Constitution and Absolute Stereochemistry of the Biphenyl Alkaloid Lythranine: *X*-Ray Analysis of the Crystal Structure of Bromolythranine Hydrobromide Ethanol Solvate ‡

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The constitution and stereochemistry of lythranine. a biphenyl alkaloid from *Lythrum anceps*. has been defined by *X*-ray analysis of the crystal structure of bromolythranine hydrobromide ethanol solvate. In the biphenyl system of the alkaloid. the dihedral angle between the benzene planes is 75°. The piperidinium ring has a chair conformation with torsion angles in the range 52—60°, mean 56°. The crystals are orthorhombic. space group $P2_12_12_1$. with $Z = 4$ in a cell of dimensions $a = 21{\cdot}768(17)$, $b = 15{\cdot}223(12)$, $c = 9{\cdot}873(7)$ Å. The atomic co-ordinates were determined by Fourier and least-squares calculations. and the analysis terminated at R 7·7% over 1443 reflections. The absolute configuration was established by the anomalous dispersion effect.

SEVERAL new biphenyl alkaloids were recently isolated from *Lythrum anceps* by Fujita and his colleagues,[1] and one of the alkaloids, lythranine, was assigned the constitution (1a) or (1b). We undertook a crystal-structure analysis of bromolythranine hydrobromide in order to establish the complete structure and stereochemistry of the alkaloid. The diffraction data were obtained by means of a computer-controlled diffractometer, and the atomic co-ordinates deduced by Fourier and least-squares methods; isotropic vibration parameters were used for all except the bromine atoms, and the refinement converged at R 7·7% over **1433** reflections. The absolute configuration was determined by the anomalous dispersion effect.

The atomic co-ordinates are listed in Table 1, and the interatomic distances, valency angles, *etc.*, are in Table 2. The arrangement of atoms in the alkaloid cation is shown in the Figure, and the results summarized by this Figure prove that bromolythranine has the constitution and absolute stereochemistry (1c), from which it follows that lythranine is represented by (1a). The absolute stereochemistry of another biphenyl alkaloid, lythridine, has been established by *X*-ray measurements,[2] and assignments of a number of these alkaloids have been made on the basis of circular dichroism spectra.[3]

The biphenyl moiety in bromolythranine hydrobromide is very considerably skewed about the central C(18)–C(20) bond, the dihedral angle between the mean planes of the benzene rings being **75°** and the O(1) · · · O(2) distance **3·39** Å. A more nearly planar biphenyl would be expected to be destabilized by repulsion between the *ortho,ortho*-substituents O(1) and O(2). Bromomyricanol has a biphenyl system bridged across

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‡ No reprint available.

[1] E. Fujita, K. Bessho, K. Fuji, and A. Sumi, *Chem. and Pharm. Bull.* (*Japan*), 1970, **18**, 2215; E. Fujita and K. Fuji, *J. Chem. Soc.* (*C*), 1971, 1651.

[2] S. C. Chu, G. A. Jeffrey, B. Douglas, J. L. Kirkpatrick, and J. A. Weisbach, *Chem. and Ind.*, 1966, 1795.

[3] J. P. Ferris, C. B. Boyce, R. C. Briner, U. Weiss, I. H. Qureshi, and N. E. Sharpless, *J. Amer. Chem. Soc.*, 1971, **93**, 2963.

TABLE 1

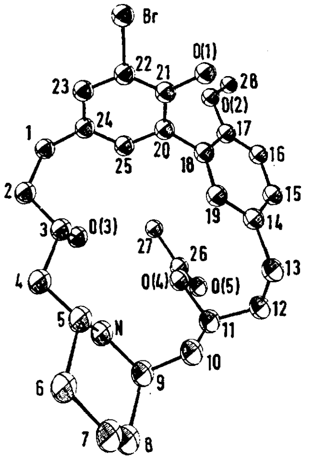
Atomic co-ordinates (as fractions of the cell edges) and thermal parameters (Å²), with standard deviations in parentheses

	x	*y*	*z*	*B*
Br(1)	0·4758(1)	0·8163(1)	−0·2784(3)	*
Br(2)	0·2373(1)	0·1545(2)	−0·0616(4)	*
O(1)	0·3433(6)	0·1824(9)	0·1298(14)	5·29(31)
O(2)	0·3398(7)	0·3183(11)	0·4024(15)	6·11(36)
O(3)	0·3777(7)	0·6008(9)	−0·0677(17)	5·91(38)
O(4)	0·5102(6)	0·5664(7)	0·1219(13)	3·57(28)
O(5)	0·5352(6)	0·6499(8)	0·3022(13)	4·81(31)
O(6) †	0·8498(10)	0·7286(13)	0·1095(22)	10·66(61)
N	0·4975(7)	0·6172(9)	−0·1716(15)	3·57(28)
C(1)	0·2588(13)	0·5060(16)	−0·0938(27)	6·66(63)
C(2)	0·3001(11)	0·5325(13)	−0·2188(27)	5·82(55)
C(3)	0·3673(10)	0·5375(12)	−0·1796(22)	4·22(49)
C(4)	0·4058(10)	0·5666(13)	−0·3045(23)	4·90(52)
C(5)	0·4713(9)	0·5531(10)	−0·2760(22)	3·57(41)
C(6)	0·5156(11)	0·5636(14)	−0·4051(23)	5·45(55)
C(7)	0·5812(10)	0·5525(14)	−0·3807(24)	5·35(57)
C(8)	0·6030(10)	0·6255(13)	−0·2732(25)	5·58(55)
C(9)	0·5646(9)	0·6156(12)	−0·1333(22)	3·96(48)
C(10)	0·5832(9)	0·5308(13)	−0·0578(22)	4·24(47)
C(11)	0·5751(10)	0·5513(13)	0·0973(22)	4·50(51)
C(12)	0·6041(9)	0·4790(11)	0·1883(19)	3·22(44)
C(13)	0·5772(9)	0·3848(12)	0·1686(21)	4·06(48)
C(14)	0·5153(9)	0·3690(11)	0·2327(20)	3·75(42)
C(15)	0·5076(9)	0·3576(13)	0·3754(21)	4·12(47)
C(16)	0·4512(8)	0·3407(12)	0·4350(20)	3·83(43)
C(17)	0·3995(10)	0·3338(14)	0·3563(22)	4·60(49)
C(18)	0·4052(8)	0·3453(11)	0·2129(21)	3·22(40)
C(19)	0·4619(9)	0·3639(11)	0·1559(18)	2·84(40)
C(20)	0·3498(9)	0·3427(14)	0·1251(20)	4·39(49)
C(21)	0·3230(9)	0·2609(13)	0·0849(22)	3·92(47)
C(22)	0·2761(11)	0·2644(16)	−0·0076(28)	6·06(62)
C(23)	0·2522(10)	0·3396(14)	−0·0662(23)	5·71(52)
C(24)	0·2781(11)	0·4227(14)	−0·0261(24)	4·92(54)
C(25)	0·3272(10)	0·4199(12)	0·0632(23)	4·30(48)
C(26)	0·4958(10)	0·6149(12)	0·2313(22)	3·95(45)
C(27)	0·4266(10)	0·6186(12)	0·2573(22)	4·92(52)
C(28)	0·3342(10)	0·3139(15)	0·5486(23)	5·89(54)
C(29) †	0·7952(18)	0·6870(26)	0·1747(38)	15·37(124)
C(30) †	0·7860(27)	0·6200(36)	0·0822(62)	15·75(199)

* The bromine atoms were assigned anisotropic temperature factors of the form: $T = \exp[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + 2b_{12}hk + 2b_{13}hl + 2b_{23}kl)]$ with parameters ($\times 10^4$):

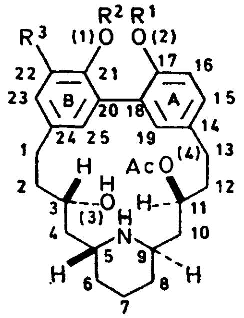
	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Br(1)	56(1)	32(1)	126(3)	− 1(1)	−12(2)	14(2)
Br(2)	35(1)	82(2)	383(7)	−13(1)	−39(2)	−63(3)

† Ethanol.



The atomic arrangement in bromolythranine.

the *m,m*-positions by a seven-membered chain of carbon atoms, and the constraint of this short bridge results in a dihedral angle of only 33° between the benzene planes, the consequent short O ··· O separation of 2·7 Å between *ortho,ortho*-substituents being made acceptable by hydrogen bonding.[4] The valency angles at the carbon atoms of the central bond of the biphenyl in bromomyricanol, moreover, exhibit marked departures from 120°, whereas in bromolythranine the greater



	R¹	R²	R³
(1a)	Me	H	H
(1b)	H	Me	H
(1c)	Me	H	Br

TABLE 2

Interatomic distances (Å) and angles (deg.)

(*a*) Bond lengths

C(1)–C(2)	1·578(37)	C(16)–C(17)	1·370(29)
C(1)–C(24)	1·493(33)	C(17)–C(18)	1·430(30)
C(2)–C(3)	1·514(32)	C(17)–O(2)	1·398(26)
C(3)–C(4)	1·554(31)	C(18)–C(19)	1·384(26)
C(3)–O(3)	1·482(26)	C(18)–C(20)	1·486(28)
C(4)–C(5)	1·468(30)	C(19)–C(14)	1·390(27)
C(5)–C(6)	1·605(31)	C(20)–C(21)	1·431(28)
C(5)–N	1·529(24)	C(20)–C(25)	1·412(29)
C(6)–C(7)	1·457(33)	C(21)–C(22)	1·371(33)
C(7)–C(8)	1·608(32)	C(22)–C(23)	1·384(33)
C(8)–C(9)	1·620(32)	C(22)–Br(2)	1·948(25)
C(9)–N	1·509(25)	C(23)–C(24)	1·442(31)
C(9)–C(10)	1·545(28)	C(24)–C(25)	1·387(32)
C(10)–C(11)	1·570(30)	O(2)–C(28)	1·449(27)
C(11)–C(12)	1·556(27)	O(4)–C(26)	1·344(24)
C(11)–O(4)	1·452(25)	O(5)–C(26)	1·228(25)
C(12)–C(13)	1·561(25)	C(26)–C(27)	1·529(30)
C(13)–C(14)	1·508(29)	O(1)–C(21)	1·350(24)
C(14)–C(15)	1·428(28)	O(6)–C(29)	1·492(44)
C(15)–C(16)	1·386(27)	C(29)–C(30)	1·383(69)

(*b*) Other interatomic distances (<3·7 Å)

O(6) ··· O(3^{I})	2·70	C(24) ··· C(2^{IV})	3·54
O(5) ··· O(1^{II})	2·77	O(1) ··· C(7^{V})	3·56
N ··· O(3)	2·81	C(29) ··· O(1^{II})	3·58
N ··· O(4)	3·01	C(25) ··· C(2^{IV})	3·58
O(5) ··· C(6^{III})	3·20	O(5) ··· C(7^{III})	3·60
N ··· Br(1)	3·24	C(29) ··· O(2^{II})	3·63
O(5) ··· C(18^{II})	3·25	O(6) ··· C(3^{I})	3·65
O(6) ··· Br(1^{I})	3·28	C(29) ··· C(21^{II})	3·68
O(5) ··· C(19^{II})	3·29	O(5) ··· C(21^{II})	3·69
O(2) ··· C(1^{IV})	3·43	C(28) ··· C(1^{IV})	3·69
O(5) ··· C(17^{II})	3·51	C(26) ··· C(6^{III})	3·69
O(5) ··· C(14^{II})	3·53	C(13) ··· Br(2^{VI})	3·69

The superscripts refer to the following transformations of the atomic co-ordinates:

I	$\frac{1}{2} + x, \frac{3}{2} - y, -z$	IV	$\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$
II	$1 - x, \frac{1}{2} + y, \frac{1}{2} - z$	V	$1 - x, -\frac{1}{2} + y, -\frac{1}{2} - z$
III	$x, y, 1 + z$	VI	$\frac{1}{2} + x, \frac{1}{2} - y, -z$

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[4] M. J. Begley and D. A. Whiting, *Chem. Comm.*, 1970, 1207.

TABLE 2 (*Continued*)

(*c*) Valency angles

C(24)–C(1)–C(2)	114·0(2·1)	C(16)–C(17)–O(2)	126·3(1·9)
C(1)–C(2)–C(3)	111·3(2·1)	C(18)–C(17)–O(2)	115·0(1·8)
C(2)–C(3)–C(4)	109·4(1·8)	C(17)–C(18)–C(19)	120·3(1·8)
C(2)–C(3)–O(3)	111·8(1·7)	C(17)–C(18)–C(20)	120·1(1·7)
C(4)–C(3)–O(3)	108·8(1·6)	C(19)–C(18)–C(20)	119·5(1·8)
C(3)–C(4)–C(5)	109·4(1·8)	C(18)–C(19)–C(14)	122·4(1·8)
C(4)–C(5)–C(6)	114·7(1·8)	C(18)–C(20)–C(21)	121·0(1·8)
C(4)–C(5)–N	113·7(1·6)	C(18)–C(20)–C(25)	120·8(1·8)
C(6)–C(5)–N	104·3(1·5)	C(21)–C(20)–C(25)	117·6(1·8)
C(5)–C(6)–C(7)	116·5(1·9)	C(20)–C(21)–C(22)	117·1(1·9)
C(6)–C(7)–C(8)	108·6(1·8)	C(20)–C(21)–O(1)	123·2(1·8)
C(7)–C(8)–C(9)	110·1(1·6)	C(22)–C(21)–O(1)	119·7(1·8)
C(8)–C(9)–N	106·6(1·6)	C(21)–C(22)–C(23)	126·1(2·2)
C(8)–C(9)–C(10)	110·7(1·6)	C(21)–C(22)–Br(2)	118·2(1·7)
N–C(9)–C(10)	112·8(1·5)	C(23)–C(22)–Br(2)	115·7(1·8)
C(9)–C(10)–C(11)	105·9(1·6)	C(22)–C(23)–C(24)	117·7(2·1)
C(10)–C(11)–C(12)	112·0(1·6)	C(23)–C(24)–C(25)	116·6(1·9)
C(10)–C(11)–O(4)	107·7(1·6)	C(23)–C(24)–C(1)	120·9(2·1)
C(12)–C(11)–O(4)	114·3(1·6)	C(25)–C(24)–C(1)	121·8(2·0)
C(11)–C(12)–C(13)	115·2(1·6)	C(24)–C(25)–C(20)	124·7(1·8)
C(12)–C(13)–C(14)	115·5(1·6)	O(4)–C(26)–O(5)	122·3(1·8)
C(13)–C(14)–C(15)	122·6(1·8)	O(4)–C(26)–C(27)	112·6(1·7)
C(13)–C(14)–C(19)	121·9(1·8)	O(5)–C(26)–C(27)	125·1(1·9)
C(15)–C(14)–C(19)	115·6(1·7)	C(17)–O(2)–C(28)	114·2(1·6)
C(14)–C(15)–C(16)	123·0(1·8)	C(11)–O(4)–C(26)	116·6(1·5)
C(15)–C(16)–C(17)	120·1(1·9)	C(5)–N–C(9)	121·3(1·4)
C(16)–C(17)–C(18)	118·7(1·9)	C(30)–C(29)–O(6)	98·3(3·4)

(*d*) Torsion angles. The angle *A–B–C–D* is defined as positive if, when viewed along the *B–C* bond, atom *A* must be rotated clockwise to eclipse atom *D*. The standard deviations are *ca*. 2·5°

C(24)–C(1)–C(2)–C(3)	−55	C(14)–C(13)–C(12)–C(11)	−76
C(1)–C(2)–C(3)–C(4)	−178	C(13)–C(12)–C(11)–C(10)	−60
C(1)–C(2)–C(3)–O(3)	−58	C(13)–C(12)–C(11)–O(4)	63
C(2)–C(3)–C(4)–C(5)	−168	C(12)–C(11)–C(10)–C(9)	−169
O(3)–C(3)–C(4)–C(5)	70	O(4)–C(11)–C(10)–C(9)	65
C(3)–C(4)–C(5)–C(6)	169	C(11)–C(10)–C(9)–C(8)	149
C(3)–C(4)–C(5)–N	−71	C(11)–C(10)–C(9)–N	−92
C(4)–C(5)–C(6)–C(7)	179	C(10)–C(9)–C(8)–C(7)	70
N–C(5)–C(6)–C(7)	54	N–C(9)–C(8)–C(7)	−53
C(5)–C(6)–C(7)–C(8)	−60	C(9)–C(8)–C(7)–C(6)	59
C(4)–C(5)–N–C(9)	−178	C(10)–C(9)–N–C(5)	−67
C(6)–C(5)–N–C(9)	−52	C(8)–C(9)–N–C(5)	55
C(2)–C(1)–C(24)–C(25)	79	C(12)–C(13)–C(14)–C(19)	106
C(2)–C(1)–C(24)–C(23)	−92	C(12)–C(13)–C(14)–C(15)	−75
C(1)–C(24)–C(25)–C(20)	−177	C(13)–C(14)–C(19)–C(18)	177
C(23)–C(24)–C(25)–C(20)	−6	C(15)–C(14)–C(19)–C(18)	−3
C(24)–C(25)–C(20)–C(18)	176	C(14)–C(19)–C(18)–C(20)	179
C(24)–C(25)–C(20)–C(21)	5	C(14)–C(19)–C(18)–C(17)	2
C(25)–C(20)–C(21)–C(22)	−2	C(19)–C(18)–C(17)–C(16)	−1
C(25)–C(20)–C(21)–O(1)	175	C(19)–C(18)–C(17)–O(2)	178
C(18)–C(20)–C(21)–O(1)	4	C(20)–C(18)–C(17)–O(2)	2
C(18)–C(20)–C(21)–C(22)	−173	C(20)–C(18)–C(17)–C(16)	−177
C(23)–C(22)–C(21)–C(20)	0	C(15)–C(16)–C(17)–C(18)	−1
C(23)–C(22)–C(21)–O(1)	−177	C(15)–C(16)–C(17)–O(2)	−179
C(21)–C(22)–C(23)–C(24)	−1	C(17)–C(16)–C(15)–C(14)	0
C(22)–C(23)–C(24)–C(25)	3	C(16)–C(15)–C(14)–C(19)	1
C(22)–C(23)–C(24)–C(1)	174	C(16)–C(15)–C(14)–C(13)	−178
Br(2)–C(22)–C(21)–C(20)	−179	C(28)–O(2)–C(17)–C(18)	−175
Br(2)–C(22)–C(21)–O(1)	4	C(28)–O(2)–C(17)–C(16)	3
Br(2)–C(22)–C(23)–C(24)	178	C(26)–O(4)–C(11)–C(12)	78
C(17)–C(18)–C(20)–C(21)	−80	C(26)–O(4)–C(11)–C(10)	−156
C(17)–C(18)–C(20)–C(25)	109	C(11)–O(4)–C(26)–O(5)	5
C(19)–C(18)–C(20)–C(25)	−67	C(11)–O(4)–C(26)–C(27)	−174
C(19)–C(18)–C(20)–C(21)	104		

(*e*) Displacements of the atoms from the benzene planes

(i) Ring A, C(14)—(19):

C(14) 0·01, C(15) 0·00, C(16) −0·01, C(17) 0·00, C(18) 0·01, C(19) −0·01, C(20) −0·05, C(23) −0·13, O(2) −0·02, C(28) −0·13, C(25) −1·21, C(21) 1·12, C(13) 0·06

(ii) Ring B, C(20)—(25)

C(20) 0·01, C(21) 0·00, C(22) −0·01, C(23) −0·01, C(24) 0·02, C(25) −0·03, C(18) −0·13, C(15) −0·38, O(1) −0·05, Br(2) 0·02, C(19) −1·34, C(17) 1·00, C(1) −0·10

flexibility of the longer eleven-membered bridging chain permits the valency angles at C(18) and C(20) to remain close to the ideal value of 120°. The central C(18)–C(20) bond in bromolythranine is slightly bent from the plane of the benzene ring B, for C(18) and C(15) are displaced to the same side of that ring by 0·13 and 0·38 Å. The displacement of the C(18)–C(20) bond from the plane of benzene ring A is smaller, and possibly not significant, for C(20) and C(23) deviate by only 0·05 and 0·13 Å from the benzene plane. A similar feature was noted in the biphenyl system of bromomyricanol.[4]

The piperidinium ring has a conformation of the chair type with torsion angles in the range 52—60°, mean 56°. The valency angles in the ring have a mean value of 111·2°.

Within the macrocyclic ring many of the corresponding torsion angles in the left-hand [C(20) · · · N] and right-hand [C(18) · · · N] branches are alike: the pair C(1)–C(2)–C(3)–C(4), −178°, and C(13)–C(12)–C(11)–C(10), −60°, provides a notable exception.

The C(sp^3)–O and C(sp^2)–O bonds have mean lengths of 1·475 and 1·364 Å. The C(sp^3)–C(sp^3) and C(sp^2)–C(sp^3) bonds have mean lengths of 1·553 and 1·510 Å, and the mean length of the aromatic C–C bonds in the biphenyl system is 1·401 Å. These distances are all close to expected values.[5]

The N · · · O(3) distance is 2·81 Å and probably represents a hydrogen-bonded interaction, for the N · · · O(4) distance is 3·01 Å. The nitrogen atom is also associated with the bromide ion (3·24 Å). The ethanol molecule of solvation takes part in two hydrogen-bonded contacts: O(6) · · · Br(1^{I}) 3·28, and O(6) · · · O(3) 2·70 Å. The alkaloid cations are also directly linked by a hydrogen bond O(5) · · · O(1^{II}) of length 2·77 Å.

The thermal parameters (*B*) of the ethanol atoms are considerably higher than those of the other atoms in the crystal, and it is likely that there is only partial occupancy of the ethanol sites.

EXPERIMENTAL

Crystal Data.—$C_{28}H_{37}Br_2NO_5,C_2H_6O$, $M = 673·56$. Orthorhombic, $a = 21·768(17)$, $b = 15·223(12)$, $c = 9·873(7)$ Å, $U = 3272$ Å^3, $Z = 4$, $D_c = 1·37$, $F(000) = 1392$. Space group $P2_12_12_1$ (D_2^4). Mo-K_α radiation, $\lambda = 0·7107$ Å; μ(Mo-K_α) = 26·7 cm^{-1}.

Crystallographic Measurements.—When exposed to air for a few hours, the crystals became opaque (presumably through loss of solvent) and the diffraction pattern deteriorated. To minimize this effect the crystal employed in the data collection was coated with Canada Balsam. The cell dimensions were determined initially from precession photographs, and were subsequently adjusted by a least-squares treatment of the θ, χ, φ setting angles of 12 reflections measured with Mo-K_α radiation on a Hilger and Watts' Y 290 four-circle automatic diffractometer controlled by a PDP 8 computer. In order to eliminate the effects of multiple reflections,[6] the crystal was placed in an arbitrary

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[6] W. H. Zachariasen, *Acta Cryst.*, 1965, **18**, 705.

orientation with respect to the axes of the instrument. The intensity data were collected by the θ—2θ step scan procedure; 50 steps of 0·02° in θ were used, with 1 s counts at each step, and background counts were taken for 12·5 s at each end of the scan range. The intensities of two standard reflections were monitored periodically, and used to ensure that all the reflections were on a common scale. The equivalent forms hkl, $\bar{h}kl$, and $\bar{h}\bar{k}l$ were collected and averaged, and 1433 independent intensities with $I > 2{\cdot}5\sigma(I)$ were obtained. The intensities were corrected for Lorentz and polarization effects, but absorption was neglected.

Structure Analysis.—Initial co-ordinates for the two bromine atoms were obtained from the Patterson function, and the carbon, nitrogen, and oxygen atoms of the alkaloid molecule were located in an electron-density distribution. After one round of least-squares adjustment of the atomic co-ordinates and isotropic temperature factors by means of the full-matrix programme ORFLS R was 22%, and a difference electron-density distribution then revealed the carbon and oxygen atoms of an ethanol molecule of solvation in the crystal structure. The additional three atoms were included in the structure-factor calculations, and one round of least-squares refinement of the atomic parameters gave R 18·6%. The bromine atoms were then assigned anisotropic temperature factors, and two further cycles of refinement gave R 12·4%.

We next allowed for anomalous dispersion with values of $\Delta f'$ and $\Delta f''$ for bromine taken from ref. 7. The co-ordinates appropriate to the absolute configuration shown in (1) led to R 11·5%, whereas the co-ordinates appropriate to the mirror image yielded R 12·3%. One cycle of least-squares refinement of the two models gave R 10·0% for configuration (1) and 11·1% for the mirror image. Hamilton's test showed that this difference is highly significant.[8] The absolute stereochemistry indicated by these calculations agrees with that deduced by Fujita on the basis of c.d. measurements.[9]

Several further rounds of least-squares calculations reached convergence at R 7·7%. The weighting scheme used in the calculations was $\sqrt{w} = (A + B|F_o|)^{-1}$, where final values adopted for the parameters were $A = 5{\cdot}2$ and $B = 0$ when $|F_o| \leqslant 25{\cdot}8$, and $A = 1{\cdot}5$ and $B = 0{\cdot}11$ when $|F_o| > 25{\cdot}8$. The calculations were performed at the S.R.C. Atlas Laboratory, with the local version of the '*X*-Ray '63' system of programmes.

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