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Dispiro-1,2,4,5-Tetraoxanes via Ozonolysis of Cycloalkanone *O*-Methyl Oximes: A Comparison with the Peroxidation of Cycloalkanones in Acetonitrile-Sulfuric Acid Media

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Supporting Information

General procedure for preparation of *O*-methyl oximes:[12] 30 mmol of the ketone, 45 mmol of methoxylamine hydrochloride, 30 ml of methanol and 4.5 ml of pyridine were added to a 100-ml round-bottomed flask and the mixture was kept at room temperature for 2 days. The reaction mixture was concentrated to a syrup by distillation at reduced pressure and 50 ml of CH_2Cl_2 and 50 ml of water were added and the organic layer was separated. The aqueous layer was extracted with 30 ml of CH_2Cl_2 and the combined organic extracts were washed with two 30-ml portions of 1 M HCl, with 30 ml of saturated aqueous sodium chloride, and then dried over magnesium sulfate. The solvent was distilled at reduced pressure to give the *O*-methyl oxime.

***O*-Methyl 2-adamantanone oxime (1a):** yield: 89%; Colorless solid; mp 70 °C (CH_3OH); ^{1}H-NMR: 1.60-2.10 (m, 12H), 2.54 (s, 1H), 3.47 (s, 1H), 3.82 (s, 3H).

***O*-Methyl 2-norbornanone oxime (1b):** yield: 88%; Colorless liquid; ^{1}H-NMR: 1.00-1.53 (m, 4H), 1.54-1.85 (m, 2H), 1.95-2.15 (m, 1H), 2.16-2.35 (m, 1H), 2.50 (s, 1H), 2.86 (s, 1H), 3.82 (s, 3H).

***O*-Methyl 4-methylcyclohexanone oxime (1c):** yield: 85%; Colorless liquid; ^{1}H-NMR: 0.94 (d, J = 6.6 Hz, 3H), 1.00-1.25 (m, 2H), 1.50-1.92 (m, 4H), 2.01-2.17 (m, 1H), 2.29-2.42 (m, 1H), 3.09-3.21 (m, 1H), 3.82 (s, 3H).

***O*-Methyl 4-*tert*-butylcyclohexanone oxime (1d):** yield: 93%; Colorless liquid; ^{1}H-NMR: 0.87 (s, 9H), 1.00-1.31 (m, 3H), 1.60-1.76 (m, 1H), 1.85-2.15 (m, 3H), 2.35-2.50 (m, 1H), 3.20-3.35 (m, 1H), 3.82 (s, 3H).

***O*-Methyl 2-*tert*-butylcyclohexanone oxime (1e):** yield: 85%; Colorless liquid; ^{1}H-NMR: 1.03 (s, 9H), 1.35-1.95 (m, 6H), 1.96-2.06 (m, 1H), 2.25-2.41 (m, 1H), 2.50-2.65 (m, 1H), 3.83 (s, 3H).

***O*-Methyl tetrahydro-4*H*-pyran-4-one oxime (1f):** yield: 68%; Colorless liquid; ^{1}H-NMR: 2.38 (t, J = 5.6 Hz, 2H), 2.63 (t, J = 5.7 Hz, 2H), 3.76 (t, J = 5.9 Hz, 2H), 3.83 (t, J = 5.7 Hz, 2H), 3.86 (s, 3H).