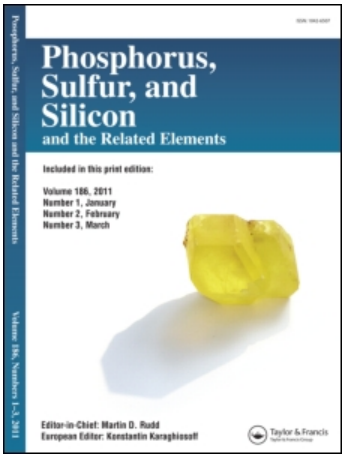

Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Deprotection Techniques for Phosphine-Borane Complexes: Methods and Extraction Coefficients

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Online publication date: 27 October 2010

To cite this Article Bradley, D. , Williams, G. , Lombard, Henriëtte , Van Niekerk, Marié and Coetzee, Paul P.(2002) 'Deprotection Techniques for Phosphine-Borane Complexes: Methods and Extraction Coefficients', Phosphorus, Sulfur, and Silicon and the Related Elements, 177: 8, 2115 — 2116
To link to this Article: DOI: 10.1080/10426500213448
URL: http://dx.doi.org/10.1080/10426500213448

Phosphorus, Sulfur and Silicon, 2002, Vol. 177:2115–2116

1042-6507/02 $12.00 + .00
DOI: 10.1080/10426500290094684

DEPROTECTION TECHNIQUES FOR PHOSPHINE-BORANE COMPLEXES: METHODS AND EXTRACTION COEFFICIENTS

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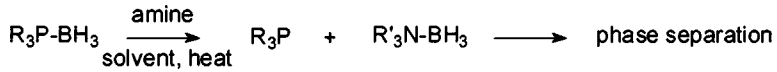
(Received July 29, 2001; accepted December 25, 2001)

Borane is often used as an efficient protecting group in the synthesis of phosphines.[1] Deprotection is facilitated by using nucleophilic amines[2] or acidic conditions.[3] Before a protected phosphine can be used as a ligand for transition metal catalysts, the P—B bond is cleaved. A systematic study by us using various deprotecting agents showed that the rate and ease of deprotection were determined by the basicity of the phosphine in conjunction with the deprotecting agent employed. It was also shown that the resulting amine-borane complexes could be extracted into water, such that the use of Pd(II) salts is permitted in certain cases (failure to extract the borane complex into water results in the precipitation of Pd black) with which to generate catalytically active species.



R = alkyl, aryl; R' = alkyl, functionalised alkyl, H

SCHEME 1

Phase separation was effected using a variety of organic solvents (e.g., DCM, EtOAc, EtOAc-hexanes), and extraction coefficients were calculated on the basis of [B] as determined by ICP analyses. EtOAc-hexanes proved to be the solvent of choice, and a source of CN^- the deprotecting agent of choice to effect the best extraction, although this

Contributions of SASOL (SA) and the Rand Afrikaans University toward this project are gratefully acknowledged.

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reagent required longer reaction times to achieve complete deprotection of the phosphine complex.

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