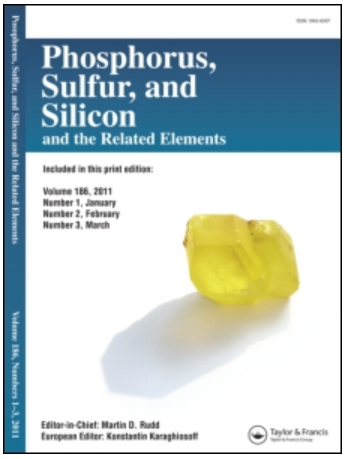

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New Macrocyclic Ligands with $P_2N_4S_2$ Rings in the Framework

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NEW MACROCYCLIC LIGANDS WITH $P_2N_4S_2$ RINGS IN THE FRAMEWORK.

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Abstract Several new macromolecules containing two $Ph_4P_2N_4S_2$ rings linked by meta-xylyl groups have been prepared.

INTRODUCTION

Over the last three years we have developed simple routes to inorganic heterocycles of the type $[Ph_4P_2N_4(SR)(SR')]$ [1] which employ the reactive alkali metal reagents $[Li(Ph_4P_2N_4S_2R)(THF)]_2$ [2] and $M_2[Ph_4P_2N_4S_2]$ (M = Li, Na).[3]

$$M_2[Ph_4P_2N_4S_2] + 2RI \xrightarrow{-2MI} \underset{(R = Me,\ allyl)}{\text{Ph}_4\text{P}_2\text{N}_4\text{S}_2\text{R}_2} \qquad (1)$$
(M = Li, Na)

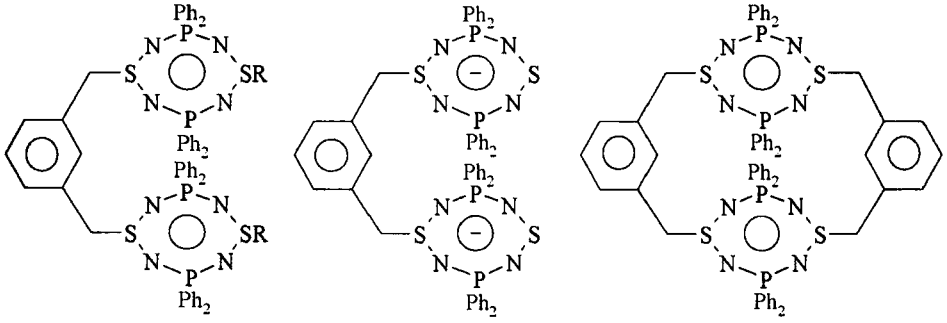
$$\tfrac{1}{2}[Li(Ph_4P_2N_4S_2R')]_2 + RI \xrightarrow{-LiI} \underset{(R = Me,\ allyl;\ R' = {}^tBu,\ Me,\ Ph)}{\text{Ph}_4\text{P}_2\text{N}_4\text{S}_2\text{RR}'} \qquad (2)$$

Very recently we began using these versatile metal reagents to construct novel macromolecules composed of $P_2N_4S_2$ rings and meta-xylyl building blocks. These supramolecular systems were prepared with the ultimate aim of employing them as host

molecules for metal cations from both sides of the periodic table.

SYNTHESIS AND CHARACTERIZATION OF 1, 2 AND 3

The neutral molecules **1a** and **1b** were first prepared by the reaction of meta-α,α′-dibromoxylene with $[Li(Ph_4P_2N_4S_2R)]_2$ (R = Me, ^{t}Bu). The dipotassium salt of the dianionic compound **2** was synthesized by mixing [meta-$(KCH_2)_2C_6H_4$] with two equiv. of 1,5-$[Ph_4P_2N_4S_2]$ in THF at -78°C. Compound **2** is readily converted to **1a** by treatment with 2 equiv. of iodomethane. The macrocycle **3** is most conveniently prepared by the reaction of $K_2[Ph_4P_2N_4S_2]$ with an equimolar quantity of meta-α,α′-dibromoxylene. This compound is also accessible via the treatment of the dipotassium salt of **2** with one equiv. of meta-α,α′-dibromoxylene.



1a: R = Me
1b: R = ^{t}Bu

2

3

Compounds **1a**, **1b** and **3** were characterized by elemental analysis, FAB mass spectroscopy and $^1H/^{13}C/^{31}P$ NMR spectroscopy. The identity of **2** was established on the basis of ^{31}P NMR spectroscopy and its derivative chemistry.

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