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

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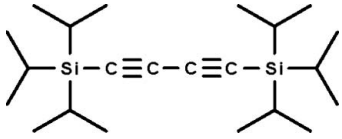
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.116; data-to-parameter ratio = 23.3.

The molecule of the title compound, $C_{22}H_{42}Si_2$, 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

$C_{22}H_{42}Si_2$
M_r = 362.74
Triclinic, $P\overline{1}$
a = 7.1213 (10) Å
b = 7.9057 (11) Å
c = 10.6937 (14) Å
α = 89.139 (2)°
β = 81.823 (2)°
γ = 79.449 (2)°
V = 585.81 (14) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 100 K
0.40 × 0.10 × 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_{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^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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).

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

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supplementary materials

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

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Comment

The compound (Scheme I) was obtained in an unsuccessful attempt at the Sonogoshira coupling of 2,9-dichloro-1,10-phenanthroline with trisisopropylsilylacetylene. The carbon–carbon triple-bond is 1.210 (2) Å long; the distance is indistinguisahble 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 trisisopropylsilyl groups are staggered (Fig. 1).

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.

Refinement

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

Figures

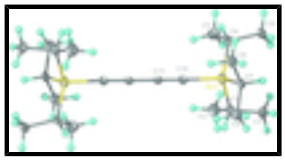

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{24}H_{42}Si_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{22}H_{42}Si_2$	Z = 1
M_r = 362.74	F(000) = 202
Triclinic, $P\overline{1}$	D_x = 1.028 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 7.1213 (10) Å	Cell parameters from 1787 reflections
b = 7.9057 (11) Å	θ = 2.6–27.7°

c = 10.6937 (14) Å	μ = 0.15 mm^{-1}
α = 89.139 (2)°	T = 100 K
β = 81.823 (2)°	Prism, colorless
γ = 79.449 (2)°	0.40 × 0.10 × 0.10 mm
V = 585.81 (14) Å^3	

Data collection

Bruker SMART APEX diffractometer	2674 independent reflections
Radiation source: fine-focus sealed tube	2190 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.036
ω scans	θ_{max} = 27.5°, θ_{min} = 1.9°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -9 \rightarrow 9$
T_{min} = 0.941, T_{max} = 0.985	$k = -10 \rightarrow 10$
5560 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.042	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.116	H-atom parameters constrained
S = 1.06	$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.057P]$ where $P = (F_o^2 + 2F_c^2)/3$
2674 reflections	$(\Delta/\sigma)_{max}$ = 0.001
115 parameters	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{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^2)

	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^{i}	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^{i}	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)
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)		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1