1,25 (2) Å; C—C—O = 117 (2)°] (Borthwick, 1980), probablement à cause de la présence dans le cristal de la liaison hydrogen N(4)···O(1) 2,624 (5) Å. En positionnant H(40) suivant une géométrie standard, dans le plan N(4), N(3), C(5), la liaison H a les caractèristiques suivantes: N(4)—H(40) 1,08 (5), O(1)—H(40) 1,66 (5) Å et N(4)—H(40)—O($1^{\rm i}$)[(i): x, y, $1+z$] 146 (1)°. Etant donné la courte liaison N(4)···O(1), il est probable que la position de H(40) pourrait être améliorée, conduisant à un angle N(4)—H(40)—O($1^{\rm i}$) plus proche de 180°, et donnant à N(4) un caractère quelque peu pyramidal. En particulier si on place H(40) exactement dans l'alignement N(4)···O($1^{\rm i}$), la somme des angles de liaison autour de N(4) vaudrait 356 (1)°. Mais dans ce cas, la position de H(40) serait fort disymétrique par rapport à C(5) et N(3): on aurait N(3)—N(4)—H(40) 111,1 (1) et C(5)—N(4)—H(40) 128,4 (1)°. H(40) occupe donc probablement une position intermédiaire entre les deux extrêmes. Le cycle triazole est plan avec des écarts au plan moyen inférieurs à 1,1σ. N(4) et C(2) sont significativement hors de ce plan, à des distances respectivement égales à −0,075 (4) et 0,184 (4) Å. La somme des angles des liaisons autour de N(3) vaut 359,8 (1) et autour de N(1) 359,3 (1)°. N(3) est distant du plan C(3), C(4), N(4) de 0,029 (3) Å tandis que N(1) s'écarte du plan C(2), C(4), N(2) de −0,066 (3) Å. Ces données mettent en évidence le caractère légèrement pyramidal de N(1) et, dans une moindre mesure, de N(3). La conformation du reste de la molécule peut être définie par les angles de torsion C(8)—C(7)—C(6)—C(5) −57,1 (5), C(7)—C(6)—C(5)—N(4) −72,2 (5), C(6)—C(5)—N(4)—N(3) −170,3 (4), C(5)—N(4)—N(3)—C(3) 90,2 (5), C(4)—N(1)—C(2)—C(1) 81,2 (5) et N(1)—C(2)—C(1)—O(1) −9,6 (4)°. L'angle dièdre C(6)—C(5)—N(4)—N(3) s'écarte de la valeur standard (180°) autour de C—N dans une liaison peptidique, où l'azote a une configuration plane. Mais on trouve également une valeur de −170,0 (7)° pour l'angle de torsion C—C—N—C équivalent dans la structure de l'acide *tert*-butoxycarbonyl-D-alanyl-D-glutamique monohydraté (Dideberg, Lamotte, Dupont & Christiaens, 1981). Par ailleurs, un échantillonnage de structures de peptides publiées en 1987 laisse apparaître des angles ω compris entre 167 et 180° et la plupart des distances C—N sont voisines de 1,33 (1) Å, tandis que les distances C=O sont comprises entre 1,22 et 1,24 Å.

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Structure and Absolute Configuration of Two Stereoisomers of α,α'-[Iminobis(methylene)]bis(3,4-dihydro-2*H*-1-benzopyran-2-methanol) Hydrobromide

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Abstract. α,α'-1,1'-Bis(3,4-dihydro-2*H*-benzopyran-2-yl)-2,2'-iminodiethanol hydrobromide. (I) $C_{22}H_{28}NO_4^+.Br^-$, $M_r = 450{\cdot}37$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}1278\ (1)$, $b = 13{\cdot}1699\ (6)$, $c = 30{\cdot}858\ (2)$ Å, $V = 2083{\cdot}9\ (2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}44$, $D_x = 1{\cdot}436$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ(Cu $K\alpha$) = 2·915 mm^{-1}, $F(000) = 936$, room temperature, final $R = 0{\cdot}054$ for 2086 observed reflections. (II) $C_{22}H_{28}NO_4^+.Br^-$, $M_r = 450{\cdot}37$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}1292\ (2)$, $b = 13{\cdot}1764\ (9)$, $c = 30{\cdot}847\ (3)$ Å, $V = 2084{\cdot}8\ (3)$ Å^3, $Z = 4$, $D_m = 1{\cdot}43$, $D_x = 1{\cdot}435$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ(Cu $K\alpha$) = 2·915 mm^{-1}, $F(000) = 936$, room temperature, final $R = 0{\cdot}054$ for 2676 observed reflections. The two structures are mirror images and the central C—C—N—C—C chain adopts the anti-

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periplanar–synclinal conformation. The active β_1-selective adrenergic receptor blocker [isomer (I)] has the *S,R,R,S* absolute configuration while the inactive isomer (II) has the *R,S,S,R* configuration. Endless chains are formed by (N—)H···Br hydrogen bonds in the **a** direction and by (O—)H···Br hydrogen bonds in the **b** direction.

Introduction. In view of the continued interest in the conformation of β-blocking agents, the crystal structure and absolute configuration of two stereoisomers of the ten possible ones of the title compound were determined. Some of these stereoisomers are very potent β_1-selective adrenergic receptor blockers and besides this they show a direct antihypertensive activity in spontaneously hypertensive rats, not common for classical β-blocking agents (Pauwels, Gommeren, Van Lommen, Janssen & Leysen, 1988).

Experimental. Crystals obtained at room temperature, of (I) from ethyl acetate/methanol and of (II) from ethyl acetate/ethanol. Density measured by flotation. X-ray diffraction data collected on a Hilger & Watts computer-controlled four-circle diffractometer with Ni-filtered Cu $K\alpha$ radiation. Experimental details are set out in Table 1. Lp corrections, absorption correction by the method of North, Phillips & Mathews (1968), scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) (for H), scattering factor for Br corrected for anomalous dispersion (*International Tables for X-ray Crystallography,* 1974). Structure (I) solved with *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, van den Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981) with the Br atom position obtained from a sharpened Patterson synthesis as input. For structure (II) the inverted coordinates of (I) were taken. Refinement with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) by block-diagonal least squares on *F*, first with isotropic temperature factors and then anisotropically; finally two cycles of full-matrix least squares performed; H positions located from ΔF syntheses, but placed in geometrically idealized positions; included in the refinement with fixed coordinates and overall temperature factor. The absolute configurations of the molecules were determined by calculating the Bijvoet coefficient according to a selection procedure by Beurskens, Noordik & Beurskens (1980) at an early stage of the refinement to avoid the possibility of systematic errors in atom coordinates (Jones, 1986). At the end of the refinement the final Bijvoet coefficient was for (I) 0·914 (5) from the 153 largest Bijvoet pairs and for (II) 0·907 (5) from the 235 largest Bijvoet pairs. The pairs were selected using the parameters $N = 1$, $N' = 2$; for larger values of N the Bijvoet coefficients were exactly 1. The computer program *PARST* (Nardelli, 1983*a*) was used to calculate the molecular parameters.

Table 1. *Experimental data*

	(I)	(II)
Crystal dimensions (mm)	0·4 × 0·1 × 0·05	0·5 × 0·2 × 0·1
Lattice parameters		
No. of reflections	24	24
2θ range (°)	$40 < 2\theta < 66$	$40 < 2\theta < 66$
Scan technique	$\omega/2\theta$	$\omega/2\theta$
Scan width (°)	1·20	1·20
Maximum 2θ (°)	140	140
hkl range	0–6, 0–16, −37–37	0–6, 0–16, −37–37
Standard reflections	0,0,10, 041	0,0,10, 060, 200, 237
Intensity variation (%)	1·86, 2·24	0·70, 0·36, 0·33, 0·75
No. of measured reflections	4190	4303
No. of unique reflections (Friedel law not obeyed)	3683	3773
R_{int} (contributing reflections)	0·041 (1014)	0·043 (1060)
No. of observed reflections ($I > 3\sigma_I$)	2086	2676
Transmission factor range	0·993–0·907	0·995–0·789
R	0·054	0·054
wR	0·063	0·070
S	0·111	0·221
w	$(400 + F_o + 0{\cdot}0022F_o^2)^{-1}$	$(50 + \sigma_{F_o}^2 + 0{\cdot}025F_o^2)^{-1}$
$(\Delta/\sigma)_{\text{ave}}$	0·09	0·09
$(\Delta/\sigma)_{\text{max}}$	0·49	0·44
$\Delta\rho$(e Å^{-3})	−1·83 to 1·01 (near Br)	−1·19 to 1·53 (near Br)

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 2, bond lengths, bond angles and selected torsion angles in Table 3.* Fig. 1 shows the molecular geometry and the crystallographic numbering scheme for structure (I).

Since the molecules of both structures consist of two identical units, linked by an imino group, four observations for each bond length and angle were obtained. Estimation of the average molecular dimensions was investigated following Taylor & Kennard (1983). There is a close correspondence between the lengths of equivalent bonds except those where atom C(3′) is involved. Careful inspection of the anisotropic thermal parameters reveals that in (I) C(2′), C(3′), C(10′) and C(11′) have unusually high values for the ratio between the maximum and minimum mean squared displacement along the principal axes of the thermal ellipsoids. Bond lengths resulting from these atomic positions are therefore suspicious and should be rejected in the calculation of the average molecular dimensions. Comparison of the bond angles revealed environmental effects for the angles in α and β positions to N, even if the doubtful atomic positions of C(2′) and C(3′) of (I) are left out of the analysis.

The geometry of the two N—C—C—O fragments in the title compound can be compared with that of

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52152 (54 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s of the refined parameters in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
(I)				
Br	9263 (1)	3216 (1)	7502 (1)	4·04 (2)
N(1)	4157 (15)	4770 (4)	7573 (2)	2·51 (19)
C(2)	4165 (23)	5191 (6)	7120 (2)	2·28 (23)
C(3)	5645 (22)	6186 (7)	7091 (3)	2·79 (25)
O(4)	8105 (13)	6043 (4)	7300 (2)	2·93 (17)
C(5)	6038 (20)	6525 (6)	6618 (3)	2·41 (24)
O(6)	7279 (13)	5677 (4)	6395 (2)	2·78 (18)
C(7)	7583 (19)	5795 (7)	5955 (3)	2·81 (28)
C(8)	6182 (20)	6500 (7)	5716 (3)	2·98 (27)
C(9)	4160 (23)	7173 (7)	5925 (3)	3·22 (25)
C(10)	3539 (16)	6812 (8)	6385 (2)	2·93 (25)
C(11)	9325 (24)	5134 (7)	5756 (3)	3·36 (28)
C(12)	9792 (23)	5188 (8)	5316 (4)	4·27 (37)
C(13)	8430 (25)	5894 (10)	5080 (4)	4·77 (39)
C(14)	6654 (25)	6526 (9)	5271 (3)	4·44 (37)
C(2′)	3237 (20)	5496 (7)	7912 (3)	2·83 (27)
C(3′)	2953 (19)	4933 (7)	8347 (2)	2·20 (23)
O(4′)	5375 (15)	4530 (5)	8473 (2)	3·90 (21)
C(5′)	1998 (19)	5646 (7)	8688 (3)	2·70 (26)
O(6′)	−269 (13)	6152 (5)	8514 (2)	3·23 (19)
C(7′)	−1504 (16)	6801 (7)	8800 (3)	2·72 (24)
C(8′)	−1316 (19)	6716 (8)	9247 (2)	2·96 (26)
C(9′)	456 (23)	5939 (8)	9444 (3)	3·67 (31)
C(10′)	1277 (22)	5135 (7)	9110 (2)	3·27 (30)
C(11′)	−3027 (23)	7549 (7)	8607 (3)	3·17 (28)
C(12′)	−4502 (23)	8188 (8)	8864 (3)	4·16 (29)
C(13′)	−4398 (25)	8113 (8)	9312 (3)	4·43 (32)
C(14′)	−2767 (25)	7391 (8)	9496 (3)	3·74 (32)
(II)				
Br	−9257 (1)	−3213 (1)	−7499 (1)	4·23 (2)
N(1)	−4171 (11)	−4767 (3)	−7568 (1)	2·71 (13)
C(2)	−4183 (17)	−5178 (5)	−7116 (2)	3·02 (18)
C(3)	−5650 (15)	−6172 (5)	−7087 (2)	2·80 (16)
O(4)	−8095 (9)	−6036 (3)	−7301 (1)	2·87 (11)
C(5)	−6038 (15)	−6521 (4)	−6615 (2)	2·79 (17)
O(6)	−7310 (10)	−5679 (3)	−6396 (1)	3·08 (13)
C(7)	−7562 (14)	−5785 (5)	−5957 (2)	2·81 (18)
C(8)	−6190 (16)	−6493 (5)	−5717 (2)	3·40 (19)
C(9)	−4179 (19)	−7181 (5)	−5928 (2)	3·88 (20)
C(10)	−3556 (13)	−6804 (6)	−6385 (2)	3·39 (19)
C(11)	−9340 (19)	−5129 (5)	−5761 (2)	3·94 (21)
C(12)	−9766 (18)	−5185 (6)	−5320 (3)	4·65 (26)
C(13)	−8425 (20)	−5905 (7)	−5076 (2)	5·01 (28)
C(14)	−6603 (19)	−6539 (6)	−5270 (2)	4·48 (25)
C(2′)	−3213 (16)	−5486 (5)	−7910 (2)	2·93 (18)
C(3′)	−2992 (15)	−4928 (5)	−8337 (2)	2·75 (16)
O(4′)	−5397 (12)	−4523 (4)	−8477 (1)	4·25 (15)
C(5′)	−2003 (15)	−5656 (5)	−8691 (2)	3·09 (18)
O(6′)	272 (10)	−6148 (4)	−8515 (1)	3·50 (14)
C(7′)	1509 (13)	−6809 (5)	−8797 (2)	3·05 (17)
C(8′)	1318 (14)	−6722 (6)	−9243 (2)	3·31 (18)
C(9′)	−453 (17)	−5937 (6)	−9443 (2)	3·82 (21)
C(10′)	−1276 (16)	−5126 (6)	−9110 (2)	3·62 (21)
C(11′)	3059 (17)	−7557 (6)	−8600 (2)	3·68 (20)
C(12′)	4522 (18)	−8199 (6)	−8864 (2)	4·31 (21)
C(13′)	4414 (20)	−8100 (6)	−9313 (2)	4·46 (22)
C(14′)	2790 (19)	−7393 (7)	−9496 (2)	4·23 (23)

Table 3. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

	(I)	(II)
N(1)—C(2)	1·506 (11)	1·495 (8)
N(1)—C(2′)	1·492 (12)	1·502 (8)
C(2)—C(3)	1·517 (13)	1·513 (10)
C(2′)—C(3′)	1·541 (13)	1·514 (9)
C(3)—O(4)	1·429 (13)	1·427 (9)
C(3′)—O(4′)	1·405 (12)	1·412 (9)
C(3)—C(5)	1·538 (13)	1·543 (9)
C(3′)—C(5′)	1·493 (13)	1·540 (10)
C(5)—O(6)	1·458 (11)	1·454 (8)
C(5′)—O(6′)	1·444 (12)	1·441 (9)
C(5)—C(10)	1·517 (13)	1·504 (10)
C(5′)—C(10′)	1·511 (13)	1·515 (10)
O(6)—C(7)	1·375 (12)	1·366 (8)
O(6′)—C(7′)	1·383 (11)	1·385 (8)
C(7)—C(8)	1·387 (14)	1·383 (10)
C(7′)—C(8′)	1·390 (13)	1·384 (10)
C(7)—C(11)	1·390 (15)	1·394 (11)
C(7′)—C(11′)	1·390 (14)	1·405 (10)
C(8)—C(9)	1·508 (14)	1·519 (11)
C(8′)—C(9′)	1·497 (15)	1·508 (11)
C(8)—C(14)	1·395 (14)	1·397 (11)
C(8′)—C(14′)	1·390 (15)	1·401 (11)
C(9)—C(10)	1·532 (13)	1·526 (10)
C(9′)—C(10′)	1·537 (14)	1·541 (11)
C(11)—C(12)	1·382 (15)	1·381 (12)
C(11′)—C(12′)	1·382 (15)	1·393 (12)
C(12)—C(13)	1·372 (17)	1·392 (13)
C(12′)—C(13′)	1·388 (16)	1·392 (11)
C(13)—C(14)	1·368 (18)	1·389 (13)
C(13′)—C(14′)	1·387 (17)	1·371 (13)
C(2)—N(1)—C(2′)	114·6 (6)	115·3 (5)
N(1)—C(2)—C(3)	112·0 (7)	111·7 (5)
N(1)—C(2′)—C(3′)	109·4 (7)	109·2 (5)
C(2)—C(3)—O(4)	107·5 (7)	107·6 (5)
C(2′)—C(3′)—O(4′)	109·8 (7)	112·6 (6)
C(2)—C(3)—C(5)	111·8 (7)	112·2 (5)
C(2′)—C(3′)—C(5′)	110·0 (7)	109·8 (5)
O(4)—C(3)—C(5)	110·5 (8)	111·1 (6)
O(4′)—C(3′)—C(5′)	109·5 (7)	107·9 (5)
C(3)—C(5)—O(6)	106·4 (6)	105·6 (5)
C(3′)—C(5′)—O(6′)	106·9 (7)	106·3 (5)
C(3)—C(5)—C(10)	114·2 (8)	114·3 (6)
C(3′)—C(5′)—C(10′)	114·0 (8)	113·5 (6)
O(6)—C(5)—C(10)	109·6 (7)	110·5 (5)
O(6′)—C(5′)—C(10′)	109·3 (8)	109·2 (6)
C(5)—O(6)—C(7)	115·4 (6)	115·1 (5)
C(5′)—O(6′)—C(7′)	114·6 (7)	114·7 (5)
O(6)—C(7)—C(8)	122·8 (8)	123·4 (6)
O(6′)—C(7′)—C(8′)	123·6 (8)	122·7 (6)
O(6)—C(7)—C(11)	116·0 (8)	115·3 (6)
O(6′)—C(7′)—C(11′)	115·0 (8)	115·3 (6)
C(8)—C(7)—C(11)	121·2 (9)	121·3 (6)
C(8′)—C(7′)—C(11′)	121·4 (9)	121·9 (7)
C(7)—C(8)—C(9)	121·5 (8)	121·3 (6)
C(7′)—C(8′)—C(9′)	120·0 (8)	120·3 (6)
C(7)—C(8)—C(14)	116·7 (9)	118·7 (7)
C(7′)—C(8′)—C(14′)	117·3 (9)	117·6 (7)
C(9)—C(8)—C(14)	121·7 (9)	120·0 (7)
C(9′)—C(8′)—C(14′)	122·6 (8)	122·1 (6)
C(8)—C(9)—C(10)	110·9 (8)	110·1 (6)
C(8′)—C(9′)—C(10′)	111·4 (8)	111·7 (6)
C(5)—C(10)—C(9)	109·9 (7)	109·8 (6)
C(5′)—C(10′)—C(9′)	109·7 (8)	108·4 (6)
C(7)—C(11)—C(12)	120·8 (10)	119·8 (7)
C(7′)—C(11′)—C(12′)	119·6 (9)	118·5 (7)
C(11)—C(12)—C(13)	118·0 (11)	119·4 (8)
C(11′)—C(12′)—C(13′)	120·5 (10)	120·2 (8)
C(12)—C(13)—C(14)	121·5 (11)	120·7 (8)
C(12′)—C(13′)—C(14′)	118·6 (10)	119·8 (8)
C(8)—C(14)—C(13)	121·7 (10)	120·1 (8)
C(8′)—C(14′)—C(13′)	122·4 (9)	121·9 (7)
C(2′)—N(1)—C(2)—C(3)	52·8 (10)	−53·4 (8)
C(2)—N(1)—C(2′)—C(3′)	172·1 (7)	−173·0 (6)
N(1)—C(2)—C(3)—O(4)	49·2 (10)	−48·9 (8)
N(1)—C(2′)—C(3′)—O(4′)	60·2 (10)	−59·9 (8)
N(1)—C(2)—C(3)—C(5)	170·6 (8)	−171·3 (6)
N(1)—C(2′)—C(3′)—C(5′)	−179·2 (8)	179·8 (6)
C(2)—C(3)—C(5)—O(6)	−54·2 (10)	55·0 (7)
C(2′)—C(3′)—C(5′)—O(6′)	50·6 (10)	−50·0 (7)
C(2)—C(3)—C(5)—C(10)	66·9 (11)	−66·7 (8)
C(2′)—C(3′)—C(5′)—C(10′)	171·4 (8)	−170·0 (6)

2-aminoethanol. A recent *ab initio* analysis of 2-aminoethanol conformers (Vanquickenborne, Coussens, Verlinde & De Ranter, 1989) showed that the angles in α and β positions to N are affected by the conformation of this three rotor molecule. The Räsänen rule (Räsänen, Aspiala, Homanen & Murto. 1982) 'if in a conformer of a primary alcohol or amine a C—C or C—H is *trans* to an *X*—H (*X*=O, N) bond, the corresponding *X*CC or *X*CH angle will be considerably smaller than for the other configurations', confirmed in the above mentioned *ab initio* analysis, can be extended to the present case. The C(2)—C(3)—O(4) angle where the O(4)—H(4) bond is antiperiplanar to C(2)—C(3), is smaller than the C(2′)—C(3′)—O(4′) angle where the O(4′)—H(4′) bond is synclinal.

The difference in the angles around C(2) and C(2′) can be compared with the situation in equatorial and axial *N*-methylpiperidine (Profeta, 1978). In the axial conformation the internal α angles (NCC) are opened to 114° because of interaction of a methyl hydrogen with axial hydrogens on position β. In our study the N(1)—C(2)—C(3) angle is enlarged to 112° because of interaction between H(3) and H(2′1) (2·34 Å).

The observed conformation of the molecule in the crystal structure is determined by intramolecular hydrogen bonds (Table 4). Two (N—)H···O hydrogen bonds stabilize the synclinal/antiperiplanar conformation of the C—C—N—C—C bridging chain of the two chromane moieties. Apart from these classical hydrogen bonds two intramolecular (C—)H···O contacts are present. These can reasonably be described as hydrogen bonds as they satisfy the description established by Taylor & Kennard (1982), *i.e.* the donor (C—)H group has a decreased electron density resulting from the inductive effect of the adjacent positively charged N atom. These (C—)H···O hydrogen bonds hold the C(2)—C(3)—C(5)—O(6) and C(2′)—C(3′)—C(5′)—O(6′) dihedral angles in a synclinal position. From puckering parameters (Cremer & Pople, 1975) and asymmetry parameters (Nardelli, 1983*b*) the 3,4-dihydropyran rings adopt a half-chair form with a twofold axis intersecting the C(5)—C(10) and C(5′)—C(10′) bonds.

The packing of the crystal (Fig. 2) results mainly from intermolecular hydrogen bonds (Table 3). (N—)H···Br, (O—)H···O and weaker (C—)H···O hydrogen bonds form endless chains in the **a** direction and (O—)H···Br hydrogen bonds in the **b** direction.

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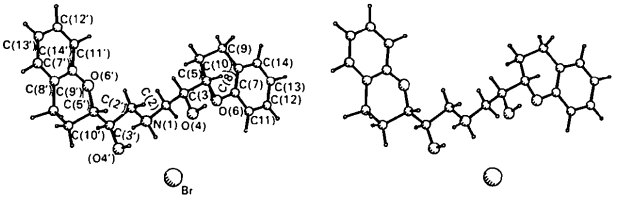

Fig. 1. Perspective view (Motherwell & Clegg, 1978) of the molecule (I) with atomic numbering scheme.

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

			(I)				(II)			
A	*B*	*C*	*AB*	*BC*	*AC*	*ABC*	*AB*	*BC*	*AC*	*ABC*
N(1)—	H(12)···	Br	1·06	2·45	3·331 (7)	139	1·06	2·46	3·322 (5)	138
N(1)—	H(1)···	Br^{i}	1·05	2·27	3·246 (7)	154	1·05	2·28	3·254 (5)	154
O(4)—	H(4)···	Br^{ii}	0·95	2·32	3·222 (6)	159	0·96	2·32	3·232 (5)	159
N(1)—	H(12)···	O(4)	1·06	2·43	2·76 (1)	96	1·06	2·42	2·743 (7)	96
N(1)—	H(12)···	O(4′)	1·06	2·57	2·86 (1)	95	1·06	2·58	2·894 (8)	96
C(2)—	H(21)···	O(6)	1·09	2·34	2·82 (1)	104	1·07	2·35	2·818 (9)	105
C(2′)—	H(2′2)···	O(6′)	1·04	2·36	2·72 (1)	98	1·04	2·37	2·728 (9)	99
C(2)—	H(22)···	O(4′)	1·09	2·50	3·35 (1)	134	1·09	2·50	3·37 (1)	136
C(2′)—	H(2′2)···	O(4′)	1·04	2·36	3·32 (1)	152	1·04	2·36	3·309 (9)	151
O(4′)—	H(4′)···	$O(6'^{iii})$	0·95	2·15	3·09 (1)	172	0·95	2·15	3·087 (8)	172

Symmetry code: (i) (I) $x-1, y, z$ (II) $x+1, y, z$; (ii) (I) $-x+2, y+0·5, -z+1·5$ (II) $-x-2, y-0·5, -z-1·5$; (iii) (I) $x+1, y, z$ (II) $x-1, y, z$.

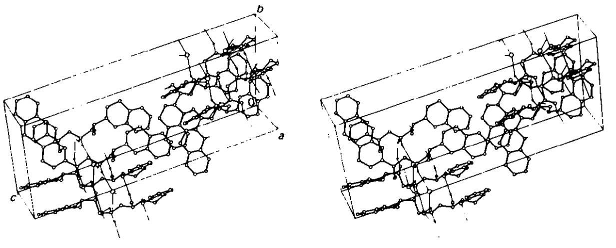

Fig. 2. Stereoscopic packing diagram (Motherwell & Clegg, 1978) of (I) with hydrogen bonds shown as thin lines.

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