Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

Cl—C23	1.740 (3)	C10—C11	1.367 (6)
O1—C1	1.409 (5)	C11—C12	1.365 (6)
O1—C30	1.371 (3)	C12—C13	1.383 (4)
O2—C2	1.431 (5)	C14—C15	1.384 (5)
O2—C29	1.377 (4)	C14—C19	1.388 (5)
O3—C3	1.429 (4)	C15—C16	1.408 (4)
O3—C13	1.367 (4)	C16—C17	1.368 (5)
N1—C5	1.466 (4)	C17—C18	1.356 (6)
N1—C6	1.472 (4)	C18—C19	1.374 (5)
N1—C7	1.398 (4)	C20—C21	1.386 (4)
N2—N3	1.417 (4)	C20—C25	1.386 (4)
N2—C6	1.466 (4)	C21—C22	1.379 (5)
N2—C14	1.396 (3)	C22—C23	1.379 (5)
N3—C7	1.281 (4)	C23—C24	1.376 (4)
C4—C5	1.539 (4)	C24—C25	1.377 (5)
C4—C26	1.502 (5)	C26—C27	1.376 (4)
C6—C8	1.524 (3)	C26—C31	1.395 (4)
C7—C20	1.463 (4)	C27—C28	1.397 (5)
C8—C9	1.374 (4)	C28—C29	1.372 (5)
C8—C13	1.398 (4)	C29—C30	1.388 (4)
C9—C10	1.387 (4)	C30—C31	1.378 (4)

C1—O1—C30	117.7 (3)	N2—C14—C19	120.3 (4)
C2—O2—C29	117.7 (2)	C15—C14—C19	119.5 (3)
C3—O3—C13	118.0 (2)	C14—C15—C16	119.0 (3)
C5—N1—C6	116.9 (2)	C15—C16—C17	121.0 (4)
C5—N1—C7	120.5 (2)	C16—C17—C18	118.8 (3)
C6—N1—C7	106.1 (3)	C17—C18—C19	122.2 (4)
N3—N2—C6	110.6 (2)	C14—C19—C18	119.5 (4)
N3—N2—C14	116.6 (2)	C7—C20—C21	119.6 (2)
C6—N2—C14	122.0 (3)	C7—C20—C25	121.8 (2)
N2—N3—C7	106.1 (2)	C21—C20—C25	118.5 (3)
C5—C4—C26	113.1 (3)	C20—C21—C22	121.1 (3)
N1—C5—C4	113.3 (3)	C21—C22—C23	118.7 (3)
N1—C6—N2	101.4 (2)	Cl—C23—C22	119.7 (2)
N1—C6—C8	113.0 (2)	Cl—C23—C24	118.9 (2)
N2—C6—C8	111.2 (2)	C22—C23—C24	121.4 (4)
N1—C7—N3	115.4 (3)	C23—C24—C25	119.0 (3)
N1—C7—C20	122.1 (2)	C20—C25—C24	121.1 (3)
N3—C7—C20	122.5 (3)	C4—C26—C27	121.1 (2)
C6—C8—C9	120.0 (3)	C4—C26—C31	120.6 (3)
C6—C8—C13	121.8 (3)	C27—C26—C31	118.2 (3)
C9—C8—C13	118.2 (2)	C26—C27—C28	120.9 (3)
C8—C9—C10	121.6 (3)	C27—C28—C29	120.1 (3)
C9—C10—C11	119.3 (3)	O2—C29—C28	124.9 (3)
C10—C11—C12	120.4 (3)	O2—C29—C30	115.5 (2)
C11—C12—C13	120.7 (3)	C28—C29—C30	119.6 (3)
O3—C13—C8	116.1 (2)	O1—C30—C29	115.9 (3)
O3—C13—C12	124.2 (3)	O1—C30—C31	124.1 (3)
C8—C13—C12	119.9 (3)	C29—C30—C31	120.0 (3)
N2—C14—C15	120.3 (3)	C26—C31—C30	121.1 (3)

Cl—O1—C30—C29	165.5 (3)	C5—N1—C7—C20	39.8 (4)
Cl—O1—C30—C31	−13.2 (4)	C6—N1—C7—N3	−5.4 (3)

Table 2 (*cont.*)

C2—O2—C29—C28	−4.0 (4)	C6—N1—C7—C20	175.6 (3)
C2—O2—C29—C30	176.7 (3)	C6—N2—N3—C7	3.5 (3)
C3—O3—C13—C8	−175.6 (3)	C14—N2—N3—C7	148.8 (3)
C3—O3—C13—C12	4.5 (4)	N3—N2—C6—N1	−6.4 (3)
C6—N1—C5—C4	88.6 (3)	N3—N2—C6—C8	−126.7 (2)
C7—N1—C5—C4	140.1 (3)	C14—N2—C6—N1	−149.4 (2)
C5—N1—C6—N2	144.3 (2)	C14—N2—C6—C8	90.3 (3)
C5—N1—C6—C8	−96.7 (3)	N2—N3—C7—N1	1.3 (3)
C7—N1—C6—N2	6.7 (3)	N2—N3—C7—C20	−179.7 (3)
C7—N1—C6—C8	125.7 (3)	C26—C4—C5—N1	66.2 (3)
C5—N1—C7—N3	−141.2 (3)		

are different [N1—C7 = 1.398 (4) and N3—C7 = 1.281 (4) Å], indicating the presence of a C7—N3 double bond. Although the triazole ring is planar within experimental error, the best least-squares plane is through the group N1—C7=N3—N2 with C6 deviating 0.107 (2) Å from it. The values of 1.466 (4) Å for N1—C5 and 1.396 (3) Å for N2—C14 can be compared with values of 1.452 (2) and 1.417 (2) Å for similar bonds in 5,5-dimethyl-4-(2-methylallyl)-2-tolyl-1,2,4-triazolidine-3-thione (Schulze, Richter & Faure, 1988).

All intermolecular contacts correspond to van der Waals interactions.

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Structure of (1R,2S)-(−)-2-(Benzylamino)-1-(*tert*-butyldimethylsiloxy)-1-phenyl-propane Hydrochloride

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Abstract. $C_{22}H_{34}NOSi^+.Cl^-$, $M_r = 381.98$, orthorhombic, $P2_12_12_1$, $a = 8.3358$ (6), $b = 14.3567$ (5), $c = 19.8068$ (7) Å, $V = 2370.4$ (3) Å^3, $Z = 4$, $D_x = 1.07$ g cm^{-3}, λ(Mo $K\alpha$) = 0.710730 Å, $\mu = 2.17$ cm^{-1}, $F(000) = 848$, $T = 293$ K, final $R = 0.0372$ for 1105 significant reflections. The asymmetric unit

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Table 1. *Positional parameters* [Cl(40) × 10⁵, *others* × 10⁴] *and equivalent thermal parameters* (Å²; N, O, Si *and* Cl × 10³, *others* × 10²) *for the non-*H *atoms*

$$B_{eq} = \tfrac{8}{3}\pi^2 \bar{U}.$$

	x	y	z	B_{eq}
N(31)	1021 (5)	−1395 (3)	381 (2)	311 (13)
O(21)	−885 (5)	13 (2)	1139 (2)	360 (12)
Si(22)	−2057 (2)	962 (1)	837 (1)	436 (6)
Cl(40)	−31951 (24)	18945 (14)	44412 (5)	503 (5)
C(01)	−2242 (7)	−1148 (3)	1708 (2)	35 (2)
C(02)	−2156 (8)	−701 (4)	2329 (2)	47 (2)
C(03)	−3006 (11)	−983 (5)	2873 (3)	65 (3)
C(04)	−4083 (11)	−1715 (5)	2804 (3)	77 (3)
C(05)	−4203 (10)	−2158 (5)	2194 (3)	71 (3)
C(06)	−3286 (10)	−1873 (4)	1657 (3)	56 (2)
C(07)	−1263 (6)	−852 (2)	1110 (2)	33 (2)
C(08)	290 (6)	−1407 (3)	1083 (2)	37 (2)
C(09)	1557 (7)	−1111 (4)	1599 (2)	52 (2)
C(23)	−4156 (6)	839 (5)	1157 (3)	83 (3)
C(24)	−2096 (1)	882 (3)	−101 (1)	70 (3)
C(25)	−1086 (8)	2060 (4)	1138 (3)	49 (2)
C(26)	711 (8)	1985 (5)	1021 (3)	86 (3)
C(27)	−1770 (11)	2908 (3)	778 (3)	98 (3)
C(32)	1881 (8)	−533 (3)	169 (2)	45 (2)
C(33)	2448 (7)	−592 (3)	−548 (2)	40 (2)
C(34)	4036 (7)	−853 (4)	−701 (3)	46 (2)
C(35)	4532 (7)	−892 (4)	−1355 (3)	54 (2)
C(36)	3505 (10)	−659 (5)	−1875 (3)	60 (3)
C(37)	1961 (10)	−399 (5)	−1736 (2)	55 (3)
C(38)	1440 (6)	−369 (4)	−1070 (2)	46 (2)
C(28)	−1324 (10)	2187 (4)	1903 (3)	78 (3)

contains one molecule of the title compound. The compound forms infinite chains along a twofold axis parallel to the *a* axis. The Cl atom bridges between two NH_2 groups. All available NH H atoms participate in hydrogen bonding.

Introduction. The title compound was prepared in the context of an investigation into the role of the Mg^{2+} ion in the diastereoselective synthesis of optically pure natural products (Brussee, Van Benthem, Kruse
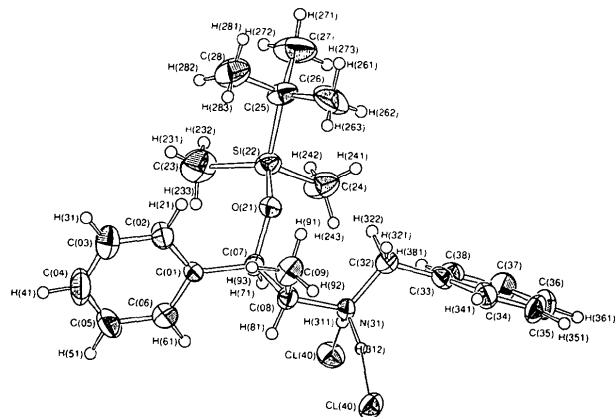

Fig. 1. Projection and atomic labeling of (1R,2S)-(−)-2-(benzylamino)-1-(*tert*-butyldimethylsiloxy)-1-phenylpropane hydrochloride
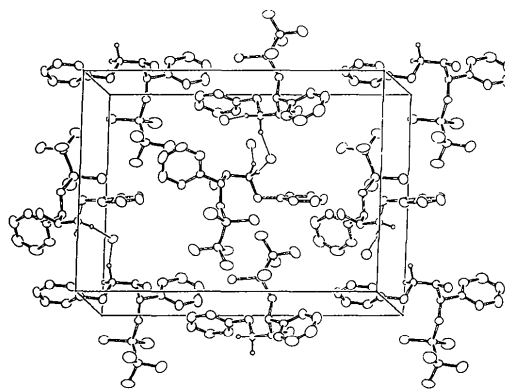
Table 2. *Intramolecular distances* (Å) *and bond angles* (°) *involving non-*H *atoms and bridging* H *atoms only*

N(31)—C(08)	1.519 (2)	C(04)—C(05)	1.369 (1)
N(31)—C(32)	1.490 (2)	C(05)—C(06)	1.371 (3)
O(21)—Si(22)	1.654 (4)	C(07)—C(08)	1.520 (2)
O(21)—C(07)	1.4504 (9)	C(08)—C(09)	1.530 (3)
Si(22)—C(23)	1.869 (3)	C(25)—C(26)	1.520 (2)
Si(22)—C(24)	1.8604 (5)	C(25)—C(27)	1.520 (3)
Si(22)—C(25)	1.869 (6)	C(25)—C(28)	1.539 (1)
Cl(40)—H(312ⁱⁱ)	1.96 (2)	C(32)—C(33)	1.499 (2)
Cl(40)—H(311ⁱⁱ)	1.95 (2)	C(33)—C(34)	1.409 (2)
C(01)—C(02)	1.390 (1)	C(33)—C(38)	1.370 (2)
C(01)—C(06)	1.360 (2)	C(34)—C(35)	1.360 (1)
C(01)—C(07)	1.501 (2)	C(35)—C(36)	1.378 (3)
C(02)—C(03)	1.351 (3)	C(36)—C(37)	1.369 (3)
C(03)—C(04)	1.391 (3)	C(37)—C(38)	1.389 (2)

C(08)—N(31)—C(32)	117.5 (4)	N(31)—C(08)—C(09)	109.4 (4)
Si(22)—O(21)—C(07)	124.0 (3)	C(07)—C(08)—C(09)	114.8 (4)
O(21)—Si(22)—C(23)	111.2 (3)	Si(22)—C(25)—C(26)	108.6 (4)
O(21)—Si(22)—C(24)	109.2 (3)	Si(22)—C(25)—C(27)	111.3 (5)
O(21)—Si(22)—C(25)	103.6 (2)	Si(22)—C(25)—C(28)	111.0 (4)
C(23)—Si(22)—C(24)	108.3 (4)	C(26)—C(25)—C(27)	110.5 (7)
C(23)—Si(22)—C(25)	112.0 (3)	C(26)—C(25)—C(28)	106.8 (6)
C(24)—Si(22)—C(25)	112.4 (3)	C(27)—C(25)—C(28)	108.5 (5)
C(02)—C(01)—C(06)	116.9 (5)	N(31)—C(32)—C(33)	111.9 (4)
C(02)—C(01)—C(07)	122.6 (5)	C(32)—C(33)—C(34)	121.0 (5)
C(06)—C(01)—C(07)	120.5 (4)	C(32)—C(33)—C(38)	120.7 (5)
C(01)—C(02)—C(03)	122.6 (5)	C(34)—C(33)—C(38)	118.3 (4)
C(02)—C(03)—C(04)	119.2 (6)	C(33)—C(34)—C(35)	120.1 (4)
C(03)—C(04)—C(05)	118.9 (7)	C(34)—C(35)—C(36)	120.7 (5)
C(04)—C(05)—C(06)	120.4 (7)	C(35)—C(36)—C(37)	120.1 (5)
C(01)—C(06)—C(05)	121.8 (5)	C(36)—C(37)—C(38)	119.3 (5)
O(21)—C(07)—C(01)	111.3 (4)	C(33)—C(38)—C(37)	121.4 (5)
O(21)—C(07)—C(08)	109.1 (4)	N(31)—H(311)—Cl(40ⁱⁱⁱ)	171 (3)
C(01)—C(07)—C(08)	111.0 (3)	N(31)—H(312)—Cl(40ⁱ)	162 (2)
N(31)—C(08)—C(07)	111.5 (3)		

Hydrogen bonds

N(31)—Cl(40ⁱ)	3.073 (4)	N(31)—Cl(40ⁱⁱⁱ)	3.088 (4)

Symmetry operations: (i) $-x$, $-\tfrac{1}{2}+y$, $\tfrac{1}{2}-z$; (ii) $-x$, $\tfrac{1}{2}+y$, $\tfrac{1}{2}-z$; (iii) $-\tfrac{1}{2}-x$, $-y$, $-\tfrac{1}{2}+z$; (iv) $-\tfrac{1}{2}-x$, $-y$, $\tfrac{1}{2}+z$.
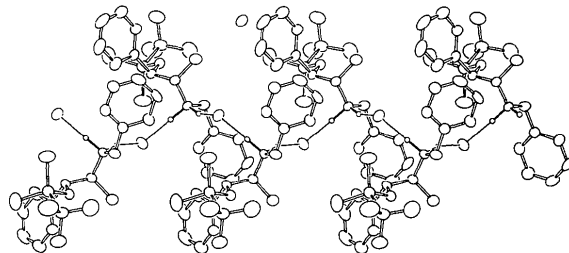

Fig. 2. Cell contents.

Fig. 3. Hydrogen bonding.

Table 3. *Specific geometry*

Plane equation used $ax + by + cz + d = 0$.

Type		Coordinates of centre of gravity			Coefficients of unit vector or plane equation				Distances (Å)
1 line	C(32) C(33)	1.805 (5)	−0.806 (3)	−0.376 (4)	−0.315 (6)	0.055 (4)	0.947 (2)		
2 line	C(01) C(07)	−1.461 (4)	−1.436 (3)	2.790 (4)	−0.545 (4)	−0.283 (4)	0.789 (3)		
3 plane	C(01)	−2.637 (3)	−2.053 (3)	4.478 (3)	−0.725 (2)	0.633 (2)	−0.271 (3)	0.60 (2)	−0.005 (5)
	C(02)								0.017 (5)
	C(03)								−0.018 (6)
	C(04)								0.009 (6)
	C(05)								0.002 (6)
	C(06)								−0.004 (6)
	C(07)								−0.009 (9)
4 plane	C(33)	2.490 (3)	−0.906 (3)	−2.406 (3)	0.297 (3)	0.953 (1)	0.059 (3)	0.265 (9)	0.000 (5)
	C(34)								0.014 (5)
	C(35)								−0.020 (5)
	C(36)								0.011 (6)
	C(37)								0.003 (6)
	C(38)								−0.009 (5)
	C(32)								0.022 (9)
5 plane	O(21) C(07) C(08)	−0.515 (3)	−1.018 (3)	2.200 (2)	−0.013 (5)	0.044 (3)	−0.999 (0)	2.236 (5)	
6 plane	N(31) C(07) C(08)	0.014 (3)	−1.748 (3)	1.699 (3)	0.516 (3)	0.823 (3)	0.237 (6)	1.03 (1)	

Angles between lines and planes

	2	3	4	5	6
1	25.3 (4)	0.4 (4)	0.8 (3)	70.0 (4)	6.2 (4)
2	—	0.1 (3)	22.7 (3)	52.5 (5)	19.1 (4)
3		—	68.2 (2)	72.1 (2)	85.3 (4)
4			—	91.2 (3)	17.9 (4)
5				—	102.0 (4)

& van der Gen, 1990). Reductive amination of (R)-O-protected α-hydroxyketones with primary amines by $NaBH_4$ in the presence of $Mg(ClO_4)_2$ led to the exclusive formation of erythro-(1R,2S)-O-protected-N-substituted ethanolamines. Discrimination between erythro and threo was determined by NMR using a procedure described for unprotected ethanolamines (Lövgren & Nilsson, 1977). The absolute configuration and conformation of this silylated ethanolamine and its related compounds are of great importance in obtaining a better understanding of the reaction mechanism of this type of synthesis.

Experimental. A colorless crystal, obtained by recrystallization from absolute ethanol, of approximate dimensions $0.5 \times 0.3 \times 0.2$ mm was used in the structure determination. Enraf–Nonius CAD-4 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.710730$ Å). Cell constants from setting angles of 24 reflections, $8 < \theta < 10°$. Corrections for Lorentz and polarization effects, transmission coefficients 0.968 to 1.037, $\mu = 2.17$ cm^{-1}, $2.0 < \theta < 27.5°$; h: 0 to 10, k: 0 to 18, l: 0 to 25. Standard reflections 05$\bar{4}$, 2$\bar{1}$7, 108; decay of scattering power 5.17%, corrected. 3113 measured reflections (ω–2θ scans), 3110 independent, $R_{int} = 0.0058$, 2005 reflections with $I < 2\sigma(I)$ were classified as unobserved.

Direct methods. F used in least-squares refinement. All H atoms placed on calculated positions. Least-squares refinement of positional and non-H anisotropic thermal parameters; isotropic thermal factors of the H atoms coupled to one another, final value 7.7 Å^2, $S = 1.506$, $w = 1/\sigma^2(F)$, $R = 0.035$, $wR = 0.037$, $(\Delta/\sigma)_{max} < 0.05$. Maximum and minimum $\Delta\rho$ values in final difference synthesis 0.13 and -0.17 e Å^{-3}, noise level 0.11. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). Leiden University Computer (IBM 3083); *MULTAN* (Main, 1978) and programs written or modified by S. Gorter, R. A. G. de Graaff and E. W. Rutten-Keulemans.

Discussion. Positional parameters and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1.* Atomic distances and intramolecular bond angles for the non-H atoms and the bridging H atoms are listed in Table 2. Definitions of

* Lists of H-atom coordinates, atomic distances involving the H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54503 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

some lines and planes and the angles between them are given in Table 3. An extended and modified version of *ORTEP* (Johnson, 1970) was used for the projection of the asymmetric unit of the title compound. The configuration of the two chiral centers is established as 1R,2S, which is in agreement with the NMR study. The projection and the atomic labeling are given in Fig. 1. In this figure two Cl atoms are displayed to demonstrate the hydrogen bonding. The second Cl atom belongs to a neighbouring molecule. In Fig. 2 the cell contents are displayed. This figure shows some of the hydrogen bonding, but the way in which the hydrogen bonding connects molecules in different cells is displayed in Fig. 3.

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Structure of 2-Amino-6-dimethylamino-4-(tricyclo[3.3.1.1³,⁷]dec-1-yl)-1,3,5-triazine

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Abstract. $C_{15}H_{23}N_5$, $M_r = 273.4$, triclinic, $P\bar{1}$, $a = 6.907$ (6), $b = 10.260$ (5), $c = 10.619$ (2) Å, $\alpha = 104.64$ (4), $\beta = 91.61$ (7), $\gamma = 104.88$ (5)°, $V = 700.2$ (14) Å^3, $Z = 2$, $D_m = 1.295$, $D_x = 1.296$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 0.648$ mm^{-1}, $F(000) = 296$, $T = 293$ K, final $R = 0.059$ for 1510 observed reflections. In pyrimidine analogs of this class of antifolate agents, a lipophilic substituent at position 5 disrupts the ring planarity [Cody (1986). *J. Mol. Graphics*, **4**(1), 69–73]. Here, the adamantyl substituent at position 4 does not have a similar effect. The adamantyl assumes one of the two observed possible conformations relative to the triazine ring.

Introduction. Dihydrofolate reductase [5,6,7,8-tetrahydrofolate:NADP$^+$ oxidoreductase (E.C. 1.5.1.3)], an enzyme which reduces dihydrofolic to tetrahydrofolic acid, is a protein of prime importance in biochemistry and medicinal chemistry (Blakley, 1969). It is used as a target for several antibacterial and antineoplasmic (antitumor) drugs. The activity of

these drugs is due to selective inhibition of the enzyme from species to species (Baccarani, Daluge & King, 1982; Hitchings & Smith, 1980). Unfortunately, the exact molecular mechanism of selective inhibition is not yet known, despite extensive efforts.

The discovery that diamino-s-triazines interfere with folic acid metabolism triggered research on the antifolate activity of this class of compounds and has shown promise in cancer chemotherapy (Modest, Foley, Pechet & Farber, 1952). A conclusion drawn from these studies is that for several antifolates the extent of their uptake and their growth inhibitory potency on tumor cells, as well as their affinity to dihydrofolate reductase (DHFR), correlate well with lipophilicity (Greco & Hakala, 1980). In the course of our investigations on compounds containing adamantane rings (Antoniadou-Vyza & Foscolos, 1986; Garoufalias, Vyza, Fytas, Foscolos & Chytiroglou, 1988) we considered it likely that attachment of this group to a triazine ring would allow it to make effective use of the enzyme hydrophobic cavity. In addition, the absence of any