# Selective Oxidation of Alcohols at the Benzylic Position by Benzeneseleninic Anhydride

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**Benzeneseleninic anhydride in the presence of *tert*-butyl hydroperoxide in chlorobenzene at about 70 °C is an effective oxidizing agent for the selective oxidation of alcohols at the benzylic position.**

**Key words** oxidation; selective oxidation; benzeneseleninic anhydride; benzyl alcohol

Oxidation of alcohols to aldehydes and ketones is obviously one of the most fundamental transformations in synthetic organic chemistry and innumerable methods have been developed.[1,2] One interesting objective in the oxidation of alcohols is the selective oxidation in polyhydroxylated molecules. Many methods for selective oxidation of primary and secondary hydroxy groups have been reported.[3—11] Selective oxidation of benzyl and allyl alcohols is also known.[12—14]

Benzeneseleninic anhydride (BSA) is easily prepared from diphenyl diselenide by oxidation with ozone or *tert*-butyl hydroperoxide (TBHP)[15] and is now commercially available.[16] Barton initially used BSA as an oxidizing agent for oxidation of phenols,[17] oxidation of ketones to enones,[18] regeneration of ketones from hydrazones, oximes, semicarbazones,[19] and thioacetals.[20] BSA was also reported to be an oxidant for alcohols to carbonyl compounds.[21]

We previously reported an angular hydroxylation of polycyclic ketones using BSA.[22,23] In continuation of our interest in BSA as an oxidizing agent, we recently found that BSA is an effective oxidant for selective oxidation of alcohols at the benzylic position.

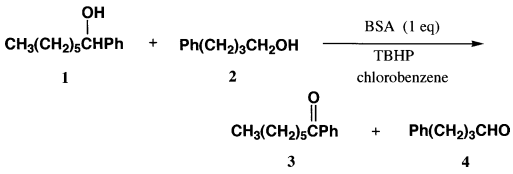
To investigate the effect of BSA as a selective oxidizing agent, we first treated a mixture of secondary alcohol **1** (1 eq) and primary alcohol **2** (1 eq) with 1 eq of BSA in chlorobenzene at 50 °C for 3 h (see Table 1; entry 1). Only ketone **3** was obtained in 34% yield with recovery of the alcohols and no aldehyde **4** was obtained. This result implied that selective oxidation of primary and secondary alcohols could be possible.

Encouraged by this result, we further studied this oxidation and found that TBHP is effective for the selective oxidation of alcohols with BSA. As shown in entry 2, the presence of 5 eq of TBHP gave a much higher yield of the ketone **3**. When this reaction was carried out at 70 °C the yield of the ketone **3** was increased up to 90% within 1.5 h (entry 3). Even under these conditions only a trace of aldehyde **4** was obtained. Later, 3 eq of TBHP was found to be enough in this selective oxidation (entry 4).

Next, we studied chemoselective oxidation of primary and secondary alcohols using diol **5** as an example (see Table 2). Under the best conditions mentioned above (Table 1; entry 4), the diol **5** was oxidized to ketoalcohol **6** in 82% yield without ketoaldehyde **7** (Table 2; entry 1). When the amount of TBHP was reduced to 1 eq, a considerable amount of the ketoaldehyde **7** was obtained (entry 2). In the case of the absence of TBHP (entry 3), the result was quite similar to the result in entry 2. When this reaction was carried out with 3 eq of 2-methyl-2-propanol instead of TBHP (entry 4) a similar result was obtained. From these results, about 3 eq of TBHP was found to be essential for the selective oxidation, though the real reason is obscure at present. To rule out the possibility that the selective oxidation takes place only by TBHP, the reaction was carried out in the presence of 3 eq of TBHP without BSA (entry 6). No oxidation at all was observed under these conditions.

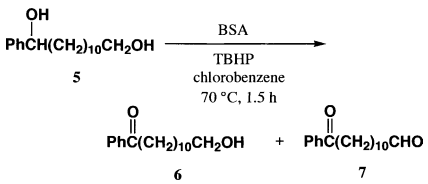
Further, we investigated this chemoselective oxidation

Table 1. Chemoselective Oxidation of Primary and Secondary Alcohols with BSA




| Entry | TBHP (eq) | Temperature (°C) | Time (h) | **3** Yield % | **4** Yield % |
|---|---|---|---|---|---|
| 1 | 0 | 50 | 3 | 34 | 0 |
| 2 | 5 | 50 | 3 | 66 | Trace |
| 3 | 5 | 70 | 1.5 | 90 | Trace |
| 4 | 3 | 70 | 1.5 | 89 | Trace |

Table 2. Chemoselective Oxidation of 1-Phenyldodecane-1,12-diol with BSA




| Entry | BSA (eq) | TBHP (eq) | **6** Yield % | **7** Yield % |
|---|---|---|---|---|
| 1 | 1 | 3 | 82 | 0 |
| 2 | 1 | 1 | 67 | 21 |
| 3 | 1 | 0 | 69 | 18 |
| 4 | 1 | —[a)] | 70 | 17 |
| 5 | 0.5 | 3 | 82 | 3.5 |
| 6 | 0 | 3 | 0[b)] | 0 |

*a*) *tert*-BuOH (3 eq) was added to the reaction mixture instead of TBHP. *b*) No reaction was observed.

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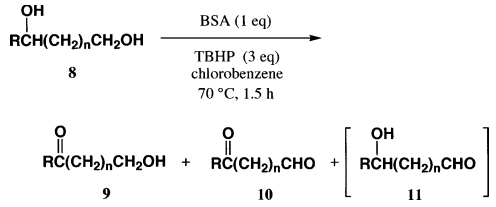
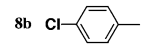
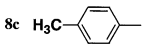
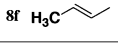
Table 3. Chemoselective Oxidation of Several Diols with BSA




| Entry | 8 R | 8 *n* | 9 | 9 Yield % | 10 | 10 Yield % |
|---|---|---|---|---|---|---|
| 1 | **8a**  | 3 | **9a** | 79 | | 0 |
| 2 | **8b**  | 10 | **9b** | 72 | **10b** | 15 |
| 3 | **8c**  | 10 | **9c** | 67 | **10c** | 16 |
| 4 | **8d**  | 10 | **9d** | 46 | **10d** | 34 |
| 5 | **8e**  | 10 | **9e** | 51 | **10e** | 32 |
| 6 | **8f**  | 10 | **9f** | 16 | | —[a] |

*a*) A complex mixture was obtained from this reaction.

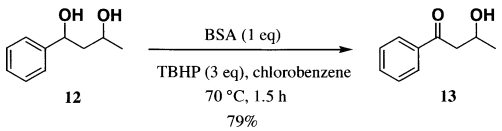



Chart 1. Intramolecular Chemoselective Oxidation of 1-Phenyl-1,3-butanediol with BSA in the Presence of TBHP

with various kinds of diols and the results are shown in Table 3. As shown in entry 1 to entry 3, benzylic secondary alcohols **8a**—**c** were selectively oxidized to ketones though the selectivity was somewhat lower when R are 4-chlorophenyl or 4-methylphenyl (entries 2, 3). The results shown in entries 4 and 5 indicated that secondary alcohols at the non-benzylic position showed low selectivity. In the case of allylic alcohol **8f**, BSA was not useful for the oxidation (entry 6). In all the reactions aldehyde **11** was not obtained.

Next, diol **12**, which has two secondary alcohols, one of which is at the benzylic position, was synthesized and the oxidation with BSA in the presence of 3 eq of TBHP at 70 °C was carried out. After 1.5 h, the reaction was quenched and we obtained the ketoalcohol **13** in 79% yield. From this reaction 16% of the starting diol **12** was recovered; however, no diketone was observed.

Finally, we investigated the selectivity for primary and secondary benzyl alcohols (Chart 2). 4-(1-Hydroxyethyl)benzyl alcohol **14** was oxidized under the same conditions described above and the reaction was stopped after 15 min. As shown in Chart 2, the aldehyde **15** was obtained in 55% yield with keto-alcohol **16** (14%). This result indicated that the speed of the oxidation of primary benzyl alcohol with BSA-TBHP is much faster than that for the secondary benzyl alcohol.

In conclusion, we have found that BSA in the presence of 3 eq of TBHP in chlorobenzene at 70 °C is effective oxidizing agent for selective oxidation of benzyl alcohols to ketones and aldehydes.

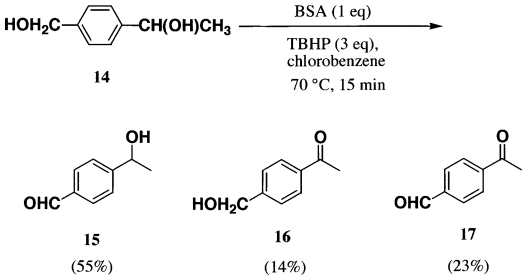



Chart 2. Oxidation of 4-(1-Hydroxyethyl)benzyl Alcohol with BSA in the Presence of TBHP

## Experimental

All melting points are uncorrected. $^1$H-NMR spectra were measured in a $CDCl_3$ solution with JEOL JNM-LA 400 and 500 spectrometer. Electron-impact mass spectra (MS) were obtained at 70 eV by direct insertion. Silica gel 60 (MERCK) containing 0.5% fluorescence reagent 254 and a quartz column were used for column chromatography and the products having UV absorption were detected by UV irradiation. In experiments requiring a dry reagent and solvent, chlorobenzene, DMSO, $CH_2Cl_2$ were distilled from $CaH_2$ and THF was distilled from diphenylketyl. TBHP (MERCK, 80% solution in di-*tert*-butylperoxide) was used without any purification.

**Chemoselective Oxidation of Secondary Alcohol 1 and Primary Alcohol 2 with BSA** To a solution of BSA (180 mg; 0.5 mmol) in 15 ml of chlorobenzene at 70 °C was added a solution of 1-phenyl-1-heptanol **1** (96 mg; 0.5 mmol) and 4-phenylbutanol **2** (75 mg; 0.5 mmol) in 0.5 ml of chlorobenzene. To the mixture was added TBHP (0.19 ml; 1.5 mmol) and the reaction mixture was stirred at 70 °C for 90 min. The reaction was quenched by sat. $Na_2S_2O_3$ and the whole was extracted with benzene. The organic layer was washed with water and brine, and then dried over $MgSO_4$. The product was purified by silica gel column chromatography to give 1-phenyl-1-heptanone (heptanophenone) **3** (85 mg; 89%) and recovery of alchol **2** (48 mg; 64%).

**1-Phenyldodecane-1,12-diol (5)** To a solution of PPTS (503 mg; 2.0 mmol) in 100 ml of dry $CH_2Cl_2$ at 0 °C were added 1,12-dodecanediol (4.04 g; 20 mmol) and dihydropyran (1.85 g; 22 mmol) and the reaction mixture was stirred at room temperature for 30 min. The mixture was diluted with $Et_2O$ and the solution was washed with brine. The product was purified by silica gel column chromatography to give mono-protected alcohol (2.97 g; 52%) as a colorless oil. IR (neat) 3400 (OH), 2927, 2855, 1466, 1353, 1201, 1137, 1122, 1078, 1034 cm$^{-1}$; $^1$H-NMR $\delta$ 1.20—1.75 (26H, m), 3.34—3.42 (1H, m), 3.47—3.53 (1H, m), 3.61—3.66 (2H, m), 3.69—3.77 (1H, m), 3.84—3.91 (1H, m), 4.58 (1H, t, $J$=3.5 Hz).

To a solution of oxalyl chloride (0.4 ml; 4.56 mmol) in 45 ml of $CH_2Cl_2$ was added DMSO (0.66 ml; 9.29 mmol) at −78 °C. After 10 min, a solution of the mono-protected alcohol (1.3 g; 4.65 mmol) in $CH_2Cl_2$ was added, and the mixture was stirred for 15 min at −60 °C. Triethylamine (23.2 mmol) was added to the reaction mixture, and the reaction mixture was stirred for 15 min at room temperature. The reaction was quenched by sat. $NH_4Cl$ and the whole was extracted with $CHCl_3$. The product was purified by silica gel column chromatography to give aldehyde (1.15 g; 89%) as a colorless oil. IR (neat) 2928, 2855, 2716, 1727 (CHO), 1466, 1353, 1201, 1137, 1122, 1078, 1034, 988 cm$^{-1}$; $^1$H-NMR $\delta$ 1.27—1.86 (24H, m), 2.40—2.44 (2H, dt, $J$=7.5, 1.9 Hz), 3.36—3.40 (1H, m), 3.48—3.52 (1H, m), 3.71—3.75 (1H, m), 3.85—3.89 (1H, m), 4.57 (1H, t, $J$=4.6 Hz), 9.76 (1H, t, $J$=1.9 Hz).

To a solution of PhMgBr (1.58 mmol) in THF (10 ml) was added the aldehyde (298 mg; 1.05 mmol) at 0 °C. After the mixture was stirred for 1 h at room temperature, the reaction was quenched with sat. $NH_4Cl$ and the whole was extracted with AcOEt. The product was purified by silica gel column chromatography to give alcohol (341 mg; 91%) as a colorless oil. IR (neat) 3437 (OH), 2927, 2854, 1455, 1136, 1121, 1077, 1024, 701 cm$^{-1}$; $^1$H-NMR $\delta$ 1.25—1.86 (26H, m), 3.35—3.40 (1H, m), 3.47—3.51 (1H, m), 3.70—3.74 (1H, m), 3.84—3.89 (1H, m), 4.57 (1H, t, $J$=3.7 Hz), 4.64—4.68 (1H, m), 7.23—7.35 (5H, m).

To a solution of the alcohol (341 mg; 0.95 mmol) in EtOH (9 ml) was added PPTS (23.8 mg; 0.09 mmol). The mixture was stirred for 2 h at 60 °C. After removal of the solvent, the product was purified by silica gel column chromatography to give diol **5** (212 mg; 81%) as colorless crystals; mp 43.5—44.5 °C (AcOEt–hexane). IR (KBr) 3307 (OH), 2919, 2849, 1468, 1054, 763, 700 cm$^{-1}$. $^1$H-NMR $\delta$ 1.25—1.88 (20H, m), 3.63 (2H, t, $J$=6.3 Hz), 4.66 (1H, t, $J$=6.3 Hz), 7.25—7.36 (5H, m). MS *m/z* (%) 278 ($M^+$,

0.6), 120 (15), 107 (100), 105 (14), 79 (9), 77 (8). Calcd for $C_{18}H_{30}O_2$: M, 278.2247. Found: *m/z* 278.2245. *Anal.* Calcd $C_{18}H_{30}O_2$: C, 77.65; H, 10.86. Found: C, 77.81; H, 10.86.

**12-Hydroxy-1-phenyl-1-dodecanone (6)** To a solution of BSA (90 mg; 0.25 mmol) in 3 ml of chlorobenzene at 70 °C was added a solution of **5** (69.5 mg; 0.25 mmol) in 1.0 ml of chlorobenzene and TBHP (0.094 ml; 0.75 mmol), and the reaction mixture was stirred at 70 °C for 90 min. The reaction was quenched by sat. $Na_2S_2O_3$, and the whole was extracted with benzene. The organic layer was washed with water and brine, and the product was purified by silica gel column chromatography to give ketone **6** (56 mg; 82%) as a colorless oil. IR (neat) 3356 (OH), 2916, 2849, 1685 (CO), 1464, 1447, 1377, 1207, 1063, 730, 688, 542 $cm^{-1}$; $^1$H-NMR $\delta$ 1.26—1.76 (18H, m), 2.96 (2H, t, $J$=7.7 Hz), 3.64 (2H, t, $J$=6.7 Hz), 7.44—7.97 (5H, m). MS *m/z* (%) 276 ($M^+$, 14), 120 (100), 105 (66), 77 (16). Calcd for $C_{18}H_{28}O_2$: M, 276.2089. Found: *m/z* 276.2088. 12-Oxo-12-phenyldodecanal **7** was obtained as by-product in entry 2—5, Table 2. Colorless oil. IR (neat) 2916, 2850, 2725, 1717 (CHO), 1680 (CO), 1450, 1366, 1211, 737, 688 $cm^{-1}$; $^1$H-NMR $\delta$ 1.29—1.76 (16H, m), 2.40—2.43 (2H, m), 2.95—2.98 (2H, m), 7.44—7.97 (5H, m), 9.76 (1H, t, $J$=1.9 Hz). MS *m/z* (%) 274 ($M^+$, 4), 133 (14), 120 (100), 105 (84), 77 (26). Calcd for $C_{18}H_{26}O_2$: M, 274.1937. Found: *m/z* 274.1932.

**Diols 8a—f** Diols in Table 3 were synthesized in a similar manner to that described above. 1-Phenyl-1,5-pentanediol **8a**: Colorless oil; IR (neat) 3340 (OH), 2938, 2864, 1454, 1055, 1028, 761, 701 $cm^{-1}$; $^1$H-NMR $\delta$ 1.22—1.86 (6H, m), 3.62 (2H, t, $J$=6.4 Hz), 4.67 (1H, t, $J$=6.4 Hz), 7.25—7.34 (5H, m). MS *m/z* (%) 180 ($M^+$, 10), 108 (8), 107 (100), 79 (36), 77 (17). Calcd for $C_{11}H_{16}O_2$: M, 180.1143. Found: *m/z* 180.1149. 1-(4-Chlorophenyl)dodecane-1,12-diol **8b**: Colorless crystals; mp 64.5—65.5 °C (AcOEt–hexane). IR (KBr) 3271 (OH), 2918, 2849, 1492, 1463, 1052, 827 $cm^{-1}$; $^1$H-NMR $\delta$ 1.25—1.87 (20H, m), 3.63 (2H, m), 4.65 (1H, m), 7.26—7.32 (4H, m). MS *m/z* (%) 312 ($M^+$, trace), 277 (12), 141 (100), 77 (13). Calcd for $C_{18}H_{29}ClO_2$: M, 312.1859. Found: *m/z* 312.1855. *Anal.* Calcd: C, 69.10; H, 9.34; Cl, 11.33. Found: C, 69.29; H, 9.38; Cl, 11.25. 1-(4-Methylphenyl)dodecane-1,12-diol **8c**: Colorless crystals; mp 54.5—55.5 °C (AcOEt–hexane). IR (KBr) 3388 (OH), 2918, 2849, 1463, 1051 $cm^{-1}$; $^1$H-NMR $\delta$ 1.25—1.80 (20H, m), 2.34 (3H, s), 3.63 (2H, m), 4.64 (1H, m), 7.16—7.24 (4H, m). MS *m/z* (%) 292 ($M^+$, 6), 121 (6), 121 (100), 93 (12), 77 (8). Calcd for $C_{19}H_{32}O_2$: M, 292.2392. Found: *m/z* 292.2340. *Anal.* Calcd: C, 78.03; H, 11.03. Found: C, 78.30; H, 11.10. 13-Phenyltridecane-1,12-diol **8d**: Colorless crystals; mp 58—59 °C (AcOEt–hexane). IR (KBr) 3373 (OH), 2920, 2850, 1084, 740, 697 $cm^{-1}$; $^1$H-NMR $\delta$ 1.24—1.62 (20H, m), 2.62—2.86 (2H, m) 3.62—3.65 (2H, m), 3.81 (1H, m), 7.21—7.52 (5H, m). MS *m/z* (%) 290 ($[M-2H]^+$, 3), 201 (19), 109 (9), 92 (100), 91 (32), 55 (12). *Anal.* Calcd for $C_{19}H_{32}O_2$: C, 78.03; H, 11.03. Found: C, 77.95; H, 11.10. 1-Cyclohexyldodecane-1,12-diol **8e**: Colorless oil; IR (neat) 3335 (OH), 2921, 2852, 1470, 1065 $cm^{-1}$; $^1$H-NMR $\delta$ 0.87—1.88 (30H, m), 3.33—3.37 (1H, m), 3.62—3.66 (2H, m). FAB-MS *m/z* 285 ($[M+H]^+$), 267, 201, 183, 137, 109, 97. Pentadec-13-ene-1,12-diol **8f**: Colorless crystals; mp 72.5—73 °C (AcOEt–hexane). IR (KBr) 3366 (OH), 2917, 2850, 1462, 1055, 730 $cm^{-1}$; $^1$H-NMR $\delta$ 1.27—1.72 (25H, m), 3.64 (2H, t, $J$=6.7 Hz), 4.46 (1H, dt, $J$=7.2, 6.7 Hz ), 5.37—5.50 (1H, m), 5.54—5.69 (1H, m). MS *m/z* (%) 242 ($M^+$, 4), 224 (6), 86 (11), 81 (9), 77 (100), 55 (14). Calcd for $C_{15}H_{30}O_2$: M, 242.2236. Found: *m/z* 242.2244.

**Ketoalcohols 9a—f** 5-Hydroxy-1-phenyl-1-pentanone **9a**: Colorless oil. IR (neat) 3419 (OH), 2929, 1668 (CO), 1621, 1288, 1038 $cm^{-1}$; $^1$H-NMR $\delta$ 0.68—1.28 (4H, m), 2.58—2.62 (2H, m), 3.85 (2H, t, $J$=6.3 Hz), 7.46—7.95 (5H, m). FAB-MS *m/z* (%) 179 ($[M+H]^+$), 177, 159, 148, 104, 68. 1-(4-Chlorophenyl)-12-hydroxy-1-dodecanone **9b**: Colorless oil. IR (neat) 3344 (OH), 2915, 2849, 1681 (CO), 1590, 1472, 1463, 1400, 1208, 1099, 1066 $cm^{-1}$; $^1$H-NMR $\delta$ 1.25—1.35 (14H, m), 1.56 (2H, quintet, $J$=7.1 Hz), 1.72 (2H, quintet, $J$=7.1 Hz), 2.93 (2H, t, $J$=7.3 Hz), 3.64 (2H, t, $J$=6.4 Hz), 7.43 (2H, d, $J$=7.3 Hz), 7.90 (2H, d, $J$=7.3 Hz). FAB-MS *m/z* 311 ($[M+H]^+$), 246, 185, 155, 154, 107, 95. Calcd for $C_{18}H_{28}O_2Cl$: M, 311.1781. Found: *m/z* 311.1772. 1-(4-Methylphenyl)-12-hydroxy-1-dodecanone **9c**: Colorless oil. IR (neat) 3430 (OH), 2915, 2849, 1682 (CO), 1608, 1463, 1379, 1184, 1069, 813 $cm^{-1}$; $^1$H-NMR $\delta$ 1.25—1.34 (14H, m), 1.56 (2H, quintet, $J$=7.3 Hz), 1.72 (2H, quintet, $J$=7.3 Hz), 2.41 (3H, s), 2.93 (2H, t, $J$=7.3 Hz), 3.64 (2H, t, $J$=6.6 Hz), 7.25 (2H, d, $J$=8.3 Hz), 7.86 (2H, d, $J$=8.3 Hz). FAB-MS *m/z* 291 ($[M+H]^+$), 289, 246, 185, 154, 137, 119, 93. Calcd for $C_{19}H_{31}O_2$: M, 291.2318. Found: *m/z* 291.2340. 13-Hydroxy-1-phenyltridecan-2-one **9d**: Colorless oil. IR (neat) 3284 (OH), 2916, 2849, 1708 (CO), 1464, 1379, 1074 , 752, 699 $cm^{-1}$; $^1$H-NMR $\delta$ 1.23—1.62 (18H, m), 2.43 (2H, m), 3.63—3.65 (2H, m), 3.68 (2H, s), 7.19—7.34 (5H, m). FAB-MS *m/z* 291 ($[M+H]^+$), 289, 246, 185, 154, 137, 119, 93. 1-Cyclohexyl-12-hydroxy-1-dodecanone **9e**: Colorless oil. IR (neat) 3435 (OH), 2928, 2852, 1702 (CO), 1471, 1382, 1057, 718 $cm^{-1}$; $^1$H-NMR $\delta$ 1.19—1.36 (24H, m), 1.53—1.83 (4H, m), 2.27—2.36 (1H, m), 2.42 (2H, t, $J$=7.3 Hz), 3.63 (2H, t, $J$=6.7 Hz). FAB-MS *m/z* 283 ($[M+H]^+$), 265, 199, 181, 137, 83. 15-Hydroxypentadec-2-en-4-one **9f**: Colorless oil. IR (neat) 3356 (OH), 2916, 2849, 1685 (CO), 1447, 1063, 730, 688 $cm^{-1}$; $^1$H-NMR $\delta$ 1.28—1.66 (18H, m), 1.90 (3H, dd, $J$=6.7, 1.6 Hz), 2.51(2H, t, $J$=7.5 Hz), 3.64 (2H, t, $J$=6.5 Hz), 6.12 (1H, dq, $J$=15.9, 1.6 Hz), 6.84 (1H, dq, $J$=15.9, 6.7 Hz). FAB-MS *m/z* 241 ($[M+H]^+$), 209, 199, 148, 132, 104, 102, 69.

**Keto-aldehyde (10b—e)** 12-(4-Chlorophenyl)-12-oxododecanal **10b**: Colorless oil. IR (neat) 2915, 2849, 1715 (CHO), 1681 (CO), 1590, 1463, 1099, 999 $cm^{-1}$; $^1$H-NMR $\delta$ 1.25—1.29 (12H, m), 1.54—1.75 (4H, m), 2.40—2.44 (2H, m), 2.91—2.94 (2H, m), 7.43 (2H, d, $J$=6.8 Hz), 7.90 (2H, d, $J$=6.8 Hz), 9.77 (1H, t, $J$=1.8 Hz). FAB-MS *m/z* 309 ($[M+H]^+$), 221, 185, 136, 92. 12-(4-Methylphenyl)-12-oxododecanal **10c**: Colorless oil. IR (neat) 2916, 2850, 1718 (CHO), 1681 (CO), 1464, 801 $cm^{-1}$; $^1$H-NMR $\delta$ 1.25—1.36 (12H, m), 1.59—1.75 (4H, m), 2.41 (3H, s), 2.42 (2H, dt, $J$=7.3, 1.9 Hz), 2.93 (2H, t, $J$=7.3 Hz), 7.25 (2H, d, $J$=8.6 Hz), 7.86 (2H, d, $J$=8.6 Hz), 9.76 (1H, t, $J$=1.6 Hz). FAB-MS *m/z* 289 ($[M+H]^+$), 255, 197, 173, 136, 91. 12-Oxo-13-phenyltridecanal **10d**: Colorless oil. IR (neat) 2916, 2849, 1714 (CHO, CO), 1462, 1414, 1084, 699 $cm^{-1}$; $^1$H-NMR $\delta$ 1.22—1.28 (14H, m), 1.52—1.64 (4H, m), 2.39—2.44 (4H, m), 3.68 (2H, s), 7.19—7.34 (5H, m), 9.76 (1H, t, $J$=1.9 Hz). FAB-MS *m/z* 289 ($[M+H]^+$), 273, 221, 147, 118, 72. 12-Cyclohexyl-12-oxododecanal **10e**: Colorless oil IR (neat) 2930, 2852, 2725, 1719 (CHO), 1698 (CO), 1471, 1410, 1387, 1148, 1082, 1002 $cm^{-1}$; $^1$H-NMR $\delta$ 1.15—1.36 (18H, m), 1.52—1.57 (2H, m), 1.59—1.68 (2H, m), 1.74—1.83 (4H, m), 2.29—2.36 (1H, m), 2.40—2.44 (4H, m), 9.76 (1H, t, $J$=1.5 Hz). FAB-MS *m/z* 281 ($[M+H]^+$), 221, 197, 135, 83, 73.

**3-Hydroxy-1-phenyl-1-butanone (13)** Colorless oil. IR (neat) 3421 (OH), 2971, 1682 (CO), 1598, 1449, 1374, 1283, 1214, 754, 690, 500 $cm^{-1}$. $^1$H-NMR $\delta$ 1.31 (3H, d, $J$=6.5 Hz), 3.05 (1H, dd, $J$=17.7, 8.9 Hz), 3.18 (1H, d, $J$=17.7, 2.8 Hz), 3.35 (1H, s, OH), 4.40—4.43 (1H, m), 7.48 (2H, t, $J$=7.6 Hz), 7.59 (1H, t, $J$=7.6 Hz), 7.96 (2H, d, $J$=7.6 Hz). MS *m/z* (%) 164 ($M^+$, 10), 146 (11), 120 (15), 105 (100), 77 (45), 51 (13). Calcd for $C_{10}H_{12}O_2$: M, 164.0830. Found: *m/z* 164.0837.

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**References and Notes**

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