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## Structure Reports

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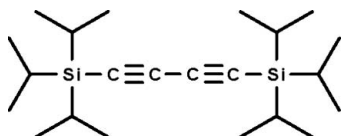
**(Butane-1,3-diyne-1,4-diyl)bis(triisopropylsilane)**Muhammad Raza Shah<sup>a</sup> and Seik Weng Ng<sup>b\*</sup><sup>a</sup>H.E.J. Research Institute of Chemistry, International Center for Chemical and Biological Sciences, University of Karachi, Karachi 75270, Pakistan, and<sup>b</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.116; data-to-parameter ratio = 23.3.The molecule of the title compound,  $\text{C}_{22}\text{H}_{42}\text{Si}_2$ , lies on a center of inversion, and the triisopropylsilyl groups are staggered.

## Related literature

For the crystal structures of the trimethyl and tris-*tert*-butyl analogs, see: Bruckmann & Krüger (1997); Vitze *et al.* (2009).

## Experimental

## Crystal data

 $\text{C}_{22}\text{H}_{42}\text{Si}_2$   
 $M_r = 362.74$   
 Triclinic,  $P\bar{1}$ 
 $a = 7.1213$  (10) Å  
 $b = 7.9057$  (11) Å  
 $c = 10.6937$  (14) Å

 $\alpha = 89.139$  (2)°  
 $\beta = 81.823$  (2)°  
 $\gamma = 79.449$  (2)°  
 $V = 585.81$  (14) Å<sup>3</sup>  
 $Z = 1$ 

 Mo  $K\alpha$  radiation  
 $\mu = 0.15$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.40 \times 0.10 \times 0.10$  mm

## Data collection

 Bruker SMART APEX  
 diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.941$ ,  $T_{\max} = 0.985$ 

 5560 measured reflections  
 2674 independent reflections  
 2190 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.036$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.116$   
 $S = 1.06$   
 2674 reflections

 115 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.34$  e Å<sup>-3</sup>

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2010).

We thank the Higher Education Commission of Pakistan and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2179).

## References

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## supporting information

*Acta Cryst.* (2010). E66, o2028 [https://doi.org/10.1107/S160053681002725X]

**(Butane-1,3-diyne-1,4-diyl)bis(triisopropylsilane)**

Muhammad Raza Shah and Seik Weng Ng

**S1. Comment**

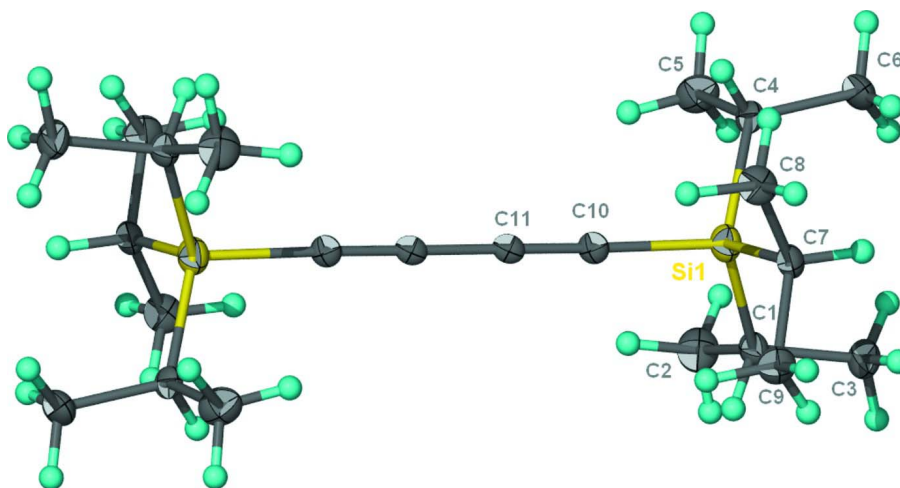
The compound (Scheme I) was obtained in an unsuccessful attempt at the Sonogoshira coupling of 2,9-dichloro-1,10-phenanthroline with triisopropylsilylacetylene. The carbon–carbon triple-bond is 1.210 (2) Å long; the distance is indistinguishable from that [1.208 (3) Å] for bis(trimethylsilyl)acetylene (Bruckmann & Krüger, 1997) as well as that [1.22 (2) Å] found in the *t*-butyl analog (Vitze *et al.* (2009). The molecule lies on a center of inversion, and the triisopropylsilyl groups are staggered (Fig. 1).

**S2. Experimental**

Copper(I) iodide (70 mg, 0.36 mmol) and dichlorobis(triphenylphosphine)palladium (10 mg, 0.014 mmol) were added to a pyridine solution (10 ml) of triisopropylsilylacetylene (440 mg, 2.4 mmol) and 2,9-dichloro-1,10-phenanthroline (200 mg, 0.8 mmol). The solution was stirred for 4 h. The pyridine was removed under vacuum and the residue dissolved in dichloromethane (10 ml). The solution was washed with 2 N hydrochloric acid (10 ml). The solvent was evaporated and the solid recrystallized from dichloromethane to afford colorless crystals.

**S3. Refinement**

Carbon-bound H-atoms were placed in calculated positions [C–H 0.98–1.00 Å,  $U(H)$  1.2–1.5 $U(C)$ ] and were included in the refinement in the riding model approximation.



**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of  $C_{24}H_{42}Si_2$  at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

**(Butane-1,3-diyne-1,4-diyl)bis(triisopropylsilane)***Crystal data*C<sub>22</sub>H<sub>42</sub>Si<sub>2</sub> $M_r = 362.74$ Triclinic,  $P\bar{1}$ 

Hall symbol: -P 1

 $a = 7.1213$  (10) Å $b = 7.9057$  (11) Å $c = 10.6937$  (14) Å $\alpha = 89.139$  (2)° $\beta = 81.823$  (2)° $\gamma = 79.449$  (2)° $V = 585.81$  (14) Å<sup>3</sup> $Z = 1$  $F(000) = 202$  $D_x = 1.028$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1787 reflections

 $\theta = 2.6$ – $27.7$ ° $\mu = 0.15$  mm<sup>-1</sup> $T = 100$  K

Prism, colorless

 $0.40 \times 0.10 \times 0.10$  mm*Data collection*

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.941$ ,  $T_{\max} = 0.985$ 

5560 measured reflections

2674 independent reflections

2190 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.036$  $\theta_{\text{max}} = 27.5$ °,  $\theta_{\text{min}} = 1.9$ ° $h = -9 \rightarrow 9$  $k = -10 \rightarrow 10$  $l = -13 \rightarrow 13$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.042$  $wR(F^2) = 0.116$  $S = 1.06$ 

2674 reflections

115 parameters

0 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

 $w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.0577P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = 0.001$  $\Delta\rho_{\text{max}} = 0.38$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.34$  e Å<sup>-3</sup>*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Si1	0.75095 (6)	0.77829 (5)	0.27093 (4)	0.01479 (15)
C1	0.7369 (2)	0.7104 (2)	0.10482 (15)	0.0195 (4)
H1	0.7950	0.5852	0.0980	0.023*
C2	0.5337 (3)	0.7257 (2)	0.07088 (18)	0.0295 (4)
H2A	0.5401	0.6716	-0.0118	0.044*
H2B	0.4745	0.8475	0.0677	0.044*
H2C	0.4559	0.6679	0.1349	0.044*
C3	0.8628 (3)	0.8009 (2)	0.00649 (16)	0.0269 (4)
H3A	0.8642	0.7528	-0.0776	0.040*
H3B	0.9947	0.7831	0.0271	0.040*
H3C	0.8096	0.9244	0.0074	0.040*
C4	0.6408 (2)	1.0072 (2)	0.31850 (16)	0.0185 (4)

H4	0.6446	1.0147	0.4113	0.022*
C5	0.4277 (3)	1.0599 (2)	0.30187 (18)	0.0264 (4)
H5A	0.3746	1.1712	0.3438	0.040*
H5B	0.3558	0.9728	0.3395	0.040*
H5C	0.4165	1.0693	0.2116	0.040*
C6	0.7578 (3)	1.1395 (2)	0.25767 (18)	0.0282 (4)
H6A	0.7061	1.2526	0.2972	0.042*
H6B	0.7489	1.1459	0.1671	0.042*
H6C	0.8932	1.1044	0.2699	0.042*
C7	1.0093 (2)	0.7272 (2)	0.30172 (16)	0.0178 (4)
H7	1.0807	0.8091	0.2514	0.021*
C8	1.0237 (3)	0.7550 (2)	0.44066 (17)	0.0233 (4)
H8A	1.1598	0.7363	0.4527	0.035*
H8B	0.9585	0.6737	0.4923	0.035*
H8C	0.9620	0.8730	0.4662	0.035*
C9	1.1074 (2)	0.5444 (2)	0.25796 (17)	0.0233 (4)
H9A	1.2368	0.5190	0.2827	0.035*
H9B	1.1173	0.5358	0.1658	0.035*
H9C	1.0307	0.4617	0.2973	0.035*
C10	0.6199 (2)	0.64130 (19)	0.37986 (15)	0.0161 (3)
C11	0.5440 (2)	0.55159 (19)	0.45656 (14)	0.0152 (3)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Si1	0.0163 (3)	0.0141 (2)	0.0141 (3)	-0.00465 (17)	-0.00050 (17)	0.00352 (16)
C1	0.0248 (9)	0.0177 (8)	0.0167 (9)	-0.0068 (7)	-0.0015 (7)	0.0022 (6)
C2	0.0319 (11)	0.0371 (11)	0.0229 (10)	-0.0123 (9)	-0.0080 (8)	0.0002 (8)
C3	0.0355 (11)	0.0301 (10)	0.0170 (9)	-0.0146 (8)	0.0009 (8)	0.0004 (7)
C4	0.0228 (9)	0.0173 (8)	0.0146 (8)	-0.0041 (7)	0.0006 (7)	0.0028 (6)
C5	0.0269 (10)	0.0230 (9)	0.0266 (10)	0.0029 (7)	-0.0043 (8)	0.0015 (7)
C6	0.0361 (11)	0.0163 (8)	0.0302 (11)	-0.0070 (8)	0.0051 (9)	0.0005 (7)
C7	0.0171 (8)	0.0166 (8)	0.0200 (9)	-0.0055 (6)	-0.0010 (7)	0.0022 (6)
C8	0.0192 (9)	0.0254 (9)	0.0263 (10)	-0.0034 (7)	-0.0077 (7)	0.0010 (7)
C9	0.0198 (9)	0.0215 (9)	0.0273 (10)	-0.0009 (7)	-0.0029 (7)	0.0016 (7)
C10	0.0159 (8)	0.0150 (7)	0.0174 (9)	-0.0017 (6)	-0.0041 (7)	0.0021 (6)
C11	0.0148 (8)	0.0142 (7)	0.0168 (9)	-0.0010 (6)	-0.0049 (7)	0.0001 (6)

*Geometric parameters (Å, °)*

Si1—C10	1.8504 (16)	C5—H5B	0.9800
Si1—C4	1.8822 (17)	C5—H5C	0.9800
Si1—C1	1.8848 (17)	C6—H6A	0.9800
Si1—C7	1.8849 (17)	C6—H6B	0.9800
C1—C2	1.524 (2)	C6—H6C	0.9800
C1—C3	1.538 (2)	C7—C8	1.526 (2)
C1—H1	1.0000	C7—C9	1.533 (2)
C2—H2A	0.9800	C7—H7	1.0000

C2—H2B	0.9800	C8—H8A	0.9800
C2—H2C	0.9800	C8—H8B	0.9800
C3—H3A	0.9800	C8—H8C	0.9800
C3—H3B	0.9800	C9—H9A	0.9800
C3—H3C	0.9800	C9—H9B	0.9800
C4—C5	1.533 (2)	C9—H9C	0.9800
C4—C6	1.535 (2)	C10—C11	1.210 (2)
C4—H4	1.0000	C11—C11 <sup>i</sup>	1.385 (3)
C5—H5A	0.9800		
C10—Si1—C4	106.04 (7)	C4—C5—H5B	109.5
C10—Si1—C1	107.42 (7)	H5A—C5—H5B	109.5
C4—Si1—C1	117.06 (8)	C4—C5—H5C	109.5
C10—Si1—C7	105.69 (7)	H5A—C5—H5C	109.5
C4—Si1—C7	110.41 (7)	H5B—C5—H5C	109.5
C1—Si1—C7	109.52 (8)	C4—C6—H6A	109.5
C2—C1—C3	110.85 (14)	C4—C6—H6B	109.5
C2—C1—Si1	115.47 (12)	H6A—C6—H6B	109.5
C3—C1—Si1	111.67 (11)	C4—C6—H6C	109.5
C2—C1—H1	106.0	H6A—C6—H6C	109.5
C3—C1—H1	106.0	H6B—C6—H6C	109.5
Si1—C1—H1	106.0	C8—C7—C9	110.89 (14)
C1—C2—H2A	109.5	C8—C7—Si1	111.21 (11)
C1—C2—H2B	109.5	C9—C7—Si1	111.98 (11)
H2A—C2—H2B	109.5	C8—C7—H7	107.5
C1—C2—H2C	109.5	C9—C7—H7	107.5
H2A—C2—H2C	109.5	Si1—C7—H7	107.5
H2B—C2—H2C	109.5	C7—C8—H8A	109.5
C1—C3—H3A	109.5	C7—C8—H8B	109.5
C1—C3—H3B	109.5	H8A—C8—H8B	109.5
H3A—C3—H3B	109.5	C7—C8—H8C	109.5
C1—C3—H3C	109.5	H8A—C8—H8C	109.5
H3A—C3—H3C	109.5	H8B—C8—H8C	109.5
H3B—C3—H3C	109.5	C7—C9—H9A	109.5
C5—C4—C6	110.60 (14)	C7—C9—H9B	109.5
C5—C4—Si1	114.57 (11)	H9A—C9—H9B	109.5
C6—C4—Si1	113.60 (11)	C7—C9—H9C	109.5
C5—C4—H4	105.7	H9A—C9—H9C	109.5
C6—C4—H4	105.7	H9B—C9—H9C	109.5
Si1—C4—H4	105.7	C11—C10—Si1	175.41 (14)
C4—C5—H5A	109.5	C10—C11—C11 <sup>i</sup>	179.4 (2)
C10—Si1—C1—C2	59.25 (14)	C1—Si1—C4—C6	-72.44 (15)
C4—Si1—C1—C2	-59.79 (14)	C7—Si1—C4—C6	53.75 (15)
C7—Si1—C1—C2	173.58 (12)	C10—Si1—C7—C8	-56.07 (12)
C10—Si1—C1—C3	-172.91 (12)	C4—Si1—C7—C8	58.19 (13)
C4—Si1—C1—C3	68.05 (14)	C1—Si1—C7—C8	-171.50 (11)
C7—Si1—C1—C3	-58.59 (14)	C10—Si1—C7—C9	68.62 (13)

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C10—Si1—C4—C5	-63.77 (14)	C4—Si1—C7—C9	-177.12 (11)
C1—Si1—C4—C5	56.00 (14)	C1—Si1—C7—C9	-46.81 (13)
C7—Si1—C4—C5	-177.80 (12)	C1—Si1—C10—C11	142.3 (18)
C10—Si1—C4—C6	167.78 (12)		

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Symmetry code: (i)  $-x+1, -y+1, -z+1$ .