

#### Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>



ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1998 American Chemical Society

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

Yuxiang Dong and Jonathan L. Vennerstrom\*

Department of Pharmaceutical Sciences, College of Pharmacy, University of Nebraska  
Medical Center, Omaha, NE 68198-6025

**Supporting Information**

**General procedure for preparation of *O*-methyl oximes:**<sup>12</sup> 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<sub>2</sub>Cl<sub>2</sub> and 50 ml of water were added and the organic layer was separated. The aqueous layer was extracted with 30 ml of CH<sub>2</sub>Cl<sub>2</sub> 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<sub>3</sub>OH); <sup>1</sup>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; <sup>1</sup>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;  $^1\text{H}$ -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;  $^1\text{H}$ -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;  $^1\text{H}$ -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;  $^1\text{H}$ -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).