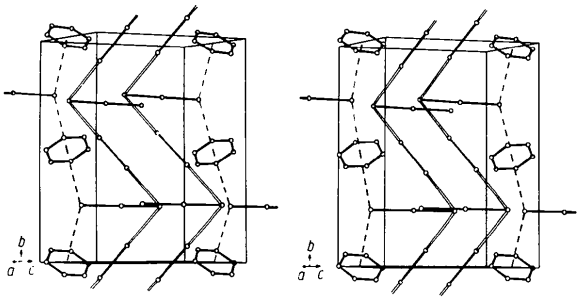

Fig. 2. Stereoskopische Ansicht der Struktur von $PyH^+[I_3.I_2]^-$ (ohne H-Atome).

3,860 (1) Å, die Abstände von I(1) zu den C- bzw. N-Atomen liegen zwischen 4,00 und 4,16 (2) Å (Mittelwert 4,11 Å). Der Abstand zwischen den Ringen und den Iodatomen ist damit kürzer als im Pyridiniumiodid (4,133 Å; Hartl, 1975), in dem einander abwechselnde PyH^+-Ringe und I^--Ionen säulenartig gestapelt sind. Eine ähnliche Packung von aromatischen Ringen zwischen Halogenatomen kennt man auch bei $PyH^+[ICl_2]^-$ (Tucker & Kroon, 1973) und beim $C_6H_6.Br_2$ (Hassel & Strømme, 1958).

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Structure of (*R*)-(—)-7-[2-(1-Hydroxy-2-butylamino)ethyl]theophylline Hydrochloride

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Abstract. $C_{13}H_{22}N_5O_3^+.Cl^-$, $M_r = 331{\cdot}803$, monoclinic, $C2$, $a = 21{\cdot}821\ (3)$, $b = 11{\cdot}810\ (2)$, $c = 6{\cdot}978\ (2)$ Å, $\beta = 100{\cdot}7\ (3)°$, $V = 1767{\cdot}01$ Å^3, $Z = 4$, $D_m = 1{\cdot}25$, $D_x = 1{\cdot}23$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 2{\cdot}051$ mm^{-1}, $F(000) = 704$, $T =$ 293 K, $R = 0{\cdot}065$ for 1512 unique observed reflections. The compound is a potential antiarrhythmic and the molecule has the typical synclinal conformation for the $-NH-CH-CH_2-OH$ fragment of the side chain [$\tau_{CC} = 56{\cdot}5\ (8)°$] exhibited by these compounds. The Cl

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Table 1. *Summary of data collection and structure refinement*

Crystal shape and size (mm)	Colourless plate 0·32 × 0·22 × 0·11
Measuring D_m	By flotation
Diffractometer	Enraf–Nonius CAD-4 (graphite-monochromated Cu $K\alpha$ radiation)
Lattice-parameter measurement:	
θ range, number of reflections	$8 \le \theta \le 17°$, 15
Intensity measurement:	
θ range	$1 \le \theta \le 72°$
Indices range	$-26 \le h \le 26, 0 \le k \le 14, 0 \le l \le 8$
Scan width (°) and mode	0·70 + 0·30tanθ, $\omega/2\theta$
Intensity control reflections	$\bar{1}12$, 202 measured every 46 reflections
Changes in intensity	< 3·4%
Number of reflections measured	2056
Criterion for observed reflections	$\|F_o\| \ge 3·92\sigma(F)$
Number of observed unique reflections	1512
Corrections applied	Lorentz, polarization effects
Extinction reflections omitted	110, 600, 001, 601
Refinement method	Full-matrix least squares on $\|F_o\|$'s in two blocks
Parameters refined	326
non-H atoms	Positional and anisotropic thermal
H atoms*	Positional and isotropic thermal for some atoms, others added as fixed contribution
Weighting scheme	$w = k[\sigma^2(F_o) + g(F_o)^2]^{-1}$; k and g converged to 5·2178 and 0·00079, respectively
R, wR, S	0·065, 0·066, 4·797
Av., max. Δ/σ	
non-H atoms	0·03, 0·11
H atoms	0·07, 0·10
Max., min. height in final difference Fourier synthesis (e Å^{-3})	0·14, −0·23

* Initial positional parameters of H atoms found from the difference Fourier maps except for disordered H atoms which were calculated.

anion is hydrogen bonded to the N atom of 2-aminobutanal: N···Cl = 3·075 (1) Å; N—H···Cl = 169·6 (5)°.

Introduction. A number of antiarrhythmically active compounds of natural origin (*e.g.* quinidine, ajmaline) as well as synthetic products such as propranolol, possess a 2-amino-1-hydroxy fragment with a secondary or tertiary N atom and a chiral C atom. A series of chiral 2-amino-1-butanol derivatives was studied as potential antiarrhythmics by Eckstein, Samochowiec & Zajaczkowska (1978). These compounds have relatively low toxicity and wide therapeutic spectrum as shown by pharmacological tests of arrhythmia induced by adrenaline and chloroform. The title compound tested on rats in intraperitoneal and intravenous application has therapeutic indices equal to 68·4 and 47, respectively.

Experimental. The compound was synthesized by J. Zajaczkowska (Eckstein, Samochowiec & Zajaczkowska, 1978). The absolute configuration (*R*) of the molecule is known from the configuration of 2-amino-1-butanol used in synthesis and established earlier by chemical methods. Crystals suitable for X-ray study were obtained from acetone–ethanol solution. A summary of data collection and structural refinement is given in Table 1. The structure was solved

Table 2. *Fractional atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic temperature factors*

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \mathbf{a}_j$$

	x	y	z	B_{eq}(Å^2)
C(17)	0·2152 (2)	0·2072 (4)	−0·0618 (6)	4·14
C(18)	0·2060 (2)	0·2792 (4)	−0·2438 (6)	4·96
O(19)	0·2610 (2)	0·3420 (3)	−0·2391 (5)	5·34
C(20*A*)*	0·1586 (5)	0·1260 (7)	−0·0812 (8)	4·85
C(20*B*)*	0·1629 (5)	0·1329 (7)	−0·0139 (8)	4·90
C(21*A*)*	0·0990 (6)	0·1845 (8)	−0·0600 (9)	7·62
C(21*B*)*	0·1110 (5)	0·1966 (8)	0·0371 (9)	7·50
N(3)	0·5283 (2)	0·0747 (4)	0·2500 (5)	3·99
C(4)	0·4644 (2)	0·0686 (5)	0·2624 (5)	3·81
C(5)	0·4383 (2)	0·1793 (4)	0·2636 (5)	3·51
C(6)	0·4716 (2)	0·2766 (4)	0·2555 (5)	3·63
N(1)	0·5339 (2)	0·2740 (4)	0·2439 (5)	4·03
C(2)	0·5633 (2)	0·1711 (5)	0·2360 (5)	4·19
N(7)	0·3789 (2)	0·2155 (4)	0·2710 (5)	3·58
C(8)	0·3808 (2)	0·3300 (4)	0·2638 (6)	4·28
N(9)	0·4366 (2)	0·3703 (4)	0·2552 (5)	4·53
C(10)	0·5695 (3)	0·3781 (5)	0·2380 (6)	5·18
O(11)	0·6175 (1)	0·1650 (4)	0·2192 (5)	5·50
C(12)	0·5610 (3)	−0·0352 (5)	0·2480 (6)	5·24
O(13)	0·4381 (2)	−0·0214 (3)	0·2700 (5)	4·83
C(14)	0·3239 (2)	0·1471 (5)	0·2915 (6)	4·33
C(15)	0·3011 (2)	0·0752 (4)	0·1148 (6)	4·04
N(16)	0·2739 (2)	0·1402 (3)	−0·0646 (5)	3·52
Cl	0·2590 (1)	−0·0592 (2)	−0·3529 (2)	6·13

* Occupancy factors fixed at 0·5.

Table 3. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N(1)–C(2)	1·380 (7)	C(4)–O(13)	1·214 (7)
C(2)–N(3)	1·384 (7)	C(2)–O(11)	1·212 (4)
N(3)–C(4)	1·415 (5)	N(7)–C(14)	1·476 (6)
C(4)–C(5)	1·427 (7)	C(14)–C(15)	1·504 (6)
C(5)–C(6)	1·366 (6)	C(15)–N(16)	1·494 (5)
C(5)–N(7)	1·375 (5)	N(16)–C(17)	1·509 (5)
N(7)–C(8)	1·354 (7)	C(17)–C(18)	1·510 (6)
N(1)–C(6)	1·377 (5)	C(18)–O(19)	1·406 (5)
C(8)–N(9)	1·319 (5)	C(17)–C(20*A*)	1·55 (1)
N(9)–C(6)	1·344 (6)	C(17)–C(20*B*)	1·53 (1)
N(1)–C(10)	1·459 (7)	C(20*A*)–C(21*A*)	1·50 (2)
N(3)–C(12)	1·482 (3)	C(20*B*)–C(21*B*)	1·46 (1)

C(6)–N(1)–C(2)	119·6 (4)	C(10)–N(1)–C(2)	119·1 (4)
C(10)–N(1)–C(6)	121·3 (4)	N(1)–C(2)–O(11)	121·7 (4)
N(1)–C(2)–N(3)	117·0 (4)	O(11)–C(2)–N(3)	121·3 (4)
C(2)–N(3)–C(4)	127·5 (4)	C(12)–N(3)–C(4)	115·9 (4)
C(2)–N(3)–C(12)	116·5 (4)	N(3)–C(4)–C(5)	110·7 (4)
N(3)–C(4)–O(13)	121·8 (5)	O(13)–C(4)–C(5)	127·6 (4)
C(4)–C(5)–C(6)	123·7 (4)	C(4)–C(5)–N(7)	131·7 (4)
C(6)–C(5)–N(7)	104·6 (3)	C(5)–C(6)–N(9)	112·7 (4)
C(5)–C(6)–N(1)	121·5 (4)	N(1)–C(6)–N(9)	125·8 (4)
C(6)–N(9)–C(8)	103·4 (4)	N(9)–C(8)–N(7)	113·4 (4)
C(5)–N(7)–C(8)	105·9 (4)	C(8)–N(7)–C(14)	125·5 (4)
C(5)–N(7)–C(14)	128·5 (4)	N(7)–C(14)–C(15)	112·4 (4)
C(14)–C(15)–N(16)	114·6 (3)	C(15)–N(16)–C(17)	117·7 (3)
N(16)–C(17)–C(18)	105·4 (3)	N(16)–C(17)–C(20*A*)	109·8 (5)
C(17)–C(18)–O(19)	107·3 (3)	N(16)–C(17)–C(20*B*)	111·9 (5)
C(18)–C(17)–C(20*A*)	107·2 (4)	C(17)–C(20*A*)–C(21*A*)	113·4 (6)
C(18)–C(17)–C(20*B*)	121·3 (4)	C(17)–C(20*B*)–C(21*B*)	113·8 (6)

by direct methods using *SHELX76* (Sheldrick, 1976) and refined with this program system. Scattering factors were as in *SHELX76*. Geometrical calculations were performed with *XANADU* (Roberts & Sheldrick, 1972) and figures drawn on an IBM PC/AT with *ORTEP* (Johnson, 1971), within *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1987). Calculations were carried out on a Cyber72.

Two C atoms of the hydroxybutyl substituent show disorder. They were localized from the difference Fourier map, each occupying two alternative positions with equal probability, and refined together with H atoms in calculated positions.

Discussion. The atomic parameters are listed in Table 2.* The bond lengths and angles are given in Table 3.

* Lists of structure factors, anisotropic temperature factors, H-atom parameters, bond lengths and least-squares-plane data, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44830 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

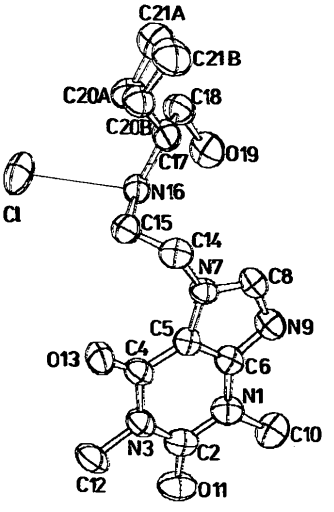

Fig. 1. The molecule with the atom-numbering scheme.

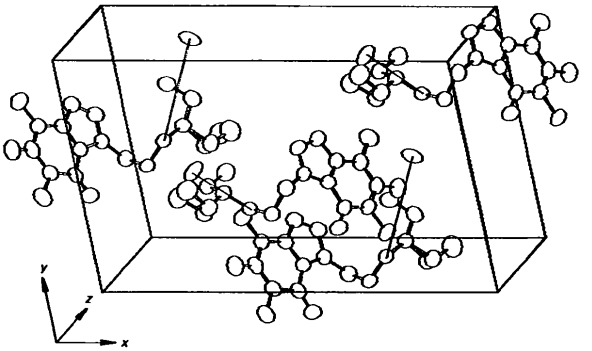

Fig. 2. Packing diagram (Johnson, 1971).

Table 4. *The intramolecular* O···N *distance* (Å) *and the torsion angle* (°) *of the side-chain fragment; e.s.d.'s are ca* 0·01 Å *and* 0·7–0·9°

	Reference	O···N	O—C—C—N
Quinidine	(*a*)	3·09	75·9
Quinidine-ethanol (1/1)	(*b*)	3·11	74·8
Propranolol	(*c*)	2·92	66·0
Propranolol. HCl	(*c*)	2·85	55·0
This work		2·69*	56·5†

References: (*a*) Kashino & Haisa (1983); (*b*) Doherty, Benson, Maienthal & Stewart (1978); (*c*) Ammon *et al.* (1977).

*O(19)···N(16).
† O(19)—C(18)—C(17)—N(16).

The molecular structure of the title compound together with the atom-numbering scheme is shown in Fig. 1. It is composed of two structural units: a theophylline moiety and a side chain with a 2-amino-1-butanol fragment. The theophylline is planar within ±0·002 Å (Table 4) and its bond lengths and angles are as expected from other studies (*e.g.* Ružić-Toroš, Kojić-Prodić & Coffou, 1981). The N(16)—C(17)—C(18)—O(19) fragment of the side chain is common to various antiarrhythmics. They were divided by Szekeres & Papp (1971) into two broad classes: specific (β-adrenergic blocking agents, *e.g.* propranolol, belong to this class) and nonspecific (quinidine is the prototype of this class). A comparison of the geometry of this fragment in the present compound and in representatives of both activity classes is given in Table 4; the geometries are basically similar, showing a typical synclinal conformation. The angle between the theophylline plane and the best plane through C(14)—C(15)—N(16)—C(17) = 76·6°; this value is in the range of the corresponding angles for some α,β-adrenergic stimulating and β-blocking drugs cited by Ammon, Balsamo, Macchia, Macchia, Howe & Keefe (1975). The packing in the structure is shown in Fig. 2. Cl^- anions are hydrogen bonded to the aminobutanol N atom with N(16)···Cl = 3·075, N(16)—H(162) = 0·81 (1), H(162)···Cl = 2·28 (1) Å, and N(16)—H(162)···Cl = 169·6 (5)°.

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Structure of *N,N*-Bis(*o*-nitrobenzyl)tryptamine

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Abstract. 3-[2-Bis(2-nitrobenzyl)aminoethyl]-1*H*-indole, $C_{24}H_{22}N_4O_4$, $M_r = 430{\cdot}46$, monoclinic, $P2_1$, $a = 7{\cdot}689$ (1), $b = 14{\cdot}041$ (1), $c = 10{\cdot}438$ (1) Å, $\beta = 106{\cdot}25$ (7)°, $V = 1081{\cdot}9$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}321$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 7{\cdot}16$ cm^{-1}, $F(000) = 452$, $T = 291$ K, final $R = 0{\cdot}0457$ for 1958 reflections and 333 parameters. The two nitrobenzyl substituents differ in conformational and thermal properties. Both nitro groups participate in an intermolecular bifurcated hydrogen bond. The geometry of the indole ring system is in agreement with previous investigations of indole amines.

Introduction. As part of a study of organic synthesis routes to quinazolinocarboline alkaloids (Bergman & Bergman, 1985), a group of naturally occurring and biologically active substances, the structure of the title reaction intermediate was established by X-ray analysis. Tryptamine derivatives can be found in mammals as well as in plants, and their biological effects on the central nervous system have been the cause of many investigations. In connection with these, several structural studies have been undertaken (Falkenberg & Carlström, 1971; Petcher & Weber, 1974; Weber & Petcher, 1974; Quarles, Templeton & Zalkin, 1974; Gartland, Freeman & Bugg, 1974; Mostad & Römming, 1974; Ohki, Takenaka, Shimanouchi & Sasada, 1977; Ishida, Inoue, Fujiwara & Tomita, 1979).

Experimental. Pale-yellow transparent rods, specimen size 0·67 × 0·30 × 0·25 mm. Data collected on a Stoe DIF4 four-circle diffractometer by $\omega/2\theta$ scan, scan width 1·05°, scan speed 1·14–3·42° min^{-1}. Three standard reflections were monitored every 90 min, intensity variation within 2%. Lattice parameters by least-squares refinement of setting angles for 18 reflections, $15{\cdot}42 \le \theta \le 24{\cdot}49°$. 3496 reflections measured, max. $\sin\theta/\lambda = 0{\cdot}6077$ Å^{-1}, $-8 \le h \le 8$, $-17 \le k \le 17$, $0 \le l \le 12$. Structure solved using *MITHRIL* (Gilmore, 1983). Anisotropic refinement minimizing $\sum w(\Delta F)^2$ with *SHELX*76 (Sheldrick, 1976), isotropic H atoms in geometrically fixed positions and with group temperature factors (five groups). H(1), on the indole N atom, was taken from the $\Delta\rho$ map and individually refined. $R = 0{\cdot}0457$ and $wR = 0{\cdot}0463$, $w = 1{\cdot}2142/[\sigma^2(F) + 0{\cdot}001F^2]$, for 1958 reflections with $F \ge 4\sigma(F)$ and 333 parameters. Max. $\Delta/\sigma = 0{\cdot}020$ for any parameter. Max. and min. peaks in final $\Delta\rho$ map 0·24 and −0·23 e Å^{-3}. The inverse structure did not alter the R values. Atomic scattering factors from Cromer & Mann (1968) for C, O and N, and from Stewart, Davidson & Simpson (1965) for H atoms. Geometrical calculations by *PARST* (Nardelli, 1983).

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* main bond lengths and angles as well as hydrogen-bond data are given in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule is shown in Fig. 1 together with the numbering scheme; the left-hand nitrobenzyl group has the same numbering as the right-hand group, but is primed. Fig. 2 illustrates the hydrogen-bond system in the direction of the *b* axis.

* Lists of structure factors, anisotropic thermal parameters, bond distances and angles, torsional angles, least-squares-planes data, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44842 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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