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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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Kentaro Watanabe^a; Makoto Yamashita^a; Yohsuke Yamamoto^a; Kin-ya Akiba^b

^a Hiroshima University, Japan ^b Waseda University, Japan

Online publication date: 27 October 2010

To cite this Article Watanabe, Kentaro , Yamashita, Makoto , Yamamoto, Yohsuke and Akiba, Kin-ya(2002) 'Synthesis and Application of New Tridentate Anthracene Ligands Bearing Donative Phosphorus(III) Atoms at 1,8-Positions', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 177: 8, 2047 — 2048

To link to this Article: DOI: 10.1080/10426500213299

URL: <http://dx.doi.org/10.1080/10426500213299>

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SYNTHESIS AND APPLICATION OF NEW TRIDENTATE ANTHRACENE LIGANDS BEARING DONATIVE PHOSPHORUS(III) ATOMS AT 1,8-POSITIONS

Kentaro Watanabe,^a Makoto Yamashita,^a Yohsuke Yamamoto,^a
 and Kin-ya Akiba^b

Hiroshima University, Japan^a and Waseda University, Japan^b

(Received July 29, 2001; accepted December 25, 2001)

A new potential tridentate ligand, 1,8-bis(diisopropylphosphino)-9-bromoanthracene **1**, was prepared in five steps from 1,8-dichloroanthraquinone **2**. 1,8-Dibromo-9-methoxyanthracene **3** could be prepared in three steps from **2** in good yields. Although 1,8-bis-(disubstituted phosphino)-9-methoxyanthracene **4a** and **4b** could be prepared from **3**, reduction by LDBB (lithium di-*tert*-butylbiphenylide) was not successful in **4b**. In contrast, 1,8-bis(diisopropylphosphino)-9-methoxyanthracene **1a** was obtained in 51% yield by reduction with LDBB followed by treatment with BrCF₂CF₂Br. ORTEP drawing of **5** is shown (Figure 1). Shorter P–B bond length is 2.14(1) Å and the longer

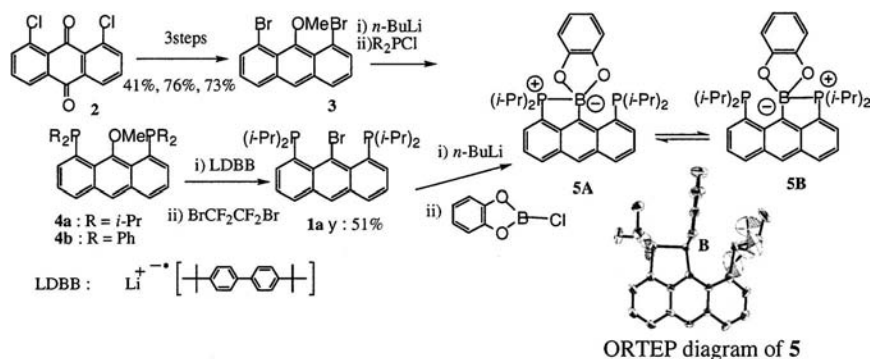


FIGURE 1 Synthesis and X-ray structure of **5**.

Address correspondence to Kin-ya Akiba, Advanced Research Center for Science and Engineering, Waseda University 3-4-1 Ohkubo, Shinjuku-ku, Tokyo 165-8555, Japan.
 E-mail: akibaky@waseda.jp

P–B length is 3.17(1) Å. Although the longer P–B length is shorter than the sum of the van der Waals radii (3.98 Å), the structure of the boron atom should be regarded to be tetracoordinated. Thus, only one of the two *i*-Pr₂P groups were coordinating toward the boron atom.

However, ¹H NMR of **5** showed a symmetrical anthracene pattern (two kinds of doublets and a triplet) at room temperature. In addition, only one signal was observed in the ³¹P NMR. These NMR data indicate that the very rapid bond switching process (**5A** ⇌ **5B**) is taking place in solution.