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Baeyer-Villiger Oxidation with $Me_3SiOOSiMe_3$ under Assistance of $SnCl_4$ or $BF_3\cdot OEt_2$

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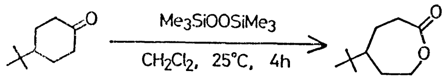
Treatment of ketones with bis(trimethylsilyl) peroxide and Lewis acid such as $SnCl_4$ or $BF_3\cdot OEt_2$ in dichloromethane at room temperature affords esters in fair to excellent yields. Jasmine lactone is synthesized from 2-[(*Z*)-2-pentenyl]cyclopentanone by means of $Me_3SiOOSiMe_3$–$BF_3\cdot OEt_2$ system without any protection of the carbon-carbon double bond. The oxidation of enol acetates of ketones to α-hydroxy (or α-acetoxy) ketones with $Me_3SiOOSiMe_3$–$FeCl_3$ system is also disclosed.

The Baeyer-Villiger oxidation is one of the most important processes in organic synthesis.[1] Recent publication[2] by Noyori and his coworkers on this oxidation using $Me_3SiOOSiMe_3$–Me_3SiOTf prompted us to disclose our independent work.

Usual Baeyer-Villiger oxidants[3] such as H_2O_2 or peroxy acids react with the coexisting carbon–carbon double bond of the substrate under epoxidation.[4] To overcome this difficulty, we examined the oxidation with $Me_3SiOOSiMe_3$[5] in the presence of Lewis acids[6] being added in order to activate the carbonyl function (Table 1). Among those examined, $SnCl_4$ was found to effectively assist the oxidation in general. No reaction occurred in the absence of Lewis acid.

The reaction rate with $BF_3\cdot OEt_2$ is comparable with that by means of $SnCl_4$ in the case of cyclopentanones. Other cycloalkanones reacted in the presence of $BF_3\cdot OEt_2$ to produce generally cyclic peroxides,[7] which decomposed slowly to afford a mixture of the desired lactones and the starting ketones. The results of the Baeyer-Villiger oxidation with $Me_3SiOOSiMe_3$–$SnCl_4$ or –$BF_3\cdot OEt_2$ system are summarized in Table 2. Although aliphatic ketones were oxidized in good to excellent yields (runs 1,2), aromatic ketones such as acetophenone gave complex mixtures.[8] The observed Baeyer-Villiger reaction in preference to C=C bond attack (runs 3—6) features the present oxidation procedure over the peroxy acid one.

Table 1. Effects of various Lewis acids on the transformation of 4-*t*-butylcyclohexanone to 4-*t*-butyl-6-hexanolide[a)]

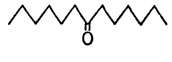
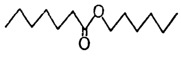
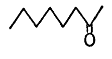
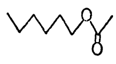
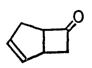
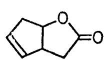
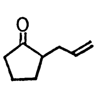
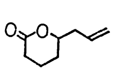
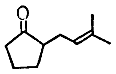
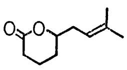


1 2

Lewis acid	Yield/%	Lewis acid	Yield/%
$SnCl_4$	84	$ZrCl_4$	34
$BF_3\cdot OEt_2$	88[b)]	$TiCl_4$	27
$FeCl_3$	72	$ZnCl_2$	<5
$AlCl_3$	38		

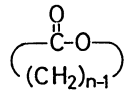
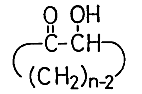
a) Two mol of $Me_3SiOOSiMe_3$ and one mol of Lewis acid were employed per mol of 4-*t*-butylcyclohexanone. b) The reaction rate was inconveniently low. The reaction required 2 d for completion.

Table 2. Baeyer-Villiger oxidation with $Me_3SiOOSiMe_3$–$SnCl_4$ and –$BF_3\cdot OEt_2$ systems[a)]

Run	Ketone	Lewis acid (mol equiv)	Time/h	Ester	Yield[b)]/%
1		$SnCl_4$ (1.0)	5		93
2		$SnCl_4$ (1.0)	11		69
3		$SnCl_4$ (1.0) $BF_3\cdot OEt_2$ (2.0)	9 7		25, 38[c)] 17, 28[c)]
4		$SnCl_4$ (1.0)	4		80[d)]
5		$SnCl_4$ (1.0) $BF_3\cdot OEt_2$ (2.0)	3 4		64 58
6		$BF_3\cdot OEt_2$[f)] (2.0)	4		44[e,f)]

a) Reactions were performed on 1.0 mmol scale at 25 °C. b) Isolated yield. c) Based on the consumed starting material. d) Ref. 1a. e) Ref. 4. f) Reaction with $SnCl_4$ gave a complex mixture in which the desired lactone was absent.

TABLE 3. OXIDATION OF SIMPLE CYCLIC KETONES TO GIVE THE CORRESPONDING LACTONES AND/OR α-HYDROXY KETONES[a)]

n		5	6	7	8	10	11	12
Time/h		3	3	3.5	4	3	4	2.5
	Yield[b)] / %	85	83	50	11	24	30	62
	Yield / %	0[c)]	0[c)]	4[c)]	42	35	33	20

a) Reactions were done at 25 °C in CH_2Cl_2. Two mol of $Me_3SiOOSiMe_3$ and 1 mol of $SnCl_4$ were employed per mol of carbonyl compounds. b) Isolated yield. c) GLPC yield.

Table 3 summarizes the reaction of simple cyclic ketones[9] in the presence of $SnCl_4$. The product distributions depend on the sizes of the ring. In the case of 5, 6, 7, and 12 membered ring the corresponding lactones were obtained in good yields. Meanwhile, medium ring (8, 10, and 11 membered) cycloalkanone gave α-hydroxy ketones.[10] This is tentatively ascribed to the oxidation of enol forms of the substrates.[11]

Treatment of silyl enol ethers with $Me_3SiOOSiMe_3$ in the presence of $SnCl_4$, $BF_3\cdot OEt_2$, or $FeCl_3$ gave the starting ketones. In contrast, enol acetates are oxidized to the corresponding α-acetoxy and/or α-hydroxy ketones by means of $Me_3SiOOSiMe_3$–Lewis acid system. Yields of α-acetoxy and α-hydroxy acetophenone in the oxidation of 1-phenylethenyl acetate and $Me_3SiOOSiMe_3$ in dichloromethane at 25 °C for 12 h were as follows: $SnCl_4$, 28%, 41%; $BF_3\cdot OEt_2$, 28%, 0%. Iron(III) chloride proved to be the best promoter for the oxidation (Fig. 1).[12]

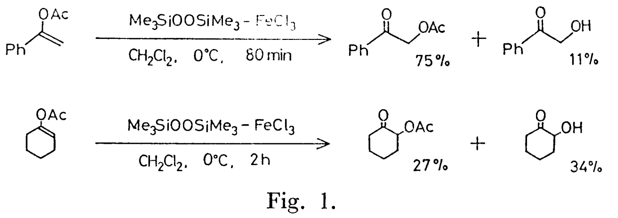


Fig. 1.

Relative migratory aptitude in the cleavage of unsymmetrical ketones with $Me_3SiOOSiMe_3$–$SnCl_4$ and –$BF_3\cdot OEt_2$ systems were examined (Fig. 2). The present reactions have the same tendency as those with peroxy acids.[13] The selectivity was poor in the case of 2-methylcyclohexanone.

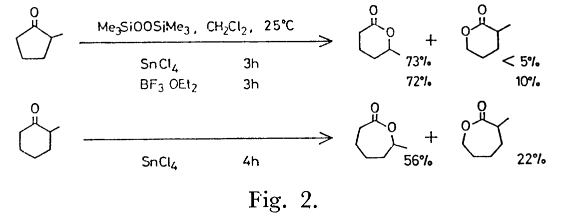


Fig. 2.

Jasmine lactone (**4**) is synthesized from 2-[(*Z*)-2-pentenyl]cyclopentanone (**3**) in several steps.[14] Protection of the olefinic bond is prerequisite in the previous procedures with perbenzoic acid, as treatment of the ketone **3** with *m*-chloroperbenzoic acid (1.5 equiv) in dichloromethane gave epoxide in 89% yield in our hand. The Baeyer-Villiger oxidation in preference to the epoxidation has now been achieved with $Me_3SiOOSiMe_3$–$BF_3\cdot OEt_2$ system (Fig. 3). Addition of portions of $Me_3SiOOSiMe_3$ (2.2 equiv) and $BF_3\cdot OEt_2$ (2.0 equiv) to the ketone **3** in dichloromethane gave the lactone **4** in 50% yield along with the hydroxy carboxylic acid (**5**) in 8% yield.[15]

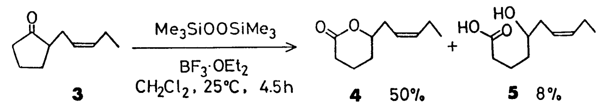


Fig. 3.

Although the yields of the oxidation with $Me_3SiOOSiMe_3$–$SnCl_4$ (or $BF_3\cdot OEt_2$) system are better than, or at least comparable to, those with Me_3SiOTf, easy accessibility of the Lewis acids characterizes this system as a useful method for the Baeyer-Villiger oxidation.

Experimental

The IR Spectra were determined on a Shimadzu IR-27-G spectrometer, the mass spectra on a Hitachi M-80 machine, and the NMR spectra on a Varian EM-390 spectrometer. The chemical shifts are given in δ, with TMS as an internal standard. The analyses were carried out by the staff at the Elemental Analyses Center of Kyoto University. Dichloromethane was dried on P_2O_5 and distilled. Bis(trimethylsilyl) peroxide was prepared according to the reported procedure and purified by distillation (bp 40—41 °C/30 Torr, 1 Torr = 133.322 Pa).[5] All the experiments were carried out under an argon atmosphere. Purification of products were performed by preparative thin layer chromatography (TLC) or column chromatography on silica gel (Wakogel C-100). Analytical GLPC was performed with a Yanagimoto GCG-550-F and a Shimadzu GC-4CPT.

4-t-Butyl-6-hexanolide (2): To a stirred solution of 4-*t*-butylcyclohexanone (**1**, 0.15 g, 1.0 mmnl) and bis(trimethylsilyl) peroxide (0.18 g, 1.0 mmol) in CH_2Cl_2 (10 ml) at 0 °C tin(IV) chloride (0.26 g, 1.0 mmol) was added slowly at this temperature. After being stirred at 0 °C for 15 min and 25 °C for 1 h, bis(trimethylsilyl) peroxide (0.18 g, 1.0 mmol) was added and the whole was stirred at 25°C for 3 h. The resulting mixture was poured into aq $Na_2S_2O_3$ (10 ml) and extracted with ether. The combined organic layers were washed with aq $NaHCO_3$ (10 ml) and brine, dried and concentrated. Purification by preparative TLC on silica gel (hexane–ethyl acetate, 2:1) gave **2** as a white solid (0.14 g, 84%). Mp 55 °C (hexane); IR (CCl_4): 1743, 1200, 1190, 1140, 1080 cm^{-1}; NMR (CCl_4): $\delta=0.90$ (s, 9H), 1.10—2.30 (m, 5H), 2.40—2.66 (m, 2H), 3.80—4.40 (m, 2H); MS *m/e* (%): 170 (M^+, 2), 155 (2), 114 (80), 86 (60), 68 (13), 57 (100). The compound was identical with an authentic sample.[16]

Hexyl Heptanoate:[17] Bp 120 °C (bath temp, 3 Torr); IR (neat): 1742, 1380, 1170, 1102 cm^{-1}; NMR (CCl_4): $\delta=$ 0.72—1.83 (m, 22H), 2.17 (t, $J=7$ Hz, 2H), 3.94 (t, $J=$ 7 Hz, 2H), MS *m/e* (%): 131 (7), 84 (25), 69 (18), 61 (19), 60 (12), 56 (44), 43 (100), 42 (27).

9-Decenyl Acetate:[18] Bp 100 °C (bath temp, 3 Torr); IR (neat): 1750, 1645, 1245, 910 cm^{-1}; NMR (CCl_4): $\delta=1.10$—2.20 (m, 14H), 1.96 (s, 3H), 3.94 (t, $J=7$ Hz, 2H), 4.70—5.10 (m, 2H), 5.68 (ddt, $J=16.5$, 10, 7 Hz, 1H); MS *m/e* (%): 198 (M^+, 0.3), 155 (6), 138 (24), 110 (40), 96 (80), 82 (90), 68 (100), 54 (50).

2-Oxabicyclo[3.3.0]oct-6-en-3-one:[1] Bp 80 °C (bath temp, 1 Torr); IR (neat): 3090, 1780, 1180, 1020, 923, 720 cm^{-1}; NMR (CCl_4): $\delta=2.10$—2.90 (m, 4H), 3.30—3.66 (m, 1H), 5.00—5.20 (m, 1H), 5.50—6.10 (m, 2H); MS *m/e* (%): 124 (M^+, 16), 96 (54), 95 (49), 79 (60), 68 (38), 67 (100), 66 (54).

7-Octen-5-olide:[14] Bp 75 °C (bath temp, 3 Torr); IR (neat): 3090, 1740, 1645, 1245, 1050, 996, 924 cm^{-1}; NMR (CCl_4): $\delta=1.15$—2.95 (m, 8H), 3.96—4.10 (m, 1H), 4.78—5.30 (m, 2H), 5.30—6.10 (m, 1H); MS *m/e* (%): 140 (M^+, 5), 100 (57), 99 (92), 71 (96), 67 (21), 55 (100), 43 (87).

8-Methyl-7-nonen-5-olide: Bp 95 °C (bath temp, 0.15 Torr); IR (neat): 1740, 1242, 1175, 1048, 920 cm^{-1}; NMR (CCl_4): $\delta=1.13$—2.50 (m, 8H), 1.63 (s, 3H), 1.72 (s, 3H), 3.80—4.40 (m, 1H), 4.90—5.30 (bt, 1H); MS *m/e* (%): 168 (M^+, 6), 99 (85), 71 (100), 69 (12), 55 (34), 43 (54): Found: C, 71.41; H, 9.47%, Calcd for $C_{10}H_{16}O_2$: C, 71.39, H, 9.59%.

7-Heptanolide: The above procedure using cycloheptanone (0.11 g, 1.0 mmol) afforded after usual workup and purification by preparative TLC (hexane–ethyl acetate, 2:1) a mixture of 7-heptanolide and 2-hydroxycycloheptanone in 54% yield (69 mg) in a ratio of 93:7 as determined by GLPC (5% PEG 20 M, 1.5 m, 128 °C). 7-Heptanolide ($T_r=4.5$ min) and 2-hydroxycycloheptanone ($T_r=6.5$ min) were identical with authentic sample.[19,10] 7-Heptanolide had bp 90 °C (bath temp, 20 Torr); IR (neat): 1715, 1240, 1150, 1098 cm^{-1}; NMR (CCl_4): $\delta=1.36$—2.13 (m, 8H), 2.35 (t, $J=6$ Hz, 2H), 4.25 (t, $J=6$ Hz, 2H): MS *m/e* (%): 128 (M^+, 12), 110 (44), 100 (78), 98 (100), 96 (60), 82 (74), 81 (85), 71 (55), 68 (92), 67 (52), 57 (75).

2-Hydroxycycloheptanone: Bp 83 °C (bath temp, 3 Torr); IR (neat): 3490. 1705, 1078, 930 cm^{-1}; NMR (CCl_4): $\delta=1.10$—3.00 (m, 10H), 3.45 (d, $J=4$ Hz, 1H), 3.90—4.30 (m, 1H); MS *m/e* (%): 128 (M^+, 0.3), 126 (0.4), 110 (5), 82 (7), 67 (100), 44 (32).

8-Octanolide: The above procedure with cyclooctanone (0.13 g, 1.0 mmol) gave a mixture of 8-octanolide (16 mg, 11%) and 2-hydroxycyclooctanone (60 mg, 42%). These compounds were identical with authentic samples.[19,10] 8-Octanolide had bp 130 °C (bath temp, 20 Torr); IR (neat): 1740, 1142, 1032 cm^{-1}; NMR (CCl_4): $\delta=1.10$—2.00 (m, 10H), 2.10—2.36 (m, 2H), 4.28 (t, $J=6$ Hz, 2H); MS *m/e* (%): 142 (M^+, 5), 124 (6), 112 (25), 99 (77), 96 (48), 84 (50), 82 (69), 70 (43), 68 (100), 43 (46).

2-Hydroxycyclooctanone: Bp 100 °C (bath temp, 3 Torr); IR (neat): 3500, 1705, 1450, 1100, 950 cm^{-1}; NMR (CCl_4): $\delta=0.80$—2.80 (m, 12H), 3.30—3.75 (m, 1H), 4.00 (dd, $J=3$, 6 Hz, 1H); MS *m/e* (%): 142 (M^+, 2), 124 (52), 98 (100), 81 (57), 80 (39), 68 (38), 57 (93), 55 (60), 44 (43).

10-Decanolide:[19] Bp 130 °C (bath temp, 20 Torr), IR (neat): 1720, 1480, 1450, 1080 cm^{-1}; NMR (CCl_4): $\delta=$ 1.10—1.93 (m, 14H), 2.05—2.40 (m, 2H), 4.00—4.30 (m, 2H); MS *m/e* (%): 170 (M^+, 2), 144 (6), 111 (14), 98 (100), 84 (34), 69 (16), 55 (20).

2-Hydroxycyclodecanone:[10] Bp 110 °C (bath temp, 0.15 Torr): IR (neat): 3500, 1700, 1475, 1100, 1085, 995 cm^{-1}; NMR (CCl_4): $\delta=0.83$—3.40 (m, 16H), 3.50 (bs, 1H), 3.97—4.27 (m, 1H); MS *m/e* (%): 170 (M^+, 2), 134 (4), 96 (19), 82 (31), 68 (47), 57 (100), 55 (44).

11-Undecanolide:[19] Bp 105 °C (bath temp, 3 Torr): IR (neat): 1705, 1480, 1192. 1090 cm^{-1}; NMR (CCl_4): $\delta=$ 1.10—3.00 (m, 18H), 4.20 (dd, $J=6$, 8Hz, 2H); MS *m/e* (%): 184 (M^+, 25), 166 (14), 125 (23), 112 (25), 98 (100), 84 (33), 82 (39), 69 (66).

2-Hydroxycycloundecanone:[10] Bp 140°C (bath temp, 3 Torr); IR (neat): 3500, 1705, 1470, 1100, 1045 cm^{-1}; NMR (CCl_4): $\delta=1.10$—3.00 (m, 18H), 3.20—3.50 (bs, 1H), 4.15 (t, $J=$ 4.5 Hz, 1H); MS *m/e* (%): 184 (M^+, 10), 119 (41), 111 (53), 98 (58). 82 (100), 81 (58), 68 (78), 55 (54).

12-Dodecanolide:[19] Bp 120 °C (bath temp, 1 Torr); IR (neat): 1740, 1465, 1280, 1140, 1050 cm^{-1}; NMR (CCl_4): $\delta=1.20$—1.95 (m, 18H), 2.20—2.50 (m, 2H), 4.25 (t, $J=$ 4.5 Hz, 2H); MS *m/e* (%): 198 (M^+, 6), 180 (16), 138 (33), 110 (34), 98 (100), 96 (67), 84 (72), 82 (63).

2-Hydroxycyclododecanone:[10] Mp 79 °C (hexane/ether); IR (CCl_4): 3500, 1710, 1470, 1075, 1010 cm^{-1}; NMR (CCl_4): $\delta=0.80$—3.30 (m, 21H), 4.25 (t, $J=4$ Hz, 1H); MS *m/e* (%): 198 (M^+, 6), 180 (10), 136 (20), 124 (22), 111 (23), 98 (53), 96 (49), 95 (47), 82 (100), 81 (44), 68 (56), 67 (37), 57 (36).

5-Hexanolide:[20] The same procedure with 2-methylcyclopentanone (98 mg, 1.0 mmol) and $BF_3 \cdot OEt_2$ gave a mixture of 5-hexanolide and 2-methyl-5-pentanolide in 81% yield (92 mg). GLPC (5% PEG 20 M, 1.5 m, 130 °C) indicated two peaks, $T_r=24$ min (5-hexanolide, 88%), $T_r=22$ min (2-methyl-5-pentanolide, 12%). 5-Hexanolide had bp 70 °C (bath temp, 3 Torr); IR (neat): 1745, 1380, 1250, 1070 cm^{-1}; NMR (CCl_4): $\delta=1.33$ (d, $J=6$ Hz, 3H), 1.44—2.10 (m, 4H), 2.20—2.50 (m, 2H), 4.33 (qt, $J=6$, 3 Hz, 1H); MS *m/e* (%): 114 (M^+, 0.5), 71 (11), 70 (17), 55 (36), 42 (100).

2-Methyl-5-pentanolide:[21] Bp 70 °C (bath temp, 3 Torr); IR (neat): 1745, 1380, 1150, 1075 cm^{-1}; NMR (CCl_4): $\delta=$ 1.28 (d, $J=6$ Hz, 3H), 1.30—2.73 (m, 5H), 4.28 (t, $J=$ 6 Hz, 2H); MS *m/e* (%): 114 (M^+, 28). 70 (22), 56 (48), 55 (100), 42 (82).

6-Heptanolide:[20] The same procedure with 2-methylcyclohexanone (0.11 g, 1.0 mmol) and $SnCl_4$ (0.26 g, 1.0 mmol) afforded a mixture of 6-heptanolide and 2-methyl-6-hexanolide in 93% yield (0.12 g) in a ratio of 72:28 as determined by ^1H-NMR (the absorption of the methyl proton). 6-Heptanolide had bp 93 °C (bath temp, 3.5 Torr); IR (neat): 1735, 1180. 1078. 1018 cm^{-1}; NMR (CCl_4): $\delta=1.31$ (d, $J=6$ Hz, 3H), 1.20—2.10 (m, 6H), 2.35—2.73 (m, 2H), 4.10—4.50 (m, 1H); MS *m/e* (%): 128 (M^+, 2), 84 (40), 56 (69), 55 (73), 41 (100).

2-Methyl-6-hexanolide: Bp 88 °C (bath temp, 3 Torr); IR (neat): 1735, 1175, 1080, 1050 cm^{-1}; NMR (CCl_4): $\delta=1.13$ (d, $J=5$ Hz, 3H), 1.20—2.13 (m, 6H), 2.30—2.80 (m, 1H), 4.00—4.30 (m, 2H); MS *m/e* (%): 128 (M^+, 4), 70 (5), 69 (14), 56 (32), 55 (38), 43 (20), 42 (100), 41 (63), 40 (14); Found: C, 65.56; H, 9.52%, Calcd for $C_7H_{12}O_2$: C, 65.59, H, 9.44%.

(Z)-7-Decen-5-olide (***4***):[14] To a stirred solution of 2-[(*Z*)-2-pentenyl]cyclopentanone (**3**, 0.15 g, 1.0 mmol) and

$Me_3SiOOSiMe_3$ (0.21 g, 1.2 mmol) in CH_2Cl_2 (10 ml) under an argon atmosphere $BF_3 \cdot OEt_2$ (0.14 g, 1.0 mmol) was added slowly at 0 °C. The resulting mixture was stirred at 0 °C for 15 min and 25 °C for 1.5 h. Additional $BF_3 \cdot OEt_2$ (0.14 g, 1.0 mmol) and $Me_3SiOOSiMe_3$ (0.18 g, 1.0 mmol) were added and the whole was stirred at 25 °C for 3 h. The mixture was poured into aq $NaHCO_3$ (10 ml) and extracted with ether. The combined organic layer was washed with brine, dried and concentrated. Purification by preparative TLC (hexane-ethyl acetate, 2:1) gave (*Z*)-7-decen-5-olide (jasmine lactone, **4**, 84 mg, 50%) along with the hydroxy carboxylic acid **5**. The hydroxy carboxylic acid **5** was heated at 100 °C under reduced pressure (10 Torr) for 1 h. Purification by preparative TLC afforded the desired lactone **4** in 8% yield (13 mg): Bp 125 °C (bath temp, 0.2 Torr); IR (neat): 3020, 1740, 1240, 1049, 928 cm^{-1}; NMR (CCl_4): $\delta = 0.99$ (t, $J = 7$ Hz, 3H), 1.20—2.93 (m, 10 Hz), 4.00—4.50 (m, 1H), 5.06—5.75 (m, 2H); MS *m/e* (%): 168 (M^+, 14), 150 (10), 127 (6), 108 (6), 99 (100), 81 (10), 71 (40), 55 (18).

2-Acetoxyacetophenone:[22] Iron(III) chloride (0.16 g, 1.0 mmol) was added in portions to a stirred solution of 1-phenylethenyl acetate (0.16 mg, 1.0 mmol) and $Me_3SiOOSiMe_3$ (0.21 g, 1.2 mmol) at 0°C under an argon atmosphere. After stirring at 0 °C for 80 min, the resulting mixture was poured into brine (5 ml) and extracted with ether. The combined organic layers were dried and concentrated. Purification by preparative TLC (hexane-ethyl acetate, 2:1) gave 2-acetoxyacetophenone in 75% yield (0.13 g) and 2-hydroxyacetophenone in 11% yield (14 mg). 2-Acetoxyacetophenone had bp 107 °C (bath temp, 3 Torr); IR (neat): 1755, 1708, 1600. 1215. 1080, 955. 750, 685 cm^{-1}; NMR (CCl_4): $\delta = 2.13$ (s, 3H), 5.13 (s, 2H), 7.15—7.63 (m, 3H), 7.70—7.93 (m, 2H); MS *m/e* (%): 178 (M^+, 1), 118 (12), 106 (23), 105 (100), 91 (12), 77 (71).

2-Acetoxycyclohexanone:[23] Bp 90 °C (bath temp, 3 Torr); IR (neat): 1755, 1728, 1375, 1240, 1082, 1065 cm^{-1}; NMR (CCl_4): $\delta = 1.00$—2.55 (m, 8H), 2.10 (s, 3H), 4.96 (dd, $J =$ 7, 11 Hz, 1H); MS *m/e* (%): 156 (M^+, 14), 114 (27), 113 (76), 85 (26), 76 (38), 57 (10), 43 (100).

2-Hydoxycyclohexanone:[10] Bp 78 °C (bath temp, 3 Torr); IR (neat): 3500, 1715, 1262, 1100, 1025, 885 cm^{-1}; NMR (CCl_4): $\delta = 1.10$—2.70 (m, 8H), 3.36 (m, 1H), 3.94 (dd, $J = 8$, 11 Hz, 1H); MS *m/e* (%): 114 (M^+, 11), 96 (7), 68 (9), 67 (15), 58 (16), 43 (100).

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