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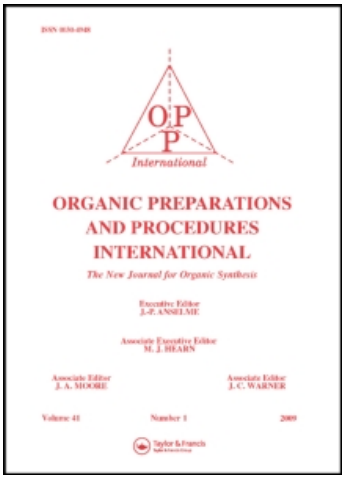

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THE SPECTRAL CHARACTERIZATION OF *endo*-4-OXATRICYCL[5,2,1,$0^{2,6}$]DEC-2-ENE

Bradford P. Mundy[a]
[a] Department of Chemistry, Montana State University, Bozeman, Montana

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THE SPECTRAL CHARACTERIZATION OF endo-4-OXATRICYCLO[5,2,1,$O^{2,6}$]DEC-2-ENE

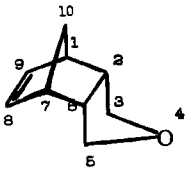
Submitted by (8/20/74)

Bradford P. Mundy

Department of Chemistry
Montana State University
Bozeman, Montana 59715

Characterization of the title compound by nmr resulted in a simple spectrum (δ values from tetramethylsilane, CCl_4 solvent) of broad singlets:

δ 1.44 [2H] (C-10); 2.76 [4H] (C-1,C-2,C-6,C-7);
3.30 [4H] (C-3,C-5); 6.01 [2H] (C-8,C-9).



Addition of 40.1 mg of $Eu(fod)_2$ to 64.5 mg of I in .45 ml of CCl_4 separated the C-1 (C-7) protons from the C-2 (C-6) protons; shifting the signals to 3.25 and 3.56 δ respectively. The other proton signals remained as broad singlets. The mass spectrum of I exhibited $^m/e$ 136 for the molecular ion with a very strong base peak at $^m/e$ 66 (cyclopentadiene) presumably arising from a retro-Diels-Alder reaction. The $^m/e$ 70 peak (Δ^3- dihydrofuran) was about 50% of the molecular ion. The infrared spectrum (KBr pellet) exhibited little of interest, with major absorptions at 3060, 2905 and 2880 (C-H) and 1095 cm^{-1} (cyclic ether).

Purification. - The crude semi-solid formed according to literature procedures[1-3] resisted attempts at crystallization, and could be purified by elution chromatography on a Florisil column with pentane leaving a dark band on the column. Evaporation of the pentane left 2.4 g (35%) of a white solid, mp. 84-86° (uncorrected).

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