

Hughes, J., Smith, T. W., Kosterlitz, H. W., Fothergill, L. A., Morgan, B. A. & Morris, H. R. (1975). *Nature (London)*, **258**, 577–579.  
 Johnson, C. K. (1976). *ORTEPII*. Report ORNI-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 Prangé, P. T. & Pascard, C. (1979). *Acta Cryst.* **B35**, 1812–1819.  
 Schiller, P. W. (1984). In *The Peptides, Analysis, Synthesis, Biology*. Vol. 6, edited by S. Udenfriend & J. Meienhofer. London: Academic Press.  
 Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Version 4.1. 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.  
 Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Smith, G. D. & Griffin, J. F. (1978). *Science*, **199**, 1214–1216.  
 Vaughan, J. R. Jr & Osato, R. L. (1952). *J. Am. Chem. Soc.* **74**, 676–678.

*Acta Cryst.* (1996). **C52**, 978–980

#### 4-*tert*-Butyloxycarbonyl-6(*S*)-(hydroxymethyl)-3(*S*)-(1-methylethyl)piperazin-2-one

THOMAS KOLTER,<sup>a</sup> FRANK RÜBSAM,<sup>a</sup> ATHANASSIOS GIANNIS<sup>a</sup> AND MARTIN NIEGER<sup>b</sup>

<sup>a</sup>*Institut für Organische Chemie und Biochemie der Universität, Gerhard-Domagk-Str. 1, 53121 Bonn, Germany,* and <sup>b</sup>*Institut für Anorganische Chemie der Universität, Gerhard-Domagk-Str. 1, 53121 Bonn, Germany. E-mail: giannis@uni-bonn.de*

(Received 21 June 1995; accepted 25 September 1995)

#### Abstract

The six-membered ring of the title ester [IUPAC name: *tert*-butyl 5-(hydroxymethyl)-2-(1-methylethyl)-3-oxo-piperazine-1-carboxylate],  $C_{13}H_{24}N_2O_4$ , adopts a distorted half-chair configuration with C5 lying out of plane. Hydrogen bonds are formed between the OH group and the protecting carbonyl group, and between NH and the piperazine oxo group.

#### Comment

Substituted piperazinones are of interest in the development of peptidomimetics, *i.e.* low-molecular weight compounds that can replace peptides in their interaction with receptors (Giannis & Kolter, 1993). The piperazinone ring can serve as a rigid template exposing substituents in a conformation resembling the side chains of a parent peptide in its receptor-bound conformation (Giannis & Kolter, 1993). Little is known about

the three-dimensional structures of these compounds (Michel, Evrard & Norberg, 1987). We report here the structure of the title compound, (I).

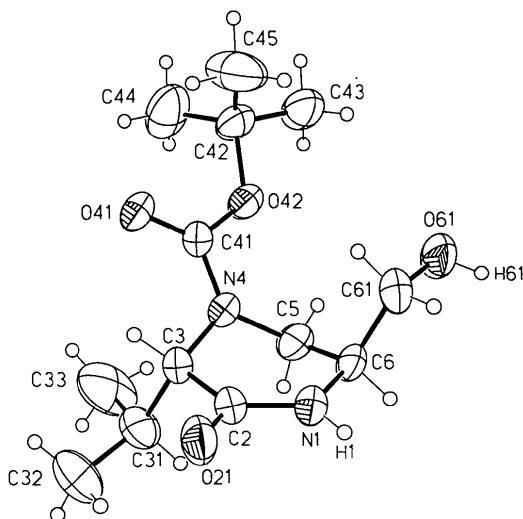
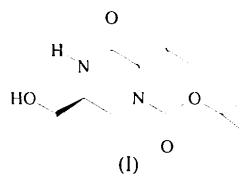


Fig. 1. Perspective drawing of (I) showing the crystallographic numbering scheme. Non-H atoms are shown with 30% probability ellipsoids.

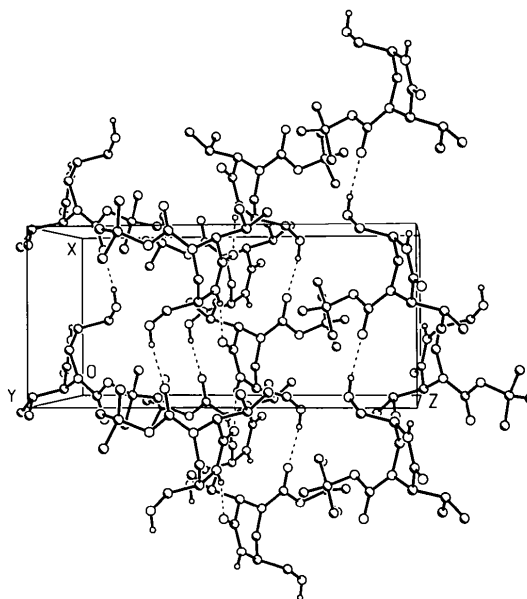


Fig. 2. A view of the unit cell showing intermolecular hydrogen bonding.

In the crystal the molecule adopts a distorted half-chair conformation with C5 lying out of the plane. Fig. 2 shows two intermolecular hydrogen bonds, O61—H61···O41 and N1—H1···O21. Interestingly, the urethane bond (N4—C41) is found to have an antiperiplanar conformation (see Table 2). All other angles and distances are as expected.

The absolute configuration of (I) has not been determined, but was assigned to agree with the known chirality as established by the synthesis, because there were no anomalous-dispersion effects.

## Experimental

The title compound was prepared by the previously published method (Kolter, Dahl & Giannis, 1995) and recrystallized from diethyl ether.

### Crystal data

C<sub>13</sub>H<sub>24</sub>N<sub>2</sub>O<sub>4</sub>  
*M<sub>r</sub>* = 272.34  
 Orthorhombic  
*P*2<sub>1</sub>2<sub>1</sub>  
*a* = 7.890 (1) Å  
*b* = 11.752 (1) Å  
*c* = 17.028 (1) Å  
*V* = 1578.9 (3) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.146 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

### Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 2θ/ω scans  
 Absorption correction:  
 none  
 2328 measured reflections  
 2019 independent reflections  
 1545 observed reflections  
 [*I* > 2σ(*I*)]  
*R*<sub>int</sub> = 0.0575  
 θ<sub>max</sub> = 59.96°

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.0674  
*wR*(*F*<sup>2</sup>) = 0.1845  
*S* = 1.061  
 2019 reflections  
 184 parameters  
 H atoms riding, except those  
 on O and N which refined  
 freely  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.128*P*)<sup>2</sup> +  
 0.6321*P*]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001

Cu *K*α radiation  
 λ = 1.54178 Å  
 Cell parameters from 25  
 reflections  
 θ = 40–46°  
 μ = 0.696 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Plate  
 0.80 × 0.60 × 0.30 mm  
 Yellowish

*h* = -8 → 7  
*k* = 0 → 13  
*l* = -19 → 0  
 2 orientation and 3 intensity  
 standard reflections,  
 monitored every 200  
 reflections for orientation  
 and every 120 min  
 for intensity; intensity  
 variation ±1%

Δρ<sub>max</sub> = 0.194 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.157 e Å<sup>-3</sup>  
 Extinction correction:  
*SHELXL93* (Sheldrick,  
 1993)  
 Extinction coefficient:  
 0.0151 (22)  
 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<sup>2</sup>)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
N1	0.1426 (5)	0.6207 (4)	0.5218 (3)	0.083 (1)
C2	0.3045 (6)	0.6436 (6)	0.5054 (3)	0.077 (2)
C3	0.4322 (6)	0.5472 (5)	0.5123 (3)	0.073 (1)
N4	0.3672 (5)	0.4589 (4)	0.5648 (2)	0.072 (1)
C5	0.1964 (7)	0.4200 (5)	0.5462 (3)	0.084 (2)
C6	0.0743 (6)	0.5178 (5)	0.5564 (3)	0.079 (2)
O21	0.3495 (5)	0.7398 (4)	0.4841 (3)	0.095 (1)
C31	0.4783 (9)	0.5015 (6)	0.4302 (4)	0.103 (2)
C32	0.5757 (12)	0.5847 (8)	0.3835 (4)	0.148 (3)
C33	0.5768 (16)	0.3889 (8)	0.4384 (5)	0.173 (4)
C41	0.4637 (6)	0.4195 (5)	0.6233 (3)	0.074 (1)
O41	0.6045 (4)	0.4563 (3)	0.6382 (2)	0.091 (1)
O42	0.3830 (4)	0.3398 (3)	0.6646 (2)	0.088 (1)
C42	0.4521 (8)	0.2940 (5)	0.7372 (4)	0.095 (2)
C43	0.3088 (10)	0.2149 (7)	0.7639 (5)	0.122 (3)
C44	0.6131 (11)	0.2300 (8)	0.7237 (7)	0.154 (4)
C45	0.4745 (14)	0.3889 (8)	0.7946 (4)	0.156 (4)
C61	0.0274 (7)	0.5377 (6)	0.6408 (3)	0.101 (2)
O61	-0.0537 (5)	0.4437 (5)	0.6743 (3)	0.127 (2)
H1	0.068 (6)	0.675 (4)	0.515 (4)	0.100
H61	-0.156 (4)	0.437 (9)	0.669 (6)	0.190

Table 2. Selected torsion angles (°)

C6—N1—C2—C3	9.5 (8)	C2—N1—C6—C5	-20.7 (7)
C2—C3—N4—C5	51.1 (6)	N4—C5—C6—N1	44.6 (6)
C3—N4—C5—C6	-63.8 (5)	C3—N4—C41—O42	-180.0 (4)

Table 3. Hydrogen-bonding geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O61—H61···O41 <sup>1</sup>	0.82 (3)	1.98 (4)	2.770 (5)	165 (11)
N1—H1···O21 <sup>11</sup>	0.88 (3)	2.00 (4)	2.837 (6)	161 (6)

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) *x* - ½, ½ - *y*, 1 - *z*.

The absolute configuration of (I) was assigned to agree with the known chirality at C3 and C6, as established by the synthesis of the compound. The absolute structure could not be determined reliably [Flack (1983) parameter = -0.0 (7)].

Data collection: *CAD-4-Express* (Enraf–Nonius, 1989). Cell refinement: *CAD-4-Express*. Data reduction: locally modified *XCAD4* (Sheldrick, 1992). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

We thank the Deutsche Forschungsgemeinschaft for financial support (Gi 204/1-2).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1074). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Enraf–Nonius (1989). *CAD-4-Express*. Enraf–Nonius, Delft, The Netherlands.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Giannis, A. & Kolter, T. (1993). *Angew. Chem.* **105**, 1303–1326; *Angew. Chem. Int. Ed. Engl.* **32**, 1244–1267.  
 Kolter, T., Dahl, C. & Giannis, A. (1995). *Liebigs Ann. Chem.* pp. 625–629.

- Michel, A., Evrard, G. & Norberg, B. (1987). *Can. J. Chem.* **65**, 1308–1312.
- Sheldrick, G. M. (1989). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1992). *XCAD4*. University of Göttingen, Germany. Modified by H. Harms, University of Marburg, Germany.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

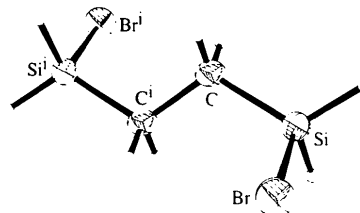


Fig. 1. A view of one molecule of (1) with the atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces and H atoms are shown as spheres of arbitrary radii.

*Acta Cryst.* (1996). **C52**, 980–982

## 1,4-Dibromo-1,4-disilabutane

NORBERT W. MITZEL, JÜRGEN RIEDE AND HUBERT SCHMIDBAUR

*Anorganisch-chemisches Institut der Technischen Universität München, Lichtenbergstrasse 4, D-85747 Garching, Germany*

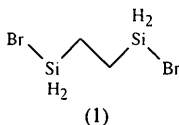
(Received 9 August 1995; accepted 20 October 1995)

### Abstract

Molecules of the title compound, C<sub>2</sub>H<sub>8</sub>Br<sub>2</sub>Si<sub>2</sub>, have a crystallographic centre of inversion. The Si—C—C—Si unit is thus planar and has a *trans* conformation. The Br—Si—C—C moieties have almost ideal *gauche* conformations with Si—Br bond lengths of 2.2362 (12) Å.

### Comment

In the course of studies on cyclic silicon–nitrogen compounds (Mitzel, Bissinger, Riede, Dreihäupl & Schmidbaur, 1993; Mitzel, Riede, Schier, Angermaier & Schmidbaur, 1995), 1,4-dibromo-1,4-disilabutane, (1), served as a synthon for the H<sub>2</sub>Si—CH<sub>2</sub>—CH<sub>2</sub>—SiH<sub>2</sub> unit. Structural information on simple molecules containing this structural element appeared desirable in order to obtain a basis for an understanding of the decomposition pathways of such molecules, which are probed as single-source precursors for the chemical vapour deposition (CVD) of epitaxially grown β-SiC (Kunstmann *et al.*, 1995).



In the crystal, the molecule of (1) has a crystallographic centre of inversion (Fig. 1). Similar results were obtained for all other open chain molecules with a symmetrical substitution pattern at the Si—C—C—Si unit studied to date (Shibayeva, Atovmyan, Rozenberg & Stryukov, 1983; Tacke, Niedner, Frohnecke, Ernst

& Sheldrick, 1980; Ovchinnikov, Shklover, Struchkov, Polyakov & Guselnikov, 1985).

The C—C and Si—C distances, as well as the angle Si—C—C, are very similar to those reported for the reference molecules. The Si—Br distance [2.2362 (12) Å] fits well into the range established by only four examples of Si—Br compounds with four-coordinate Si atoms studied in the solid state so far [according to a search in the Cambridge Crystal Structure Database and the Inorganic Crystal Structure Database (Gurkova, Gusev, Sharapov, Gar & Alexeev, 1979; Kratky, Hengge, Stüger & Rheingold, 1985; Schubert & Steib, 1982)].

The most interesting structural feature of (1) is the *gauche* conformation of each Br—Si—C—C unit. The corresponding torsion angle [−61.7 (5)°] is close to the ideal value for the *gauche* conformation of interpenetrating tetrahedra. The overall *gauche-trans-gauche* conformation has also been found by Ovchinnikov *et al.* (1985) in 2,5-dichloro-2,5-dimethyl-2,5-disilohexane and by Tacke *et al.* (1980) for ([Me<sub>3</sub>N(CH<sub>2</sub>)<sub>3</sub>]Me<sub>2</sub>SiCH<sub>2</sub>)<sub>2</sub>I<sub>2</sub>, taking the most electronegative substituent at silicon as a reference. This conformational preference appears to be an inherent characteristic of this class of compounds, although in the present case it may also be affected by Si···Br and Br···Br intermolecular interactions. There are some weak intermolecular interactions as indicated by the shortest lattice contacts Si···Br [3.741 (1) Å] and Br···Br [3.839 (1) Å], which are very close to the corresponding sums of the van der Waals radii (Si···Br

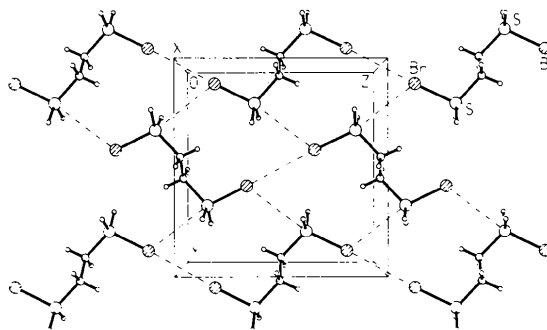


Fig. 2. Packing plot showing the Si···Br and Br···Br contacts in crystals of (1), and the unit cell.