

Supporting Information

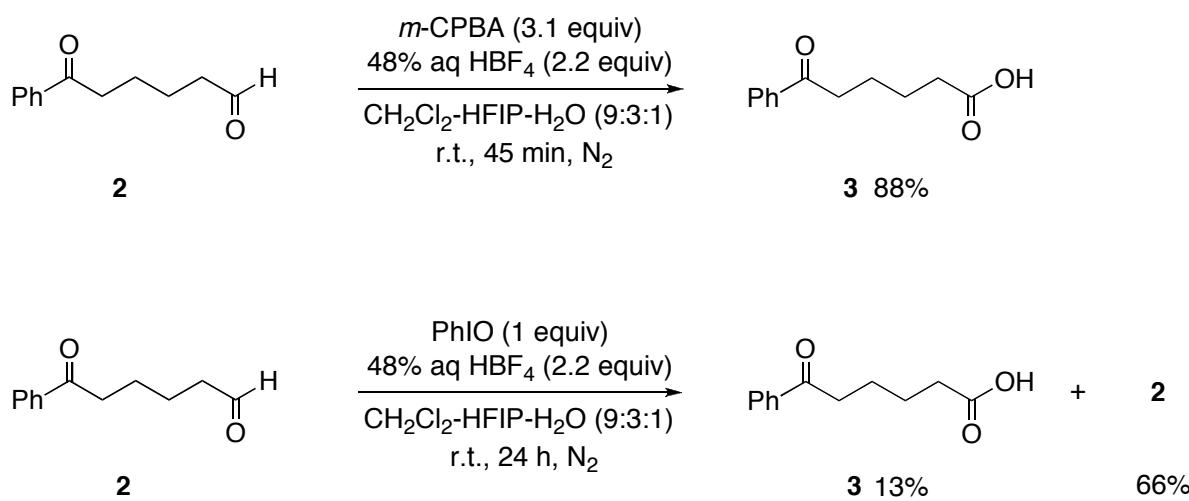
Iodomethylene-Catalyzed Oxidative Cleavage of Carbon-Carbon Double and Triple Bonds Using *m*-Chloroperbenzoic Acid as a Terminal Oxidant

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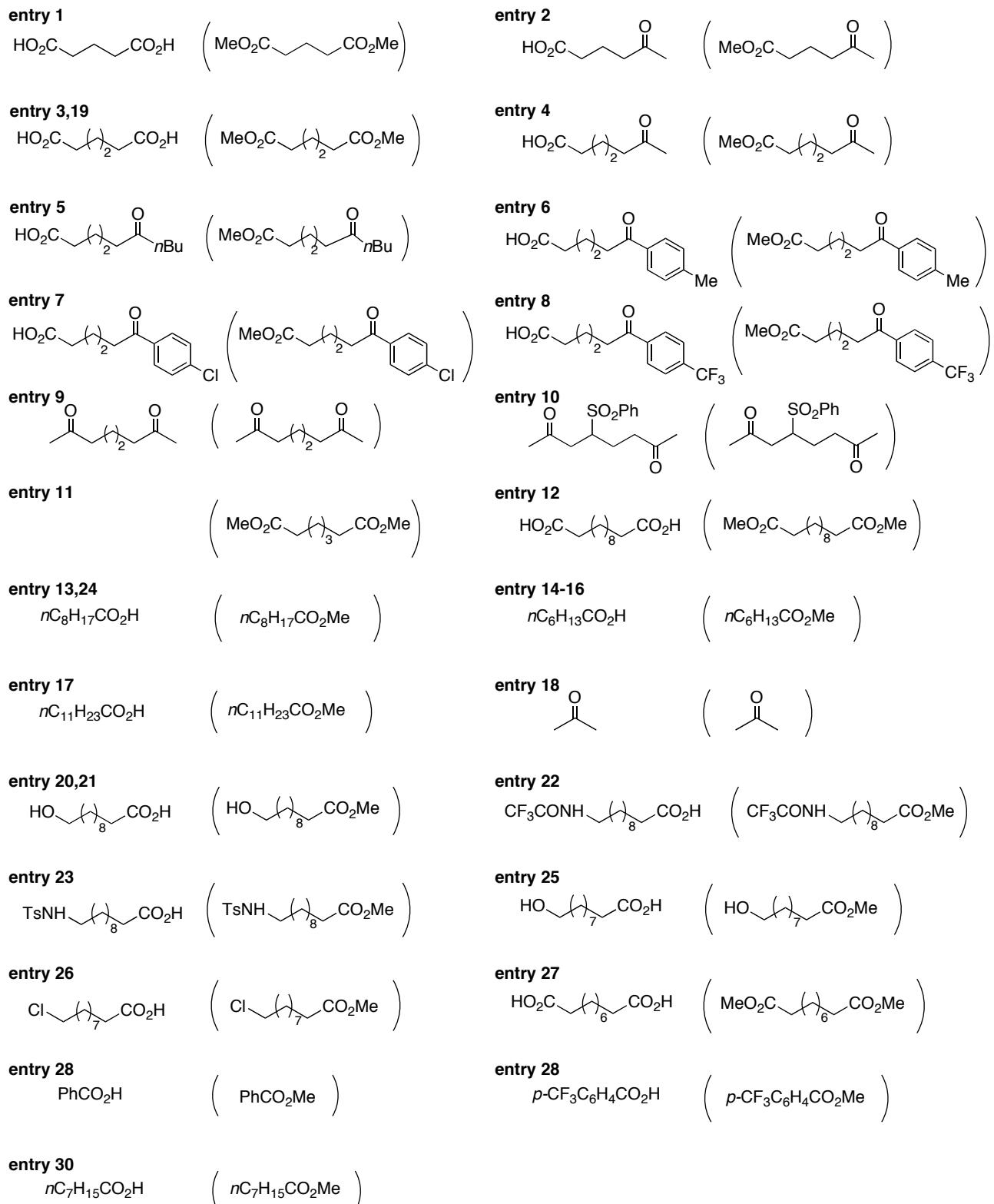
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Scheme S1. Oxidation of Keto Aldehyde **2** to Carboxylic Acid **3**^a



^a Oxidation of aldehyde **2** with *m*-CPBA under acidic conditions is fast, while that with iodosylbenzene is slow.

Scheme S2. Structures of the Products in Table 2



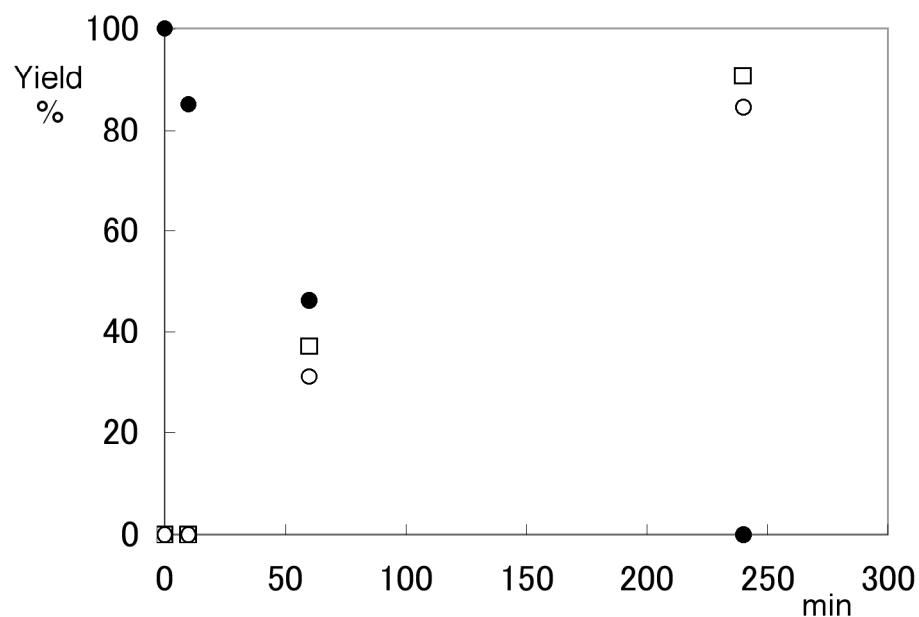
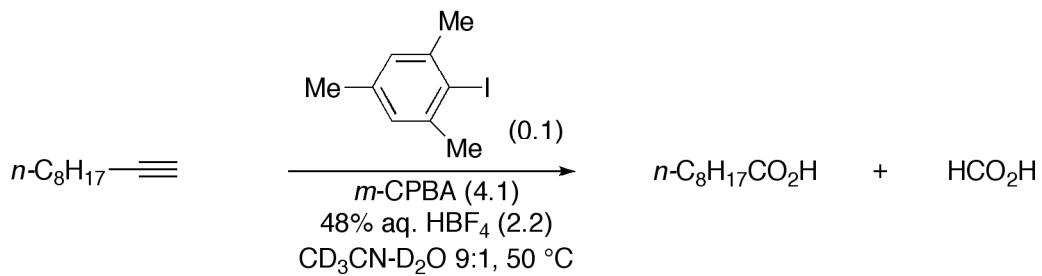


Figure S1. Time course for oxidative cleavage of 1-decyne: yields of (●) 1-decyne, (□) nonanoic acid, and (○) formic acid, measured by ^1H NMR.

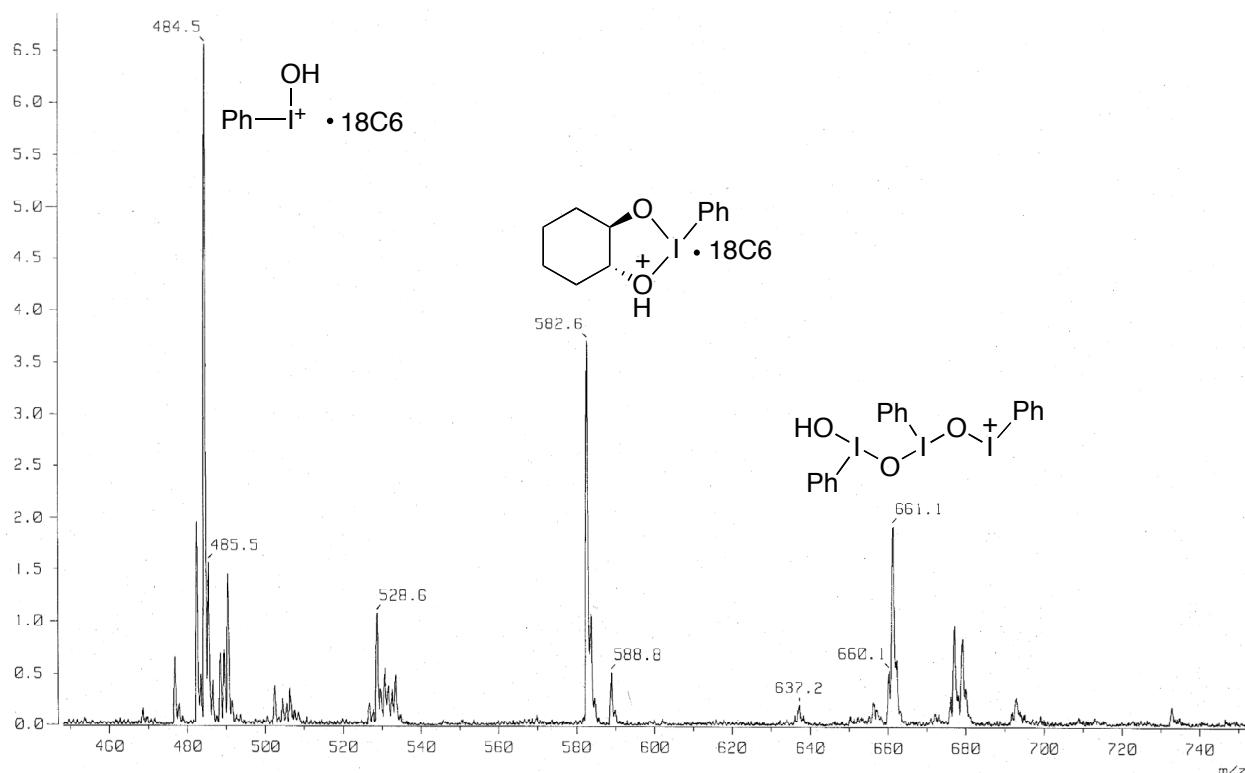
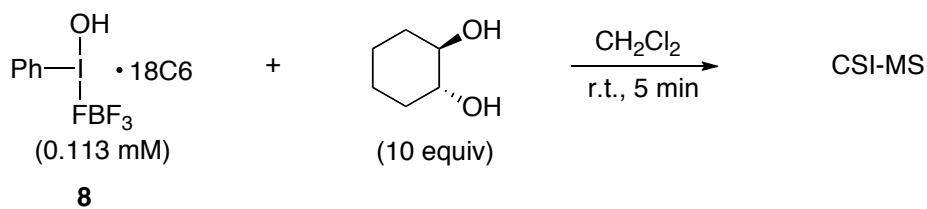


Figure S2. CSI-MS spectrum of a mixture of $\text{PhI(OH)}\text{BF}_4\cdot 18\text{-crown-6}$ (**8**) (0.113 mM) and *trans*-1,2-cyclohexanediol (10 equiv) in CH_2Cl_2 at room temperature, measured after 5 min. The complex **8** as well as the oxidant **5** prepared *in situ* undergo oxidative cleavage of carbon-carbon double bonds. See: Miyamoto, K.; Tada, N.; Ochiai, M. *J. Am. Chem. Soc.* **2007**, 129, 2772.

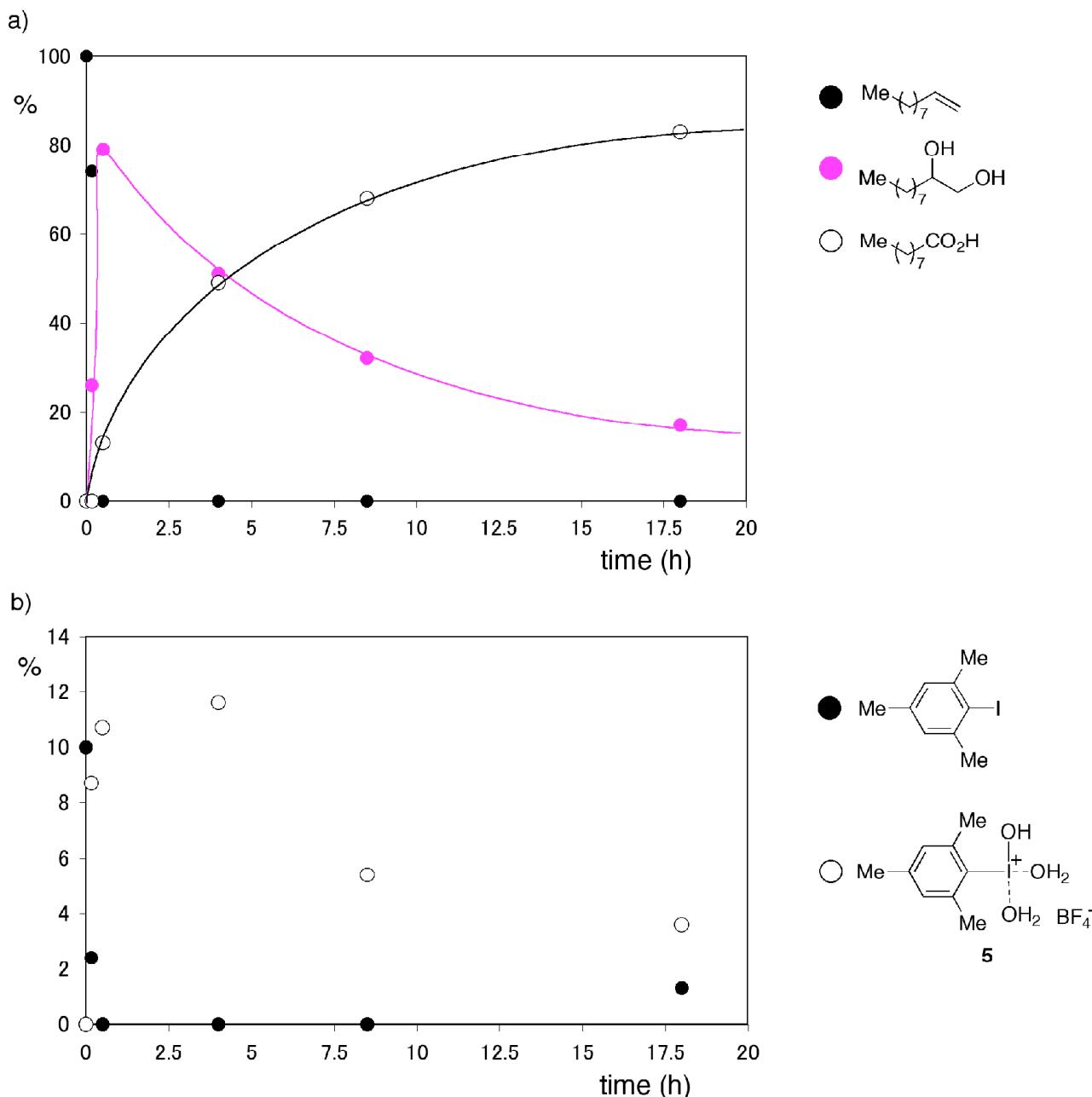
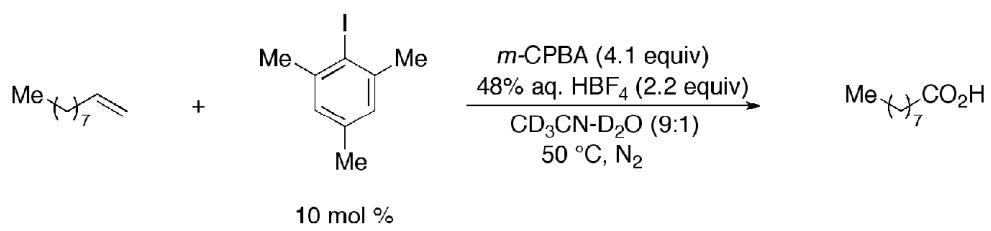


Figure S3. Time course for the catalytic cleavage of 1-decene with method B, measured by ^1H NMR: a) 1-decene, decane-1,2-diol, and nonanoic acid; b) iodomesitylene and hydroxy- λ^3 -iodane **5**.

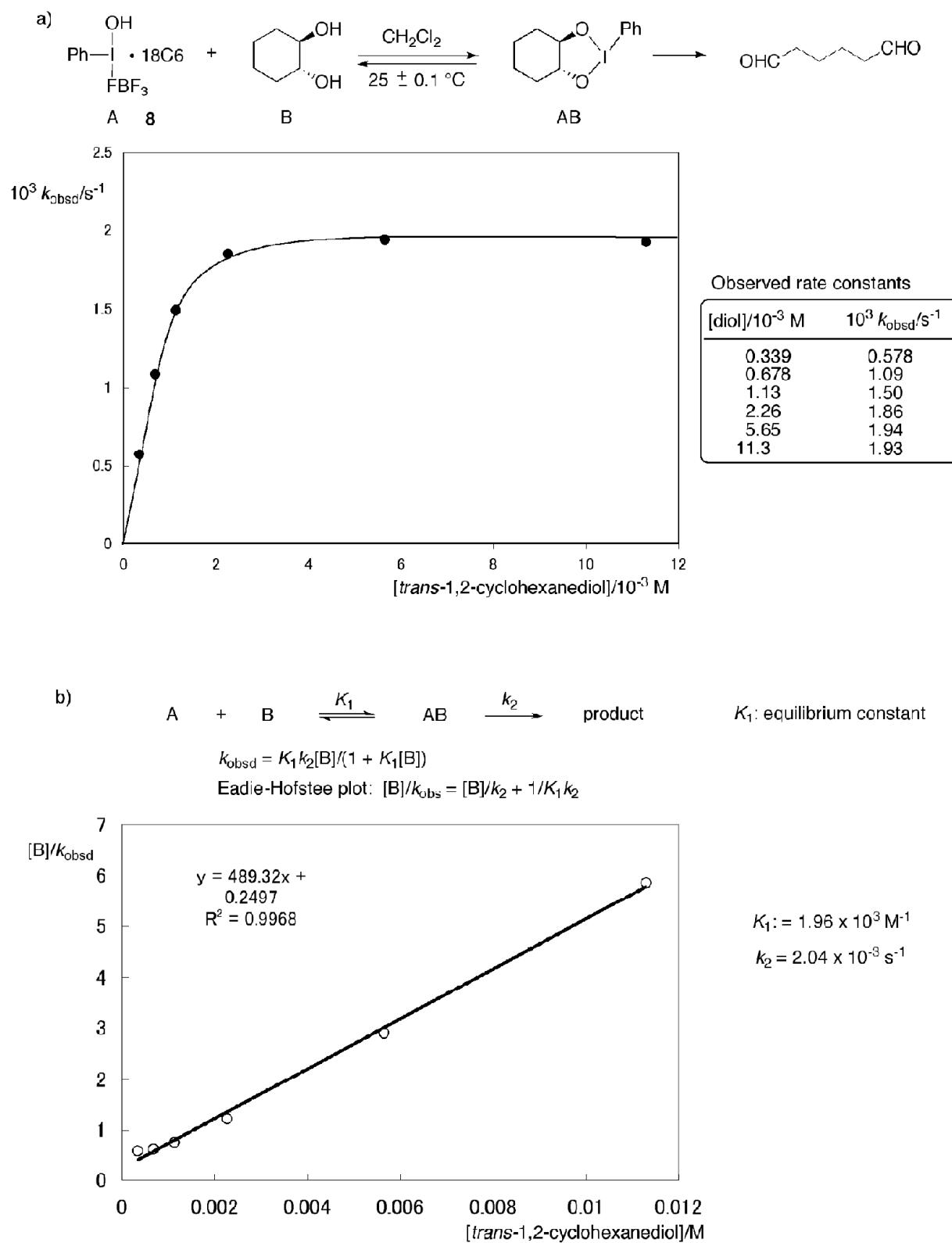


Figure S4. a) Observed rate constants k_{obsd} for oxidative cleavage of a glycol with $\text{PhI(OH)}\cdot 18\text{-crown-6}$ (**8**) ($0.113 \times 10^{-3} \text{ M}$) in dichloromethane at 25°C plotted against concentrations of *trans*-1,2-cyclohexanediol; b) Kinetic analysis using Eadie-Hofstee plot. The complex **8** as well as the oxidant **5** prepared *in situ* undergo oxidative cleavage of carbon-carbon double bonds. See: Miyamoto, K.; Tada, N.; Ochiai, M. *J. Am. Chem. Soc.* **2007**, *129*, 2772.

General Information. IR spectra were recorded on Perkin Elmer 1720 FT-IR spectrometers. ¹H NMR and ¹³C NMR spectra were obtained on a JEOL JNM-AL300 or JNM-AL400 spectrometers. Chemical shifts (δ) are reported in parts per million (ppm) downfield from internal Me₄Si. Mass spectra (MS) were obtained on either a JEOL JMX-SX 102A, Waters LCT Premier, or SHIMADZU Model GCMS-QP 505 spectrometer. Preparative thin-layer chromatography (TLC) was carried out on precoated plates of silica gel (MERCK, silica gel F-254). Melting points were determined with a Yanaco micro melting points apparatus and are uncorrected.

General Procedure for Iodomethylene-Catalyzed Oxidative Cleavage of Olefins (Method A). A Typical Example (Table 1, entry 12). *m*-Chloroperbenzoic acid (72% purity, 74 mg, 0.31 mmol) was dried under vacuum for 15 min at room temperature prior to use. To a stirred solution of *m*-CPBA in dichloromethane (0.82 mL), 1,1,1,3,3,3-hexafluoro-2-propanol (0.27 mL), and water (91 μ L) was added a 48% (wt.) aqueous solution of tetrafluoroboric acid (29 μ L, 0.22 mmol), 0.11 M dichloromethane solution of 2,4,6-trimethyliodobenzene (9 μ L, 0.001 mmol), and then 1-phenyl-1-cyclohexene (**1**) (16 mg, 0.10 mmol) at room temperature under nitrogen and the mixture was stirred for 14 h. The reaction mixture was poured into water and extracted with dichloromethane four times. The combined organic phases were dried over Na₂SO₄, filtered, and concentrated at 0 °C under aspirator vacuum to give a mixture of 6-oxo-6-phenylhexanoic acid (**3**) (96%) and adipic acid monophenyl ester (**4**) (<1%). The yields were determined by ¹H NMR. The mixture was treated with (trimethylsilyl)diazomethane (2 M solution in hexane, 0.3 mL, 0.6 mmol) in benzene (31 mL) and methanol (9 mL) at room temperature for 1 h. The reaction mixture was evaporated under aspirator vacuum to give an oil, which was purified by preparative TLC (ethyl acetate-hexane, 2:8) to give methyl 6-phenyl-6-oxohexanoate (19.4 mg, 88%) as a colorless oil: ¹IR (neat) 2951, 1736, 1684, 1448, 1363, 1261, 1209, 1176, 1001, 692 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.96 (dd, *J* = 8.7, 1.4 Hz, 2H), 7.56 (tt, *J* = 7.4, 1.4 Hz, 1H), 7.46 (dd, *J* = 8.7, 7.4 Hz, 2H), 3.68 (s, 3H), 3.01 (t, *J* = 7.1 Hz, 2H), 2.39 (t, *J* = 7.1 Hz, 2H), 1.84-1.68 (m, 4H); MS *m/z* (relative intensity) 220 (0.5%, M⁺), 143 (12), 120 (35), 105 (100), 77 (46), 55 (10), 51 (16). The authentic sample of **4**² was prepared by the reaction of adipoyl chloride with phenol and obtained as colorless needles: mp 101.5-102 °C (recrystallized from benzene-hexane); IR (KBr) 3430-2430, 1757, 1697, 1591, 1132 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.37 (dd, *J* = 8.8, 7.7 Hz, 2H), 7.23 (tt, *J* = 7.7, 1.2 Hz, 1H), 7.08 (dd, *J* = 8.8, 1.2 Hz, 2H), 2.61 (t, *J* = 7.4 Hz, 2H), 2.44 (t, *J* = 7.4 Hz, 2H), 1.88-1.73 (m, 4H).

General Procedure for Iodomethylene-Catalyzed Oxidative Cleavage of Olefins (Method B). A Typical Example (Table 2, entry 12). *m*-CPBA (72% purity, 98 mg, 0.41 mmol) was dried under vacuum for 15 min at room temperature prior to use. To a stirred solution of *m*-CPBA in acetonitrile (0.82 mL) and water (91 μ L) was added a 48% (wt.) aqueous solution of tetrafluoroboric acid (29 μ L, 0.22 mmol), 0.81 M dichloromethane solution of 2,4,6-trimethyliodobenzene (12 μ L, 0.01 mmol), and then cyclododecene (*cis:trans* = 70:30, 17 mg, 0.1 mmol) at room temperature under nitrogen and the mixture was heated at 50 °C for 10 h. After cooling, the mixture was poured into water and extracted with dichloromethane four times. The combined organic phases were dried over Na₂SO₄, filtered, and concentrated under aspirator vacuum to give dodecanedioic acid (77%) as a white solid. The yield was determined by ¹H NMR. To a solution of the solid in methanol (3 mL) was added H₂SO₄ (0.2 mL), and the mixture was refluxed with stirring for 2 h. The mixture was poured into water and extracted with dichloromethane four times. The combined organic phases were dried over Na₂SO₄, filtered, and

concentrated under aspirator vacuum to give an oil, which was purified by preparative TLC (ethyl acetate-hexane, 1:9) to give dimethyl 1,12-dodecanedioate (16.8 mg, 65%) as a colorless powder.³ mp 32-32.5 °C (recrystallized from hexane); IR (neat) 2918, 2848, 1741, 1464, 1437, 1412, 1176, 883, 735 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.67 (s, 6H), 2.31 (t, J = 7.3 Hz, 4H), 1.62 (quint, J = 7.3 Hz, 4H), 1.37-1.23 (m, 12H); MS *m/z* (relative intensity) 259 [7%, (M+1)⁺], 227 (33), 98 (29), 84 (21), 74 (51), 69 (40), 59 (77), 55 (75), 41 (100).

Dimethyl Glutarate (Table 2, entry 1):⁴ a colorless oil; IR (neat) 2954, 1739, 1439, 1319, 1203, 1174, 1066, 1024, 995 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.68 (s, 6H), 2.39 (t, J = 7.3 Hz, 4H), 1.96 (quint, J = 7.3 Hz, 2H); MS *m/z* (relative intensity) 161 (<1%, M⁺), 129 (65), 100 (84), 87 (25), 59 (100), 55 (76).

Methyl 5-Oxohexanoate (Table 2, entry 2):⁵ a colorless oil; IR (neat) 2954, 1734, 1716, 1437, 1362, 1254, 1200, 1155, 1001 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.68 (s, 3H), 2.51 (t, J = 7.1 Hz, 2H), 2.35 (t, J = 7.1 Hz, 2H), 2.15 (s, 3H), 1.90 (quint, J = 7.1, 2H); MS *m/z* (relative intensity) 144 (2%, M⁺), 129 (2), 112 (34), 101 (8), 85 (18), 74 (38), 59 (47), 43 (100).

Dimethyl Hexanedioate (Table 2, entry 3):⁶ a colorless oil; IR (neat) 2954, 1739, 1437, 1367, 1173, 1082 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.68 (s, 6H), 2.38-2.25 (m, 4H), 1.72-1.61 (m, 4H); MS *m/z* (relative intensity) 175 [<1%, (M+1)⁺], 143 (28), 114 (56), 101 (51), 74 (34), 59 (100), 55 (75).

Methyl 6-Oxoheptanoate (Table 2, entry 4):⁷ a colorless oil; IR (neat) 2951, 1736, 1714, 1437, 1363, 1254, 1198, 1173, 1086 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.67 (s, 3H), 2.46 (t, J = 7.0 Hz, 2H), 2.33 (t, J = 7.0 Hz, 2H), 2.14 (s, 3H), 1.68-1.55 (m, 4H); MS *m/z* (relative intensity) 158 (2%, M⁺), 143 (1), 126 (28), 101 (40), 87 (37), 81 (27), 59 (58), 43 (100).

Methyl 6-Oxodecanoate (Table 2, entry 5):⁸ a colorless oil; IR (neat) 2954, 2933, 2871, 1738, 1714, 1437, 1373, 1257, 1196, 1173 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.67 (s, 3H), 2.42 (t, J = 7.4 Hz, 2H), 2.39 (t, J = 7.4 Hz, 2H), 2.33 (t, J = 7.4 Hz, 2H), 1.67-1.49 (m, 4H), 1.55 (quint, J = 7.4 Hz, 2H), 1.36-1.25 (m, 2H), 0.91 (t, J = 7.7 Hz, 3H); *m/z* (relative intensity) 200 (0.2%, M⁺), 169 (4), 158 (32), 143 (14), 126 (36), 111 (83), 85 (79), 57 (100).

Methyl 6-(4-Methylphenyl)-6-oxohexanoate (Table 2, entry 6):⁹ colorless plates; mp 47-48 °C (recrystallized from hexane); IR (KBr) 2951, 2870, 1734, 1674, 1606, 1574, 1261, 1171, 997 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, J = 8.6 Hz, 2H), 7.26 (d, J = 8.6 Hz, 2H), 3.67 (s, 3H), 2.97 (t, J = 7.3 Hz, 2H), 2.41 (s, 3H), 2.38 (t, J = 7.3 Hz, 2H), 1.81-1.69 (m, 4H); MS *m/z* (relative intensity) 234 (1%, M⁺), 157(15), 119 (100), 91 (63), 65 (28), 55 (11).

Methyl 6-(4-Chlorophenyl)-6-oxohexanoate (Table 2, entry 7):¹⁰ colorless needles; mp 69.5-70 °C (recrystallized from hexane); IR (KBr) 2949, 2871, 1726, 1676, 1589, 1462, 1404, 1259, 1176, 1093, 993, 850, 835, 737 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 3.67 (s, 3H), 2.98 (t, J = 6.7 Hz, 2H), 2.38 (t, J = 6.7 Hz, 2H), 1.85-1.65 (m, 4H); MS *m/z* (relative intensity) 254 (0.6%, M⁺), 177 (6), 154 (39), 139 (100), 111 (33), 75 (14), 55 (11).

Methyl 6-Oxo-6-[4-(trifluoromethyl)phenyl]hexanoate (Table 2, entry 8): colorless needles; mp 77-77.5 °C (recrystallized from hexane); IR (KBr) 2937, 2866, 1728, 1680, 1579, 1510, 1329, 1259, 1165, 1130, 1111, 1068, 993, 866, 729 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, J = 8.8 Hz, 2H), 7.73 (d, J = 8.8 Hz, 2H), 3.68 (s, 3H), 3.03 (t, J = 7.3 Hz, 2H), 2.39 (t, J = 7.3 Hz, 2H), 1.85-1.68 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 198.2, 173.4, 139.2, 134.0 (q, ²J_{CF} = 32.5 Hz), 128.0, 125.4

(q, $^3J_{CF} = 4.2$ Hz), 123.3 (q, $^1J_{CF} = 271.8$ Hz), 51.5, 38.4, 33.8, 24.4, 23.3; MS m/z (relative intensity) 288 (0.7%, M $^+$), 211 (11), 188 (25), 173 (100), 145 (45), 101 (19), 74 (12), 55 (15); HRMS calcd for C₁₄H₁₅F₃O₃ (M $^+$) 288.0973, found 288.0971. Anal. Calcd for C₁₄H₁₅F₃O₃: C, 58.33; H, 5.24. Found: C, 58.04; H, 5.21.

2,7-Octanedione (Table 2, entry 9):¹¹ a colorless oil; IR (neat) 2937, 2862, 1712, 1414, 1362, 1174 cm $^{-1}$; 1H NMR (400 MHz, CDCl₃) δ 2.53-2.40 (4H), 2.14 (s, 6H), 1.61-1.50 (4H); MS m/z (relative intensity) 142 (<1%, M $^+$), 84 (44), 71 (19), 58 (25), 43 (100).

4-Phenylsulfonyl-2,7-octanedione (Table 2, entry 10): a colorless oil; IR (neat) 3064, 2924, 1716, 1585, 1446, 1419, 1363, 1302, 1144, 1084, 731, 692 cm $^{-1}$; 1H NMR (400 MHz, CDCl₃) δ 7.88 (br d, $J = 7.9$ Hz, 2H), 7.68 (tt, $J = 7.4, 1.5$ Hz, 1H), 7.59 (dd, $J = 7.9, 7.4$ Hz, 2H), 3.75-3.66 (m, 1H), 3.14 (dd, $J = 8.7, 3.7$ Hz, 1H), 2.61 (t, $J = 7.3$ Hz, 2H), 2.65-2.55 (1H), 2.17 (s, 3H), 2.09 (s, 3H), 2.13-2.0 (m, 1H), 1.83-1.72 (m, 1H); ^{13}C NMR (100 MHz, CDCl₃) δ 206.4, 203.4, 136.9, 133.7, 129.0, 128.5, 58.5, 41.7, 40.2, 30.0, 29.8, 22.7; HRMS (ESI) m/z calcd for C₁₄H₁₈NaO₄S [(M+Na) $^+$] 305.0824, found 305.0823.

Dimethyl 1,7-Heptanedioate (Table 2, entry 11):¹² a colorless oil; IR (neat) 2952, 2866, 1739, 1437, 1363, 1200, 1173 cm $^{-1}$; 1H NMR (400 MHz, CDCl₃) δ 3.67 (s, 6H), 2.32 (t, $J = 7.6$ Hz, 4H), 1.65 (quint, $J = 7.6$ Hz, 4H), 1.43-1.29 (m, 2H); MS m/z (relative intensity) 188 (<1%, M $^+$), 157 (30), 115 (78), 97 (24), 83 (49), 74 (69), 59 (63), 55 (100).

Methyl 11-Hydroxyundecanoate (Table 2, entry 20):¹³ a colorless oil; IR (neat) 3390, 2927, 2854, 1739, 1464, 1437, 1254, 1198, 1174, 1057, 723 cm $^{-1}$; 1H NMR (300 MHz, CDCl₃) δ 3.67 (s, 3H), 3.64 (t, $J = 6.6$ Hz, 2H), 2.31 (t, $J = 7.7$ Hz, 2H), 1.71-1.47 (m, 2H), 1.42-1.20 (m, 14H); MS m/z (relative intensity) 217 (27%, (M+1) $^+$), 199 (8), 185 (13), 167 (14), 149 (9), 98 (17), 87 (34), 74 (71), 69 (38), 55 (70), 41 (84), 31 (100).

Methyl 11-[N-(Trifluoroacetyl)amino]undecanoate (Table 2, entry 22): colorless needles; mp 48-48.5 °C (recrystallized from hexane); IR (neat) 3311, 3110, 2925, 2854, 1736, 1701, 1564, 1260-1090, 999, 891, 721 cm $^{-1}$; 1H NMR (400 MHz, CDCl₃) δ 6.39-6.23 (br s, 1H), 3.67 (s, 1H), 3.36 (q, $J = 6.9$ Hz, 2H), 2.31 (t, $J = 7.4$ Hz, 2H), 1.66-1.53 (m, 4H), 1.38-1.23 (m, 12H); ^{13}C NMR (100 MHz, CDCl₃) δ 173.9, 156.7 (q, $^2J_{CF} = 36.7$ Hz), 115.6 (q, $^1J_{CF} = 286.8$ Hz), 51.3, 39.9, 34.0, 29.2, 29.2, 29.1, 29.0, 29.0, 28.8, 26.5, 24.8; HRMS (ESI) m/z calcd for C₁₄H₂₄F₃NNaO₃ [(M+Na) $^+$] 334.1606, found 334.1603. Anal. Calcd for C₁₄H₂₄F₃NO₃: C, 54.01; H, 7.77; N, 4.50. Found: C, 53.62; H, 7.61, N, 4.51.

Methyl 11-(N-Tosylamino)undecanoate (Table 2, entry 23): colorless needles; mp 69.5-70 °C (recrystallized from hexane); IR (neat) 3292, 2920, 2850, 1736, 1597, 1429, 1323, 1159, 1065, 885, 820, 687 cm $^{-1}$; 1H NMR (400 MHz, CDCl₃) δ 7.75 (d, $J = 8.4$ Hz, 2H), 7.31 (d, $J = 8.4$ Hz, 2H), 4.46-4.34 (m, 1H), 3.67 (s, 3H), 2.92 (q, $J = 6.7$ Hz, 2H), 2.43 (s, 3H), 2.30 (t, $J = 7.4$ Hz, 2H), 1.66-1.53 (m, 2H), 1.44 (quint, $J = 6.7$ Hz, 2H), 1.33-1.14 (m, 12H); ^{13}C NMR (100 MHz, CDCl₃) δ 173.9, 142.9, 136.6, 129.3, 126.8, 51.3, 43.1, 34.0, 29.5, 29.2, 29.2, 29.1, 29.0, 29.0, 26.4, 24.8, 21.5; HRMS (ESI) m/z calcd for C₁₉H₃₁NNaO₄S [(M+Na) $^+$] 392.1872, found 392.1859. Anal. Calcd for C₁₉H₃₁NO₄S: C, 61.76; H, 8.46; N, 3.79. Found: C, 61.45; H, 8.29; N, 3.76.

Methyl 10-Hydroxydecanoate (Table 2, entry 25):¹⁴ a colorless oil; IR (neat) 3415, 2929, 2856, 1739, 1437, 1362, 1173, 1057 cm $^{-1}$; 1H NMR (400 MHz, CDCl₃) δ 3.67 (s, 3H), 3.64 (t, $J = 7.0$ Hz, 2H), 2.30 (t, $J = 7.5$ Hz, 2H), 1.67-1.50 (m, 2H), 1.40-1.22 (m, 12H); MS m/z (relative intensity) 203 [3%, (M+1) $^+$], 172 (61), 152 (50), 98 (54), 87 (92), 74 (100), 69 (55), 55 (73), 43 (29), 41 (55).

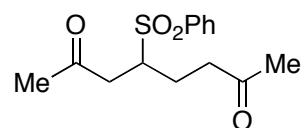
Methyl 10-Chlorodecanoate (Table 2, entry 26):¹⁵ a colorless oil; IR (neat) 2931, 2856, 1739, 1462, 1437, 1360, 1254, 1173, 725 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.67 (s, 3H), 3.53 (t, J = 6.5 Hz, 2H), 2.31 (t, J = 7.4 Hz, 2H), 1.77 (quint, J = 7.4 Hz, 2H), 1.68-1.25 (m, 12H); MS m/z (relative intensity) 220 (<1%, M⁺), 189 (3), 177 (3), 143 (3), 101 (4), 87 (36), 74 (100), 59 (11), 55 (26).

Dimethyl 1,10-Decanedioate (Table 2, entry 27):¹⁶ a colorless oil; IR (neat) 2931, 2856, 1739, 1437, 1362, 1250, 1198, 1173, 1018, 862 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.67 (s, 6H), 2.30 (t, J = 7.7 Hz, 4H), 1.70-1.54 (m, 4H), 1.37-1.23 (m, 8H); MS m/z (relative intensity) 231 [<1%, (M+1)⁺], 199 (27), 166 (22), 157 (21), 138 (26), 125 (51), 98 (51), 84 (41), 74 (66), 59 (40), 55 (100).

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Table 2, entry 10



^1H NMR (400 MHz, CDCl_3)

