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Synthesis and Structure of a Kinetically Stabilized 1,3,6-Triphosphafulvene

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SYNTHESIS AND STRUCTURE OF A KINETICALLY STABILIZED 1,3,6-TRIPHOSPHAFULVENE

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Kinetically stabilized phosphanylidene carbenoid 1 is one of the precursors for novel low-coordinated phosphorus compounds. We report the novel trimerization of phosphanylidene carbenoid 1 affording 1,3,6-triphosphafulvene 3. Dibromophosphaethene 2 was allowed to react with 2 molar equivalent of t-butyllithium at -78° C to generate the phosphanylidene carbenoid 1, and the reaction mixture was warmed to 25° C. After purification, 1,3,6-triphosphafulvene 3 was obtained as a deep red solid. The NMR and MS spectrum of 3 supported the 1,3,6-triphosphafulvene structure. Furthermore, triphosphafulvene 3 was treated with an excess amount of W(CO)₅(thf) to afford the pentacarbonyltungsten(0) complex $3\mathbf{w}$ as a deep red solid. Complex $3\mathbf{w}$ was recrystallized from toluene at 0° C to afford a suitable crystal for x-ray analysis. The x-ray crystallography of $3\mathbf{w}$ was confirmed the 1,3,6-triphosphafulvene structure with the coordination of tungsten at the P3 position. 1,*

Mes* Br
$$t$$
-BuLi t

SCHEME 1

*In ref. [1], the phosphorus chemical shifts at the P6 position for **3** and **3w** should read $\delta_P = 399.0$ and $\delta_P = 397.1$, respectively.

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