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[Si1—Si2—Si3—C23 = −177.9 (1)°] conformation, with the two aromatic systems pointing away from each other.

The angle between the best planes through the pyrene and phenyl atoms is 56.2 (1)°. The maximum deviation from the best plane through the pyrene atoms is 0.030 (3) Å (for atom C1) and through the phenyl atoms the maximum deviation is 0.004 (3) Å. This structure is comparable to the recently reported structure of 1,3-bis(1-pyrenyl)hexamethyltrisilane (Van Meervelt, 1993), where the angle between the pyrene ring systems is 71° with a −anticlinal, +synclinal conformation for the trisilane chain.

1-[4-(Dimethylamino)phenyl]-3-(1-pyrenyl)hexamethyltrisilane

LUC VAN MEERVELT

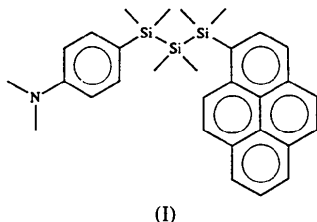*Laboratorium voor Macromoleculaire Structuurchemie, KU Leuven, Celestijnenlaan 200F, B-3001 Leuven, Belgium*

Abstract

The trisilane chain C1—Si1—Si2—Si3—C23 of the title compound, 1-[4-(dimethylamino)phenyl]-1,1,2,2,3,3-hexamethyl-3-(1-pyrenyl)trisilane, $C_{30}H_{37}NSi_3$, adopts a +synclinal, −antiperiplanar conformation. The two aromatic systems make an angle of 56.2 (1)° with each other.

Comment

Donor–acceptor substituted oligosilanes can be used as model systems for the study of through-bond interactions. The structure determination of the title compound, (I), was undertaken in order to establish the mutual orientation of the two aromatic systems, pyrene and phenyl, and to determine the conformation of the trisilane chain.

(I)

The molecular structure with atomic labelling is depicted in Fig. 1. The trisilane chain adopts a +synclinal [C1—Si1—Si2—Si3 = 48.2 (2)°], −antiperiplanar
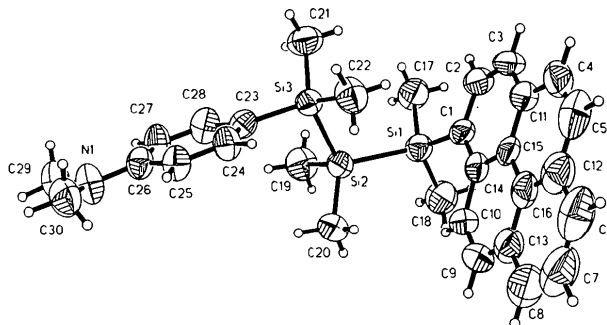

Fig. 1. View (*SHELXTL/PC*; Sheldrick 1990) of the title molecule with atomic labelling. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary size.

Experimental

Crystal data

$C_{30}H_{37}NSi_3$	Cu $K\alpha$ radiation
$M_r = 495.88$	$\lambda = 1.54178$ Å
Monoclinic	Cell parameters from 25
$P2_1/n$	reflections
$a = 7.8980$ (10) Å	$\theta = 7–23°$
$b = 26.463$ (4) Å	$\mu = 1.626$ mm^{-1}
$c = 14.099$ (2) Å	$T = 289$ (2) K
$\beta = 99.69$ (2)°	Block
$V = 2904.7$ (7) Å^3	0.20 × 0.20 × 0.10 mm
$Z = 4$	Yellow
$D_x = 1.134$ Mg m^{-3}	Crystal source: grown from cyclohexane

Data collection

Siemens *P4/PC* diffractometer	$R_{int} = 0.0520$
θ–2θ scans	$\theta_{max} = 50.41°$
Absorption correction:	$h = -1 \rightarrow 7$
empirical	$k = -1 \rightarrow 26$
$T_{min} = 0.479$, $T_{max} = 0.991$	$l = -14 \rightarrow 14$
4081 measured reflections	3 standard reflections
3030 independent reflections	monitored every 100
1827 observed reflections	reflections
$[I > 2\sigma(I)]$	intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0640$
$wR(F^2) = 0.1746$
$S = 1.058$
3030 reflections
308 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0748P)^2 + 0.5736P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.063$
$\Delta\rho_{max} = 0.308$ e Å^{-3}
$\Delta\rho_{min} = -0.204$ e Å^{-3}

Extinction correction:
$F_c^* = 1/kF_c[1 + (0.001 \times F_c^2\lambda^3/\sin2\theta)]^{1/4}$
Extinction coefficient:
0.00023 (11)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Si1	0.0343 (2)	0.28680 (5)	0.93410 (9)	0.0625 (4)
Si2	−0.0069 (2)	0.34352 (5)	0.80437 (8)	0.0564 (4)
Si3	−0.2972 (2)	0.35587 (5)	0.74315 (8)	0.0555 (4)
C1	−0.0999 (5)	0.2287 (2)	0.9023 (3)	0.0559 (14)
C2	−0.2453 (6)	0.2204 (2)	0.9452 (3)	0.068 (2)
C3	−0.3594 (6)	0.1813 (2)	0.9185 (3)	0.075 (2)
C4	−0.4560 (6)	0.1079 (2)	0.8128 (3)	0.081 (2)
C5	−0.4322 (7)	0.0767 (2)	0.7416 (4)	0.095 (2)
C6	−0.2623 (8)	0.0488 (2)	0.6205 (4)	0.109 (2)
C7	−0.1176 (9)	0.0546 (2)	0.5780 (4)	0.118 (3)
C8	−0.0009 (8)	0.0924 (2)	0.6047 (3)	0.108 (2)
C9	0.0953 (7)	0.1652 (2)	0.7097 (3)	0.088 (2)
C10	0.0722 (6)	0.1976 (2)	0.7804 (3)	0.071 (2)
C11	−0.3391 (6)	0.1487 (2)	0.8455 (3)	0.0670 (15)
C12	−0.2852 (7)	0.0822 (2)	0.6954 (3)	0.085 (2)
C13	−0.0218 (7)	0.1261 (2)	0.6787 (3)	0.077 (2)
C14	−0.0742 (6)	0.1947 (2)	0.8282 (3)	0.0579 (14)
C15	−0.1925 (6)	0.1547 (2)	0.8006 (3)	0.0598 (14)
C16	−0.1660 (7)	0.1210 (2)	0.7251 (3)	0.068 (2)
C17	−0.0438 (7)	0.3195 (2)	1.0367 (3)	0.085 (2)
C18	0.2670 (6)	0.2726 (2)	0.9758 (3)	0.092 (2)
C19	0.0880 (6)	0.4057 (2)	0.8513 (3)	0.082 (2)
C20	0.0965 (6)	0.3229 (2)	0.6996 (3)	0.080 (2)
C21	−0.4093 (6)	0.3860 (2)	0.8354 (3)	0.089 (2)
C22	−0.4021 (6)	0.2945 (2)	0.7023 (3)	0.080 (2)
C23	−0.3017 (5)	0.3996 (2)	0.6394 (3)	0.0522 (13)
C24	−0.3470 (6)	0.3851 (2)	0.5431 (3)	0.0595 (14)
C25	−0.3410 (6)	0.4167 (2)	0.4674 (3)	0.0591 (14)
C26	−0.2900 (6)	0.4667 (2)	0.4812 (3)	0.0585 (14)
C27	−0.2447 (6)	0.4822 (2)	0.5768 (3)	0.072 (2)
C28	−0.2524 (6)	0.4498 (2)	0.6502 (3)	0.071 (2)
N1	−0.2763 (5)	0.49944 (14)	0.4074 (3)	0.0767 (14)
C29	−0.2279 (7)	0.5517 (2)	0.4269 (3)	0.089 (2)
C30	−0.3514 (7)	0.4865 (2)	0.3104 (3)	0.086 (2)

Table 2. *Selected geometric parameters* (Å, °)

Si1—C18	1.871 (5)	C7—C8	1.369 (8)
Si1—C17	1.877 (4)	C8—C13	1.404 (7)
Si1—C1	1.878 (4)	C9—C10	1.351 (6)
Si1—Si2	2.346 (2)	C9—C13	1.406 (7)
Si2—C19	1.882 (5)	C10—C14	1.434 (6)
Si2—C20	1.885 (5)	C11—C15	1.419 (6)
Si2—Si3	2.332 (2)	C12—C16	1.408 (7)
Si3—C23	1.860 (4)	C13—C16	1.413 (7)
Si3—C21	1.871 (5)	C14—C15	1.422 (6)
Si3—C22	1.869 (5)	C15—C16	1.431 (6)

C1—C2	1.403 (6)	C23—C28	1.385 (6)
C1—C14	1.420 (6)	C23—C24	1.398 (5)
C2—C3	1.382 (6)	C24—C25	1.364 (5)
C3—C11	1.372 (6)	C25—C26	1.387 (6)
C4—C5	1.337 (7)	C26—N1	1.372 (5)
C4—C11	1.445 (6)	C26—C27	1.397 (6)
C5—C12	1.432 (8)	C27—C28	1.355 (6)
C6—C7	1.385 (9)	N1—C30	1.437 (5)
C6—C12	1.413 (7)	N1—C29	1.449 (6)
C18—Si1—C17	106.4 (2)	Si3—Si2—Si1	112.14 (7)
C18—Si1—C1	113.3 (2)	C23—Si3—C21	109.4 (2)
C17—Si1—C1	108.4 (2)	C23—Si3—C22	110.4 (2)
C18—Si1—Si2	111.9 (2)	C21—Si3—C22	110.3 (2)
C17—Si1—Si2	106.6 (2)	C23—Si3—Si2	105.4 (1)
C1—Si1—Si2	109.8 (1)	C21—Si3—Si2	110.7 (2)
C19—Si2—C20	109.2 (2)	C22—Si3—Si2	110.5 (2)
C19—Si2—Si3	108.4 (2)	C2—C1—Si1	119.7 (3)
C20—Si2—Si3	106.2 (2)	C14—C1—Si1	123.4 (3)
C19—Si2—Si1	106.9 (2)	C28—C23—Si3	122.8 (3)
C20—Si2—Si1	113.8 (2)	C24—C23—Si3	124.1 (3)

The H atoms were refined using a rigid model (C_{sp^2}—H = 0.93, C_{sp^3}—H = 0.96 Å) with U_{iso}(H) = 1.2U_{eq}(C). Dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C), Tables 4.2.6.8 and 4.2.4.2, respectively.

Data collection: *P3/PC Data Collection Program* (Siemens, 1989). Cell refinement: *P3/PC Data Collection Program*. Data reduction: *XDISK* (Siemens, 1991). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*.

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