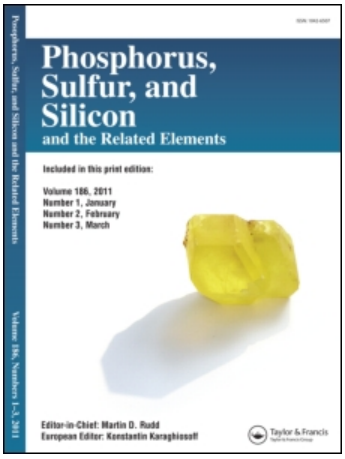


## Phosphorus, Sulfur, and Silicon and the Related Elements

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# Diastereoselective Synthesis of Phosphines and Phosphoranes using Fluorinated Acetylacetones

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# Diastereoselective Synthesis of Phosphines and Phosphoranes using Fluorinated Acetylacetones

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A two step formal insertion of 1,1,1,5,5,5-hexafluoro- (**1**) and 1,1,1-trifluoropentane-2,4-dione (**2**) into the P–H bonds of phosphane gave the primary α-hydroxyphosphanes, precursors for 2-phospha-6-oxa-9-oxabicyclo[3.3.1]-nonane and 2,4,8-trioxa-6-phospha-adamantane, both formed diastereospecifically. The molecular structures of the two latter compounds were established by single-crystal X-ray structure analysis. Compound **1** reacted diastereospecifically with phosphonous acid dichlorides, $RPCl_2$ (R = Me, Et, *i*Pr, *t*Bu, $Me_3SiCH_2$, $PhCH_2$, Ph) to give in a concerted mechanism thermally stable tricyclic $\lambda^5\sigma^5$P phosphoranes containing two five- and one six-membered ring. In one case hydrolysis gave 3,5-dihydroxy-2-oxo-1,2$\lambda^5\sigma^4$-oxaphospholane, whereas methanol added to the double bond in the six-membered ring furnishing two isomeric phosphoranes. When **2** was reacted with $RPCl_2$ (R = Et, $Me_3SiCH_2$, $PhCH_2$, Ph), diastereomerically pure regioisomeric phosphoranes were obtained. The solid state molecular structures of three $\lambda^5\sigma^5$P species exhibited two oxygen atoms in the axial position of a slightly distorted trigonal-bipyramidal geometry at phosphorus.