Electronic Supplementary Material (ESI) for Dalton Transactions.
This journal is © The Royal Society of Chemistry 2015

Supporting Information:

Reaction of an Allylstannylene with Adamantyl Phosphaalkyne

Kilian M. Krebs, Jellian Jamin, Lars Wesemann*

Institute of Inorganic Chemistry, University of Tübingen, Auf der Morgenstelle 18, D-72076 Tübingen

Table of contents

1 Experimental Details

1.1 Methods

1.1.1 General procedures

All manipulations were carried out under an argon atmosphere using standard Schlenk techniques and gloveboxes. *n*-Hexane was dried using a M.Braun – Solvent Purification System (SPS). All other solvents were destilled from sodium or sodium/benzophenone. All solvents were subsequently degassed by 3 × freeze/pump/thaw. Tin(II)chloride was purified by sublimation. 2,6-$Trip_2C_6H_3SnCl$ was prepared according to literature procedures[1] and converted into 2,6-$Trip_2C_6H_3SnC_3H_5$ as reported.[2] Further chemicals were purchased commercially and used as received.

Elemental analyses were performed at the Institute of Inorganic Chemistry, University of Tübingen using a Vario MICRO EL analyzer.

1.1.2 NMR Spectroscopy

NMR spectra were recorded on a Bruker DRX-250 NMR spectrometer (^1H, 250.13 MHz; ^{13}C, 62.90 MHz ^{119}Sn, 93.28 MHz) equipped with a 5 mm ATM probe head, a Bruker AvanceII+400 NMR spectrometer (^1H, 400.13 MHz;

^{13}C, 100.61 MHz) equipped with a 5 mm QNP (quad nucleus probe) head and a Bruker AvanceII+500 NMR-spectrometer (^{1}H, 500.13 MHz; ^{13}C, 125.76 MHz; ^{119}Sn, 186.50 MHz) equipped with a 5 mm ATM or a 5 mm TBO probe head and a setup for variable temperature. The chemical shifts are reported in δ values in ppm relative to external $SiMe_4$ (^{1}H, ^{13}C) or $SnMe_4$ (^{119}Sn) using the chemical shift of the solvent ^{2}H resonance frequency and Ξ = 25.145020 % for ^{13}C and 37.290632 % for ^{119}Sn.[3] The multiplicity of the signals is abbreviated as s = singlet, d = doublet, t = triplet, quint = quintet, sept = septet and m = multiplet or unresolved. The proton and carbon signals were assigned by detailed analysis of ^{1}H, $^{13}C\{^{1}H\}$, ^{1}H-^{1}H COSY, ^{1}H-^{13}C HSQC, ^{1}H-^{13}C HMBC and $^{13}C\{^{1}H\}$ DEPT 135 spectra. For variable temperature measurements the sample temperature was stabilized with a Bruker BVT 3200 temperature controller. The temperatures given are uncorrected.

2 Crystallographic Details

2.1 General crystallographic considerations

2.1.1 *Hardware and refinement details*

X-ray data were collected with a Bruker Smart APEX II diffractometer with graphite-monochromated Mo Kα radiation. The programs used were Bruker's APEX2 v2011.8-0, including SADABS for absorption correction and SAINT for data reduction, as well as the WinGX suite of programs version 1.70.01 or the GUI ShelXle, including SHELXS for structure solution and SHELXL for structure refinement.[4]

2.1.2 *General remarks*

Compound **1** crystalized in the triclinic space group P-1. One allyl moiety shows a disorder of 40/60 and was treated with ISOR. Two severely disordered hexane molecules and some disordered isopropyl groups were treated with SIMU, DELU, ISOR, EADP, DFIX and DANG as well as the PART instruction. Disorders and flexible parts of the molecule resulted in enlarged ellipsoids and the solvent hexane was refined to approximately 1.7 eq in a void filled with either two or one hexane molecules.

2.2 Crystal Structure Refinement Tables

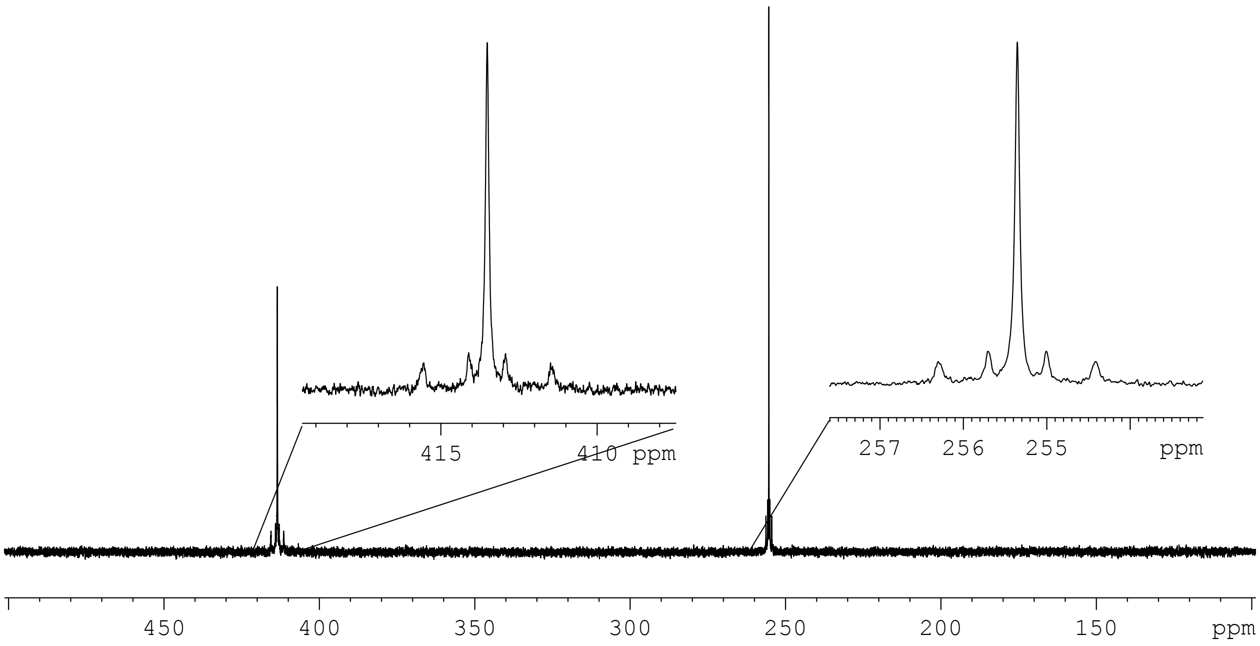
Table S1. Crystal structure refinement table of **1** × ca 1.7 eq hexane.

	1 × ca 1.7 eq hexane
Empirical formula	$C_{110.15}H_{161.65}P_2Sn_2$
M_r / g mol^{-1}	1785.15
λ / Å	0.71073
T / K	100(2)
Crystal system	triclinic
Space group	P -1
Z	2
a / Å	14.9605(3)
b / Å	17.8145(4)
c / Å	19.5352(4)
α / °	79.9460(10)
β / °	82.2560(10)
γ / °	83.4230(10)
V / Å^3	5057.55(19)

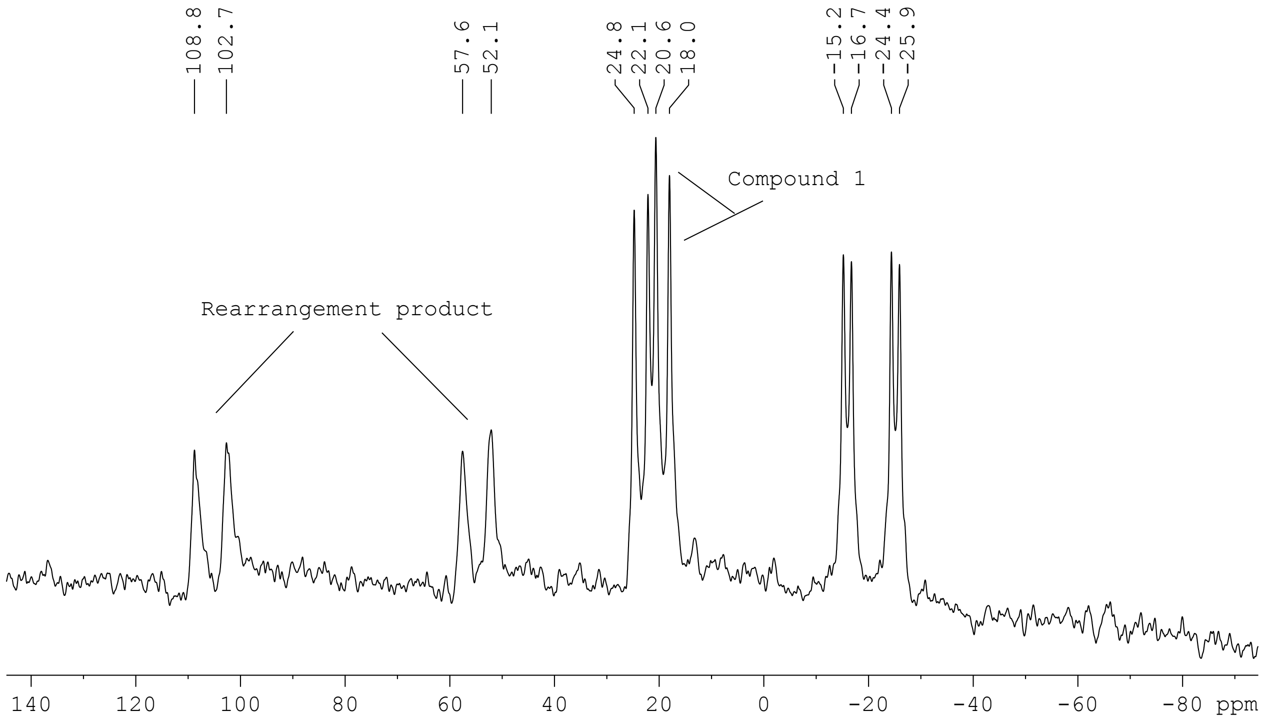
D_c / g cm^{-3}	1.172
μ / mm^{-1}	0.570
F(000)	1905
Crystal size / mm	0.303 × 0.179 × 0.046
θ range / °	1.831 – 25.681
Limiting indices	$-16 \leq h \leq 18$
	$-19 \leq k \leq 21$
	$-23 \leq l \leq 22$
Reflects. collect.	70071
Indepdnt Reflects	18828
R_{int}	0.0504
Completeness	98.1
Absorp. Corr.	numerical
Trans.(max., min.)	0.82, 1.00
Parameters/restraints	1135/384
R_1, ωR_2 [$I > 2\sigma(I)$]	0.0535, 0.1275
R_1, ωR_2 (all data)	0.0930, 0.1455
GooF on F^2	1.029
$\Delta\rho_{max,min}$ / e·Å^{-3}	1.458, –0.892
CCDC	1427413

3 Additional NMR Data

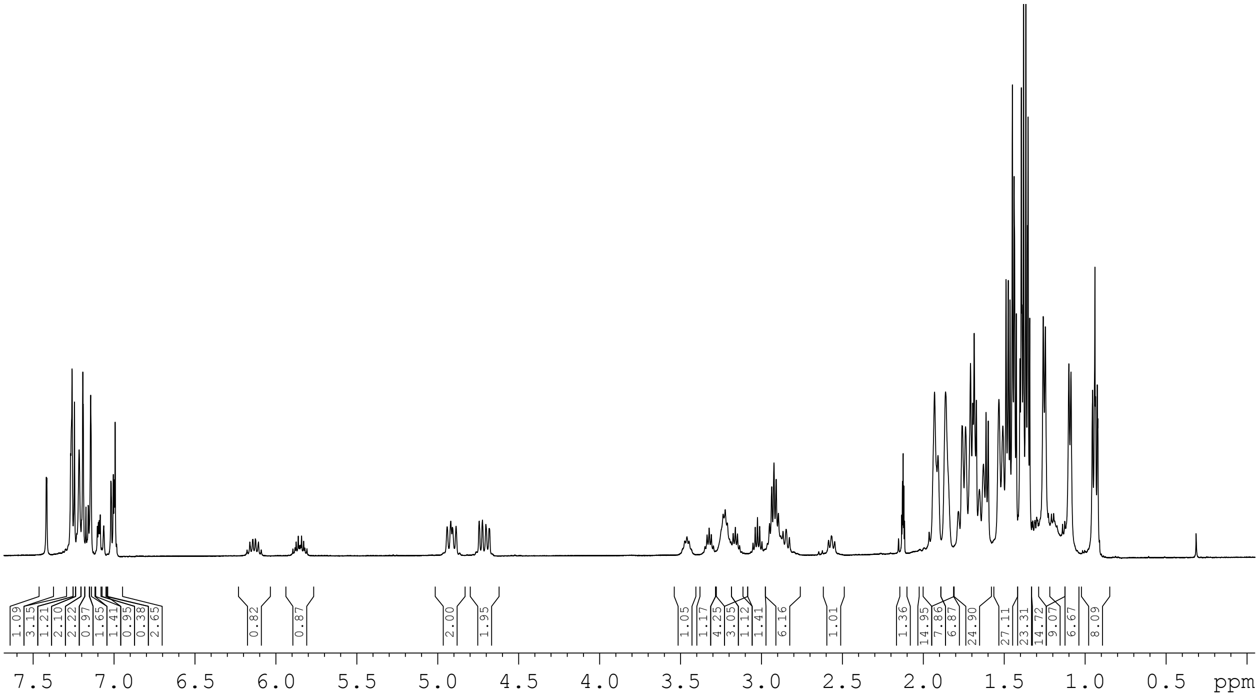
3.1 $^{31}P\{^{1}H\}$ NMR spectrum for compound **1** (Tol-d8, 0°C)



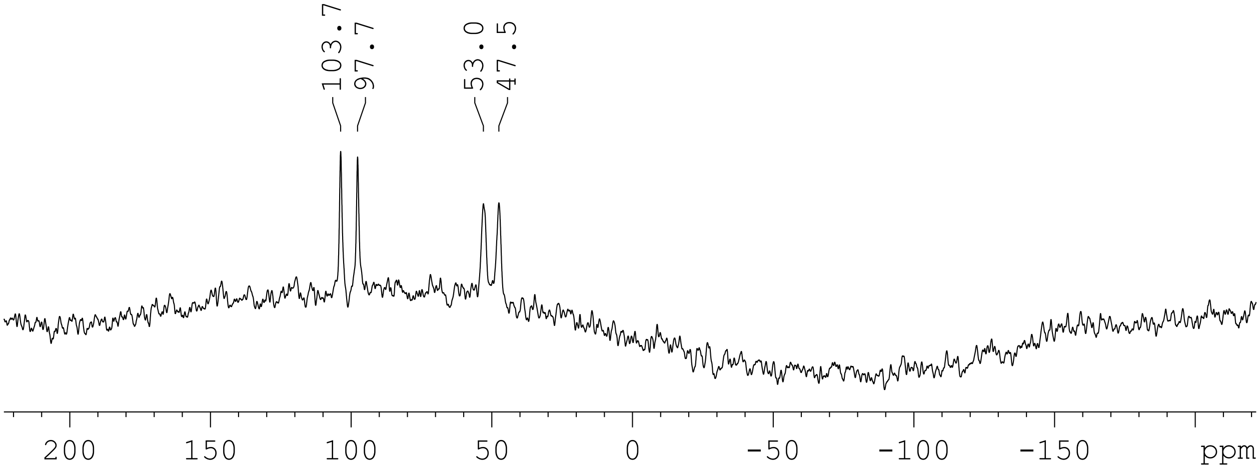
3.2 $^{119}Sn\{^{1}H\}$ NMR spectrum for compound **1**; partially rearranged over night (Tol-d8)

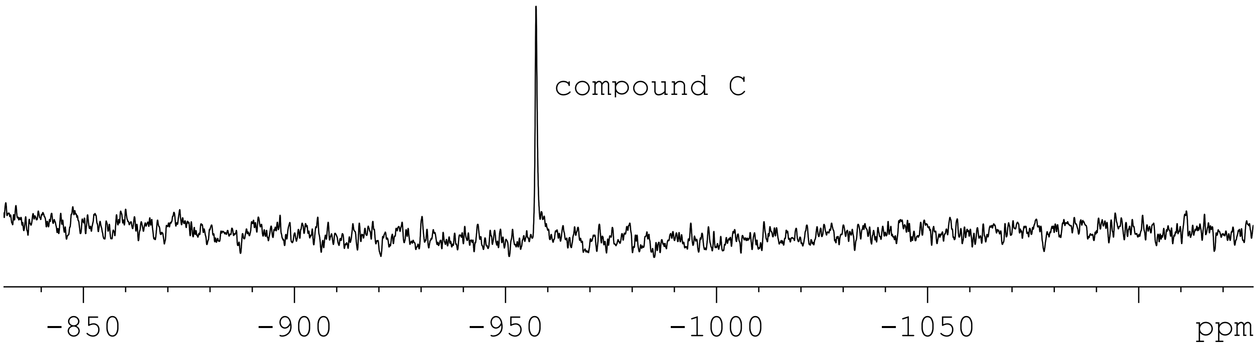


3.3 ^{1}H NMR spectrum for compound **1** (Tol-d8, 0°C)

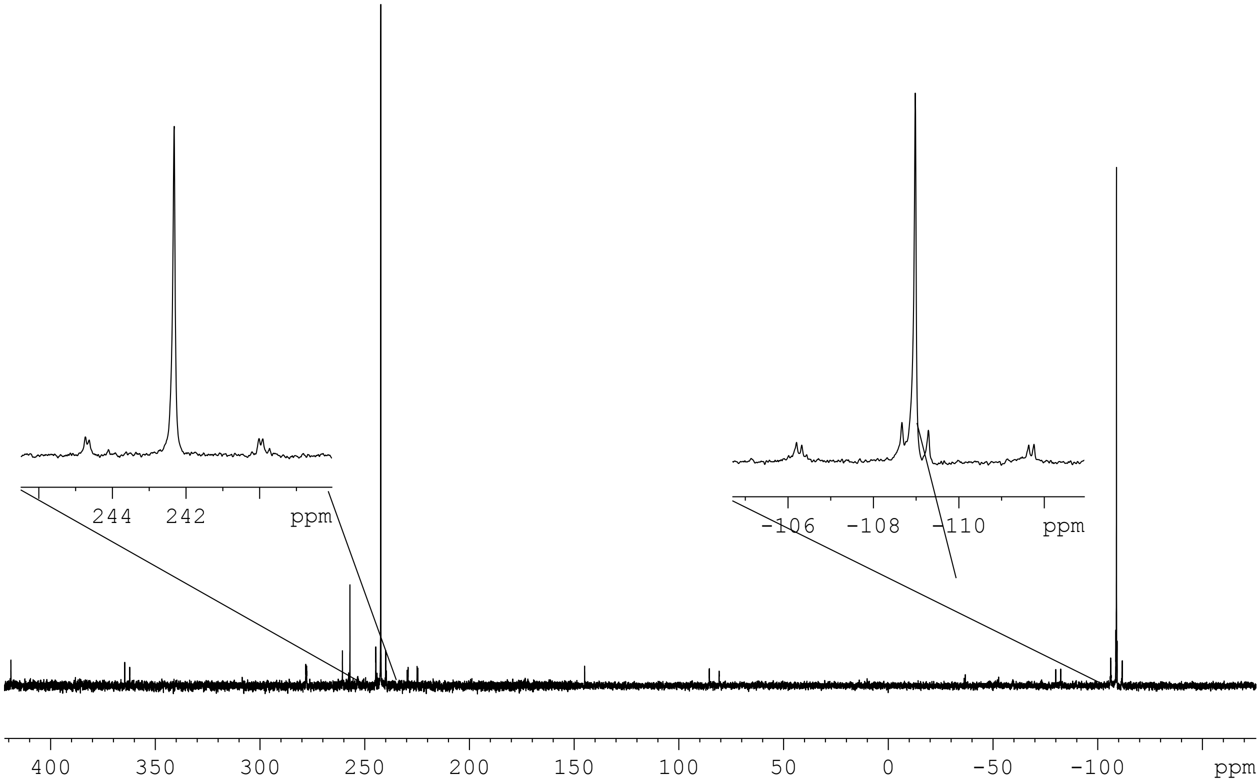


3.4 ^{119}Sn{^{1}H} NMR after the rearrangement of **1** (C_6D_6)





3.5 $^{31}P\{^{1}H\}$ NMR spectrum after the rearrangement compound **1** (Tol-d8)



4 References

[1] a) B. Schiemenz, P. P. Power, *Organometallics* **1996**, *15*, 958-964; b) R. S. Simons, S. T. Haubrich, B. V. Mork, M. Niemeyer, P. P. Power, *Main Group Chem.* **1998**, *2*, 275-283; c) B. E. Eichler, L. Pu, M. Stender, P. P. Power, *Polyhedron* **2001**, *20*, 551-556.

[2] K. M. Krebs, J. Wiederkehr, J. Schneider, H. Schubert, K. Eichele, L. Wesemann, *Angew. Chem., Int. Ed.* **2015**, *54*, 5502-5506.

[3] K. Harris Robin, D. Becker Edwin, M. Cabral de Menezes Sonia, R. Goodfellow, P. Granger, in *Pure Appl. Chem., Vol. 73*, **2001**, p. 1795.

[4] a) SAINT, APEX2; Bruker AXS Inc.: Madison, WI., **2007**; b) Sheldrick, G. M., *SADABS*, University of Göttingen, Germany, **1996**; c) L. J. Farrugia, *J. Appl. Crystallogr.* **1999**, *32*, 837-838; d) C. B. Hübschle, G. M. Sheldrick, B. Dittrich, *J. Appl. Crystallogr.* **2011**, *44*, 1281-1284; e) G. M. Sheldrick, *Acta Crystallographica Section A* **2008**, *64*, 112-122.