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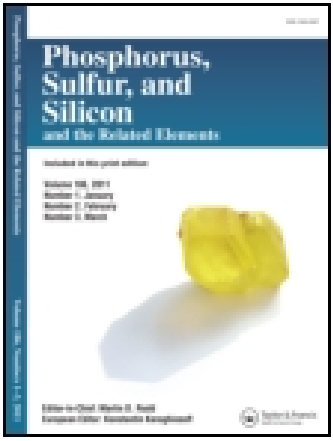

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2,2,2-Tribromonaphtho[2,3-d]-1,3,2-Dioxaphosphole: Obtaining and Reaction with Phenylacetylene

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2,2,2-Tribromonaphtho[2,3-*d*]-1,3,2-Dioxaphosphole: Obtaining and Reaction with Phenylacetylene

A. V. Bogdanov, V. F. Mironov, B. I. Buzykin, A. B. Dobrynin, D. B. Krivolapov, and A. I. Konovalov
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The reaction of 2,3-dihydroxynaphthalene with phosphorus tribromide followed by bromide treatment has been shown to lead to quantitative formation of the new 2,2,2 tribromonaphtho [2,3-d]-1,3,2-dioxaphosphole. Its interaction with phenylacetylene proceeds by several pathways and leads to unexpected formation of 2-phenyl-9-(2-dihydroxyphosphoryl-1-phenylethen-1-yl)naphtho[1,2-d]furane. The structure of the last compound was established by NMR and single crystal X-ray diffraction.

Keywords 2,3-dihydroxynaphthalenes; phosphorus tribromides; phosphole; phenylacehylene; naphthofurane; crystal structure

The interaction of 2,2,2-trichlorobenzo[*d*]-1,3,2-dioxaphosphole with arylacetylenes is a versatile approach to the synthesis of benzo[*e*]-1,2-oxaphosphorinine derivatives–P-analogues of coumarines.[1] Here, we report the obtaining of 2,2,2-tribromonaphtho[2,3-*d*]-1,3,2-dioxaphosphole **1** from 1,2-naphthalenediol, PBr_3 and bromine and its reaction with phenylacetylene. The reaction of phosphole **1** with phenylacetylene proceeds in several directions (10–15°C) (Scheme 1).

The first and second pathways include the formation of heterocycles **2** (2–3%) and **3** (20–23%) after the reaction mixture hydrolysis. The third pathway is the disproportionation of the phosphole **1,** with the following bromination resulting in the formation of phosphoranes **4**, **5**. The phosphates **6**, **7** were obtained as a result of the hydrolysis. We succeeded in isolation of compound **3**, **7**. Phosphate **6** was obtained by the independent synthesis also. The structure of 2-phenyl-9-(2-dihydroxy phosphoryl-1-phenylethen-1-yl)naphtho[1,2-*d*]furane **3** was confirmed by the X-ray single crystal diffraction (Figure 1).

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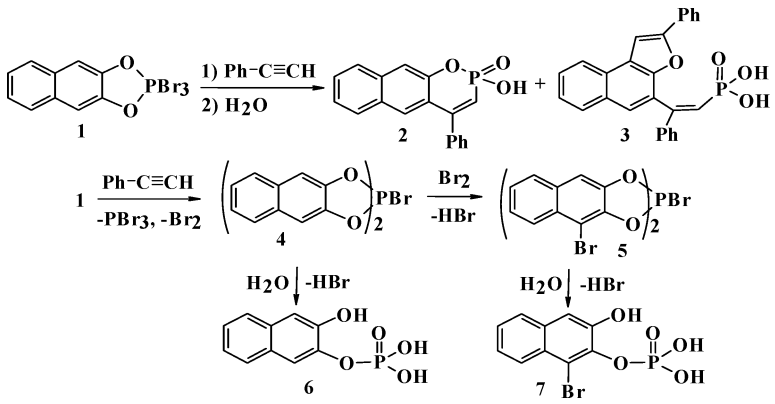

SCHEME 1

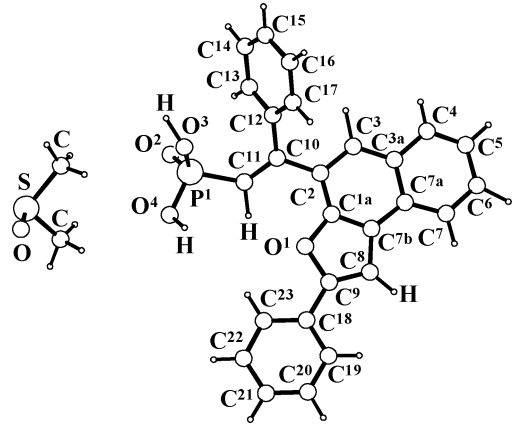

FIGURE 1 The geometry of molecule 3 (solvate with DÃS) in crystal.

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