

Crystal data

C₁₆H₁₀Cl₂
M_r = 273.16
 Monoclinic
*P*2₁/*c*
a = 11.217 (1) Å
b = 8.1008 (3) Å
c = 14.4403 (8) Å
 β = 104.34 (1)°
V = 1271.3 (3) Å³
Z = 4
D_x = 1.427 Mg m⁻³
D_m not measured

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\theta/2\theta$ scans
 Absorption correction:
 ψ scan (North, Phillips
 & Mathews, 1968)
T_{min} = 0.36, *T_{max}* = 0.47
 2841 measured reflections
 2532 independent reflections

Refinement

Refinement on *F*²
R = 0.039
wR = 0.059
S = 2.301
 2326 reflections
 204 parameters
 All H atoms refined
 $w = 4F_o^2/[\sigma^2(F_o^2) + 0.0009F_o^4]$
 $(\Delta/\sigma)_{\max} = 0.023$

Cu *K*α radiation
 $\lambda = 1.54184$ Å
 Cell parameters from 25
 reflections
 $\theta = 19\text{--}29^\circ$
 $\mu = 4.47$ mm⁻¹
T = 297 K
 Prism
 0.30 × 0.22 × 0.17 mm
 Colorless

2326 reflections with
 $I > \sigma(I)$
R_{int} = 0.012
 $\theta_{\max} = 75^\circ$
 $h = 0 \rightarrow 14$
 $k = 0 \rightarrow 9$
 $l = -18 \rightarrow 17$
 3 standard reflections
 frequency: 120 min
 intensity decay: 3.6%

$\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.14$ e Å⁻³
 Extinction correction:
 isotropic (Zachariasen,
 1963)
 Extinction coefficient:
 $3.9(2) \times 10^{-6}$
 Scattering factors from *Inter-*
national Tables for X-ray
Crystallography (Vol. IV)

Table 1. Selected geometric parameters (Å, °)

C11—C1	1.741 (2)	C10—C14	1.520 (3)
C12—C8	1.744 (2)	C10—C16	1.529 (2)
C9—C11	1.528 (2)	C11—C12	1.397 (2)
C9—C13	1.520 (3)	C13—C14	1.399 (2)
C9—C15	1.528 (2)	C15—C16	1.312 (3)
C10—C12	1.521 (2)		
C11—C9—C13	104.5 (1)	C10—C12—C11	112.5 (1)
C11—C9—C15	105.8 (1)	C8—C13—C9	128.4 (2)
C13—C9—C15	105.6 (1)	C8—C13—C14	118.7 (2)
C12—C10—C14	105.4 (1)	C9—C13—C14	112.9 (2)
C14—C10—C16	106.0 (1)	C5—C14—C10	126.7 (2)
C1—C11—C9	128.5 (2)	C5—C14—C13	120.9 (2)
C1—C11—C12	118.7 (2)	C10—C14—C13	112.5 (2)
C9—C11—C12	112.8 (1)	C9—C15—C16	114.7 (2)
C4—C12—C10	126.5 (2)	C10—C16—C15	113.9 (1)
C4—C12—C11	121.1 (1)		

C—H distances are in the range 0.86 (2)–0.99 (2) Å, while *B_{iso}* values for H atoms are in the range 3.3 (4)–7.1 (7) Å².

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980). Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *CIFIN* in *MolEN*.

The purchase of the diffractometer was made possible by a National Science Foundation chemical instrumentation grant, which we gratefully acknowledge.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1334). Services for accessing these data are described at the back of the journal.

References

- Benites, M. R., Fronczek, F. R. & Maverick, A. W. (1996). *J. Organomet. Chem.* **516**, 17–24.
 Benites, M. R., Maverick, A. W. & Fronczek, F. R. (1996). *Acta Cryst.* **C52**, 647–648.
 Dellaca, R. J., Penfold, B. R. & Robinson, W. T. (1969). *Acta Cryst.* **B25**, 1589–1594.
 Desvergne, J.-P., Gaultier, J. & Hauw, C. (1970). *C. R. Acad. Sci. Ser. C*, **271**, 1298–1300.
 Enraf–Nonius (1977). *CAD-4 Operations Manual*. Enraf–Nonius, Delft, The Netherlands.
 Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 Oliver, S. W., Fallon, G. D. & Smith, T. D. (1986). *Acta Cryst.* **C42**, 1047–1048.
 Paquette, L. A., Moerck, R. E., Harirchian, B. & Magnus, P. D. (1978). *J. Am. Chem. Soc.* **100**, 1597–1599.
 Pokkuluri, P. R., Scheffer, J. R. & Trotter, J. (1993). *Acta Cryst.* **B49**, 107–116.
 Rettig, S. J., Scheffer, J. R., Trotter, J. & Yang, J. (1995). *Acta Cryst.* **C51**, 2685–2687.
 Trotter, J. & Wireko, F. C. (1990). *Acta Cryst.* **C46**, 103–106.
 Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1997). **C53**, 1335–1337

1,1,1,4,4,4-Hexachloro-1,4-disilabutane

NORBERT W. MITZEL, JÜRGEN RIEDE AND HUBERT SCHMIDBAUR

Anorganisch-Chemisches Institut, Technische Universität München, Lichtenbergstrasse 4, D-85747 Garching, Germany. E-mail: n.w.mitzel@lrz.tu-muenchen.de

(Received 26 March 1997; accepted 17 April 1997)

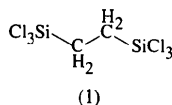
Abstract

Molecules of the title compound, C₂H₄Cl₆Si₂, have a crystallographic centre of inversion and adopt a *trans* conformation in the crystal. Important bond lengths

are C—C 1.536 (3), Si—C 1.8469 (15), and Si—Cl 2.0229 (6), 2.0225 (6) and 2.0283 (6) Å. The Cl atom *trans* to the C—C bond is tilted towards the C atom. There are no important intermolecular interactions.

Comment

We have recently reported the gas-phase and solid-state structures of 1,4-disilabutane and 1,5-disilapentane (Mitzel, Smart, Blake, Robertson & Rankin, 1996) and the crystal structure of 1,4-dibromo-1,4-disilabutane (Mitzel, Riede & Schmidbaur, 1996). Simple carbosilane molecules and their halogen derivatives are currently being probed as single-source precursors for the epitaxial growth of β -SiC layers by chemical vapour deposition (Kunstmann *et al.*, 1995). 1,4-Disilabutane and 1,5-disilapentane and their α,ω -dibromo derivatives tend to eliminate ethene and propene, respectively, in the low-temperature regime of the deposition experiments with formation of Si layers, whereas in the high-temperature regime SiC is formed without loss of the carbon units. These observations led us to determine the molecular structures of these and related compounds in order to provide a structural basis for a discussion of the decomposition chemistry. An investigation of methyltrichlorosilane and 1,1,1,4,4,4-hexachloro-1,4-disilabutane, (1), as precursors has now been commenced and the molecular structures of these compounds are being determined in order to make a parallel comparison with the structure and decomposition chemistry of the corresponding carbosilanes and their bromo derivatives.



In the crystal, 1,1,1,4,4,4-hexachloro-1,4-disilabutane has a crystallographic centre of inversion (Fig. 1). 1,4-Dibromo-1,4-disilabutane (Mitzel, Riede & Schmidbaur, 1996), as well as other open-chain molecules with a symmetrical substitution pattern at the Si—C—C—Si unit that have been studied by crystallographic methods (Shibayeva, Atovmyan, Rozenberg & Stryukov, 1983; Tacke, Niedner, Frohnecke, Ernst & Sheldrick, 1980; Ovchinnikov, Shklover, Struchkov, Polyakov & Guselnikov, 1985), also have crystallographic inversion centres. Compound (1) also has almost perfect $2/m$ symmetry, reflecting its ideal all-staggered conformation, which is not distorted by intermolecular forces in the crystal.

The C—C distance in (1) is very similar to those in the related compounds mentioned above, as is the Si—C distance. The C—C—Si angle also is equal to that in 1,4-dibromo-1,4-disilabutane [113.6 (4)°] within the limits of error.

The SiCl₃ group is tilted in a way that two C—Si—Cl angles are larger than the third [Cl2] by about

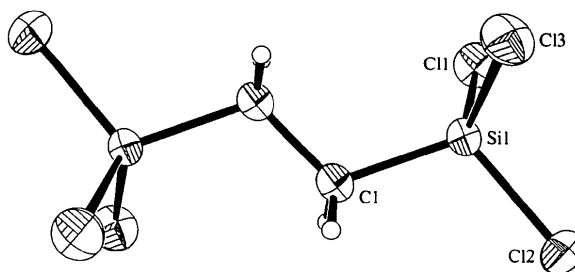


Fig. 1. A view of one molecule of (1) with the atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces and H atoms are shown as spheres of arbitrary radii. The crystallographic centre of inversion is located in the middle of the C—C bond.

1.6°. The two Cl atoms Cl1 and Cl3, which are bent away from the ethanediyl bridge, have slightly shorter Si—Cl distances (2.023 Å on average) than the Si—Cl2 distance [2.0283 (6) Å].

There are no important intermolecular interactions. The shortest such Cl···Cl non-bonded distances are 3.52 Å, which is about the sum of the van der Waals radii (3.62 Å; Emsley, 1991). The shortest intermolecular Si···Cl distance is 4.30 Å, indicating the absence of any Cl···Si donor–acceptor interaction comparable with the Si···Br interactions observed in 1,4-dibromo-1,4-disilabutane.

Experimental

1,1,1,4,4,4-Hexachloro-1,4-disilabutane, (1), was prepared by a hydrosilylation reaction from vinyltrichlorosilane and trichlorosilane (Mitzel *et al.*, 1997). Crystals were grown by slowly cooling the melt to 290 K. For the X-ray diffraction experiment, one of these was transferred into a glass capillary and sealed under an atmosphere of dry argon at 223 K.

Crystal data

C₂H₄Cl₆Si₂

$M_r = 296.93$

Monoclinic

$P2_1/n$

$a = 6.231 (1) \text{ \AA}$

$b = 9.447 (1) \text{ \AA}$

$c = 9.394 (1) \text{ \AA}$

$\beta = 92.320 (10)^\circ$

$V = 552.52 (12) \text{ \AA}^3$

$Z = 2$

$D_r = 1.785 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 96 reflections

$\theta = 18\text{--}21^\circ$

$\mu = 1.706 \text{ mm}^{-1}$

$T = 205 (2) \text{ K}$

Block

$0.35 \times 0.35 \times 0.30 \text{ mm}$

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

ω scans

Absorption correction:

ψ scans (North, Phillips

& Mathews, 1968)

$T_{\min} = 0.533$, $T_{\max} = 0.599$

1085 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\text{max}} = 26.94^\circ$

$h = -7 \rightarrow 7$

$k = 0 \rightarrow 12$

$l = 0 \rightarrow 11$

1259 measured reflections
1190 independent reflections

3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.062$
 $S = 1.120$
1181 reflections
55 parameters
All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0267P)^2 + 0.1264P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.310 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.171 \text{ e } \text{\AA}^{-3}$
Extinction correction:
SHELXL93 (Sheldrick, 1993)
Extinction coefficient:
0.058 (3)
Scattering factors from
International Tables for Crystallography (Vol. C)

Acta Cryst. (1997). **C53**, 1337–1341

1-Aryl-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole Derivatives

GIUSEPPE BRUNO,^a ALBA CHIMIRRI,^b ANNA MARIA MONFORTE,^b FRANCESCO NICOLÓ^a AND ROSARIO SCOPELLITI^a

^a*Dipartimento di Chimica Inorganica, Chimica Analitica e Chimica Fisica, Università di Messina, 98166 Vill. Sant'Agata, Messina, Italy, and* ^b*Dipartimento Farmaco-Chimico, Università di Messina, 98168 Viale Annunziata, Messina, Italy. E-mail: bruno@medif0.unime.it*

(Received 31 January 1997; accepted 1 April 1997)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Si1—C1	1.8469 (15)	Si1—Cl2	2.0283 (6)
C1—C1 ¹	1.536 (3)	Si1—Cl3	2.0225 (6)
Si1—Cl1	2.0229 (6)		
C1—Si1—Cl1	111.44 (6)	Cl1—Si1—Cl3	107.17 (3)
C1—Si1—Cl2	109.80 (5)	Cl2—Si1—Cl3	108.50 (3)
C1—Si1—Cl3	111.27 (6)	C1 ¹ —C1—Si1	113.93 (13)
Cl1—Si1—Cl2	108.55 (3)		
Cl1—Si1—Cl1—C1 ¹	-61.7 (2)	Cl3—Si1—Cl1—C1 ¹	57.8 (2)
Cl2—Si1—Cl1—C1 ¹	177.96 (14)		

Symmetry code: (i) $-x, 2 - y, 1 - z$.

Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1992). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1006). Services for accessing these data are described at the back of the journal.

References

- Emsley, J. (1991). In *The Elements*. Oxford: Clarendon Press.
- Kunstmann, T., Angerer, H., Knecht, J., Vepfek, S., Mitzel, N. W. & Schmidbauer, H. (1995). *Chem. Mater.* **7**, 1675–1679.
- Mitzel, N. W., Riede, J. & Schmidbauer, H. (1996). *Acta Cryst.* **C52**, 980–982.
- Mitzel, N. W., Schmidbauer, H., Rankin, D. W. H., Smart, B. A., Hofmann, M. & Schleyer, P. v. R. (1997). *Inorg. Chem.* In the press.
- Mitzel, N. W., Smart, B. A., Blake, A. J., Robertson, H. E. & Rankin, D. W. H. (1996). *J. Phys. Chem.* **100**, 12280–12287.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Ovchinnikov, Y. E., Shklover, V. E., Struchkov, Y. T., Polyakov, Y. P. & Gusevnikov, L. E. (1985). *Acta Cryst.* **C41**, 1055–1057.
- Sheldrick, G. M. (1992). *SHELXTL/PC*. Version 4.3. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Shibayeva, R. P., Atovmyan, L. O., Rozenberg, L. P. & Stryukov, V. B. (1983). *Dokl. Akad. Nauk SSSR*, **210**, 833–836.
- Tacke, R., Niedner, R., Frohnecke, J., Ernst, L. & Sheldrick, W. S. (1980). *Justus Liebig's Ann. Chem.* pp. 1859–1876.

Abstract

The structural features of some 1-aryl-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazoles active against human immunodeficiency virus (HIV) are reported. The diffractometric analysis reveals that the title compounds, 1-(2,4-difluorophenyl)-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole, $\text{C}_{15}\text{H}_{10}\text{F}_2\text{N}_2\text{S}$, 1-(2,6-dichlorophenyl)-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole, $\text{C}_{15}\text{H}_{10}\text{Cl}_2\text{N}_2\text{S}$, and 1-(2-chloro-6-fluorophenyl)-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole, $\text{C}_{15}\text{H}_{10}\text{ClFN}_2\text{S}$, all adopt a butterfly-like conformation which appears to be particularly important for the inhibition of HIV-1 reverse transcriptase (RT), an enzyme which plays a key role in the viral life-cycle.

Comment

In previous papers, we reported the synthesis and evaluation of anti-HIV activity of a series of 1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole derivatives (Chimirri, Grasso, Monforte, Monforte & Zappalá, 1991; Monforte *et al.*, 1993; Bruno, Chimirri *et al.*, 1996; Chimirri *et al.*, 1996; Bruno, Monforte, Nicoló & Scopelliti, 1996). Some of the studied compounds exhibited significant activity (Chimirri *et al.*, 1991, 1996) and in particular the 1-(2,6-difluorophenyl)- derivative (TBZ) was found to be a highly potent non-nucleoside HIV-1 reverse transcriptase (RT) inhibitor (NNRTI) (Schultz *et al.*, 1992; Buckheit *et al.*, 1993; Bruno, Chimirri *et al.*, 1996). It was recently ascertained (Scafer *et al.*, 1993; Ding *et al.*, 1995) that some structural features, such as two systems arranged in a butterfly-like conformation and a hydrogen-bond acceptor in an appropriate position, are necessary for the inhibition of the RT enzyme. On this basis, it is very important to determine the geometry of thiazolobenzimidazole derivatives, a new class of anti-HIV agents, in order to clarify which molecular requirements are common to NNRTIs. As part of a broader structural and pharmacological study of these compounds, we report here the results of the X-ray