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Dimethyl 4,4′-(dimethylsilanediyl)dibenzoate

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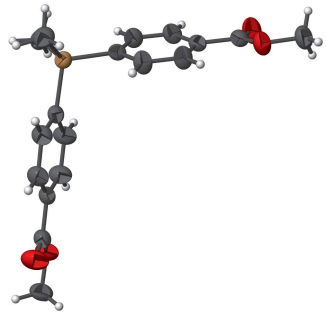
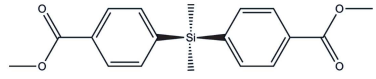
The complete molecule of the title compound, $C_{18}H_{20}O_4Si$, is generated by crystallographic twofold symmetry, with the Si atom lying on the rotation axis. The molecule adopts a V-shape: the dihedral angle between the benzene ring and it attached methyl formate unit is 9.3 (2)°, and the dihedral angle between the benzene rings is 68.8 (1)°. In the crystal, weak C—H···O hydrogen bonds link the molecules into [101] chains.

3D view



Chemical scheme



Structure description

Recently, several reports have indicated that Si-based tetrahedral organic molecules have excellent emission properties (Li *et al.*, 2015; Zhao *et al.*, 2015; Shimada *et al.*, 2015). As a typical example, Liu and co-workers recently reported a series of tetrahedral luminescent materials comprising SiAr4 cores (Tang *et al.*, 2014). They found that their fluorene derivatives were efficient blue-light-emitting materials and that Si-centered materials were superior with regard to film formation ability and quantum efficiency. A noteworthy feature of Si-centered tetrahedral materials is their high photoluminescence efficiency (nearly 100%) in the condensed state. In this paper, we report the crystal structure of the title compound, which is a precursor of these organosilicon compounds.

The complete molecule (Fig. 1) is generated by crystallographic twofold symmetry with the silicon atom lying on the rotation axis. The C—Si—C angles vary from 106.0 (1)° to 112.3 (1)°. The Si—C bond lengths of 1.866 (2) and 1.887 (2) Å are comparable with those in related structures (Ziller *et al.*, 1993; Yoshida *et al.*, 2005; Tsutsui & Sakamoto, 2003). The molecule adopts a V-shape and the dihedral angle between the benzene rings is 68.78 (7)°.

In the crystal, there are weak C—H···O hydrogen bonds (Fig. 2 and Table 1). Atom O2 accepts two such bonds, resulting in an aggregation of three molecules. The trimers

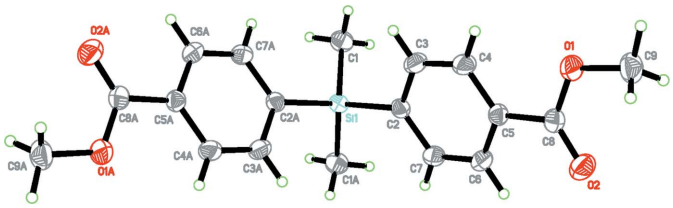


Figure 1
The molecular structure of the title compound.

are further linked to each other to form a double zigzag chain propagating along the [101] direction.

Synthesis and crystallization

The title compound was prepared according to a literature method (Tang *et al.*, 2007). Colourless blocks were prepared by recrystallization from a solvent mixture of dichloromethane and petroleum.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O2^{i}	0.96	2.62	3.511 (2)	154
C9—H9C···O2^{ii}	0.96	2.53	3.465 (3)	164

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+2$; (ii) $-x, y, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{20}O_4Si$
M_r	328.43
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	296
a, b, c (Å)	15.869 (3), 9.991 (2), 12.328 (3)
β (°)	117.79 (3)
V (Å^3)	1729.1 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.15
Crystal size (mm)	0.36 × 0.35 × 0.18
Data collection	
Diffractometer	Rigaku R-AXIS RAPID CCD
Absorption correction	Multi-scan (*RAPID-AUTO*; Rigaku, 1998)
T_{min}, T_{max}	0.948, 0.974
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8251, 1974, 1647
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.130, 1.06
No. of reflections	1974
No. of parameters	107
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.15

Computer programs: *RAPID-AUTO* (Rigaku, 1998), *CrystalStructure* (Rigaku/MSC and Rigaku, 2002), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008).

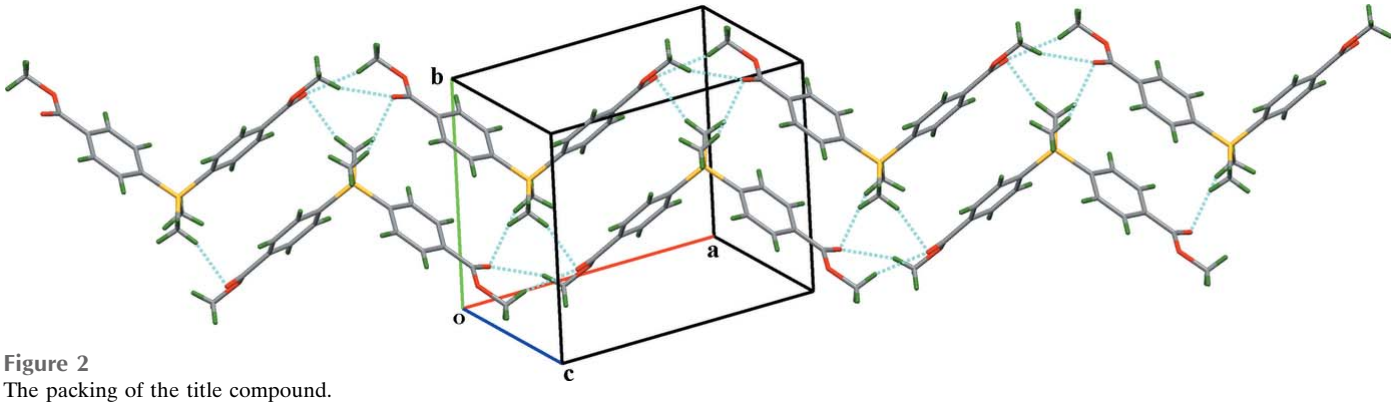

Figure 2
The packing of the title compound.

full crystallographic data

IUCrData (2016). **1**, x161887 [https://doi.org/10.1107/S2414314616018873]

Dimethyl 4,4′-(dimethylsilanediyl)dibenzoate

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Dimethyl 4,4′-(dimethylsilanediyl)dibenzoate

Crystal data

$C_{18}H_{20}O_4Si$
M_r = 328.43
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 15.869 (3) Å
b = 9.991 (2) Å
c = 12.328 (3) Å
β = 117.79 (3)°
V = 1729.1 (6) Å^3
Z = 4
$F(000)$ = 696
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7030 reflections
θ = 3.5–27.5°
μ = 0.15 mm^{-1}
T = 296 K
Block, colorless
0.36 × 0.35 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (RAPID-AUTO; Rigaku, 1998)
T_{min} = 0.948, T_{max} = 0.974
8251 measured reflections
1974 independent reflections
1647 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°, θ_{min} = 3.5°
h = −20→17
k = −12→12
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
S = 1.06
1974 reflections
107 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
$w = 1/[\sigma^2(F_o^2) + (0.0867P)^2 + 0.2156P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.54323 (13)	0.86561 (16)	1.16029 (15)	0.0529 (4)
H1A	0.4953	0.9300	1.1125	0.079*
H1B	0.5560	0.8090	1.1068	0.079*
H1C	0.6005	0.9113	1.2155	0.079*
C2	0.40084 (10)	0.64788 (13)	1.14363 (12)	0.0378 (3)
C3	0.41296 (11)	0.56740 (17)	1.05964 (16)	0.0522 (4)
H3	0.4702	0.5715	1.0562	0.063*
C4	0.34246 (11)	0.48152 (17)	0.98120 (15)	0.0513 (4)
H4	0.3521	0.4303	0.9249	0.062*
C5	0.25723 (9)	0.47202 (14)	0.98686 (13)	0.0400 (3)
C6	0.24381 (11)	0.54968 (16)	1.07046 (15)	0.0487 (4)
H6	0.1871	0.5434	1.0752	0.058*
C7	0.31455 (11)	0.63684 (16)	1.14723 (14)	0.0470 (4)
H7	0.3042	0.6891	1.2023	0.056*
C8	0.17949 (10)	0.37917 (15)	0.90651 (13)	0.0437 (4)
C9	0.12150 (13)	0.23692 (19)	0.73424 (17)	0.0623 (5)
H9A	0.1119	0.1643	0.7783	0.094*
H9B	0.1401	0.2020	0.6761	0.094*
H9C	0.0633	0.2866	0.6918	0.094*
O1	0.19538 (8)	0.32396 (12)	0.81941 (11)	0.0559 (3)
O2	0.10978 (9)	0.35693 (14)	0.91819 (12)	0.0667 (4)
Si1	0.5000	0.76151 (5)	1.2500	0.0378 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0619 (10)	0.0484 (8)	0.0529 (9)	−0.0023 (7)	0.0306 (8)	0.0075 (7)
C2	0.0385 (7)	0.0404 (7)	0.0336 (6)	0.0032 (5)	0.0160 (5)	0.0029 (5)
C3	0.0419 (8)	0.0650 (10)	0.0570 (9)	−0.0078 (7)	0.0292 (7)	−0.0168 (7)
C4	0.0490 (8)	0.0608 (9)	0.0518 (9)	−0.0065 (7)	0.0301 (7)	−0.0167 (7)
C5	0.0361 (7)	0.0427 (7)	0.0376 (7)	0.0020 (5)	0.0140 (5)	0.0029 (5)
C6	0.0376 (7)	0.0605 (9)	0.0518 (9)	−0.0015 (6)	0.0241 (7)	−0.0045 (7)
C7	0.0466 (8)	0.0536 (8)	0.0454 (8)	0.0013 (6)	0.0253 (7)	−0.0074 (6)
C8	0.0385 (7)	0.0453 (8)	0.0424 (8)	0.0021 (5)	0.0148 (6)	0.0022 (6)
C9	0.0545 (10)	0.0659 (11)	0.0546 (10)	−0.0100 (8)	0.0153 (8)	−0.0173 (8)
O1	0.0473 (6)	0.0658 (7)	0.0525 (7)	−0.0100 (5)	0.0215 (5)	−0.0177 (5)
O2	0.0516 (7)	0.0819 (9)	0.0720 (9)	−0.0195 (6)	0.0333 (6)	−0.0199 (7)
Si1	0.0413 (3)	0.0377 (3)	0.0356 (3)	0.000	0.0188 (2)	0.000

Geometric parameters (Å, º)

C1—Si1	1.8662 (16)	C5—C8	1.491 (2)
C1—H1A	0.9600	C6—C7	1.387 (2)
C1—H1B	0.9600	C6—H6	0.9300
C1—H1C	0.9600	C7—H7	0.9300
C2—C3	1.393 (2)	C8—O2	1.2009 (19)
C2—C7	1.395 (2)	C8—O1	1.3318 (19)
C2—Si1	1.8869 (15)	C9—O1	1.4440 (19)
C3—C4	1.383 (2)	C9—H9A	0.9600
C3—H3	0.9300	C9—H9B	0.9600
C4—C5	1.390 (2)	C9—H9C	0.9600
C4—H4	0.9300	Si1—C1^{i}	1.8662 (16)
C5—C6	1.383 (2)	Si1—C2^{i}	1.8869 (15)
Si1—C1—H1A	109.5	C7—C6—H6	119.9
Si1—C1—H1B	109.5	C6—C7—C2	121.47 (14)
H1A—C1—H1B	109.5	C6—C7—H7	119.3
Si1—C1—H1C	109.5	C2—C7—H7	119.3
H1A—C1—H1C	109.5	O2—C8—O1	123.46 (14)
H1B—C1—H1C	109.5	O2—C8—C5	123.95 (14)
C3—C2—C7	117.11 (13)	O1—C8—C5	112.59 (13)
C3—C2—Si1	120.29 (11)	O1—C9—H9A	109.5
C7—C2—Si1	122.57 (11)	O1—C9—H9B	109.5
C4—C3—C2	122.03 (14)	H9A—C9—H9B	109.5
C4—C3—H3	119.0	O1—C9—H9C	109.5
C2—C3—H3	119.0	H9A—C9—H9C	109.5
C3—C4—C5	119.74 (14)	H9B—C9—H9C	109.5
C3—C4—H4	120.1	C8—O1—C9	116.16 (13)
C5—C4—H4	120.1	C1^{i}—Si1—C1	112.26 (11)
C6—C5—C4	119.38 (14)	C1^{i}—Si1—C2	109.23 (7)
C6—C5—C8	118.47 (13)	C1—Si1—C2	109.96 (7)
C4—C5—C8	122.15 (14)	C1^{i}—Si1—C2^{i}	109.96 (7)
C5—C6—C7	120.25 (14)	C1—Si1—C2^{i}	109.23 (7)
C5—C6—H6	119.9	C2—Si1—C2^{i}	106.02 (9)
C7—C2—C3—C4	1.1 (2)	C4—C5—C8—O2	171.12 (16)
Si1—C2—C3—C4	179.15 (13)	C6—C5—C8—O1	171.45 (13)
C2—C3—C4—C5	−1.3 (3)	C4—C5—C8—O1	−9.3 (2)
C3—C4—C5—C6	0.5 (2)	O2—C8—O1—C9	2.1 (2)
C3—C4—C5—C8	−178.70 (15)	C5—C8—O1—C9	−177.48 (13)
C4—C5—C6—C7	0.5 (2)	C3—C2—Si1—C1^{i}	174.94 (12)
C8—C5—C6—C7	179.71 (14)	C7—C2—Si1—C1^{i}	−7.11 (14)
C5—C6—C7—C2	−0.7 (2)	C3—C2—Si1—C1	51.33 (14)
C3—C2—C7—C6	−0.1 (2)	C7—C2—Si1—C1	−130.71 (13)

Si1—C2—C7—C6	−178.09 (12)	C3—C2—Si1—C2^{i}	−66.63 (12)
C6—C5—C8—O2	−8.1 (2)	C7—C2—Si1—C2^{i}	111.33 (13)

Symmetry code: (i) −*x*+1, *y*, −*z*+5/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2^{ii}	0.96	2.62	3.511 (2)	154
C9—H9*C*···O2^{iii}	0.96	2.53	3.465 (3)	164

Symmetry codes: (ii) −*x*+1/2, −*y*+3/2, −*z*+2; (iii) −*x*, *y*, −*z*+3/2.