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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

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To cite this Article Nazarov, A. A. , Hartinger, Ch. , Brehmer, T. H. , Giester, G. , Galanski, M. and Keppler, B. K.(2001) 'New C_2 -Chiral 1,1'-Bis(Oxazoline-2-YL)-Ferrocenes - Synthesis and Crystal Structure', Phosphorus, Sulfur, and Silicon and the Related Elements, 169: 1, 141 - 144

To link to this Article: DOI: 10.1080/10426500108546610 URL: http://dx.doi.org/10.1080/10426500108546610

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New C₂-Chiral 1,1'-Bis(Oxazoline-2-YL)-Ferrocenes – Synthesis and Crystal Structure

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We report here about the synthesis of a few 1,1'-bis(oxazoline-2-yl)-ferrocene derivatives from pure bis(β -hydroxylamide)s via the reaction with methylsulfonylchloride and triethylamine. The bis(β -hydroxylamide)s were prepared from enantiomeric pure amino alcohols and 1,1'-ferrocenedicarbonyl chloride.

Keywords: ferrocene; oxazoline; chiral

INTRODUCTION

For more than twenty years now chiral ferrocenes have been used in the metal catalysed enantioselective synthesis which plays an important role in preparative organic chemistry [1].

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The demand for drugs and agro chemicals in an enantiomerical pure form leads to syntheses where racemates are avoided [2]. The major part of the enantioselective catalysts used are soluble metal complexes of low nuclearity. Alternative metal complexes with chiral ligands which are bound to an insoluble achiral polymeric matrix are also known. Heterogeneous catalysts where chiral ligands are absorbed [3] form a minority. A new approach concerns synthetic catalysts with chiral polymers as ligands. This combination is well known from natural catalysts (enzymes and DNA/RNA) [4].

SYNTHESIS OF 1,1'-BIS[(S)-4-ISOPROPYL-OXAZOLINE-2-YL]-FERROCENE

Under argon atmosphere a solution of 2.32 g ferrocenedicarbonyl dichloride (7.5 mmol) in 10 ml CH₂Cl₂ was added very slowly to 1.55 g (S)-(+)-amino-3-methyl-1-butanol (15 mmol) and 3.88 ml triethylamine (28 mmol) in 50 ml of dry CH₂Cl₂. This reaction mixture was stirred for 72 hours, 60 ml of ether was added and it was quenched with 100 ml of saturated NaHCO₃. The organic phase was washed with 75 ml of saturated NaCl-solution and dried over Na₂SO₄. The solvent was evaporated and the residue dried at high vacuum (yield: 81.7 %).

1.04 g 1,1'-bis[(S)-N-(1-hydroxy-3-methylbutyl)]-ferrocene diamide (2.3 mmol) and 1.33 ml triethylamine (9.6 mmol) were solvated in 30 ml dry dichloromethane under argon atmosphere. A solution of 0.63 g methanesulfonylchloride (5.5 mmol) in 10 ml CH₂Cl₂ was added slowly at 0°C. This mixture was stirred for 48 hours, washed with cold water and saturated NaCl solution and dried over Na₂SO₄. The solvent was evaporated and the resulting solid dried at high vacuum (yield: 66.0 % on ferrocenedicarbonyl chloride).

R = (S)-cthyl, (S)-iso-propyl, (R)-iso-propyl, (S)-2-iso-butyl, (S)-3-iso-butyl, (S)-tert-butyl

FIGURE 1: A general route to synthesise several chiral ferrocene derivatives.

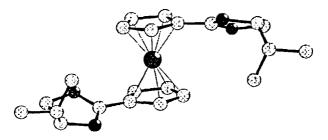


FIGURE 2: Molecular geometry of compound 1 1,1'-bis[(S)-4-isopropyl-oxazoline-2-yl]-ferrocene in crystal structure 1.

CHARACTERISATION

The characterisation was done by ¹H- and ¹³C-NMR-measurement, elemental analysis and X-ray diffraction.

Acknowledgement

We gratefully acknowledge the FWF (Austrian Science Fund) for financing the project.

References

- [1] A. Togni; T. Hayashi in Ferrocenes, 168 (1995).
- [2] R. A. Sheldon, Chirotechnology, Marcel Dekker (1993).
- [3] J. W. ApSimpson, T. L. Collier, Tetrahedron, 42, 5157 (1986).
- [4] H. Alper, N. Hamel, J. Chem. Soc., Chem. Commun., 135 (1990).