



Supporting Information

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The First Highly Efficient Cross-Dehydrogenative-Coupling (CDC) between Ethers and Active Methylene Compounds

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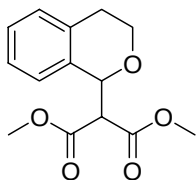
Contents

- 1) Experimental details and characterization data for all new compounds;
- 2) PDF file of copies of ¹H NMR and ¹³C NMR spectra for all new compounds.

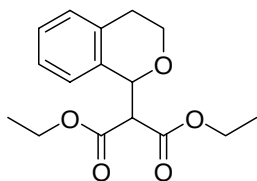
1) Experimental details and characterization data for all new compounds

General information: ^1H NMR spectra were recorded on 400 MHz spectrometer in CDCl_3 (or acetone- d_6) solution and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm). The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; dd, doublet of doublet; dt, doublet of triplet; ddd, doublet of doublet of doublet; dddd, doublet of doublet of doublet of doublet; m, multiplet; q, quartet. The coupling constants, J , are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at 400 MHz and 300 MHz and referenced to the internal solvent signals (central peak is 77.00 ppm). MS data were obtained by Agilent 6890N Network GC System/Agilent 5973 Mass Selective Detector. HRMS were made by McGill University. IR spectra were recorded by an ABB Bomem MB100 instrument. Flash column chromatography was performed over SORBENT silica gel 30-60 μm . All reagents were weighed and handled in air, and backfilled under an inert atmosphere of nitrogen at room temperature. All reagents were purchased from Aldrich and Acros and used without further purification. 3-Methylisochroman (**1b**) was prepared by the literature method ^[1].

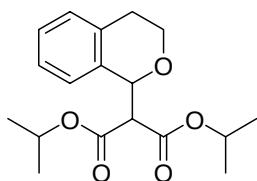
General procedure: To a mixture of isochroman (68 mg, 0.5 mmol) and dimethyl malonate (81 mg, 0.6 mmol), InCl_3 (5.5 mg, 0.025 mmol) and $\text{Cu}(\text{OTf})_2$ (9.0 mg, 0.025 mmol) were added. Then 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) (139 mg, 0.6 mmol) was added and 2-3 mL CH_2Cl_2 was followed at room temperature. The reaction tube was sealed and the resulting mixture was stirred at room temperature for 24 to 36 hours. The resulting suspension was diluted with diethyl ether and filtered through a short silica gel in a pipette eluting with diethyl ether. Solvent was evaporated and the residue was purified by column chromatography on silica gel (eluting with hexane/ethyl acetate = 3:1), and the fraction with an R_f = 0.5 was collected and concentrated to give the desired product **3a**.



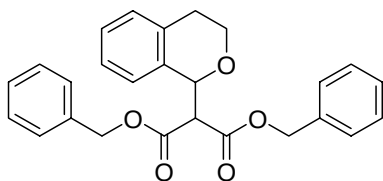
Dimethyl 2-(isochroman-1-yl)malonate (3a). Isolated by flash column chromatography (hexane/ethyl acetate = 3:1, R_f = 0.5). IR (KBr): $\nu_{\max}$ 2997, 2853, 1758, 1735, 1491, 1452, 1433, 1333, 1283, 1195, 1149, 1109, 1056, 1027, 997, 750 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.18-7.08(m, 3H), 6.99(d, J = 7.6 Hz, 1H), 5.45(d, J = 6 Hz, 1H), 4.15(dddd, J = 11.2, 4.8, 4.8, 1.6 Hz, 1H), 3.98(dd, J = 6.0, 2.0 Hz, 1H), 3.80-3.74(m, 1H), 3.73(d, J = 2.0 Hz, 3H), 3.63(d, J = 2.0 Hz, 3H), 3.01-2.94(m, 1H), 2.69(d, J = 16.4 Hz, 1H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 168.2, 167.3, 134.8, 134.6, 129.3, 127.4, 126.5, 124.7, 74.3, 63.7, 58.2, 53.0, 52.7, 28.8; MS (EI) m/z (%) 264, 205, 173, 146, 133(100), 115, 105, 79, 77, 59, 39; HRMS calcd for $\text{C}_{14}\text{H}_{16}\text{O}_5$: 264.0998; found: 264.0994.



Diethyl 2-(isochroman-1-yl)malonate (3b). Isolated by flash column chromatography (hexane/ethyl acetate = 5:1, R_f = 0.4). IR (KBr): $\nu_{\max}$ 2979, 2903, 1758, 1734, 1496, 1451, 1367, 1334, 1301, 1277, 1250, 1175, 1151, 1100, 1032, 915, 730 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.17-7.07(m, 3H), 7.03(d, J = 7.6 Hz, 1H), 5.45(d, J = 6.4 Hz, 1H), 4.22-4.08(m, 5H), 3.95(dd, J = 6.4, 1.2 Hz, 1H), 3.80-3.73(m, 1H), 3.01-2.94(m, 1H), 2.69(ddd, J = 16, 7.6, 3.6 Hz, 1H), 1.21(dt, J = 7.2, 1.2 Hz, 3H), 1.12 (dt, J = 7.2, 1.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 167.8, 167.0, 135.1, 134.6, 129.2, 127.3, 126.4, 124.9, 74.3, 63.6, 61.9, 61.5, 58.4, 28.8, 14.2, 14.1; MS (EI) m/z (%) 292, 219, 173, 146, 133(100), 115, 91, 77, 55; HRMS calcd for $\text{C}_{16}\text{H}_{20}\text{O}_5$: 292.1311; found: 292.1305.



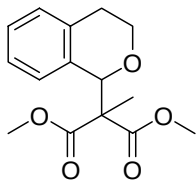
Diisopropyl 2-(isochroman-1-yl)malonate (3c). Isolated by flash column chromatography (hexane/ethyl acetate = 10:1, R_f = 0.4). IR (KBr): $\nu_{\max}$ 2979, 2935, 1754, 1727, 1490, 1463, 1373, 1297, 1182, 1104, 995, 910, 832, 744, 642, 557 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.14-7.07(m, 4H), 5.43(d, J = 5.6 Hz, 1H), 5.09-5.03(m, 1H), 5.01-4.95(m, 1H), 4.17-4.14(m, 1H), 3.89(dd, J = 6.4, 2.4 Hz, 1H), 3.79-3.74(m, 1H), 2.98-2.94(m, 1H), 2.70(d, J = 16.4 Hz, 1H), 1.22(dd, J = 6.0, 2.0 Hz, 3H), 1.17(dd, J = 6.0, 2.0 Hz, 3H), 1.16-1.12(m, 6H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 167.4, 166.6, 135.4, 134.5, 129.2, 127.2, 126.3, 125.0, 74.2, 69.4, 68.9, 63.6, 58.7, 28.8, 21.8, 21.6; MS (EI) m/z (%) 320, 233, 191, 173, 133(100), 115, 105, 77, 43; HRMS calcd for $\text{C}_{18}\text{H}_{24}\text{O}_5$: 320.1624; found: 320.1616.



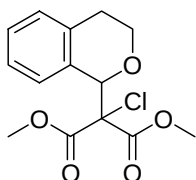
Dibenzyl 2-(isochroman-1-yl)malonate (3e). Isolated by flash column chromatography (hexane/ethyl acetate = 5:1, R_f = 0.4). IR (KBr): $\nu_{\max}$ 2958, 2853, 1757, 1735, 1496, 1452, 1381, 1331, 1279, 1142, 1111, 1055, 1002, 950, 909, 748, 698 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.34-7.29(m, 8H), 7.19-7.16(m, 3H), 7.09-7.06(m, 2H), 7.00(d, J = 8.0 Hz, 1H), 5.53(d, J = 6.0 Hz, 1H), 5.22(d, J = 12.8 Hz, 1H), 5.16(d, J = 12 Hz, 1H), 5.11(s, 2H), 4.17-4.11(m, 2H), 3.79-3.73(m, 1H), 2.90(ddd, J = 15.2, 8.8, 5.6 Hz, 1H), 2.66(d, J = 16.4 Hz, 1H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 167.6, 166.7, 135.6, 135.4, 134.9, 134.7, 129.4, 128.8, 128.7, 128.6(two peaks), 128.5, 128.4, 127.3, 126.5, 124.8, 74.4, 67.6, 67.3, 63.8, 58.3, 28.8;

MS (EI) m/z (%) 325($M^+ - CH_2C_6H_5$), 281, 219, 205, 173, 146, 133, 115, 107, 91(100), 79, 77;

HRMS calcd for $C_{26}H_{24}O_5$: 325.1076($M^+ - CH_2C_6H_5$); found: 325.1073.

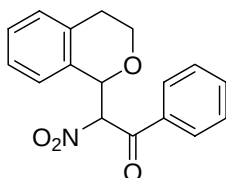


Dimethyl 2-(isochroman-1-yl)-2-methylmalonate (3f). Isolated by flash column chromatography (hexane/ethyl acetate = 5:1, R_f = 0.5). IR (KBr): ν_{max} 2970, 2852, 1734, 1457, 1439, 1375, 1308, 1256, 1227, 1120, 1099, 1059, 1004, 968, 923, 868, 802, 764, 759 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz, ppm) δ 7.17-7.09(m, 3H), 7.03(d, J = 7.6 Hz, 1H), 5.72(s, 1H), 4.12(dd, J = 11.2, 5.6 Hz, 1H), 3.77(s, 3H), 3.76(s, 3H), 3.64(dt, J = 11.2, 1.6 Hz, 1H), 3.00(ddd, J = 16, 11.2, 5.6 Hz, 1H), 2.54(d, J = 16 Hz, 1H), 1.21(s, 3H); ^{13}C NMR ($CDCl_3$, 400 MHz, ppm) δ 171.4, 170.6, 136.1, 134.2, 129.3, 127.1, 126.4, 125.8, 78.2, 64.6, 60.9, 53.1, 53.0, 29.7, 15.0; MS (EI) m/z (%) 278, 218, 187, 133(100), 115, 105, 91, 79, 77; HRMS calcd for $C_{15}H_{18}O_5$: 278.1154; found: 278.1156.

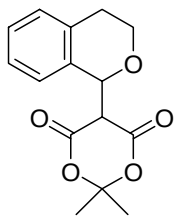


Dimethyl 2-chloro-2-(isochroman-1-yl)malonate (3g). Isolated by flash column chromatography (hexane/ethyl acetate = 4:1, R_f = 0.4). IR (KBr): ν_{max} 2976, 2858, 1769, 1742, 1457, 1431, 1388, 1262, 1209, 1150, 1113, 1020, 901, 773, 765, 753, 729 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz, ppm) δ 7.25-7.19(m, 2H), 7.16-7.11(m, 2H), 5.92(s, 1H), 4.23(dddd, J = 10.8, 4.4, 3.6, 1.6 Hz, 1H), 3.85(d, J = 1.6 Hz, 3H), 3.83(d, J = 1.6 Hz, 3H), 3.71(ddt, J = 10.4, 3.6, 1.6 Hz, 1H), 3.03(ddd, J = 16.6, 9.2, 5.2 Hz, 1H), 2.68-2.63(m, 1H); ^{13}C NMR($CDCl_3$,

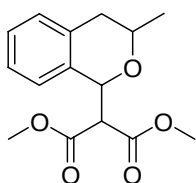
400 MHz, ppm) δ 166.6, 165.4, 136.2, 132.3, 129.1, 127.8, 126.4, 126.1, 78.4, 64.0, 54.4, 54.3, 29.1; ^{13}C NMR (acetone- d_6 , 300 MHz, ppm) δ 166.3, 164.7, 136.2, 132.6, 129.1, 127.6, 126.2, 126.1, 78.3, 77.7, 63.6, 53.8, 53.7, 29.0; MS (EI) m/z (%) 263(M^+ -Cl), 231, 133(100), 115, 105, 91, 82, 79, 77, 59; HRMS calcd for $\text{C}_{14}\text{H}_{15}\text{ClO}_5$: 263.0924(M^+ -Cl); found: 263.0916.



2-(Isochroman-1-yl)-2-nitro-1-phenylethanone (3h). The ratio of two diastereoisomers is 2.5:1. Isolated by flash column chromatography (hexane/ethyl acetate = 3:1, R_f = 0.5). IR (KBr) of two isomers: ν_{max} 2976, 2958, 2858, 1764, 1742, 1457, 1436, 1305, 1291, 1264, 1217, 1207, 1186, 1112, 1012, 953, 910, 891, 777, 765, 739, 666, 649, 635 cm^{-1} ; The major isomer: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 6.57(dd, J = 6.8, 1.6 Hz, 1H), 5.85(d, J = 6.8 Hz, 1H), 4.21-4.17(m, 1H), 3.81-3.76(m, 1H), 3.03-2.95(m, 1H), 2.77-2.73(m, 1H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 187.8, 92.8, 74.4, 63.6, 28.7; The minor isomer: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 6.39(dd, J = 4.8, 1.6 Hz, 1H), 5.90(d, J = 3.2 Hz, 1H), 4.01-3.98(m, 1H), 3.67-3.61(m, 1H), 2.61-2.48(m, 2H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 187.6, 93.9, 74.9, 64.3, 28.6; Other overlapped peaks of two isomers: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.86-7.84(m), 7.68-7.66(m), 7.60-7.51(m), 7.45-7.41(m), 7.35-7.31(m), 7.24-7.22(m), 7.15-6.99(m); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 135.3, 135.2, 135.0, 134.9, 134.3, 132.8, 131.3, 129.6, 129.5, 129.3, 129.0, 128.9, 128.2, 128.1, 127.3, 126.8, 125.6, 124.8; MS (EI) m/z (%) 251(M^+ - NO_2), 173, 158, 148, 133, 115, 105(100), 91, 77, 65, 51; HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{NO}_4$: 251.1072(M^+ - NO_2); found: 251.1070.

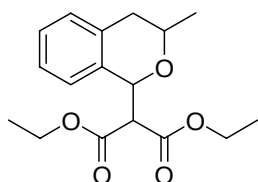


6-Hydroxy-5-(isochroman-1-yl)-2,2-dimethyl-4H-1,3-dioxin-4-one (3i). Isolated by flash column chromatography (hexane/ethyl ether = 2:1, R_f = 0.4). IR (KBr): $\nu_{\max}$ 2925, 2853, 1791, 1743, 1500, 1398, 1380, 1315, 1298, 1201, 1108, 1039, 1008, 915, 897, 842, 742, 631 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.24-7.17(m, 2H), 7.13-7.07(m, 2H), 5.75(s, 1H), 4.14(dd, J = 11.2, 5.6 Hz, 1H), 3.96(s, 1H), 3.72(t, J = 11.2 Hz, 1H), 3.14-3.06(m, 1H), 2.54(d, J = 16 Hz, 1H), 1.79(s, 3H), 1.72(s, 3H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 165.9, 163.0, 135.0, 134.4, 129.4, 127.1, 126.7, 124.0, 106.0, 76.1, 65.2, 51.9, 28.8, 28.7, 28.5; MS (EI) m/z (%) 221, 205, 190, 173, 148, 133, 118, 115, 105, 90, 74, 59, 45, 43(100).



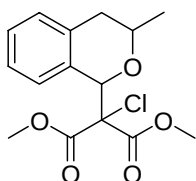
Dimethyl 2-(3-methylisochroman-1-yl)malonate (3j). The ratio of two diastereoisomers is 1:1. Isolated by flash column chromatography (hexane/ethyl acetate = 5:1, R_f = 0.5). IR (KBr) of the two isomers: $\nu_{\max}$ 2945, 2892, 1768, 1728, 1494, 1455, 1431, 1382, 1079, 1033, 944, 790, 753, 645, 599, 559 cm^{-1} ; The first isomer: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.18-7.12(m, 2H), 7.08-7.06(m, 1H), 7.00-6.98(m, 1H), 5.46(d, J = 5.6 Hz, 1H), 3.91(d, J = 6 Hz, 1H), 3.82-3.76(m, 1H), 3.74(s, 3H), 3.64(s, 3H), 2.74(dd, J = 16, 10.8 Hz, 1H), 2.62(dd, J = 15.6, 2.4 Hz, 1H), 1.30(d, J = 6 Hz, 3H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 168.3, 167.5, 135.3, 135.1, 129.0, 127.2, 126.5, 124.2, 75.2, 71.1, 58.9, 52.9, 52.6, 36.6, 21.9; The second isomer: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.20-7.04(m, 4H), 5.56(d, J = 9.6 Hz, 1H), 4.15-

4.10(m, 1H), 4.04(dd, $J = 9.6, 1.2$ Hz, 1H), 3.78(d, $J = 1.6$ Hz, 3H), 3.71(d, $J = 1.6$ Hz, 3H), 2.77(dd, $J = 16.4, 3.6$ Hz, 1H), 2.64(dd, $J = 16, 10.4$ Hz, 1H), 1.23(dd, $J = 6, 1.6$ Hz, 3H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 168.0, 167.3, 134.6, 133.6, 129.4, 127.7, 126.3, 125.4, 73.7, 65.9, 58.7, 53.0, 52.9, 35.7, 21.6; MS (EI) m/z (%) 278, 260, 219, 205, 187, 175, 160, 147(100), 129, 115, 103, 91, 77, 59, 43; HRMS calcd for $\text{C}_{15}\text{H}_{18}\text{O}_5$: 278.1154; found: 278.1151.

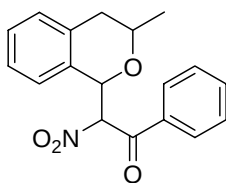


Diethyl 2-(3-methylisochroman-1-yl)malonate (3k). The ratio of two diastereoisomers is 1:1. The first isomer: Isolated by flash column chromatography (hexane/ethyl acetate = 6:1, $R_f = 0.55$). IR (KBr): ν_{max} 2979, 2932, 1758, 1733, 1382, 1367, 1345, 1299, 1273, 1251, 1177, 1144, 1039, 750 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.19-7.15(m, 1H), 7.10-7.07(m, 3H), 5.57(d, $J = 9.2$ Hz, 1H), 4.28-4.11(m, 5H), 3.99(d, $J = 9.2$ Hz, 1H), 2.77(dd, $J = 16.4, 3.6$ Hz, 1H), 2.64(dd, $J = 16.4, 10$ Hz, 1H), 1.28(t, $J = 7.2$ Hz, 3H), 1.24(d, $J = 6$ Hz, 3H), 1.20(t, $J = 7.2$ Hz, 3H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 167.7, 166.9, 134.9, 133.6, 129.3, 127.6, 126.2, 125.5, 73.6, 65.8, 61.9, 61.8, 59.1, 35.7, 21.6, 14.3, 14.2; MS (EI) m/z (%) 306, 288, 233, 220, 205, 187, 160, 147(100), 129, 115, 91, 77, 57, 43; HRMS calcd for $\text{C}_{17}\text{H}_{22}\text{O}_5$: 306.1467; found: 306.1472. The second isomer: Isolated by flash column chromatography (hexane/ethyl acetate = 6:1, $R_f = 0.5$). IR (KBr): ν_{max} 2976, 2935, 1758, 1731, 1492, 1452, 1385, 1367, 1297, 1277, 1253, 1174, 1142, 1076, 1035, 752 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.12-7.05(m, 2H), 7.01(d, $J = 7.2$ Hz, 1H), 6.97(d, $J = 7.6$ Hz, 1H), 5.41(d, $J = 5.6$ Hz, 1H), 4.22-4.13(m, 2H), 4.06(q, $J = 7.2$ Hz, 2H), 3.83(d, $J = 5.6$ Hz, 1H), 3.77-3.70(m, 1H), 2.69(dd, $J = 16, 11.2$ Hz, 1H), 2.57(dd, $J = 16, 2.4$ Hz, 1H), 1.24(d, $J = 6$

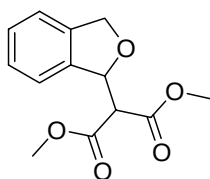
Hz, 3H), 1.18(t, J = 6.4 Hz, 3H), 1.07(t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 168.0, 167.1, 135.4, 135.3, 128.9, 127.1, 126.4, 124.3, 75.2, 71.0, 61.8, 61.4, 58.8, 36.6, 21.8, 14.2, 14.1; MS (EI) m/z (%) 306, 288, 233, 187, 160, 147(100), 129, 115, 91, 77, 43; HRMS calcd for $\text{C}_{17}\text{H}_{22}\text{O}_5$: 306.1467; found: 306.1476.



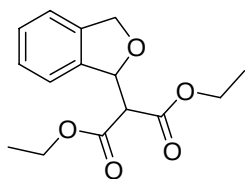
Dimethyl 2-chloro-2-(3-methylisochroman-1-yl)malonate (3I). The ratio of two diastereoisomers is 1:0.7. Isolated by flash column chromatography (hexane/ethyl acetate = 5:1, R_f = 0.4). IR (KBr) of two isomers: ν_{max} 2952, 2880, 1770, 1740, 1454, 1431, 1388, 1262, 1209, 1152, 1113, 1089, 1065, 1020, 902, 773, 754, 729 cm^{-1} ; The major isomer: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 5.92(s, 1H), 3.81(d, J = 2.8 Hz, 3H), 3.78-3.72(m, 1H), 1.27(dd, J = 6, 2.4 Hz, 3H); The minor isomer: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 6.04(s, 1H), 4.48-4.42(m, 1H), 3.87(d, J = 2.8 Hz, 3H), 1.16(dd, J = 6, 2.4 Hz, 3H); Other overlapped peaks of two isomers: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.34-7.07(m), 3.86-3.85(m), 2.83-2.74(m), 2.63-2.56(m); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) of two isomers δ 167.3, 166.6, 165.4, 136.8, 134.9, 132.4, 132.1, 129.1, 129.0, 127.9, 127.8, 126.4, 126.3, 126.2, 125.9, 79.8, 77.5, 77.2, 76.7, 71.0, 66.7, 54.3, 54.2, 54.1, 36.8, 35.6, 21.7; ^{13}C NMR (acetone- d_6 , 300 MHz, ppm) of the two isomers δ 167.1, 166.2, 164.9, 164.7, 136.7, 134.9, 132.6, 132.2, 129.1, 128.9, 127.8, 127.5, 126.2(possibly overlapped), 126.1, 125.8, 79.8, 77.6, 77.5, 76.5, 70.9, 66.5, 53.9, 53.8(possibly two peaks), 53.6, 36.6, 35.4, 21.2, 21.1; MS (EI) m/z (%) 277($\text{M}^+ - \text{Cl}$), 245, 205, 147, 129, 115, 91, 86, 84(100), 59, 47; HRMS calcd for $\text{C}_{15}\text{H}_{17}\text{ClO}_5$: 277.1076($\text{M}^+ - \text{Cl}$); found: 277.1072.



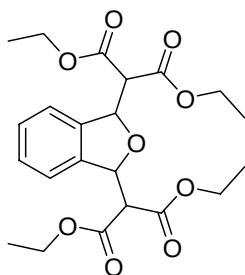
2-(3-Methylisochroman-1-yl)-2-nitro-1-phenylethanone (3m). The ratio of four diastereoisomers is 1:0.5:1.5:1. Isolated by flash column chromatography (hexane/ethyl acetate = 4:1, R_f = 0.5). IR (KBr) of the four isomers: $\nu_{\max}$ 1708, 1694, 1595, 1568, 1490, 1450, 1383, 1366, 1320, 1259, 1143, 1120, 1082, 1037, 999, 749, 692 cm^{-1} ; Isomer 1: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 6.67(dd, J = 9.2, 2 Hz, 1H); Isomer 2: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 6.48(dd, J = 7.6, 2 Hz, 1H), 3.88-3.84(m, 1H), 0.89(dd, J = 6.4, 2 Hz, 3H); Isomer 3: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 6.37(dd, J = 6, 2 Hz, 1H); Isomer 4: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 6.31(dd, J = 4.8, 2 Hz, 1H); Other overlapped peaks of four isomers: ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.94-7.81(m), 7.66-6.92(m), 5.99-5.84(m), 4.35-4.29(m), 3.78-3.69(m), 2.88-2.83(m), 2.75-2.47(m), 2.26-2.19(m), 1.31-1.28(m), 1.10-1.08(m); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) for the four isomers δ 188.1, 188.0, 187.3, 135.7, 135.6, 135.5, 135.3, 135.1, 135.0, 134.7, 134.1, 134.0, 133.7, 133.0, 132.2, 131.5, 130.8, 129.6, 129.5, 129.3, 129.2, 129.1, 129.0, 128.9, 128.7, 128.6, 128.4, 128.0, 127.9, 127.3, 127.1, 126.8, 126.6, 126.4, 125.3, 125.1, 124.5, 94.6, 94.3, 91.2, 90.9, 75.6, 75.3, 74.0, 73.8, 71.2, 70.9, 67.6, 66.7, 36.5, 36.1, 35.6, 35.3, 21.7, 21.6, 21.4, 20.5; MS (EI) m/z (%) 265($\text{M}^+ - \text{NO}_2$), 187, 147, 129, 115, 105(100), 91, 77, 51, 43; HRMS calcd for $\text{C}_{18}\text{H}_{17}\text{NO}_4$: 265.1229($\text{M}^+ - \text{NO}_2$); found: 265.1224.



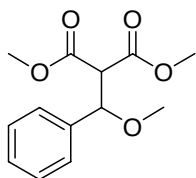
Dimethyl 2-(1,3-dihydroisobenzofuran-1-yl)malonate (3n). Isolated by flash column chromatography (hexane/ethyl acetate = 3:1, R_f = 0.5). IR (KBr): $\nu_{\max}$ 2952, 1758, 1732, 1475, 1460, 1437, 1364, 1317, 1300, 1253, 1198, 1154, 1047, 950, 749, 711 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.26-7.14(m, 4H), 5.82(d, J = 7.6 Hz, 1H), 5.10(dd, J = 12.8, 1.6 Hz, 1H), 5.00(d, J = 12.4 Hz, 1H), 3.68-3.66(m, 7H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 167.5, 167.4, 139.7, 138.6, 128.6, 127.7, 122.3, 121.3, 82.2, 73.2, 58.2, 52.9, 52.8; MS (EI) m/z (%) 250, 220, 205, 191, 159, 131, 119(100), 103, 91, 77, 65, 51, 39; HRMS calcd for $\text{C}_{13}\text{H}_{14}\text{O}_5$: 250.0841; found: 250.0845.



Diethyl 2-(1,3-dihydroisobenzofuran-1-yl)malonate (3o). Isolated by flash column chromatography (hexane/ethyl acetate = 4:1, R_f = 0.5). IR (KBr): $\nu_{\max}$ 2978, 2932, 1755, 1728, 1463, 1370, 1303, 1248, 1187, 1149, 1041, 848, 750 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.23-7.14(m, 4H), 5.83(d, J = 6.8 Hz, 1H), 5.10(d, J = 12 Hz, 1H), 5.01(d, J = 12 Hz, 1H), 4.14(q, J = 7.2 Hz, 4H), 3.65(d, J = 7.2 Hz, 1H), 1.16(t, J = 7.2 Hz, 6H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 167.1(two peaks), 139.7, 138.8, 128.5, 127.6, 122.4, 121.2, 82.2, 73.2, 61.8(two peaks), 58.5, 14.2(two peaks); MS (EI) m/z (%) 278, 229, 220, 205, 177, 159, 133, 119(100), 105, 91, 77, 51, 41; HRMS calcd for $\text{C}_{15}\text{H}_{18}\text{O}_5$: 278.1154; found: 278.1151.



Tetraethyl 2,2'-(1,3-dihydroisobenzofuran-1,3-diyl)dimalonate (3p). The ratio of two isomers is 1.5:1. Isolated by flash column chromatography (hexane/ethyl acetate = 3:1, R_f = 0.5). IR (KBr) of the two isomers: $\nu_{\max}$ 2985, 2939, 1758, 1732, 1460, 1444, 1372, 1299, 1252, 1185, 1153, 1097, 1036, 853, 758 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) of two isomers δ 7.25-7.20(m, 4H), 5.89(d, J = 6.4 Hz, aH), 5.82(d, J = 8 Hz, bH)[a+b=2], 4.25-4.09(m, 8H), 3.72(d, J = 7.2 Hz, 2H), 1.28-1.13(m, 12H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) of two isomers δ 167.0, 166.9, 166.8, 139.6, 139.4, 128.7, 128.6, 122.5, 122.4, 81.6, 81.5, 61.9, 61.8, 61.7, 59.2, 58.3, 14.3, 14.2, 14.1; MS (EI) m/z (%) 435(M^+ -H), 391, 317, 276(100), 243, 231, 203(100), 175, 159, 131(100), 115, 103, 91, 77, 59; HRMS calcd for $\text{C}_{22}\text{H}_{28}\text{O}_9$: 435.1655(M^+ -H); found: 435. 1663.



Dimethyl 2-(methoxy(phenyl)methyl)malonate (3q). Isolated by flash column chromatography (hexane/ethyl acetate = 4:1, R_f = 0.5). IR (KBr): $\nu_{\max}$ 2953, 2825, 1762, 1743, 1455, 1432, 1351, 1313, 1264, 1146, 1087, 1028, 976, 765, 699, 601 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.34-7.29(m, 5H), 4.77(d, J = 10 Hz, 1H), 3.78(s, 3H), 3.76(d, J = 10.4 Hz, 1H), 3.46(s, 3H), 3.17(s, 3H); ^{13}C NMR (CDCl_3 , 400 MHz, ppm) δ 167.9, 166.8, 137.9, 128.9, 128.7, 127.8, 81.9, 60.0, 57.1, 53.0, 52.6; MS (EI) m/z (%) 252, 237, 193, 161, 131, 121(100), 105, 91, 77, 59, 51; HRMS calcd for $\text{C}_{13}\text{H}_{16}\text{O}_5$: 252.0998; found: 252.1007.

Reference:

[1] The 3-Methylisochroman was prepared by the reaction of the corresponding homobenzylic alcohol with dimethoxymethane in the presence of BF_3 etherate or TMSOTf, see: DeNinno, M. P.; Perner, R. J.; Morton, H. E.; DiDomenico, S., Jr. *J. Org. Chem.* **1992**, *57*, 7115.

Active directory: /export/chem/13cdata
Sample directory: /export/chem/13cdata/13c-17-13
File: 13c17-13

Pulse sequence: zgpg30

Solvent: CDCl₃

Temperature: 300.2 K

Time: 0.00000000

Pulse delay: 1.000 sec

Pulse: 45.0 degrees

Mag: 100.0 MHz

Mag: 100.0 MHz

256 repetitions

Acquire: 13C, 100.625000 MHz

Time: 4.00, 200.741400 MHz

Continuously on

WALTZ-16

13C, 100.625000 MHz

13C, 100.625000 MHz

PT: 1.00000000

PT: 1.00000000

PT: 1.00000000

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S-15

220 200 180 160 140 120 100 80 60 40 20 0 ppm

Archive directory: /archive/chem/1/compens/1data
 Sample directory: comp/eq/1001000-4/15113
 File: C0000

Pulse sequence: zgpg30

Software: CDE13

Temperature: 300.2 K

INSTRUM: spect

Relax: delay 1.000 sec

Pulse: 45.0 degrees

Acq: 1.000 sec

Width: 2513.0 Hz

250 repetitions

DECEIVE: C13, 100.614000 MHz

PROBE: 5 mm QNP-1H/13

Power: 41 dB

continuously on

WALTZ-16, 200.140000 MHz

WALTZ-16, 200.140000 MHz

Lock: 100.614000 MHz

FT size: 15526

Total time: 10 min, 38 sec



220 200 180 160 140 120 100 80 60 40 20 0 ppm

S-17

Acquire directory: /data4/chem11/vawwly/data
 Sample directory: 20010_1207505

F1 file: PROTON

Pulse sequence: zgpg30

Solvent: CDCl₃
 NSI: none
 INOVA-400 "ONEWATER"

Relax. delay 1.00 sec

Pulse: zgpg30, zgpg30ac

NUC1: 13C, 13C

NUC2: 1H, 1H

WALTZ: 6000, 1.00 sec

6-CPD(110000)

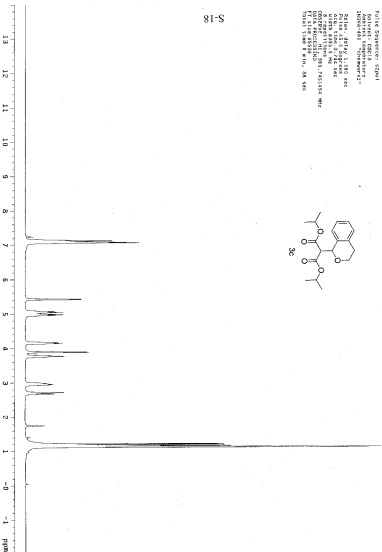
00020000, 01.000, 7461404 Hz

FT size 15528

Total time 8 min, 38 sec



S-18

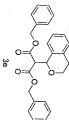


Archive directory: /usr/local/chem/113/chem113/0010
 Sample directory: 0086_1301003
 File: proton

Pulse sequence: zgpg30

solvent: CDCl3
 Name of the solvent: chloroform
 Name of the solvent: chloroform

Relax. delay: 1.000 sec
 Pulse: zgpg30
 Number of scans: 16
 Width: 6000 Hz
 8 repetitions
 Relaxation delay: 2.000000 sec
 Data processing
 FT size: 65535
 Total time: 0 min, 30 sec



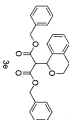
S-20



Pulse Sequence: zgpg30

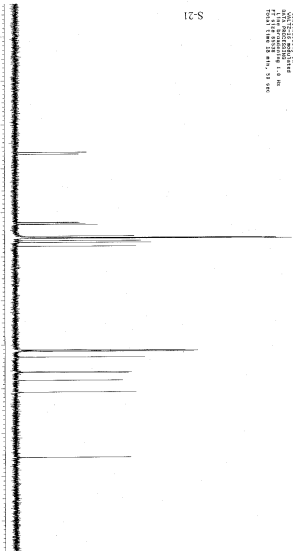
Solvent: CDCl3
 Sample Temperature: 300.2 K
 INVD=980 13C NMR=45

Relax. delay 1.000 sec
 Pulse delay 4.000 sec
 Delay 1.000 sec
 Width 1013.6 Hz
 255 repetitions
 OBSERVE C13, 140.518132 MHz
 PULSE C13, 140.518132 MHz
 Power 41.00 W
 Continuously on
 VOLTAGE 200.000000 V
 Q1 1.000000
 Q2 1.000000
 Line broadening 1.0 Hz
 FT SIZE 95936
 Total time 15 min, 39 sec



S-21

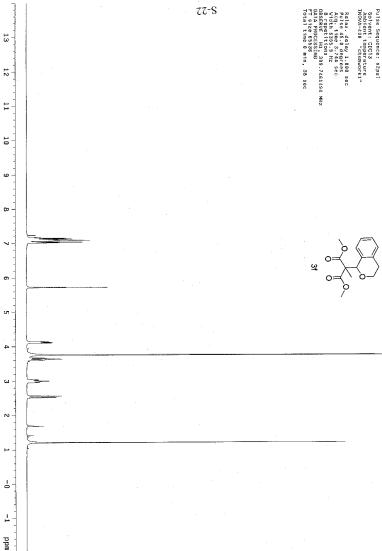
220 200 180 160 140 120 100 80 60 40 20 0 ppm



Acquire directory: /data1/0000/11/00000000/00000000
 Name: 11-00000000_00000000_00000000
 File: 00000000

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Sample Temperature: 25.00
 INNOVATION: "Chemical"

Relax. delay 1.000 sec
 Pulse pr. 8.000000
 Width 500.0 Hz
 8 repetitions
 Observed: 101.255, 76.8334 MHz
 100.00000000000000
 FT size 65536
 Total time 0 min, 30 sec



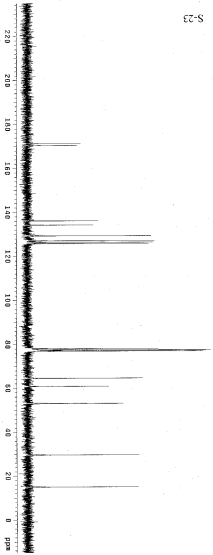
Date: Tue 4/16/2008 7:40:21 PM
 Sample directory: 07807-2_12012008
 File: C08008

Pulse Sequence: zgpg30
 Acquisition Date: 4/16/2008
 Acquisition Time: 12:01:20
 INVO: 480 °C, 100 MHz

Relax delay: 1.000 sec
 Pulse: zgpg30, zgpg30
 Delay: 1.000 sec
 Width: 20.00 Hz
 Size: 1024
 Resolution: 0.19 Hz
 Frequency: 125.761 MHz
 Power: 41.00
 Continuity: 1.00
 Delay: 1.000 sec
 Delay: 1.000 sec
 Line broadening: 1.0 Hz
 F2 size: 65536
 Total time: 18 min, 58 sec



S-23




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Archive directory: /export/home/114/venkays/data
Sample directory: 021861-1_150612605
File: PROTON
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put the sequence in a2pro1

DO NOT WRITE IN THESE SPACES
 Student Name: _____
 Date: _____

Refers to delay 1.000 sec
 between 45th character

[illegible]

Q What is the date?
ANSWER 12-20-76
Q What is the time?
ANSWER 11:00

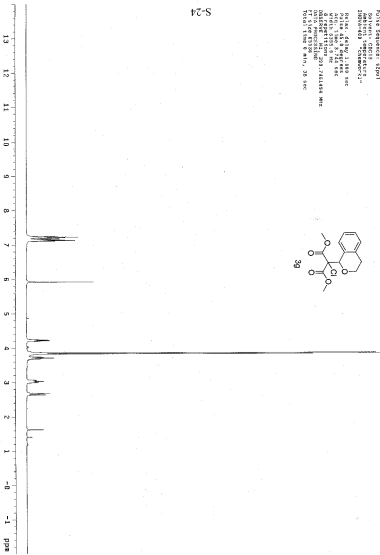
PT 4 sec 873206
Total time 6 min, 35 sec

$$S$$

-24-

[illegible]

13 12



DATA Collected on: 0001-09-26/56/310
 Archive directory: /temp/1/name/56/56w/56/56a
 Sample directory:
 File: 000500

Pulse Sequence: zgpg30

Solvent: acetone

Temp: 22.2 C / 295.4 K

Pulse: zgpg30 1.00 sec

Pulse: 45.8 degrees

45.8 198.1 615 sec

90deg 100.0 1.9 sec

2.000 100.0 1.9 sec

0001000 013.0 0.75 458404 sec

0001000 013.0 0.75 458404 sec

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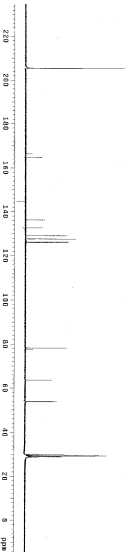
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0001000 013.0 0.75 458404 sec



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 Sample directory: 01/01/2001/000000-000000
 File: PROTON

Pulse sequence: zgpg30

Solvent: CDCl3

Temperature: 300.2 K

INSTR: spect

Pulse delay: 1.00 sec

Pulse: zgpg30

Acquisition: 0.00 sec

Width: 6550.0 Hz

Frequency: 300.13 MHz

Processing: 0.00 sec

Observed: 0.00 sec

FT size: 65500

Total time: 8 min, 30 sec



S-26

13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm

active directory: /sage/6/786/1/5/chem/3/6/active
 active directory: 07/10/2012_15012385
 File: carbon

Pulse Sequence: zgpg30

Solvent: CDCl₃
 Name: 3-nitro-4,5,6,7-tetrahydro-2H-chromene
 Name: 3n

Pulse delay 1.00 sec
 Pulse 45.0 degrees
 128 repetitions
 width 2323.5 Hz
 OBSERVE CH: 103.146132 MHz
 power 45.00 W
 continuously on
 Waltz-16 decoupled
 0.0000000000000000
 Line broadening 1.0 Hz
 FT size 65536
 Total time 16 min, 36 sec



8-27

220 200 180 160 140 120 100 80 60 40 20 0 ppm

Active directory: /export/home/jjw/psf/data
 Sample directory: 01181-1_143/1285
 File: PROTON

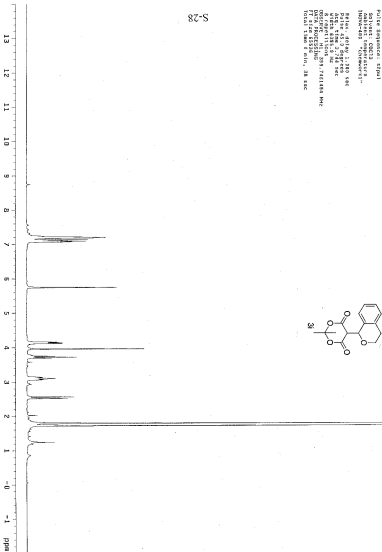
Pulse sequence: zgpg30

Solvent: CDCl3
 Solvent temperature:
 INSTRUM: spect

Relax: delay 1.200 sec
 D1 1.20000000
 AQC time 0.284 sec
 WDEL 0.050 sec
 R 1.000113205
 0.000000000
 DATA PROCESSING
 FT size 65536
 Total time 8 min, 38 sec



S-28



Active directory: /usr/local/lib/chemsys/data
 Sample directory: W1131-E_14311285

File: COBOL

Pulse Program: STD01

Polymat: COBOL
 Analytical temperature
 120000-000 "Chemomart"

Pulse delay: 1.081 sec

Pulse delay: 1.081 sec

Pulse delay: 1.081 sec

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Pulse delay: 1.081 sec

Pulse delay: 1.081 sec

Pulse delay: 1.081 sec

Pulse delay: 1.081 sec

Pulse delay: 1.081 sec



S-29

220 200 180 160 140 120 100 80 60 40 20 0 ppm

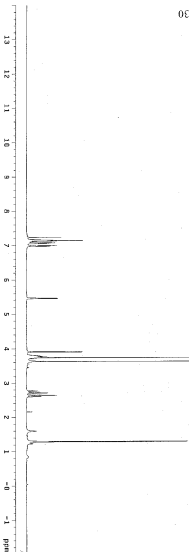
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 Solvent temperature: 25°C
 Probe: 5 mm QNP-1H/13C
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 d1: 2.00 s
 Temperature: 300.2 K
 Pressure: 1.00 bar
 Acquisition time: 0.10000000
 F2: 125.7618 MHz
 F1: 500.1360 MHz
 AQ: 327.359 s
 SI: 327.359 s
 SC: 1.00000000
 SD: 0.00000000
 SE: 0.00000000
 SF: 0.00000000
 SS: 0.00000000
 SWH: 6553.600 MHz
 FIDRES: 0.34498 Hz
 AQRES: 0.34498 Hz
 SFO: 500.1360 MHz
 SF1: 125.7618 MHz
 SF2: 125.7618 MHz
 SF3: 125.7618 MHz
 SF4: 125.7618 MHz
 SF5: 125.7618 MHz
 SF6: 125.7618 MHz
 SF7: 125.7618 MHz
 SF8: 125.7618 MHz
 SF9: 125.7618 MHz
 SF10: 125.7618 MHz
 SF11: 125.7618 MHz
 SF12: 125.7618 MHz
 SF13: 125.7618 MHz
 SF14: 125.7618 MHz
 SF15: 125.7618 MHz
 SF16: 125.7618 MHz
 SF17: 125.7618 MHz
 SF18: 125.7618 MHz
 SF19: 125.7618 MHz
 SF20: 125.7618 MHz
 SF21: 125.7618 MHz
 SF22: 125.7618 MHz
 SF23: 125.7618 MHz
 SF24: 125.7618 MHz
 SF25: 125.7618 MHz
 SF26: 125.7618 MHz
 SF27: 125.7618 MHz
 SF28: 125.7618 MHz
 SF29: 125.7618 MHz
 SF30: 125.7618 MHz
 SF31: 125.7618 MHz
 SF32: 125.7618 MHz
 SF33: 125.7618 MHz
 SF34: 125.7618 MHz
 SF35: 125.7618 MHz
 SF36: 125.7618 MHz
 SF37: 125.7618 MHz
 SF38: 125.7618 MHz
 SF39: 125.7618 MHz
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 SF68: 125.7618 MHz
 SF69: 125.7618 MHz
 SF70: 125.7618 MHz
 SF71: 125.7618 MHz
 SF72: 125.7618 MHz
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 SF96: 125.7618 MHz
 SF97: 125.7618 MHz
 SF98: 125.7618 MHz
 SF99: 125.7618 MHz
 SF100: 125.7618 MHz
 SF101: 125.7618 MHz
 SF102: 125.7618 MHz
 SF103: 125.7618 MHz
 SF104: 125.7618 MHz
 SF105: 125.7618 MHz
 SF106: 125.7618 MHz
 SF107: 125.7618 MHz
 SF108: 125.7618 MHz
 SF109: 125.7618 MHz
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 SF160: 125.7618 MHz
 SF161: 125.7618 MHz
 SF162: 125.7618 MHz
 SF163: 125.7618 MHz
 SF164: 125.7618 MHz
 SF165: 125.7618 MHz
 SF166: 125.7618 MHz
 SF167: 125.7618 MHz
 SF168: 125.7618 MHz
 SF169: 125.7618 MHz
 SF170: 125.7618 MHz
 SF171: 125.7618 MHz
 SF172: 125.7618 MHz
 SF173: 125.7618 MHz
 SF174: 125.7618 MHz
 SF175: 125.7618 MHz
 SF176: 125.7618 MHz
 SF177: 125.7618 MHz
 SF178: 125.7618 MHz
 SF179: 125.7618 MHz
 SF180: 125.7618 MHz
 SF181: 125.7618 MHz
 SF182: 125.7618 MHz
 SF183: 125.7618 MHz
 SF184: 125.7618 MHz
 SF185: 125.7618 MHz
 SF186: 125.7618 MHz
 SF187: 125.7618 MHz
 SF188: 125.7618 MHz
 SF189: 125.7618 MHz
 SF190: 125.7618 MHz
 SF191: 125.7618 MHz
 SF192: 125.7618 MHz
 SF193: 125.7618 MHz
 SF194: 125.7618 MHz
 SF195: 125.7618 MHz
 SF196: 125.7618 MHz
 SF197: 125.7618 MHz
 SF198: 125.7618 MHz
 SF199: 125.7618 MHz
 SF200: 125.7618 MHz
 SF201: 125.7618 MHz
 SF202: 125.7618 MHz
 SF203: 125.7618 MHz
 SF204: 125.7

```
Relax delay 1.000 sec  
Pulse 47.8 degrees  
Acq time 5.724 sec  
Width 6595.9 Hz  
16 channels  
Observed at -287.746404 MHz  
Data processed on  
at time 65536  
Total time 1 min, 16 sec
```

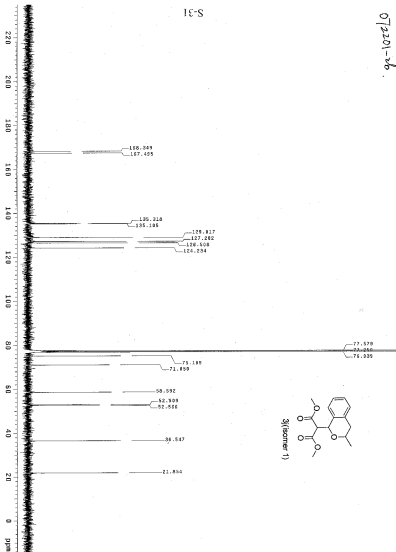


21(isomer 1)



072201-26.

S-31



Active directory: J:\spect\8000\11\acmryr\data
 Sample directory: 012281-1_24011985
 File: PROTON

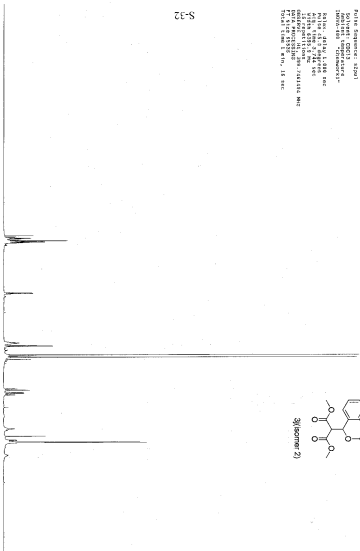
Pulse sequence: zgpg1
 SOLVENT: CDCl3
 NS: 1024
 DS: 4
 SWH: 6000.000 MHz
 FIDRES: 0.001300 Hz
 AQ: 0.1960000 sec
 RG: 327.5
 IS: 16 repetitions
 ACQ: 012281-1_24011985
 NAME: PROTON
 FT size: 65520
 Total time: 1 min, 15 sec



3v (isomer 2)

S-32

13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm



Active directory: \\sccwr1\share\gc\data
 Sample directory: 97251-46_15011503
 File: C0000

Pulse sequence: zgpg30

Solvent: CDCl3
 Acquired: 05/04/99
 Name: 46a - CHM000003

Pulse delay: 1.000 sec
 Pulse: 45.0 deg/sec
 Relaxation delay: 1.000 sec
 Width: 2512.0 Hz
 SFOFF: 100.6261500 MHz
 OBSERVE: C13, 100.6261500 MHz
 AQUEOUS: 100.6261500 MHz
 Power: 41 dB
 continuously on
 MULTIZ: 16, 160.0000000 MHz
 160.0000000 MHz
 Live broadening: 1.0 Hz
 FT size: 65536
 Total time: 2 hr, 6 min, 1 sec



3 (isomer 2)

220 200 180 160 140 120 100 80 60 40 20 0 ppm

8-33

acquire directory: /home/134user/spc/data
 sample directory: /home/134user/spc/data
 file: 070000

pulse sequence: zgpg30

solvent: CDCl3

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

name: 134user

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name: 134user

S-34



Archive directory: /asport/home/11/sonnerj/data
 Sample directory: /jsteward/eva/0195
 File: C19508

Pulse sequence: zgpg30

Solvent: CDCl3

Ambient temperature

INSTRUM: spect

Relax delay: 1.000 sec

Acq time: 0.189 sec

Width: 2115.5 Hz

3800 repetitions

Decouple: no

Power: 41.95

Continuously on

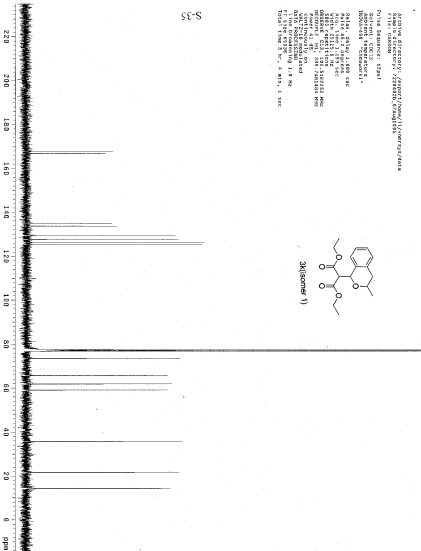
1 line breaking 1.0 Hz

Total time: 3 hr, 4 min, 1 sec



3k[isomer 1]

S-35



archive directory: /srapc/hsap/11/overage/data
 NAME directory: srapc_1_0301000
 FILE: PROTIN

Polite sequence: srapc

Solvent: CDCl₃
 Temperature: 25.00°C
 INOVA-100 100MHzCL

Pulse sequence: zgpg30
 Relax: delay 1.500 sec
 Pulse: dcl, 8 degrees
 Acquire: 600.13 MHz
 Width: 6385.3 Hz
 S: repetitions
 OBSERVE: nu1 250.1361364 MHz
 P1: 12.0000000 sec
 P2: 12.0000000 sec
 FT size: 65536
 Total time: 0 min, 30 sec



13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm

Data Collected on: 03/06/2003
 Archive directory: /export/home/ajay/newsp/2003
 Sample directory:
 8161 C0000

Pulse Sequence: zgpg30

Solvent: Acetone

Temp: 22.3 C / 245.4 K

Pulse: zgpg30 1.00 sec

Acq: 1000 1.015 sec

Width: 1600 1.8 Hz

RG: 655 1.000

DS: 4 1.000

OS: 16 1.000

RECORD: 12 200 0.025 sec

Power: 35 dB

CG: 1.000 1.000

WALTZ-16: 1.000 1.000

DATA PROCESSING

FT size: 131872

10000: 1000 20 Hz

S-39



Sample directory: /home/1/chem/1/1246955/data
 Sample directory: 071517_1_25011985
 File: PROTON

Pulse sequence: zgpg30

SOI Name: CDE1A
 NO Name: CDE1A
 INOVA-400 "Chemical"

Pulse: delay 1.000 sec

Pulse: 45.0 degrees

Width: 6380.3 Hz

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

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Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

Q: 0.00000000

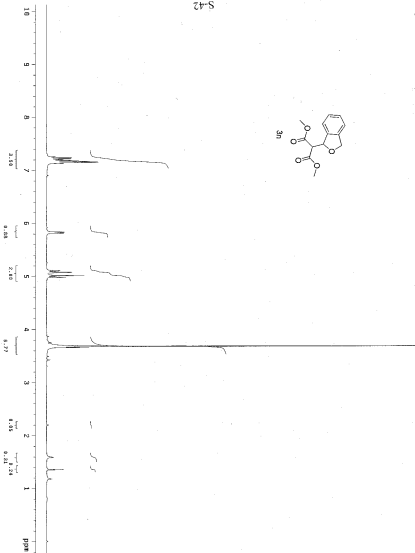
Q: 0.00000000

Q: 0.00000000



S-41

13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm





220 200 180 160 140 120 100 80 60 40 20 0 ppm

187.666
187.489

139.651
135.637

126.580

127.713

123.251

122.298

82.164

77.587

77.267

76.946

73.816

56.150

52.925

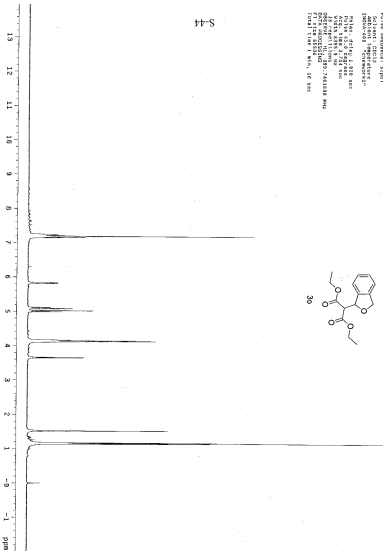
52.835

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient Temperature:
 INMR-400 Chemshifts:
 Relaxation delay: 1.010 sec
 Pulse delay: 0.060000 sec
 Acq. time: 3.734 sec
 F2: 100.625130 MHz
 F1: 500.136091 MHz
 OBSERVE: 1H, 350.765100 MHz
 PULPROG: zgpg30
 Total time: 1 min, 36 sec



30

S-44



Archive directory: /export/home/1/chemsys/data
 Sample directory: 755001b_1samples

File: C:\MSWIN

Pulse sequence: zgpg1

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

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Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

Relaxation delay: 1.000 sec

Number of scans: 128

Acquisition time: 1.000 sec

S-45



active directory: /export/home/11/nmr/sy/data
sample directory: 780202b_namg2e85

file: 00000

pulse sequence: zgpg30

solvent: CDCl3

acquisition temperature

INSTRUMENT: spect

probe: zgpg30

pulse delay: 1.000 sec

pulse: 45.0 degrees

acq. time: 1.18 sec

width: 25135.8 Hz

offset: 0.000000 MHz

observed: C13 100.618235 MHz

DECOUPLE: 1H 395.748104 MHz

power: 0.1 dB

acquisition: 0.000000 sec

DATA PROCESSING

line broadening: 1.0 Hz

frequency: 100.618235 MHz

total time: 3 hr, 4 min, 1 sec



3p

S-46

220 200 180 160 140 120 100 80 60 40 20 0 ppm

Pulse Sequence: zgpg30

SOIvent: CDCl3

SOIvent: CDCl3

INOVA-800 125MHzX13

Relax: delay 1.000 sec

Pulse: 45.0 degree

Width: 6380.0 Hz

16 repetitions

Acquire: 01.100.700000 Hz

FT size: 65536

Total time 1 min, 16 sec



S-48

13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm

Pulse sequence: zgpg30

Acquire date: 000113
 Acquire time: 13:05:05
 Instrument: spect
 INOVA-400 13C NMR

Relax. delay: 1.000 sec

Pulse: zgpg30

Power: 40.00 dB

Width: 20.00 dB

Size: 1000000

Resolution: 0.180 Hz

Frequency: 100.626 MHz

Power: 40.00 dB

Consistency: 99.99%

Acquire date: 000113

Acquire time: 13:05:05

PT size: 65536

Total time: 10 min, 58 sec



S-50

220 200 180 160 140 120 100 80 60 40 20 0 ppm

Active directory: J:\MSDC1\2005\11\04\MS-SP\data
 Sample directory: 070602-1_20051205
 File: C00101

Pulse Sequence: zgpg30

NUC1: 13C
 NUC2: 1H
 solvent: CDCl3
 INSTR: spect
 FID: 160 "Chemshifts"

Relax: delay 1.000 sec
 Pulse: 40.0 dB/90deg
 Acq: 10.000 sec
 Width: 2025.6 Hz
 SIZ: repetitions
 SFO: 125.760 MHz
 SFO2: 500.136 MHz
 P1: 12.00 sec
 Power: 41.00 W
 Continuously on
 Lock: 10.000 sec
 LYNA: Locking 1.0 Hz
 FT size: 65536
 Total time: 10 min, 50 sec



15-8

