

Supporting Information

Acceptorless dehydrogenative lactonization of diols by Pt-loaded SnO₂ catalysts

Abeda Sultana Touchy,^a Ken-ichi Shimizu*^{a,b}

^a Catalysis Research Center, Hokkaido University, N-21, W-10, Sapporo 001-0021, Japan

^b Elements Strategy Initiative for Catalysts and Batteries, Kyoto University, Katsura, Kyoto 615-8520, Japan

*Corresponding author

Ken-ichi Shimizu

Catalysis Research Center, Hokkaido University, N-21, W-10, Sapporo 001-0021, Japan

E-mail: kshimizu@cat.hokudai.ac.jp, Fax: +81-11-706-9163

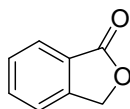
Experimental Section

General: Commercially available organic and inorganic compounds (from Tokyo Chemical Industry, Wako Pure Chemical Industries, Kishida Chemical, or Mitsuwa Chemicals) were used without further purifications. The GC (Shimadzu GC-14B) and GCMS (Shimadzu GCMS-QP2010) analyses were carried out with Ultra ALLOY capillary column UA⁺-5 (Frontier Laboratories Ltd.) using nitrogen as the carrier gas.

NMR and GC/MS analysis

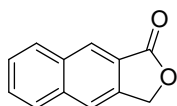
¹H and ¹³C NMR spectra for lactones of Table-3 were assigned and reproduced to the corresponding literature. ¹H and ¹³C NMR spectra were recorded using at ambient temperature on JEOL-ECX 600 operating at 600.17 and 150.92 MHz, respectively with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts are reported relative to tetramethylsilane and *d*-solvent peaks 77.00 ppm chloroform. Abbreviations used in the NMR experiments: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. GC-MS spectra was taken by SHIMADZU QP2010.

Isobenzofuran-1(3*H*)-one:¹



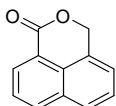
^1H NMR (600.17 MHz, CDCl_3 , TMS): δ 7.93 (d, $J = 7.8$ Hz, 1H), 7.71-7.68 (m, 1H), 7.55-7.50 (m, 2H), 5.33 (s, 2H); ^{13}C NMR (150.92 MHz, CDCl_3) δ 171.06, 146.46, 133.96, 128.95, 125.62, 125.58, 121.93, 69.60; GC-MS m/e 134.08.

3H-Naptho[2,3-c]furan-1-one:²



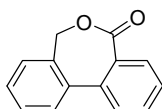
^1H NMR (600.17 MHz, CDCl_3 , TMS): δ 8.50 (s, 1H), 8.04 (d, $J = 8.28$ Hz, 1H), 7.94 (d, $J = 8.22$ Hz, 1H), 7.89 (s, 1H), 7.68-7.65 (m, 1H), 7.61-7.58 (m, 1H), 5.48 (s, 2H); ^{13}C NMR (150.92 MHz, CDCl_3) δ 170.96, 139.93, 136.23, 133.11, 129.93, 128.98, 128.11, 127.06, 126.93, 123.31, 120.85, 69.61; GC-MS m/e 184.05.

3H-Benzo[d,e]isochromen-1-one:²



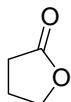
^1H NMR (600.17 MHz, CDCl_3 , TMS): δ 8.41 (d, $J = 7.50$ Hz, 1H), 8.12 (d, $J = 8.22$ Hz, 1H), 7.87 (d, $J = 8.18$ Hz, 1H), 7.67 (t, $J = 7.56$ Hz, 1H), 7.57 (t, $J = 8.28$ Hz, 1H), 7.38 (d, $J = 6.18$ Hz, 1H), 5.85 (s, 2H); ^{13}C NMR (150.92 MHz, CDCl_3) δ 164.32, 135.33, 133.44, 131.72, 130.40, 127.47, 126.55, 125.79, 125.25, 121.54, 118.60, 70.14; GC-MS m/e 184.01.

Dibenzo[c,e]oxepin-5(7H)-one:¹



^1H NMR (600.17 MHz, CDCl_3 , TMS): δ 7.98 (d, $J = 8.28$ Hz, 1H), 7.72-7.62 (m, 2H), 7.60-7.58 (m, 1H), 7.54-7.48 (m, 2H), 7.44-7.41 (m, 2H), 5.03 (s, 1H), 4.97 (s, 1H); ^{13}C NMR (150.92 MHz, CDCl_3) δ 170.21, 138.90, 137.20, 134.26, 132.52, 131.86, 130.58, 130.07, 128.63, 128.60, 128.49, 128.36, 127.52, 69.12; GC-MS m/e 210.10.

Dihydrofuran-2(3H)-one:³



¹H NMR (600.17 MHz, CDCl₃, TMS): δ 4.27 (t, *J* = 7.02 Hz, 2H), 2.41 (t, *J* = 7.79 Hz, 2H), 2.22-2.17 (m, 2H); ¹³C NMR (150.92 MHz, CDCl₃) δ 177.65, 68.36, 27.54, 21.92; GC-MS m/e 86.07.

Tetrahydro-2H-pyran-2-one:¹



¹H NMR (600.17 MHz, CDCl₃, TMS): δ 4.35 (t, *J* = 5.58 Hz, 2H), 2.56 (t, *J* = 7.20 Hz, 2H), 1.92-1.86 (m, 4H); ¹³C NMR (150.92 MHz, CDCl₃) δ 171.27, 69.31, 29.66, 22.12, 18.91; GC-MS m/e 100.055.

Oxepan-2-one:⁴



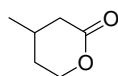
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 4.16 (t, *J* = 4.53 Hz, 2H), 2.56 (t, *J* = 6.08 Hz, 2H), 1.79-1.77 (m, 2H), 1.71-1.67 (m, 4H); ¹³C NMR (150.92 MHz, CDCl₃) δ 176.07, 69.10, 34.32, 29.05, 28.68, 22.71; GC-MS m/e 114.05.

4-Methyl-oxetan-2-one:⁵



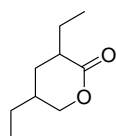
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 4.56-4.52 (m, 1H), 3.52-3.50 (m, 1H), 3.12-3.10 (m, 1H), 1.51 (d, *J* = 6.28 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃) δ 171.05, 64.39, 35.82, 20.96; GC-MS m/e 86.11

4-Methyl-tetrahydro-pyran-2-one:⁶



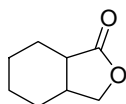
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 4.43-4.39 (m, 1H), 4.18-4.17 (m, 1H), 2.60-2.58 (m, 1H), 2.22-2.26 (m, 2H), 1.85-1.79 (m, 1H), 1.60-1.55 (m, 1H), 1.09 (d, *J* = 6.18 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃) δ 171.27, 68.43, 37.96, 30.35, 26.29, 21.18; GC-MS *m/e* 114.06.

3,5-Diethyl-tetrahydro-pyran-2-one:



¹H NMR (600.17 MHz, CDCl₃, TMS): δ 4.21-4.19 (m, 1H), 4.12-4.11 (m, 1H), 2.30-2.28 (m, 2H), 2.14-2.199 (m, 2H), 1.62-1.58 (m, 2H), 1.38-1.35 (m, 2H), 0.83-0.85 (m, 6H); ¹³C NMR (150.92 MHz, CDCl₃) δ 175.01, 72.53, 40.90, 38.32, 34.67, 24.54, 22.35, 11.09, 10.87; GC-MS *m/e* 156.15.

Hexahydro-isobenzo-furan-1-one:⁷



¹H NMR (600.17 MHz, CDCl₃, TMS): δ 4.21-4.19 (m, 1H), 3.95-3.91 (m, 1H), 2.64-2.70 (m, 1H), 2.54-2.49 (m, 1H), 1.94-1.92 (m, 1H), 1.68-1.57 (m, 4H), 1.28-1.20 (m, 3H); ¹³C NMR (150.92 MHz, CDCl₃) δ 178.26, 71.84, 39.23, 35.77, 26.57, 23.41, 22.84, 22.43; GC-MS *m/e* 140.10.

References

1. S. Jhulki, S. Seth, M. Mondal, J. N. Moorthy, *Tetrahedron*, 2014, **70**, 2286–2293.
2. K. I. Fujita, w. Ito, R. Yamaguchi, *ChemCatChem.*, 2014, **6**, 109–112.
3. X. Li, Y. Cui, X. Yang, W. L. Dai, K. Fan, *Appl. Catal. A : General*, 2013, **458**, 63–70.
4. T. Buntara, S. Noel, P. H. Phua, I. M. Cabrera, J. G. Vries and H. J. Heeres, *Angew. Chem. Int. Ed.*, 2011, **50**, 7083–7087.
5. M. C. Bagley, Z. Lin, D. J. Philips, A. E. Graham, *Tetrahedron Lett.*, 2009, **50**, 6823–6825.
6. T. Tokoroyama, H. Kusaka, *Can. J. Chem.*, 1996, **74**, 2487–2502.
7. Y. Endo, J. E. Bäckvall, *Chem. Eur. J.*, 2011, **17**, 12596–12601.