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SUPPORTING INFORMATION

Title: Highly Efficient Direct Alkylation of Activated Methylene by Cycloalkanes
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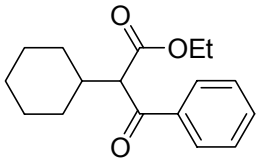
Ref. No.: O200700686

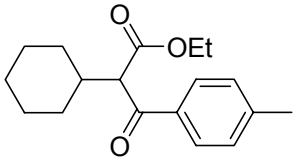
Experimental details and characterization data for all new compounds

General information: ^{1}H NMR spectra were recorded on 400 MHz spectrometer in $CDCl_3$ 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; ddd, doublet of doublet of doublet; m, multiplet; q, quartet; dq, doublet of quartet. The coupling constants, *J*, are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at 100 MHz and referenced to the internal solvent signals (central peak is 77.00 ppm). MS data were obtained by Varian Saturn 2100D GC/MS Spectrometer. HRMS were made by McGill University. IR spectra were recorded by a Nexus 670 Avator FTIR Spectrometer. Flash column chromatography was performed over SORBENT silica gel 30-60 μm. Thin layer chromatography was performed by using Sorbent Silica Gel 60 F_{254} TLC plates and visualized with ultraviolet light. 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.

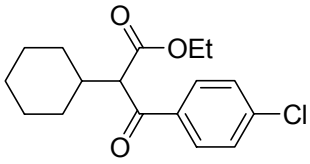
General procedure: To a mixture of ethyl benzoylacetate (107 mg, 0.5 mmol) and $FeCl_2{\cdot}4H_2O$ (20 mg, 0.1 mmol), cyclohexane (1.07 mL, 10 mmol) and *tert*-butyl peroxide (0.19 mL, 1.0 mmol) were added by syringe. Then the reaction mixture was stirred at 100°C for 12hrs under nitrogen atmosphere. After that, the resulting mixture was filtered through a short silica gel in a pipette eluting with methylene chloride. The solvent was evaporated and the residue was purified by column separation (eluting with hexane/methylene chloride = 2:1); to give the desired product **1c** (122 mg, 88%).



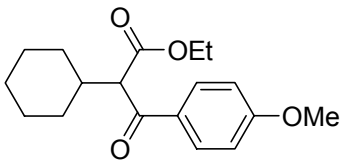
Ethyl 2-cyclohexyl-3-oxo-3-phenylpropanoate (3a). IR (liquid): ν_{max} 2980, 2930, 2853, 1734, 1684, 1597, 1581, 1448, 1288, 1241, 1155, 1131, 1028, 1001 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 8.03-8.01(m, 2H), 7.60-7.56(m, 1H), 7.49-7.45(m, 2H), 4.17(d, J = 9.6 Hz, 1H), 4.13(q, J = 7.2 Hz, 2H), 2.44-2.34(m, 1H), 1.80-1.72(m, 2H), 1.70-1.60(m, 3H), 1.40-1.24(m, 2H), 1.21-1.06(m, 5H), 1.00-0.88(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 195.0, 169.2, 137.3, 133.7, 128.9, 128.7, 61.4, 60.8, 38.5, 31.6, 31.0, 26.4, 26.2(two peaks), 14.3; MS (EI) *m/z* (%) 275, 192, 146, 120, 105(100); HRMS calcd for $C_{17}H_{22}O_3$: 274.1570; found: 274.1567.



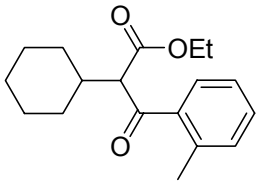
Ethyl 2-cyclohexyl-3-oxo-3-p-tolylpropanoate (3b). IR (liquid): ν_{max} 2980, 2928, 2853, 1734, 1684, 1607, 1573, 1448, 1290, 1152, 1030, 1003 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 7.94-7.91(m, 2H), 7.28-7.25(m, 2H), 4.15(d, J = 9.6 Hz, 1H), 4.12(q, J = 7.2 Hz, 2H), 2.43-2.33(m, 4H), 1.81-1.70(m, 2H), 1.69-1.59(m, 3H), 1.40-1.22(m, 2H), 1.21-1.06(m, 5H), 0.97-0.86(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 194.5, 169.3, 144.6, 134.9, 129.6, 128.9, 61.3, 60.7, 38.4, 31.6, 31.1, 26.4, 26.2(two peaks), 21.8, 14.3; MS (EI) *m/z* (%) 289, 207, 135, 120(100); HRMS calcd for $C_{18}H_{24}O_3$: 288.1725; found: 288.1735.



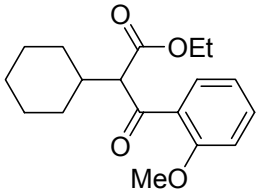
Ethyl 3-(4-chlorophenyl)-2-cyclohexyl-3-oxopropanoate (3c). IR (liquid): ν_{max} 2979, 2928, 2854, 1734, 1690, 1589, 1570, 1448, 1400, 1287, 1093, 1000 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 7.99-7.95(m, 2H), 7.46-7.43(m, 2H), 4.14(q, J = 7.2 Hz, 2H), 4.11(d, J = 10 Hz, 1H), 2.43-2.32(m, 1H), 1.80-1.71(m, 2H), 1.70-1.61(m, 3H), 1.38-1.23(m, 2H), 1.21-1.08(m, 5H), 0.98-0.88(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 193.7, 169.0, 140.2, 135.6, 130.2, 129.2, 61.5, 60.9, 38.4, 31.6, 31.0, 26.3, 26.2, 26.1, 14.3; MS (EI) *m/z* (%) 309, 220, 177, 140(100), 106, 81; HRMS calcd for $C_{17}H_{21}ClO_3$: 308.1179; found: 308.1175.



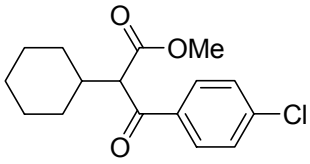
Ethyl 2-cyclohexyl-3-(4-methoxyphenyl)-3-oxopropanoate (3d). Register Number [58987-20-5]. IR (liquid): ν_{max} 2985, 2930, 2853, 1740, 1678, 1598, 1575, 1511, 1449, 1421, 1264, 1174, 1030, 997 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 8.04-8.00(m, 2H), 6.97-6.93(m, 2H), 4.13(q, J = 7.2 Hz, 2H), 4.12(d, J = 10.4 Hz, 1H), 3.87(s, 3H), 2.42-2.32(m, 1H), 1.80-1.71(m, 2H), 1.70-1.60(m, 3H), 1.40-1.23(m, 2H), 1.21-1.02(m, 5H), 0.96-0.86(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 193.3, 169.4, 164.0, 131.1, 130.4, 114.1, 61.3, 60.5, 55.7, 38.4, 31.6, 31.1, 26.4, 26.2(two peaks), 14.3; MS (EI) *m/z* (%) 305, 257, 223, 135(100).



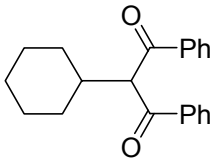
Ethyl 2-cyclohexyl-3-oxo-3-o-tolylpropanoate (3e). IR (liquid): ν_{max} 2979, 2928, 2853, 1734, 1695, 1600, 1576, 1448, 1292, 1239, 1155, 1030 cm^{-1}; ^1H NMR (400 MHz, ppm) δ 7.73(d, J = 7.6 Hz, 1H), 7.37(ddd, J = 7.6, 7.6, 1.2 Hz, 1H), 7.29-7.23(m, 2H), 4.11(q, J = 7.2 Hz, 2H), 4.06(d, J = 9.6 Hz, 1H), 2.49(s, 3H), 2.39-2.29(m, 1H), 1.78-1.62(m, 5H), 1.39-1.26(m, 2H), 1.20-0.95(m, 6H); ^{13}C NMR (100 MHz, ppm) δ 198.4, 169.2, 138.9, 138.2, 132.2, 131.8, 128.9, 125.9, 63.3, 61.3, 38.7, 31.7, 30.9, 26.4(possibly overlapped), 26.3, 21.3, 14.3; MS (EI) *m/z* (%) 289, 228, 207, 161, 132, 120(100), 87; HRMS calcd for $C_{18}H_{24}O_3$: 288.1725; found: 288.1722.



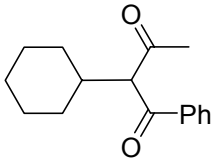
Ethyl 2-cyclohexyl-3-(2-methoxyphenyl)-3-oxopropanoate (3f). IR (liquid): ν_{max} 2979, 2928, 2854, 1739, 1680, 1598, 1580, 1486, 1294, 1246, 1024 cm^{-1}; ^1H NMR (400 MHz, ppm) δ 7.64(dd, J = 7.6, 1.6 Hz, 1H), 7.45(ddd, J = 7.6, 7.6, 1.6 Hz, 1H), 6.99(ddd, J = 7.6, 7.6, 1.2 Hz, 1H), 6.94(d, J = 8.4 Hz, 1H), 4.18-4.08(m, 3H), 3.87(s, 3H), 2.35-2.25(m, 1H), 1.78-1.61(m, 5H), 1.35-1.23(m, 2H), 1.21-1.03(m, 6H); ^{13}C NMR (100 MHz, ppm) δ 197.4, 169.6, 158.4, 133.9, 130.7, 129.0, 121.0, 111.6, 64.6, 60.9, 55.4, 38.8, 31.9, 30.8, 26.5, 26.4(possibly overlapped), 14.4; MS (EI) *m/z* (%) 305, 257, 230, 205, 176, 136(100); HRMS calcd for $C_{18}H_{24}O_4$: 304.1675; found: 304.1680.



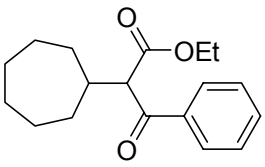
Methyl 3-(4-chlorophenyl)-2-cyclohexyl-3-oxopropanoate (3g). IR (liquid): ν_{max} 2929, 2854, 1740, 1690, 1589, 1570, 1400, 1286, 1093, 998 cm^{-1}; ^1H NMR (400 MHz, ppm) δ 7.98-7.95(m, 2H), 7.47-7.43(m, 2H), 4.14(d, J = 10 Hz, 1H), 3.67(s, 3H), 2.44-2.32(m, 1H), 1.79-1.70(m, 2H), 1.70-1.60(m, 3H), 1.39-1.24(m, 2H), 1.21-1.05(m, 2H), 0.98-0.86(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 193.7, 169.4, 140.3, 135.5, 130.2, 129.3, 60.7, 52.6, 38.5, 31.6, 31.1, 26.3, 26.2, 26.1; MS (EI) *m/z* (%) 295, 212, 180, 139(100), 111; HRMS calcd for $C_{16}H_{19}ClO_3$: 294.1023; found: 294.1027.



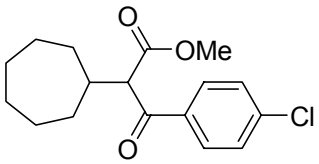
2-Cyclohexyl-1,3-diphenylpropane-1,3-dione (3h). Register Number [891821-75-2]. ^1H NMR (400 MHz, ppm) δ 8.01-7.99(m, 4H), 7.56-7.51(m, 2H), 7.45-7.41(m, 4H), 5.16(d, J = 9.2 Hz, 1H), 2.72-2.62(m, 1H), 1.74-1.62(m, 5H), 1.40-1.25(m, 2H), 1.21-1.02(m, 3H); ^{13}C NMR (100 MHz, ppm) δ 195.8, 137.4, 133.6, 129.0, 128.9, 64.2, 40.1, 32.1, 26.5, 26.4; MS (EI) *m/z* (%) 307, 281, 249, 223, 201, 147, 105(100).



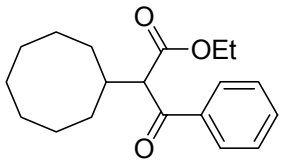
2-Cyclohexyl-1,3-diphenylpropane-1,3-dione (3i). Register Number [891821-74-2]. ^{1}H NMR (400 MHz, ppm) δ 8.02-7.99(m, 2H), 7.61-7.56(m, 1H), 7.50-7.45(m, 2H), 4.32(d, J = 10 Hz, 1H), 2.50-2.40(m, 1H), 2.15(s, 3H), 1.80-1.71(m, 1H), 1.70-1.57(m, 4H), 1.38-1.25(m, 2H), 1.21-1.02(m, 2H), 0.92-0.83(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 204.8, 196.6, 137.7, 133.9, 129.0, 128.9, 71.2, 39.3, 31.6, 31.1, 27.7, 26.3, 26.2(two peaks); MS (EI) *m/z* (%) 245, 201, 161, 147, 120, 105(100).



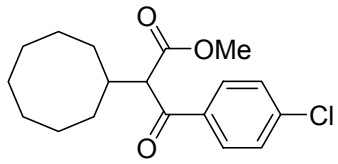
Ethyl 2-cycloheptyl-3-oxo-3-phenylpropanoate (3j). IR (liquid): ν_{max} 2979, 2927, 2856, 1734, 1688, 1597, 1581, 1462, 1448, 1291, 1147, 1027 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 8.04-8.01(m, 2H), 7.60-7.55(m, 1H), 7.49-7.45(m, 2H), 4.28(d, J = 9.2 Hz, 1H), 4.13(dd, J = 7.2, 1.2 Hz, 2H), 2.62-2.53(m, 1H), 1.78-1.32(m, 11H), 1.29-1.20(m, 1H), 1.17(t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, ppm) δ 195.2, 169.4, 137.3, 133.6, 128.9, 128.8, 61.4, 61.0, 39.8, 32.8, 32.1, 28.3(two peaks), 26.7(two peaks), 14.3; MS (EI) *m/z* (%) 289, 243, 223, 192, 147, 120, 105(100); HRMS calcd for $C_{18}H_{24}O_3$: 288.1725; found: 288.1720.



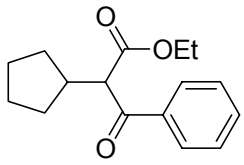
Methyl 3-(4-chlorophenyl)-2-cycloheptyl-3-oxopropanoate (3k). IR (liquid): ν_{max} 2926, 2855, 1739, 1688, 1588, 1571, 1461, 1400, 1287, 1215, 1093, 1012 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 7.98-7.95(m, 2H), 7.47-7.43(m, 2H), 4.24(d, J = 9.6 Hz, 1H), 3.67(s, 3H), 2.60-2.51(m, 1H), 1.75-1.18(m, 12H); ^{13}C NMR (100 MHz, ppm) δ 193.9, 169.7, 140.3, 135.5, 130.2, 129.3, 60.9, 52.6, 39.9, 32.7, 32.2, 28.3, 28.2, 26.6(possibly overlapped); MS (EI) *m/z* (%) 309, 278, 249, 213, 178, 139(100), 107; HRMS calcd for $C_{17}H_{21}ClO_3$: 308.1179; found: 308.1176.



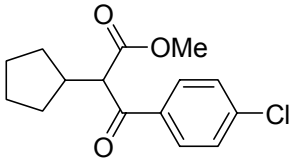
Ethyl 2-cyclooctyl-3-oxo-3-phenylpropanoate (3l). IR (liquid): ν_{max} 2979, 2922, 2852, 1734, 1688, 1597, 1580, 1472, 1448, 1283, 1151, 1033 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 8.04-8.01(m, 2H), 7.59-7.54(m, 1H), 7.49-7.44(m, 2H), 4.28(d, J = 9.6 Hz, 1H), 4.13(dq, J = 7.2, 1.6 Hz, 2H), 2.70-2.60(m, 1H), 1.73-1.26(m, 14H), 1.18(t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, ppm) δ 195.3, 169.5, 137.4, 133.6, 128.9, 128.8, 61.5, 61.4, 38.0, 31.2, 30.1, 26.9, 26.8, 26.6, 25.8, 25.6, 14.3; MS (EI) *m/z* (%) 303, 194, 120, 106(100); HRMS calcd for $C_{19}H_{26}O_3$: 302.1182; found: 302.1177.



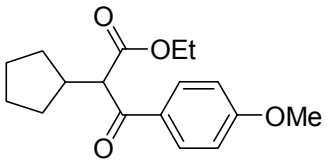
Methyl 3-(4-chlorophenyl)-2-cyclooctyl-3-oxopropanoate (3m). IR (liquid): ν_{max} 2922, 2852, 1739, 1690, 1588, 1571, 1441, 1400, 1283, 1092, 1012 cm^{-1}; ^1H NMR (400 MHz, ppm) δ 7.99-7.95(m, 2H), 7.47-7.43(m, 2H), 4.24(d, J = 10 Hz, 1H), 3.67(s, 3H), 2.69-2.60(m, 1H), 1.76-1.26(m, 14H); ^{13}C NMR (100 MHz, ppm) δ 194.0, 169.7, 140.3, 135.5, 130.2, 129.3, 61.3, 52.6, 38.0, 31.0, 30.6, 26.9, 26.7, 26.6, 25.7, 25.5; MS (EI) *m/z* (%) 323, 291, 245, 213, 139(100), 107; HRMS calcd for $C_{18}H_{23}ClO_3$: 322.1336; found: 322.1329.



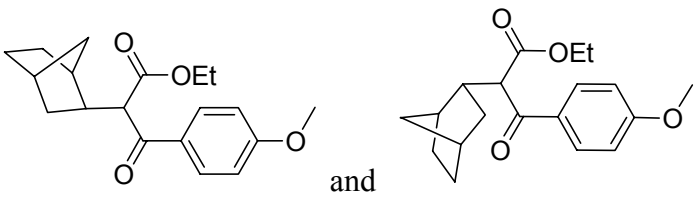
Ethyl 2-cyclopentyl-3-oxo-3-phenylpropanoate (3n). IR (liquid): ν_{max} 2957, 2870, 1734, 1688, 1598, 1581, 1448, 1294, 1218,1032 cm^{-1}; ^1H NMR (400 MHz, ppm) δ 8.04-8.01(m, 2H), 7.60-7.56(m, 1H), 7.50-7.46(m, 2H), 4.16(d, J = 10 Hz, 1H), 4.13(dq, J = 7.2, 2.4 Hz, 2H), 2.79-2.68(m, 1H), 1.93-1.81(m, 2H), 1.72-1.53(m, 4H), 1.38-1.31(m, 1H), 1.70(t, J = 7.2 Hz, 3H), 1.08-0.98(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 195.0, 169.7, 137.0, 133.6, 128.9, 128.8, 61.5, 60.2, 40.1, 31.4, 30.9, 25.2, 25.0, 14.2; MS (EI) *m/z* (%) 261, 217, 192, 151, 120, 105(100); HRMS calcd for $C_{16}H_{20}O_3$: 260.1412; found: 260.1410.



Methyl 3-(4-chlorophenyl)-2-cyclopentyl-3-oxopropanoate (3o). IR (liquid): ν_{max} 2954, 2870, 1740, 1690, 1589, 1570, 1400, 1270, 1218, 1093 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 7.99-7.95(m, 2H), 7.47-7.43(m, 2H), 4.14(d, J = 10.4 Hz, 1H), 3.67(s, 3H), 2.78-2.67(m, 1H), 1.93-1.78(m, 2H), 1.70-1.53(m, 4H), 1.36-1.24(m, 1H), 1.06-0.97(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 193.7, 169.9, 140.3, 135.2, 130.3, 129.3, 60.0, 52.7, 40.1, 31.3, 30.9, 25.1, 25.0; MS (EI) *m/z* (%) 281, 249, 212, 139(100), 111; HRMS calcd for $C_{15}H_{17}ClO_3$: 280.0853; found: 280.0858.

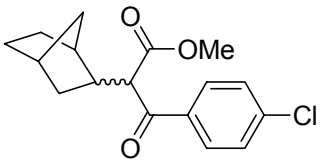


Ethyl 2-cyclopentyl-3-(4-methoxyphenyl)-3-oxopropanoate (3p). Register number [58987-19-2]. IR (liquid): ν_{max} 2957, 2870, 2840, 1740, 1684, 1598, 1576, 1511, 1464, 1420, 1263, 1175, 1031 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 8.05-8.01(m, 2H), 6.97-6.93(m, 2H), 4.17-4.08(m, 3H), 3.87(s, 3H), 2.79-2.68(m, 1H), 1.93-1.79(m, 2H), 1.71-1.52(m, 4H), 1.37-1.23(m, 1H), 1.18(t, J = 7.2 Hz, 3H), 1.08-0.98(m, 1H); ^{13}C NMR (100 MHz, ppm) δ 193.3, 169.9, 164.0, 131.2, 130.0, 114.1, 61.4, 60.0, 55.7, 40.1, 31.4, 30.9, 25.2, 25.0, 14.3; MS (EI) *m/z* (%) 291, 223, 167, 136(100), 101;

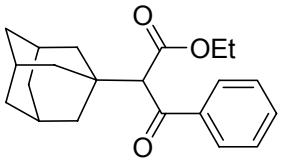


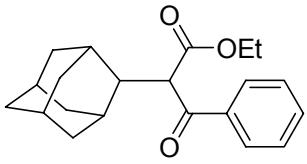
and

Ethyl 2-(bicyclo[2.2.1]heptan-2-yl)-3-oxo-3-phenylpropanoate (3q). Register number of first isomer is [891821-70-8]. The ratio of two isomers is 1:1. IR (liquid) of the two isomers: ν_{max} 2954, 2912, 2871, 1739, 1688, 1597, 1581, 1449, 1368, 1292, 1225, 1032 cm^{-1}; ^1H NMR (400 MHz, ppm) of the two isomers δ 8.06-8.00(m, 4H), 7.61-7.54(m, 2H), 7.50-7.44(m, 4H), 4.20-4.06(m, 6H), 2.54-2.45(m, 2H), 2.28-2.24(m, 1H), 2.20-2.16(m, 1H), 2.13-2.10(m, 1H), 1.85-1.82(m, 2H), 1.62-1.09(m, 20H), 0.93-0.86(m, 1H); ^{13}C NMR (100 MHz, ppm) of the two isomers δ 195.2, 194.7, 169.4, 169.1, 137.2(two peaks), 133.7(two peaks), 129.0, 128.9(possibly overlapped), 128.8, 61.5(possibly overlapped), 60.5, 60.2, 42.3(possibly overlapped), 40.1, 39.6, 37.3, 37.0, 36.9, 36.0(two peaks), 35.8, 30.1(possibly overlapped), 28.7(possibly overlapped), 14.3, 14.2; MS (EI) *m/z* (%) 287, 240, 192, 106(100).



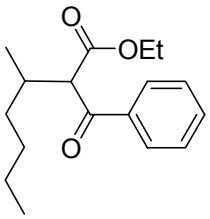
Methyl 2-(bicyclo[2.2.1]heptan-2-yl)-3-(4-chlorophenyl)-3-oxopropanoate (3r). The ratio of two isomers is 1:1. IR (liquid) of the two isomers: ν_{max} 2953, 2912, 2871, 1739, 1684, 1588, 1570, 1456, 1401, 1285, 1092, 983 cm^{-1}; ^1H NMR (400 MHz, ppm) of the two isomers δ 8.00-7.94(m, 4H), 7.48-7.42(m, 4H), 4.10(d, J = 11.2 Hz, 1H), 4.08(d, J = 11.6 Hz, 1H), 3.68(s, 3H), 3.65(s, 3H), 2.52-2.44(m, 2H), 2.29-2.25(m, 1H), 2.21-2.18(m, 1H), 2.10-2.07(m, 1H), 1.81-1.78(m, 1H), 1.61-1.10(m, 14H), 0.89-0.83(m, 2H); ^{13}C NMR (100 MHz, ppm) δ 193.9, 193.4, 169.7, 169.4, 140.4, 140.3, 135.4(two peaks), 130.3(two peaks), 129.3(two peaks), 60.3, 59.9, 52.8, 52.7, 42.3(two peaks), 40.1, 39.6, 37.3, 37.0, 36.8, 36.1, 36.0, 35.8, 30.1(possibly overlapped), 28.6(two peaks); MS (EI) *m/z* (%) 307, 271, 249, 212, 140(100), 107, 90; HRMS calcd for $C_{17}H_{19}ClO_3$: 306.1023; found: 306.1018.

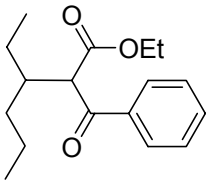


(**3s**) and


(**3t**)

(3s and 3t). Register number of **3s** [572915-11-8]. The NMR ratio of these two compounds is 4:1. IR (liquid) of the mixture of two compounds: ν_{max} 2972, 2906, 2849, 1745, 1692, 1596, 1581, 1447, 1145, 1033 cm^{-1}; ^1H NMR (400 MHz, ppm) of major compound **3s** δ 7.98-7.95(m, 2H), 4.23(s, 1H), 4.13(dq, J = 7.2, 2 Hz, 2H); ^1H NMR (400 MHz, ppm) of minor compound **3t** δ 8.09-8.06(m, 2H), 4.86(d, J = 11.6 Hz, 1H), 2.86(d, J = 11.6 Hz, 1H); ^1H NMR (400 MHz, ppm) of other overlapped peaks δ 7.60-7.54(m), 7.50-7.44(m), 2.10-1.60(m), 1.22-1.14(m); ^{13}C NMR (100 MHz, ppm) of **3s** δ 195.1, 168.1, 138.8, 133.3, 128.9, 128.5, 62.9, 61.0, 40.5, 37.7, 36.9, 28.8, 14.4; ^{13}C NMR (100 MHz, ppm) of **3t** δ 194.6, 169.2, 137.5, 133.7, 129.0, 128.8, 61.5, 56.3, 44.6, 39.1, 38.8, 38.2, 32.2, 32.1, 30.4, 29.9, 28.0, 27.8, 14.3; MS (EI) of **3s** *m/z* (%) 326, 308, 279, 253, 235, 225, 195, 175, 135, 105(100), 91; MS (EI) of **3t** *m/z* (%) 326, 279, 252, 235, 223, 193, 105(100), 91; HRMS calcd for $C_{17}H_{26}O_3$: 326.1882; found: 326.1887.



(**3u**) and


(**3v**)

The NMR ratio of **3u** and **3v** is 1:2. IR (liquid) of the mixture of two compounds: ν_{max} 2961, 2933, 2873, 2851, 1734, 1690, 1597, 1581, 1448, 1284, 1157, 1033 cm^{-1}; ^1H NMR (400 MHz, ppm) of minor compound **3u** δ 4.41-4.37(m, 1H), 2.47-2.38(m, 1H), 1.03-1.01(m, 3H); ^1H NMR (400 MHz, ppm) of major compound **3v** δ 4.22-4.19(m, 1H), 2.57-2.47(m, 1H),; ^1H NMR (400 MHz, ppm) of other overlapped peaks δ 8.04-7.98(m), 7.60-7.56(m), 7.49-7.45(m), 4.17-4.10(m), 1.43-1.15(m), 0.93-0.81(m); ^{13}C NMR (100 MHz, ppm) of the mixture of **3u** and **3v** δ 195.2, 195.1(two peaks), 169.5(two peaks), 169.4, 169.3, 137.4, 137.3(two peaks), 137.2, 133.6(three peaks), 128.9(possibly overlapped), 128.8, 128.7(two

peaks), 61.4, 61.3(possibly overlapped), 60.7, 60.4, 57.9, 57.8, 39.3, 39.2, 34.8, 34.1, 33.8, 33.6, 32.8, 32.6, 29.3, 29.0, 23.6, 23.3, 22.9, 22.8, 19.9, 19.7, 17.7, 17.1, 14.5, 14.4, 14.2(possibly overlapped), 14.1, 10.8, 10.6; MS (EI) of **3u** *m/z* (%) 277, 247, 192, 146, 120, 105(100), 91; MS (EI) of **3v** *m/z* (%) 277, 230, 192, 146, 120, 105(100), 91; HRMS calcd for $C_{17}H_{24}O_3$: 276.1725; found: 276.1721.