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Key indicators

Single-crystal synchrotron study

$T = 150$ K

Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å

R factor = 0.081

wR factor = 0.187

Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

3-(Trimethylsilylethynyl)carbazole

The title compound, $[\text{Si}(\text{CH}_3)_3(\text{C}_{14}\text{H}_8\text{N})]$ is a TMS-protected mono-alkyne. It is used as a model species for platinum σ -acetylide compounds of which it is a precursor. Such compounds are of interest due to the extended π -conjugation through the hetero-aromatic linker unit in the backbone.

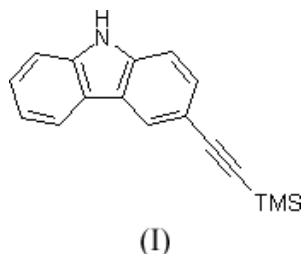
Received 4 August 2003

Accepted 12 August 2003

Online 23 August 2003

Comment

Here we report the structural characterization of the title compound, (I), which is a TMS-protected mono-alkyne and a precursor of the mononuclear platinum(II) di-yne species, $\text{trans}[\text{Pt}(\text{P}^i\text{Bu}_3)_2(-\text{C}\equiv\text{CR})_2]$ ($R = \text{carbazol-3-yl}$). Such platinum-containing species form the building blocks for rigid-rod organometallic poly-yne of general formula $\text{trans}[\text{Pt}(\text{P}^i\text{Bu}_3)_2-\text{C}\equiv\text{C}-R-\text{C}\equiv\text{C}-]_\infty$ ($R = \text{aromatic or heteroaromatic linker unit}$). Platinum poly-yne are of immense current interest owing to π -electron conjugation along the backbone, novel donor-acceptor interactions between the metal centres and the conjugated ligands, and the unique photophysical properties arising from the large spin-orbit coupling (Wittmann *et al.*, 1994; Beljonne *et al.*, 1996; Younus *et al.*, 1998; Chawdhury *et al.*, 1998, 1999; Wilson *et al.*, 2000; Wilson, Chawdhury *et al.*, 2001; Wilson, Dhoot *et al.*, 2002; Khan, Al-Mandhary, Al-Suti, Feeder *et al.*, 2002; Khan, Al-Mandhary, Al-Suti, Hisahm *et al.*, 2002; Khan, Al-Mandhary, Al-Suti, Ahrens *et al.*, 2003; Khan, Al-Mandhary, Al-Suti, Corcoran *et al.*, 2003; Khan, Al-Suti *et al.*, 2003). Precursors to these species, such as the title compound, are studied as models of the molecular and electronic properties and structure-property relationships in the metal poly ynes.



In the crystal structure of (I) there are three independent molecules per asymmetric unit, as shown in Fig. 1. Selected geometric parameters are given in Table 1.

Experimental

3-Trimethylsilylethynylcarbazole was synthesized by the following procedure. To a stirred solution of 3-bromocarbazole (2.0 g,

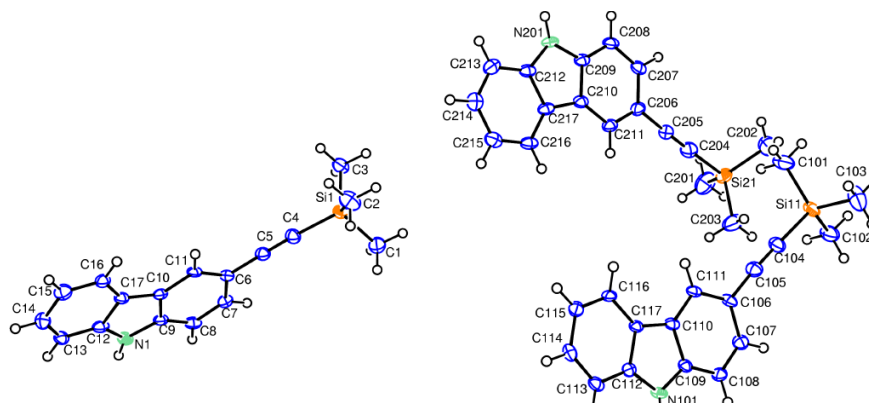


Figure 1

The molecular structure of 3-trimethylsilyl-1H-1,2,3,4,5-pentahydrocarbazole, showing the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

6.13 mmol) in $i\text{Pr}_2\text{NH}/\text{THF}$ (70 ml, 1:1 v/v) under nitrogen was added a catalyst mixture of CuI (15 mg), $\text{Pd}(\text{OAc})_2$ (15 mg) and PPh_3 (50 mg). The solution was stirred for 20 min. at 323 K and then trimethylsilyl-1H-1,2,3,4,5-pentahydrocarbazole (1.5 g, 15.3 mmol) was added and the mixture stirred for another 20 min. The reaction temperature was then raised to 348 K and left with stirring for 20 h. The completion of the reaction was determined by silica TLC and IR spectroscopy. The solution was allowed to cool down to room temperature. The crude product was filtered to eliminate the ammonium salt and the solvent was removed under vacuum. The residue was subjected to silica column chromatography using hexane/ CH_2Cl_2 (1:1) as eluant to afford the target compound as a colorless solid in 77% yield.

Crystal data

$[\text{Si}(\text{CH}_3)_3(\text{C}_{14}\text{H}_8\text{N})]$	Synchrotron radiation
$M_r = 263.41$	$\lambda = 0.688 \text{ \AA}$
Monoclinic, $P2_1/c$	Cell parameters from 13603 reflections
$a = 33.402 (3) \text{ \AA}$	$\theta = 2.3\text{--}29.1^\circ$
$b = 5.8135 (4) \text{ \AA}$	$\mu = 0.15 \text{ mm}^{-1}$
$c = 22.8511 (17) \text{ \AA}$	$T = 150 (2) \text{ K}$
$\beta = 98.394 (2)^\circ$	Plate, white
$V = 4389.7 (6) \text{ \AA}^3$	$0.20 \times 0.20 \times 0.04 \text{ mm}$
$Z = 12$	
$D_x = 1.196 \text{ Mg m}^{-3}$	

Data collection

Bruker AXS SMART 1K CCD diffractometer	7939 independent reflections
ω rotation with narrow-frame scans	5999 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.047$
$T_{\text{min}} = 0.971$, $T_{\text{max}} = 0.994$	$\theta_{\text{max}} = 24.5^\circ$
21435 measured reflections	$h = -39 \rightarrow 40$
	$k = -7 \rightarrow 6$
	$l = -20 \rightarrow 27$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 9.4726P]$
$R[F^2 > 2\sigma(F^2)] = 0.081$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.187$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.14$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
7939 reflections	$\Delta\rho_{\text{min}} = -0.42 \text{ e \AA}^{-3}$
514 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$) for (section).

C1—Si1	1.862 (4)	C109—N101	1.381 (5)
C2—Si1	1.864 (5)	C109—C110	1.423 (5)
C3—Si1	1.864 (4)	C110—C117	1.445 (5)
C4—C5	1.207 (5)	C112—N101	1.391 (5)
C4—Si1	1.827 (4)	C112—C117	1.419 (5)
C5—C6	1.455 (5)	C201—Si21	1.869 (6)
C9—N1	1.377 (5)	C202—Si21	1.856 (5)
C9—C10	1.418 (5)	C203—Si21	1.847 (5)
C10—C17	1.445 (5)	C204—C205	1.205 (6)
C12—N1	1.385 (5)	C204—Si21	1.833 (4)
C12—C17	1.417 (5)	C205—C206	1.448 (5)
C101—Si11	1.860 (5)	C209—N201	1.385 (5)
C102—Si11	1.855 (4)	C209—C210	1.426 (5)
C103—Si11	1.862 (5)	C210—C217	1.450 (5)
C104—C105	1.206 (6)	C212—N201	1.379 (5)
C104—Si11	1.828 (4)	C212—C217	1.422 (5)
C105—C106	1.438 (5)		
C5—C4—Si1	176.8 (4)	C1—Si1—C3	109.2 (2)
C4—C5—C6	178.9 (4)	C4—Si1—C2	109.0 (2)
N1—C9—C10	108.5 (3)	C1—Si1—C2	111.3 (2)
C9—C10—C17	106.8 (3)	C3—Si1—C2	109.1 (2)
N1—C12—C17	108.5 (3)	C104—Si11—C102	107.44 (19)
C12—C17—C10	106.5 (3)	C104—Si11—C101	108.6 (2)
C105—C104—Si11	178.1 (4)	C102—Si11—C101	111.8 (2)
C104—C105—C106	176.0 (5)	C104—Si11—C103	110.5 (2)
N101—C109—C110	108.8 (3)	C102—Si11—C103	108.0 (2)
C109—C110—C117	106.3 (3)	C101—Si11—C103	110.6 (3)
N101—C112—C117	108.0 (3)	C204—Si21—C203	108.0 (2)
C112—C117—C110	107.3 (3)	C204—Si21—C202	107.7 (2)
C205—C204—Si21	178.7 (4)	C203—Si21—C202	111.8 (3)
C204—C205—C206	176.4 (5)	C204—Si21—C201	110.7 (2)
N201—C209—C210	108.4 (3)	C203—Si21—C201	109.6 (3)
C209—C210—C217	106.6 (3)	C202—Si21—C201	109.2 (3)
N201—C212—C217	108.8 (3)	C9—N1—C12	109.7 (3)
C212—C217—C210	106.4 (3)	C109—N101—C112	109.6 (3)
C4—Si1—C1	107.1 (2)	C212—N201—C209	109.7 (3)
C4—Si1—C3	111.1 (2)		
C5—C6—C7—C8	178.4 (4)	C5—C4—Si1—C2	-123 (7)
N1—C9—C10—C17	-0.2 (4)	C105—C104—Si11—C102	7 (13)
N1—C12—C17—C10	-1.2 (4)	C105—C104—Si11—C101	128 (13)
C9—C10—C17—C12	0.9 (4)	C105—C104—Si11—C103	-110 (13)
C105—C106—C107—C108	176.9 (4)	C205—C204—Si21—C203	105 (21)
N101—C109—C110—C117	0.4 (4)	C205—C204—Si21—C202	-16 (21)
N101—C112—C117—C110	-1.1 (4)	C205—C204—Si21—C201	-135 (21)
C109—C110—C117—C112	0.4 (4)	C10—C9—N1—C12	-0.6 (4)
C205—C206—C207—C208	176.3 (4)	C17—C12—N1—C9	1.1 (4)
N201—C209—C210—C217	-0.2 (4)	C110—C109—N101—C112	-1.1 (4)
N201—C212—C217—C210	-1.7 (4)	C117—C112—N101—C109	1.3 (4)
C209—C210—C217—C212	1.1 (4)	C217—C212—N201—C209	1.6 (4)
C5—C4—Si1—C1	-2 (7)	C210—C209—N201—C212	-0.9 (4)
C5—C4—Si1—C3	117 (7)		

Aromatic and methyl H atoms were constrained as riding atoms, fixed to the parent atoms with distances of 0.95 Å and 0.98 Å, respectively. The isotropic displacement parameters were fixed to 120% of that of the parent atom for aromatic and 150% for methyl H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

We thank Sultan Qaboos University, Oman, the EPSRC, UK, and Cambridge Crystallographic Data Centre, UK for funding.

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