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研究论文

6-烷氧基乙氧/硫基-6-氧/硫-双苯并[1,3,2]-二氧磷杂八环的合成与波谱特征

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摘要 通过双(3,5-二氯-2-羟基-苯基)甲烷(**1**)分别与$POCl_3$或$PSCl_3$在三乙胺存在下关环, 再与单烷氧基乙醇钠作用, 得到了新化合物**5**或**6**; 以**1**与P_4S_{10}及三乙胺关环的产物**4**与烷氧基溴乙烷反应得到了**7**. 测试了**5**, **6**和**7**的^1H, ^{13}C和^{31}P NMR及MS数据, 并讨论了其特征. 观察到了磷与C^1H ($C^{11}H$)或C^3H (C^9H)与P的$^5J_{PH}$偶合常数(约为0.9 Hz). 在**1**与$POCl_3$关环产物与单甲氧基乙醇反应的产物中, 分离到了一个副产物**8**并确定了它的化学结构. **1**与$POCl_3$按3∶2的物质的量比在三乙胺存在下反应, 得到收率75%的**8**.

关键词 6-烷氧基乙氧/硫基-6-氧/硫-双苯并[1,3,2]-二氧磷杂八环 合成 核磁共振 质谱

分类号

Synthesis and Spectral Characteristics of 6-*O*/*S*-Alkoxyethyl- 12*H*-[1,3,2]dioxaphosphocin-6-oxides/sulfides

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Abstract From bis(3,5-dichloro-2-hydrxy-phenyl)methane (**1**), the new [1,3,2] dioxaphosphocins **5**, **6** and **7** were synthesized. **1** reacted with $POCl_3$ or $PSCl_3$ to give cyclic compound **2** or **3**, which reacted with monoalkyl-1,2-ethandiol to offer **5** or **6**. **7** was prepared by the reaction of **1** with P_4S_{10} in the presence of triethylamine, and then reacted with 2-alkoxyl ethyl bromide. The ^1H, ^{13}C and ^{31}P NMR and MS spec-tra of the new compounds were evaluated and discussed. The coupling $^5J_{PH}$ (about 0.9 Hz) of P and C^1H or C^3H was observed in the first time. A solid compound **8** was separated from the crude product **5** and its structure was determined. When **1** and $POCl_3$ reacted at the molar ratio of 3∶2, **8** was obtained in yield of 70%.

Key words [1 3 2]dioxaphosphocin-6-oxides/sulfide synthesis NMR MS

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