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Synthesis of 3-Ethoxy-2-Oxo-3-Phenyl-1,2-Oxaphosphorinane-3,5-Diene

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Synthesis of 3-Ethoxy-2-Oxo-3-Phenyl-1,2-Oxaphosphorinane-3,5-Diene

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In view of the limited number of examples of 2-oxo-1,2-oxaphosphorinane-3,5-diene derivatives^{1,2}, we investigated the preparation of the title compound and derivatives of the same. Of additional interest were the spectroscopic properties of this class of compounds, their stability, and the outcome of their reaction with nucleophiles. Prior work in our laboratory on the synthesis of simple phostones and their derivatives³, led us to use these as starting materials. The presence of the phenyl substituent in 1a,b (cis and trans isomers) permitted facile introduction of a bromo group through free radical bromination with NBS/AIBN to give 2a,b in 79% overall yield. The individual isomers of 2 were separated; tentative stereochemical assignments were made using NMR spectroscopy. Treatment of 2 with LiCl/DMF gave 3; the trans isomer (phenyl and OEt trans) 2b reacted much faster than the cis isomer. Treatment of 3 with NBS/AIBN gave 4a,b and 5 which were separated by flash chromatography. Dehydrobromination of 4 (71% yield) was achieved by heating with an excess of Et₃N in toluene at 95 °C to produce 6; likewise, dehydrobromination of 5 at 70 °C gave 7.

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