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Structure of 1,3-Bis(1-pyrenyl)hexamethyltrisilane

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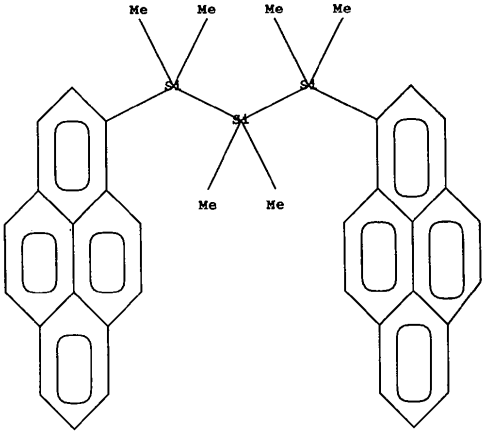
Abstract

The trisilane chain C(1)—Si(1)—Si(2)—Si(3)—C(1*B*) adopts a −anticlinal, +synclinal conformation. The pyrene rings are slightly folded and make and angle of 71° to each other.

Comment

The structure determination of the title compound was undertaken to establish the mutual orientation of the pyrene rings. Details of the spectroscopic properties of dipyrenyl-substituted oligosilanes are reported by Declercq, De Schryver & Miller (1991).



The final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1. The molecular structure with labelling is depicted in Fig. 1. Bond distances and selected bond angles are listed in Table 2.

The trisilane chain adopts a −anticlinal [C(1*B*)—Si(1)—Si(2)—Si(3) = −117.6 (2)°], +synclinal [Si(1)—Si(2)—Si(3)—C(1) = 62.8 (2)°] conformation, with the pyrene rings almost perpendicular to each other. The angle between the best planes through the pyrene atoms is 71°. Both pyrene rings are slightly folded, significantly more than found in the crystal structure of pyrene (Hazell, Larsen & Lehmann, 1972). The maximum deviation from the best plane through the pyrene atoms is 0.055 Å for ring *A* and −0.047 Å for ring *B*, compared with −0.020 Å for pyrene. Both pyrene rings show a very short C(4)—C(5) bond distance of 1.331 (8) Å (ring *A*) and 1.329 (6) Å (ring *B*).

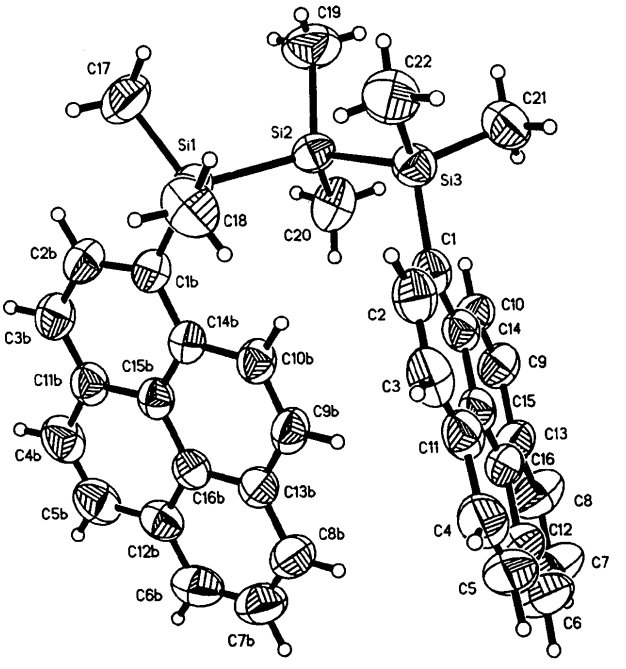


Fig. 1. View of the molecule with labelling. Thermal ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radius (*SHELXTL PC*, Sheldrick 1990).

A search in the January 1992 version of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed two comparable 1,8-bis(1-pyrenyl)naphthalenes (*cis* and *trans*; Wahl, Krieger, Schweitzer & Staab, 1984). Here the trisilane chain is replaced by a planar Csp^2—Csp^2—Csp^2 chain bringing the two pyrene planes parallel to each other.

Experimental

Crystal data

$C_{38}H_{36}Si_3$
M_r = 576.9
Monoclinic
$P2_1/c$
a = 11.213 (2) Å
b = 22.656 (4) Å
c = 13.397 (3) Å
β = 110.70 (1)°
V = 3183.7 (11) Å^3
Z = 4
D_x = 1.204 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 30 reflections
θ = 4–13°
μ = 1.522 mm^{-1}
T = 289 K
Parallelepiped
0.4 × 0.3 × 0.3 mm
Colourless with yellow fluorescence
Crystal source: crystallized by evaporation from cyclohexane

0108-2701/93/040831-03$06.00

Data collection

Siemens *P4/PC* diffractometer
2θ–θ scans
Absorption correction: Semi-empirical
T_{min} = 0.5655, T_{max} = 0.6690
4606 measured reflections
4352 independent reflections

3459 observed reflections [$F>4.0\sigma(F)$]
R_{int} = 0.0365
θ_{max} = 58°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 24$
$l = -14 \rightarrow 13$
3 standard reflections monitored every 50 reflections
intensity variation: none

Refinement

Refinement on F
Final R = 0.0424
wR = 0.0451
S = 1.23
3459 reflections
406 parameters
Only H-atom U's refined [riding model (C—H = 0.96 Å)]

Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.081
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

U_{eq} = 1/3(trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
Si(1)	0.5706 (1)	0.3922 (1)	0.7183 (1)	0.058 (1)
Si(2)	0.4217 (1)	0.3735 (1)	0.8019 (1)	0.055 (1)
Si(3)	0.4712 (1)	0.2875 (1)	0.9076 (1)	0.057 (1)
C(17)	0.4796 (6)	0.3965 (3)	0.5699 (3)	0.097 (3)
C(18)	0.6881 (4)	0.3303 (2)	0.7406 (4)	0.085 (3)
C(19)	0.2619 (4)	0.3564 (3)	0.6933 (4)	0.099 (2)
C(20)	0.3971 (4)	0.4405 (2)	0.8755 (3)	0.074 (2)
C(21)	0.3373 (4)	0.2707 (2)	0.9576 (4)	0.079 (2)
C(22)	0.4781 (5)	0.2238 (2)	0.8196 (4)	0.086 (2)
C(1*B*)	0.6581 (3)	0.4644 (2)	0.7626 (3)	0.053 (1)
C(2*B*)	0.6524 (4)	0.5076 (2)	0.6860 (3)	0.065 (2)
C(3*B*)	0.7124 (4)	0.5614 (2)	0.7126 (3)	0.068 (2)
C(4*B*)	0.8429 (4)	0.6319 (2)	0.8492 (4)	0.075 (2)
C(5*B*)	0.9083 (4)	0.6450 (2)	0.9506 (4)	0.079 (2)
C(6*B*)	0.9922 (4)	0.6152 (2)	1.1407 (4)	0.080 (2)
C(7*B*)	1.0063 (4)	0.5734 (2)	1.2186 (4)	0.083 (2)
C(8*B*)	0.9532 (4)	0.5181 (2)	1.1930 (3)	0.071 (2)
C(9*B*)	0.8208 (3)	0.4479 (2)	1.0565 (3)	0.061 (2)
C(10*B*)	0.7496 (3)	0.4358 (2)	0.9549 (3)	0.056 (2)
C(11*B*)	0.7814 (3)	0.5760 (2)	0.8175 (3)	0.058 (2)
C(12*B*)	0.9228 (3)	0.6032 (2)	1.0342 (3)	0.063 (2)
C(13*B*)	0.8817 (3)	0.5038 (2)	1.0874 (3)	0.054 (1)
C(14*B*)	0.7315 (3)	0.4772 (2)	0.8700 (3)	0.049 (1)
C(15*B*)	0.7924 (3)	0.5329 (2)	0.8982 (3)	0.049 (1)
C(16*B*)	0.8659 (3)	0.5467 (2)	1.0065 (3)	0.051 (1)
C(1)	0.6281 (3)	0.2907 (2)	1.0232 (3)	0.053 (1)
C(2)	0.7301 (4)	0.2549 (2)	1.0226 (3)	0.064 (2)
C(3)	0.8447 (4)	0.2537 (2)	1.1072 (4)	0.070 (2)
C(4)	0.9814 (4)	0.2870 (2)	1.2885 (4)	0.082 (2)
C(5)	0.9989 (4)	0.3215 (2)	1.3729 (4)	0.091 (2)
C(6)	0.9218 (6)	0.4005 (3)	1.4631 (4)	0.107 (3)
C(7)	0.8272 (6)	0.4385 (3)	1.4645 (4)	0.119 (3)
C(8)	0.7116 (5)	0.4401 (2)	1.3815 (3)	0.092 (2)
C(9)	0.5711 (4)	0.4033 (2)	1.2048 (3)	0.062 (2)
C(10)	0.5525 (3)	0.3676 (2)	1.1206 (3)	0.054 (1)
C(11)	0.8645 (3)	0.2876 (2)	1.1973 (3)	0.063 (2)
C(12)	0.9046 (4)	0.3616 (2)	1.3781 (3)	0.081 (2)
C(13)	0.6892 (4)	0.4027 (2)	1.2937 (3)	0.066 (2)
C(14)	0.6481 (3)	0.3269 (2)	1.1129 (3)	0.051 (1)
C(15)	0.7662 (3)	0.3257 (2)	1.2010 (3)	0.054 (1)
C(16)	0.7862 (4)	0.3632 (2)	1.2905 (3)	0.062 (2)

Table 2. *Geometric parameters* (Å, °)

Si(1)—Si(2)	2.355 (2)	Si(1)—C(17)	1.890 (4)
Si(1)—C(18)	1.875 (5)	Si(1)—C(1*B*)	1.892 (4)
Si(2)—Si(3)	2.356 (2)	Si(2)—C(19)	1.906 (4)
Si(2)—C(20)	1.881 (5)	Si(3)—C(21)	1.886 (6)
Si(3)—C(22)	1.883 (5)	Si(3)—C(1)	1.891 (3)
C(1*B*)—C(2*B*)	1.403 (6)	C(1*B*)—C(14*B*)	1.411 (4)
C(2*B*)—C(3*B*)	1.376 (6)	C(3*B*)—C(11*B*)	1.384 (5)
C(4*B*)—C(5*B*)	1.329 (6)	C(4*B*)—C(11*B*)	1.434 (6)
C(5*B*)—C(12*B*)	1.431 (6)	C(6*B*)—C(7*B*)	1.377 (7)
C(6*B*)—C(12*B*)	1.389 (6)	C(7*B*)—C(8*B*)	1.376 (7)
C(8*B*)—C(13*B*)	1.396 (5)	C(9*B*)—C(10*B*)	1.341 (5)
C(9*B*)—C(13*B*)	1.429 (5)	C(10*B*)—C(14*B*)	1.431 (5)
C(11*B*)—C(15*B*)	1.429 (5)	C(12*B*)—C(16*B*)	1.420 (5)
C(13*B*)—C(16*B*)	1.419 (5)	C(14*B*)—C(15*B*)	1.422 (5)
C(15*B*)—C(16*B*)	1.427 (4)	C(1)—C(2)	1.402 (6)
C(1)—C(14)	1.406 (5)	C(2)—C(3)	1.381 (5)
C(3)—C(11)	1.382 (6)	C(4)—C(5)	1.331 (8)
C(4)—C(11)	1.442 (5)	C(5)—C(12)	1.415 (7)
C(6)—C(7)	1.372 (9)	C(6)—C(12)	1.398 (8)
C(7)—C(8)	1.378 (7)	C(8)—C(13)	1.399 (6)
C(9)—C(10)	1.344 (5)	C(9)—C(13)	1.434 (5)
C(10)—C(14)	1.446 (5)	C(11)—C(15)	1.414 (6)
C(12)—C(16)	1.428 (5)	C(13)—C(16)	1.420 (6)
C(14)—C(15)	1.429 (4)	C(15)—C(16)	1.421 (5)
Si(2)—Si(1)—C(17)	107.5 (2)	Si(2)—Si(1)—C(18)	111.5 (2)
C(17)—Si(1)—C(18)	107.1 (3)	Si(2)—Si(1)—C(1*B*)	112.5 (1)
C(17)—Si(1)—C(1*B*)	108.2 (2)	C(18)—Si(1)—C(1*B*)	109.9 (2)
Si(1)—Si(2)—Si(3)	112.2 (1)	Si(1)—Si(2)—C(19)	107.8 (2)
Si(3)—Si(2)—C(19)	104.4 (2)	Si(1)—Si(2)—C(20)	111.3 (2)
Si(3)—Si(2)—C(20)	113.5 (1)	C(19)—Si(2)—C(20)	106.9 (2)
Si(2)—Si(3)—C(21)	109.4 (2)	Si(2)—Si(3)—C(22)	108.1 (2)
C(21)—Si(3)—C(22)	106.4 (2)	Si(2)—Si(3)—C(1)	114.5 (1)
C(21)—Si(3)—C(1)	110.1 (2)	C(22)—Si(3)—C(1)	108.0 (2)
Si(1)—C(1*B*)—C(2*B*)	119.3 (2)	Si(1)—C(1*B*)—C(14*B*)	123.2 (3)
Si(3)—C(1)—C(2)	119.9 (3)	Si(3)—C(1)—C(14)	122.9 (3)

Data collection, data reduction and absorption correction: Siemens *P3/PC* software (Siemens Analytical X-ray Instruments, Inc., 1989). The program package *SHELXTL PC* (Sheldrick, 1990) was used for all calculations and figures. Refinement was by full-matrix least-squares methods.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55696 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1013]

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Structure of the Photostable Form of *p*-Nitrocinnamic Acid

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Abstract

The molecule is planar to within 0.08 (2) Å. The molecules form a centrosymmetric dimer through O—H···O hydrogen bonds between the carboxylic groups. The dimers are stacked along **b** with an interplanar distance of 3.374 (2) Å. The C=C double bonds of the nearest neighbors are related by a **b** translation.

Comment

The structure of the photostable form of *p*-nitrocinnamic acid has been determined as part of studies into solid-state photoreactions (Iwamoto, Kashino & Haisa, 1989). The lattice parameters and space groups of the photostable and photoreactive forms of the title compound have been reported by Schmidt (1964). The crystals used in the present study were obtained by slow evaporation from an ethanol solution. The crystal structure is similar to that of photoreactive *p*-formylcinnamic acid (Nakanishi, Hasegawa & Mori, 1985). However, the length of the *b* axis is 0.206 Å longer than in *p*-formylcinnamic acid.

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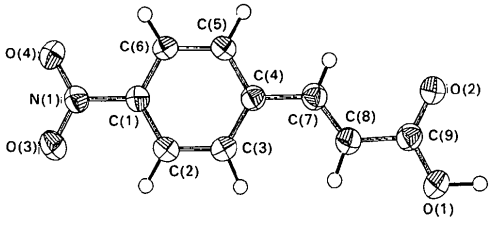

Fig. 1. The thermal ellipsoids with atomic numbering. Ellipsoids are drawn at 50% probability for the non-H atoms; the H atoms are represented as spheres equivalent to $B = 1.0$ Å^2.

Experimental

Crystal data

$C_9H_7NO_4$
$M_r = 193.16$
Monoclinic
$P2_1/a$
$a = 27.729$ (5) Å
$b = 5.0311$ (7) Å
$c = 6.105$ (1) Å
$\beta = 99.57$ (2)°
$V = 839.9$ (3) Å^3
$Z = 4$
$D_x = 1.527$ Mg m^{-3}
$D_m = 1.50$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 18 reflections
$\theta = 8$–18°
$\mu = 1.06$ mm^{-1}
$T = 295$ K
Prismatic
0.28 × 0.28 × 0.15 mm
Colorless

Data collection

Rigaku AFC-5 diffractometer
ω-2θ scans
1548 measured reflections
1329 independent reflections
1284 observed reflections [$F_o > 1\sigma(F_o)$]
$R_{int} = 0.011$ for 138 $hk0$ reflections
$\theta_{max} = 62.5°$
$h = -31 \rightarrow 30$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 6$
3 standard reflections monitored every 57 reflections
intensity variation: 1%

Refinement

Refinement on F
Final $R = 0.045$
$wR = 0.055$
$S = 2.91$
1284 reflections
156 parameters
All H-atom parameters refined
$w = 1/[\sigma(F_o)^2 - 0.4488|F_o| + 0.0228|F_o|^2]$
$(\Delta/\sigma)_{max} = 0.23$
$\Delta\rho_{max} = 0.2$ e Å^{-3}
$\Delta\rho_{min} = -0.3$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\beta_{ii}/a_{ii}^{*2}$.

	x	*y*	*z*	B_{eq}
O(1)	0.50023 (4)	1.3330 (2)	0.2491 (2)	4.57 (5)
O(2)	0.45399 (4)	1.2762 (2)	−0.0802 (2)	4.36 (5)
O(3)	0.30860 (4)	−0.0074 (2)	0.6531 (2)	4.33 (5)
O(4)	0.26096 (4)	−0.0400 (2)	0.3379 (2)	4.05 (5)
N(1)	0.29527 (4)	0.0591 (2)	0.4595 (2)	3.09 (5)
C(1)	0.32246 (5)	0.2730 (3)	0.3712 (2)	2.79 (5)
C(2)	0.36232 (5)	0.3803 (3)	0.5065 (2)	3.43 (6)
C(3)	0.38838 (5)	0.5765 (3)	0.4236 (2)	3.55 (6)
C(4)	0.37501 (5)	0.6666 (3)	0.2051 (2)	2.89 (6)
C(5)	0.33429 (5)	0.5554 (3)	0.0745 (2)	3.21 (6)
C(6)	0.30776 (5)	0.3572 (3)	0.1563 (2)	3.12 (6)
C(7)	0.40240 (5)	0.8759 (3)	0.1126 (2)	3.22 (6)
C(8)	0.44095 (5)	1.0036 (3)	0.2189 (2)	3.60 (7)
C(9)	0.46586 (5)	1.2160 (3)	0.1198 (2)	3.41 (6)

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