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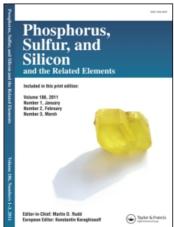
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## Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information: <a href="http://www.informaworld.com/smpp/title~content=t713618290">http://www.informaworld.com/smpp/title~content=t713618290</a>

## Diastereoselective Synthesis of Phosphines and Phosphoranes using Fluorinated Acetylacetones

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**To cite this Article** Bekiaris, Georgios and Röschenthaler, Gerd-Volker(1999) 'Diastereoselective Synthesis of Phosphines and Phosphoranes using Fluorinated Acetylacetones', Phosphorus, Sulfur, and Silicon and the Related Elements, 147: 1, 359

To link to this Article: DOI: 10.1080/10426509908053659 URL: http://dx.doi.org/10.1080/10426509908053659

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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 1,1,1trifluoropentane-2,4-dione (2) into the P-H bonds of phosphane gave the primary α-hydroxyphosphanes, precursors for 2-phospha-6-oxa-9oxabicyclo[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<sub>2</sub> (R = Me, Et, iPr, tBu, Me<sub>3</sub>SiCH<sub>2</sub>, PhCH<sub>2</sub>, 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<sub>2</sub> (R = Et, Me<sub>3</sub>SiCH<sub>2</sub>, PhCH<sub>2</sub>, 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.