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A Novel Resolution Procedure for the Preparation of P-Stereogenic Phosphine Oxides

Andersen, Neil G.; Ramsden, Philip D.; Che, Daqing; Parvez, Masood; Keay, Brian A.

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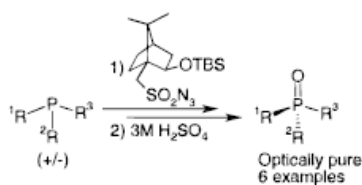
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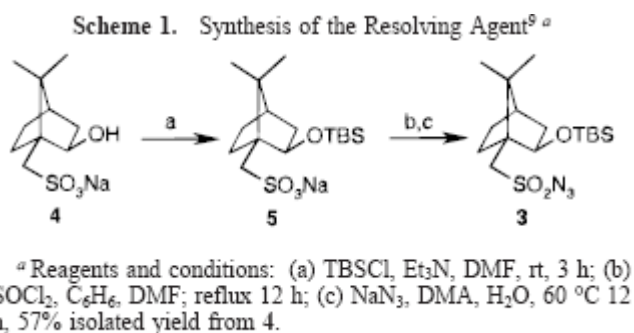
A Novel Resolution Procedure for the Preparation of P-Stereogenic Phosphine Oxides
Neil G. Andersen, Philip D. Ramsden, Daqing Che, Masood Parvez, and Brian A. Keay
pp 2009 – 2011.

Abstract:



A new general route for preparing enantiomerically pure P-stereogenic phosphine oxides has been developed by exploiting the Staudinger reaction between racemic tertiary phosphines and an enantiomerically pure organoazide. The resulting phosphinimines are easily resolved by either crystallization or flash chromatography and serve as synthetic intermediates toward enantiomerically pure phosphine oxides.

Schemes:



Figures:

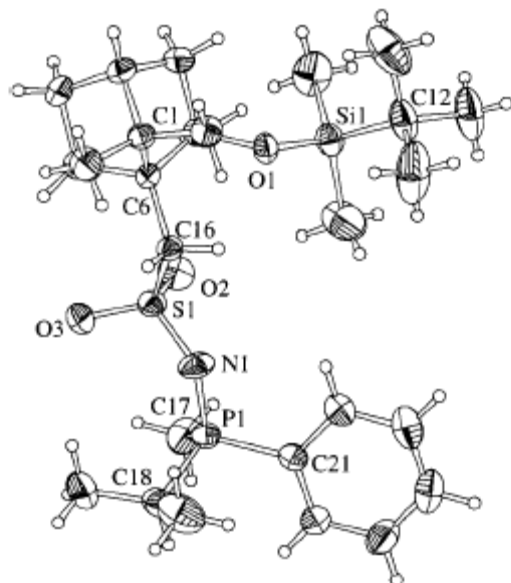
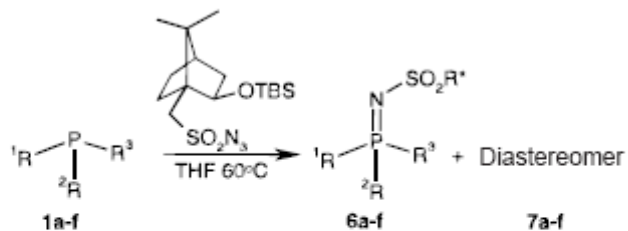


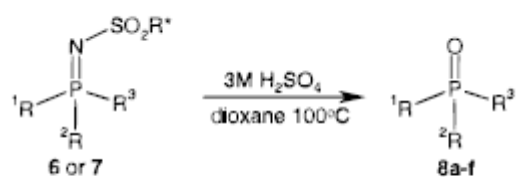
Figure 1. ORTEP drawing of phosphinimine **6c**¹⁸ drawn with 30% probability ellipsoids, except for the hydrogen atoms which are represented as spheres of arbitrary size.

Tables:

Table 1. Treatment of Racemic Phosphines with Azide 3¹⁴



entry	R ¹	R ²	R ³	yield (%)
1a	Ph	Me	C ₆ H ₁₁	94
1b	Ph	Me	C ₅ H ₉	90
1c	Ph	Me	CH(CH ₃) ₂	87
1d	Ph	Me	1-naphthyl	94
1e	Ph	Me	9-phenanthryl	89
1f	Ph	1-naphthyl	<i>p</i> -PhC ₆ H ₄	91

Table 2. Hydrolysis of Isomerically Pure Phosphinimines

SM	R ¹	R ²	R ³	yield (%)
6a	Ph	Me	C ₆ H ₁₁	93
7b	Ph	Me	C ₅ H ₉	93
6c	Ph	Me	CH(CH ₃) ₂	94
6d	Ph	Me	1-naphthyl	96
6e	Ph	Me	9-phenanthryl	>99
7f	Ph	1-naphthyl	<i>p</i> -PhC ₆ H ₄	93

Table 3. Assignment of Phosphinimine Absolute Configuration from Hydrolysis Product Optical Rotation Data

SM	yield (%)	product α_{D}^{20} (<i>c</i> , solvent)	configuration	
			product 8	SM
6a	93	+19.2 (0.93, MeOH) ²¹	<i>R</i> _P	<i>S</i> _P
7b	93	+33.3 (1.62, MeOH)		
6c	94	-22.6 (1.00, MeOH) ²²	<i>S</i> _P	<i>R</i> _P
6d	96	+19.8 (2.92, MeOH) ²³	<i>S</i> _P	<i>R</i> _P
6e	>99	+71.4 (1.14, MeOH) ²⁴		
7f	93	+26.9 (0.62, CHCl ₃) ²⁵	<i>R</i> _P	<i>S</i> _P

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