Fig. 2. Unit cell of droperidol viewed along the *b* axis (the hydrogen bonds are shown with broken lines).

Table 4. *Hydrogen-bond distances* (Å) *and angles* (°)

$A-B\cdots C$	$\angle A-B\cdots C$	$A-B$	$B\cdots C$	$A\cdots C$
O(29)–H(29A)···O(21)[i]	158·0	1·03	1·89	2·880 (5)
O(29)–H(29B)···N(14)	171·0	1·06	1·73	2·785 (4)
O(30)–H(30A)···O(1)[ii]	165·7	0·89	2·17	3·044 (5)
O(30)–H(30B)···O(29)	171·9	1·17	1·81	2·982 (5)
N(8)[iii]– H(8)···O(29)	162·9	1·00	1·87	2·847 (5)

Symmetry code

(i) $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$ (iii) $1+x, y, z$

(ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$

bonds with N(14) and a screw-axis-related (*y* direction) O(21) atom, and as an acceptor with the second water molecule O(30) and a translation-equivalent (*x* direction) imidazolic NH group.

The second water molecule O(30) forms, in addition to the hydrogen bond with O(29) mentioned above, a second hydrogen bond with a symmetry-related (screw axis) O(1).

Table 4 gives relevant hydrogen-bond distances and angles.

References

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A24, 321–324.

DECLERCQ, J. P., GERMAIN, G. & KOCH, M. H. J. (1973). *Acta Cryst.* B29, 2311–2313.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.

STEWART, J. M., MACHIN, P. A., DICKINSON, C., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY 76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

3-|Bis(trimethylsilyl)aminofluoro(methyl)silyl|-2,2,4,4,6,8,8-heptamethyl-5,7-bis(trimethylsilyl)-1,3,5,7-tetraaza-2,4,6,8-tetrasilabicyclo[4.2.0]octane

BY WILLIAM CLEGG

Anorganisch Chemisches Institut der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

Abstract. $C_{20}H_{60}FN_5Si_9$, $M_r = 642\cdot6$, monoclinic, $P2_1/c$, $a = 14\cdot107$ (4), $b = 9\cdot468$ (3), $c = 29\cdot874$ (8) Å, $\beta = 102\cdot41$ (2)°, $U = 3896\cdot9$ Å³, $Z = 4$, $D_x = 1\cdot095$ Mg m⁻³; final $R = 0\cdot061$ for 4889 reflexions. The molecule contains an almost planar Si_2N_2 ring fused to a Si_3N_3 ring which approximates to a boat conformation.

Introduction. This structure was determined as part of a study of Si–N ring compounds. Air-stable, colour-

less crystals were obtained from a solution in light petrol. The preparation has been described (Skoda, Klingebiel & Meller, 1980).

Intensities were measured with graphite-monochromated Mo *Kα* radiation ($\lambda = 0\cdot71069$ Å) and a Stoe–Siemens AED diffractometer by a profile-fitting procedure (Clegg, 1981). 4889 reflexions with $7 \le 2\theta \le 60°$ and $I \ge 2\sigma(I)$ were collected. No corrections were made for absorption ($\mu = 0\cdot32$ mm⁻¹). Cell dimensions were obtained by least squares from the setting angles of 24 reflexions with $20 \le 2\theta \le 25°$.

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic thermal parameters* (Å² ×10⁴)

	x	y	z	U
Si(1)	4329 (1)	1552 (1)	1207 (1)	647 (5)
C(11)	3384 (3)	1872 (6)	1556 (2)	1002 (24)
C(12)	4645 (4)	−351 (4)	1289 (2)	921 (23)
C(13)	3780 (4)	1934 (6)	604 (2)	1165 (28)
N(1)	5308 (2)	2644 (3)	1402 (1)	540 (12)
Si(2)	5329 (1)	4129 (1)	1748 (1)	655 (5)
C(21)	4445 (3)	5531 (5)	1497 (2)	1079 (27)
C(22)	5219 (4)	3774 (6)	2346 (2)	1194 (28)
N(2)	6532 (2)	4397 (3)	1724 (1)	492 (11)
Si(3)	6544 (1)	2848 (1)	1410 (1)	483 (4)
C(31)	7280 (3)	1416 (4)	1730 (1)	764 (20)
N(3)	6980 (2)	3306 (3)	936 (1)	467 (11)
Si(4)	6993 (1)	2128 (1)	484 (1)	646 (5)
C(41)	6293 (4)	2820 (6)	−66 (1)	1002 (23)
C(42)	8240 (4)	1676 (6)	430 (2)	1018 (24)
C(43)	6424 (4)	420 (5)	593 (2)	1073 (26)
Si(5)	7316 (1)	5092 (1)	907 (1)	474 (4)
C(51)	8045 (3)	5312 (5)	459 (1)	759 (19)
C(52)	6224 (3)	6209 (4)	720 (1)	705 (18)
N(4)	8016 (2)	5716 (3)	1436 (1)	452 (11)
Si(6)	7433 (1)	5594 (1)	1898 (1)	502 (4)
C(61)	6873 (3)	7295 (4)	2016 (1)	777 (19)
C(62)	8225 (3)	4963 (5)	2438 (1)	810 (20)
Si(7)	8939 (1)	6910 (1)	1453 (1)	546 (4)
F(7)	8477 (2)	8206 (2)	1128 (1)	782 (11)
C(71)	9362 (3)	7684 (6)	2025 (1)	1078 (24)
N(5)	9943 (2)	6410 (3)	1245 (1)	558 (12)
Si(8)	10484 (1)	7717 (2)	960 (1)	768 (6)
C(81)	10524 (4)	9465 (5)	1240 (2)	1068 (26)
C(82)	11764 (4)	7329 (7)	944 (2)	1469 (39)
C(83)	9816 (4)	7948 (6)	355 (1)	1091 (27)
Si(9)	10558 (1)	4827 (1)	1408 (1)	691 (5)
C(91)	11657 (3)	5119 (6)	1877 (2)	1065 (25)
C(92)	10905 (4)	3949 (6)	903 (2)	1095 (26)
C(93)	9798 (3)	3532 (5)	1633 (2)	877 (22)

The structure was solved by direct methods and refined to a minimum value of $\sum w\Delta^2$ $[\Delta = |F_o| - |F_c|$: $w^{-1} = \sigma^2(F_o) + 0.001F_o^2]$. A common isotropic thermal parameter was refined for all H atoms and the methyl groups were refined as rigid groups with C–H = 0.96 Å, H–C–H = 109.5°. All other atoms were refined freely, with anisotropic thermal parameters.

The final R is 0.061, and $R_w = (\sum w\Delta^2/\sum wF_o^2)^{1/2} = 0.076$. Coordinates and geometrical parameters are given in Tables 1 and 2.*

Discussion. The molecule (Fig. 1) is bicyclic. The four-membered ring is almost planar [r.m.s. deviation from least-squares plane = 0.029 (4) Å]. The dimensions are typical for Si₂N₂ rings, with N–Si–N angles less than and Si–N–Si greater than 90° (Wheatley,

Table 2. *Bond lengths* (Å) *and angles* (°)

Si(1)–N(1)	1·723 (3)	Si(2)–N(1)	1·742 (3)
Si(2)–N(2)	1·733 (3)	Si(3)–N(1)	1·749 (3)
Si(3)–N(2)	1·742 (3)	Si(3)–N(3)	1·717 (3)
Si(4)–N(3)	1·755 (3)	Si(5)–N(3)	1·763 (3)
Si(5)–N(4)	1·776 (3)	Si(6)–N(2)	1·698 (3)
Si(6)·N(4)	1·755 (3)	Si(7)–N(4)	1·717 (3)
Si(7)–N(5)	1·729 (3)	Si(8)–N(5)	1·767 (4)
Si(9)–N(5)	1·747 (3)	Si(7)–F(7)	1·613 (3)
Si(1)–C(11)	1·885 (6)	Si(1)–C(12)	1·859 (4)
Si(1)–C(13)	1·839 (5)	Si(2)–C(21)	1·864 (5)
Si(2)–C(22)	1·857 (5)	Si(3)–C(31)	1·844 (4)
Si(4)–C(41)	1·845 (4)	Si(4)–C(42)	1·850 (5)
Si(4)·C(43)	1·866 (5)	Si(5)–C(51)	1·865 (5)
Si(5)·C(52)	1·853 (4)	Si(6)–C(61)	1·861 (4)
Si(6)–C(62)	1·853 (4)	Si(7)–C(71)	1·837 (5)
Si(8)–C(81)	1·850 (5)	Si(8)–C(82)	1·855 (6)
Si(8)–C(83)	1·862 (5)	Si(9)–C(91)	1·873 (5)
Si(9)–C(92)	1·878 (6)	Si(9)·C(93)	1·848 (5)

C(11)–Si(1)–C(12)	105·1 (2)	C(11)–Si(1)–C(13)	108·1 (2)
C(12)–Si(1)·C(13)	111·0 (3)	C(11)–Si(1)–N(1)	109·1 (2)
C(12)–Si(1)–N(1)	112·7 (2)	C(13)–Si(1)–N(1)	110·5 (2)
N(1)·Si(2)–C(21)	114·3 (2)	N(1)–Si(2)–C(22)	115·5 (2)
C(21)·Si(2)–C(22)	109·2 (3)	N(1)–Si(2)–N(2)	89·2 (2)
C(21)·Si(2)–N(2)	117·1 (2)	C(22)–Si(2)–N(2)	110·5 (2)
N(1)–Si(3) N(2)	88·6 (1)	N(1)–Si(3)–C(31)	112·1 (2)
N(2)·Si(3)·C(31)	114·2 (2)	N(1)–Si(3)–N(3)	123·2 (1)
N(2)–Si(3)–N(3)	106·1 (1)	C(31)–Si(3)–N(3)	110·5 (2)
N(3)–Si(4)–C(41)	111·6 (2)	N(3)–Si(4)–C(42)	112·5 (2)
C(41)–Si(4)–C(42)	110·0 (2)	N(3)–Si(4)–C(43)	109·9 (2)
C(41)–Si(4)·C(43)	107·1 (2)	C(42)–Si(4)·C(43)	105·5 (3)
N(3)–Si(5)–C(51)	109·8 (2)	N(3)–Si(5)·C(52)	110·3 (2)
C(51)–Si(5)–C(52)	105·7 (2)	N(3)–Si(5)·N(4)	112·3 (1)
C(51)–Si(5)–N(4)	108·3 (2)	C(52)–Si(5)–N(4)	110·2 (2)
N(2)–Si(6)·N(4)	104·5 (1)	N(2)–Si(6)·C(61)	108·4 (2)
N(4)·Si(6)–C(61)	112·5 (2)	N(2)–Si(6)–C(62)	108·8 (2)
N(4)–Si(6)–C(62)	114·1 (2)	C(61)–Si(6)–C(62)	108·3 (2)
N(4)–Si(7)–F(7)	106·1 (1)	N(4)–Si(7)–C(71)	112·5 (2)
F(7)·Si(7)–C(71)	105·3 (2)	N(4)·Si(7)–N(5)	118·9 (2)
F(7)–Si(7)–N(5)	104·4 (2)	C(71)–Si(7)–N(5)	108·5 (2)
N(5)–Si(8)·C(81)	112·5 (2)	N(5)·Si(8)–C(82)	113·4 (3)
C(81)–Si(8)–C(82)	104·7 (3)	N(5)–Si(8)·C(83)	111·7 (2)
C(81)–Si(8) C(83)	107·1 (2)	C(82)–Si(8)–C(83)	106·9 (3)
N(5)·Si(9)–C(91)	111·3 (2)	N(5)·Si(9)–C(92)	110·9 (2)
C(91) Si(9)–C(92)	110·4 (2)	N(5)–Si(9)–C(93)	112·2 (2)
C(91)–Si(9)–C(93)	106·1 (2)	C(92)–Si(9)–C(93)	105·7 (2)
Si(1)–N(1)–Si(2)	126·5 (2)	Si(1)–N(1)–Si(3)	142·7 (2)
Si(2)–N(1)–Si(3)	90·7 (1)	Si(2)–N(2)–Si(3)	91·2 (1)
Si(2)–N(2)–Si(6)	139·5 (2)	Si(3)–N(2)–Si(6)	129·3 (2)
Si(3)–N(3)·Si(4)	122·7 (2)	Si(3)–N(3)–Si(5)	115·6 (2)
Si(4)–N(3)–Si(5)	121·6 (2)	Si(5)–N(4)·Si(6)	114·4 (2)
Si(5)·N(4)·Si(7)	121·2 (2)	Si(6)·N(4)–Si(7)	120·1 (2)
Si(7)·N(5) Si(8)	116·9 (2)	Si(7)–N(5)–Si(9)	122·1 (2)
Si(8)·N(5)–Si(9)	119·7 (2)		

1962; Clegg, Klingebiel, Krampe & Sheldrick, 1980; Clegg, Klingebiel & Sheldrick, 1980). The six-membered ring is far from planar [r.m.s. deviation = 0.293 (4) Å]. Torsion angles for the ring bonds are shown in Fig. 1. The ring does not correspond to any of the ideal conformations, but approximates most closely to the boat conformation, for which the ideal torsion angles are 0, 60, −60, 0, 60, −60° in cyclic order (Bucourt & Hainaut, 1965). The simply substituted Si₃N₃ ring in (PhSiF.NMe)₃ is almost planar (Clegg, Noltemeyer, Sheldrick & Vater, 1980). For the structure described here, a planar Si₃N₃ ring is rendered impossible by the ring fusion and also, probably, by the bulky (Me₃Si)₂NSiFMe substituent at N(4). The ring in
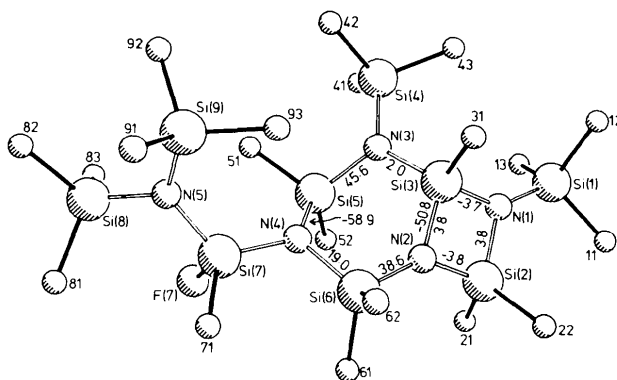
Fig. 1. A view of the molecule showing the atom numbering and torsion angles (°) for the ring bonds. Torsion angles have an e.s.d. of 0·2°. The sign convention is that of Klyne & Prelog (1960).

$(SiMe_2.NSiMe_3)_3$, which has bulky substituents, is also non-planar (Adamson & Daly, 1970).

The Si—N lengths cover a wide range [1·698 (3) to 1·776 (3) Å]. The shortest are those involving Si(7), which is bonded to an electronegative F atom, and those which are exocyclic to the four-membered ring. This shortening of Si—N bonds exocyclic to Si_2N_2 rings appears to be general (Wheatley, 1962; Clegg,

Klingebiel, Krampe & Sheldrick, 1980; Clegg, Klingebiel & Sheldrick, 1980).

All N atoms have essentially planar coordination, as is usual in such compounds (Bürger, 1973).

References

ADAMSON, G. W. & DALY, J. J. (1970). *J. Chem. Soc. A*, pp. 2724–2728.
BUCOURT, R. & HAINAUT, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366–1378.
BÜRGER, H. (1973). *Angew. Chem. Int. Ed. Engl.* 12, 474–487.
CLEGG, W. (1981). *Acta Cryst.* A37. In the press.
CLEGG, W., KLINGEBIEL, U., KRAMPE, C. & SHELDRICK, G. M. (1980). *Z. Naturforsch. Teil B*, 35, 275–281.
CLEGG, W., KLINGEBIEL, U. & SHELDRICK, G. M. (1980). *Z. Naturforsch. Teil B*. In the press.
CLEGG, W., NOLTEMEYER, M., SHELDRICK, G. M. & VATER, N. (1980). *Acta Cryst.* B36, 2461–2462.
KLYNE, W. & PRELOG, V. (1960). *Experientia*, 16, 521–523.
SKODA, L., KLINGEBIEL, U. & MELLER, A. (1980). *Chem. Ber.* 113, 2342–2347.
WHEATLEY, P. J. (1962). *J. Chem. Soc.* pp. 1721–1724.

The Structure of Benzoin

BY MASAO HAISA, SETSUO KASHINO AND MICHIHIRO MORIMOTO

Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700, Japan

Abstract. $C_{14}H_{12}O_2$, m.p. 409–410 K, monoclinic, $P2_1/a$, $a = 18.76$ (2), $b = 5.765$ (5), $c = 10.44$ (2) Å, $\beta = 107.1$ (2)°, $D_m = 1.29$, $D_x = 1.307$ Mg m^{-3}, $Z = 4$. The structure was refined to $R = 0.082$ for 1820 non-zero reflexions. Molecules related by a 2_1 axis are linked together by bifurcated hydrogen bonds to form columns along b. The columns are held together by van der Waals interactions along a and c. The keto group makes a dihedral angle of 12·0 (1)° with the plane of the phenyl ring attached to it. The dihedral angle between the two terminal phenyl rings is 64·5 (1)°. The torsion angle O=C—C—O is 26·0 (4)°. The O···O distance is 2·585 (4) Å, but there is no intramolecular hydrogen bond between the O atoms.

Introduction. The present work forms part of a study on the relationship between molecular conformation and crystal structure for aromatic compounds in which two benzene rings are separated by two bonding atoms. It has been shown previously that when the two bonding atoms are different the two benzene rings usually make a dihedral angle of about 60° (Kashino, Ito & Haisa, 1979). The molecular conformation of benzoin is of particular interest because of its ability to form intramolecular hydrogen bonds in solution (Joris & Schleyer, 1968), and its use as a chelating agent (Koester & Rotermund, 1965). The unit-cell dimensions have been obtained (Prasad & Shanker, 1936), but the crystal structure has not been determined.