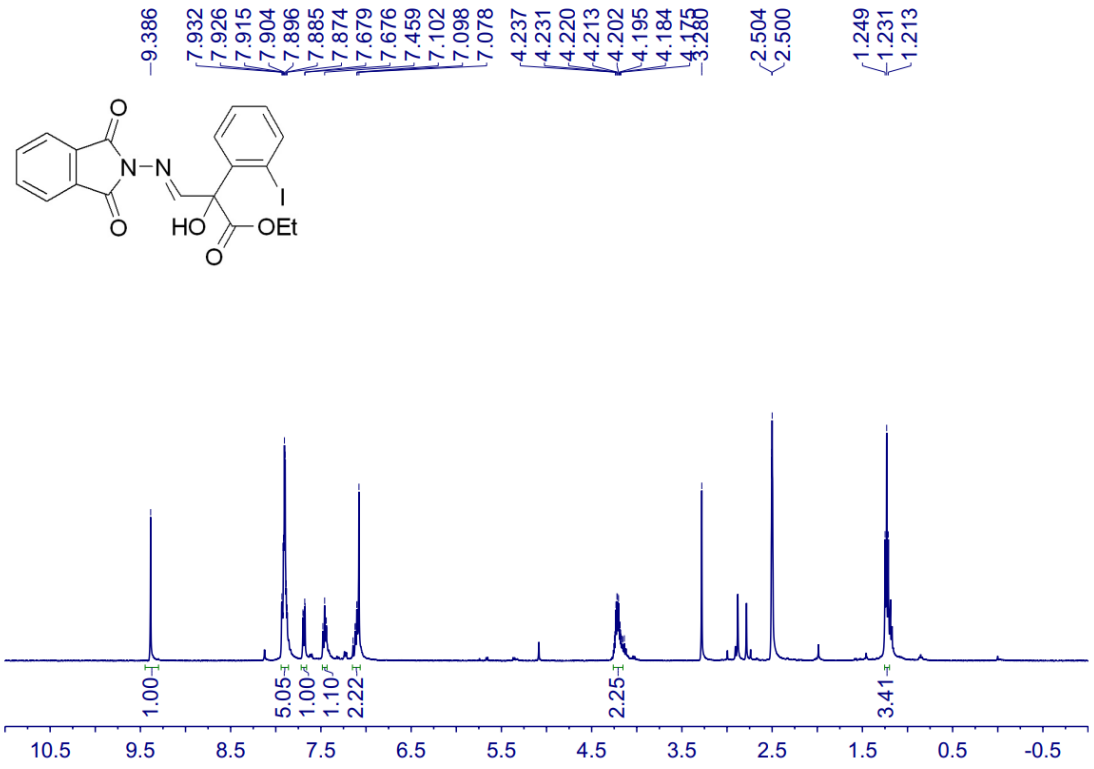
^{19}F NMR Spectrum of Compound **4e** (376 MHz, DMSO-d_6)



^{1}H NMR Spectrum of Compound **4f** (400 MHz, DMSO-d_6)



^{13}C NMR Spectrum of Compound **4f** (100 MHz, DMSO-d_6)



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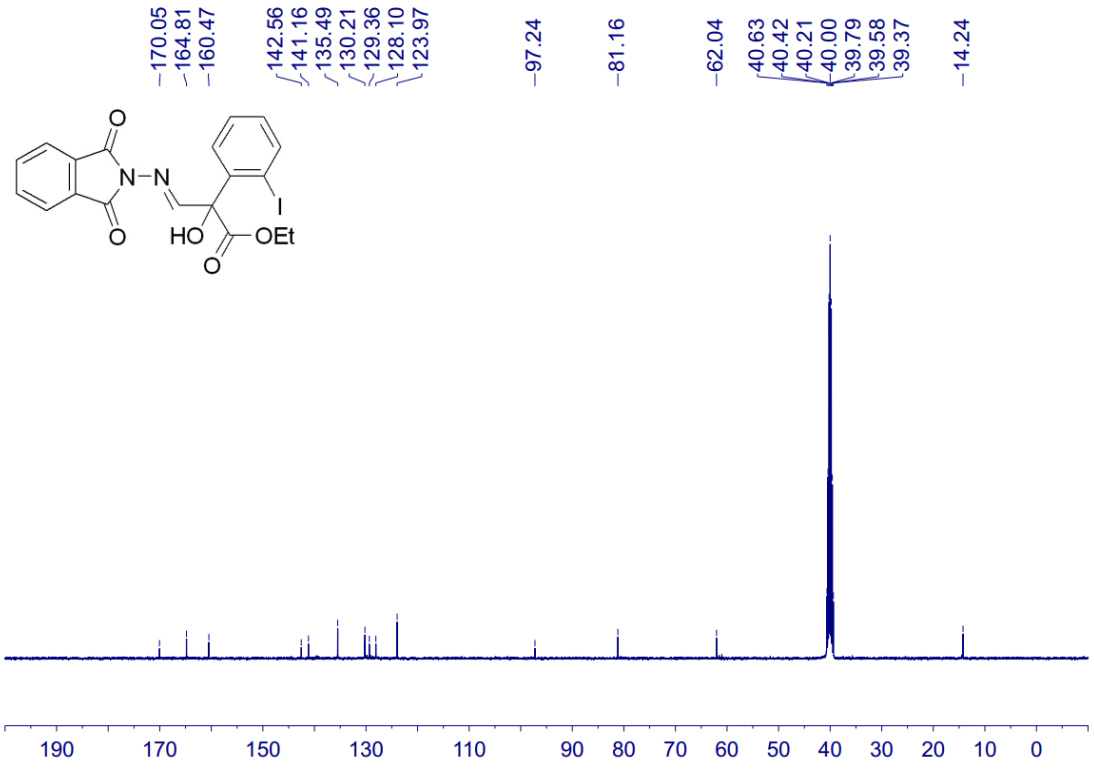
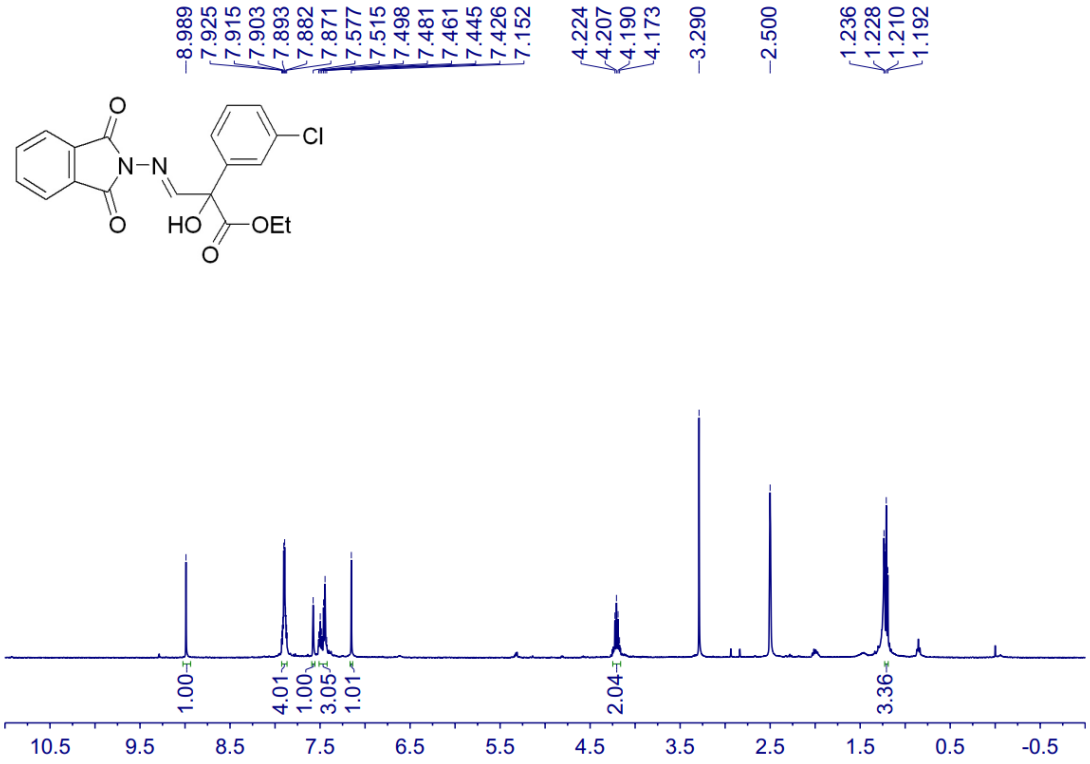
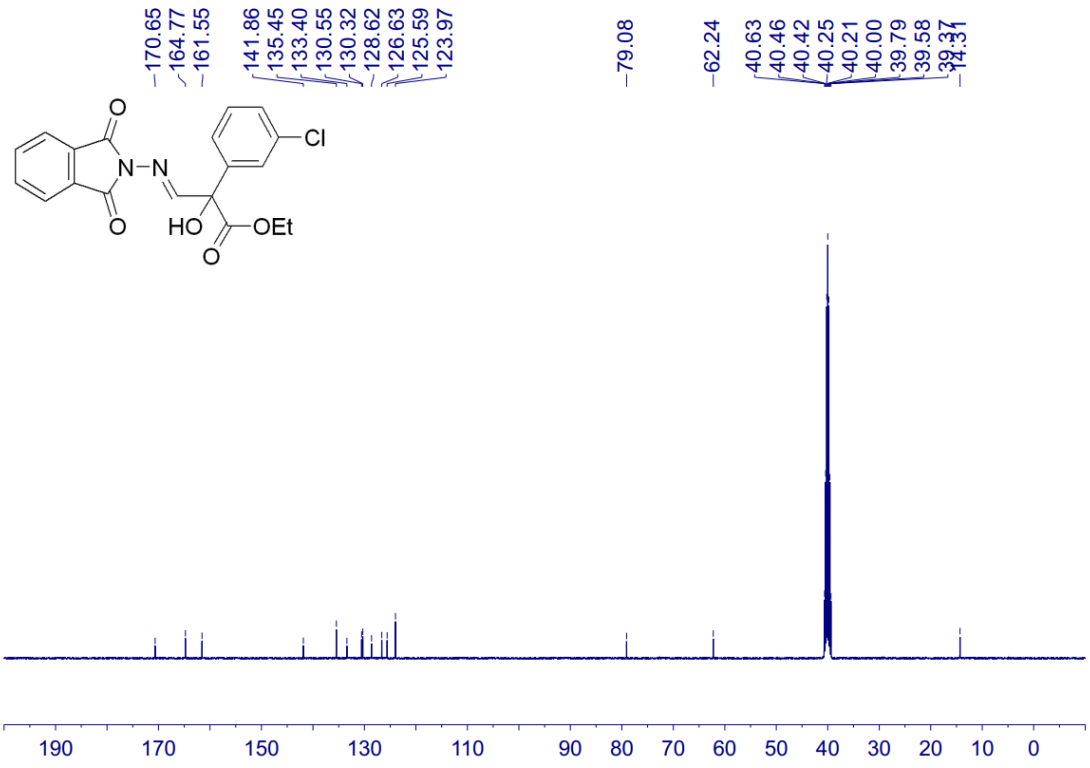


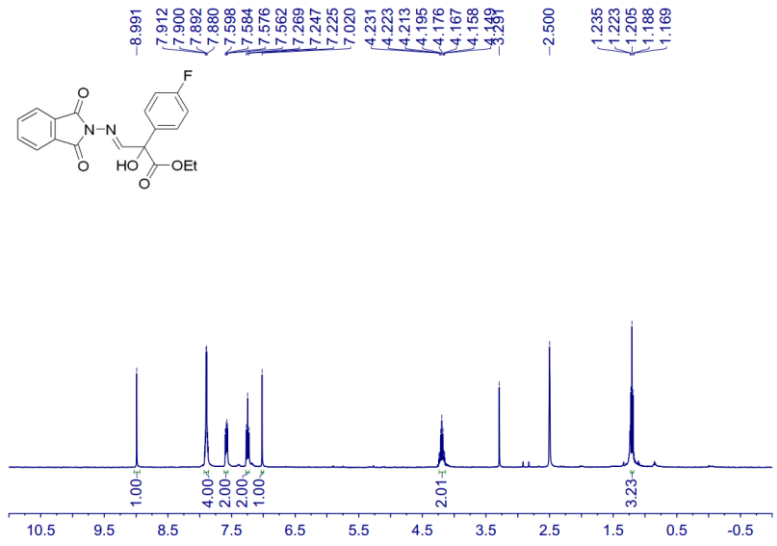
^{13}C NMR Spectrum of Compound **4g** (100 MHz, DMSO-d_6)



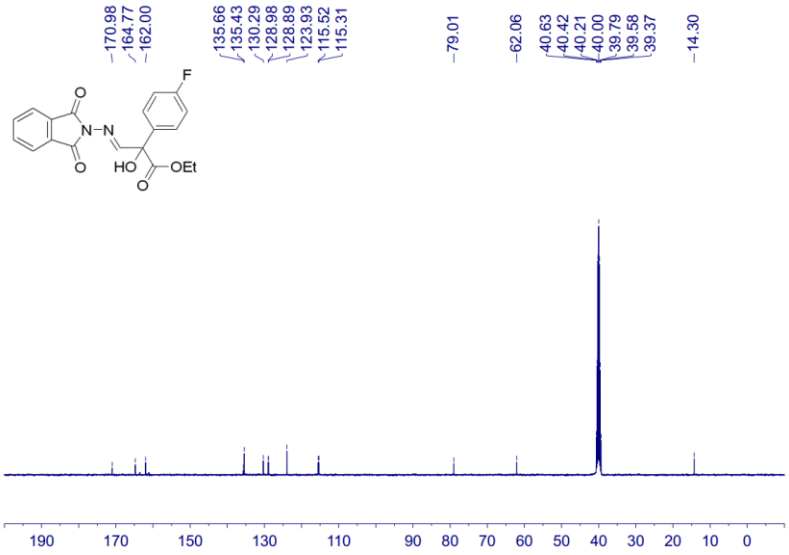
^{1}H NMR Spectrum of Compound **4h** (400 MHz, DMSO-d_6)



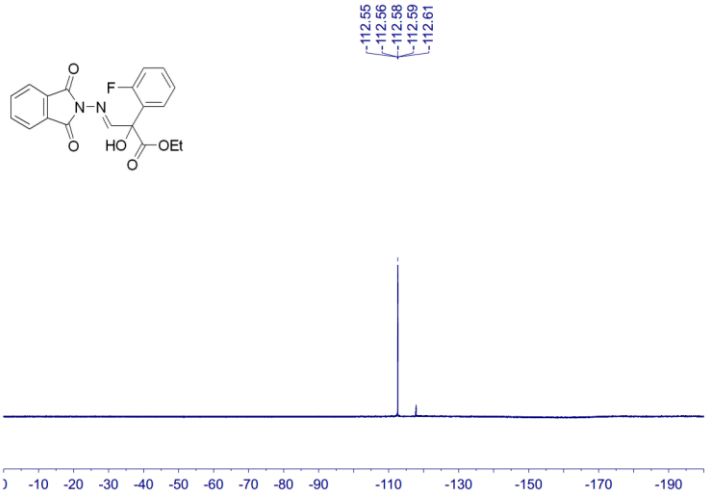
^{13}C NMR Spectrum of Compound **4h** (100 MHz, DMSO-d_6)



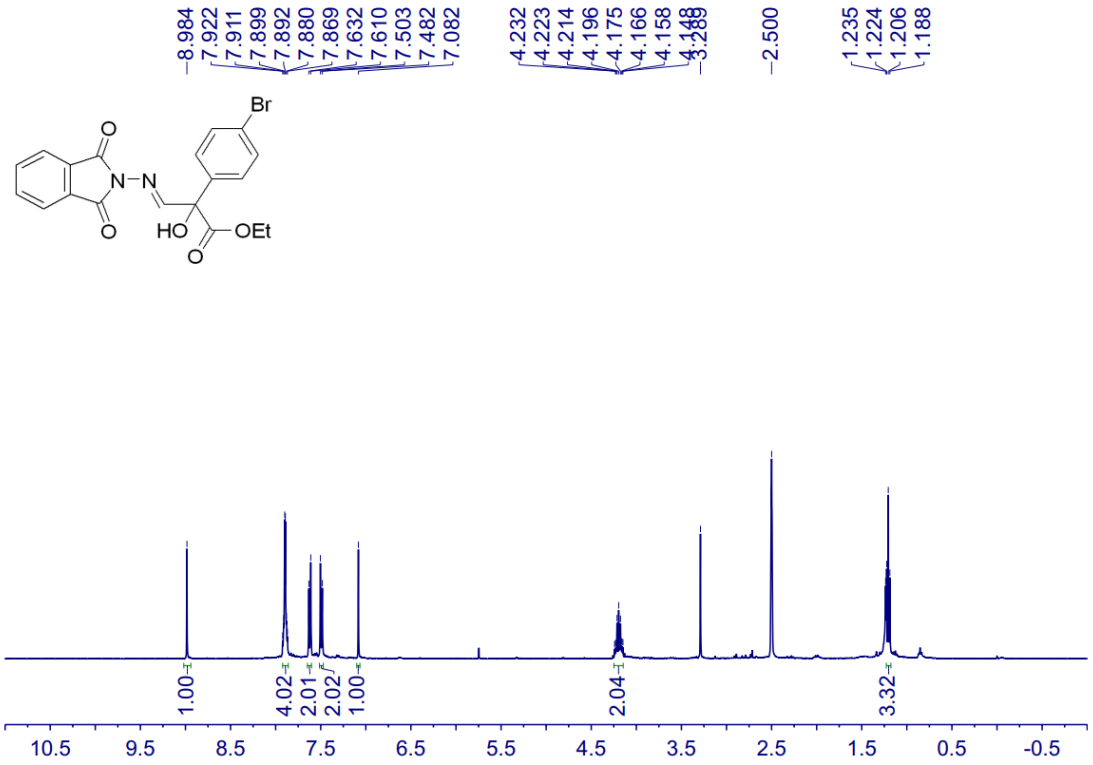
^{1}H NMR Spectrum of Compound **4i** (400 MHz, DMSO-d_6)



^{13}C NMR Spectrum of Compound **4i** (100 MHz, DMSO-d_6)



^{19}F NMR Spectrum of Compound **4i** (376 MHz, DMSO-d_6)



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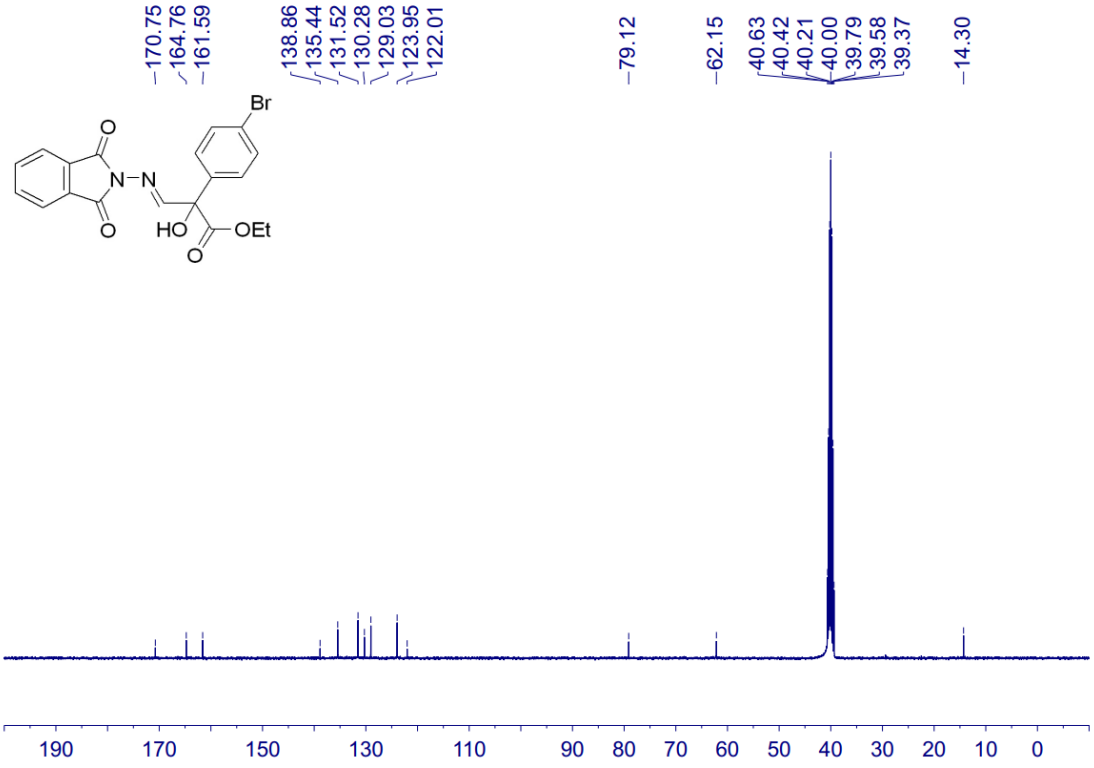
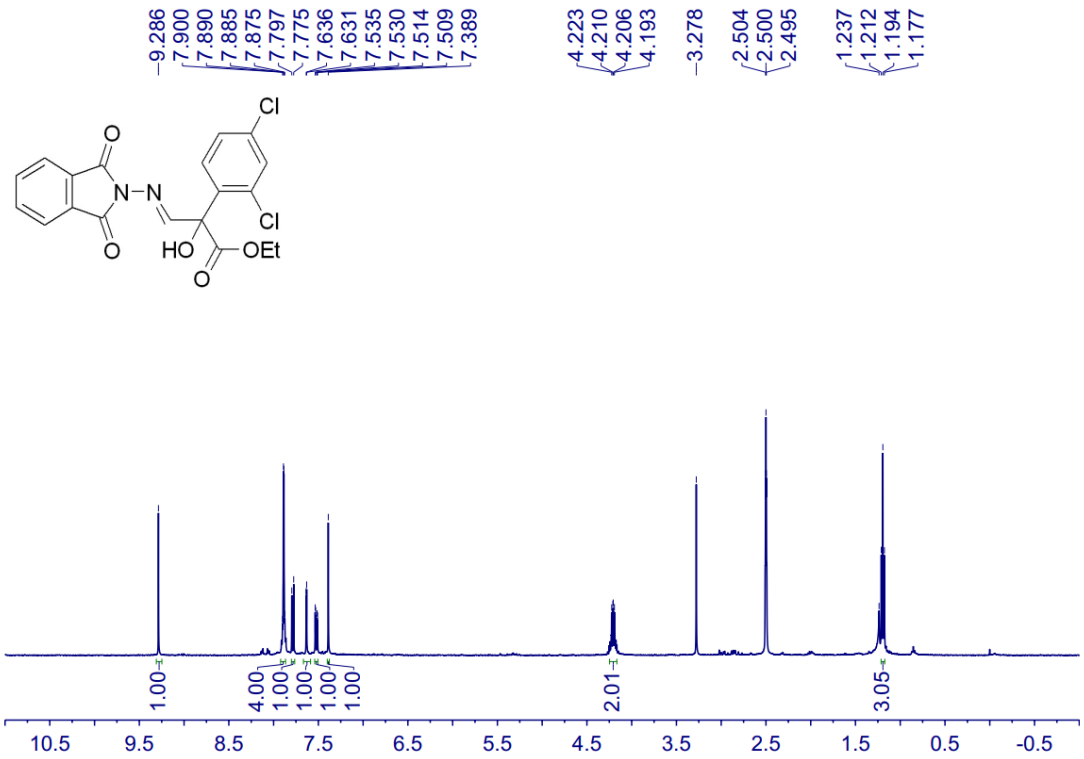
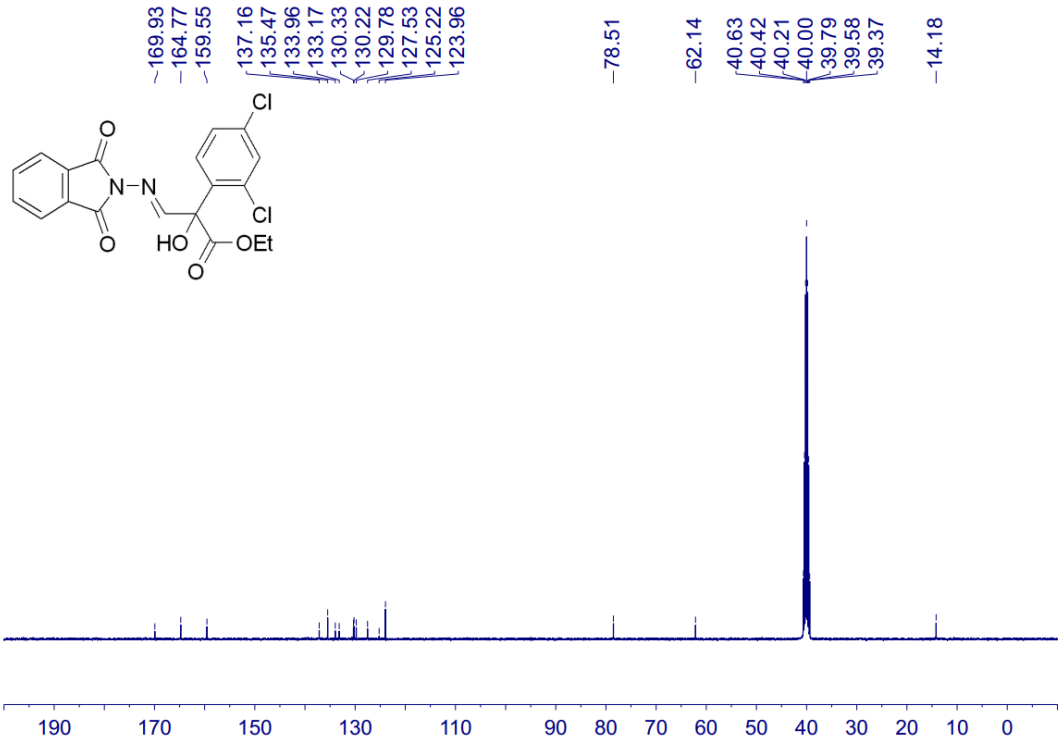


^{13}C NMR Spectrum of Compound **4j** (100 MHz, DMSO-d_6)



^{1}H NMR Spectrum of Compound **4k** (400 MHz, DMSO-d_6)



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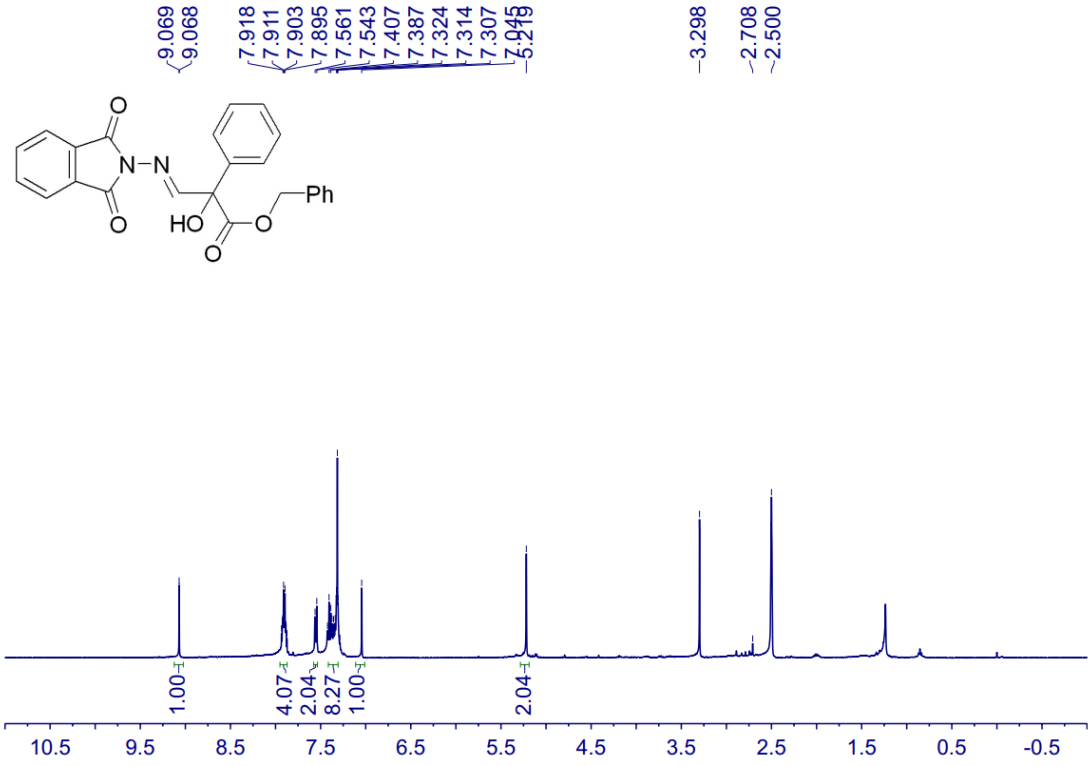

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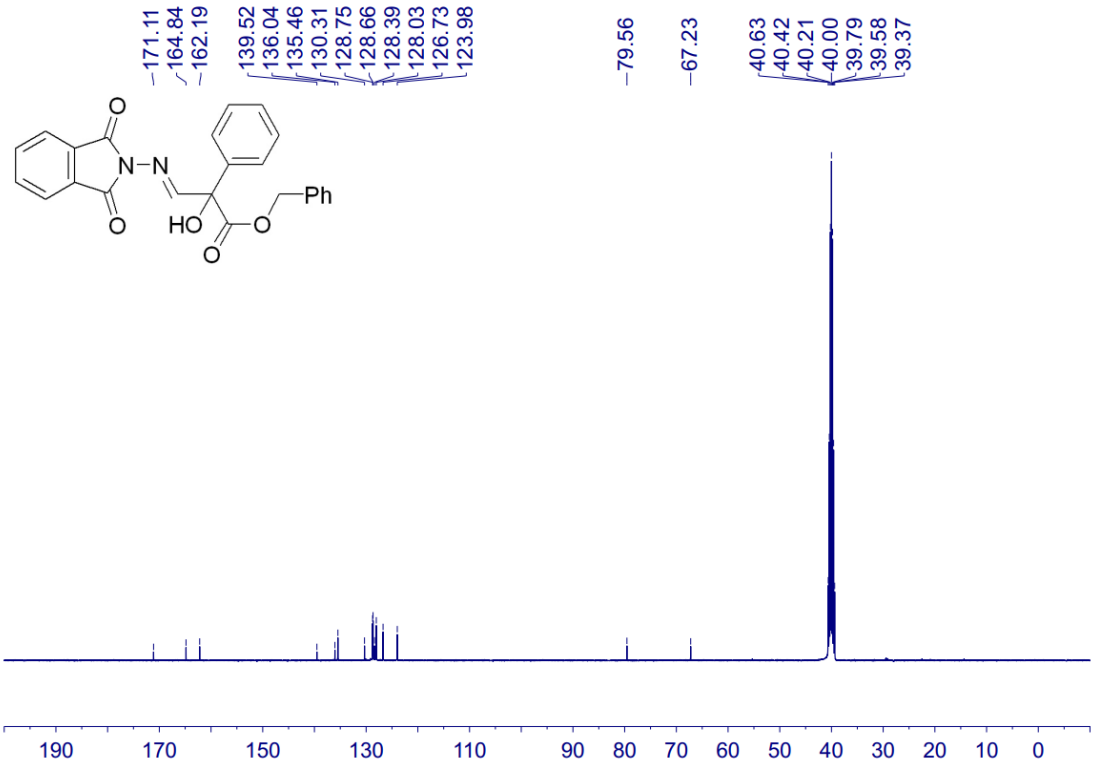

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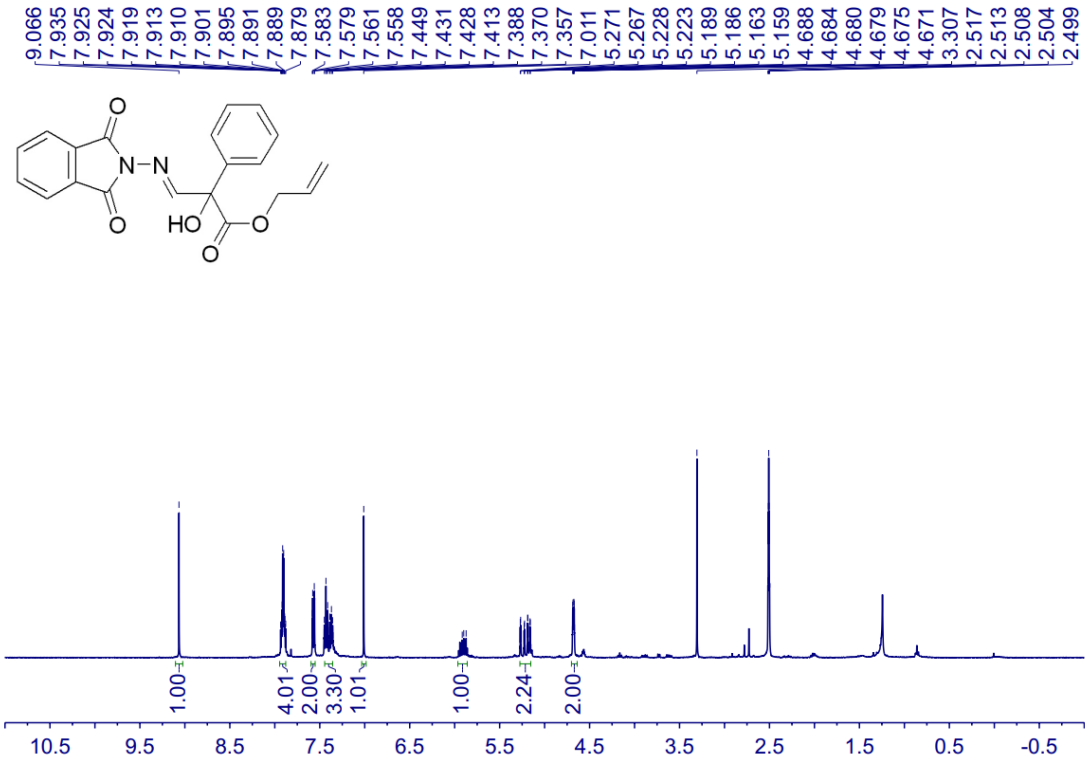
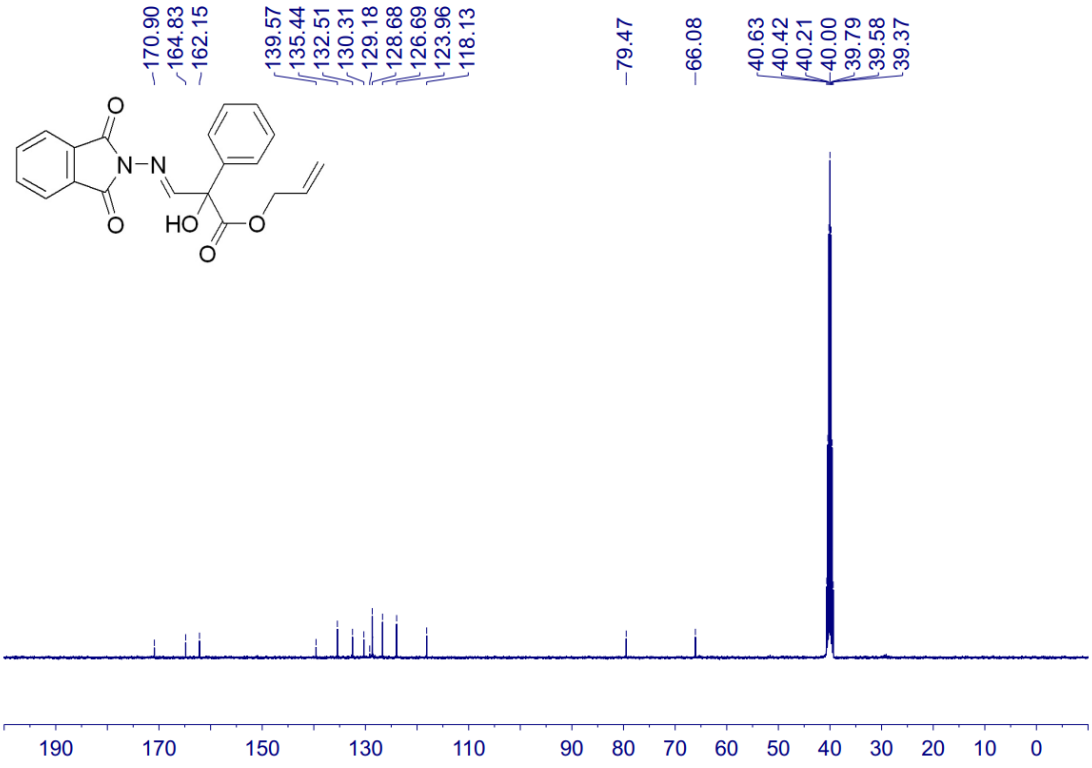
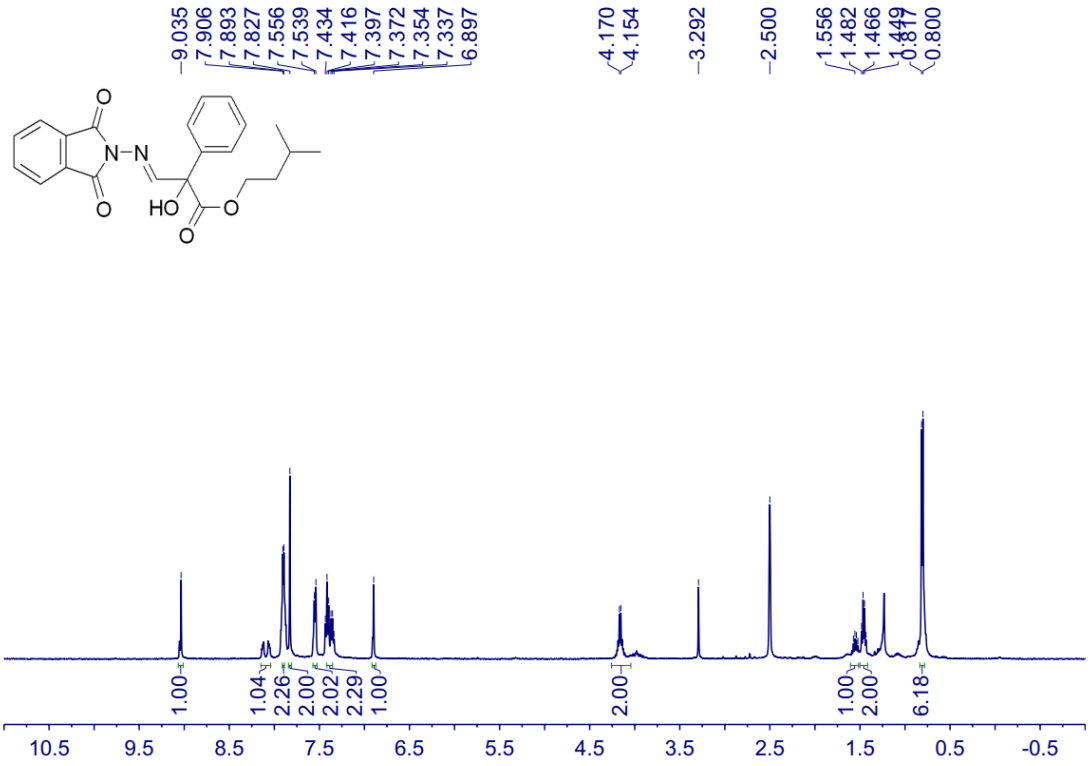


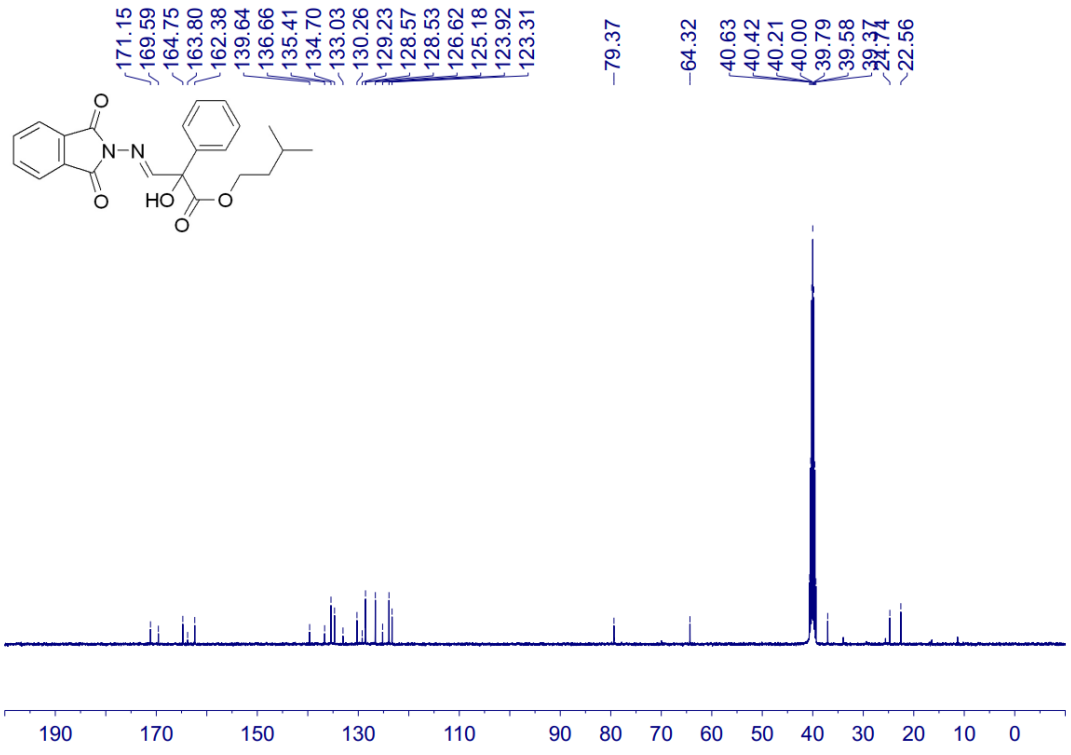
^{1}H NMR Spectrum of Compound **4m** (400 MHz, DMSO-d_6)



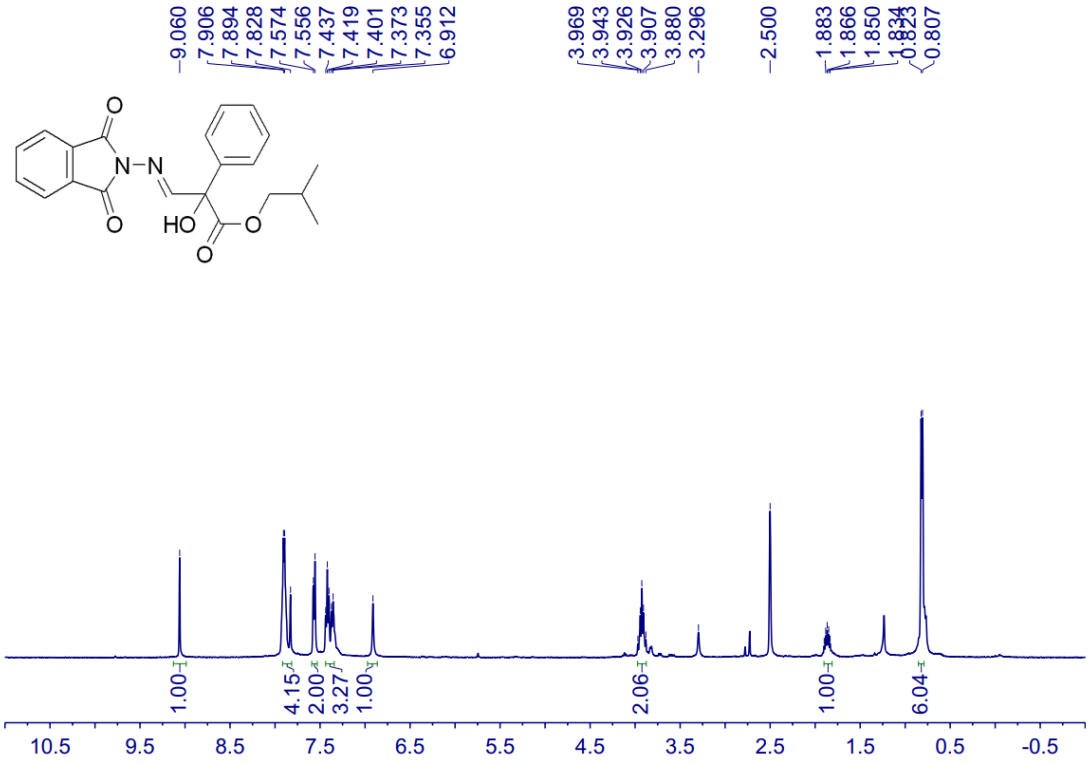
^{13}C NMR Spectrum of Compound **4m** (100 MHz, DMSO-d_6)



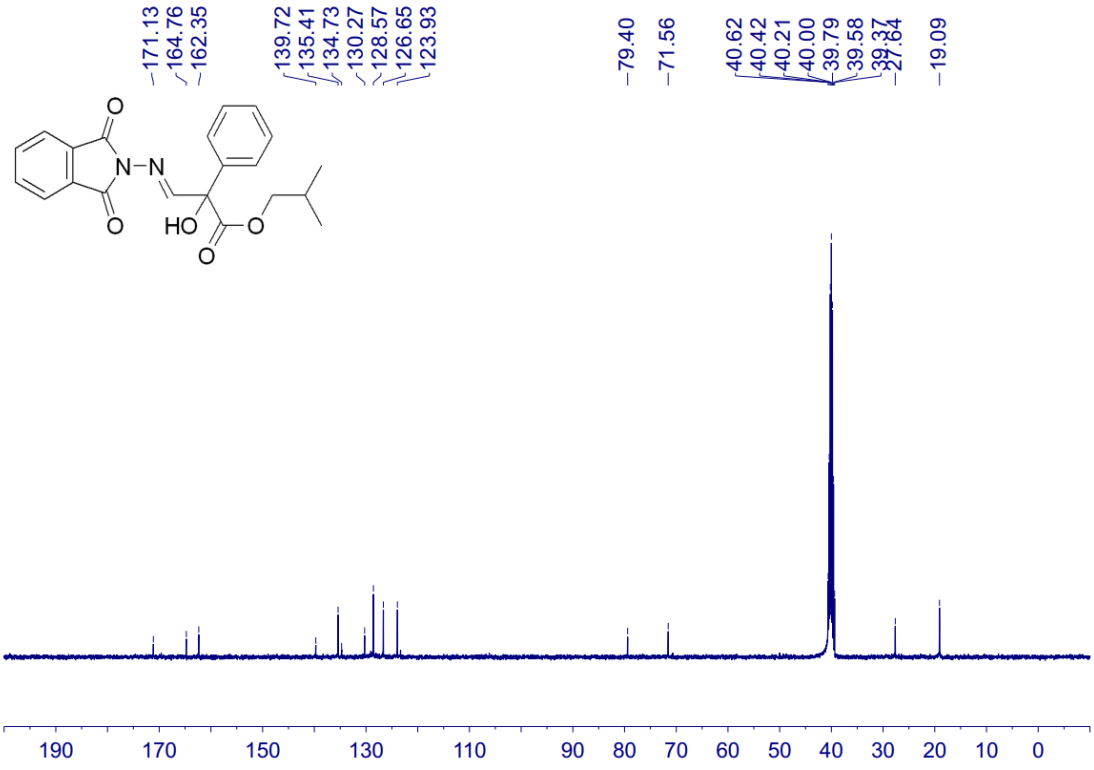
^{1}H NMR Spectrum of Compound **4n** (400 MHz, DMSO-d_6)



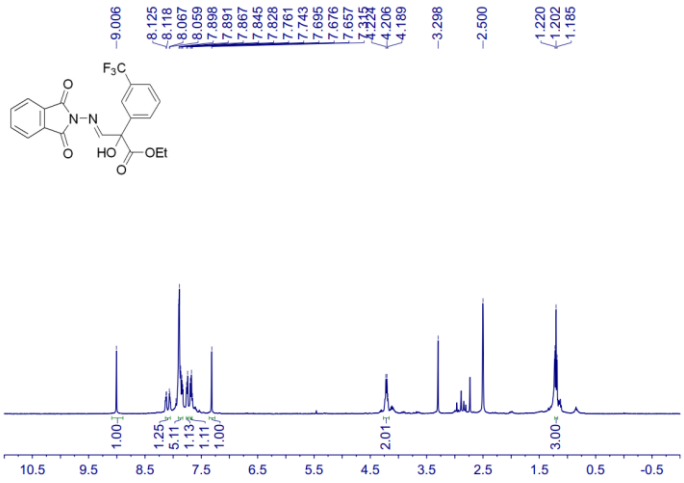
^{13}C NMR Spectrum of Compound **4n** (100 MHz, DMSO-d_6)



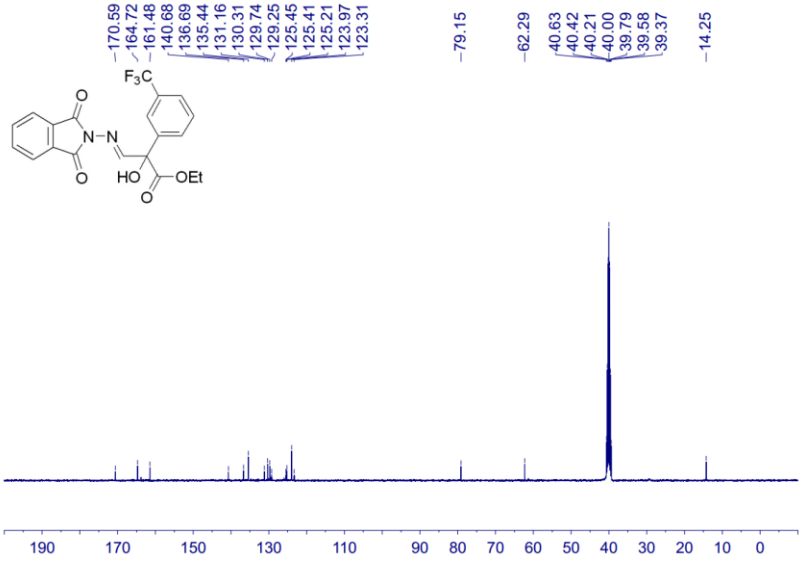
^{1}H NMR Spectrum of Compound **4o** (400 MHz, DMSO-d_6)



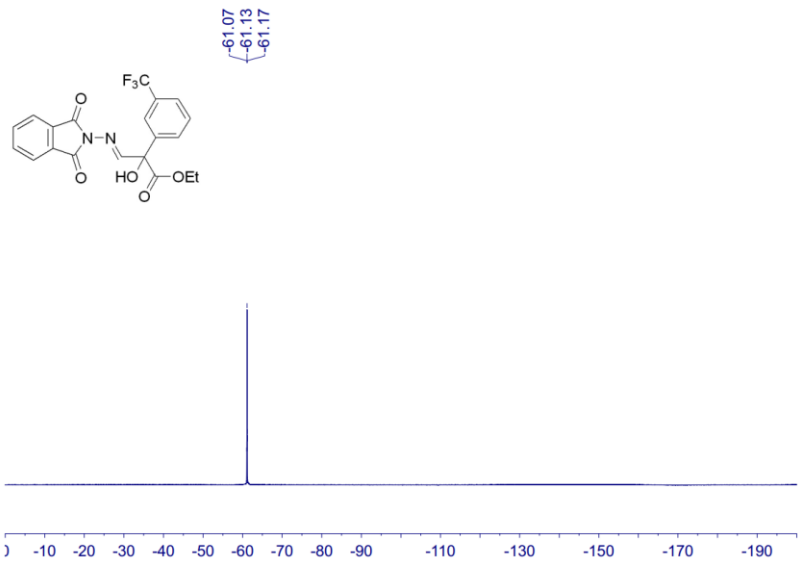
^{13}C NMR Spectrum of Compound **4o** (100 MHz, DMSO-d_6)



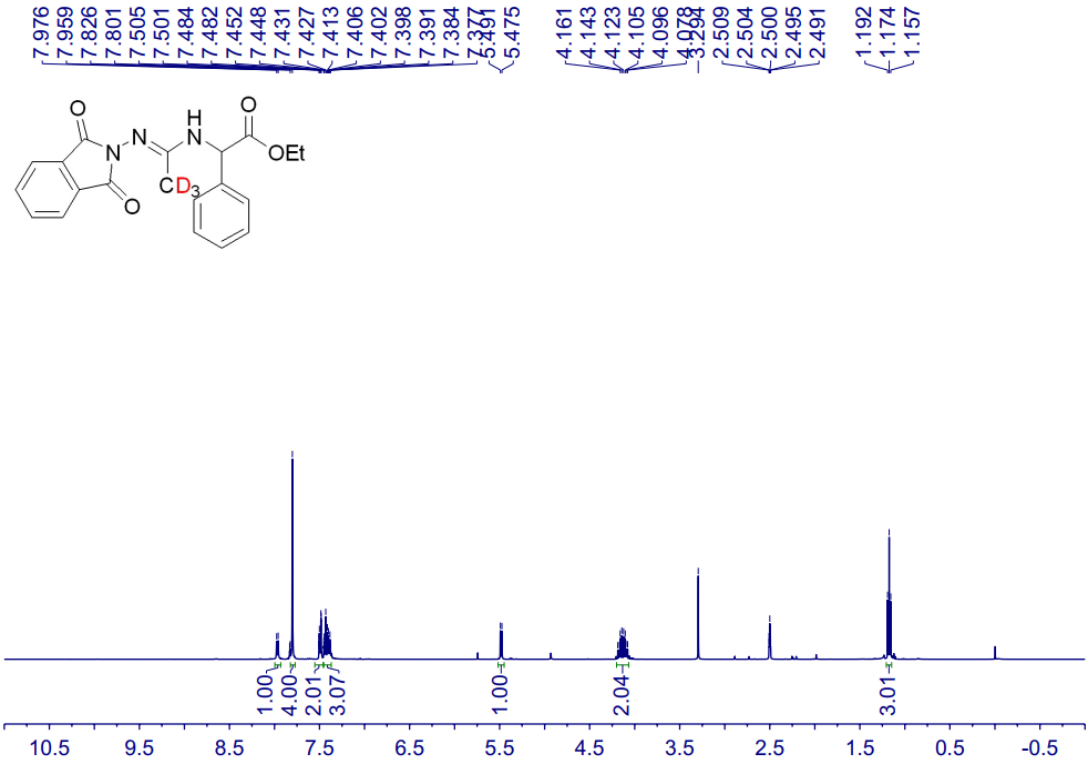
^{1}H NMR Spectrum of Compound **4q** (400 MHz, DMSO-d_6)



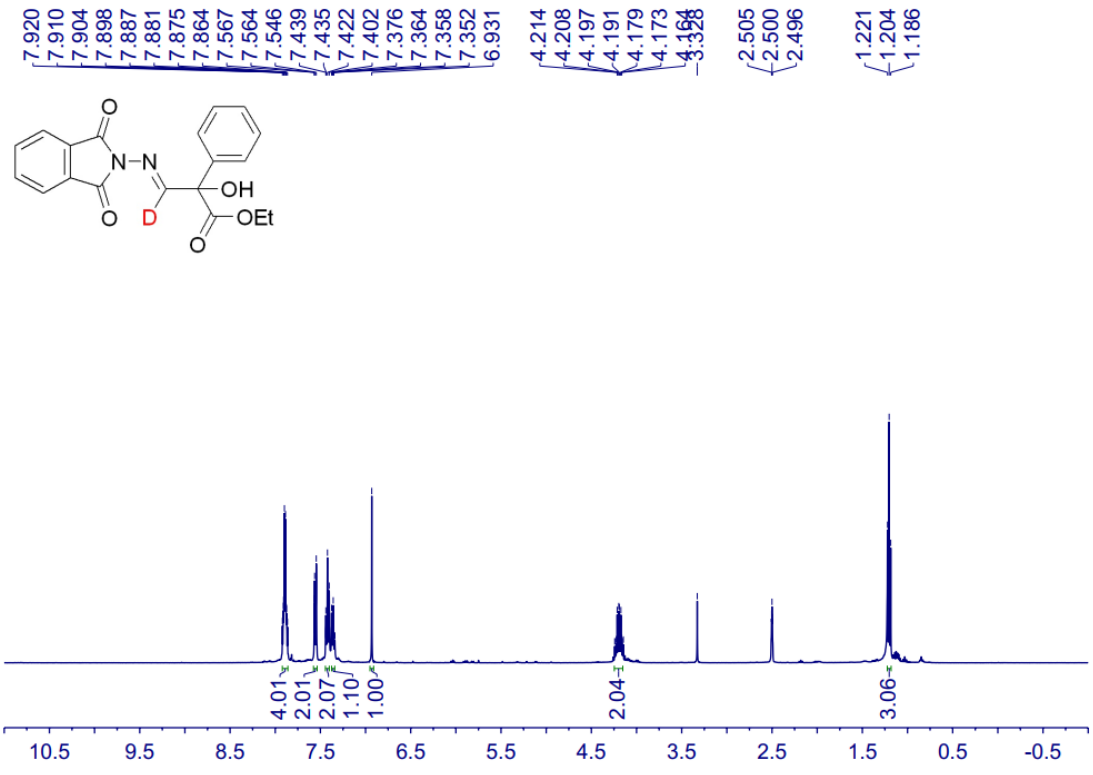
^{13}C NMR Spectrum of Compound **4q** (100 MHz, DMSO-d_6)



^{19}F NMR Spectrum of Compound **4i** (376 MHz, DMSO-d_6)

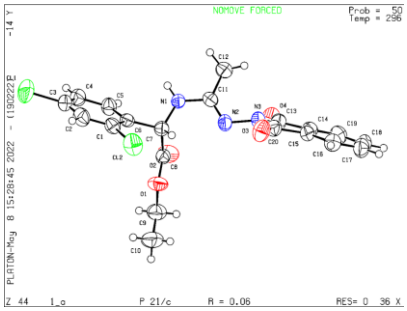


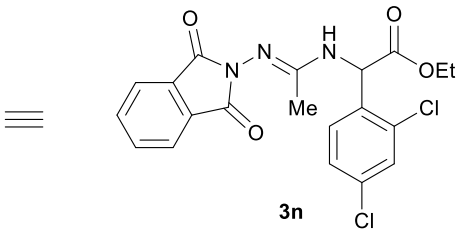
^{1}H NMR Spectrum of Compound **3a'** (400 MHz, DMSO-d_6)



^{1}H NMR Spectrum of Compound **4a'** (400 MHz, DMSO-d_6)

7. X-ray Diffraction Parameters and Data of 3n and 4k





3n

Bond precision: C-C = 0.0045 A Wavelength=0.71073

Cell: a=11.74000 b=19.00700 c=8.97600
alpha=90 beta=95.3100 gamma=90
Temperature: 296 K

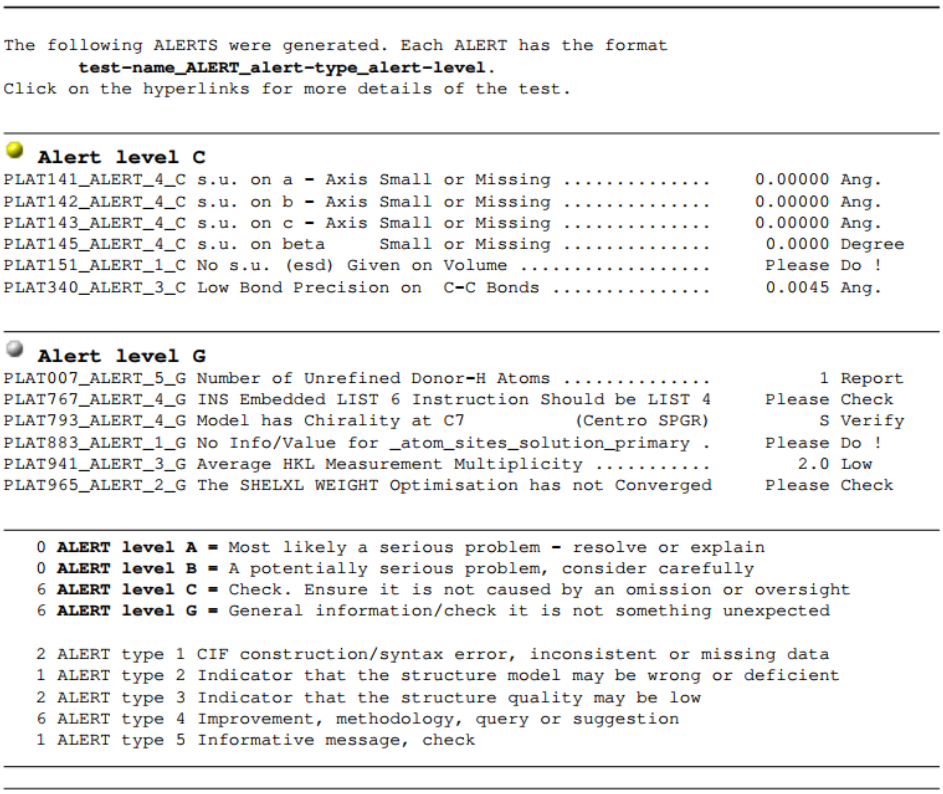
	Calculated	Reported
Volume	1994.329	1994
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C20 H17 Cl2 N3 O4	?
Sum formula	C20 H17 Cl2 N3 O4	C20 H17 Cl2 N3 O4
Mr	434.27	434.26
Dx,g cm-3	1.446	1.446
Z	4	4
Mu (mm-1)	0.358	0.358
F000	896.0	896.0
F000'	897.56	
h,k,lmax	13,22,10	13,22,10
Nref	3540	3536
Tmin,Tmax	0.931,0.931	0.864,0.864
Tmin'	0.931	

Correction method= # Reported T Limits: Tmin=0.864 Tmax=0.864
AbsCorr = MULTI-SCAN

Data completeness= 0.999 Theta(max)= 25.064

R(reflections)= 0.0644(2265) wR2(reflections)= 0.1288(3536)

S = 1.138 Npar= 264



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The following ALERTS were generated. Each ALERT has the format
      test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

 Alert level C
PLAT141_ALERT_4_C s.u. on a - Axis Small or Missing ..............    0.00000 Ang.
PLAT142_ALERT_4_C s.u. on b - Axis Small or Missing ..............    0.00000 Ang.
PLAT143_ALERT_4_C s.u. on c - Axis Small or Missing ..............    0.00000 Ang.
PLAT145_ALERT_4_C s.u. on beta     Small or Missing ..............     0.0000 Degree
PLAT151_ALERT_1_C No s.u. (esd) Given on Volume ..................     Please Do !
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ...............     0.0045 Ang.

 Alert level G
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............         1 Report
PLAT767_ALERT_4_G INS Embedded LIST 6 Instruction Should be LIST 4     Please Check
PLAT793_ALERT_4_G Model has Chirality at C7          (Centro SPGR)         S Verify
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .     Please Do !
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........       2.0 Low
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged     Please Check

   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   6 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   6 ALERT level G = General information/check it is not something unexpected

   2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   1 ALERT type 2 Indicator that the structure model may be wrong or deficient
   2 ALERT type 3 Indicator that the structure quality may be low
   6 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
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Figure S1 The single crystal analysis for **3n** (**CCDC number: 2205769**)

Table 1-1. Crystal data and structure refinement for 1_a.

Identification code	1_a	
Empirical formula	C20 H17 Cl2 N3 O4	
Formula weight	434.26	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.740 Å	α= 90°.
	b = 19.007 Å	β= 95.31°.
	c = 8.976 Å	γ = 90°.
Volume	1994.2 $Å^3$	
Z	4	
Density (calculated)	1.446 Mg/m^3	
Absorption coefficient	0.358 mm^{-1}	
F(000)	896	
Crystal size	0.200 x 0.200 x 0.200 mm^3	
Theta range for data collection	2.143 to 25.064°.	
Index ranges	0<=h<=13, -22<=k<=22, -10<=l<=10	

Reflections collected	6914
Independent reflections	3536 [R(int) = 0.0453]
Completeness to theta = 25.064°	99.9 %
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3536 / 0 / 264
Goodness-of-fit on F^2	1.138
Final R indices [I>2sigma(I)]	R1 = 0.0644, wR2 = 0.1111
R indices (all data)	R1 = 0.1256, wR2 = 0.1288
Extinction coefficient	n/a
Largest diff. peak and hole	0.185 and -0.226 e.$Å^{-3}$

Table 1-2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for 1_a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(7)	5042(2)	4014(1)	2247(3)	40(1)
C(15)	8943(2)	5153(2)	6757(3)	43(1)
C(11)	5746(3)	5100(2)	3463(3)	41(1)
C(8)	5861(2)	3908(2)	1047(3)	43(1)
C(6)	3885(2)	3699(1)	1729(3)	37(1)
C(5)	3211(3)	4014(2)	575(3)	48(1)
C(14)	9116(3)	5651(2)	5685(3)	44(1)
C(13)	8169(3)	5606(2)	4492(4)	47(1)
C(20)	7873(3)	4775(2)	6294(3)	47(1)
C(1)	3450(3)	3099(2)	2353(3)	45(1)
C(12)	5522(3)	5865(2)	3674(4)	55(1)
C(16)	9737(3)	5046(2)	7950(3)	54(1)
C(3)	1741(3)	3181(2)	769(4)	53(1)
C(4)	2137(3)	3767(2)	95(3)	54(1)
C(2)	2380(3)	2840(2)	1886(4)	55(1)
C(19)	10096(3)	6054(2)	5776(4)	57(1)
C(17)	10720(3)	5454(2)	8054(4)	61(1)
C(18)	10896(3)	5948(2)	6977(4)	62(1)
C(9)	7008(3)	3079(2)	-80(4)	68(1)
C(10)	7413(3)	2345(2)	219(5)	93(1)
Cl(1)	382(1)	2875(1)	165(1)	96(1)
Cl(2)	4250(1)	2640(1)	3763(1)	69(1)
N(1)	4925(2)	4748(1)	2600(3)	44(1)
N(2)	6601(2)	4729(1)	4020(3)	46(1)
N(3)	7431(2)	5096(1)	4956(3)	45(1)
O(1)	6270(2)	3259(1)	1075(2)	57(1)
O(2)	6093(2)	4344(1)	170(3)	64(1)
O(3)	7449(2)	4286(1)	6887(3)	66(1)
O(4)	8033(2)	5929(1)	3332(3)	64(1)

Table 1-3. Bond lengths [Å] and angles [°] for 1_a.

C(7)-N(1)	1.440(3)
C(7)-C(6)	1.517(4)
C(7)-C(8)	1.522(4)
C(7)-H(7)	0.9800
C(15)-C(16)	1.368(4)
C(15)-C(14)	1.379(4)
C(15)-C(20)	1.473(4)
C(11)-N(2)	1.288(3)
C(11)-N(1)	1.357(3)
C(11)-C(12)	1.493(4)
C(8)-O(2)	1.192(3)
C(8)-O(1)	1.323(3)
C(6)-C(5)	1.381(4)
C(6)-C(1)	1.388(4)
C(5)-C(4)	1.377(4)
C(5)-H(5)	0.9300
C(14)-C(19)	1.378(4)
C(14)-C(13)	1.472(4)
C(13)-O(4)	1.206(3)
C(13)-N(3)	1.390(4)
C(20)-O(3)	1.201(3)
C(20)-N(3)	1.403(4)
C(1)-C(2)	1.377(4)
C(1)-Cl(2)	1.740(3)
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(16)-C(17)	1.386(4)
C(16)-H(16)	0.9300
C(3)-C(2)	1.361(4)
C(3)-C(4)	1.368(4)
C(3)-Cl(1)	1.737(3)
C(4)-H(4)	0.9300
C(2)-H(2)	0.9300
C(19)-C(18)	1.377(4)
C(19)-H(19)	0.9300

C(17)-C(18)	1.378(4)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
C(9)-O(1)	1.452(4)
C(9)-C(10)	1.489(4)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
N(1)-H(1)	0.8600
N(2)-N(3)	1.411(3)

N(1)-C(7)-C(6)	110.3(2)
N(1)-C(7)-C(8)	111.3(2)
C(6)-C(7)-C(8)	109.7(2)
N(1)-C(7)-H(7)	108.5
C(6)-C(7)-H(7)	108.5
C(8)-C(7)-H(7)	108.5
C(16)-C(15)-C(14)	121.0(3)
C(16)-C(15)-C(20)	130.5(3)
C(14)-C(15)-C(20)	108.3(3)
N(2)-C(11)-N(1)	116.0(3)
N(2)-C(11)-C(12)	128.6(3)
N(1)-C(11)-C(12)	115.4(3)
O(2)-C(8)-O(1)	123.8(3)
O(2)-C(8)-C(7)	125.0(3)
O(1)-C(8)-C(7)	111.2(3)
C(5)-C(6)-C(1)	116.9(3)
C(5)-C(6)-C(7)	119.6(3)
C(1)-C(6)-C(7)	123.5(2)
C(4)-C(5)-C(6)	122.1(3)
C(4)-C(5)-H(5)	119.0
C(6)-C(5)-H(5)	119.0
C(19)-C(14)-C(15)	121.1(3)
C(19)-C(14)-C(13)	130.2(3)
C(15)-C(14)-C(13)	108.5(3)
O(4)-C(13)-N(3)	125.2(3)

O(4)-C(13)-C(14)	129.0(3)
N(3)-C(13)-C(14)	105.8(3)
O(3)-C(20)-N(3)	125.4(3)
O(3)-C(20)-C(15)	129.0(3)
N(3)-C(20)-C(15)	105.6(3)
C(2)-C(1)-C(6)	122.0(3)
C(2)-C(1)-Cl(2)	117.2(2)
C(6)-C(1)-Cl(2)	120.8(2)
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(15)-C(16)-C(17)	118.2(3)
C(15)-C(16)-H(16)	120.9
C(17)-C(16)-H(16)	120.9
C(2)-C(3)-C(4)	121.8(3)
C(2)-C(3)-Cl(1)	119.9(3)
C(4)-C(3)-Cl(1)	118.3(3)
C(3)-C(4)-C(5)	118.6(3)
C(3)-C(4)-H(4)	120.7
C(5)-C(4)-H(4)	120.7
C(3)-C(2)-C(1)	118.6(3)
C(3)-C(2)-H(2)	120.7
C(1)-C(2)-H(2)	120.7
C(18)-C(19)-C(14)	118.1(3)
C(18)-C(19)-H(19)	121.0
C(14)-C(19)-H(19)	121.0
C(18)-C(17)-C(16)	120.8(3)
C(18)-C(17)-H(17)	119.6
C(16)-C(17)-H(17)	119.6
C(19)-C(18)-C(17)	120.9(3)
C(19)-C(18)-H(18)	119.5
C(17)-C(18)-H(18)	119.5
O(1)-C(9)-C(10)	107.1(3)
O(1)-C(9)-H(9A)	110.3
C(10)-C(9)-H(9A)	110.3

O(1)-C(9)-H(9B)	110.3
C(10)-C(9)-H(9B)	110.3
H(9A)-C(9)-H(9B)	108.5
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(11)-N(1)-C(7)	121.7(2)
C(11)-N(1)-H(1)	119.1
C(7)-N(1)-H(1)	119.1
C(11)-N(2)-N(3)	115.5(2)
C(13)-N(3)-C(20)	111.6(2)
C(13)-N(3)-N(2)	125.6(2)
C(20)-N(3)-N(2)	118.4(2)
C(8)-O(1)-C(9)	116.5(2)

Symmetry transformations used to generate equivalent atoms:

Table 1-4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 1_a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(7)	40(2)	36(2)	42(2)	2(1)	0(1)	1(1)
C(15)	41(2)	46(2)	42(2)	-7(2)	1(2)	-1(2)
C(11)	46(2)	37(2)	40(2)	-3(2)	9(2)	-3(2)
C(8)	40(2)	35(2)	52(2)	-1(2)	-3(2)	0(2)
C(6)	40(2)	32(2)	40(2)	-3(1)	5(1)	2(1)
C(5)	50(2)	43(2)	50(2)	3(2)	-1(2)	-6(2)
C(14)	45(2)	38(2)	49(2)	-4(2)	7(2)	-2(2)
C(13)	45(2)	46(2)	48(2)	1(2)	2(2)	-2(2)
C(20)	44(2)	46(2)	50(2)	1(2)	5(2)	-3(2)
C(1)	53(2)	36(2)	45(2)	1(2)	4(2)	-3(2)
C(12)	54(2)	42(2)	68(2)	-6(2)	4(2)	1(2)
C(16)	53(2)	56(2)	54(2)	0(2)	4(2)	-2(2)
C(3)	39(2)	51(2)	69(2)	-15(2)	1(2)	-7(2)
C(4)	53(2)	53(2)	55(2)	-3(2)	-9(2)	3(2)
C(2)	60(2)	44(2)	63(2)	0(2)	10(2)	-14(2)
C(19)	60(2)	41(2)	69(2)	-1(2)	6(2)	-9(2)
C(17)	57(2)	65(2)	59(2)	-4(2)	-8(2)	-4(2)
C(18)	47(2)	56(2)	80(3)	-7(2)	-6(2)	-11(2)
C(9)	59(2)	64(2)	85(3)	-15(2)	21(2)	3(2)
C(10)	72(3)	66(3)	142(4)	-24(3)	12(3)	20(2)
Cl(1)	56(1)	89(1)	140(1)	-19(1)	-11(1)	-23(1)
Cl(2)	90(1)	46(1)	68(1)	18(1)	-9(1)	-7(1)
N(1)	38(1)	35(2)	56(2)	-6(1)	-6(1)	4(1)
N(2)	44(2)	44(2)	49(2)	-5(1)	-4(1)	-5(1)
N(3)	44(2)	44(2)	44(2)	-2(1)	-2(1)	-6(1)
O(1)	60(1)	39(1)	76(2)	-1(1)	23(1)	9(1)
O(2)	73(2)	53(2)	71(2)	12(1)	28(1)	4(1)
O(3)	57(2)	68(2)	72(2)	18(1)	-1(1)	-15(1)
O(4)	63(2)	71(2)	58(1)	16(1)	1(1)	-9(1)

Table 1-5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 1_a.

	x	y	z	U(eq)
H(7)	5350	3767	3154	47
H(5)	3491	4406	107	57
H(12A)	5906	6019	4608	82
H(12B)	4714	5942	3679	82
H(12C)	5803	6127	2869	82
H(16)	9621	4708	8671	65
H(4)	1690	3993	-671	65
H(2)	2101	2440	2326	66
H(19)	10214	6387	5047	68
H(17)	11268	5393	8861	74
H(18)	11563	6215	7062	74
H(9A)	7653	3399	-49	82
H(9B)	6589	3109	-1061	82
H(10A)	7841	2324	1182	140
H(10B)	7893	2204	-538	140
H(10C)	6767	2035	204	140
H(1)	4318	4969	2254	52

Table 1-6. Torsion angles [°] for 1_a.

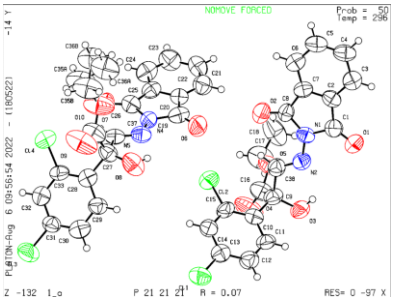
N(1)-C(7)-C(8)-O(2)	-22.6(4)
C(6)-C(7)-C(8)-O(2)	99.8(3)
N(1)-C(7)-C(8)-O(1)	158.8(2)
C(6)-C(7)-C(8)-O(1)	-78.8(3)
N(1)-C(7)-C(6)-C(5)	53.3(3)
C(8)-C(7)-C(6)-C(5)	-69.7(3)
N(1)-C(7)-C(6)-C(1)	-126.8(3)
C(8)-C(7)-C(6)-C(1)	110.2(3)
C(1)-C(6)-C(5)-C(4)	2.5(4)
C(7)-C(6)-C(5)-C(4)	-177.6(3)
C(16)-C(15)-C(14)-C(19)	0.3(5)
C(20)-C(15)-C(14)-C(19)	-175.7(3)
C(16)-C(15)-C(14)-C(13)	175.5(3)
C(20)-C(15)-C(14)-C(13)	-0.5(3)
C(19)-C(14)-C(13)-O(4)	-1.8(6)
C(15)-C(14)-C(13)-O(4)	-176.4(3)
C(19)-C(14)-C(13)-N(3)	177.6(3)
C(15)-C(14)-C(13)-N(3)	3.0(3)
C(16)-C(15)-C(20)-O(3)	1.0(6)
C(14)-C(15)-C(20)-O(3)	176.6(3)
C(16)-C(15)-C(20)-N(3)	-177.7(3)
C(14)-C(15)-C(20)-N(3)	-2.1(3)
C(5)-C(6)-C(1)-C(2)	-2.2(4)
C(7)-C(6)-C(1)-C(2)	178.0(3)
C(5)-C(6)-C(1)-Cl(2)	177.4(2)
C(7)-C(6)-C(1)-Cl(2)	-2.5(4)
C(14)-C(15)-C(16)-C(17)	0.2(5)
C(20)-C(15)-C(16)-C(17)	175.3(3)
C(2)-C(3)-C(4)-C(5)	-0.3(5)
Cl(1)-C(3)-C(4)-C(5)	179.6(2)
C(6)-C(5)-C(4)-C(3)	-1.3(5)
C(4)-C(3)-C(2)-C(1)	0.6(5)
Cl(1)-C(3)-C(2)-C(1)	-179.3(2)
C(6)-C(1)-C(2)-C(3)	0.7(5)
Cl(2)-C(1)-C(2)-C(3)	-179.0(2)
C(15)-C(14)-C(19)-C(18)	-0.5(5)

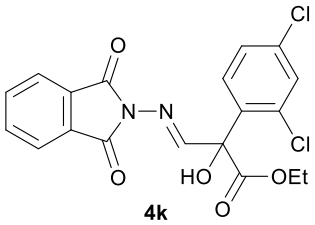
C(13)-C(14)-C(19)-C(18)	-174.5(3)
C(15)-C(16)-C(17)-C(18)	-0.6(5)
C(14)-C(19)-C(18)-C(17)	0.1(5)
C(16)-C(17)-C(18)-C(19)	0.5(5)
N(2)-C(11)-N(1)-C(7)	-4.0(4)
C(12)-C(11)-N(1)-C(7)	177.4(3)
C(6)-C(7)-N(1)-C(11)	161.3(2)
C(8)-C(7)-N(1)-C(11)	-76.6(3)
N(1)-C(11)-N(2)-N(3)	-178.0(2)
C(12)-C(11)-N(2)-N(3)	0.3(4)
O(4)-C(13)-N(3)-C(20)	175.0(3)
C(14)-C(13)-N(3)-C(20)	-4.4(3)
O(4)-C(13)-N(3)-N(2)	19.1(5)
C(14)-C(13)-N(3)-N(2)	-160.3(2)
O(3)-C(20)-N(3)-C(13)	-174.6(3)
C(15)-C(20)-N(3)-C(13)	4.1(3)
O(3)-C(20)-N(3)-N(2)	-16.8(4)
C(15)-C(20)-N(3)-N(2)	161.9(2)
C(11)-N(2)-N(3)-C(13)	-69.0(4)
C(11)-N(2)-N(3)-C(20)	136.7(3)
O(2)-C(8)-O(1)-C(9)	-2.4(4)
C(7)-C(8)-O(1)-C(9)	176.3(2)
C(10)-C(9)-O(1)-C(8)	177.0(3)

Symmetry transformations used to generate equivalent atoms:

Table 1-7. Hydrogen bonds for 1_a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)





4k

Bond precision: C-C = 0.0119 A Wavelength=0.71073

Cell: a=13.1812(18) b=14.144(2) c=20.391(3)
alpha=90 beta=90 gamma=90
Temperature: 296 K

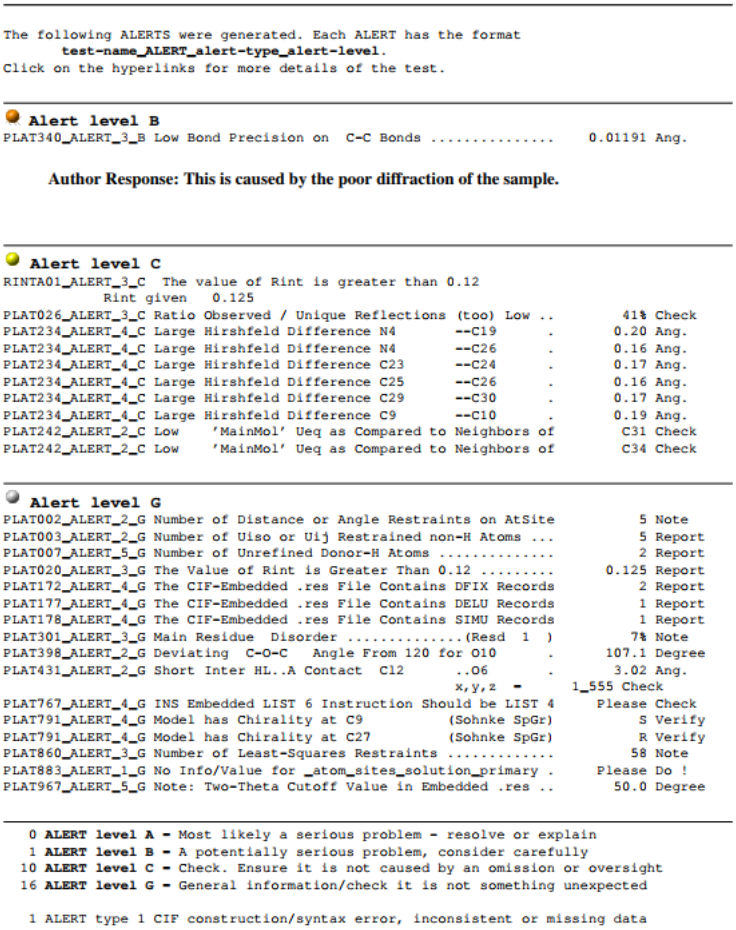
	Calculated	Reported
Volume	3801.6(9)	3801.6(9)
Space group	P 21 21 21	P 21 21 21
Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	C19 H14 Cl2 N2 O5	?
Sum formula	C19 H14 Cl2 N2 O5	C19 H14 Cl2 N2 O5
Mr	421.22	421.22
Dx,g cm-3	1.472	1.472
Z	8	8
Mu (mm-1)	0.376	0.376
F000	1728.0	1728.0
F000'	1731.17	
h,k,lmax	15,16,24	15,16,24
Nref	6682[3738]	6675
Tmin,Tmax	0.928,0.928	0.864,0.864
Tmin'	0.928	

Correction method= # Reported T Limits: Tmin=0.864 Tmax=0.864
AbsCorr = MULTI-SCAN

Data completeness= 1.79/1.00 Theta(max)= 24.998

R(reflections)= 0.0690(2768) wR2(reflections)= 0.1926(6675)

S = 0.970 Npar= 477



The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT340_ALERT_3_B Low Bond Precision on C-C Bonds ..... 0.01191 Ang.

Author Response: This is caused by the poor diffraction of the sample.

Alert level C

RINTA01_ALERT_3_C The value of Rint is greater than 0.12
Rint given 0.125
PLAT026_ALERT_3_C Ratio Observed / Unique Reflections (too) Low .. 41% Check
PLAT234_ALERT_4_C Large Hirshfeld Difference N4 --C19 . 0.20 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference N4 --C26 . 0.16 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C23 --C24 . 0.17 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C25 --C26 . 0.16 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C29 --C30 . 0.17 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C9 --C10 . 0.19 Ang.
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C31 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C34 Check

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 5 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 5 Report
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 2 Report
PLAT020_ALERT_3_G The Value of Rint is Greater Than 0.12 ..... 0.125 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 2 Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records 1 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 1 Report
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 7% Note
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O10 . 107.1 Degree
PLAT431_ALERT_2_G Short Inter HL..A Contact C12 ..O6 . 3.02 Ang.
x,y,z = 1_555 Check
PLAT767_ALERT_4_G INS Embedded LIST 6 Instruction Should be LIST 4 Please Check
PLAT791_ALERT_4_G Model has Chirality at C9 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C27 (Sohnke SpGr) R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 58 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res .. 50.0 Degree

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
10 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
16 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data

Figure S2 The single crystal analysis for **4k** (**CCDC number: 2205670**)

Table 2-1. Crystal data and structure refinement for 1_a.

Identification code	1_a	
Empirical formula	C19 H14 Cl2 N2 O5	
Formula weight	421.22	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 13.1812(18) Å	α= 90°.
	b = 14.144(2) Å	β= 90°.
	c = 20.391(3) Å	γ = 90°.
Volume	3801.6(9) $Å^3$	
Z	8	
Density (calculated)	1.472 Mg/m^3	
Absorption coefficient	0.376 mm^{-1}	
F(000)	1728	
Crystal size	0.200 x 0.200 x 0.200 mm^3	

Theta range for data collection	2.112 to 24.998°.
Index ranges	-13<=h<=15, -16<=k<=16, -20<=l<=24
Reflections collected	32402
Independent reflections	6675 [R(int) = 0.1249]
Completeness to theta = 24.998°	99.9 %
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6675 / 58 / 477
Goodness-of-fit on F^2	0.970
Final R indices [I>2sigma(I)]	R1 = 0.0690, wR2 = 0.1484
R indices (all data)	R1 = 0.2151, wR2 = 0.1926
Absolute structure parameter	0.05(6)
Extinction coefficient	n/a
Largest diff. peak and hole	0.223 and -0.245 e.$Å^{-3}$

Table 2-2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 1_a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2165(9)	6066(8)	9109(5)	72(3)
C(2)	2861(5)	6675(4)	9476(4)	66(3)
C(3)	2745(5)	7142(5)	10071(4)	75(3)
C(4)	3533(7)	7689(5)	10319(3)	83(3)
C(5)	4435(6)	7770(5)	9971(5)	89(4)
C(6)	4551(5)	7303(6)	9376(4)	82(3)
C(7)	3764(6)	6755(5)	9129(3)	67(3)
C(8)	3697(8)	6181(7)	8533(5)	65(3)
C(9)	2248(8)	4141(8)	7191(5)	74(3)
C(10)	2038(6)	4569(4)	6517(3)	64(3)
C(15)	2823(4)	4872(5)	6113(4)	67(3)
C(14)	2605(5)	5293(5)	5513(3)	72(3)
C(13)	1603(6)	5410(4)	5318(3)	74(3)
C(12)	818(4)	5107(5)	5722(4)	82(3)
C(11)	1036(5)	4687(5)	6322(3)	76(3)
C(16)	2917(10)	3240(8)	7146(7)	87(4)
C(17)	4112(11)	2320(10)	7721(7)	121(5)
C(18)	4513(12)	2204(11)	8373(6)	139(6)
C(19)	6820(9)	3826(8)	6867(6)	79(3)
C(20)	7479(5)	3189(5)	7211(4)	70(3)
C(21)	7363(6)	2762(6)	7821(4)	86(3)
C(22)	8130(8)	2193(5)	8071(3)	87(3)
C(23)	9014(6)	2050(5)	7712(5)	97(4)
C(24)	9130(5)	2476(6)	7103(4)	86(3)
C(25)	8363(6)	3046(5)	6852(3)	68(3)
C(26)	8308(9)	3585(7)	6243(6)	70(3)
C(27)	6955(9)	5683(8)	4932(5)	73(3)
C(28)	6862(6)	5286(4)	4231(3)	65(3)
C(33)	7713(4)	5129(5)	3843(4)	68(3)
C(32)	7603(5)	4780(5)	3210(3)	76(3)
C(31)	6641(6)	4587(4)	2965(3)	69(3)
C(30)	5790(5)	4744(5)	3352(4)	81(3)
C(29)	5901(5)	5094(5)	3985(3)	73(3)

C(34)	7577(11)	6616(9)	4922(7)	96(4)
C(35A)	9120(20)	7350(20)	5440(20)	123(6)
C(36A)	8750(40)	7910(20)	5999(16)	126(8)
C(36B)	9440(20)	7510(20)	5958(14)	121(7)
C(35B)	8690(20)	7601(15)	5425(16)	125(5)
Cl(1)	1330(2)	5905(2)	4574(1)	96(1)
Cl(2)	4075(2)	4699(2)	6305(1)	90(1)
Cl(3)	6524(3)	4203(3)	2164(1)	108(1)
Cl(4)	8912(2)	5342(3)	4106(2)	103(1)
N(1)	2723(7)	5794(6)	8549(4)	63(2)
N(2)	2276(6)	5125(6)	8135(5)	69(2)
C(38)	2767(8)	4890(7)	7631(5)	71(3)
N(4)	7343(6)	4033(6)	6266(4)	65(2)
N(5)	6945(6)	4685(6)	5856(5)	72(2)
C(37)	7439(8)	4951(7)	5355(5)	69(3)
O(1)	1329(6)	5800(6)	9238(4)	90(2)
O(2)	4308(6)	6061(6)	8116(4)	95(3)
O(3)	1340(5)	3826(5)	7470(3)	83(2)
O(4)	2886(8)	2718(6)	6709(4)	126(4)
O(5)	3462(6)	3147(5)	7686(4)	90(2)
O(6)	5995(6)	4105(7)	6996(4)	106(3)
O(7)	8913(5)	3650(5)	5813(4)	88(2)
O(8)	5985(7)	5917(6)	5163(4)	105(3)
O(9)	7467(9)	7202(6)	4520(5)	140(4)
O(10)	8214(7)	6646(5)	5426(5)	127(3)

Table 2-3. Bond lengths [Å] and angles [°] for 1_a.

C(1)-O(1)	1.193(12)
C(1)-N(1)	1.412(12)
C(1)-C(2)	1.464(12)
C(2)-C(3)	1.3900
C(2)-C(7)	1.3900
C(3)-C(4)	1.3900
C(3)-H(3)	0.9300
C(4)-C(5)	1.3900
C(4)-H(4)	0.9300
C(5)-C(6)	1.3900
C(5)-H(5)	0.9300
C(6)-C(7)	1.3900
C(6)-H(6)	0.9300
C(7)-C(8)	1.465(12)
C(8)-O(2)	1.182(10)
C(8)-N(1)	1.396(13)
C(9)-O(3)	1.397(11)
C(9)-C(10)	1.527(11)
C(9)-C(38)	1.547(13)
C(9)-C(16)	1.552(15)
C(10)-C(15)	1.3900
C(10)-C(11)	1.3900
C(15)-C(14)	1.3900
C(15)-Cl(2)	1.714(6)
C(14)-C(13)	1.3900
C(14)-H(14)	0.9300
C(13)-C(12)	1.3900
C(13)-Cl(1)	1.708(6)
C(12)-C(11)	1.3900
C(12)-H(12)	0.9300
C(11)-H(11)	0.9300
C(16)-O(4)	1.156(12)
C(16)-O(5)	1.322(13)
C(17)-C(18)	1.440(15)
C(17)-O(5)	1.451(13)
C(17)-H(17A)	0.9700

C(17)-H(17B)	0.9700
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(19)-O(6)	1.186(12)
C(19)-C(20)	1.436(12)
C(19)-N(4)	1.436(13)
C(20)-C(21)	1.3900
C(20)-C(25)	1.3900
C(21)-C(22)	1.3900
C(21)-H(21)	0.9300
C(22)-C(23)	1.3900
C(22)-H(22)	0.9300
C(23)-C(24)	1.3900
C(23)-H(23)	0.9300
C(24)-C(25)	1.3900
C(24)-H(24)	0.9300
C(25)-C(26)	1.459(12)
C(26)-O(7)	1.190(12)
C(26)-N(4)	1.422(13)
C(27)-O(8)	1.402(12)
C(27)-C(37)	1.490(13)
C(27)-C(28)	1.542(11)
C(27)-C(34)	1.553(16)
C(28)-C(33)	1.3900
C(28)-C(29)	1.3900
C(33)-C(32)	1.3900
C(33)-Cl(4)	1.695(6)
C(32)-C(31)	1.3900
C(32)-H(32)	0.9300
C(31)-C(30)	1.3900
C(31)-Cl(3)	1.727(6)
C(30)-C(29)	1.3900
C(30)-H(30)	0.9300
C(29)-H(29)	0.9300
C(34)-O(9)	1.174(13)
C(34)-O(10)	1.328(14)
C(35A)-C(36A)	1.47(3)

C(35A)-O(10)	1.55(2)
C(35A)-H(35A)	0.9700
C(35A)-H(35B)	0.9700
C(36A)-H(36A)	0.9600
C(36A)-H(36B)	0.9600
C(36A)-H(36C)	0.9600
C(36B)-C(35B)	1.48(2)
C(36B)-H(36D)	0.9600
C(36B)-H(36E)	0.9600
C(36B)-H(36F)	0.9600
C(35B)-O(10)	1.490(19)
C(35B)-H(35C)	0.9700
C(35B)-H(35D)	0.9700
N(1)-N(2)	1.399(10)
N(2)-C(38)	1.260(11)
C(38)-H(38)	0.9300
N(4)-N(5)	1.350(10)
N(5)-C(37)	1.269(12)
C(37)-H(37)	0.9300
O(3)-H(3A)	0.8199
O(8)-H(8)	0.8201

O(1)-C(1)-N(1)	125.0(11)
O(1)-C(1)-C(2)	130.7(11)
N(1)-C(1)-C(2)	104.3(9)
C(3)-C(2)-C(7)	120.0
C(3)-C(2)-C(1)	131.1(8)
C(7)-C(2)-C(1)	108.9(8)
C(2)-C(3)-C(4)	120.0
C(2)-C(3)-H(3)	120.0
C(4)-C(3)-H(3)	120.0
C(3)-C(4)-C(5)	120.0
C(3)-C(4)-H(4)	120.0
C(5)-C(4)-H(4)	120.0
C(6)-C(5)-C(4)	120.0
C(6)-C(5)-H(5)	120.0
C(4)-C(5)-H(5)	120.0
C(5)-C(6)-C(7)	120.0

C(5)-C(6)-H(6)	120.0
C(7)-C(6)-H(6)	120.0
C(6)-C(7)-C(2)	120.0
C(6)-C(7)-C(8)	130.9(7)
C(2)-C(7)-C(8)	109.0(7)
O(2)-C(8)-N(1)	125.9(10)
O(2)-C(8)-C(7)	129.4(10)
N(1)-C(8)-C(7)	104.6(9)
O(3)-C(9)-C(10)	109.7(8)
O(3)-C(9)-C(38)	111.2(9)
C(10)-C(9)-C(38)	109.3(8)
O(3)-C(9)-C(16)	104.4(9)
C(10)-C(9)-C(16)	111.9(9)
C(38)-C(9)-C(16)	110.2(9)
C(15)-C(10)-C(11)	120.0
C(15)-C(10)-C(9)	121.4(6)
C(11)-C(10)-C(9)	118.5(6)
C(14)-C(15)-C(10)	120.0
C(14)-C(15)-Cl(2)	117.5(5)
C(10)-C(15)-Cl(2)	122.5(5)
C(15)-C(14)-C(13)	120.0
C(15)-C(14)-H(14)	120.0
C(13)-C(14)-H(14)	120.0
C(14)-C(13)-C(12)	120.0
C(14)-C(13)-Cl(1)	120.3(5)
C(12)-C(13)-Cl(1)	119.7(5)
C(11)-C(12)-C(13)	120.0
C(11)-C(12)-H(12)	120.0
C(13)-C(12)-H(12)	120.0
C(12)-C(11)-C(10)	120.0
C(12)-C(11)-H(11)	120.0
C(10)-C(11)-H(11)	120.0
O(4)-C(16)-O(5)	126.6(12)
O(4)-C(16)-C(9)	123.4(12)
O(5)-C(16)-C(9)	109.9(10)
C(18)-C(17)-O(5)	110.7(11)
C(18)-C(17)-H(17A)	109.5
O(5)-C(17)-H(17A)	109.5

C(18)-C(17)-H(17B)	109.5
O(5)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	108.1
C(17)-C(18)-H(18A)	109.5
C(17)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(17)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
O(6)-C(19)-C(20)	130.9(12)
O(6)-C(19)-N(4)	124.2(11)
C(20)-C(19)-N(4)	104.8(10)
C(21)-C(20)-C(25)	120.0
C(21)-C(20)-C(19)	130.0(8)
C(25)-C(20)-C(19)	109.9(8)
C(20)-C(21)-C(22)	120.0
C(20)-C(21)-H(21)	120.0
C(22)-C(21)-H(21)	120.0
C(23)-C(22)-C(21)	120.0
C(23)-C(22)-H(22)	120.0
C(21)-C(22)-H(22)	120.0
C(24)-C(23)-C(22)	120.0
C(24)-C(23)-H(23)	120.0
C(22)-C(23)-H(23)	120.0
C(23)-C(24)-C(25)	120.0
C(23)-C(24)-H(24)	120.0
C(25)-C(24)-H(24)	120.0
C(24)-C(25)-C(20)	120.0
C(24)-C(25)-C(26)	130.7(8)
C(20)-C(25)-C(26)	109.3(8)
O(7)-C(26)-N(4)	126.0(10)
O(7)-C(26)-C(25)	129.5(11)
N(4)-C(26)-C(25)	104.5(9)
O(8)-C(27)-C(37)	111.1(9)
O(8)-C(27)-C(28)	108.9(8)
C(37)-C(27)-C(28)	108.5(8)
O(8)-C(27)-C(34)	106.6(9)
C(37)-C(27)-C(34)	111.9(10)

C(28)-C(27)-C(34)	109.8(9)
C(33)-C(28)-C(29)	120.0
C(33)-C(28)-C(27)	121.4(7)
C(29)-C(28)-C(27)	118.6(7)
C(28)-C(33)-C(32)	120.0
C(28)-C(33)-Cl(4)	122.9(5)
C(32)-C(33)-Cl(4)	117.1(5)
C(31)-C(32)-C(33)	120.0
C(31)-C(32)-H(32)	120.0
C(33)-C(32)-H(32)	120.0
C(32)-C(31)-C(30)	120.0
C(32)-C(31)-Cl(3)	118.9(5)
C(30)-C(31)-Cl(3)	121.0(5)
C(31)-C(30)-C(29)	120.0
C(31)-C(30)-H(30)	120.0
C(29)-C(30)-H(30)	120.0
C(30)-C(29)-C(28)	120.0
C(30)-C(29)-H(29)	120.0
C(28)-C(29)-H(29)	120.0
O(9)-C(34)-O(10)	126.5(13)
O(9)-C(34)-C(27)	123.0(13)
O(10)-C(34)-C(27)	110.5(11)
C(36A)-C(35A)-O(10)	96(2)
C(36A)-C(35A)-H(35A)	112.5
O(10)-C(35A)-H(35A)	112.5
C(36A)-C(35A)-H(35B)	112.5
O(10)-C(35A)-H(35B)	112.5
H(35A)-C(35A)-H(35B)	110.0
C(35A)-C(36A)-H(36A)	109.5
C(35A)-C(36A)-H(36B)	109.5
H(36A)-C(36A)-H(36B)	109.5
C(35A)-C(36A)-H(36C)	109.5
H(36A)-C(36A)-H(36C)	109.5
H(36B)-C(36A)-H(36C)	109.5
C(35B)-C(36B)-H(36D)	109.5
C(35B)-C(36B)-H(36E)	109.5
H(36D)-C(36B)-H(36E)	109.5
C(35B)-C(36B)-H(36F)	109.5

H(36D)-C(36B)-H(36F)	109.5
H(36E)-C(36B)-H(36F)	109.5
C(36B)-C(35B)-O(10)	101.8(17)
C(36B)-C(35B)-H(35C)	111.4
O(10)-C(35B)-H(35C)	111.4
C(36B)-C(35B)-H(35D)	111.4
O(10)-C(35B)-H(35D)	111.4
H(35C)-C(35B)-H(35D)	109.3
C(8)-N(1)-N(2)	129.7(9)
C(8)-N(1)-C(1)	113.1(9)
N(2)-N(1)-C(1)	116.9(9)
C(38)-N(2)-N(1)	117.0(8)
N(2)-C(38)-C(9)	115.2(10)
N(2)-C(38)-H(38)	122.4
C(9)-C(38)-H(38)	122.4
N(5)-N(4)-C(26)	129.2(9)
N(5)-N(4)-C(19)	118.8(9)
C(26)-N(4)-C(19)	111.4(9)
C(37)-N(5)-N(4)	120.1(9)
N(5)-C(37)-C(27)	116.9(10)
N(5)-C(37)-H(37)	121.5
C(27)-C(37)-H(37)	121.5
C(9)-O(3)-H(3A)	108.2
C(16)-O(5)-C(17)	116.3(9)
C(27)-O(8)-H(8)	109.3
C(34)-O(10)-C(35B)	107.2(12)
C(34)-O(10)-C(35A)	121.4(19)

Symmetry transformations used to generate equivalent atoms:

Table 2-4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 1_a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	76(8)	68(7)	71(8)	7(6)	-3(7)	-6(7)
C(2)	78(8)	59(7)	60(7)	3(6)	-11(6)	12(6)
C(3)	74(8)	66(7)	86(9)	1(7)	-2(7)	12(7)
C(4)	91(9)	67(7)	91(9)	-17(7)	-26(8)	6(7)
C(5)	91(10)	84(9)	90(10)	-15(8)	-23(8)	-8(8)
C(6)	66(8)	86(8)	95(10)	1(8)	-6(7)	-2(7)
C(7)	62(7)	62(7)	77(8)	1(7)	-13(7)	2(6)
C(8)	55(8)	68(7)	71(8)	6(6)	7(6)	5(6)
C(9)	66(7)	68(7)	87(8)	-14(7)	-6(6)	-10(7)
C(10)	77(8)	54(6)	60(7)	0(5)	-13(6)	0(6)
C(15)	40(6)	78(8)	84(8)	-15(7)	-4(6)	15(6)
C(14)	76(8)	67(7)	72(8)	-16(6)	3(6)	-7(6)
C(13)	76(7)	60(7)	86(8)	-8(6)	-3(7)	-1(6)
C(12)	72(7)	90(9)	83(8)	8(7)	-9(7)	2(7)
C(11)	61(7)	85(8)	82(8)	-21(7)	-10(6)	-11(7)
C(16)	120(10)	59(8)	81(9)	-12(7)	-18(9)	5(8)
C(17)	134(12)	108(11)	122(13)	-24(9)	-21(9)	55(10)
C(18)	178(14)	152(14)	86(10)	11(10)	2(10)	85(12)
C(19)	64(8)	77(8)	96(10)	-8(7)	-17(8)	-1(7)
C(20)	66(8)	78(8)	65(7)	-4(7)	-7(6)	-4(6)
C(21)	82(9)	92(9)	85(10)	-7(8)	3(7)	11(8)
C(22)	116(10)	74(8)	71(8)	-2(7)	7(8)	-3(8)
C(23)	106(11)	92(9)	94(10)	14(8)	-14(8)	21(9)
C(24)	77(8)	64(8)	117(11)	-14(8)	1(8)	13(7)
C(25)	64(7)	51(7)	88(8)	-13(6)	-4(7)	-5(6)
C(26)	87(10)	54(7)	70(8)	2(6)	-6(7)	-3(7)
C(27)	83(8)	73(8)	64(7)	-3(6)	-5(6)	14(7)
C(28)	71(7)	44(6)	82(8)	8(6)	-5(7)	2(6)
C(33)	66(7)	63(7)	76(7)	-3(6)	-16(6)	-13(6)
C(32)	78(8)	66(7)	85(8)	1(6)	5(7)	-1(7)
C(31)	78(7)	67(7)	63(6)	-7(6)	-6(6)	-7(6)
C(30)	83(8)	57(7)	102(9)	3(7)	-13(7)	-5(7)
C(29)	69(7)	76(8)	74(7)	-9(6)	-11(6)	8(6)

C(34)	133(11)	67(9)	86(10)	-11(8)	-15(9)	2(9)
C(35A)	173(12)	65(10)	131(10)	4(10)	-56(11)	-22(9)
C(36A)	172(18)	79(14)	128(15)	16(12)	-40(16)	4(14)
C(36B)	147(17)	75(14)	141(14)	17(13)	-40(13)	-23(13)
C(35B)	180(12)	57(9)	137(10)	5(9)	-54(10)	-13(9)
Cl(1)	100(2)	107(2)	82(2)	8(2)	-26(2)	4(2)
Cl(2)	63(2)	111(2)	96(2)	-5(2)	-5(2)	8(2)
Cl(3)	126(3)	118(2)	80(2)	-21(2)	-7(2)	-29(2)
Cl(4)	71(2)	130(3)	107(2)	-4(2)	-9(2)	-15(2)
N(1)	71(6)	60(6)	60(5)	-2(5)	-12(5)	7(5)
N(2)	65(5)	65(6)	75(6)	3(5)	-4(5)	0(5)
C(38)	65(7)	73(8)	74(7)	1(7)	1(6)	5(6)
N(4)	56(6)	73(6)	65(6)	-1(5)	0(5)	4(5)
N(5)	75(6)	65(6)	76(6)	-1(5)	-17(5)	6(5)
C(37)	68(7)	65(7)	73(7)	-2(7)	-21(6)	4(6)
O(1)	64(5)	112(6)	95(5)	-8(5)	4(4)	-11(5)
O(2)	76(5)	111(7)	97(6)	-7(5)	16(5)	-12(5)
O(3)	75(5)	85(5)	90(5)	4(4)	-3(4)	-25(4)
O(4)	207(10)	82(6)	90(6)	-23(5)	-40(7)	38(7)
O(5)	118(6)	82(6)	70(5)	-4(4)	-19(5)	24(5)
O(6)	69(5)	160(8)	90(6)	4(6)	5(5)	26(6)
O(7)	67(5)	101(6)	95(6)	15(5)	17(5)	9(5)
O(8)	123(7)	109(6)	85(5)	-8(5)	-3(5)	49(6)
O(9)	247(12)	60(5)	114(7)	2(5)	-55(8)	-1(7)
O(10)	178(8)	69(5)	134(7)	7(5)	-68(7)	-26(5)

Table 2-5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 1_a.

	x	y	z	U(eq)
H(3)	2141	7087	10304	90
H(4)	3455	8002	10717	100
H(5)	4962	8137	10137	106
H(6)	5155	7357	9143	98
H(14)	3130	5495	5243	86
H(12)	148	5186	5591	98
H(11)	511	4484	6592	91
H(17A)	3727	1762	7600	146
H(17B)	4667	2387	7412	146
H(18A)	5030	1726	8369	208
H(18B)	3978	2019	8665	208
H(18C)	4800	2792	8519	208
H(21)	6772	2858	8061	104
H(22)	8053	1908	8479	104
H(23)	9527	1669	7880	117
H(24)	9721	2380	6862	103
H(32)	8172	4675	2951	91
H(30)	5146	4616	3188	97
H(29)	5331	5199	4244	87
H(35A)	9756	7039	5532	148
H(35B)	9167	7717	5040	148
H(36A)	9224	8411	6091	190
H(36B)	8100	8184	5893	190
H(36C)	8681	7515	6378	190
H(36D)	9808	8094	6005	181
H(36E)	9102	7365	6361	181
H(36F)	9912	7012	5854	181
H(35C)	9016	7736	5008	150
H(35D)	8198	8091	5520	150
H(38)	3397	5149	7530	85
H(37)	8073	4701	5255	83
H(3A)	1250	4115	7814	125

H(8)	5888	5656	5516	158

Table 2-6. Torsion angles [°] for 1_a.

O(1)-C(1)-C(2)-C(3)	-2.0(16)
N(1)-C(1)-C(2)-C(3)	-179.0(5)
O(1)-C(1)-C(2)-C(7)	178.2(10)
N(1)-C(1)-C(2)-C(7)	1.2(8)
C(7)-C(2)-C(3)-C(4)	0.0
C(1)-C(2)-C(3)-C(4)	-179.8(8)
C(2)-C(3)-C(4)-C(5)	0.0
C(3)-C(4)-C(5)-C(6)	0.0
C(4)-C(5)-C(6)-C(7)	0.0
C(5)-C(6)-C(7)-C(2)	0.0
C(5)-C(6)-C(7)-C(8)	-178.3(8)
C(3)-C(2)-C(7)-C(6)	0.0
C(1)-C(2)-C(7)-C(6)	179.8(6)
C(3)-C(2)-C(7)-C(8)	178.6(6)
C(1)-C(2)-C(7)-C(8)	-1.6(7)
C(6)-C(7)-C(8)-O(2)	-1.0(15)
C(2)-C(7)-C(8)-O(2)	-179.4(10)
C(6)-C(7)-C(8)-N(1)	179.7(5)
C(2)-C(7)-C(8)-N(1)	1.3(8)
O(3)-C(9)-C(10)-C(15)	-175.1(6)
C(38)-C(9)-C(10)-C(15)	62.7(9)
C(16)-C(9)-C(10)-C(15)	-59.7(10)
O(3)-C(9)-C(10)-C(11)	7.5(9)
C(38)-C(9)-C(10)-C(11)	-114.7(8)
C(16)-C(9)-C(10)-C(11)	122.9(8)
C(11)-C(10)-C(15)-C(14)	0.0
C(9)-C(10)-C(15)-C(14)	-177.4(7)
C(11)-C(10)-C(15)-Cl(2)	-176.8(5)
C(9)-C(10)-C(15)-Cl(2)	5.8(7)
C(10)-C(15)-C(14)-C(13)	0.0
Cl(2)-C(15)-C(14)-C(13)	177.0(5)
C(15)-C(14)-C(13)-C(12)	0.0
C(15)-C(14)-C(13)-Cl(1)	-178.6(5)
C(14)-C(13)-C(12)-C(11)	0.0
Cl(1)-C(13)-C(12)-C(11)	178.7(5)
C(13)-C(12)-C(11)-C(10)	0.0

C(15)-C(10)-C(11)-C(12)	0.0
C(9)-C(10)-C(11)-C(12)	177.5(7)
O(3)-C(9)-C(16)-O(4)	85.9(15)
C(10)-C(9)-C(16)-O(4)	-32.7(16)
C(38)-C(9)-C(16)-O(4)	-154.6(13)
O(3)-C(9)-C(16)-O(5)	-90.7(10)
C(10)-C(9)-C(16)-O(5)	150.7(9)
C(38)-C(9)-C(16)-O(5)	28.9(13)
O(6)-C(19)-C(20)-C(21)	-4.8(16)
N(4)-C(19)-C(20)-C(21)	179.5(5)
O(6)-C(19)-C(20)-C(25)	177.7(11)
N(4)-C(19)-C(20)-C(25)	2.0(8)
C(25)-C(20)-C(21)-C(22)	0.0
C(19)-C(20)-C(21)-C(22)	-177.3(8)
C(20)-C(21)-C(22)-C(23)	0.0
C(21)-C(22)-C(23)-C(24)	0.0
C(22)-C(23)-C(24)-C(25)	0.0
C(23)-C(24)-C(25)-C(20)	0.0
C(23)-C(24)-C(25)-C(26)	178.1(8)
C(21)-C(20)-C(25)-C(24)	0.0
C(19)-C(20)-C(25)-C(24)	177.8(7)
C(21)-C(20)-C(25)-C(26)	-178.5(7)
C(19)-C(20)-C(25)-C(26)	-0.7(7)
C(24)-C(25)-C(26)-O(7)	1.2(15)
C(20)-C(25)-C(26)-O(7)	179.5(10)
C(24)-C(25)-C(26)-N(4)	-179.2(5)
C(20)-C(25)-C(26)-N(4)	-0.9(8)
O(8)-C(27)-C(28)-C(33)	170.1(6)
C(37)-C(27)-C(28)-C(33)	-68.8(10)
C(34)-C(27)-C(28)-C(33)	53.8(10)
O(8)-C(27)-C(28)-C(29)	-9.2(10)
C(37)-C(27)-C(28)-C(29)	111.9(8)
C(34)-C(27)-C(28)-C(29)	-125.6(8)
C(29)-C(28)-C(33)-C(32)	0.0
C(27)-C(28)-C(33)-C(32)	-179.3(7)
C(29)-C(28)-C(33)-Cl(4)	-179.8(6)
C(27)-C(28)-C(33)-Cl(4)	0.9(7)
C(28)-C(33)-C(32)-C(31)	0.0

Cl(4)-C(33)-C(32)-C(31)	179.8(5)
C(33)-C(32)-C(31)-C(30)	0.0
C(33)-C(32)-C(31)-Cl(3)	177.2(5)
C(32)-C(31)-C(30)-C(29)	0.0
Cl(3)-C(31)-C(30)-C(29)	-177.2(5)
C(31)-C(30)-C(29)-C(28)	0.0
C(33)-C(28)-C(29)-C(30)	0.0
C(27)-C(28)-C(29)-C(30)	179.4(7)
O(8)-C(27)-C(34)-O(9)	-75.6(15)
C(37)-C(27)-C(34)-O(9)	162.8(13)
C(28)-C(27)-C(34)-O(9)	42.2(17)
O(8)-C(27)-C(34)-O(10)	103.5(11)
C(37)-C(27)-C(34)-O(10)	-18.1(14)
C(28)-C(27)-C(34)-O(10)	-138.7(10)
O(2)-C(8)-N(1)-N(2)	6.9(16)
C(7)-C(8)-N(1)-N(2)	-173.8(7)
O(2)-C(8)-N(1)-C(1)	-179.9(10)
C(7)-C(8)-N(1)-C(1)	-0.6(10)
O(1)-C(1)-N(1)-C(8)	-177.6(10)
C(2)-C(1)-N(1)-C(8)	-0.3(10)
O(1)-C(1)-N(1)-N(2)	-3.4(14)
C(2)-C(1)-N(1)-N(2)	173.8(7)
C(8)-N(1)-N(2)-C(38)	-7.8(13)
C(1)-N(1)-N(2)-C(38)	179.2(9)
N(1)-N(2)-C(38)-C(9)	179.4(8)
O(3)-C(9)-C(38)-N(2)	-3.6(13)
C(10)-C(9)-C(38)-N(2)	117.7(9)
C(16)-C(9)-C(38)-N(2)	-118.9(10)
O(7)-C(26)-N(4)-N(5)	-7.5(16)
C(25)-C(26)-N(4)-N(5)	173.0(8)
O(7)-C(26)-N(4)-C(19)	-178.2(10)
C(25)-C(26)-N(4)-C(19)	2.3(10)
O(6)-C(19)-N(4)-N(5)	9.5(15)
C(20)-C(19)-N(4)-N(5)	-174.5(7)
O(6)-C(19)-N(4)-C(26)	-178.7(11)
C(20)-C(19)-N(4)-C(26)	-2.7(10)
C(26)-N(4)-N(5)-C(37)	2.9(14)
C(19)-N(4)-N(5)-C(37)	173.0(9)

N(4)-N(5)-C(37)-C(27)	-179.2(8)
O(8)-C(27)-C(37)-N(5)	-2.8(13)
C(28)-C(27)-C(37)-N(5)	-122.6(10)
C(34)-C(27)-C(37)-N(5)	116.2(11)
O(4)-C(16)-O(5)-C(17)	2(2)
C(9)-C(16)-O(5)-C(17)	178.7(10)
C(18)-C(17)-O(5)-C(16)	-169.1(13)
O(9)-C(34)-O(10)-C(35B)	5(3)
C(27)-C(34)-O(10)-C(35B)	-173.6(18)
O(9)-C(34)-O(10)-C(35A)	-17(2)
C(27)-C(34)-O(10)-C(35A)	163.5(16)
C(36B)-C(35B)-O(10)-C(34)	-175(3)
C(36A)-C(35A)-O(10)-C(34)	114(3)

Symmetry transformations used to generate equivalent atoms:

Table 2-7. Hydrogen bonds for 1_a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)