

1,3-Distanna-2-phospha-[3]ferrocenophanes - Synthesis, Reactivity and NMR Spectroscopic Properties

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Ferrocene, Tin, Phosphorus, NMR Data, Coupling Signs

1,1,3,3-Tetramethyl-2-organo(R)-1,3-distanna-2-phospha-[3]ferrocenophanes [R = Me (**2a**), ^tBu (**2b**), C₆H₁₁ (**2c**), Ph (**2d**)] and one arsa-analogue **2d(As)** were obtained from the reaction of 1,1'-bis(chlorodimethylstannyl)ferrocene **1** with either bis(trimethylsilyl)methylphosphane or the dilithio derivatives, Li₂PR and Li₂AsPh, respectively. All compounds **2** react with chalcogens (oxygen, sulfur, selenium) by cleavage of the Sn-P bonds and formation of the known 1,3-distanna-2-chalcogena-[3]ferrocenophanes. In contrast, **2d** traps pentacarbonylmetal fragments [M(CO)₅] to give the stable phosphane complexes [M = Cr (**4d**), Mo (**5d**), W (**6d**)]. The 1,1'-bis(diorganophosphanostannyl)ferrocenes [R = ^tBu (**3b**), Ph (**3d**)] were prepared for comparison of NMR data. The ferrocenophanes **2** are fluxional with respect to fast movement of the cyclopentadienyl rings which induces inversion at the pyramidal phosphorus atom. This dynamic process is slow in the cases of **2d(As)** and of the pentacarbonyl complexes **4d** - **6d**. All new compounds were characterised by ¹H, ¹³C, ³¹P and ¹¹⁹Sn NMR spectroscopy. Various 2D heteronuclear shift correlations (*e.g.* ³¹P/¹H and ¹¹⁹Sn/¹H) were carried out for the compounds **2** and also for non-cyclic derivatives such as bis(trimethylstannyl)phenylphosphane and -arsane in order to determine absolute signs of coupling constants [*e.g.* ¹J(¹¹⁹Sn, ³¹P) > 0 and ²J(¹¹⁹Sn, ¹¹⁷Sn) < 0]. The NMR data suggest that the molecular frameworks of the ferrocenophanes **2** are not particularly strained.

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