Experimental Section

General Methods. General details are as previously described.⁶

4,4-Dimethoxybutanal (1a). The relative rate of ozonolysis was tested by a small scale diozonolysis. A solution of 1,5cyclooctadiene (8) (1.0 g, 9.2 mmol) in 10 mL of methylene chloride and 10 mL of methanol was ozonized at -78 °C. After 18 min,⁷ the solution turned blue, indicating the complete ozonolysis. Under these conditions, a solution of 1,5-cyclooctadiene (8) (15.0 g, 138.6 mmol) in 150 mL of methylene chloride and 150 mL of methanol was ozonized at -78 °C. After 135 min of ozonolysis,⁷ TsOH·H₂O (2 g, 10.5 mmol) was then added. After the solution was stirred at room temperature for 2 h, dimethyl sulfide (15 mL) was added, and this mixture was stirred at room temperature overnight. After workup with aqueous NaHCO3 and CHCl3, the crude product 9 was treated with ozone at -78 °C until the solution turned blue. Dissolved ozone was removed by flushing the solution with argon. Dimethyl sulfide (15 mL) was then added. After the solution was stirred at room temperature for 3-4 h, the solvents were removed in the hood by distillation. The residue was dissolved in diethyl ether (250 mL) and washed with water (2×100 mL). After the removal of diethyl ether by evaporation, the residue

(7) The reaction time depends on the setup of ozone generator.

was purified by vacuum distillation (69–72 °C, 10 mmHg) to give 4,4-dimethoxybutanal (**1a**) (30 g, 227.0 mmol, 82% overall yield) as a colorless oil: ¹H NMR (200 MHz, CDCl₃) δ 9.73 (1H, t, *J* = 1.5 Hz), 4.35 (1H, t, *J* = 5.5 Hz), 3.30 (6H, s), 2.48 (2H, dt, *J* = 1.5, 7.2 Hz), 1.91 (2H, dt, *J* = 5.5, 7.2 Hz); ¹³C NMR (50 MHz, CDCl₃) δ 202.0, 104.3, 53.9, 39.4, 26.0; MS *m*/*z* (intensity) 132 (0.01, M⁺), 131 (0.6, M⁺ – 1), 101 (29, M⁺ – CH₃O), 75 (100, M⁺ – CH₂CH₂CHO).

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Supporting Information Available: Copies of ¹H and ¹³C NMR spectra of compounds **1a** and **9** (4 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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Additions and Corrections

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Robert Reinhard and Brigitte F. Schmidt. Nitrobenzyl-Based Photosensitive Phosphoramide Mustards: Synthesis and Photochemical Properties of Potential Prodrugs for Cancer Therapy.

Page 2441. **Supporting Information Available** paragraph should be added.

¹H and ¹³C NMR spectra for compounds prepared (48 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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⁽⁶⁾ Gi, H.; Xiang, Y.; Schinazi, R.; Zhao, K. J. Org. Chem. 1997, 62, 88.