Oxidative Cleavage of *vic*-Diols to Aldehydes with Dioxygen Catalyzed by $Ru(PPh_3)_3Cl_2$ on Activate Carbon

Supporting Information

Eiichiro Takezawa, Satoshi Sakaguchi, Yasutaka Ishii*

Department of Applied Chemistry, Faculty of Engineering,

& High Technology Research Center, Kansai University, Suita, Osaka 564-8680, Japan

Experimental Procedure and Compound Characterization Data

General. ^1H and ^{13}C NMR spectra were recorded at 400 and 100 MHz, respectively, using $CDCl_3$ with tetramethylsilane as the internal standard. Infrared (IR) spectra were measured using NaCl or KBr pellets. Flash chromatography was performed with use of silica gel (MERCK, Silica gel 60, 70-230 mesh). Gas chromatography was carried out on Shimazu GC-17A with a flame ionization detector using a 0.22 mm x 25 m capillary column (SGE BP–5). All starting materials and solvent were purchased from commercial sources and used without further treatment.

Synthesis of *tans*-2-methoxycyclooctanol: Epoxidation of cyclooctene was performed by methyltrioxorhenium/pyridine/H_2O_2 system.[1)] The following methanolysis of cyclooctene oxide (10 mmol) with 1-N H_2SO_4 aqueous solution (4 mL) in methanol (20 mL) gave *trans*-2-methoxycyclooctanol. ^1H NMR ($CDCl_3$, 400 MHz) δ 1.47-1.90 (m, 12H), 2.75 (brs, 1H), 3.10-3.15 (td, 1H, *J*=2.20 Hz), 3.38 (S, 3H), 3.58-3.63 (td, 1H, *J*=8.25 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 23.54, 24.59, 25.70, 26.23, 26.78, 30.17, 56.38, 74.64, 86.37.

Compound Characterization Data. Aldehydes **2**, butanal, pentanal, hexanal, benzaldehyde, *o*-phthalaldehyde, and dicarboxylic acid adipic acid, suberic acid, dodecanedioic acid, and phthalide were identified through the comparison of their ^{1}H and ^{13}C NMR with those authentic samples. Acetals were identified with that obtained from the acetalization of diol and aldehyde under acidic conditions separately.

1,8-Octanedial. (^{1}H NMR, $CDCl_3$, 400 MHz) δ 1.34-1.38 (m, 4H), 1.62-1.66 (t, 4H, J=7.33 Hz), 2.42-2.46 (td, 4H, *J*=1.47, 7.33 Hz), 9.76-9.77 (t, 2H, *J*=1.47 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.76, 28.82, 43.71, 202.53.

1,12-Dodecanedial. (^{1}H NMR, $CDCl_3$, 400 MHz) δ 1.29 (m, 12H), 1.61-1.64 (t, 4H, *J*=7.33 Hz), 2.40-2.44 (td, 4H, *J*=1.83, 7.33 Hz), 9.76-9.77 (t, 1H, *J*=1.83); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 22.04, 29.11, 29.29, 202.89.

1-Formylcyclopentene. (^{1}H NMR, $CDCl_3$, 400 MHz) δ 1.97-2.05 (m, 2H, *J*=7.33, 7.69 Hz), 2.51-2.55 (m, 2H, *J*=1.83, 7.69 Hz), 2.56-2.64 (m, 2H, *J*=2.20, 7.33 Hz), 6.88-6.89 (t, 1H, *J*=1.83 Hz), 9.80 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 22.89, 28.27, 33.64, 147.89, 153.20, 189.92.

Reference

1. Rudolph, J.; Reddy, K. L.; Chiang, J. P.; Sharpless, K. B., *J. Am. Chem. Soc.* **1997**, *119*, 6189.