

This article was downloaded by:

On: 30 January 2011

Access details: Access Details: Free Access

Publisher Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713618290>

The First Chromatographic Resolution of a Chiral Phosphane. An Efficient Synthesis of (R,R)- and (S,S)-DIPAMP

H. Bokel^a; H. Pflug^a; E. Merck^a

^a Pharmaforschung Postfach, Darmstadt

To cite this Article Bokel, H. , Pflug, H. and Merck, E.(1987) 'The First Chromatographic Resolution of a Chiral Phosphane. An Efficient Synthesis of (R,R)- and (S,S)-DIPAMP', Phosphorus, Sulfur, and Silicon and the Related Elements, 30: 3, 832

To link to this Article: DOI: 10.1080/03086648708079319

URL: <http://dx.doi.org/10.1080/03086648708079319>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

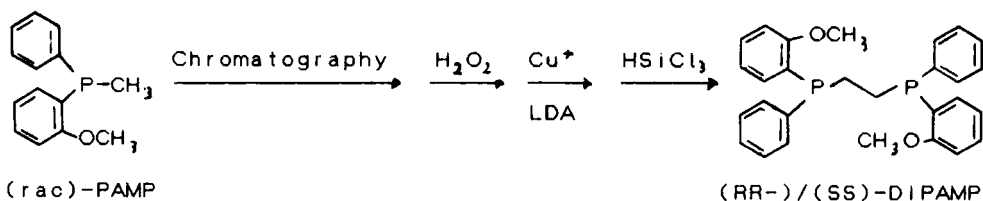
The First Chromatographic Resolution of a Chiral Phosphane. An Efficient Synthesis of (R,R)- and (S,S)-DIPAMP

H. Bokel*, H. Pflug

E. MERCK, Pharmaforschung
Postfach 4119
D-6100 Darmstadt

Resolution of racemic compounds by chromatography on chiral stationary phases (CSPs) offers several advantages compared with "classical" resolution: no waste of either antipode, no dependence on the e.e. of the chiral auxiliary (CSP), no necessity for chemical transformation. The mean loadability of most CSPs can be compensated if a racemic catalyst is resolved: the chromatographically produced "chiral information" will subsequently be multiplied.

PAMP (Phenyl-o-Anisyl-Methyl-Phosphane), is a suitable P-chiral precursor of the very important bis-phosphane DIPAMP (1,2-ethanediylbis-((o-methoxyphenyl)phenylphosphane)), which is used as a Rh(I)-ligand in the L-DOPA-synthesis (1).



Racemic PAMP was synthesised according to a one-pot literature procedure (2) and resolved on cellulose triacetate (CTA) (separation factor = 1.9). Fortunately, CTA is a CSP which is not too easily overloaded (3).

The oxidation of chiral phosphanes with hydrogenperoxide is known to proceed via retention of P-configuration (4). Oxygen must strictly be excluded since racemic phosphaneoxide would be produced.

The enantiomerically pure (R)- respectively (S)-PAMP-oxide was coupled and reduced to DIPAMP as described in literature (1).

- (1) Vineyard, B.D., Knowles, W.S., Sabacky, M.J., Bachman, G.L., Weinkauff, D.J., J. Am. Chem. Soc. 99: 5946 (1977)
- (2) Chodkiewicz, W., Guillermin, D., Jore, D., Mathieu, E., Wodzki, W., J. Organometallic Chem. 269: 107 (1984)
- (3) Rimböck, K.-H., Kastner, F., Mannschreck, A., J. Chromatography 329: 307 (1985)
- (4) Horner, L., Winkler, H., Tetrahedron Lett. 175, (1964)