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## Preparation and Reactions of Bulky Ethynylphosphines

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# PREPARATION AND REACTIONS OF BULKY ETHYNYLPHOSPHINES

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Ethynylphosphine system is expected to show the interaction between the lonepair of phosphorus and  $\pi$ -orbital of C=C group, and some spectroscopic and theoretical investigations have been performed so far. Although some ethynylphosphines have been derived, their instability limits the synthetic and structural study. We report the preparation and some reactions of ethynylphosphines possessing the bulky 2,4,6-tri-t-butylphenyl (Mes\*) group. Ethynylbenzene 1 was lithiated and allowed to react with chlorobis(dimethylamino)phosphine to afford 2, and 2 was chlorinated with HCl to afford 3 as a thermally stable solid. Dichlorophosphine 3 was allowed to react with LiAlH<sub>4</sub> to give the corresponding primary phosphine 4 as a thermally-stable compound and the product was characterized by spectroscopic study. The Tungsten complex 5 derived from 4 reacted with Mes\*PCl<sub>2</sub> to afford a tungsten complex of diphosphene 6.

Mes\*C=CH 
$$\xrightarrow{1) \text{ $r$-BuLi}}$$
 Mes\*C=C-R  $\xrightarrow{X}$  LiAlH<sub>4</sub> Mes\*C=C-R  $\xrightarrow{H}$  HCI  $\xrightarrow{2) \text{CIP}(\text{NMe}_2)_2}$  HCI  $\xrightarrow{2} (X = \text{NMe}_2)$   $\xrightarrow{4}$  Mes\*C=C-R  $\xrightarrow{H}$  Mes\*

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