Supporting Information for

Efficient Palladium-Catalyzed Cross-Coupling of β-chloro-alkylidene/arylidene Malonates using Microwave Chemistry

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General experimental information:

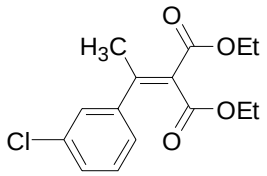
Unless otherwise noted, all materials were obtained from commercial suppliers and used without further purification. THF was dried and distilled from sodium/benzophenone. K_2CO_3 was purchased dry from commercial suppliers and dried under vacuum at 120 ^{0}C for 24h.Thin-Layer Chromatography was performed on precoated plates, silica gel 60 F_{254} and visualizing with ultraviolet light and iodine spray. Flash chromatography on silica gel. All melting points are uncorrected. Infrared spectra were observed as KBr pellets or neat. All ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ and DMSO-d_6 solutions using the residual solvent peak as internal reference. Spin multiplicities are given as s (singlet), d (doublet), t (triplet), q (quartet), and m (multiplet) as well as b (broad). Coupling constants (J) are given in hertz. Mass spectra Electro spray and HRMS were obtained.

All microwave irradiation experiments were carried out using the Explorer PLS.™ The reactions were performed in heavy-walled Pyrex tubes (10 ml, 1= 150 mm) sealed with a septum utilizing the standard absorbance level (300 W maximum power). The reaction volume filled not more than ¼th of the total volume of the tube. All couplings and other reactions were conducted in the presence of stirring and external cooling.

General Procedure for the Reaction of β-chloro-arylidene/alkylidenemalonates with arylboronic acids:

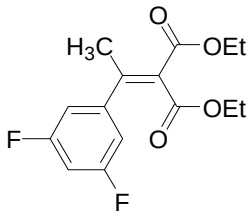
POPd (1mol %), β-Chloro-alkylidene/arylidenemalonate (1.00 mmol), arylboronic acid (1.50 to 2.00 mmol), and K_2CO_3 (3.00 mmol) were weighed in a microwave tube, equipped with a magnetic stirrer bar, and sealed with a silicon septum. THF (2 to 3 mL) was injected into the tube *via* a syringe and the reaction mixture was subjected to microwave irradiation for 30 min at 100 ^{0}C.The reaction vessel was allowed to cool to room temperature and the crude reaction mixture transferred to a separating funnel and diluted with hexane (50mL) and H_2O (15mL). The layers were separated, the organic layer was washed with H_2O (20 mL) and brine (20 mL), dried over $MgSO_4$, and filtered, and solvents were removed from the filtrate by rotary evaporation. The resulting residue was chromatographed on silica gel using ethyl acetate/hexane as eluant.

2-[1-(3-Chlorophenyl) ethylidene] malonic acid diethyl ester (Entry 1):



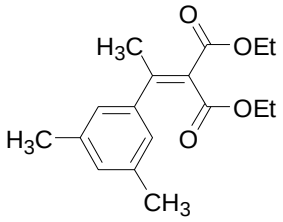
2-[1-(3-Chlorophenyl) ethylidene] malonic acid diethyl ester was prepared using the general procedure. POPd (12 mg, 0.023 mmol, 1% mol), 2-(1-Chloro-ethylidene)-malonic acid diethyl ester (0.5 g, 2.26 mmol), 3-chlorophenyl boronic acid (0.532 g, 3.40 mmol), and K_2CO_3 (0.940 g, 6.80 mmol) in THF (3mL) yielded 0.470 g (70% yield) of the title product as a colorless liquid. R_f = 0.5 (hexane/ethyl acetate = 9:1); IR (Neat): 2982, 1725, 1628, 1563, 1473, 1445, 1228 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$): δ 7.45 (m, 3H), 7.31 (s, 1H), 4.49 (q, *J* = 7.14 Hz, 2H), 4.20 (q, *J* = 7.15 Hz, 2H), 2.59 (s, 3H), 1.51 (t, *J* = 7.13 Hz, 3H), 1.21 (t, *J* = 7.13 Hz, 3H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 165.5, 164.4, 153.5, 143.1, 134.1, 129.5, 128.3, 126.7, 124.7, 61.8, 22.5, 13.9. HRMS (EI$^+$): m/z calculated for $C_{15}H_{17}ClO_4$, 296.0815; found, 296.0813.

2-[1-(3, 5-Difluoro phenyl) ethylidene] malonic acid diethyl ester (Entry 2):



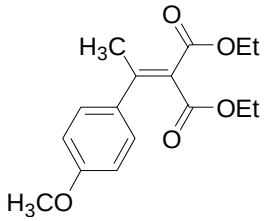
2-[1-(3,5-Difluoro phenyl) ethylidene] malonic acid diethyl ester was prepared using the general procedure. POPd, (11.3 mg, 0.02 mmol, 1 % mol), 2-(1-Chloro-ethylidene)-malonic acid diethyl ester (0.5 g, 2.26 mmol), 3,5-fluorophenyl boronic acid (0.716 g, 4.53 mmol), and K_2CO_3 (0.940g, 6.80 mmol) in THF (3mL) yielded 0.4 g (59 % yield) of the title product as a colorless liquid. R_f = 0.4 (hexane/ethyl acetate = 8:2); IR (Neat): 2984, 1726, 1641, 1620, 1590, 1492, 1446, 1236 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 7.25 (m, 2H), 7.11 (m, 1H), 4.55 (q, *J* = 7.12 Hz, 2H), 4.25 (q, *J* = 7.12 Hz, 2H), 2.57 (s, 3H), 1.58 (t, *J* = 7.11 Hz, 3H), 1.28 (t, *J* = 7.12 Hz, 3H). HRMS (EI^+): m/z calculated for $C_{15}H_{16}F_2O_4$, 298.1017; found, 298.1020.

2-[1-(3, 5-Dimethyl phenyl) ethylidene] malonic acid diethyl ester (Entry 3):



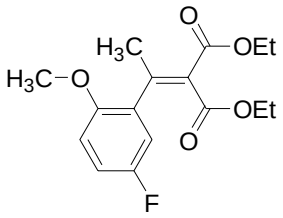
2-[1-(3,5-Dimethyl phenyl) ethylidene] malonic acid diethyl ester was prepared using the general procedure. POPd, (6 mg, 0.011 mmol, 1 % mol), 2-(1-Chloro-ethylidene)-malonic acid diethyl ester (0.250 g, 1.13 mmol), 3,5-methylphenyl boronic acid (0.255 g, 1.70 mmol), and K_2CO_3 (0.470 g, 3.40 mmol) in THF (2mL) yielded 0.2 g (60 % yield) of the title product as a colorless liquid. R_f = 0.5 (hexane/ethyl acetate = 9:1); IR (Neat): 2981, 1724, 1623, 1445, 1266, 1223 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 7.11 (s, 1H), 7.01 (s, 2H), 4.43 (q, *J* = 7.12 Hz, 2H), 4.15 (q, *J* = 7.13 Hz, 2H), 2.56 (s, 3H), 2.45 (s, 6H) 1.47 (t, *J* = 7.11 Hz, 3H), 1.15 (t, *J* = 7.12 Hz, 3H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 166.2, 164.6, 156.0, 141.4, 137.6, 129.9, 125.7, 124.2, 60.8, 22.6, 13.9. HRMS (EI^+): m/z calculated for $C_{17}H_{12}O_4$, 290.1518; found, 290.1520.

2-[1-(4-Methoxyphenyl)-ethylidene]-malonic acid diethyl ester (Entry 4):



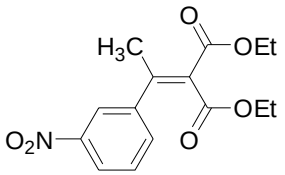
2-[1-(4-Methoxyphenyl)-ethylidene]-malonic acid diethyl ester was prepared using the general procedure. POPd, (6 mg, 0.011 mmol, 0.1 % mol), 2-(1-Chloro-ethylidene)-malonic acid diethyl ester (0.250 g, 1.13 mmol), 4-methoxyphenyl boronic acid (0.258 g, 1.70mmol), and K_2CO_3 (0.470 g, 3.40 mmol) in THF (3mL) yielded 0.210 g (65% yield) of the title product as a colorless liquid. R_f = 0.5 (hexane/ethyl acetate = 8:2). ^{1}H NMR (250 MHz, $CDCl_3$): δ 7.13 (d, J = 8.48 Hz, 2H), 6.78 (d, *J* = 8.57 Hz, 2H), 4.50 (q, *J* = 7.10 Hz, 2H), 3.94 (q, J = 7.10 Hz, 2H), 3.73 (s, 3H), 2.35 (s, 3H), 1.25 (t, *J* = 7.11 Hz, 3H), 0.97 (t, J = 7.12 Hz, 3H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 166.5, 164.7, 159.7, 155.4, 133.6, 128.1, 125.4, 113.5, 60.8, 55.1, 22.6, 13.9. MS (ES^+): m/z (relative intensity) 292(M+, 15), 260 (15), 172 (20), 110 (55), 97 (100), 80 (15).

2-[1-(5-Fluoro-2-methoxyphenyl) ethylidene]malonic acid diethyl ester (Entry 5):



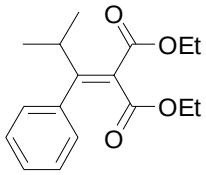
2-[1-(5-Fluoro-2-methoxyphenyl) ethylidene] malonic acid diethyl ester was prepared using the general procedure. POPd, (11.5 mg, 0.02 mmol, 1 % mol), 2-(1-Chloro-ethylidene)-malonic acid diethyl ester (0.5 g, 2.26 mmol), 2-methoxy-5-fluorophenyl boronic acid (0.770g, 4.53 mmol), and K_2CO_3 (0.940g, 6.80 mmol) in THF (3mL) yielded 0.280 g (66 % yield) of the title product as a colorless liquid. R_f = 0.5 (hexane/ethyl acetate = 9:1); IR (Neat): 2982, 1725, 1637, 1610, 1594, 1465 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$): δ 7.27 (m, 3H), 4.55 (q, *J* = 7.11 Hz, 2H), 4.27 (q, *J* = 7.15 Hz, 2H), 4.06 (s, 3H), 2.60 (s, 3H), 1.57 (t, *J* = 7.15 Hz, 3H), 1.23 (t, J = 7.14 Hz, 3H). HRMS (EI^+): m/z calculated for $C_{16}H_{19}FO_5$, 310.1217; found 310.1216.

2-[-(3-Nitrophenyl) ethylidene] malonic acid diethyl ester (Entry 6):



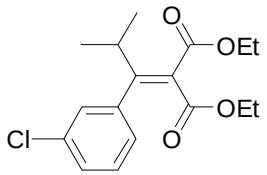
2-[-(3-Nitrophenyl) ethylidene] malonic acid diethyl ester was prepared using the general procedure. POPd, (11.3 mg, 0.02 mmol, 1 % mol), 2-(1-Chloro-ethylidene)-malonic acid diethyl ester (0.5 g, 2.26 mmol), 3-nitrophenyl boronic acid (0.757g, 4.53 mmol), and K_2CO_3 (0.940g, 6.80 mmol) in THF (3mL) yielded 0.3 g (72 % yield) of the title product as a colorless liquid. R_f = 0.3 (hexane/ethyl acetate = 9:1); IR (Neat): 2984, 1724, 1631, 1574, 1474, 1446 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 8.12 (m, 1H), 8.05 (s, 1H), 7.48 (m, 2H), 4.25 (q, *J* = 7.17 Hz, 2H), 3.92 (q, *J* = 7.13 Hz, 2H), 2.33 (s, 3H), 1.29 (t, *J* = 7.09 Hz, 3H), 0.97 (t, *J* = 7.11 Hz, 3H). HRMS (EI^+): m/z calculated for $C_{15}H_{17}NO_6$, 307.1056; found, 307.1049.

2-(2-Methyl-1-phenylpropylidene) malonic acid diethyl ester (Entry 7):



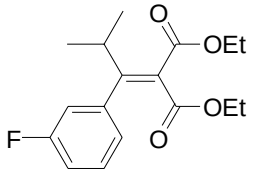
2-(2-Methyl-1-phenylpropylidene) malonic acid diethyl ester was prepared using the general procedure. POPd, (10 mg, 0.012 mmol, 1 % mol), 2-(1-Chloro-2-methyl propylidene) malonic acid diethyl ester (0.5g, 2.0 mmol), 3-phenyl boronic acid (0.294 g, 2.40 mmol), and K_2CO_3 (0.834 g, 6.03 mmol) in THF (3mL) yielded 0.250 g (71 % yield) of the title product as a colorless liquid. R_f = 0.4 (hexane/ethyl acetate = 9:1). ^1H NMR (250 MHz, $CDCl_3$): δ 7.37 (m, 3H), 7.12 (m, 2H), 4.40 (q, *J* = 7.14 Hz, 2H), 3.94 (q, *J* = 7.17 Hz, 2H), 3.57 (m, 1H), 1.40 (t, *J* = 7.14 Hz, 3H), 1.07 (d, *J* = 6.84 Hz, 6H), 0.99 (t, *J* = 7.14 Hz, 3H). MS (ES^+): m/z (relative intensity) 313 (M+ Na, 100), 240 (5), 163 (25).

2-[1-(3-Chlorophenyl)-2-methylpropylidene] malonic acid diethyl ester (Entry 8):



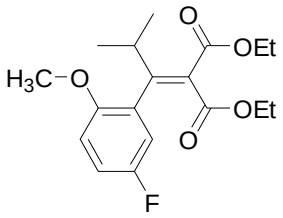
2-[1-(3-Chlorophenyl)-2-methylpropylidene] malonic acid diethyl ester was prepared using the general procedure. POPd, (6 mg, 0.012 mmol, 1% mol), 2-(1-Chloro-2-methyl propylidene) malonic acid diethyl ester (0.3g, 1.20 mmol), 3-chlorophenyl boronic acid (0.283 g, 0.181 mmol) K_2CO_3 (0.5 g, 3.62 mmol) in THF (2mL) yielded (0.15 g, 57% yield) of the title product as a colorless liquid. IR (Neat): 2978, 1726, 1624, 1563, 1466, 1446 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 7.45 (m, 2H), 7.26 (m, 1H), 7.14 (m, 1H), 4.49 (q, *J* = 3.45 Hz, 2H), 4.07 (q, *J* = 7.14 Hz, 2H), 3.60 (m, 1H), 1.52 (t, *J* = 3.74 Hz, 3H), 1.20 (s, 3H), 1.87 (s, 3H), 1.13 (t, *J* = 7.14 Hz, 3H); HRMS (EI^+): m/z calculated for $C_{17}H_{21}ClO_4$, 324.1128; found, 324.1297.

2-[1-(3-Fluoro phenyl)-2-methylpropylidene] malonic acid diethyl ester(Entry 9):



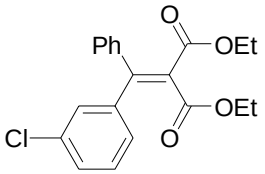
2-[1-(3-Fluoro phenyl)-2-methyl propylidene] malonic acid diethyl ester was prepared using the general procedure. POPd, (10 mg, 0.02 mmol, 1 % mol), 2-(1-Chloro-2-methyl propylidene) malonic acid diethyl ester (0.5 g, 2.0 mmol), 3-fluorophenyl boronic acid (0.337 g, 2.40 mmol), and K_2CO_3 (0.834 g, 6.0 mmol) in THF (3mL) yielded 0.340 g (55 % yield) of the title product as a colorless liquid. R_f = 0.6 (hexane/ethyl acetate = 9:1). ^1H NMR (250 MHz, $CDCl_3$): δ 7.28 (m, 1H), 7.02 (m, 1H), 6.84 (m, 2H), 4.30 (q, *J* = 7.13 Hz, 2H), 3.92 (q, *J* = 7.13 Hz, 2H), 3.46 (m, 1H), 1.04 (d, *J* = 7.13 Hz, 6H), 0.96 (t, *J* = 7.14 Hz, 3H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 164.8, 163.8, 161.4, 159.9, 138.3, 129.0, 126.1, 123.6, 115.3, 61.2, 31.9, 20.5, 13.9. MS (ES^+): m/z (relative intensity) 331(M+, +Na, 5), 300 (40), 198 (18), 130 (100), 73 (10), 61(50).

2-[1-(5-Fluoro-2-methoxyphenyl)-2-methylpropylidene] malonic acid diethyl ester (Entry 10):



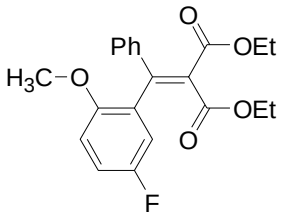
2-[1-(5-Fluoro-2-methoxyphenyl)-2-methylpropylidene] malonic acid diethyl ester was prepared using the general procedure. POPd, (10 mg, 0.019 mmol, 1 % mol), 2-(1-Chloro-2-methyl propylidene) malonic acid diethyl ester (0.5 g, 2.0 mmol), 2-methoxy-5-fluorophenyl boronic acid (0.684 g, 4.0 mmol), and K_2CO_3 (0.830 g, 6.0 mmol) in THF (3mL) yielded 0.250 g (52 % yield) of the title product as a colorless liquid. R_f = 0.5 (hexane/ethyl acetate = 9:1); IR (Neat): 2979, 1726, 1628, 1607, 1495, 1465 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 7.18 (m, 1H), 6.98 (m, 1H), 6.88 (m, 1H), 4.51 (m, 2H), 4.11 (q, *J* = 7.16 Hz, 2H), 3.96 (s, 3H), 3.57 (m, 1H), 1.35 (t, *J* = 7.14 Hz, 3H), 1.21 (m, 9H). HRMS (EI^+): m/z calculated for $C_{18}H_{23}FO_5$, 338.1530; found, 338.1528.

2-[(3-Chloro-phenyl)-phenylmethylene] malonic acid diethyl ester (Entry 11):



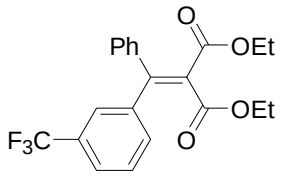
2-[(3-Chloro-phenyl)-phenylmethylene] malonic acid diethyl ester was prepared using the general procedure. POPd, (3.5 mg, 0.0069 mmol, 1% mol), 2-(Chloro-phenyl methylene) malonic acid diethyl ester (0.2 g, 0.707mmol), 3-chlorophenyl boronic acid (0.166 g, 1.06 mmol), and K_2CO_3 (0.293 g, 2.12 mmol) in THF (2mL) yielded 0.14 g (55% yield) of the title product as a colorless liquid. R_f = 0.5 (hexane/ethyl acetate = 9:1); IR (Neat): 2982, 1728, 1591, 1472, 1444 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 7.18 (m, 9H), 4.04(m, 4H), 0.97(m, 6H). HRMS (EI^+): m/z calculated for $C_{20}H_{19}ClO_4$, 358.0972; found, 358.0975.

2-[(5-Fluoro-2-methoxyphenyl) phenylmethylene] malonic acid diethyl ester (Entry 12):



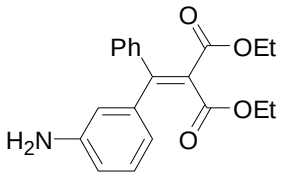
2-[(5-Fluoro-2-methoxyphenyl) phenylmethylene] malonic acid diethyl ester was prepared using the general procedure. POPd, (9 mg, 0.017 mmol 1%mol), 2-(Chloro-phenyl methylene) malonic acid diethyl ester (0.5 g, 1.76), 2-methoxy-5-fluorophenyl boronic acid (0.601 g, 3.53 mmol), and K_2CO_3 (0.735 g, 5.31 mmol) in THF (3mL) yielded 0.2 g (50 % yield) of the title product as a colorless liquid. R_f = 0.5 (hexane/ethyl acetate = 8.5:1.5); IR (Neat): 2982, 1728, 1619, 1591, 1494 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 7.17 (m, 6H), 6.72 (m, 2H), 3.97 (m, 4H), 3.59 (s, 3H), 1.01 (m, 6H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 166.0, 164.6, 158.4, 154.6, 152.5, 150.5, 139.5, 130.9, 128.8, 128.1, 126.7, 116.6, 112.1, 60.2, 56.1, 13.6. HRMS (EI^+): m/z calculated for $C_{21}H_{21}FO_5$, 372.1373; found 372.1368.

2-[phenyl- (3-trifluoromethyl-phenyl)-methylene] malonic acid diethyl ester (Entry 13):



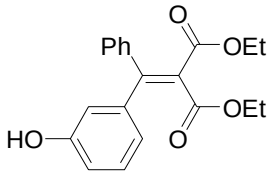
2-[phenyl- (3-trifluoromethyl-phenyl)-methylene] malonic acid diethyl ester was prepared using the general procedure. POPd, (3.5 mg, 0.0070 mmol, 1% mol), 2-(Chloro-phenyl methylene) malonic acid diethyl ester (0.2 g, 0.707 mmol), 3-trifluorophenyl boronic acid (0.161 g, 0.84 mmol), and K_2CO_3 (0.29 g, 2.12 mmol) in THF (2mL) yielded 0.19 g (68% yield) of the title product as a pale brown solid. R_f = 0.3 (hexane/ethyl acetate = 9.5:0.5), mp = 58-59.5 ^0C. IR (KBr): 2980, 1721, 1698, 1604, 1444 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 7.64 (m, 9H), 4.27(m, 4H), 1.21(m, 6H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 153.2, 140.1, 138.5, 131.8, 128.9, 128.4, 128.1, 127.7, 125.1, 76.9, 76.4, 75.9, 60.9, 12.9. HRMS (FAB): m/z calculated for $C_{21}H_{19}F_3O_4$, 392.1235; found, 392.1346.

2-[(3-Amino-phenyl)-phenylmethylene] malonic acid diethyl ester (Entry 14):

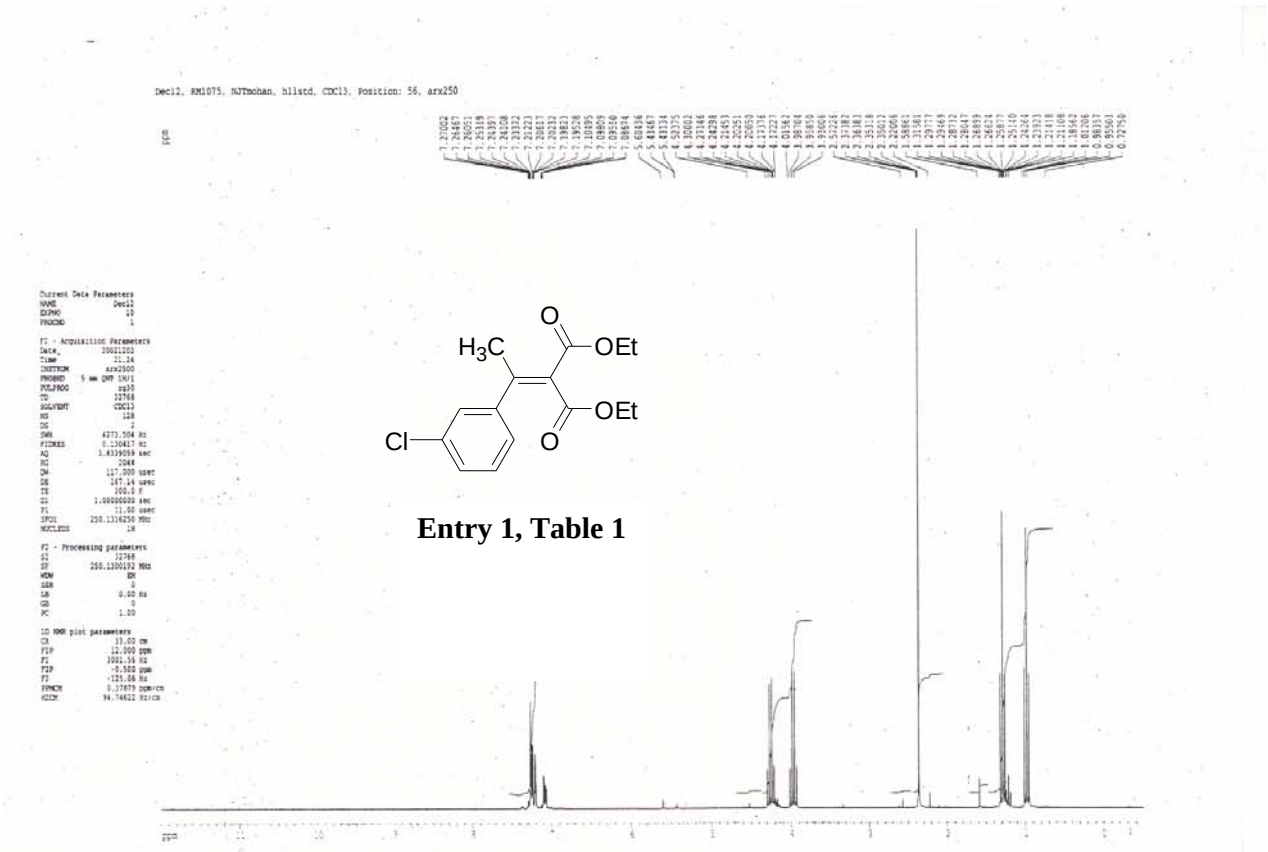


2-[(3-Amino-phenyl)-phenylmethylene] malonic acid diethyl ester was prepared using the general procedure. POPd, (9 mg, 0.017 mmol, 1% mol), 2-(Chloro-phenyl methylene) malonic acid diethyl ester (0.5g, 1.76 mmol), 3-aminophenyl boronic acid (0.493 g, 2.65 mmol), and K_2CO_3 (0.733 g, 5.30 mmol) in THF (3mL) yielded 0.250 g (60% isolated yield) of the title product as a light brown solid. R_f = 0.5 (hexane/ethyl acetate = 7:3), mp = 73-74 ^{0}C. IR (KBr): 3462, 3372, 2988, 1731, 1597, 1488, 1445 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$): δ 7.14 (m, 6H), 6.46(m, 2H), 6.37(s, 1H), 4.00(m, 4H), 3.56(bs, 2H), 0.95 (m, 6H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 166.0, 165.8, 155.7, 146.0, 141.0, 139.8, 128.9, 127.9, 125.9, 119.3, 115.7, 115.4, 61.0, 13.5. HRMS (FAB): m/z calculated for $C_{20}H_{21}NO_4$, 339.1471; found, 339.1468.

2-[(3-Hydroxy-phenyl)-phenyl methylene] malonic acid diethyl ester (Entry 15):



2-[(3-Hydroxy-phenyl)-phenyl methylene] malonic acid diethyl ester was prepared using the general procedure. POPd, (9 mg, 0.017 mmol, 1% mol), 2-(Chloro-phenyl methylene) malonic acid diethyl ester (0.5 g, 1.76 mmol), 3-hydroxyphenyl boronic acid (0.366 g, 2.65 mmol), and K_2CO_3 (0.733 g, 5.30 mmol) in THF (3mL) yielded 0.380 g (63% yield) of the title product as a white colour solid. R_f = 0.5 (hexane/ethyl acetate = 7:3), mp = 94-95.5 ^{0}C. IR (KBr): 3389, 2989, 1707, 1673, 1578, 1444 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$): δ 7.32 (m, 6H), 6.66 (m, 2H), 6.43(s, 1H), 5.70(bs, 1H), 4.14(m, 2H), 1.10(m, 6H). ^{13}C NMR (63 MHz, $CDCl_3$): δ 166.3, 155.6, 141.2, 139.6, 129.2, 129.1, 129.0, 128.0, 121.2, 116.3, 61.2, 13.5. HRMS (FAB): m/z calculated for $C_{20}H_{20}O_5$, 340.1311; found, 340.1374.


Dec12, RM1075, NJTmohan, h1std, CDCl3, Position: 56, arx250
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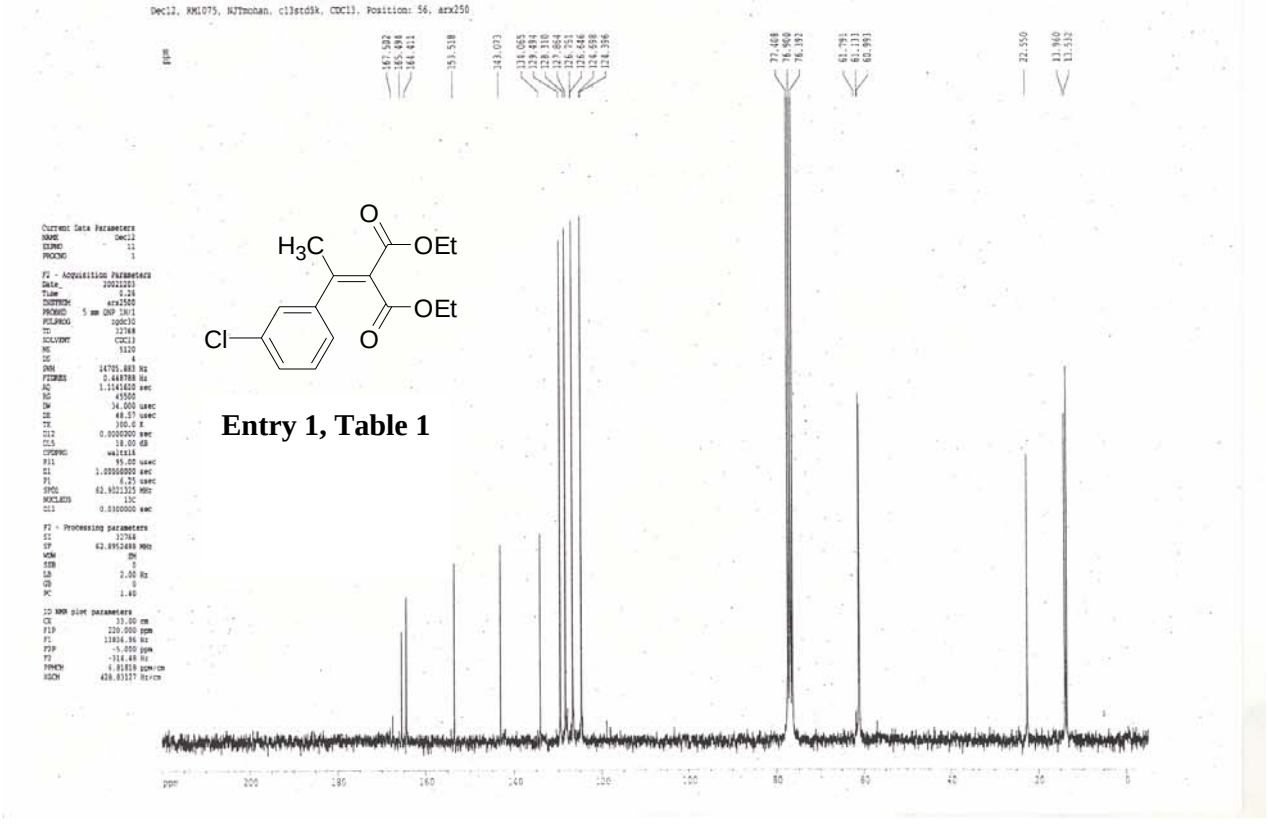
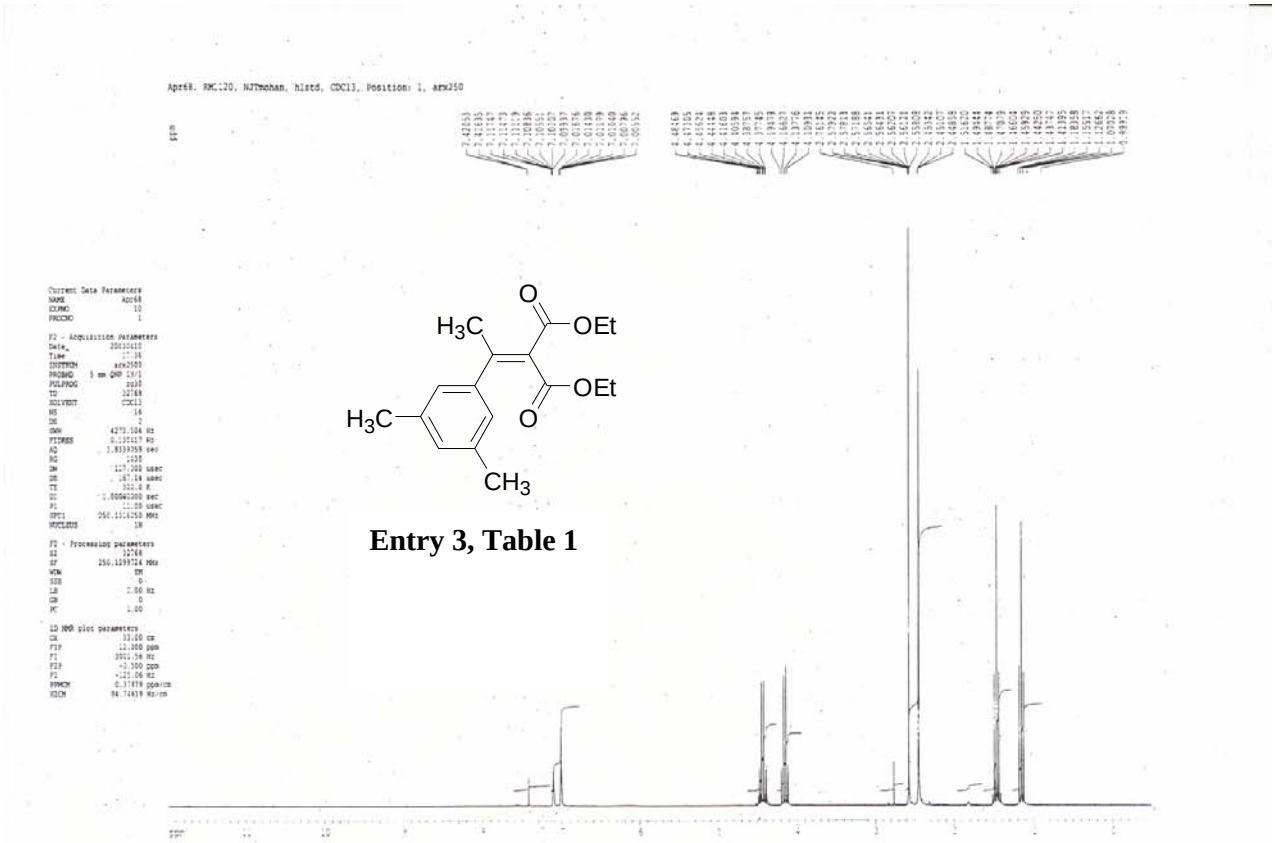
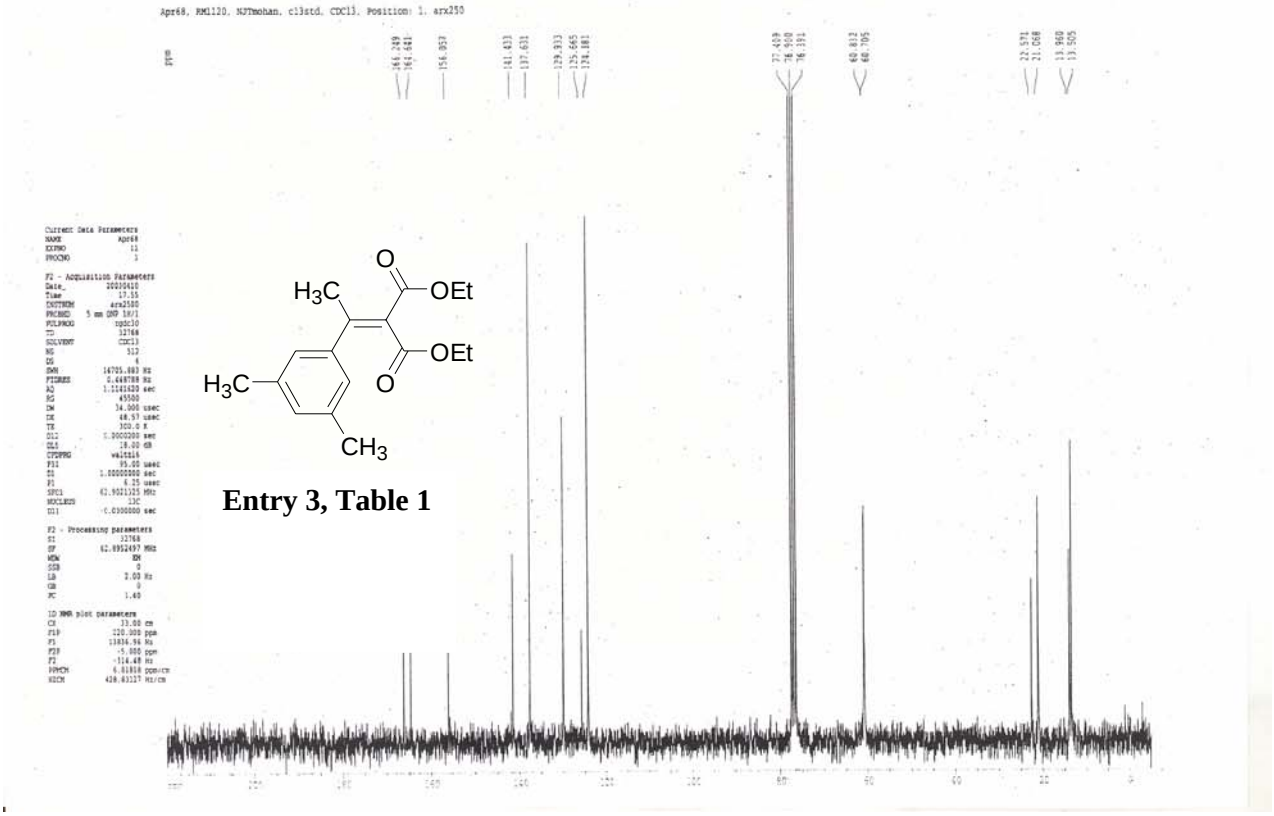
EXPNO 10
PROCNO 1
F1 - Acquisition Parameters
Date_ 20021202
Time 21.24
INSTRM arx250
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 128
DS 2
SWH 4273.504 Hz
FIDRES 0.130417 Hz
AQ 3.8139059 sec
RG 2048
DW 117.000 usec
DE 167.14 usec
TE 300.0 K
D1 1.00000000 sec
F1 11.50 usec
SFO1 250.1316250 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 250.1300192 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 13.00 cm
F1P 12.000 ppm
F1 3001.56 Hz
F2P -0.500 ppm
F2 -125.06 Hz
PPMCM 0.37879 ppm/cm
HZCM 94.74622 Hz/cm
O
H3C
OEt
OEt
Cl
O
Entry 1, Table 1

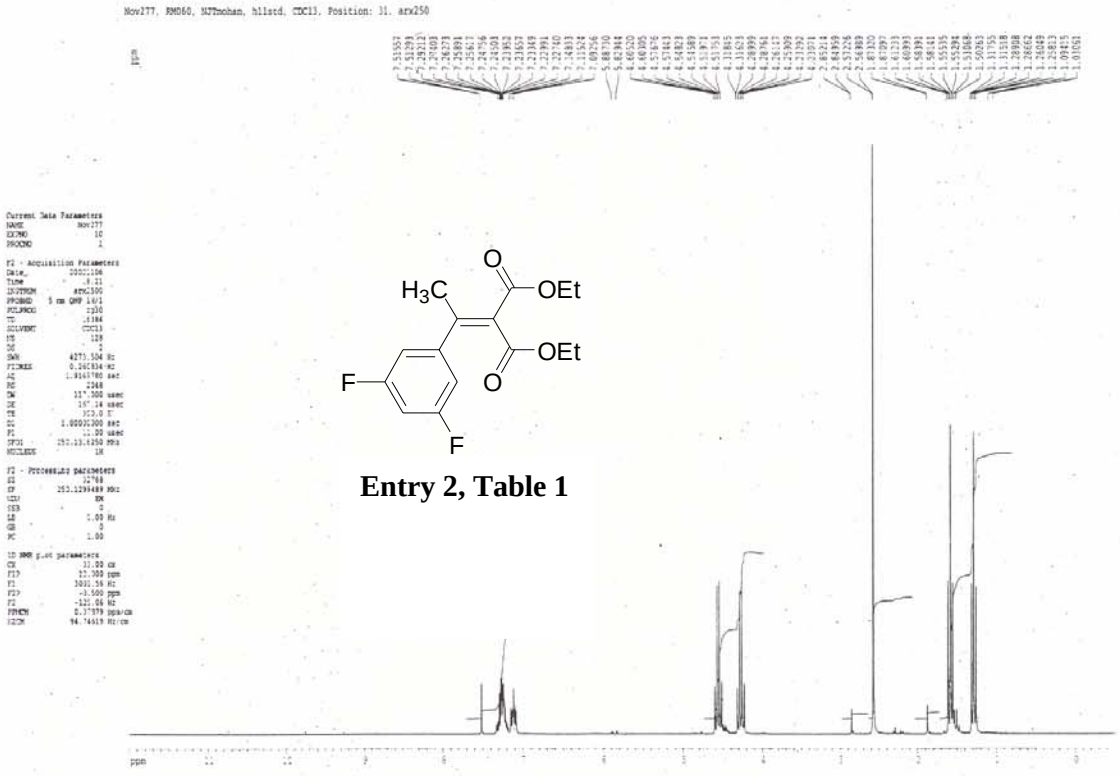

Dec12, RM1075, NJTmohan, c13std5k, CDCl3, Position: 56, arx250
Current Data Parameters
NAME Dec12
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20021203
Time 0.26
INSTRM arx250
PROBHD 5 mm QNP 1H/1
PULPROG zgdc30
TD 32768
SOLVENT CDCl3
NS 5120
DS 4
SWH 14705.883 Hz
FIDRES 0.448788 Hz
AQ 1.1141620 sec
RG 45500
DW 34.000 usec
DE 68.57 usec
TE 300.0 K
D12 0.0000200 sec
CLS 18.00 dB
CPDPRG waltz16
P31 95.00 usec
D1 1.00000000 sec
F1 6.25 usec
SFO1 62.9021325 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 - Processing parameters
SI 32768
SF 62.8952489 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 33.00 cm
F1P 220.000 ppm
F1 13836.96 Hz
F2P -5.000 ppm
F2 -314.48 Hz
PPMCM 6.81818 ppm/cm
HZCM 428.83127 Hz/cm
O
H3C
OEt
OEt
Cl
O
Entry 1, Table 1



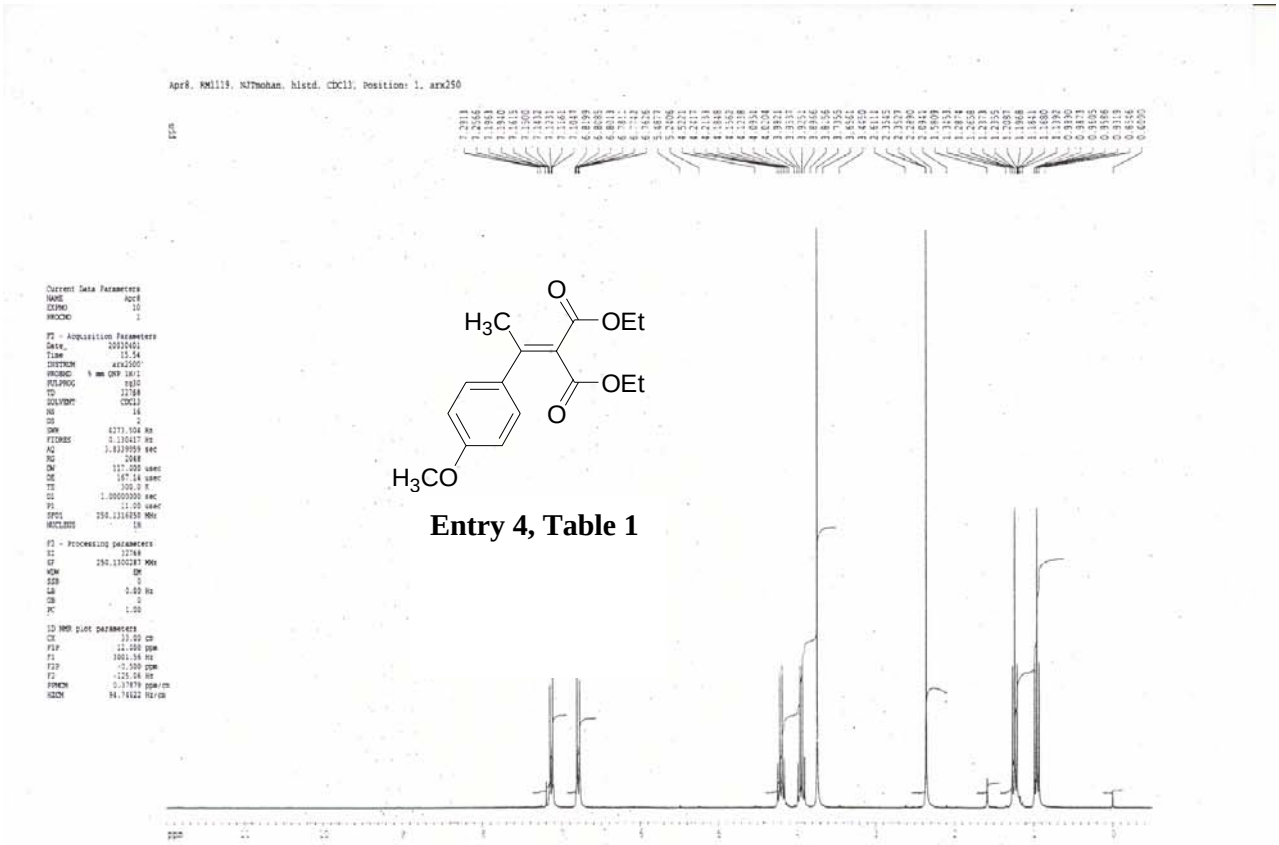
Entry 3, Table 1



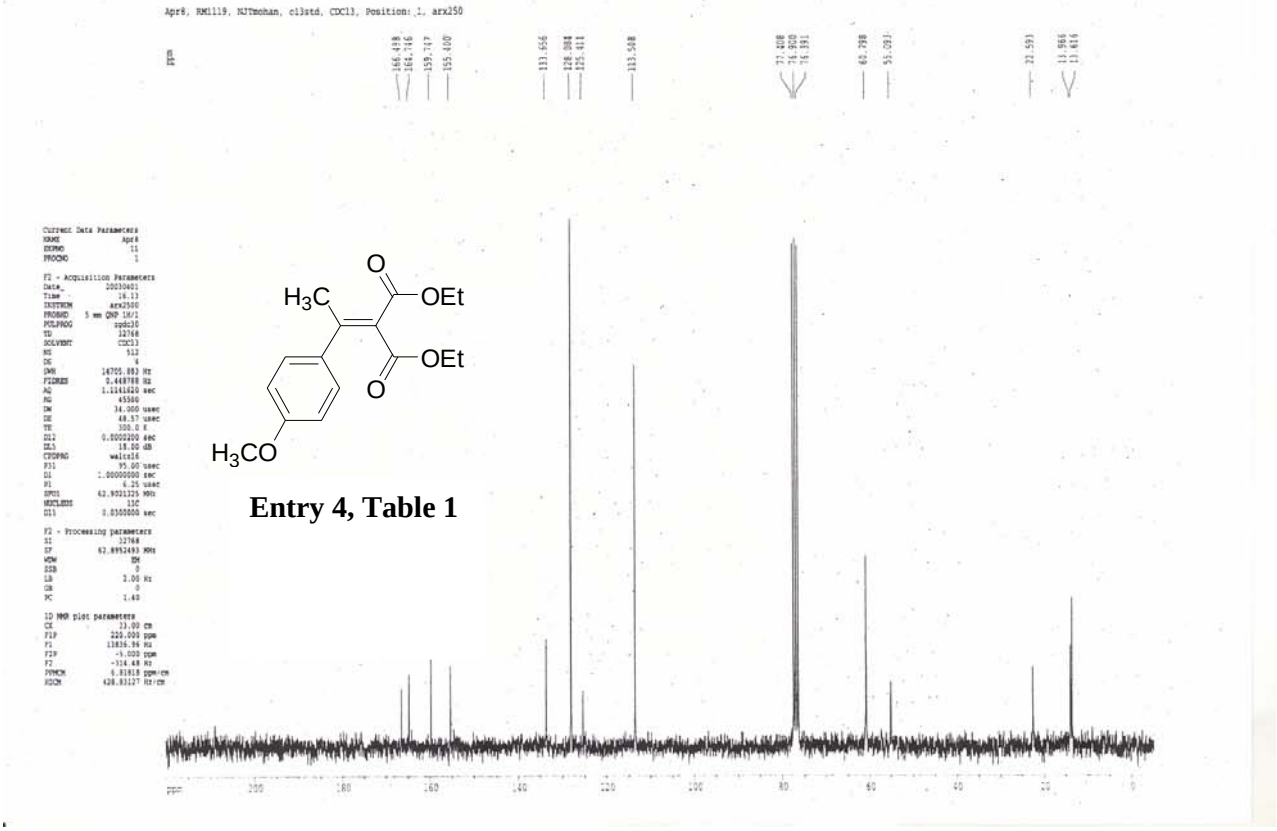
Entry 3, Table 1



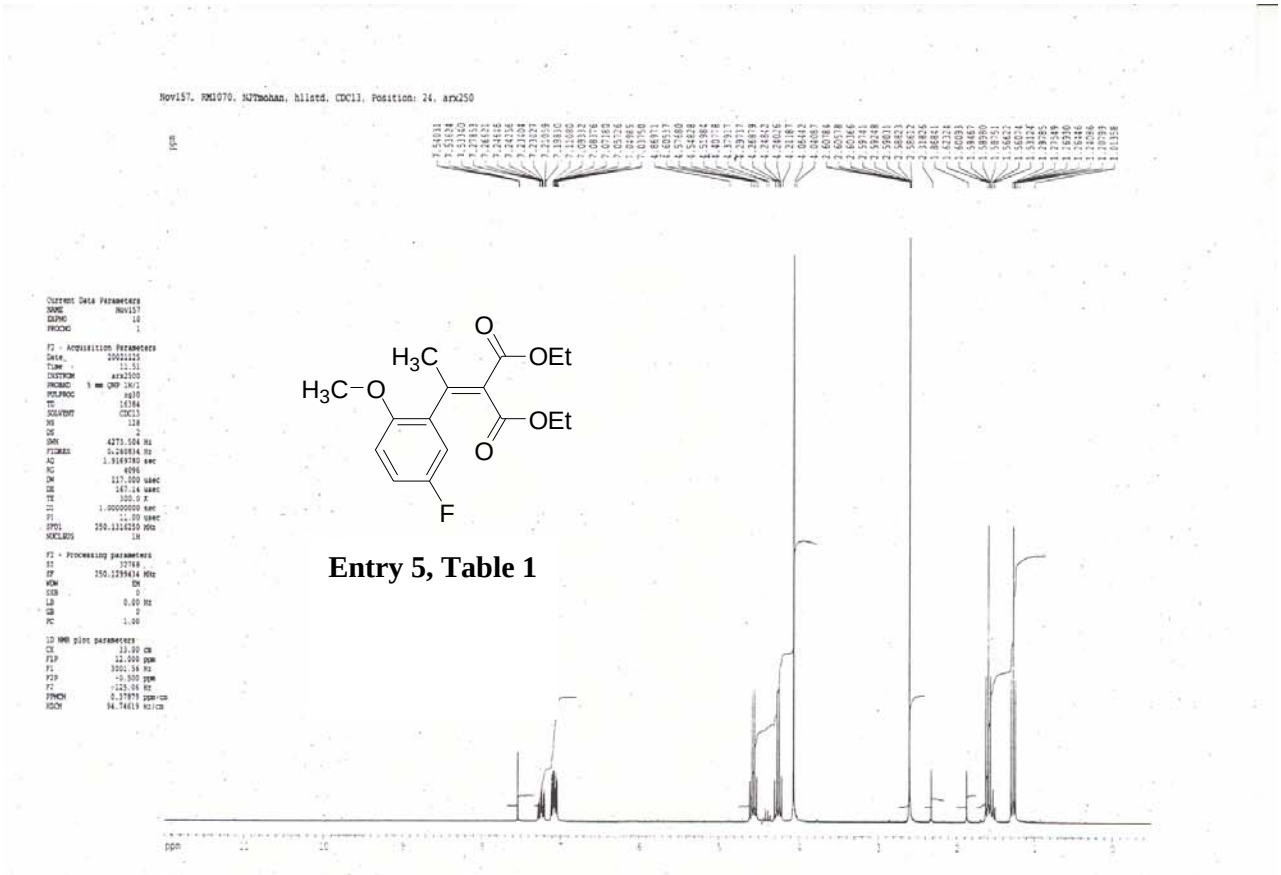
Entry 2, Table 1



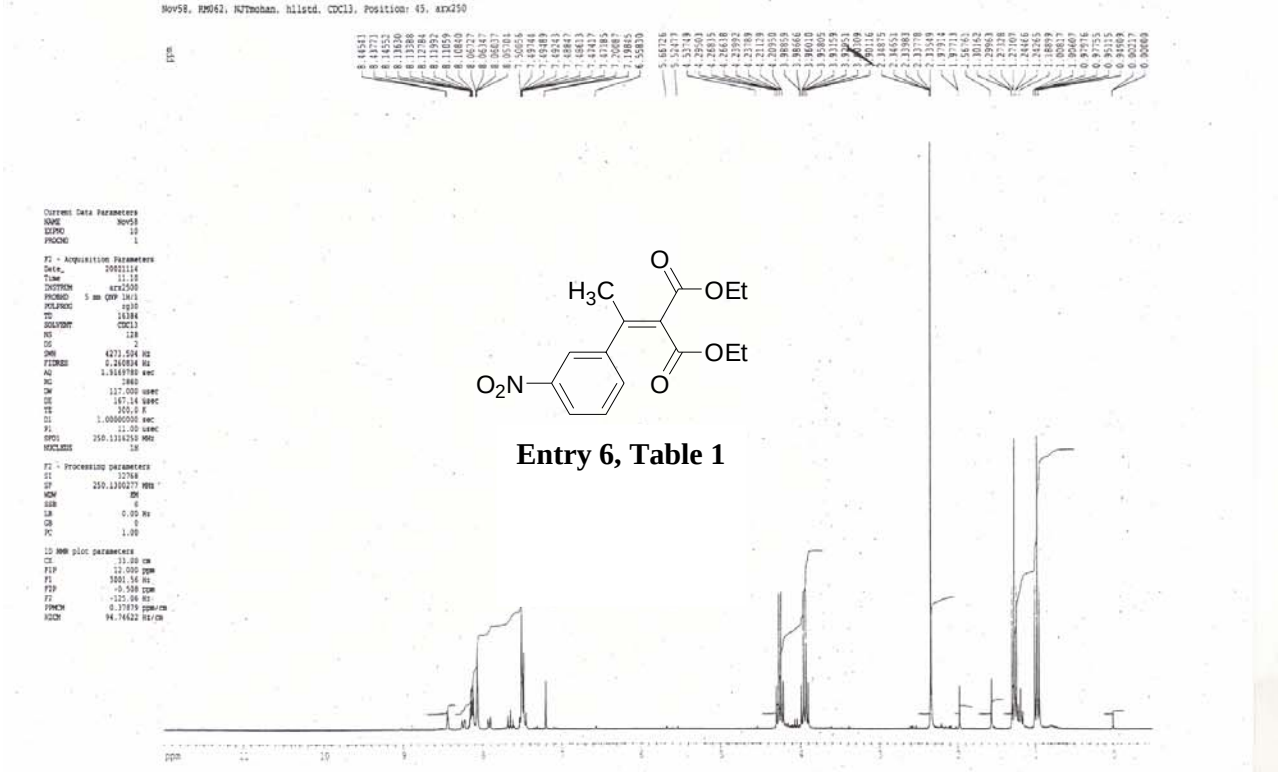
Entry 4, Table 1



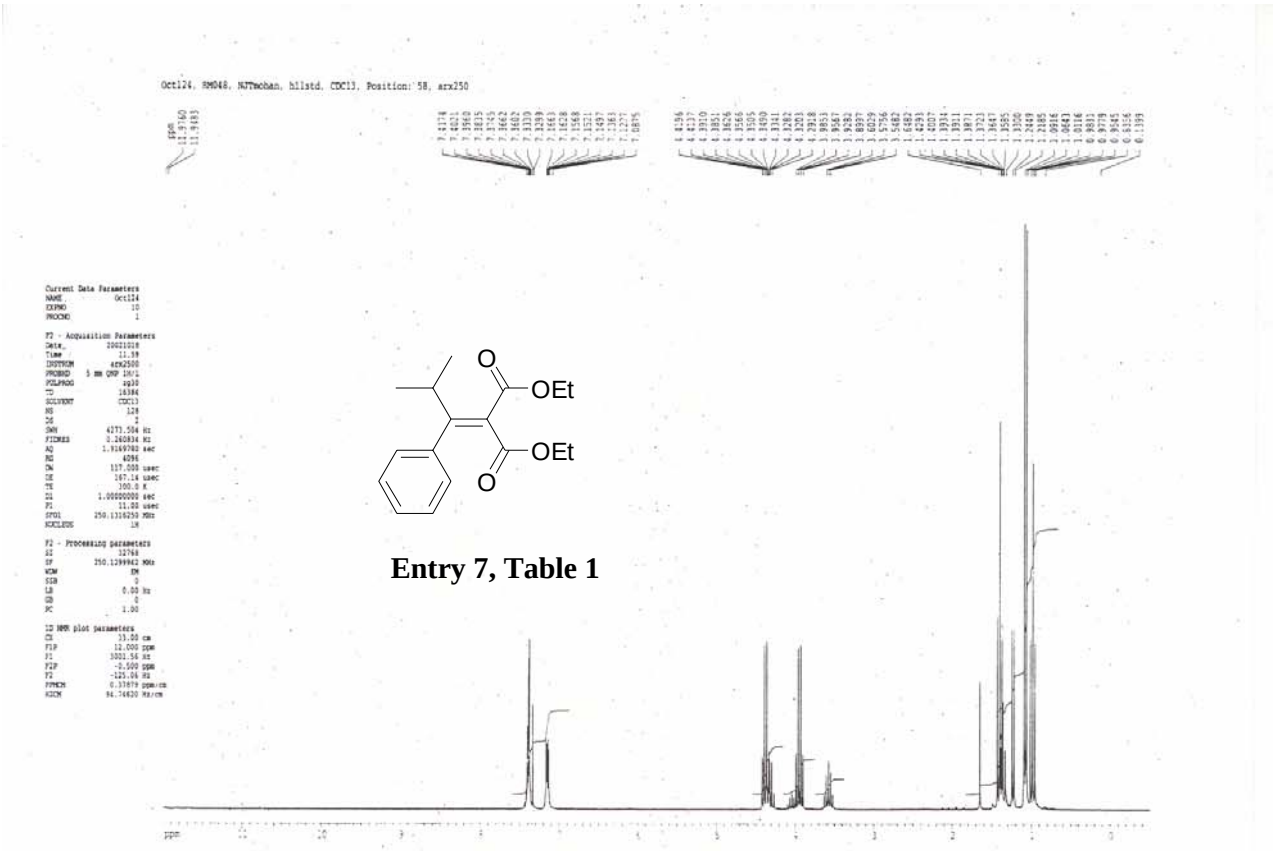
Entry 4, Table 1



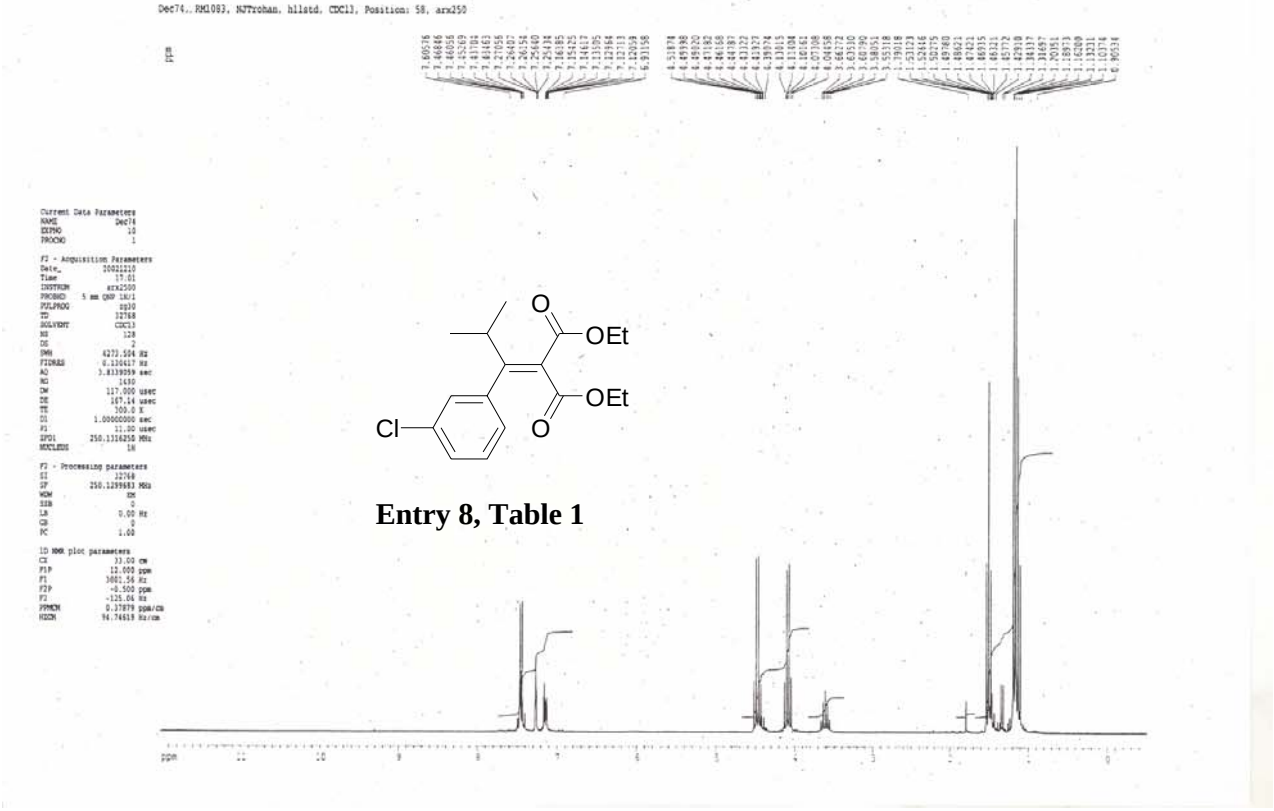
Entry 5, Table 1



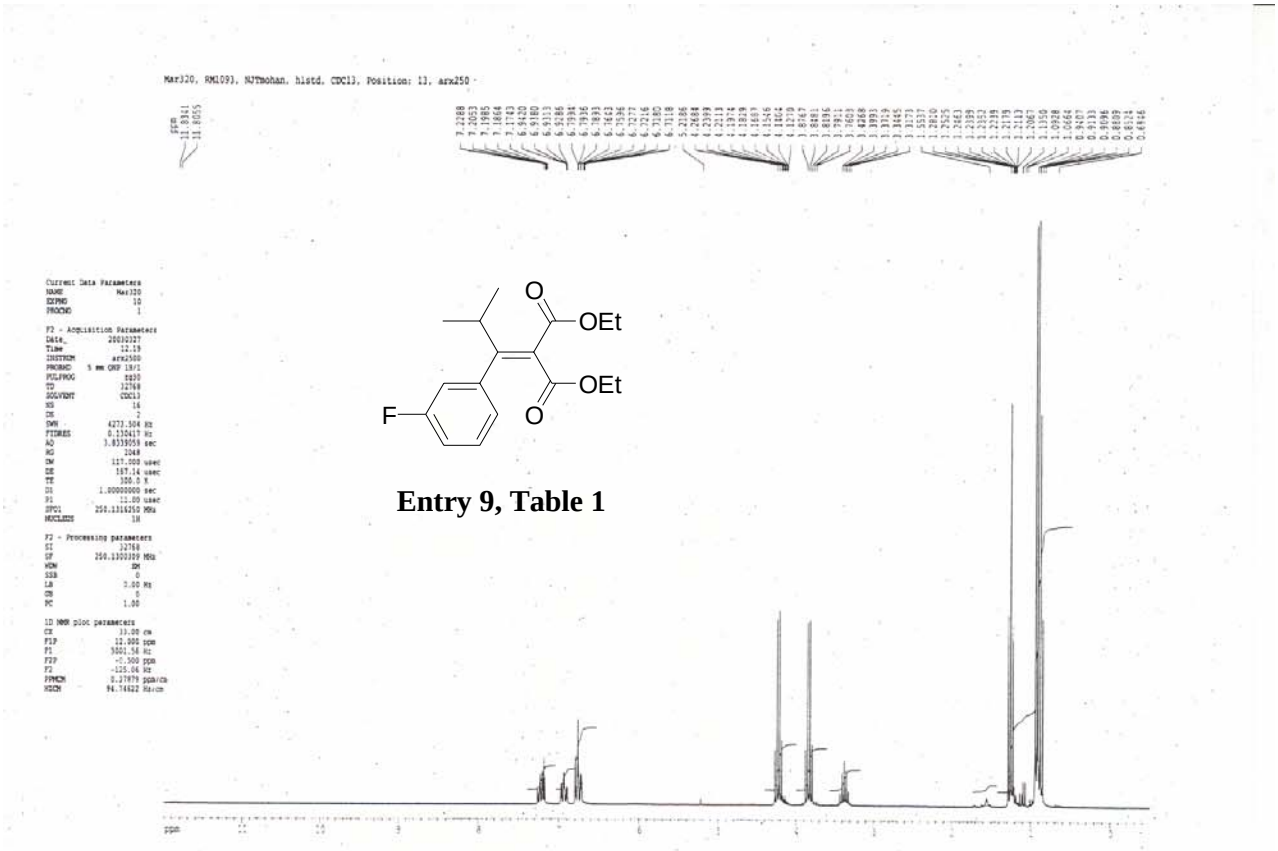
Entry 6, Table 1



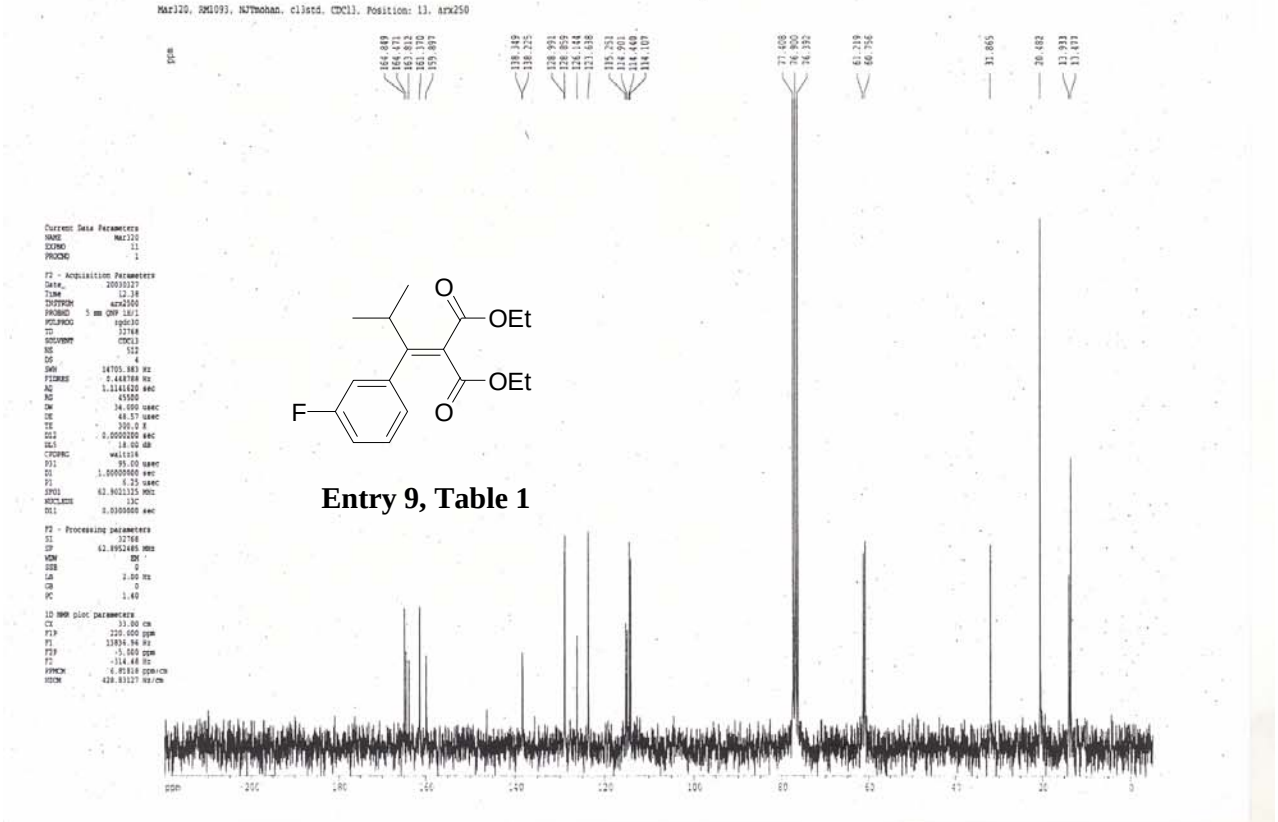
Entry 7, Table 1



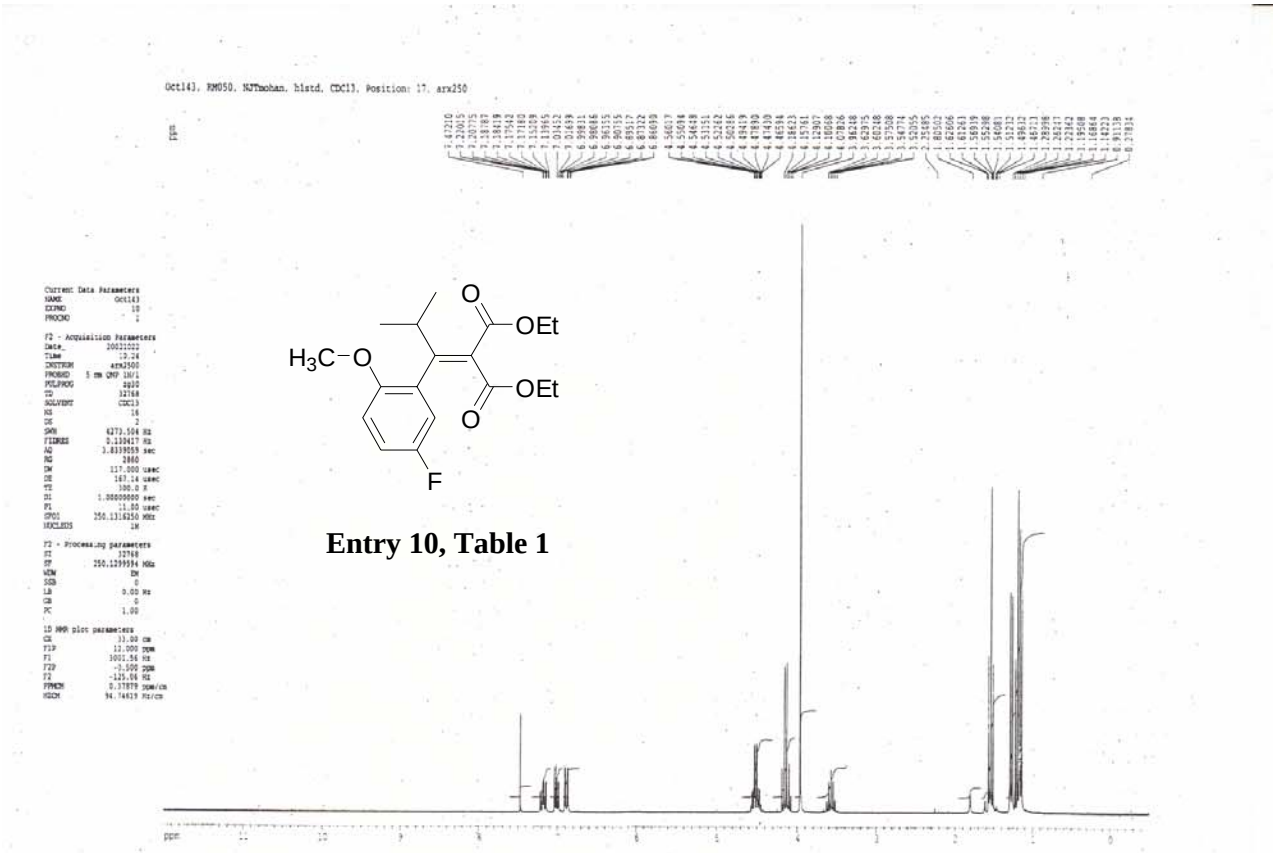
Entry 8, Table 1



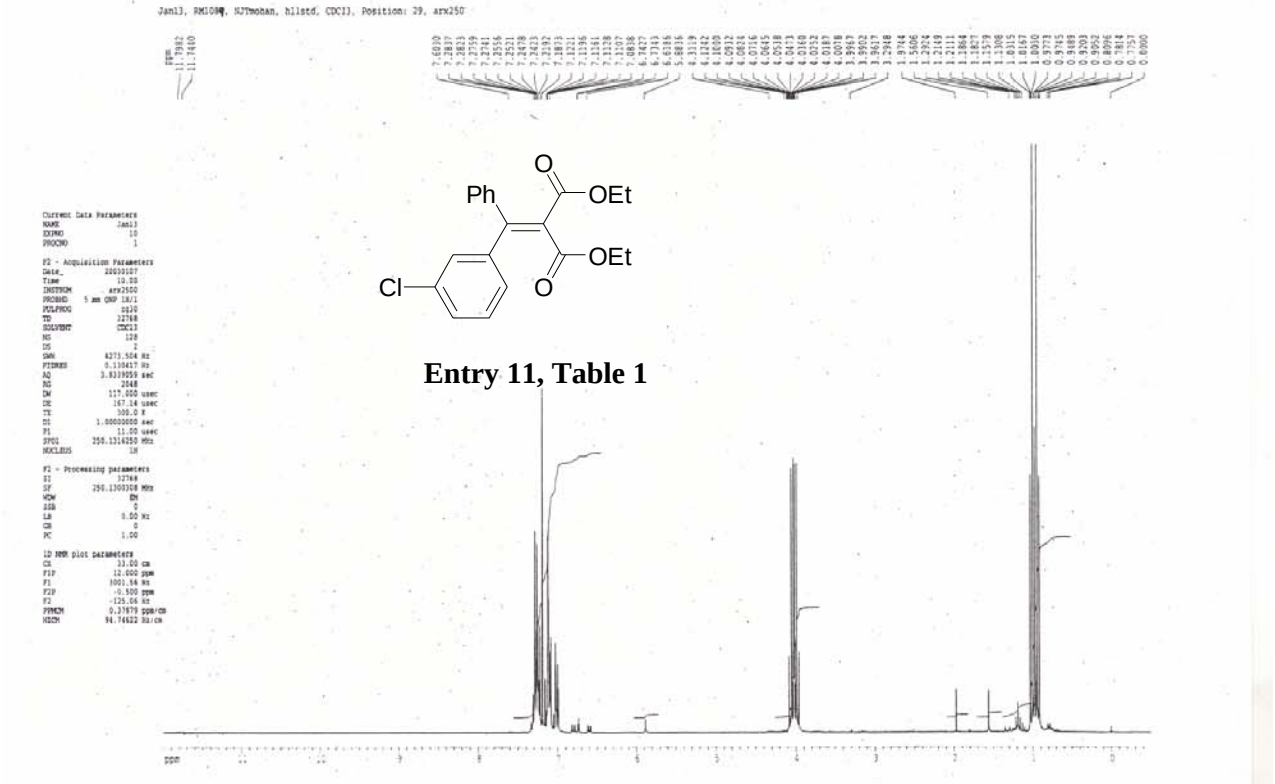
Entry 9, Table 1



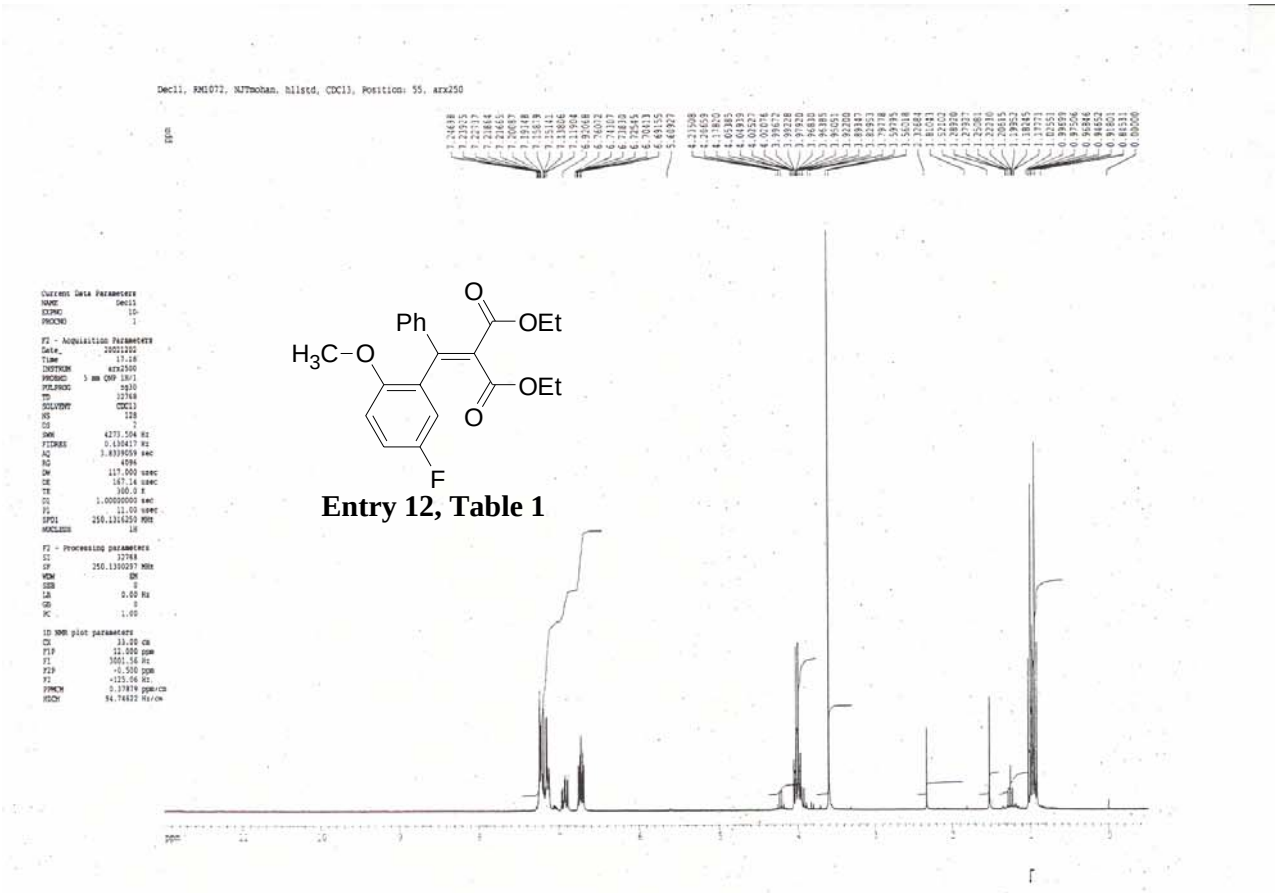
Entry 9, Table 1



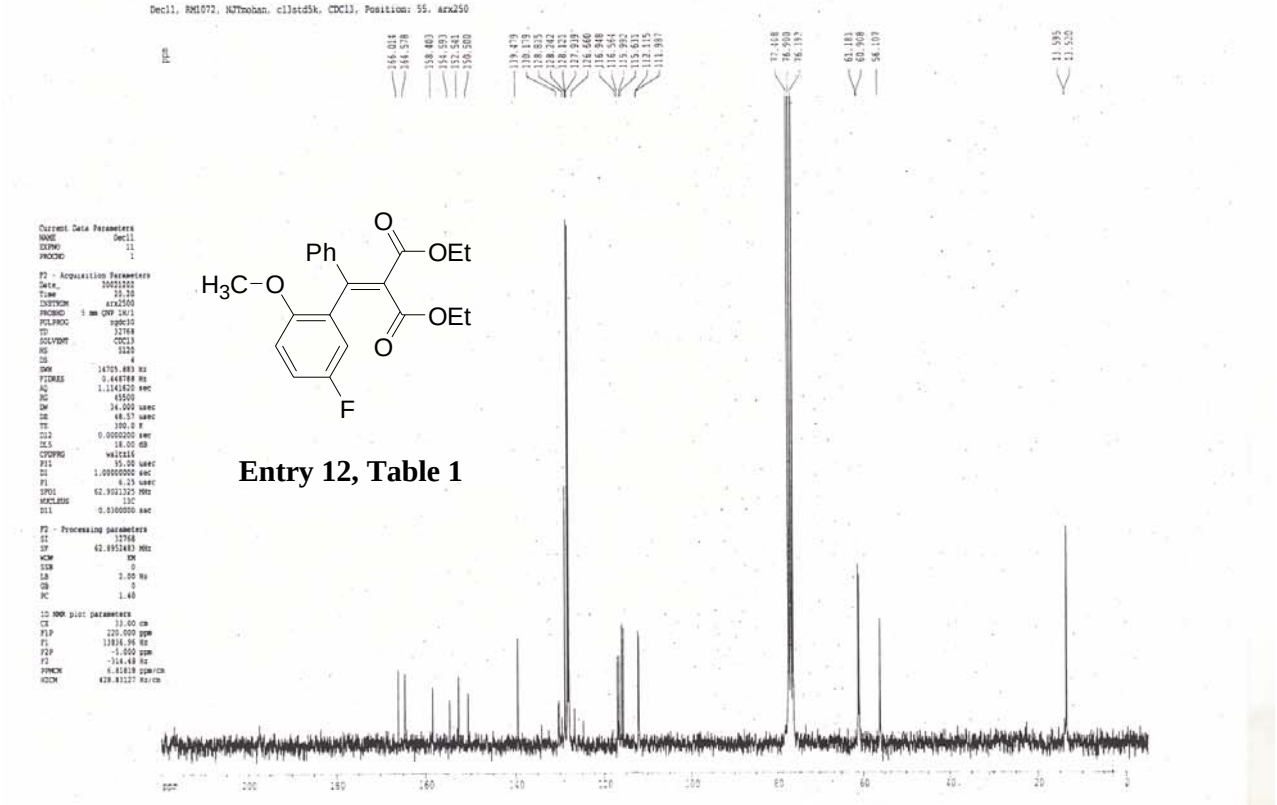
Entry 10, Table 1



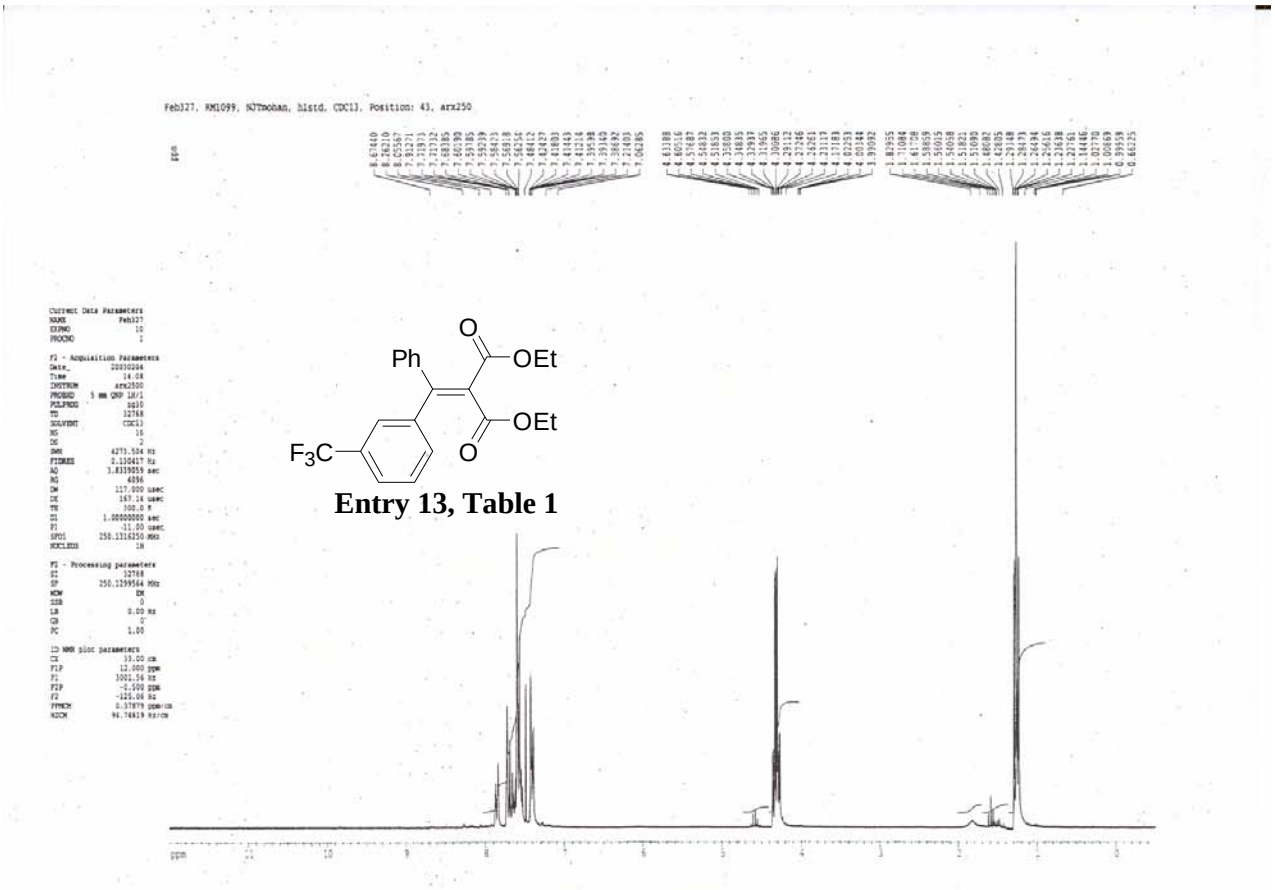
Entry 11, Table 1



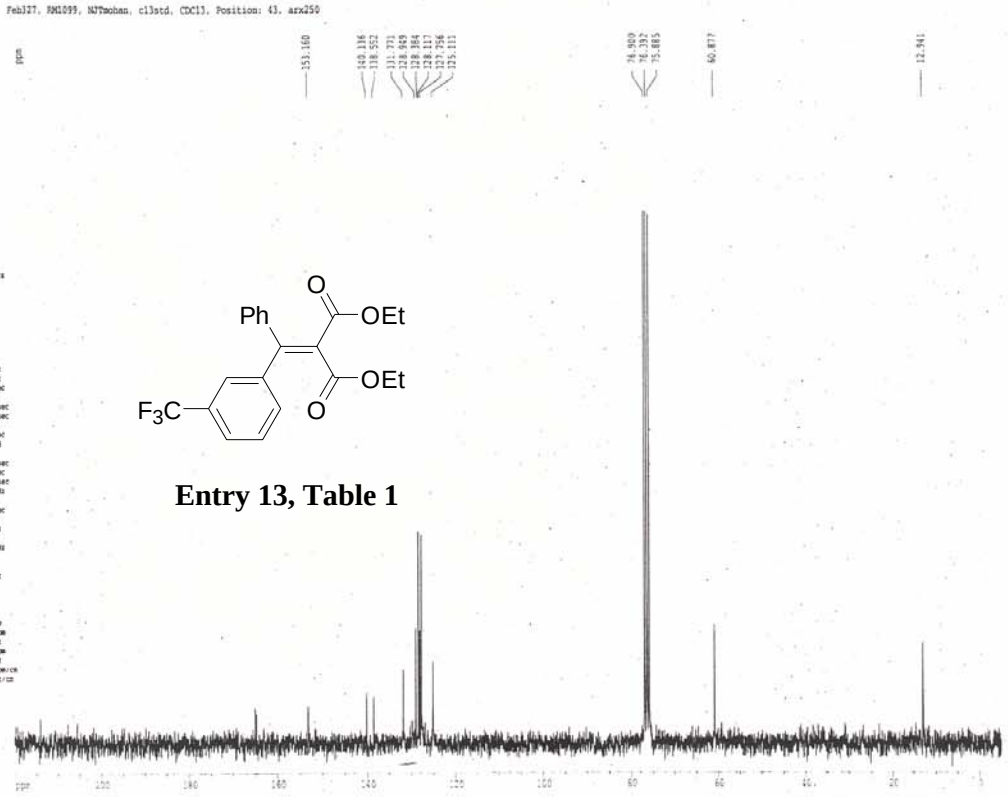
Entry 12, Table 1



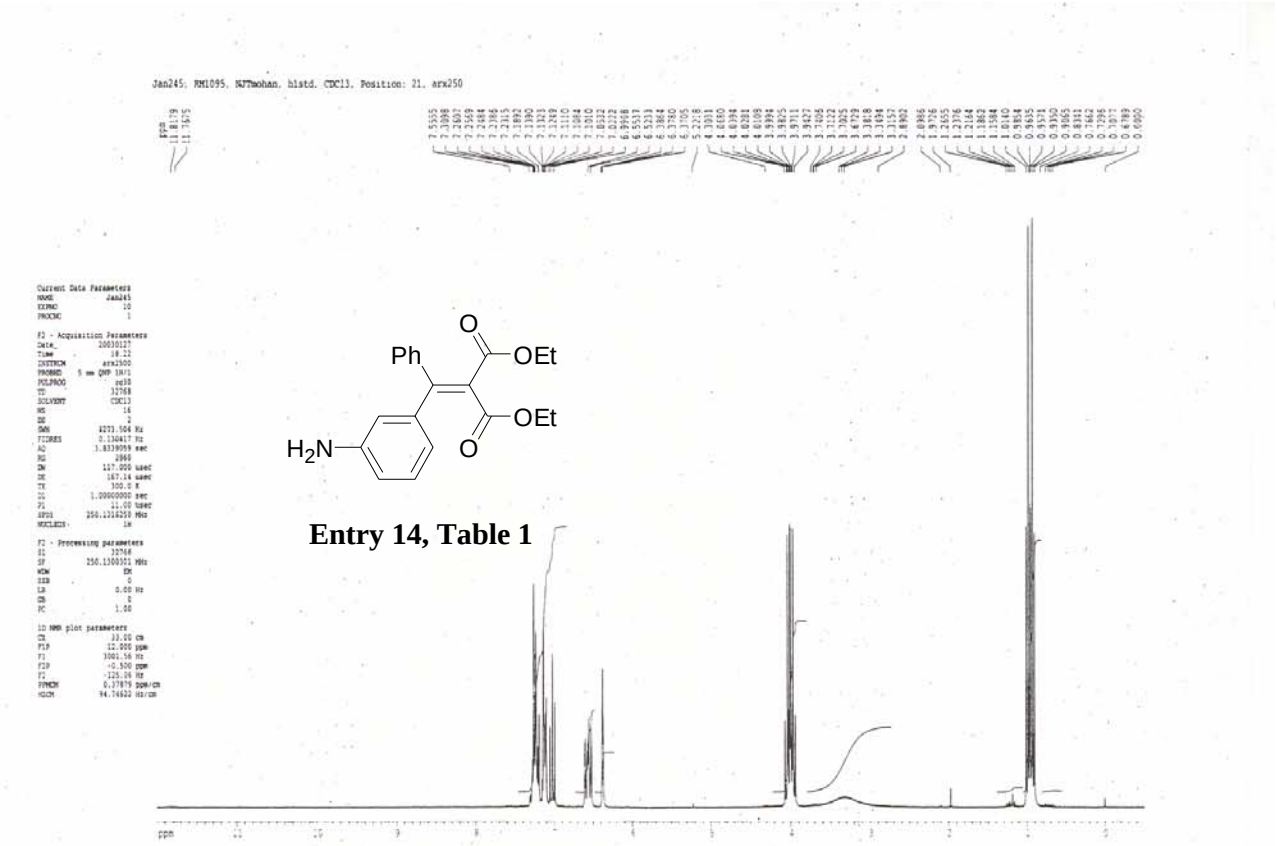
Entry 12, Table 1



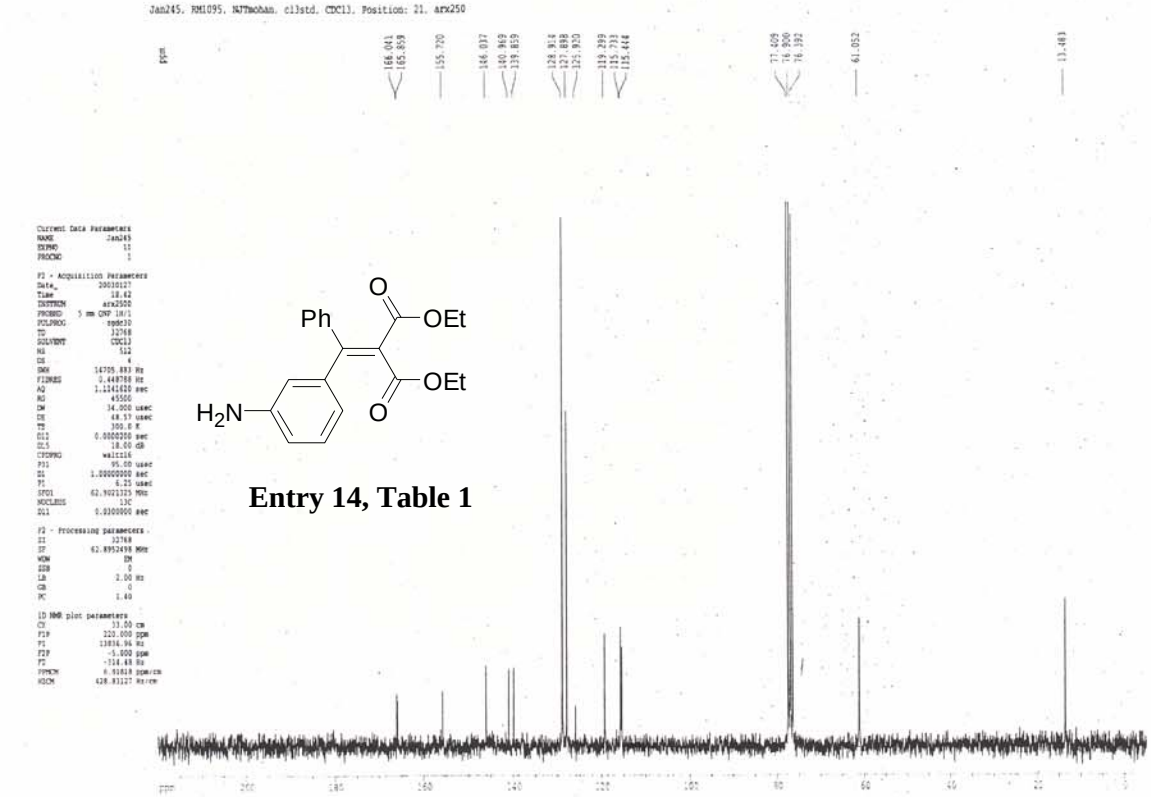
Entry 13, Table 1



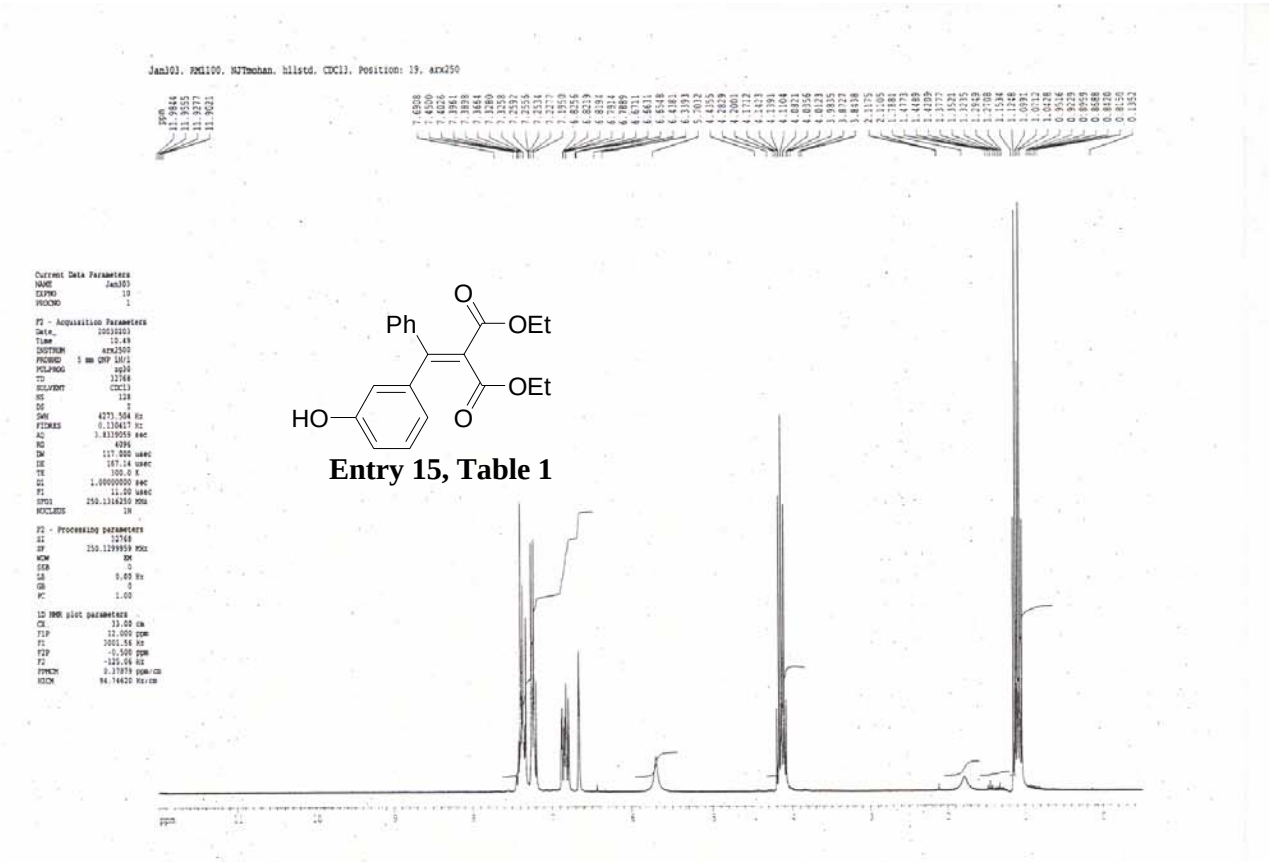
Entry 13, Table 1

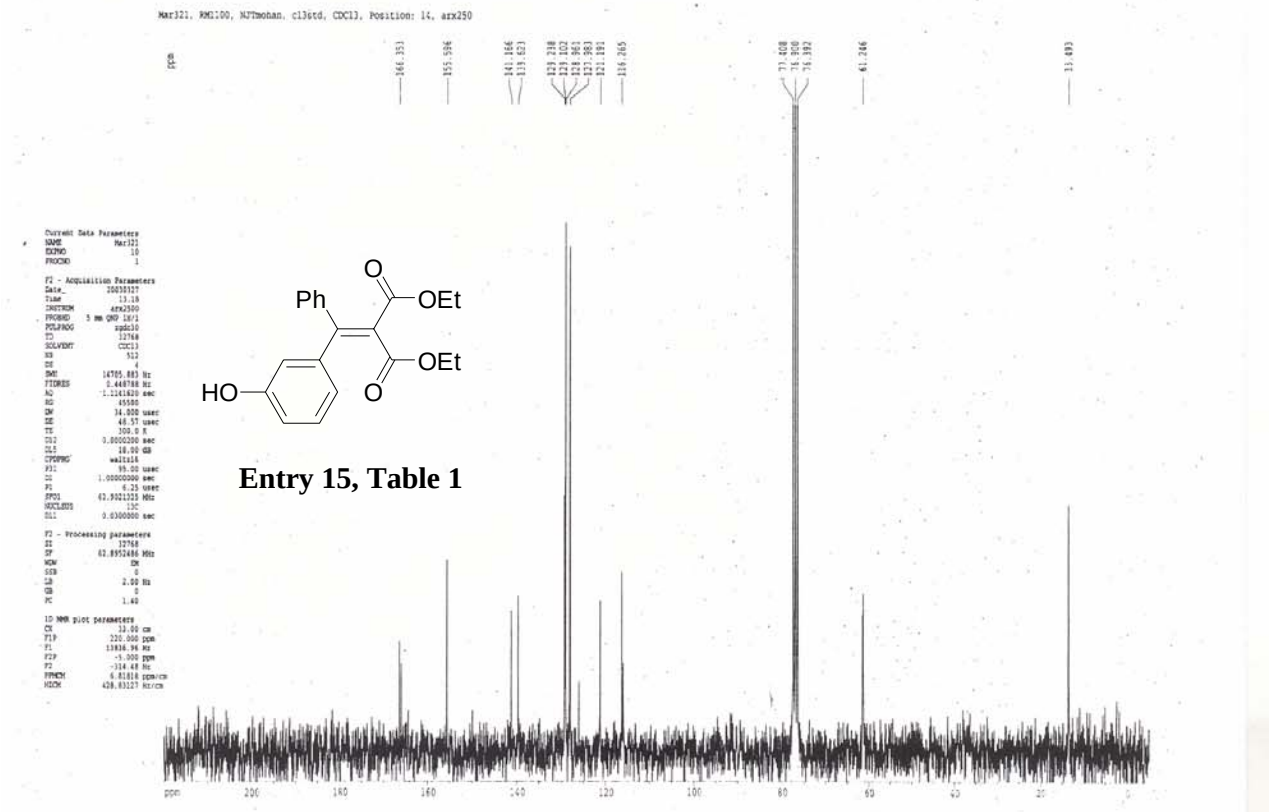


Entry 14, Table 1



Entry 14, Table 1


Jan303, RM1100, MJTmohan, h1std, CDCl3, Position: 19, arx250
Current Data Parameters
NAME Jan303
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030203
Time 10.49
INSTRUM arx2500
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 12768
SOLVENT CDCl3
NS 128
DS 1
SWH 4273.504 Hz
FIDRES 0.130417 Hz
AQ 3.8339059 sec
RG 4096
DW 117.000 usec
DE 167.14 usec
TE 300.0 K
D1 1.00000000 sec
P1 11.00 usec
SFO1 250.1316250 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 250.1299959 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 33.00 cm
F1P 12.000 ppm
F1 3001.56 Hz
F2P -0.500 ppm
F2 -125.06 Hz
PPMCM 0.37879 ppm/cm
HZCM 94.74620 Hz/cm
O
Ph
OEt
OEt
HO
O
Entry 15, Table 1


Mar321, RM1100, MJTmohan, c13std, CDCl3, Position: 14, arx250
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EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030327
Time 13.18
INSTRUM arx2500
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PULPROG zgdc30
TD 12768
SOLVENT CDCl3
NS 512
DS 4
SWH 14705.883 Hz
FIDRES 0.448788 Hz
AQ 1.1141820 sec
RG 65536
DW 34.000 usec
DE 46.57 usec
TE 300.0 K
D11 0.03000000 sec
D12 0.00002000 sec
D15 10.00 GB
CPDPRG waltz16
P31 99.00 usec
D1 1.00000000 sec
P1 6.25 usec
SFO1 62.9021253 MHz
NUCLEUS 13C
D11 0.03000000 sec
F2 - Processing parameters
SI 32768
SF 62.8952486 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 33.00 cm
F1P 220.000 ppm
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F2P -5.000 ppm
F2 -314.48 Hz
PPMCM 6.81818 ppm/cm
HZCM 428.83127 Hz/cm
O
Ph
OEt
OEt
HO
O
Entry 15, Table 1