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5,5′-Bis(trimethylsilylethynyl)-2,2′-bipyridine

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Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.110
Data-to-parameter ratio = 15.3

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

The title compound, 5,5′-bis(trimethylsilylethynyl)-2,2′-bipyridine, $C_{20}H_{24}N_2Si_2$, is a trimethylsilyl-protected dialkyne. It is a precursor in the preparation of platinum di-yne complexes and platinum poly-yne polymers. Such organic compounds are of interest because of the extended π-conjugation that occurs through the hetero-aromatic linker unit in the molecular backbone. Within the molecule, the silyl-alkyne groups are essentially linear and the bipyridine unit is approximately planar with a dihedral angle of 5.3 (1)° between the planes.

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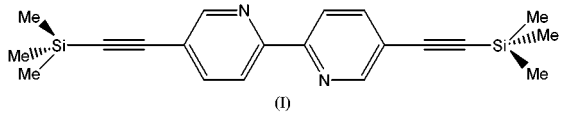
Comment

In this paper, we report the structural characterization of the title compound, (I), which is a trimethylsilyl-protected di-alkyne and a precursor of the dinuclear platinum(II) di-yne species, *trans*-[(Ph)(PEt$_3$)$_2$Pt—C≡C—*R*—C≡C—Pt(PEt$_3$)$_2$-(Ph)] (*R* = 2,2′-bipyridne-5,5′-diyl). Such organoplatinum species forms the building blocks for rigid-rod platinum poly-ynes with the general formula *trans*-[(^{n}Bu$_3$P)$_2$Pt—C≡C—*R*—C≡C—]$_\infty$ (*R* = aromatic or heteroaromatic spacer group). Platinum poly-ynes are of immense current interest because of the π-electron conjugation that occurs along the polymer backbone, novel donor-acceptor interaction between the metal centres and the conjugated ligands, and the unique photophysical properties arising from the large spin–orbit coupling associated with the presence of the heavy metal atoms (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.*, 2001; Khan, Al-Mandhary, Al-Suti, Hisahm *et al.*, 2002; Khan, Al-Mandhary, Al-Feeder *et al.*, 2002; Khan, Al-Mandhary, Al-Suti, Corcoran *et al.*, 2003; Khan, Al-Mandhary, Al-Suti, Raithby, Ahrens, Male *et al.*, 2003; Khan, Al-Mandhary, Al-Suti, Raithby, Ahrens, Mahon *et al.*, 2003).



(I)

Precursors to organometallic polymers, such as the title compound, (I), are studied as models of the molecular and electronic properties and structure-property relationships that occur in metal poly-ynes. The central ring system of (I) is approximately planar, with a dihedral angle of 5.3 (1)° between the planes of the two pyridine rings. The Si—C≡C and the C≡C—C(ring) units are essentially linear. There are no short intermolecular contacts within the crystal structure.

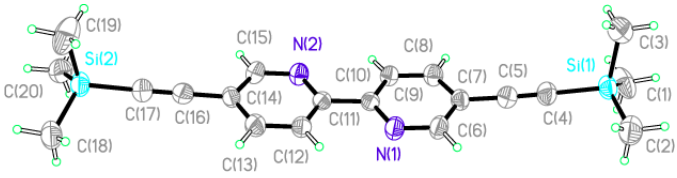


Figure 1
View of (I) (50% probability displacement ellipsoids). The disorder in the methyl groups has been omitted for clarity.

Experimental

5,5′-Bis(trimethylsilylethynyl)-2,2′-bipyridine was synthesized according to the procedure of Khan, Al-Mandhary, Al-Suti, Hisahm *et al.* (2002). To a solution of 5,5′-dibromo-2,2′-bipyridine (2.0 g, 6.37 mmol) in diisopropylamine/THF (60 ml, 1:1 *v*/*v*) under nitrogen was added a catalytic mixture of CuI (15 mg), $Pd(OAc)_2$ (16 mgl) and PPh_3 (50 mg). The solution was stirred for 20 min at 323 K and then trimethylsilylethyne (2.24 ml, 15.92 mmol) was added and the mixture stirred for another 20 min. The temperature was then raised to 348 K and the reaction left under reflux with stirring for 20 h. The completion of the reaction was determined by silica thin-layer chromatography and IR spectroscopy. The solution was allowed to cool to room temperature, was filtered and the solvent mixture removed. The residue was subjected to silica column chromatography using hexane/CH_2Cl_2 (1:2) as eluant to afford (I) as colourless needles (1.77 g, 80% yield).

Crystal data

$C_{20}H_{24}N_2Si_2$
M_r = 348.59
Monoclinic, $P2_1/c$
a = 6.1910 (6) Å
b = 25.697 (2) Å
c = 13.2450 (11) Å
β = 92.249 (5)°
V = 2105.5 (3) Å^3
Z = 4
D_x = 1.1 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 483 reflections
θ = 2.9–25.0°
μ = 0.17 mm^{-1}
T = 180 (2) K
Needle, colourless
0.18 × 0.11 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.94, T_{max} = 0.99
8978 measured reflections
3715 independent reflections
2301 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.1°
$h = -7 \rightarrow 7$
$k = -30 \rightarrow 30$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.110
S = 1.00
3715 reflections
243 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

The two trimethylsilyl groups are partially disordered, and one CH_3 group on each terminal group was refined over two positions with occupancies of 0.3 (1) and 0.7 (1) for C1 and C1′, respectively, and 0.56 (2) and 0.44 (2) for C18 and C18′, respectively; the associated H atoms were assigned the same occupancies. All aromatic and methyl H atoms were constrained as riding atoms, fixed to the parent atoms with distances of 0.93 and 0.96 Å, respectively. U_{iso} values were set equal to $1.2U_{eq}$ (1.5 for methyl H) of the parent atom.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* and *DENZO* (Otwinowski & Minor); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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(I)

Crystal data

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$M_r = 348.59$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.1910$ (6) Å
$b = 25.697$ (2) Å
$c = 13.2450$ (11) Å
$\beta = 92.249$ (5)°
$V = 2105.5$ (3) Å^3
$Z = 4$
$F(000) = 744$
$D_x = 1.1$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 22483 reflections
$\theta = 2.9$–25.0°
$\mu = 0.17$ mm^{-1}
$T = 180$ K
Tablet, colourless
0.18 × 0.11 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
CCD scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.94$, $T_{max} = 0.99$
8978 measured reflections
3715 independent reflections
2301 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 25.1°$, $\theta_{min} = 3.7°$
$h = 0 \rightarrow 7$
$k = -30 \rightarrow 30$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.110$
$S = 1.00$
3715 reflections
243 parameters
28 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Si1	1.15745 (10)	0.22354 (3)	0.20143 (5)	0.0452 (2)	
Si2	0.10692 (10)	0.59904 (3)	0.98707 (5)	0.0484 (2)	
C1	1.337 (4)	0.2506 (12)	0.1051 (14)	0.083 (8)	0.30
H1A	1.2521	0.2700	0.0559	0.124*	0.30
H1B	1.4092	0.2226	0.0722	0.124*	0.30
H1C	1.4423	0.2731	0.1374	0.124*	0.30
C1′	1.2693 (16)	0.2406 (6)	0.0769 (6)	0.074 (3)	0.70
H1′1	1.3616	0.2704	0.0846	0.111*	0.70
H1′2	1.1527	0.2483	0.0293	0.111*	0.70
H1′3	1.3511	0.2117	0.0526	0.111*	0.70
C2	0.9326 (4)	0.17786 (12)	0.1731 (2)	0.0854 (10)	
H2A	0.8593	0.1704	0.2340	0.128*	
H2B	0.9888	0.1462	0.1462	0.128*	
H2C	0.8330	0.1933	0.1245	0.128*	
C3	1.3557 (4)	0.19401 (12)	0.2916 (2)	0.0754 (9)	
H3A	1.4700	0.2184	0.3067	0.113*	
H3B	1.4146	0.1632	0.2625	0.113*	
H3C	1.2858	0.1851	0.3527	0.113*	
C4	1.0400 (3)	0.27990 (10)	0.26492 (17)	0.0467 (6)	
C5	0.9560 (3)	0.31345 (10)	0.31314 (17)	0.0435 (6)	
N1	0.5516 (3)	0.37694 (8)	0.47131 (14)	0.0459 (5)	
C6	0.6455 (3)	0.34556 (10)	0.40625 (17)	0.0480 (6)	
H6	0.5650	0.3179	0.3794	0.058*	
C7	0.8572 (3)	0.35155 (9)	0.37577 (16)	0.0389 (6)	
C8	0.9676 (3)	0.39481 (9)	0.41289 (16)	0.0422 (6)	
H8	1.1070	0.4015	0.3926	0.051*	
C9	0.8727 (3)	0.42769 (9)	0.47926 (15)	0.0358 (5)	
H9	0.9468	0.4568	0.5039	0.043*	
C10	0.6666 (3)	0.41736 (9)	0.50926 (15)	0.0327 (5)	
N2	0.6783 (3)	0.48926 (8)	0.62732 (14)	0.0421 (5)	
C11	0.5640 (3)	0.44926 (8)	0.58700 (15)	0.0324 (5)	
C12	0.3595 (3)	0.43736 (9)	0.61818 (16)	0.0408 (6)	
H12	0.2838	0.4094	0.5897	0.049*	
C13	0.2693 (3)	0.46696 (10)	0.69111 (17)	0.0474 (7)	
H13	0.1308	0.4594	0.7116	0.057*	
C14	0.3826 (3)	0.50797 (9)	0.73444 (16)	0.0382 (6)	
C15	0.5874 (3)	0.51767 (9)	0.69937 (16)	0.0425 (6)	
H15	0.6657	0.5454	0.7273	0.051*	
C16	0.2949 (3)	0.53950 (10)	0.81337 (18)	0.0442 (6)	
C17	0.2193 (4)	0.56410 (10)	0.88083 (18)	0.0489 (7)	
C18	−0.1937 (14)	0.5921 (7)	0.9876 (8)	0.069 (3)	0.56
H18A	−0.2579	0.6099	0.9302	0.103*	0.56
H18B	−0.2459	0.6069	1.0485	0.103*	0.56
H18C	−0.2315	0.5559	0.9843	0.103*	0.56
C18′	−0.1855 (17)	0.6039 (9)	0.9470 (10)	0.066 (4)	0.44

H18D	−0.1995	0.6220	0.8836	0.098*	0.44
H18E	−0.2618	0.6226	0.9972	0.098*	0.44
H18F	−0.2454	0.5696	0.9396	0.098*	0.44
C19	0.1993 (5)	0.56474 (12)	1.10385 (19)	0.0854 (10)	
H19A	0.1375	0.5305	1.1044	0.128*	
H19B	0.1542	0.5838	1.1617	0.128*	
H19C	0.3541	0.5621	1.1060	0.128*	
C20	0.2088 (4)	0.66656 (10)	0.98830 (19)	0.0596 (7)	
H20A	0.3634	0.6663	0.9977	0.089*	
H20B	0.1466	0.6854	1.0426	0.089*	
H20C	0.1695	0.6831	0.9252	0.089*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0495 (4)	0.0455 (5)	0.0411 (4)	0.0099 (3)	0.0095 (3)	−0.0041 (4)
Si2	0.0470 (4)	0.0500 (5)	0.0486 (5)	0.0025 (3)	0.0082 (3)	−0.0143 (4)
C1	0.122 (17)	0.079 (17)	0.049 (11)	0.062 (12)	0.024 (10)	0.024 (11)
C1′	0.087 (5)	0.082 (5)	0.056 (5)	0.022 (4)	0.039 (4)	0.004 (5)
C2	0.0666 (18)	0.078 (2)	0.112 (3)	0.0040 (16)	−0.0007 (16)	−0.041 (2)
C3	0.0790 (19)	0.078 (2)	0.069 (2)	0.0226 (17)	−0.0047 (15)	−0.0051 (17)
C4	0.0506 (14)	0.0484 (17)	0.0415 (15)	0.0035 (12)	0.0079 (11)	−0.0045 (14)
C5	0.0458 (14)	0.0428 (16)	0.0420 (15)	−0.0010 (12)	0.0039 (11)	−0.0024 (13)
N1	0.0415 (11)	0.0463 (13)	0.0505 (13)	−0.0045 (10)	0.0083 (9)	−0.0145 (11)
C6	0.0443 (14)	0.0476 (17)	0.0523 (16)	−0.0052 (12)	0.0062 (11)	−0.0198 (14)
C7	0.0409 (13)	0.0412 (16)	0.0348 (13)	0.0061 (11)	0.0059 (10)	−0.0019 (12)
C8	0.0406 (13)	0.0440 (16)	0.0425 (15)	−0.0024 (12)	0.0102 (10)	−0.0010 (13)
C9	0.0369 (12)	0.0343 (14)	0.0367 (13)	−0.0046 (10)	0.0057 (10)	−0.0030 (12)
C10	0.0364 (12)	0.0300 (14)	0.0317 (13)	0.0024 (10)	−0.0003 (9)	0.0018 (11)
N2	0.0455 (11)	0.0384 (12)	0.0428 (12)	−0.0019 (10)	0.0055 (9)	−0.0054 (10)
C11	0.0362 (12)	0.0289 (13)	0.0319 (13)	0.0023 (10)	−0.0015 (9)	0.0023 (11)
C12	0.0330 (12)	0.0447 (16)	0.0450 (14)	−0.0057 (11)	0.0046 (10)	−0.0109 (13)
C13	0.0362 (13)	0.0565 (19)	0.0500 (16)	−0.0027 (12)	0.0075 (11)	−0.0105 (14)
C14	0.0423 (13)	0.0396 (15)	0.0329 (14)	0.0109 (11)	0.0044 (10)	0.0007 (12)
C15	0.0502 (14)	0.0386 (15)	0.0388 (15)	−0.0039 (11)	0.0040 (11)	−0.0067 (12)
C16	0.0448 (14)	0.0441 (16)	0.0434 (16)	0.0043 (11)	−0.0020 (11)	−0.0004 (13)
C17	0.0497 (14)	0.0485 (18)	0.0485 (16)	0.0053 (12)	0.0022 (12)	−0.0069 (14)
C18	0.053 (4)	0.083 (8)	0.072 (8)	−0.003 (4)	0.017 (4)	−0.034 (7)
C18′	0.048 (4)	0.076 (10)	0.076 (10)	−0.001 (4)	0.029 (5)	−0.026 (8)
C19	0.140 (3)	0.067 (2)	0.0503 (18)	−0.004 (2)	0.0209 (17)	0.0006 (17)
C20	0.0637 (15)	0.0567 (19)	0.0580 (17)	0.0067 (14)	−0.0019 (12)	−0.0049 (15)

Geometric parameters (Å, º)

Si1—C4	1.839 (3)	C8—C9	1.369 (3)
Si1—C3	1.842 (2)	C8—H8	0.9300
Si1—C2	1.848 (3)	C9—C10	1.377 (3)
Si1—C1	1.860 (13)	C9—H9	0.9300

Si1—C1′	1.866 (6)	C10—C11	1.479 (3)
Si2—C17	1.829 (3)	N2—C15	1.343 (3)
Si2—C20	1.846 (3)	N2—C11	1.347 (3)
Si2—C19	1.851 (3)	C11—C12	1.381 (3)
Si2—C18	1.870 (9)	C12—C13	1.366 (3)
Si2—C18′	1.871 (11)	C12—H12	0.9300
C1—H1A	0.9600	C13—C14	1.379 (3)
C1—H1B	0.9600	C13—H13	0.9300
C1—H1C	0.9600	C14—C15	1.390 (3)
C1′—H1′1	0.9600	C14—C16	1.445 (3)
C1′—H1′2	0.9600	C15—H15	0.9300
C1′—H1′3	0.9600	C16—C17	1.204 (3)
C2—H2A	0.9600	C18—H18A	0.9600
C2—H2B	0.9600	C18—H18B	0.9600
C2—H2C	0.9600	C18—H18C	0.9600
C3—H3A	0.9600	C18′—H18D	0.9600
C3—H3B	0.9600	C18′—H18E	0.9600
C3—H3C	0.9600	C18′—H18F	0.9600
C4—C5	1.203 (3)	C19—H19A	0.9600
C5—C7	1.435 (3)	C19—H19B	0.9600
N1—C6	1.330 (3)	C19—H19C	0.9600
N1—C10	1.345 (3)	C20—H20A	0.9600
C6—C7	1.395 (3)	C20—H20B	0.9600
C6—H6	0.9300	C20—H20C	0.9600
C7—C8	1.385 (3)		

C4—Si1—C3	107.05 (12)	C6—C7—C5	121.2 (2)
C4—Si1—C2	106.55 (11)	C9—C8—C7	120.3 (2)
C3—Si1—C2	110.31 (15)	C9—C8—H8	119.8
C4—Si1—C1	106.1 (10)	C7—C8—H8	119.8
C3—Si1—C1	101.4 (8)	C8—C9—C10	119.5 (2)
C2—Si1—C1	124.3 (8)	C8—C9—H9	120.2
C4—Si1—C1′	112.7 (5)	C10—C9—H9	120.2
C3—Si1—C1′	114.1 (4)	N1—C10—C9	121.6 (2)
C2—Si1—C1′	105.8 (3)	N1—C10—C11	116.91 (18)
C1—Si1—C1′	18.8 (10)	C9—C10—C11	121.5 (2)
C17—Si2—C20	109.14 (12)	C15—N2—C11	117.93 (19)
C17—Si2—C19	107.14 (13)	N2—C11—C12	121.6 (2)
C20—Si2—C19	110.24 (13)	N2—C11—C10	117.76 (18)
C17—Si2—C18	111.4 (5)	C12—C11—C10	120.61 (19)
C20—Si2—C18	115.4 (6)	C13—C12—C11	119.5 (2)
C19—Si2—C18	103.1 (3)	C13—C12—H12	120.2
C17—Si2—C18′	102.0 (6)	C11—C12—H12	120.2
C20—Si2—C18′	105.5 (7)	C12—C13—C14	120.3 (2)
C19—Si2—C18′	122.2 (4)	C12—C13—H13	119.8
C18—Si2—C18′	19.1 (6)	C14—C13—H13	119.8
Si1—C1—H1A	109.5	C13—C14—C15	117.0 (2)
Si1—C1—H1B	109.5	C13—C14—C16	122.0 (2)

Si1—C1—H1C	109.5	C15—C14—C16	121.0 (2)
Si1—C1′—H1′1	109.5	N2—C15—C14	123.6 (2)
Si1—C1′—H1′2	109.5	N2—C15—H15	118.2
H1′1—C1′—H1′2	109.5	C14—C15—H15	118.2
Si1—C1′—H1′3	109.5	C17—C16—C14	177.6 (3)
H1′1—C1′—H1′3	109.5	C16—C17—Si2	177.5 (2)
H1′2—C1′—H1′3	109.5	Si2—C18—H18A	109.5
Si1—C2—H2A	109.5	Si2—C18—H18B	109.5
Si1—C2—H2B	109.5	Si2—C18—H18C	109.5
H2A—C2—H2B	109.5	Si2—C18′—H18D	109.5
Si1—C2—H2C	109.5	Si2—C18′—H18E	109.5
H2A—C2—H2C	109.5	H18D—C18′—H18E	109.5
H2B—C2—H2C	109.5	Si2—C18′—H18F	109.5
Si1—C3—H3A	109.5	H18D—C18′—H18F	109.5
Si1—C3—H3B	109.5	H18E—C18′—H18F	109.5
H3A—C3—H3B	109.5	Si2—C19—H19A	109.5
Si1—C3—H3C	109.5	Si2—C19—H19B	109.5
H3A—C3—H3C	109.5	H19A—C19—H19B	109.5
H3B—C3—H3C	109.5	Si2—C19—H19C	109.5
C5—C4—Si1	173.7 (2)	H19A—C19—H19C	109.5
C4—C5—C7	176.6 (3)	H19B—C19—H19C	109.5
C6—N1—C10	118.16 (18)	Si2—C20—H20A	109.5
N1—C6—C7	124.0 (2)	Si2—C20—H20B	109.5
N1—C6—H6	118.0	H20A—C20—H20B	109.5
C7—C6—H6	118.0	Si2—C20—H20C	109.5
C8—C7—C6	116.2 (2)	H20A—C20—H20C	109.5
C8—C7—C5	122.48 (19)	H20B—C20—H20C	109.5