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Supporting Information

Electrochemical synthesis of α-enaminones from acetophenones

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Experimental Section

General Information

^{1}H NMR and ^{13}C NMR were recorded on a Bruker-400MHz Spectrometer (^{1}H NMR: 400MHz, ^{13}C NMR: 100MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (*J*) were expressed in ppm and Hz, respectively. HRMS (ESI) were recorded on a Waters^{TM} Q-TOF Premier. GC-MS was Shimadzu QP-5050 GC-MS system. IR spectra were recorded on Thermo Scientific Nicolet iS10. Commercially available compounds were used without further purification. Solvents were purified according to the standard procedures unless otherwise noted.

Instruments

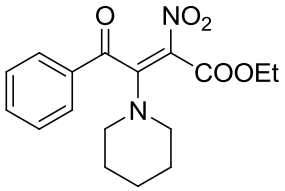
The instrument for electrolysis is dual display potentiostat (CJS-292) (made in China). The anode electrode and cathode electrode all are Pt (1.5×1.5 cm^2).

Experimental procedure

A mixture of acetophenone derivatives (0.5mmol), ethyl nitroacetate (1mmol) and piperidine (1mmol), KI (1mmol) and EtOH (8mL) was added to an undivided cell. The cell was equipped with platinum electrodes (1.5×1.5 cm^2) as both the anode and cathode. The reaction mixture was stirred and electrolyzed at a constant current of 40 mA under room temperature for corresponding time. When the reaction was finished, the solution was extracted with EtOAc (3×10mL). The combined organic layer was dried with Na_2SO_4, filtered. The solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel to afford the desired product.

Characterization data for the products

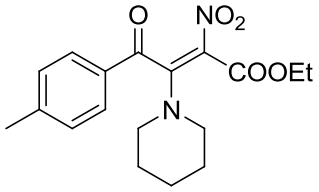
(*E*)-ethyl 2-nitro-4-oxo-4-phenyl-3-(piperidin-1-yl)but-2-enoate (4aa)



The title compound was prepared according to the general working procedure (3 h) and purified

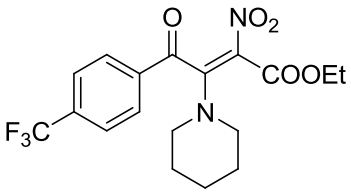
by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.95 (d, *J* = 6.8 Hz, 2H), 7.65-7.61 (m, 1H), 7.53-7.49 (m, 2H), 4.23-4.18 (m, 2H), 3.31-3.24 (m, 4H), 1.68-1.67 (m, 6H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.8, 162.1, 159.5, 135.0, 134.5, 129.1, 128.5, 120.5, 61.7, 52.7, 25.8, 22.7, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1712, 1677, 1556, 1507, 1471, 1448, 1406, 1299, 1241, 1209, 1163, 1121, 1077, 1026, 890, 867, 856, 828, 771, 704, 643, 614, 516. HRMS (ESI) m/z calcd for $C_{17}H_{20}N_2O_5$ $[M+Na]^+$ 355.1270, found 355.1273.

(*E*)-ethyl 2-nitro-4-oxo-3-(piperidin-1-yl)-4-*p*-tolylbut-2-enoate (4ab)



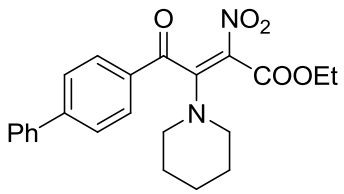
The title compound was prepared according to the general working procedure (4 h) and purified by column chromatography (petroleum ether / ethyl acetate = 4:1) to give the product as a yellow oil: 85% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.85 (d, *J* = 8 Hz, 2H), 7.30 (d, *J* = 8 Hz, 2H), 4.21-4.19 (m, 2H), 3.31-3.23 (m, 4H), 2.42 (s, 3H), 1.67 (m, 6H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.5, 162.2, 159.8, 145.8, 132.6, 129.8, 128.7, 120.5, 61.7, 52.7, 25.9, 22.8, 21.8, 13.9. IR (film, ν/cm^{-1}): 2987, 2920, 1715, 1679, 1603, 1550, 1487, 1442, 1304, 1243, 1210, 1177, 1138, 1088, 1016, 951, 825, 788, 724, 608, 487. HRMS (ESI) m/z calcd for $C_{18}H_{22}N_2O_5$ $[M+Na]^+$ 369.1426, found 369.1432.

(*E*)-ethyl 2-nitro-4-oxo-3-(piperidin-1-yl)-4-(4-(trifluoromethyl)phenyl)but-2-enoate (4ac)



The title compound was prepared according to the general working procedure (1.5 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 70% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 8.07 (d, *J* = 8.4 Hz, 2H), 7.78 (d, *J* = 8.0 Hz,2H), 4.25 (q, *J* = 7.2 Hz, 2H), 3.31-3.25 (m, 4H), 1.70 (m, 6H), 1.27 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 187.6, 162.0, 158.5, 137.6, 135.5 (q, *J* = 32.8 Hz), 128.6, 126.3 (q, *J* = 10.9 Hz), 123.3 (d, *J* = 271.2 Hz), 120.7, 62.0, 52.7, 25.9, 22.7, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1689, 1557, 1488, 1409, 1309, 1247, 1065, 1014, 823, 777, 699, 627, 516. HRMS (ESI) m/z calcd for $C_{18}H_{19}N_2O_5F_3$ $[M+Na]^+$ 423.1144, found 423.1145.

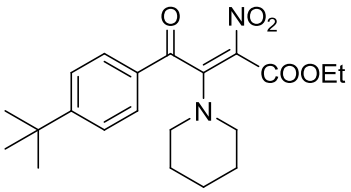
(*E*)-ethyl 4-(biphenyl-4-yl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ad)



The title compound was prepared according to the general working procedure (2.5 h) and purified

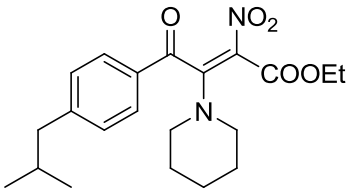
by column chromatography (petroleum ether / ethyl acetate = 4:1) to give the product as a yellow oil: 64% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 8.03 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.61 (d, *J* = 7.2 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.43-7.40 (m, 1H), 4.26-4.21 (m, 2H), 3.35-3.26 (m, 4H), 1.69 (m, 6H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.4, 162.2, 159.6, 147.2, 139.4, 133.7, 129.1, 129.0, 128.5, 127.8, 127.3, 120.6, 61.8, 52.7, 25.9, 22.8, 13.9. IR (film, ν/cm^{-1}): 2987, 2919, 1710, 1675, 1645, 1601, 1557, 1436, 1416, 1281, 1246, 1191, 1175, 1133, 1115, 1075, 1057, 1027, 1016, 889, 852, 825, 759, 688, 647, 631. HRMS (ESI) m/z calcd for $C_{23}H_{24}N_2O_5$ $[M+Na]^+$ 431.1583, found 431.1586.

(*E*)-ethyl 4-(4-*tert*-butylphenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ae)



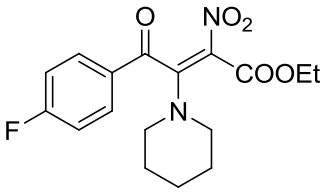
The title compound was prepared according to the general working procedure (4 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 74% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.88 (d, *J* = 8.8 Hz, 2H), 7.51 (d, *J* = 8.8 Hz, 2H), 4.23-4.20 (m, 2H), 3.33-3.24 (m, 4H), 1.68 (m, 6H), 1.33 (s, 9H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.4, 162.2, 159.9, 158.6, 132.4, 128.4, 126.1, 120.5, 61.7, 52.8, 35.3, 30.9, 25.9, 22.8, 13.9. IR (film, ν/cm^{-1}): 2970, 2900, 1705, 1676, 1634, 1602, 1544, 1488, 1393, 1307, 1247, 1210, 1158, 1139, 1076, 870, 854, 764, 747, 729, 658, 631, 545. HRMS (ESI) m/z calcd for $C_{21}H_{28}N_2O_5$ $[M+Na]^+$ 411.1896, found 411.1901.

(*E*)-ethyl 4-(4-isobutylphenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4af)



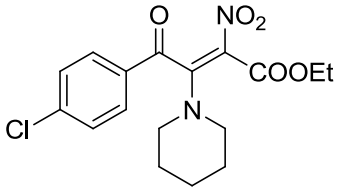
The title compound was prepared according to the general working procedure (4 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 86% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.86 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 4.23-4.16 (m, 2H), 3.32-3.23 (m, 4H), 2.54 (d, *J* = 7.2 Hz, 2H), 1.95-1.85 (m, 1H), 1.67 (m, 6H), 1.21 (t, *J* = 7.2 Hz, 3H), 0.91 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.6, 162.2, 159.9, 149.4, 132.8, 129.8, 128.6, 120.5, 61.6, 52.7, 45.5, 30.0, 25.9, 22.8, 22.3, 13.9. IR (film, ν/cm^{-1}): 2952, 2866, 1715, 1682, 1603, 1553, 1489, 1444, 1385, 1305, 1253, 1214, 1140, 1118, 1094, 856, 756, 666, 516. HRMS (ESI) m/z calcd for $C_{21}H_{28}N_2O_5$ $[M+Na]^+$ 411.1896, found 411.1898.

(*E*)-ethyl 4-(4-fluorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ag)



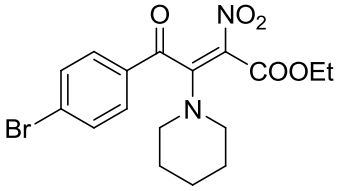
The title compound was prepared according to the general working procedure (2 h) and purified by column chromatography (petroleum ether / ethyl acetate = 3:1) to give the product as a yellow oil: 72% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 8.01-7.96 (m, 2H), 7.18 (t, *J* = 8.6 Hz, 2H), 4.22 (q, *J* = 7.2 Hz, 2H), 3.30-3.23 (m, 4H), 1.68 (m, 6H), 1.25 (t, *J* = 7.2 Hz, 3H); [13] C NMR (100 MHz, $CDCl_3$, ppm): δ = 187.3, 166.5 (d, *J* = 256.1 Hz), 162.1, 159.1, 131.6 (d, *J* = 2.9 Hz), 131.3 (d, *J* = 9.6 Hz), 120.7, 116.5 (d, *J* = 22.3 Hz), 61.8, 52.7, 25.9, 22.7, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1709, 1677, 1594, 1556, 1508, 1488, 1457, 1407, 1394, 1299, 1242, 1225, 1209, 1123, 1077, 921, 873, 855, 828, 733, 697, 633, 511. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_5F$ $[M+Na]^+$ 373.1176, found 373.1180.

(*E*)-ethyl 4-(4-chlorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ah)



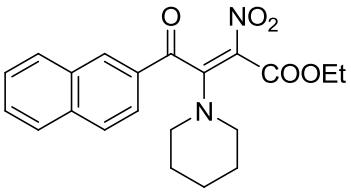
The title compound was prepared according to the general working procedure (4 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 75% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.90 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 3.30-3.23 (m, 4H), 1.69-1.68 (m, 6H), 1.26 (t, *J* = 7.2 Hz, 3H); [13] C NMR (100 MHz, $CDCl_3$, ppm): δ = 187.6, 162.1, 158.9, 141.0, 133.4, 129.8, 129.5, 120.6, 61.9, 52.7, 25.9, 22.7, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1716, 1684, 1587, 1557, 1488, 1403, 1394, 1301, 1242, 1209, 1170, 1140, 1076, 954, 867, 757, 692, 642, 625, 545. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_5Cl$ $[M+Na]^+$ 389.0880, found 389.0880.

(*E*)-ethyl 4-(4-bromophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ai)



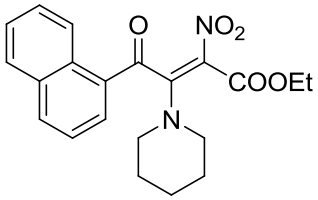
The title compound was prepared according to the general working procedure (3.5 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 69% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.79 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.0 Hz, 2H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.27-3.21 (m, 4H), 1.66 (m, 6H), 1.23 (t, *J* = 7.1 Hz, 3H); [13] C NMR (100 MHz, $CDCl_3$, ppm): δ = 187.7, 162.0, 158.8, 133.8, 132.5, 129.8, 129.8, 120.6, 61.8, 52.7, 25.8, 22.7, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1716, 1682, 1583, 1552, 1488, 1394, 1303, 1242, 1137, 1066, 950, 891, 821, 726, 697, 663, 516. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_5Br$ $[M+Na]^+$ 433.0375, found 433.0377.

(*E*)-ethyl 4-(naphthalen-2-yl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4aj)



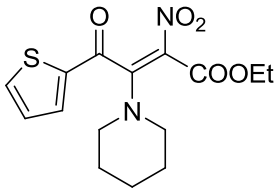
The title compound was prepared according to the general working procedure (3 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow solid: 72% yield; mp = 144-145 ℃; ^{1} H NMR (400 MHz, $CDCl_3$, ppm): δ = 8.44 (s, 1H), 8.04-8.01 (m, 1H), 7.96 (t, *J* = 8.6 Hz, 2H), 7.89 (d, *J* = 8.1 Hz, 1H), 7.66-7.62 (m, 1H), 7.60-7.56 (m, 1H), 4.24-4.18 (m, 2H), 3.37-3.28 (m, 4H), 1.67 (m, 6H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.8, 162.2, 159.7, 136.2, 132.5, 132.4, 130.8, 129.9, 129.3, 129.3, 127.9, 127.2, 123.4, 120.7, 61.8, 52.8, 25.9, 22.8, 13.9. IR (film, ν/cm^{-1}): 2971, 2921, 1714, 1675, 1625, 1595, 1551, 1439, 1304, 1248, 1211, 1181, 1160, 1088, 1017, 856, 814, 751, 715, 663, 602, 583. HRMS (ESI) m/z calcd for $C_{21}H_{22}N_2O_5$ $[M+Na]^+$ 405.1426, found 405.1427.

(*E*)-ethyl 4-(naphthalen-1-yl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ak)



The title compound was prepared according to the general working procedure (3 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 63% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.08 (d, J = 8.6 Hz, 1H), 8.06 (d, *J* = 8.2 Hz, 1H), 7.89-7.86 (m, 2H), 7.72-7.69 (m, 1H), 7.60-7.56 (m, 1H), 7.50-7.46 (m, 1H), 4.20-4.18 (m, 2H), 3.35-3.34 (m, 4H), 1.71 (m, 6H), 1.20 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 189.6, 162.6, 162.3, 135.4, 134.0, 131.2, 130.9, 130.6, 129.2, 128.5, 127.1, 125.9, 124.2, 120.7, 61.7, 53.3, 26.1, 22.8, 14.0. IR (film, ν/cm^{-1}): 2923, 2855, 1715, 1674, 1552, 1507, 1485, 1441, 1303, 1265, 1244, 1211, 1182, 1117, 1089, 1012, 891, 832, 801, 774, 735, 648, 604, 501. HRMS (ESI) m/z calcd for $C_{21}H_{22}N_2O_5$ $[M+Na]^+$ 405.1426, found 405.1431.

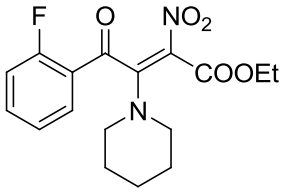
(*E*)-ethyl 2-nitro-4-oxo-3-(piperidin-1-yl)-4-(thiophen-2-yl)but-2-enoate (4al)



The title compound was prepared according to the general working procedure (2.5 h) and purified by column chromatography (petroleum ether / ethyl acetate = 3:1) to give the product as a yellow oil: 55% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.78-7.76 (m, 2H), 7.15-7.12 (m, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 3.28 (m, 4H), 1.71 (m, 6H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 181.0, 162.1, 159.1, 141.7, 136.4, 134.2, 128.7, 120.9, 61.7, 52.9, 26.0, 22.7,

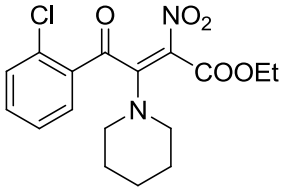
13.9. IR (film, ν/cm^{-1}): 2941, 2861, 1711, 1655, 1551, 1485, 1442, 1406, 1306, 1247, 1211, 1160, 1137, 1115, 1090, 1058, 1017, 983, 951, 912, 856, 730, 662, 623, 562. HRMS (ESI) m/z calcd for $C_{15}H_{18}N_2O_5S$ $[M+Na]^+$ 361.0834, found 361.0833.

(*E*)-ethyl 4-(2-fluorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4am)



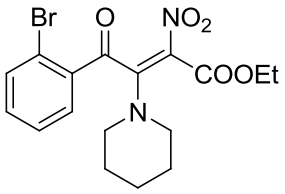
The title compound was prepared according to the general working procedure (3 h) and purified by column chromatography (petroleum ether / ethyl acetate = 4:1) to give the product as a yellow oil: 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 8.00-7.96 (m, 1H), 7.63-7.58 (m, 1H), 7.32-7.28 (m, 1H), 7.20-7.14 (m, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 3.26 (m, 4H), 1.64 (m, 6H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 185.0, 162.2, 161.3, 161.8 (d, *J* = 256.5 Hz), 136.2 (d, *J* = 9.2 Hz), 130.6, 124.9 (d, *J* = 3.3 Hz), 123.9 (d, *J* = 9.8 Hz), 119.3, 116.9 (d, *J* = 21.8 Hz), 61.6, 52.7, 25.6, 22.6, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1771, 1715, 1675, 1635, 1608, 1557, 1481, 1454, 1407, 1303, 1249, 1213, 1137, 1075, 891, 869, 808, 765, 733, 634, 519. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_5F$ $[M+Na]^+$ 373.1176, found 373.1175.

(*E*)-ethyl 4-(2-chlorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4an)



The title compound was prepared according to the general working procedure (2 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 66% yield;^{1} H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.90 (dd, *J* = 7.8 Hz, 1.4 Hz, 1H), 7.52-7.47 (m, 1H), 7.46-7.37 (m, 2H), 4.24 (q, *J* = 7.1 Hz, 2H), 3.30 (m, 4H), 1.70 (m, 6H), 1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 187.2, 162.1, 161.6, 134.2, 133.8, 133.8, 132.3, 131.3, 127.1, 119.9, 61.6, 53.4, 25.8, 22.7, 14.0. IR (film, ν/cm^{-1}): 2987, 2920,1697, 1586, 1556, 1435, 1393, 1303, 1241, 1218, 1160, 1137, 1066, 1027, 950, 920, 855, 758, 728, 647, 631. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_5Cl$ $[M+Na]^+$ 389.0880, found 389.0881.

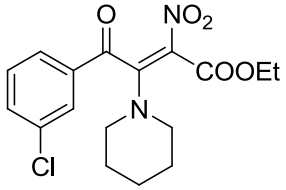
(*E*)-ethyl 4-(2-bromophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ao)



The title compound was prepared according to the general working procedure (1.5 h) and purified by column chromatography (petroleum ether / ethyl acetate = 4:1) to give the product as a yellow

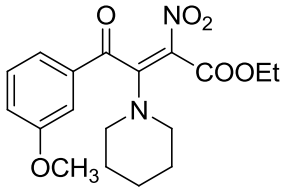
oil: 57% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.84 (dd, *J* = 7.6 Hz, 2.0 Hz, 1H), 7.68-7.66 (m, 1H), 7.45-7.37 (m, 2H), 4.24 (q, *J* = 7.1 Hz, 2H), 3.31 (m, 4H), 1.71 (m, 6H), 1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 187.8, 162.1, 161.2, 135.2, 134.9, 134.1, 132.7, 127.5, 122.1, 120.3, 61.7, 53.6, 25.9, 22.7, 14.1. IR (film, ν/cm^{-1}): 2987, 2900, 1697, 1552, 1430, 1393, 1303, 1241, 1211, 1160, 1137, 1088, 950, 890, 856, 764, 725, 697, 641, 616, 452. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_5Br$ $[M+Na]^+$ 433.0375, found 433.0381.

(*E*)-ethyl 4-(3-chlorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ap)



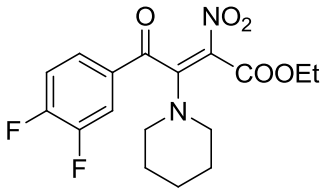
The title compound was prepared according to the general working procedure (1 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 73% yield;[1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.95 (t, *J* = 1.8 Hz, 1H), 7.82-7.79 (m, 1H), 7.61-7.58 (m, 1H), 7.46 (t, *J* = 7.9 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 3.30-3.24 (m, 4H), 1.69 (m, 6H), 1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 187.4, 162.0, 158.6, 136.5, 135.5, 134.3, 130.5, 128.3, 126.5, 120.6, 61.9, 52.7, 25.9, 22.7, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1762, 1705, 1678, 1635, 1557, 1470, 1394, 1337, 1297, 1241, 1161, 1144, 1077, 1048, 1027, 951, 912, 892, 879, 858, 785, 769, 743, 715, 669, 643, 502. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_2O_5Cl$ $[M+Na]^+$ 389.0880, found 389.0885.

(*E*)-ethyl 4-(3-methoxyphenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4aq)



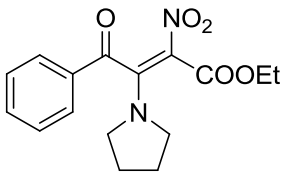
The title compound was prepared according to the general working procedure (3.5 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 58% yield; [1] H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.49 (d, *J* = 7.4 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.17-7.14 (m, 1H), 4.24-4.19 (m, 2H), 3.85 (s, 3H), 3.31-3.23 (m, 4H), 1.67 (m, 6H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.6, 162.2, 160.1, 159.5, 136.2, 130.1, 121.5, 121.3, 120.6, 111.9, 61.8, 55.5, 52.7, 25.9, 22.8, 13.9. IR (film, ν/cm^{-1}): 2987, 2900, 1708, 1677, 1594, 1556, 1508, 1488, 1471, 1456, 1406, 1394, 1299, 1242, 1225, 1160, 1143, 1123, 1077, 921, 893, 873, 855, 828, 814, 797, 756, 733, 697, 675, 662, 634, 601, 511. HRMS (ESI) m/z calcd for $C_{18}H_{22}N_2O_6$ $[M+Na]^+$ 385.1376, found 385.1374.

(*E*)-ethyl 4-(3,4-difluorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ar)



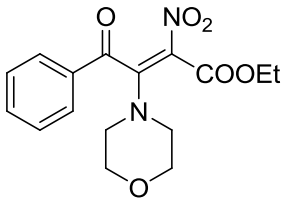
The title compound was prepared according to the general working procedure (2.5 h) and purified by column chromatography (petroleum ether / ethyl acetate = 5:1) to give the product as a yellow oil: 61% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.78-7.73 (m, 1H), 7.67-7.64 (m, 1H), 7.24-7.20 (m, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 3.23-3.16 (m, 4H), 1.62 (m, 6H), 1.20 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 185.3, 161.0, 157.2, 153.4 (dd, *J* = 258.2 Hz, *J* = 12.8 Hz), 149.8 (dd, *J* = 251.5 Hz, *J* = 13.1 Hz), 131.2 (m), 124.6 (dd, *J* = 7.5 Hz, *J* = 3.5 Hz), 119.7, 117.3 (d, *J* = 18.1 Hz), 116.6 (d, *J* = 18.3 Hz), 61.0, 51.7, 24.9, 21.7, 12.9. IR (film, ν/cm^{-1}): 2987, 2900, 1708, 1677, 1594, 1556, 1508, 1488, 1471, 1406, 1394, 1299, 1242, 1160, 1143, 1123, 1077, 1027, 892, 873, 855, 828, 815, 756, 733, 697, 675, 634, 601, 511. HRMS (ESI) m/z calcd for $C_{17}H_{18}N_2O_5F_2$ $[M+Na]^+$ 391.1081, found 391.1087.

(*E*)-ethyl 2-nitro-4-oxo-4-phenyl-3-(pyrrolidin-1-yl)but-2-enoate (4as)



The title compound was prepared according to the general working procedure (4.5 h) and purified by column chromatography (petroleum ether / ethyl acetate = 3:1) to give the product as a yellow oil: 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.97-7.94 (m, 2H), 7.64-7.60 (m, 1H), 7.52-7.48 (m, 2H), 4.23-4.22 (m, 2H), 3.48-3.29 (m, 4H), 1.96-1.95 (m, 4H), 1.25 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 186.5, 161.2, 153.5, 133.4, 133.2, 128.3, 127.3, 119.2, 60.9, 50.3, 24.0, 12.9. IR (film, ν/cm^{-1}): 2987, 2900, 1732, 1697, 1596, 1558, 1506, 1488, 1451, 1405, 1381, 1291, 1250, 1075, 1066, 1056, 1027, 891, 869, 702, 643, 457. HRMS (ESI) m/z calcd for $C_{16}H_{18}N_2O_5$ $[M+H]^+$ 319.1289, found 319.1295.

(*E*)-ethyl 3-morpholino-2-nitro-4-oxo-4-phenylbut-2-enoate (4at)

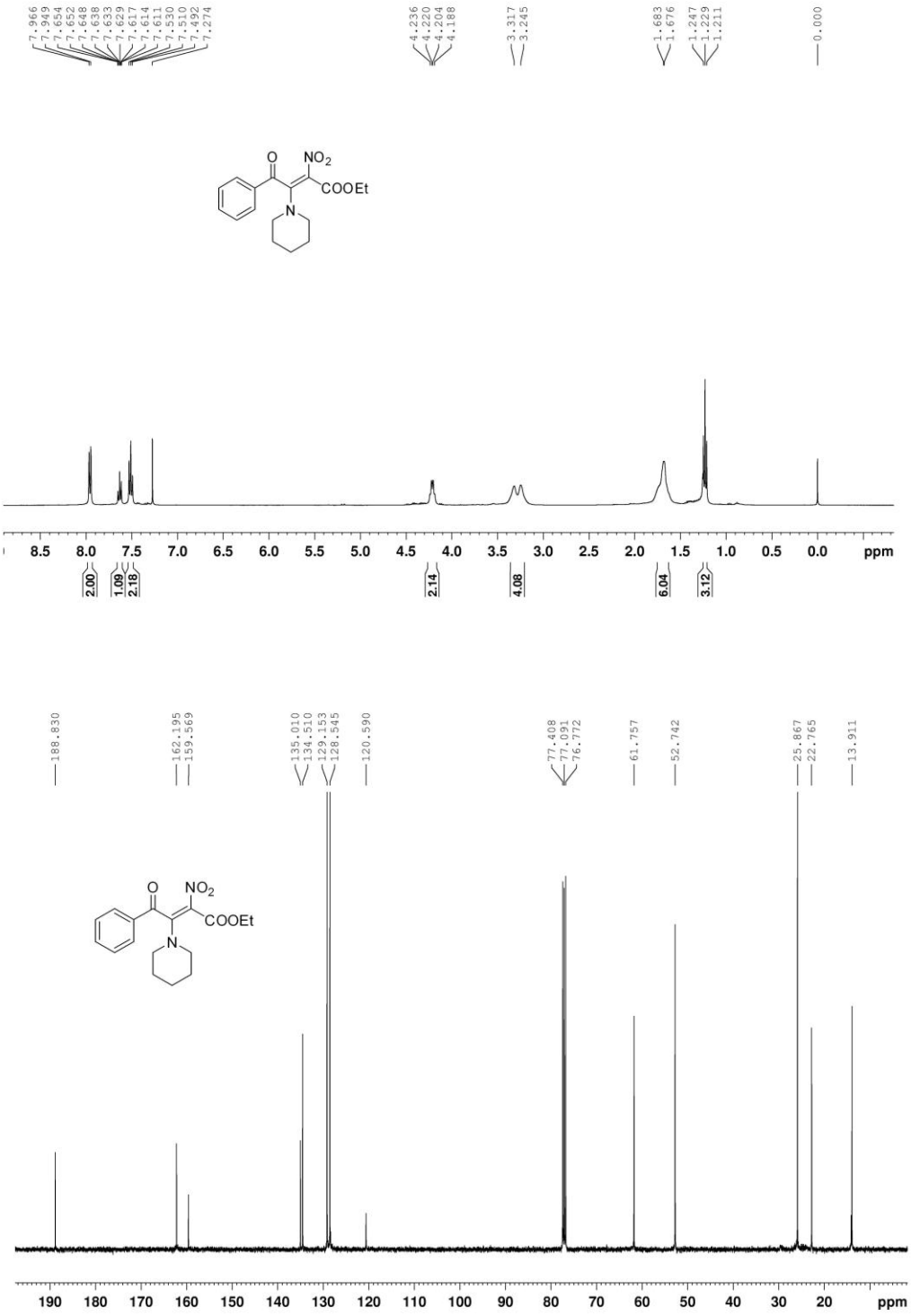


The title compound was prepared according to the general working procedure (3 h) and purified by column chromatography (petroleum ether / ethyl acetate = 4:1) to give the product as a yellow oil: 56% yield; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.96-7.94 (m, 2H), 7.67-7.62 (m, 1H), 7.54-7.49 (m, 2H), 4.19-4.18 (m, 2H), 3.77-3.72 (m, 4H), 3.37-3.24 (m, 4H), 1.21 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 188.5, 161.8, 158.1, 134.9, 134.8, 129.4, 128.5, 121.3, 66.2, 62.1, 51.2, 13.9. IR (film, ν/cm^{-1}): 2980, 2922, 1717, 1683, 1596, 1556, 1488, 1449,

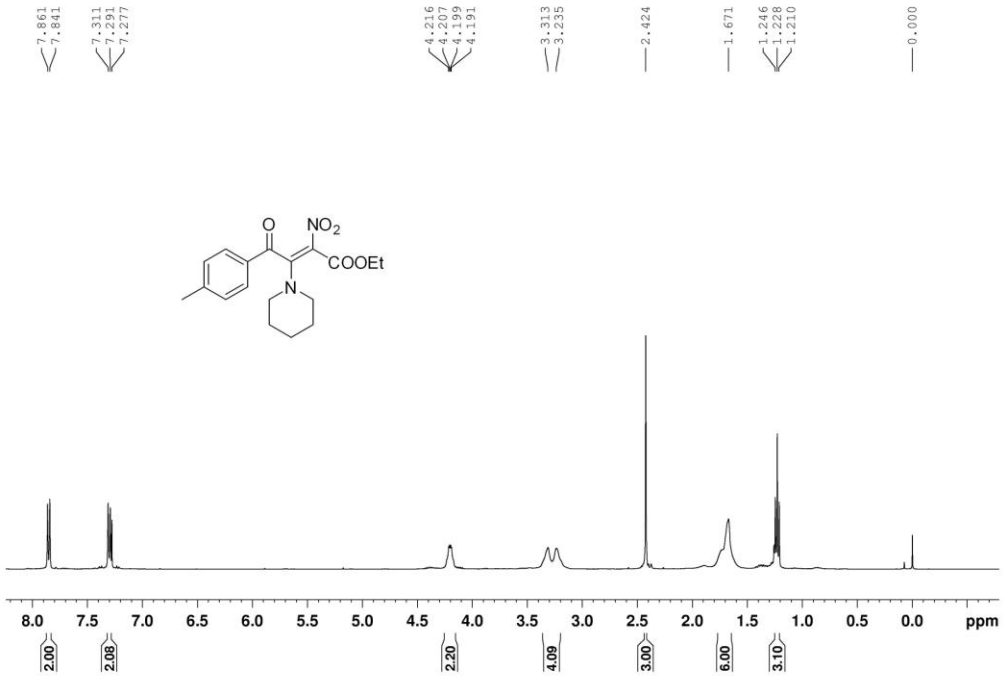
1370, 1296, 1249, 1176, 1118, 1098, 1046, 1026, 895, 873, 844, 815, 768, 702, 645, 615, 502, 435. HRMS (ESI) m/z calcd for $C_{16}H_{18}N_2O_6$ $[M+H]^+$ 335.1238, found 335.1243.

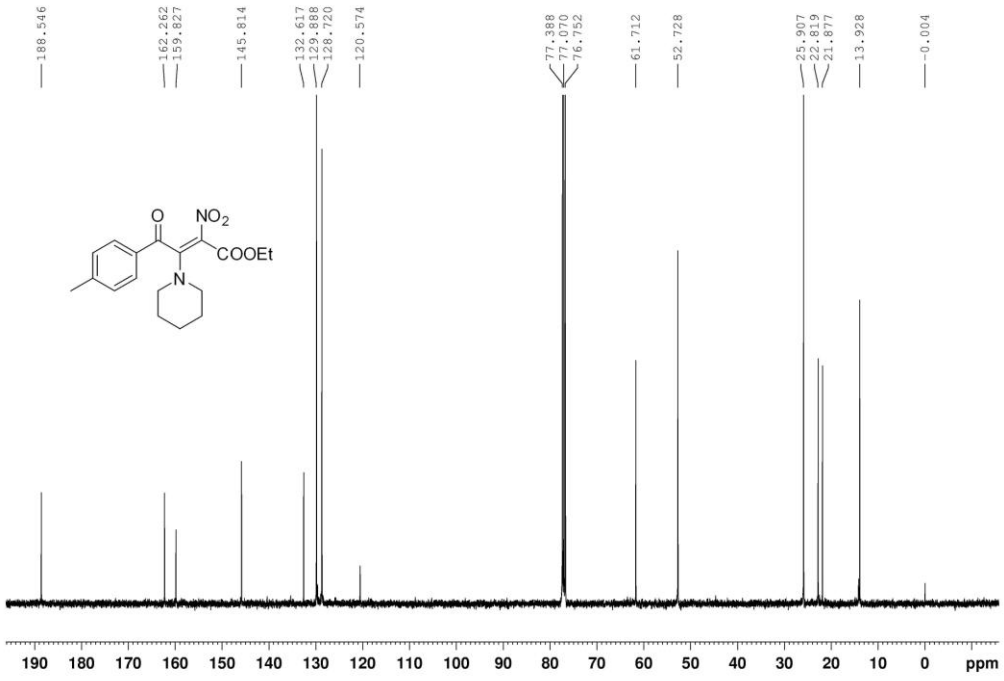
NMR spectra for the products

(*E*)-ethyl 2-nitro-4-oxo-4-phenyl-3-(piperidin-1-yl)but-2-enoate (4aa)

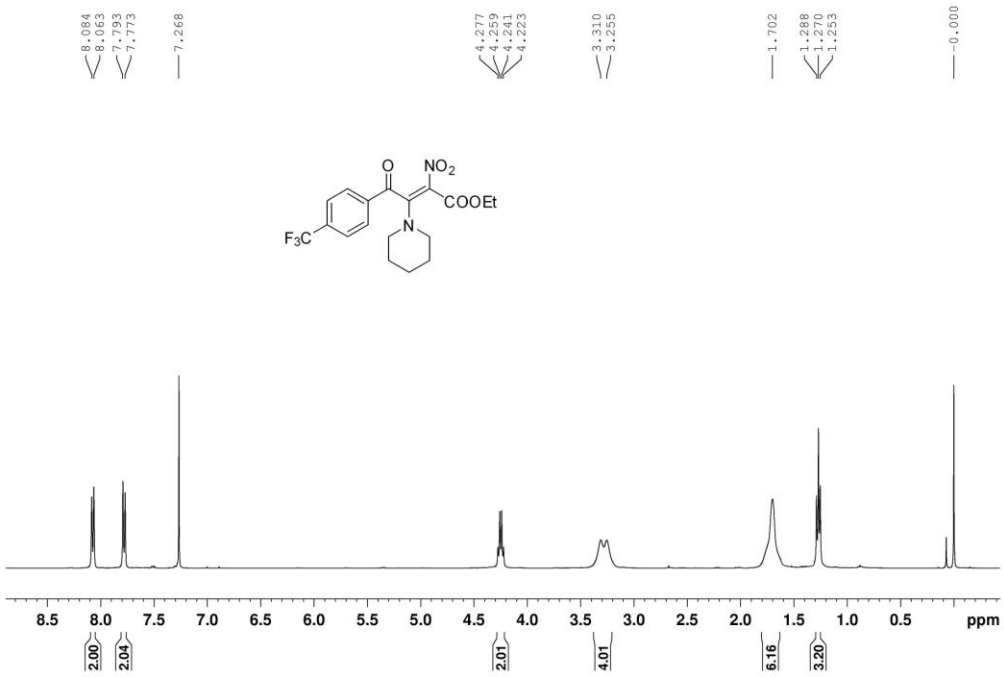


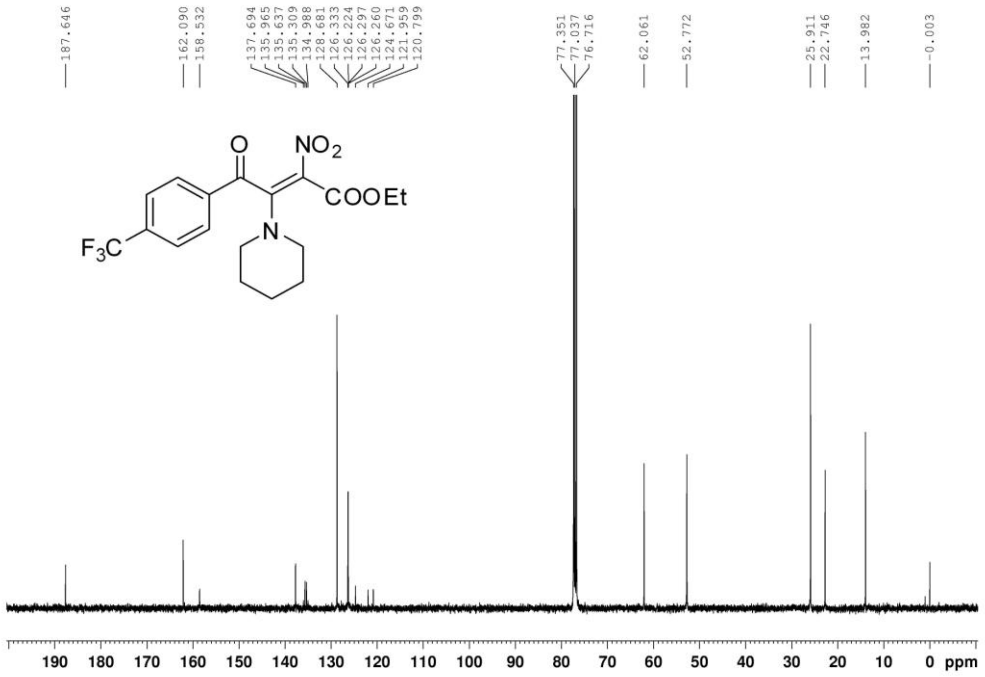
(*E*)-ethyl 2-nitro-4-oxo-3-(piperidin-1-yl)-4-*p*-tolylbut-2-enoate (4ab)



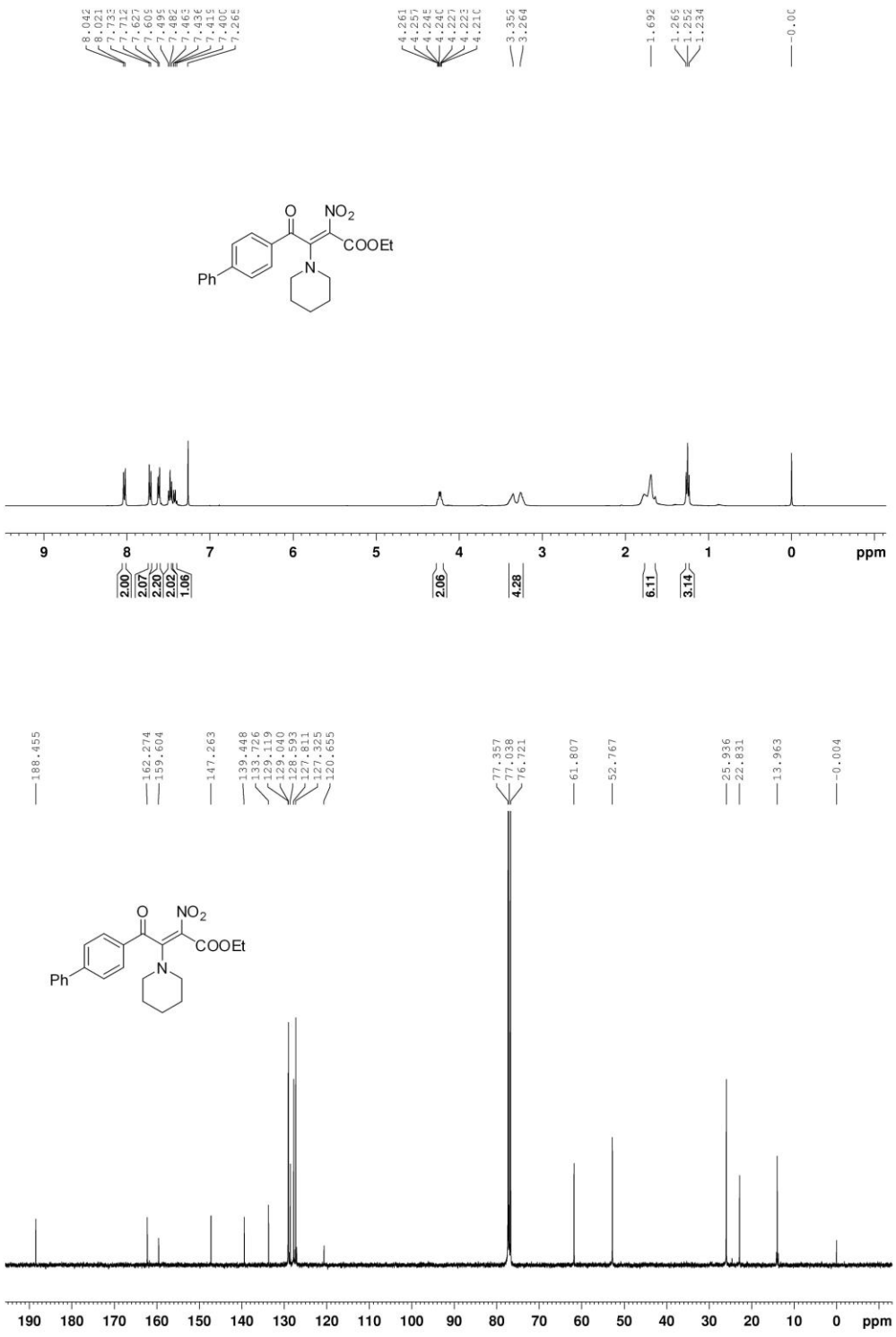


(*E*)-ethyl 2-nitro-4-oxo-3-(piperidin-1-yl)-4-(4-(trifluoromethyl)phenyl)but-2-enoate (4ac)

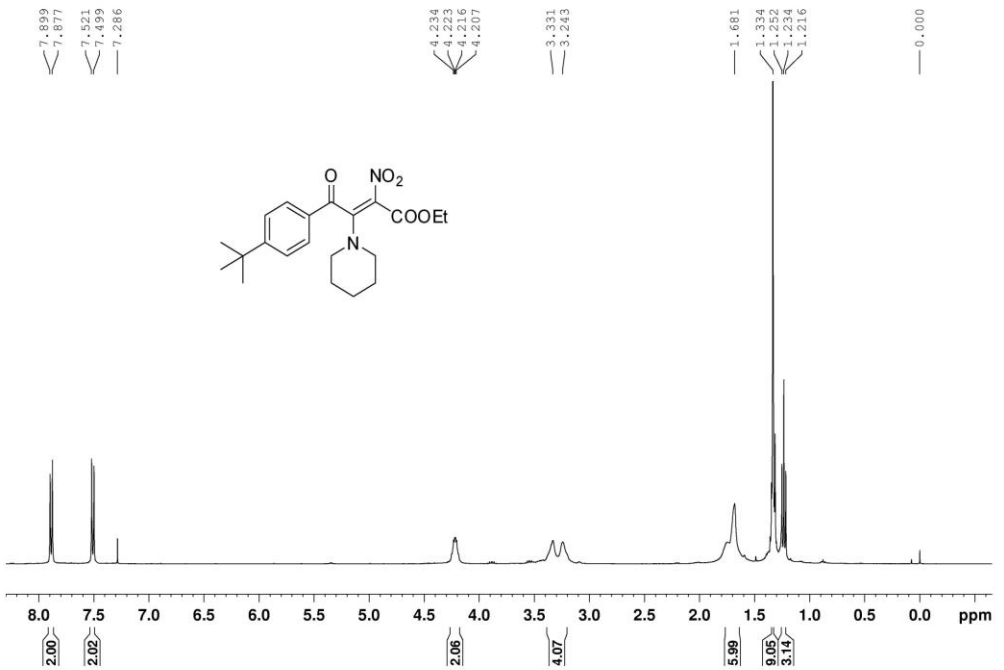


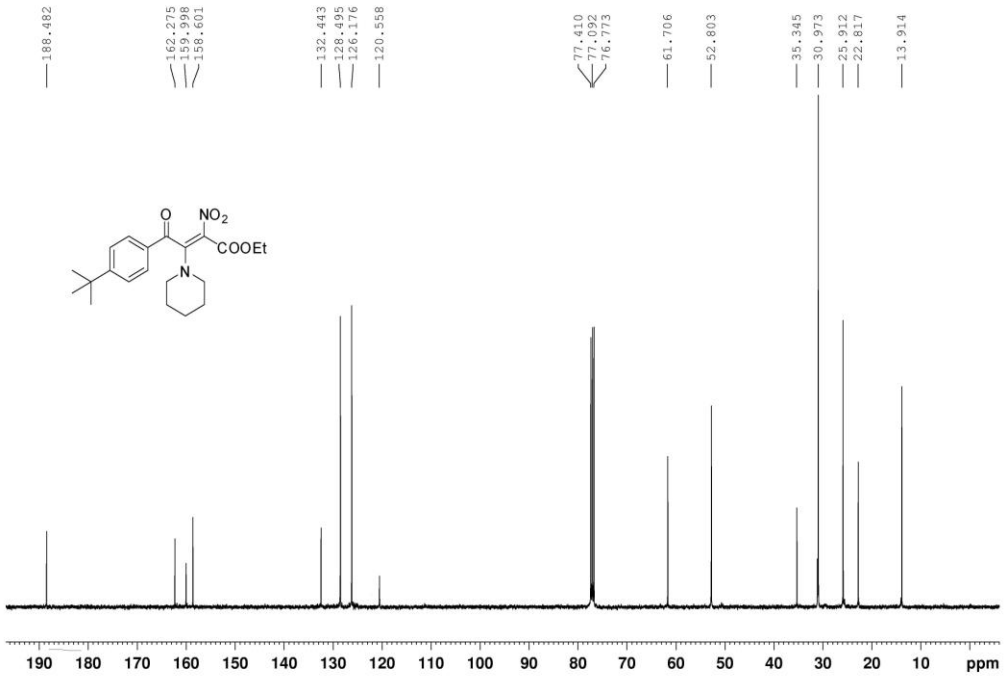


(*E*)-ethyl 4-(biphenyl-4-yl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ad)

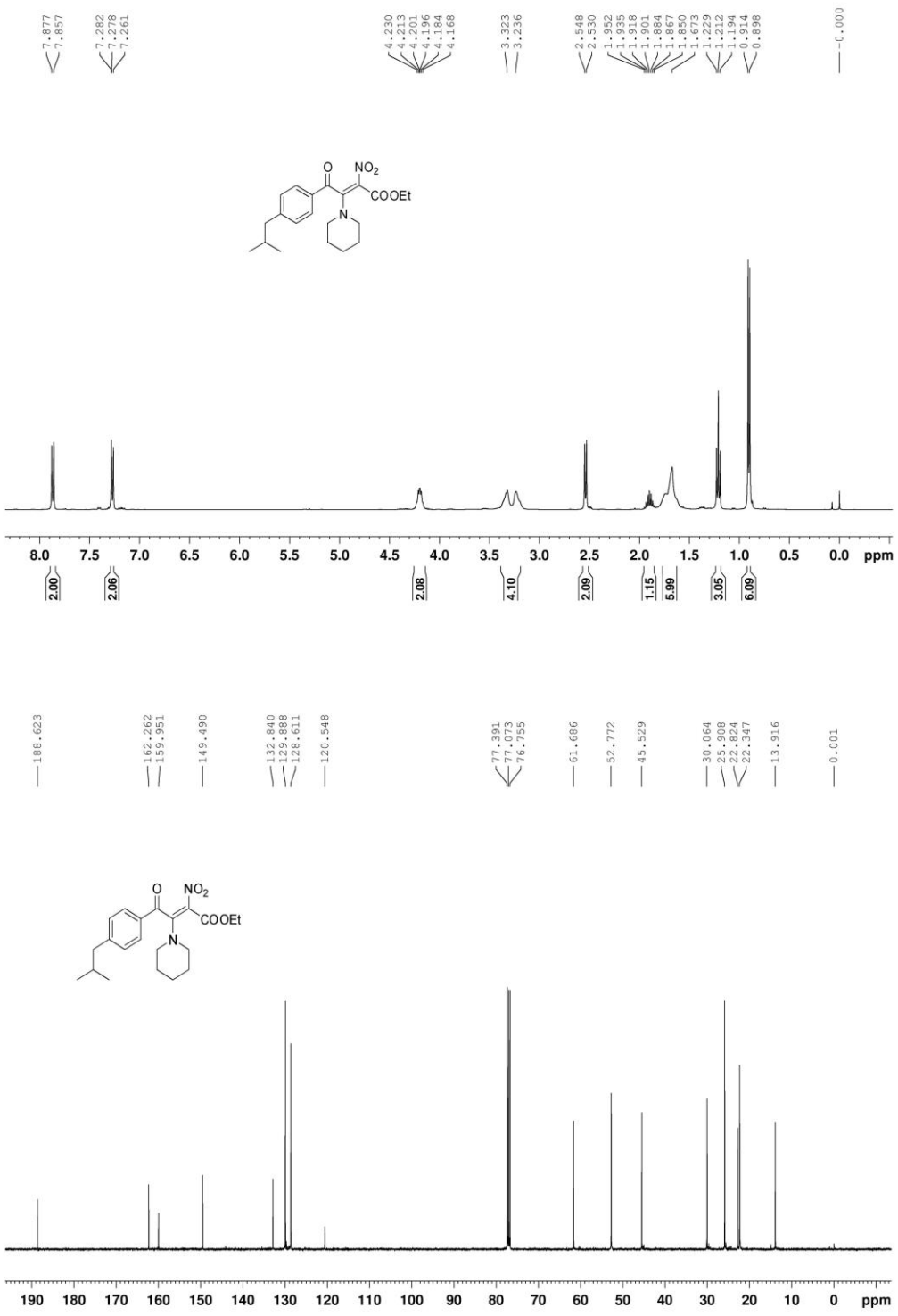


(*E*)-ethyl 4-(4-*tert*-butylphenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ae)

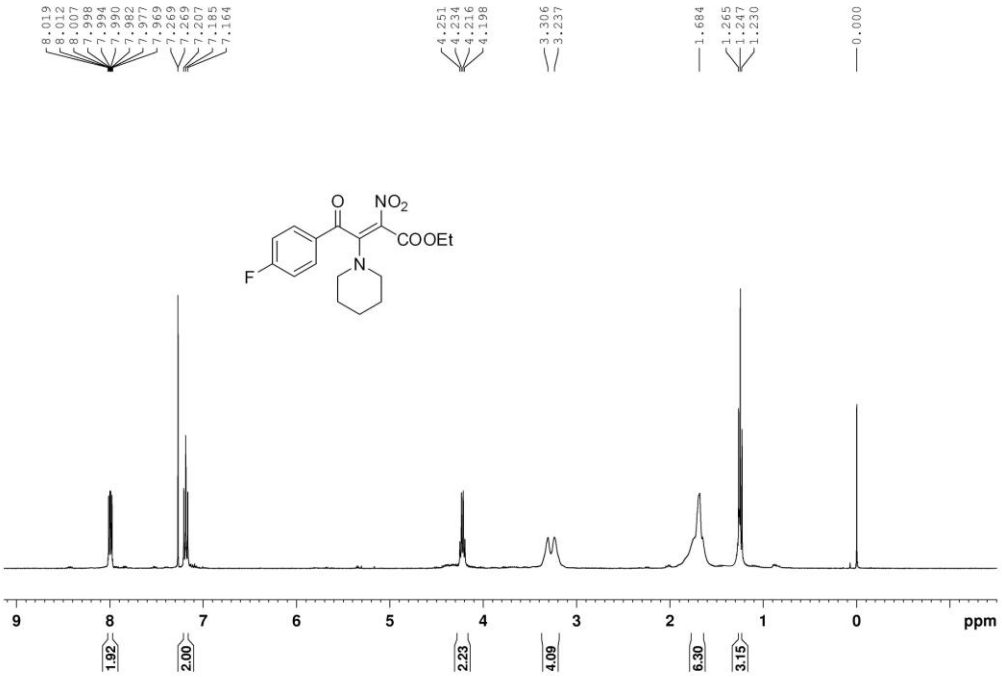


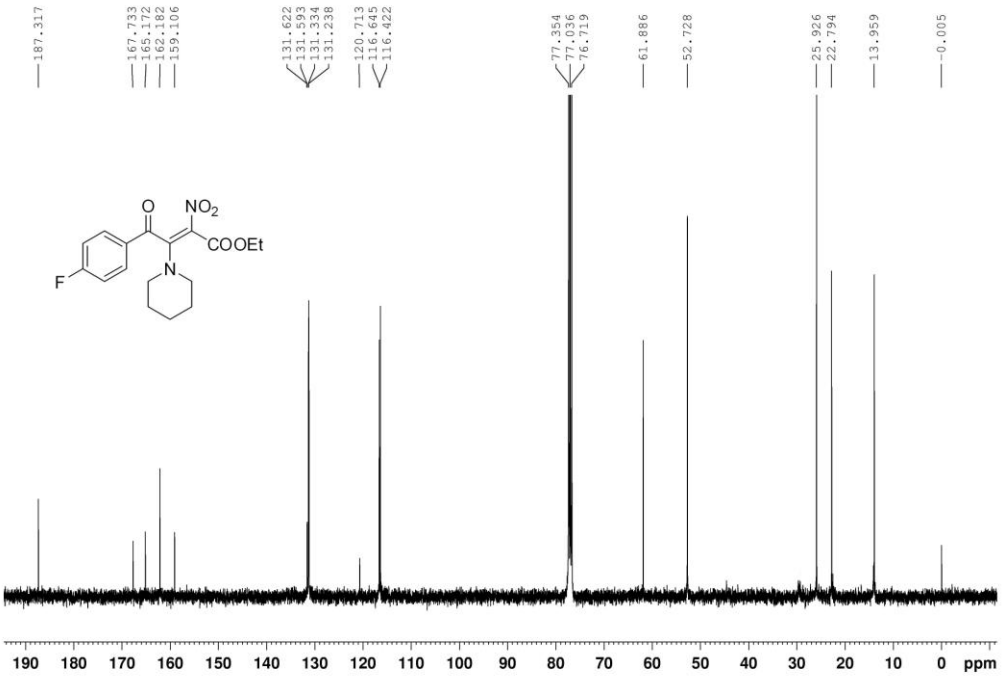


(*E*)-ethyl 4-(4-isobutylphenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4af)

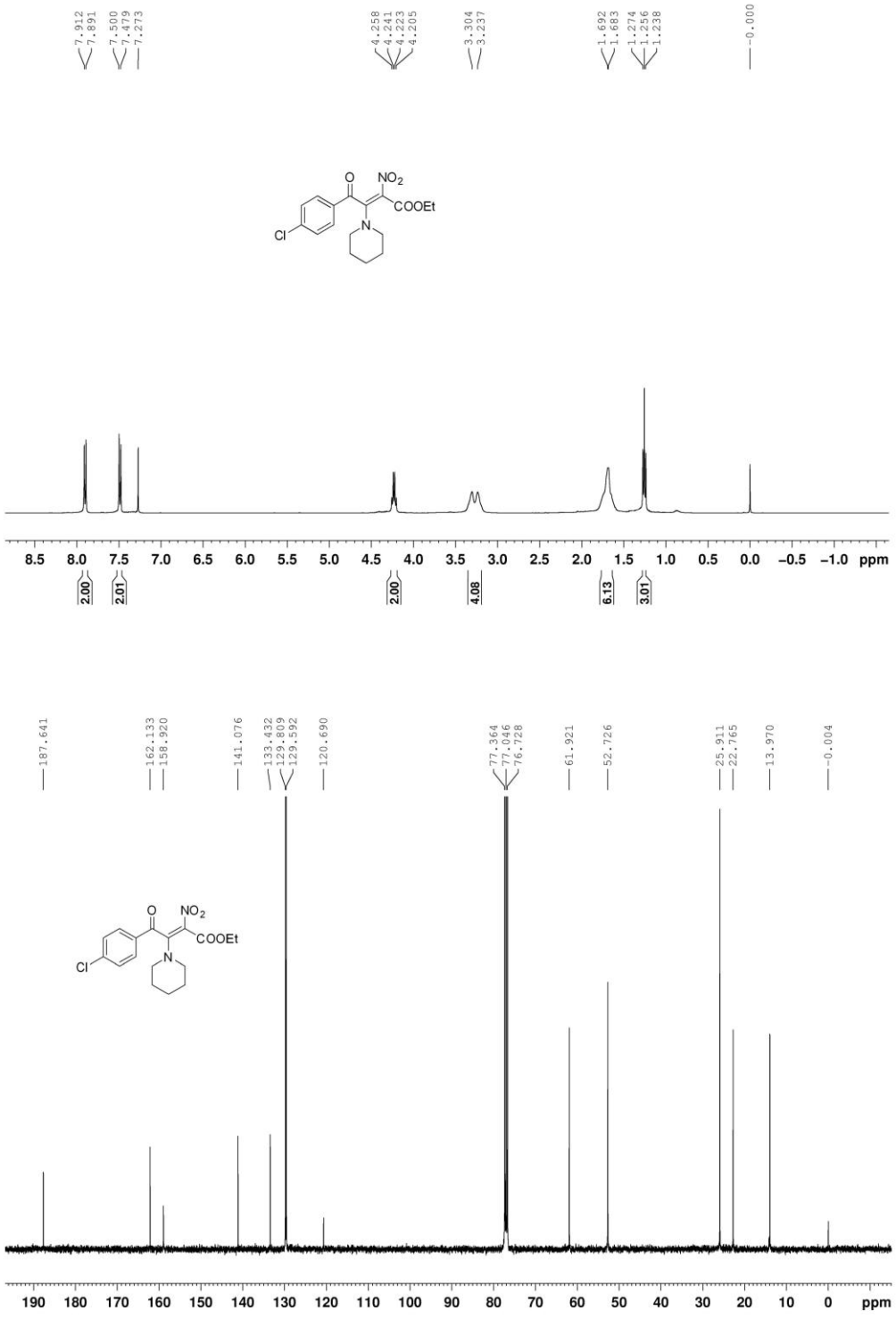


(*E*)-ethyl 4-(4-fluorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ag)

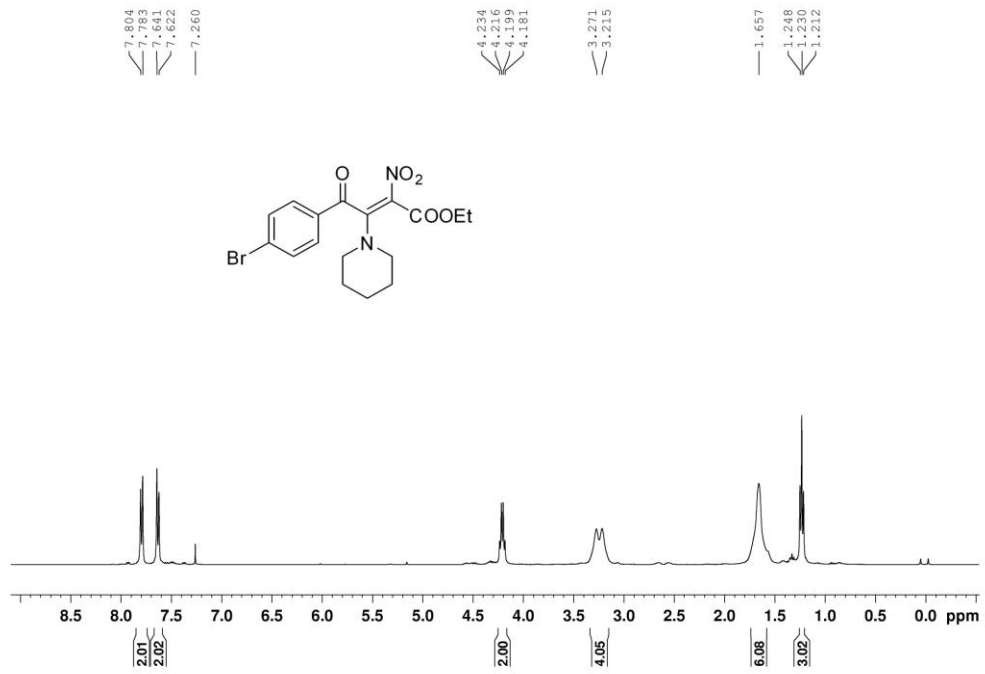


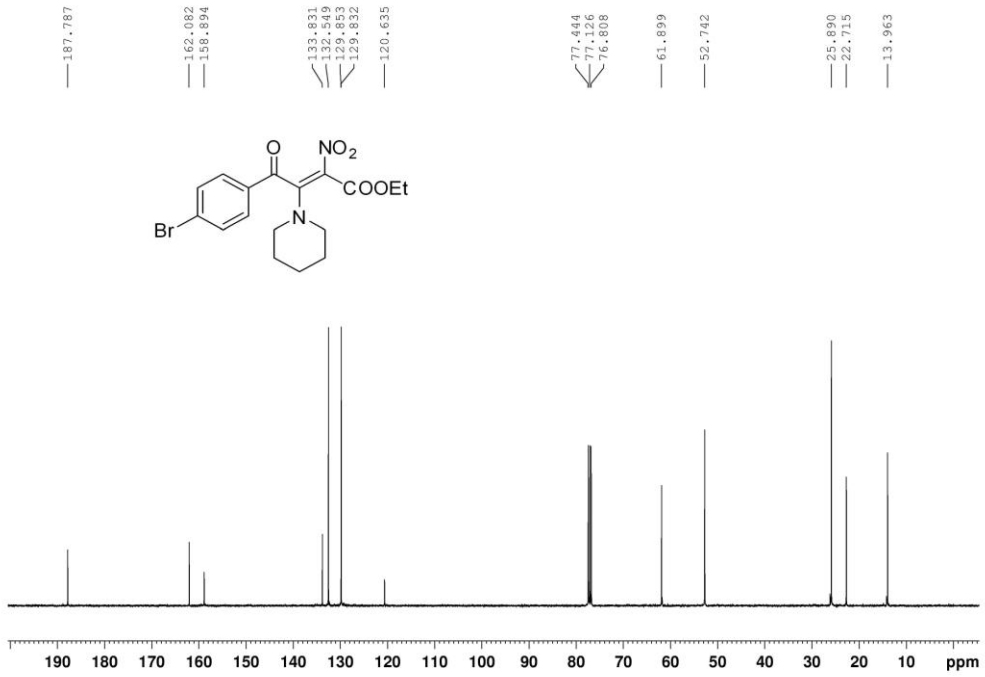


(*E*)-ethyl 4-(4-chlorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ah)

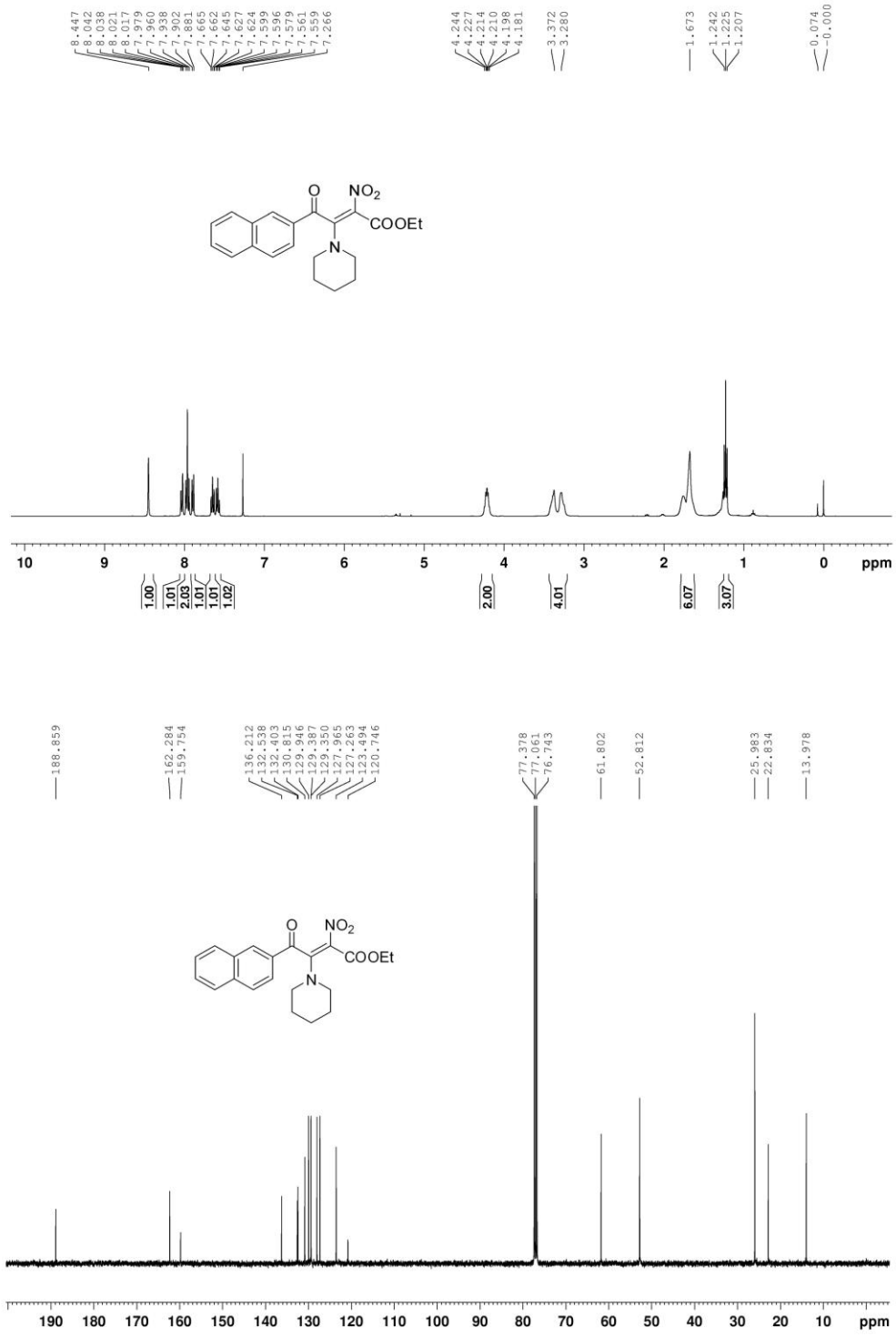


(*E*)-ethyl 4-(4-bromophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ai)

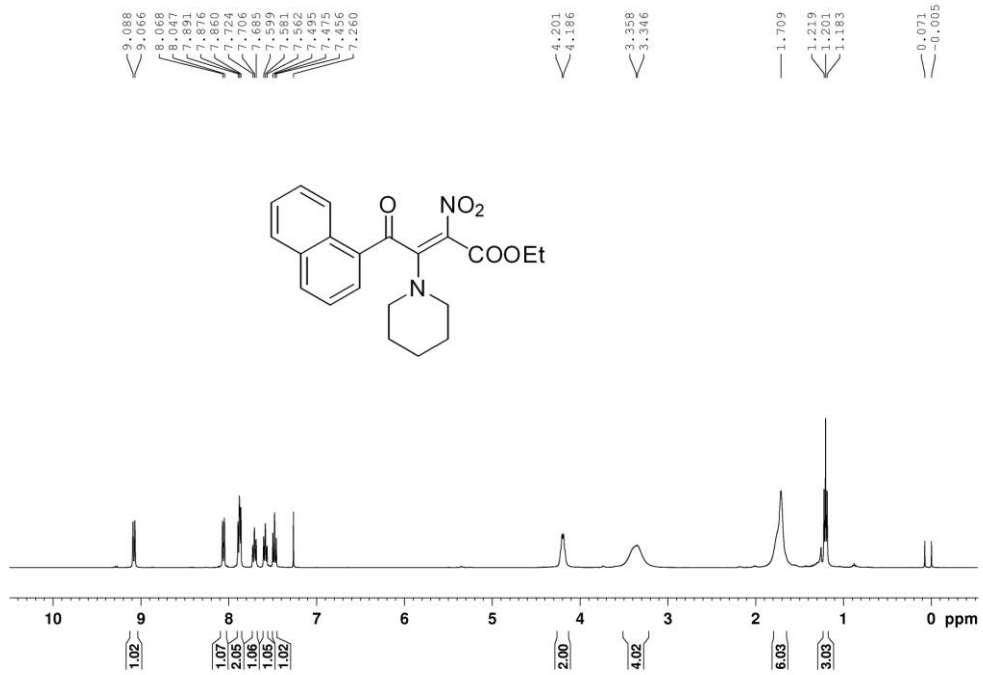


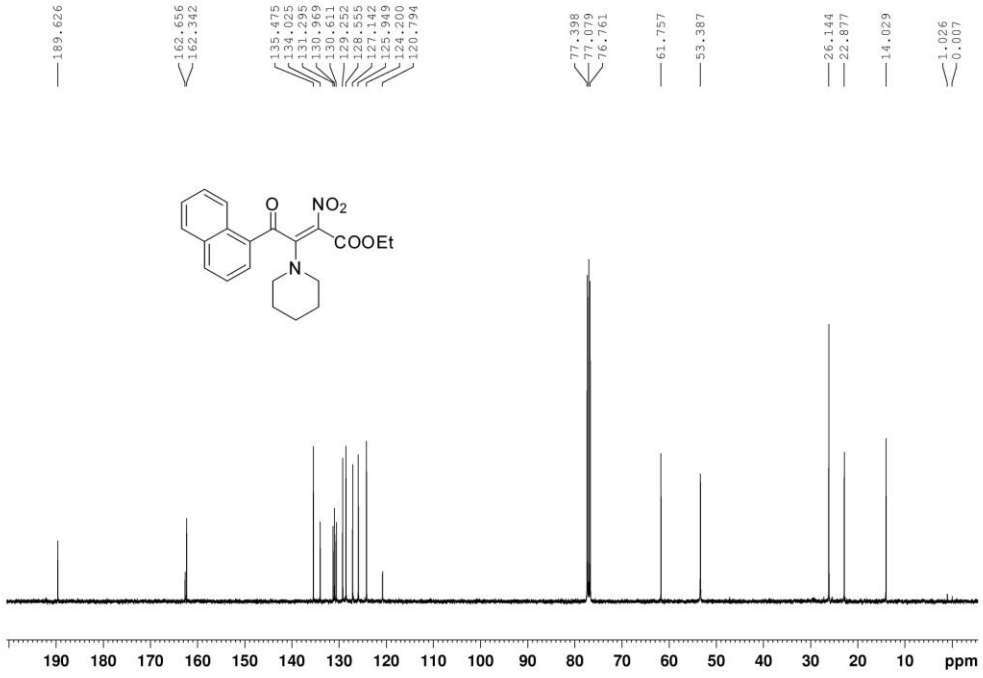


(*E*)-ethyl 4-(naphthalen-2-yl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4aj)

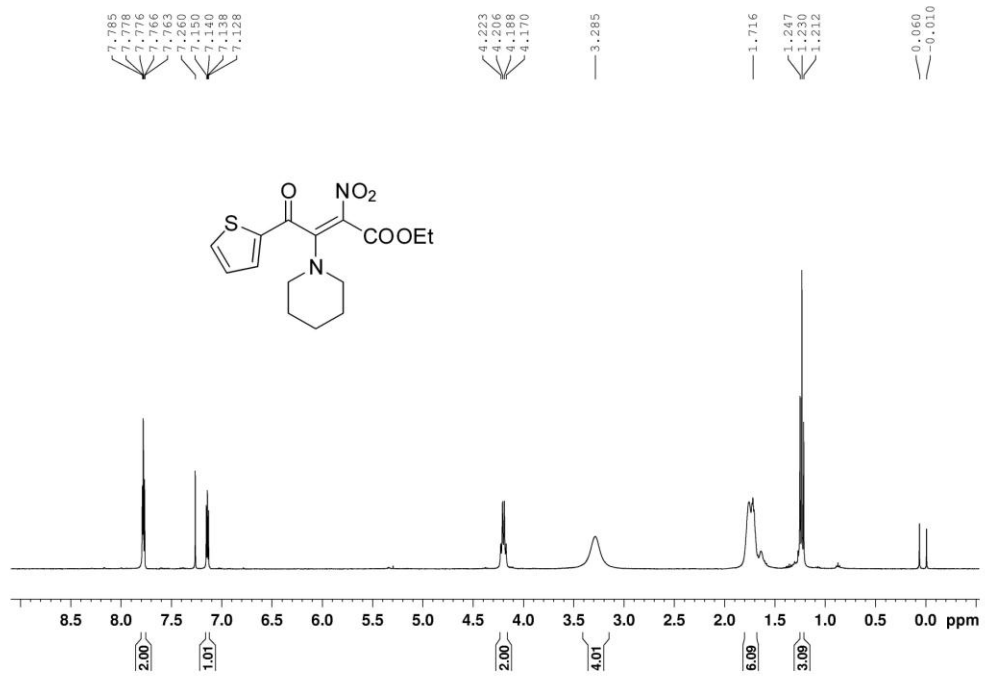


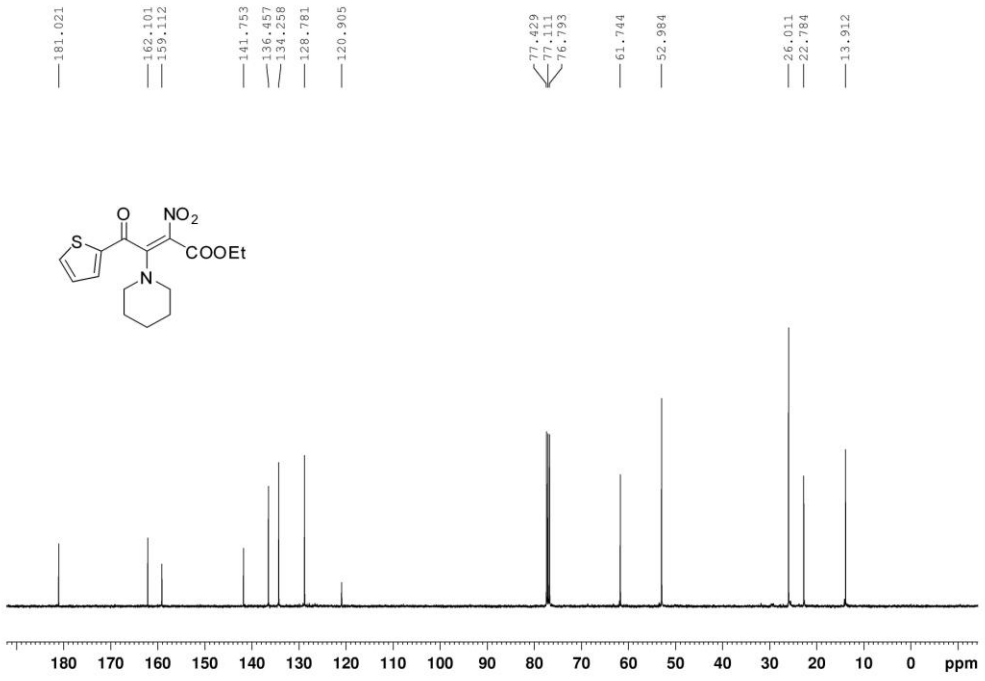
(*E*)-ethyl 4-(naphthalen-1-yl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ak)



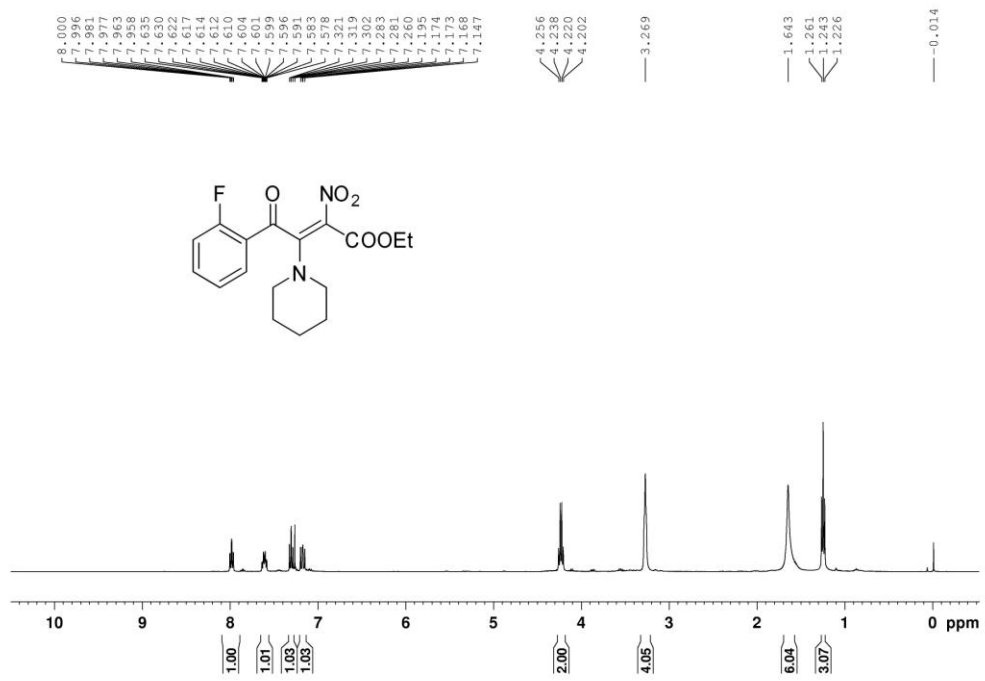


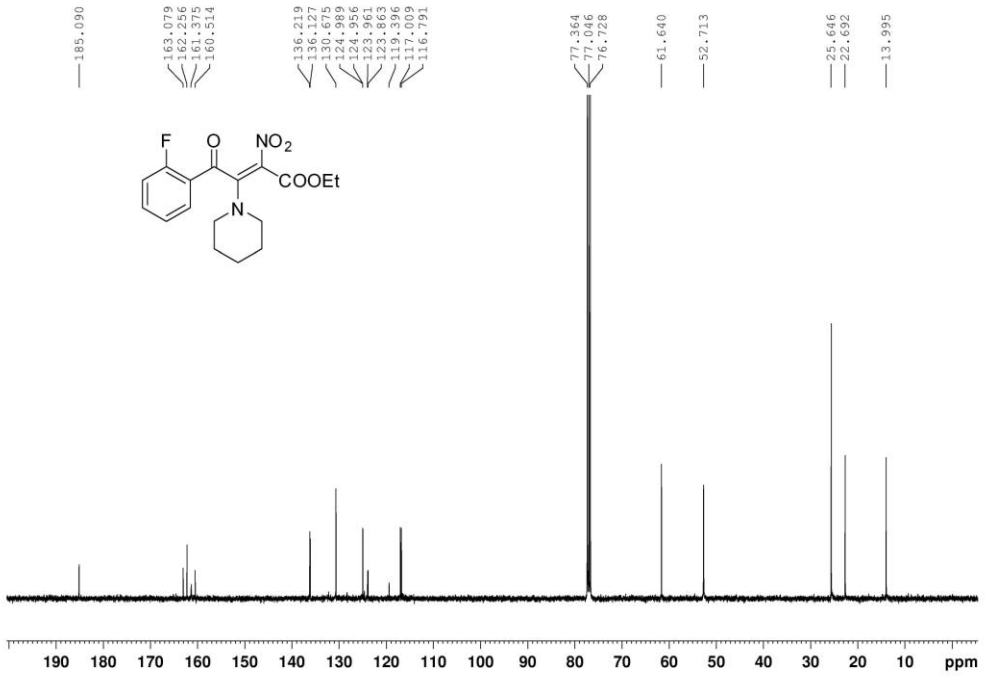
(*E*)-ethyl 2-nitro-4-oxo-3-(piperidin-1-yl)-4-(thiophen-2-yl)but-2-enoate (4al)



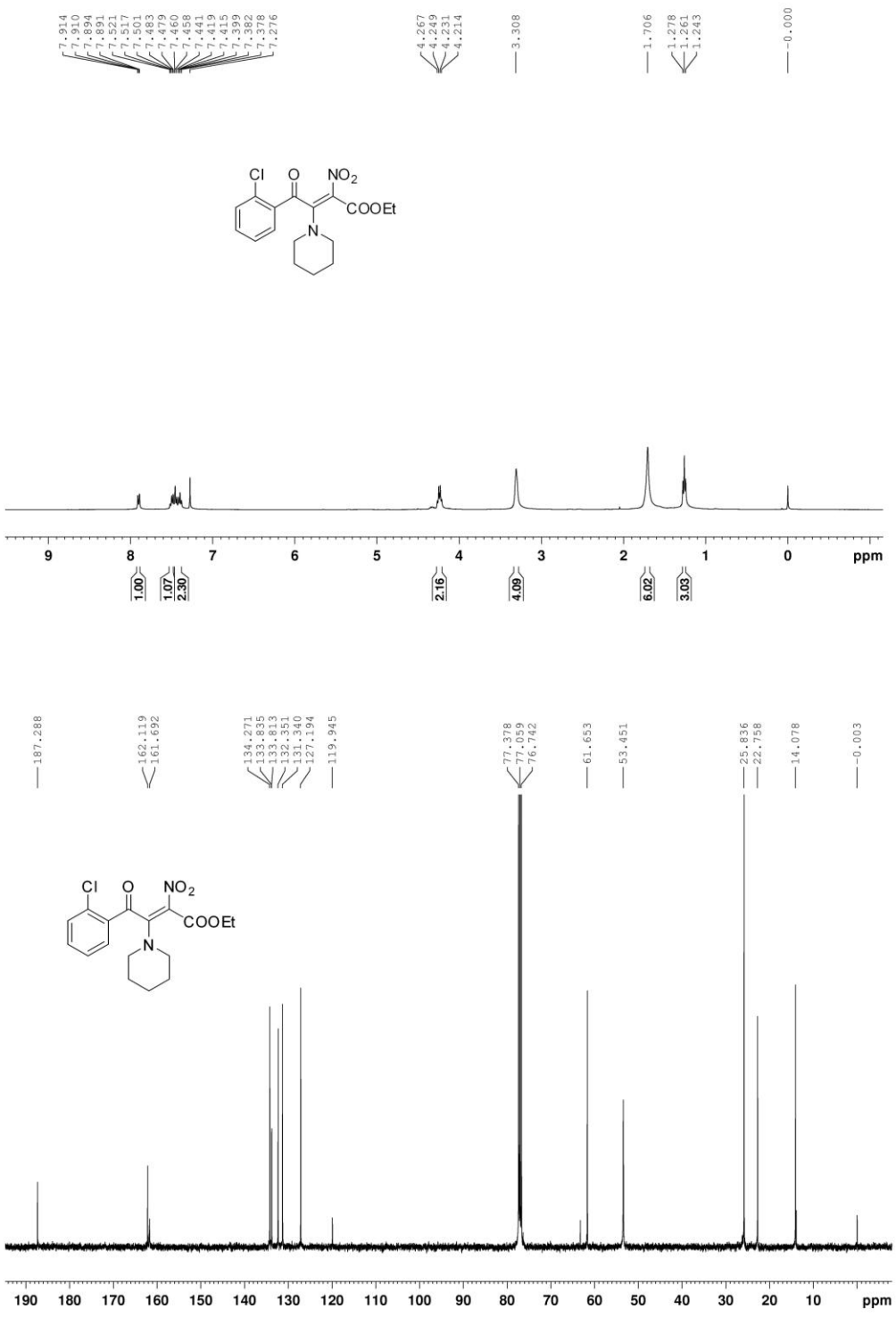


(*E*)-ethyl 4-(2-fluorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4am)

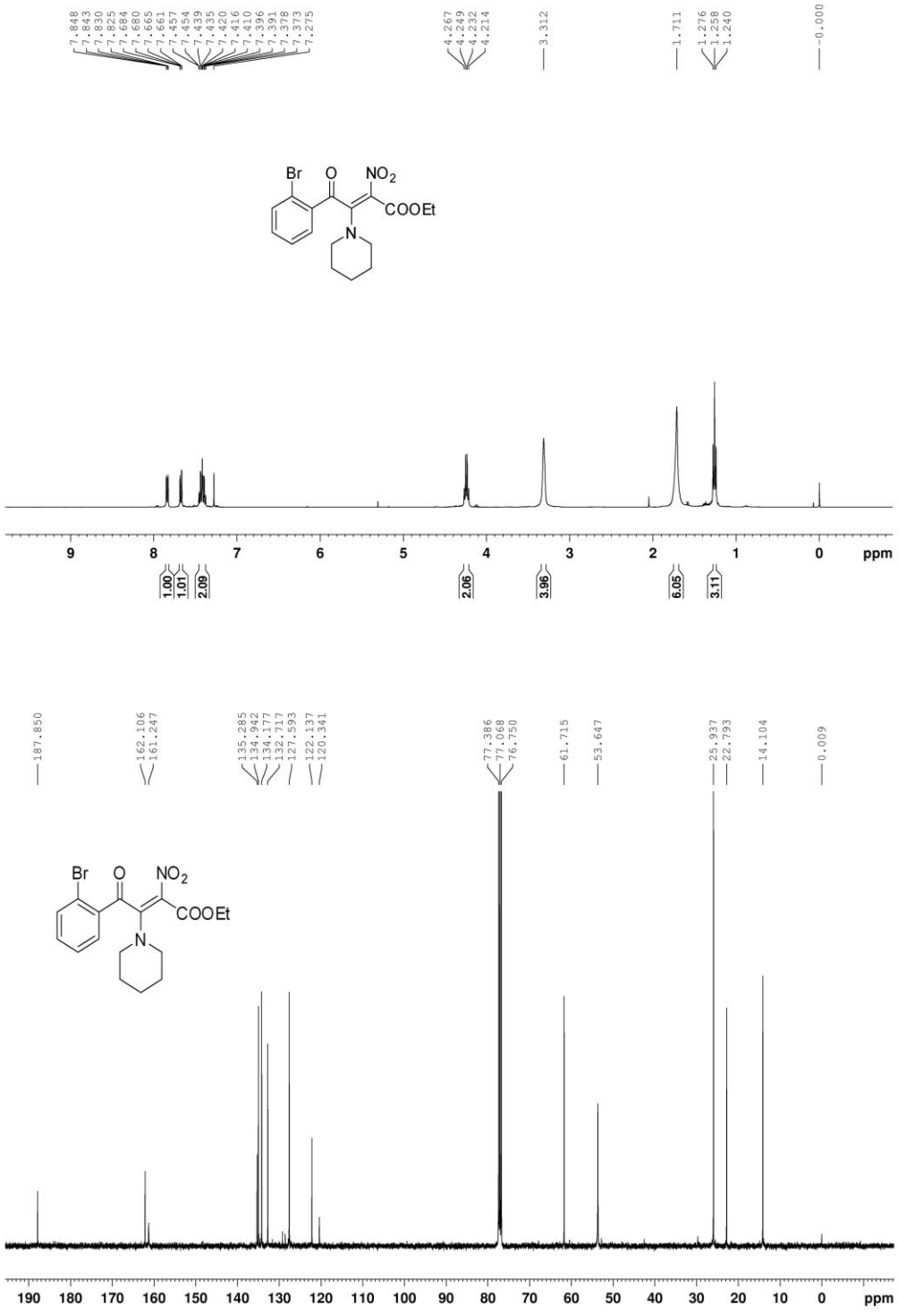




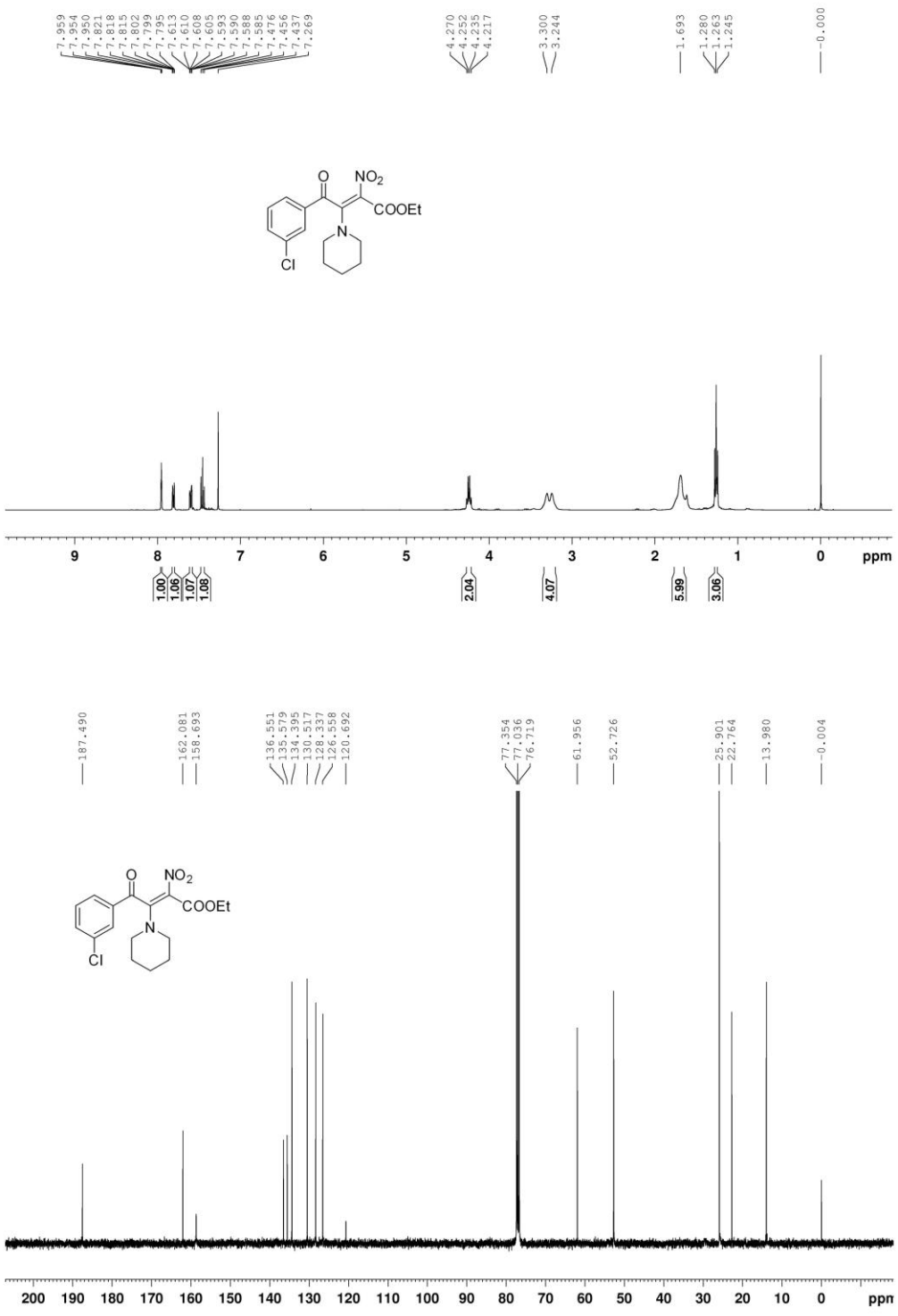
(*E*)-ethyl 4-(2-chlorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4an)



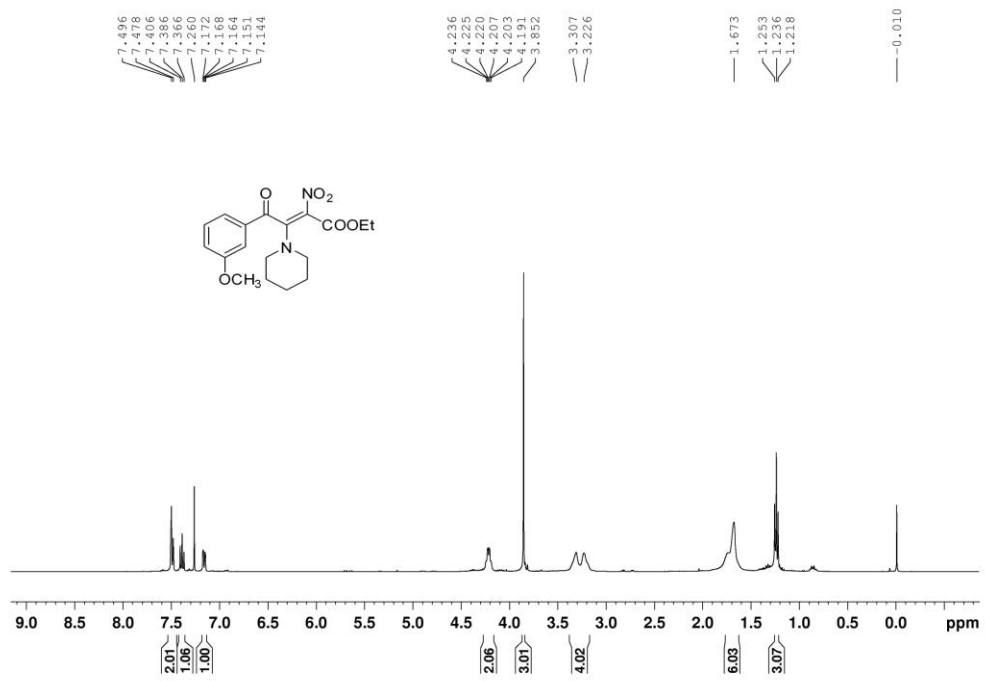
(*E*)-ethyl 4-(2-bromophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ao)

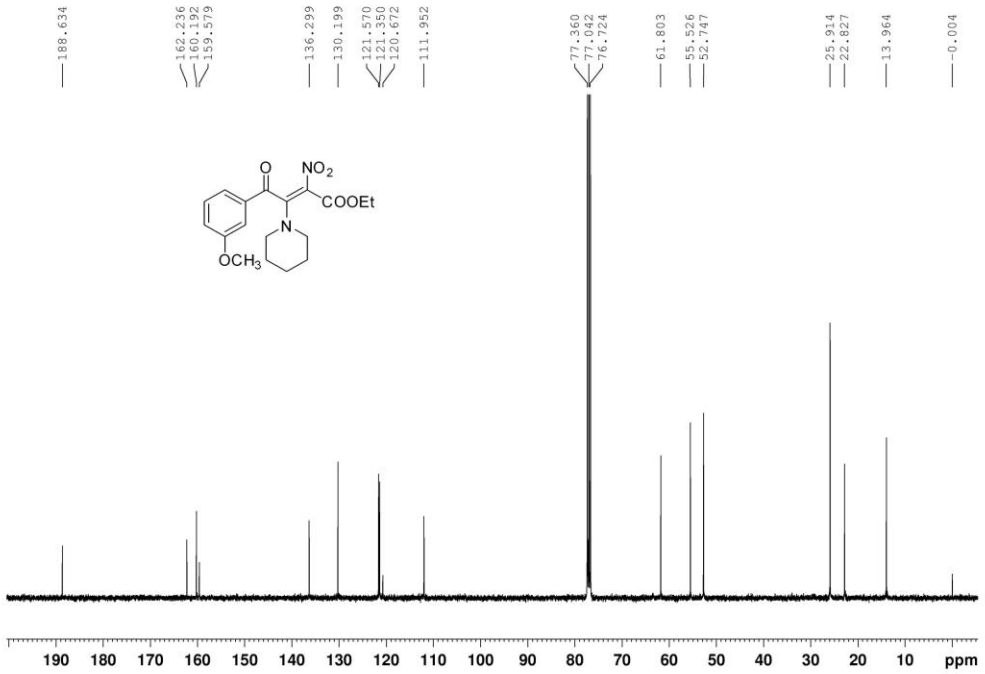


(*E*)-ethyl 4-(3-chlorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ap)

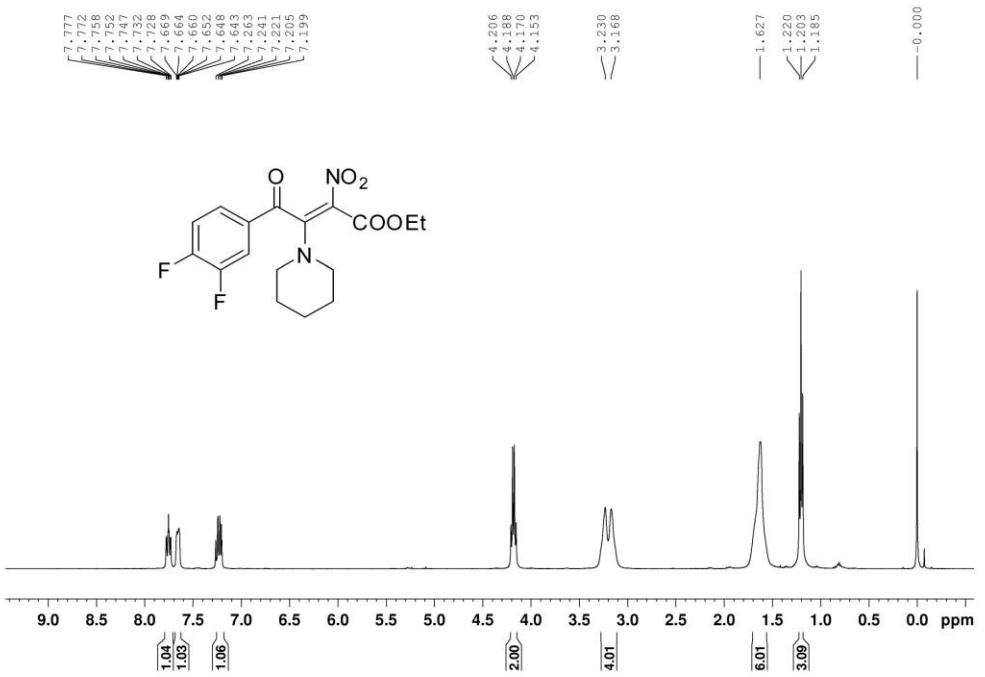


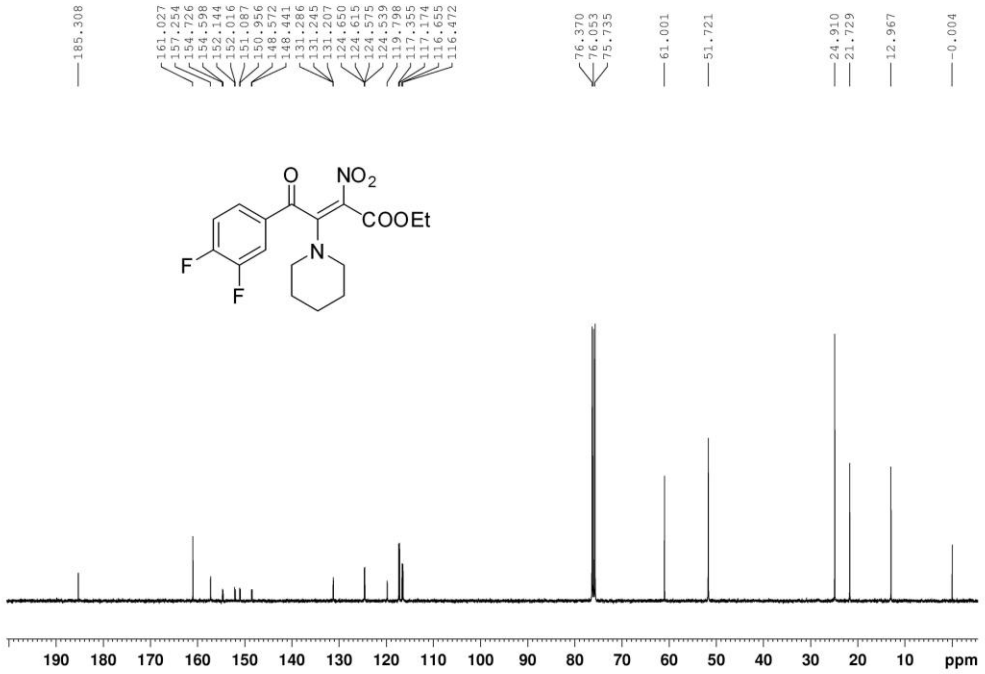
(*E*)-ethyl 4-(3-methoxyphenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4aq)



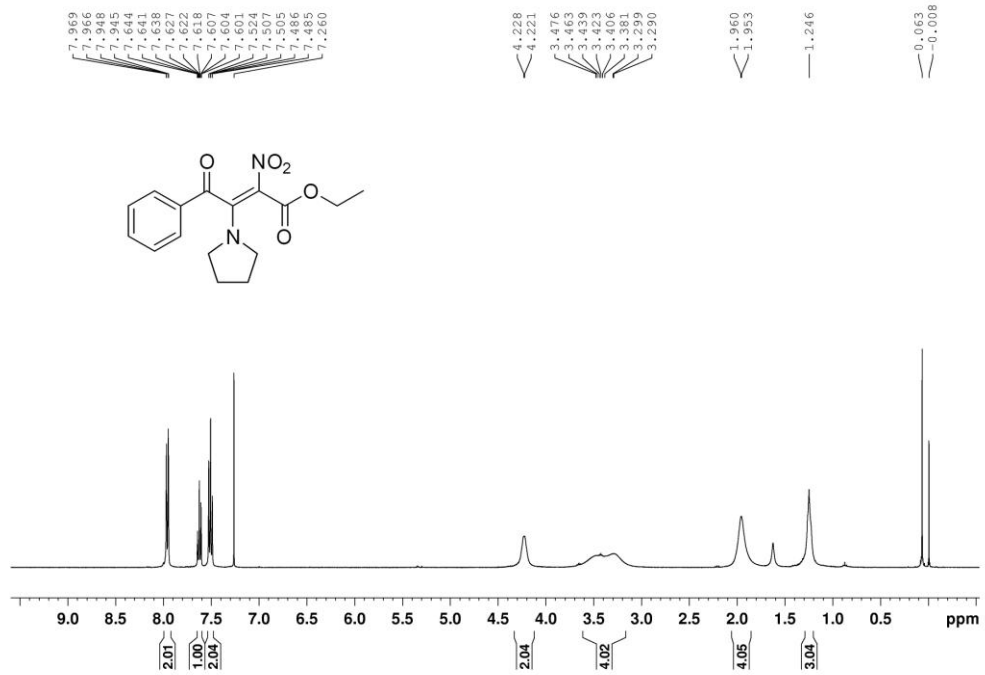


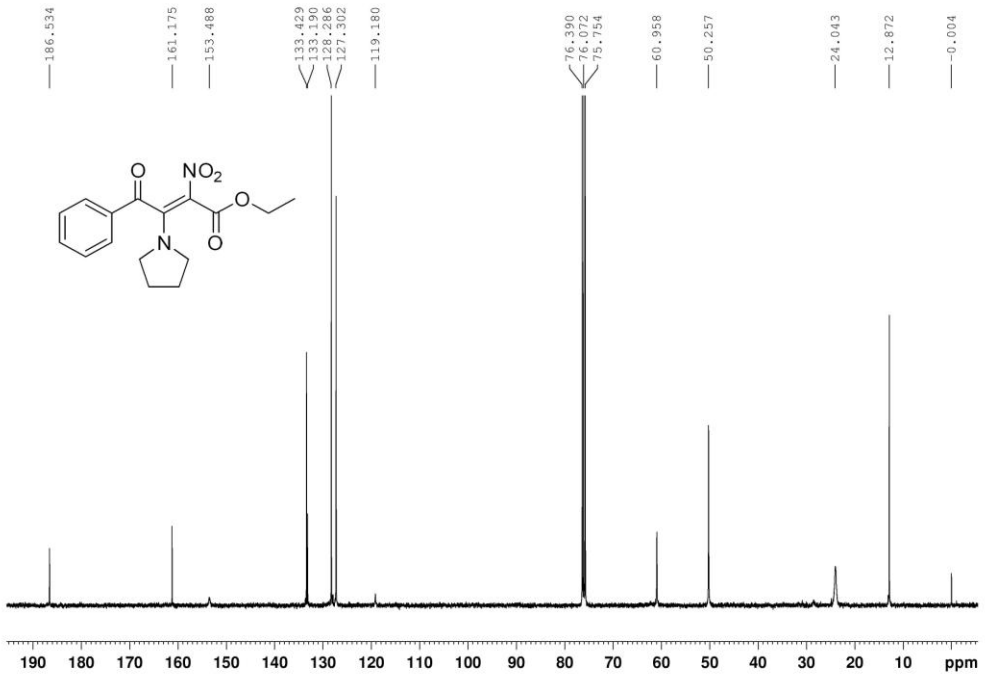
(*E*)-ethyl 4-(3,4-difluorophenyl)-2-nitro-4-oxo-3-(piperidin-1-yl)but-2-enoate (4ar)



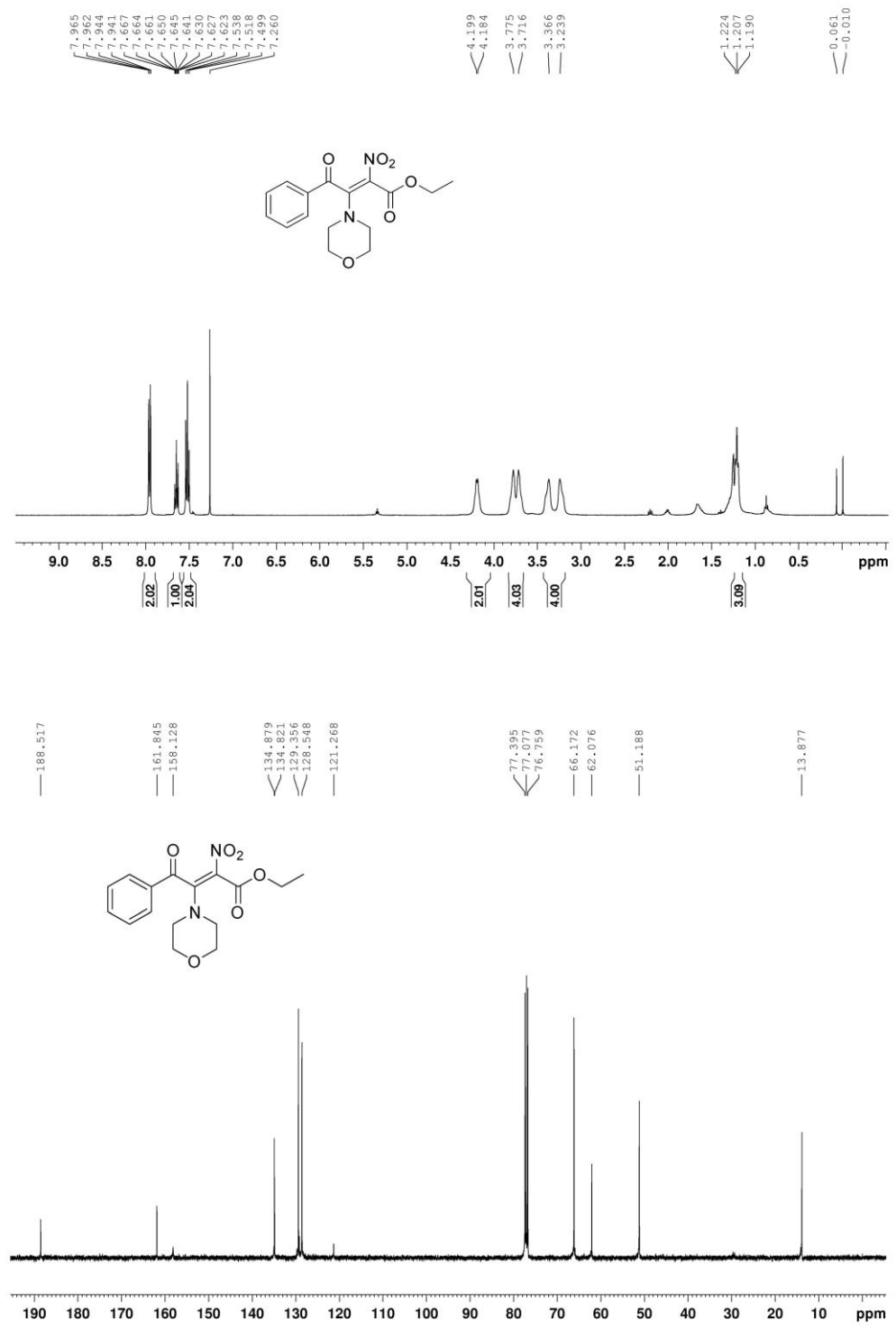


(*E*)-ethyl 2-nitro-4-oxo-4-phenyl-3-(pyrrolidin-1-yl)but-2-enoate (4as)



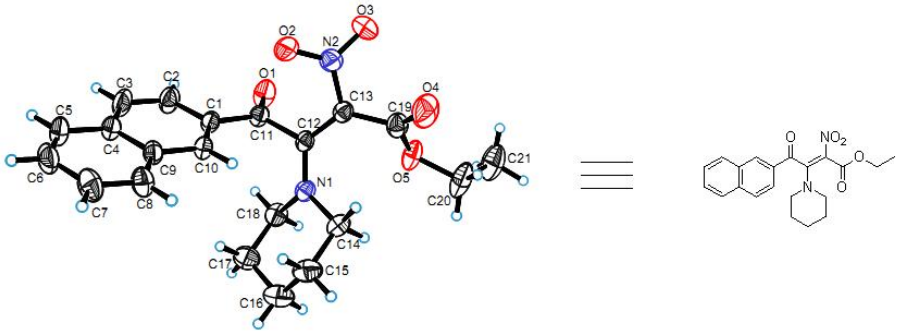


(*E*)-ethyl 3-morpholino-2-nitro-4-oxo-4-phenylbut-2-enoate (4at)



Crystal structure data

A single crystal for X-ray analysis of **4aj** was obtained by recrystallation from acetone/petroleum ether.



CCDC-1473985

Table 1 Crystal data and structure refinement for 4aj.

Identification code	lyn16031801
Empirical formula	$C_{21}H_{22}N_2O_5$
Formula weight	382.40
Temperature/K	290(2)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	14.2787(9)
b/Å	9.4954(4)
c/Å	14.5637(8)
α/°	90
β/°	98.003(5)
γ/°	90
Volume/Å ^{3}	1955.34(18)
Z	4
ρ_{calc}g/cm^3	1.299
μ/mm^{-1}	0.093
F(000)	808.0
Crystal size/mm^3	0.360 × 0.350 × 0.300
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	7.096 to 52.742
Index ranges	$-17 \le h \le 17$, $-10 \le k \le 11$, $-16 \le l \le 18$
Reflections collected	11149
Independent reflections	3993 [R_{int} = 0.0361, R_{sigma} = 0.0473]
Data/restraints/parameters	3993/0/254
Goodness-of-fit on F^2	1.067
Final R indexes [I>=2σ (I)]	R_1 = 0.0575, wR_2 = 0.1511
Final R indexes [all data]	R_1 = 0.0990, wR_2 = 0.2113

Largest diff. peak/hole / e Å $^{-3}$ 0.36/-0.36

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å ^{2}×10^3) for 4aj. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	6562.9(15)	-1690(2)	3129.4(15)	76.7(7)
O5	7315.7(14)	327(2)	479.1(14)	70.6(6)
N1	6210.7(14)	1271(2)	2006.5(14)	47.2(5)
O2	5131.2(18)	-2533(2)	1538(2)	89.6(8)
O3	5994(2)	-3093(2)	504.1(18)	93.6(9)
C1	5046.8(19)	-705(3)	3164.2(17)	48.2(6)
C9	3472.4(19)	291(3)	3104.8(18)	50.6(6)
C12	6082.2(16)	-114(3)	1892.6(17)	42.4(6)
N2	5718.7(18)	-2227(2)	1033.1(17)	58.7(6)
C10	4333.8(18)	129(3)	2736.8(17)	48.1(6)
C4	3345(2)	-451(3)	3921(2)	59.5(7)
C11	5934.7(19)	-931(3)	2773.6(19)	50.5(6)
C13	6102.2(17)	-848(3)	1079.7(18)	46.5(6)
C14	6108(2)	2299(3)	1242(2)	57.3(7)
O4	6183(2)	-680(3)	-526.9(16)	102.6(10)
C5	2466(3)	-319(4)	4265(3)	77.9(10)
C19	6517(2)	-418(3)	251(2)	59.2(7)
C18	6571(2)	1884(3)	2921(2)	62.3(8)
C8	2735(2)	1156(4)	2678(2)	66.9(8)
C2	4919(2)	-1423(3)	3987(2)	70.3(9)
C17	5978(3)	3118(3)	3155(2)	79.5(10)
C6	1776(3)	503(4)	3831(3)	80.2(10)
C15	5504(3)	3534(3)	1479(3)	75.6(9)
C3	4086(3)	-1292(4)	4344(2)	78.1(10)
C7	1905(2)	1253(4)	3037(3)	79.4(10)
C20	7720(3)	1075(5)	-260(3)	106.7(15)
C16	5893(3)	4213(4)	2392(3)	90.5(12)
C21	8606(3)	523(6)	-395(4)	120.9(17)

Table 3 Anisotropic Displacement Parameters (Å ^{2}×10^3) for 4aj. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	57.6(13)	94.5(16)	80.7(15)	39.5(13)	18.7(11)	28.0(12)
O5	47.7(12)	101.7(16)	65.9(13)	28.1(12)	19.9(9)	-0.8(11)
N1	40.9(12)	51.2(13)	49.6(12)	5(1)	6.2(9)	-1.8(9)

O2	84.0(17)	67.1(14)	130(2)	-13.9(14)	58.0(16)	-21.5(12)
O3	135(2)	61.5(14)	93.6(18)	-19.2(13)	47.9(17)	0.1(14)
C1	47.6(15)	51.1(15)	48.2(14)	8.3(12)	14.3(11)	0.8(11)
C9	46.6(15)	57.5(15)	50.2(15)	-5.9(12)	15.6(11)	-4.2(12)
C12	30.2(12)	49.8(14)	48.2(14)	6.5(11)	9.1(10)	1(1)
N2	56.2(15)	53.9(14)	67.6(15)	-0.8(12)	14.0(12)	2.5(11)
C10	45.7(15)	57.3(15)	43.4(14)	5.6(12)	13.3(11)	-0.4(11)
C4	60.4(18)	62.3(17)	60.9(17)	-2.1(14)	26.9(14)	-9.2(14)
C11	44.6(15)	54.5(15)	53.4(15)	8.8(12)	11.0(11)	2.1(12)
C13	38.8(13)	48.2(14)	53.3(15)	6.1(11)	9.6(11)	2.3(11)
C14	57.0(17)	54.0(16)	60.5(17)	11.6(13)	7.1(13)	-6.5(13)
O4	135(3)	125(2)	46.9(14)	5.8(13)	6.6(14)	-28.7(18)
C5	75(2)	88(2)	80(2)	-1.2(19)	45.7(19)	-10.3(19)
C19	61.7(19)	66.2(18)	50.4(17)	10.0(14)	10.2(13)	9.5(14)
C18	60.1(18)	69.0(18)	56.2(17)	-3.1(14)	2.2(13)	-12.6(14)
C8	52.0(18)	87(2)	64.2(18)	0.8(16)	16.6(14)	10.3(15)
C2	72(2)	78(2)	65.9(19)	29.4(16)	25.1(15)	15.3(16)
C17	94(3)	67(2)	78(2)	-17.8(18)	11.5(19)	-11.9(18)
C6	55(2)	95(3)	98(3)	-22(2)	39.6(19)	-8.2(18)
C15	80(2)	52.8(18)	92(2)	8.6(17)	5.9(18)	5.8(16)
C3	90(3)	83(2)	70(2)	28.9(18)	40.6(18)	4.8(19)
C7	50.5(19)	102(3)	89(2)	-12(2)	19.5(16)	12.7(17)
C20	90(3)	140(4)	99(3)	56(3)	45(2)	9(3)
C16	106(3)	55.8(19)	109(3)	-12(2)	11(2)	-1.7(19)
C21	89(3)	164(5)	120(4)	32(3)	52(3)	-15(3)

Table 4 Bond Lengths for 4aj.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C11	1.210(3)	C12	C11	1.539(3)
O5	C19	1.343(4)	N2	C13	1.418(3)
O5	C20	1.472(4)	C4	C3	1.399(5)
N1	C12	1.334(3)	C4	C5	1.420(4)
N1	C14	1.473(3)	C13	C19	1.475(4)
N1	C18	1.478(3)	C14	C15	1.523(4)
O2	N2	1.224(3)	O4	C19	1.192(4)
O3	N2	1.228(3)	C5	C6	1.343(5)
C1	C10	1.369(4)	C18	C17	1.512(5)
C1	C2	1.412(4)	C8	C7	1.365(4)
C1	C11	1.476(4)	C2	C3	1.368(4)
C9	C8	1.410(4)	C17	C16	1.515(5)

C9	C4	1.415(4)	C6	C7	1.392(5)
C9	C10	1.417(4)	C15	C16	1.512(5)
C12	C13	1.378(4)	C20	C21	1.408(6)

Table 5 Bond Angles for 4aj.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C19	O5	C20	118.6(3)	O1	C11	C12	118.6(2)
C12	N1	C14	124.1(2)	C1	C11	C12	118.2(2)
C12	N1	C18	121.6(2)	C12	C13	N2	117.0(2)
C14	N1	C18	113.9(2)	C12	C13	C19	128.5(2)
C10	C1	C2	119.3(3)	N2	C13	C19	114.4(2)
C10	C1	C11	122.2(2)	N1	C14	C15	109.8(2)
C2	C1	C11	118.4(2)	C6	C5	C4	120.8(3)
C8	C9	C4	119.0(3)	O4	C19	O5	124.0(3)
C8	C9	C10	122.0(3)	O4	C19	C13	124.5(3)
C4	C9	C10	118.9(3)	O5	C19	C13	111.5(2)
N1	C12	C13	125.9(2)	N1	C18	C17	111.9(3)
N1	C12	C11	115.2(2)	C7	C8	C9	120.4(3)
C13	C12	C11	118.9(2)	C3	C2	C1	120.0(3)
O2	N2	O3	121.6(3)	C18	C17	C16	110.9(3)
O2	N2	C13	118.9(2)	C5	C6	C7	121.0(3)
O3	N2	C13	119.5(2)	C16	C15	C14	112.2(3)
C1	C10	C9	121.4(2)	C2	C3	C4	121.9(3)
C3	C4	C9	118.4(3)	C8	C7	C6	120.4(3)
C3	C4	C5	123.2(3)	C21	C20	O5	111.9(3)
C9	C4	C5	118.4(3)	C15	C16	C17	109.5(3)
O1	C11	C1	123.1(2)				

Table 6 Torsion Angles for 4aj.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C14	N1	C12	C13	19.5(4)	C12	N1	C14	C15	132.3(3)
C18	N1	C12	C13	-153.9(3)	C18	N1	C14	C15	-53.8(3)
C14	N1	C12	C11	-162.7(2)	C3	C4	C5	C6	-179.9(3)
C18	N1	C12	C11	23.8(3)	C9	C4	C5	C6	-0.7(5)
C2	C1	C10	C9	0.3(4)	C20	O5	C19	O4	11.1(5)
C11	C1	C10	C9	177.6(2)	C20	O5	C19	C13	-168.1(3)
C8	C9	C10	C1	179.4(3)	C12	C13	C19	O4	-143.1(3)
C4	C9	C10	C1	-1.3(4)	N2	C13	C19	O4	40.2(4)
C8	C9	C4	C3	-179.3(3)	C12	C13	C19	O5	36.0(4)
C10	C9	C4	C3	1.4(4)	N2	C13	C19	O5	-140.7(2)

C8	C9	C4	C5	1.6(4)	C12	N1	C18	C17	-131.6(3)
C10	C9	C4	C5	-177.8(3)	C14	N1	C18	C17	54.3(3)
C10	C1	C11	O1	-178.2(3)	C4	C9	C8	C7	-1.3(5)
C2	C1	C11	O1	-0.8(4)	C10	C9	C8	C7	178.0(3)
C10	C1	C11	C12	4.3(4)	C10	C1	C2	C3	0.6(5)
C2	C1	C11	C12	-178.3(3)	C11	C1	C2	C3	-176.8(3)
N1	C12	C11	O1	-108.3(3)	N1	C18	C17	C16	-54.3(4)
C13	C12	C11	O1	69.6(3)	C4	C5	C6	C7	-0.4(5)
N1	C12	C11	C1	69.3(3)	N1	C14	C15	C16	55.3(4)
C13	C12	C11	C1	-112.7(3)	C1	C2	C3	C4	-0.5(6)
N1	C12	C13	N2	-164.4(2)	C9	C4	C3	C2	-0.5(5)
C11	C12	C13	N2	17.8(3)	C5	C4	C3	C2	178.6(3)
N1	C12	C13	C19	18.9(4)	C9	C8	C7	C6	0.2(5)
C11	C12	C13	C19	-158.8(3)	C5	C6	C7	C8	0.7(5)
O2	N2	C13	C12	23.9(4)	C19	O5	C20	C21	-114.6(4)
O3	N2	C13	C12	-154.7(3)	C14	C15	C16	C17	-56.8(4)
O2	N2	C13	C19	-159.0(3)	C18	C17	C16	C15	55.5(4)
O3	N2	C13	C19	22.4(4)					

Table 7 Hydrogen Atom Coordinates (Å $\times 10^4$) and Isotropic Displacement Parameters (Å $^2 \times 10^3$) for 4aj.

Atom	*x*	*y*	*z*	U(eq)
H10	4417	599	2194	58
H14A	5812	1851	676	69
H14B	6726	2635	1139	69
H5	2367	-807	4797	94
H18A	7218	2193	2920	75
H18B	6571	1166	3395	75
H8	2816	1664	2148	80
H2	5401	-1985	4285	84
H17A	6265	3541	3732	95
H17B	5352	2789	3239	95
H6	1204	571	4065	96
H15A	5474	4232	989	91
H15B	4866	3207	1510	91
H3	4011	-1777	4883	94
H7	1421	1824	2750	95
H19A	7288	1003	-834	128
H19B	7790	2064	-99	128
H16A	5476	4965	2532	109

H16B 6510	4617	2349	109
H20A 8864	1074	-852	181
H20B 8530	-434	-604	181
H20C 9027	553	179	181

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait
checkCIF / PLATON (basic structural check)

Structure factors have been supplied for datablock(s) shelxl
THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.
No syntax errors found. CIF dictionary
Please wait while processing Interpreting this report
Structure factor report

Datablock: shelxl

Bond precision: C-C = 0.0047 A Wavelength = 0.71073
Cell: a = 14.2787(9) b = 9.4954(4) c = 14.5637(8)
alpha = 90 beta = 98.003(5) gamma = 90
Temperature: 290 K

	Calculated	Reported
Volume	1955.34(18)	1955.34(18)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	-P 2yn
Moiety formula	C21 H22 N2 O5	?
Sum formula	C21 H22 N2 O5	C21 H22 N2 O5
Mr	382.41	382.40
Dx,g cm-3	1.299	1.299
Z	4	4
Mu (mm-1)	0.093	0.093
F000	808.0	808.0
F000'	808.41	
h, k, lmax	17,11,18	17,11,18
Nref	4000	3993

Tmin, Tmax 0.967,0.972 0.884,1.000

Tmin' 0.967

Correction method = # Reported T Limits: Tmin = 0.884 Tmax = 1.000 AbsCorr = MULTI-SCAN

Data completeness = 0.998 Theta(max) = 26.371

R(reflections) = 0.0575(2582) wR2(reflections) = 0.2113(3993)

S = 1.067 Npar = 254

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

PLAT934_ALERT_3_B Number of (Iobs-Icalc)/SigmaW > 10 Outliers 6 Check

---

Alert level C

PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of N2 Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds 0.00465 Ang.
PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C20 - C21 1.41 Ang.
PLAT369_ALERT_2_C Long C(sp2)-C(sp2) Bond C11 - C12 1.54 Ang.
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min) 8 Note
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) 5 Check
PLAT939_ALERT_3_C Large Value of Not (SHELXL) Weight Optimized S 13.91

Alert level G

PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density 1 Note

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

0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
4 ALERT type 2 Indicator that the structure model may be wrong or deficient
5 ALERT type 3 Indicator that the structure quality may be low
0 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.
start Validation Reply Form
_vrf_PLAT242_shelxl
;

PROBLEM: Low 'MainMol' Ueq as Compared to Neighbors of N2 Check
RESPONSE: ...
;
_vrf_PLAT340_shelxl
;
PROBLEM: Low Bond Precision on C-C Bonds ..... 0.00465 Ang.
RESPONSE: ...
;
_vrf_PLAT360_shelxl
;
PROBLEM: Short C(sp3)-C(sp3) Bond C20 - C21 1.41 Ang.
RESPONSE: ...
;
_vrf_PLAT369_shelxl
;
PROBLEM: Long C(sp2)-C(sp2) Bond C11 - C12 .. 1.54 Ang.
RESPONSE: ...
;
_vrf_PLAT910_shelxl
;
PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min) 8 Note
RESPONSE: ...
;
_vrf_PLAT918_shelxl
;
PROBLEM: Reflection(s) with I(obs) much Smaller I(calc) . 5 Check
RESPONSE: ...
;
_vrf_PLAT939_shelxl
;
PROBLEM: Large Value of Not (SHELXL) Weight Optimized S 13.91
RESPONSE: ...
;
end Validation Reply Form

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

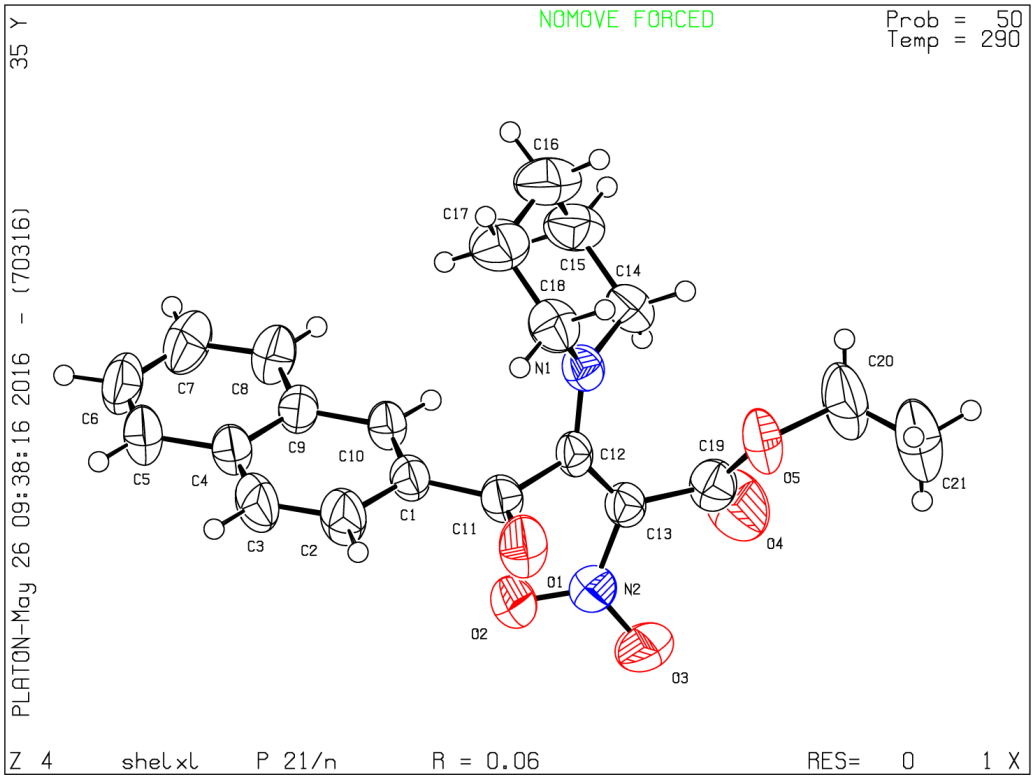
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 06/05/2016; check.def file version of 05/05/2016

Datablock shelxl - ellipsoid plot



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