

## (*R,R*)-Labetalol Hydrochloride: \*Determination of the Absolute Configuration of the High-Melting Polymorph, C<sub>19</sub>H<sub>25</sub>N<sub>2</sub>O<sub>3</sub><sup>+</sup>·Cl<sup>-</sup>

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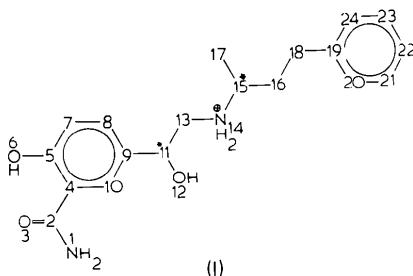
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**Abstract.**  $M_r = 364.9$ , orthorhombic,  $P2_12_12_1$ ,  $a = 31.04$  (6),  $b = 19.21$  (4),  $c = 6.57$  (3) Å,  $V = 3888.2$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.25$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54051$  Å,  $\mu = 1.796$  mm<sup>-1</sup>,  $F(000) = 1552$ ,  $T = 293$  K. Final  $R = 0.075$  for 3766 observed reflexions. The absolute configuration was determined as *RR*. There are two crystallographically independent molecules which have different conformations in the propyl chain. Both molecules adopt a conformation intermediate between 'perpendicular-trans' and 'planar-trans'.

**Introduction.** The crystal studied was that of one of the four isomers present in the adrenoceptor-blocking drug labetalol hydrochloride (Brittain, Drew & Levy, 1982). The single enantiomer ( $[\alpha]_D^{20.5^\circ\text{C}} = -29.2^\circ$  [MeOH, 5.1 g dm<sup>-3</sup>]) (Hartley, Lunts, Collins, Hallett, Clifton & Wicks, 1982) was shown to be >99% isomerically pure as determined by GLC (Munro, Hunt, Rowe & Evans, 1978). This compound crystallizes in at least two polymorphs, and the one studied here (I) had a melting point of 468–469 K. The structure analysis has shown the absolute configuration to be *RR*.



**Experimental.** (I) recrystallized from methanol,  $\sim 0.5 \times 0.4 \times 0.3$  mm. Systematic absences indicated space group  $P2_12_12_1$ . Data collected for  $\pm h k 0 - 5$  with  $\theta_{\text{max}} = 60^\circ$  on a Stoe Stadi-2 two-circle diffractometer,

\* 2-Hydroxy-5-[1-hydroxy-2-(1-methyl-3-phenylpropylamino)-ethyl]benzamide hydrochloride.

graphite-monochromated Cu  $K\alpha$  radiation. 5090 data, 3770 reflexions with  $I > 3\sigma(I)$  used in subsequent calculations. No absorption correction. Structure solution using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). *SHELX76* (Sheldrick, 1976) used for subsequent calculations. Complex neutral-atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974). Weighted full-matrix least-squares refinement (including isotropic H atoms) converged at  $R = 0.075$  for 3766 observed reflexions,  $R_w = 0.084$ ;  $w = 1.00/\sigma^2(F_o) + 0.011692F_o^2$ . In final cycle all shifts in parameters less than their standard deviations.  $R$  for enantiomeric structure refined to 0.083 ( $R_w = 0.097$ ).

**Discussion.** Atomic parameters are given in Table 1 and bond distances and angles in Table 2.†

The crystal contains two independent cations (*A* and *B*) closely related by an approximate non-crystallographic translation of  $\mathbf{b}/4$  (Fig. 1), *i.e.* about 5 Å. They are in very similar crystal environments and the hydrogen-bonding scheme is essentially the same for both, differing only in the lengths of the bonds. A schematic diagram of the hydrogen bonding is shown in Fig. 2. The  $\mathbf{c}$  translation results in  $A \cdots A$  and  $B \cdots B$  hydrogen bonds (amide $\cdots$ HOAr).  $A \cdots B$  hydrogen bonding occurs through amide $\cdots$ amide contacts round the  $2_1$  ( $z$ ) axis and they also share common chloride anions.

The two independent cations have fairly similar conformations although there are important differences. In one molecule (*B*) the aliphatic chain is essentially fully extended since the torsion angles (Table 3) differ by an average of only  $5^\circ$  from a perfectly staggered arrangement. In the other molecule (*A*) most of the backbone is extended, but at one point [C(15)–C(16)

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38505 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\times 10^4$ ) with *e.s.d.*'s in parentheses
$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$U_{eq}$ ( $\text{\AA}^2$ )
<b>Molecule A</b>				
C(2)	-3156 (2)	-1339 (3)	-7084 (11)	284 (33)
C(4)	-3574 (2)	-1675 (3)	-6485 (11)	279 (41)
C(5)	-3632 (2)	-1857 (3)	-4430 (11)	303 (40)
C(7)	-4006 (2)	-2204 (4)	-3808 (12)	448 (46)
C(8)	-4320 (2)	-2365 (3)	-5216 (12)	449 (43)
C(9)	-4275 (2)	-2170 (3)	-7278 (11)	305 (40)
C(10)	-3894 (2)	-1832 (3)	-7900 (10)	221 (31)
C(11)	-4623 (2)	-2363 (3)	-8840 (11)	277 (33)
C(13)	-5071 (2)	-2188 (3)	-8052 (12)	313 (39)
C(15)	-5871 (2)	-2276 (4)	-8842 (15)	476 (51)
C(16)	-6189 (3)	-2048 (5)	-10338 (20)	761 (73)
C(17)	-5949 (2)	-2990 (4)	-7926 (16)	608 (54)
C(18)	-6207 (4)	-2465 (6)	-12079 (20)	950 (87)
C(19)	-6600 (3)	-2203 (5)	-13611 (16)	710 (68)
C(20)	-6543 (4)	-1567 (5)	-14605 (18)	838 (77)
C(21)	-6845 (3)	-1356 (5)	-16117 (17)	653 (60)
C(22)	-7187 (3)	-1736 (6)	-16559 (17)	749 (71)
C(23)	-7270 (3)	-2343 (6)	-15458 (19)	796 (77)
C(24)	-6988 (3)	-2575 (5)	-13979 (17)	753 (67)
Cl(1)	-5317 (1)	-968 (1)	-2815 (3)	424 (11)
N(1)	-3065 (2)	-1230 (3)	-9038 (10)	428 (36)
N(14)	-5406 (1)	-2261 (2)	-9672 (9)	285 (29)
O(3)	-2890 (1)	-1155 (3)	-5756 (8)	438 (30)
O(6)	-3334 (1)	-1708 (3)	-2998 (8)	429 (31)
O(12)	-4580 (2)	-3090 (2)	-9234 (9)	558 (34)
<b>Molecule B</b>				
C(52)	-3033 (2)	571 (3)	-6885 (12)	335 (38)
C(54)	-3468 (2)	336 (3)	-6377 (10)	264 (36)
C(55)	-3563 (2)	113 (4)	-4328 (12)	351 (41)
C(57)	-3954 (2)	-168 (3)	-3843 (11)	362 (41)
C(58)	-4277 (2)	-205 (3)	-5272 (12)	320 (38)
C(59)	-4218 (2)	35 (3)	-7279 (11)	236 (33)
C(60)	-3801 (2)	287 (3)	-7767 (11)	273 (33)
C(61)	-4566 (2)	4 (3)	-8895 (11)	271 (34)
C(63)	-4993 (2)	287 (3)	-8085 (12)	296 (35)
C(65)	-5765 (2)	584 (3)	-9260 (13)	377 (41)
C(66)	-6009 (2)	652 (4)	-11277 (14)	508 (45)
C(67)	-5965 (3)	50 (5)	-7755 (16)	689 (67)
C(68)	-6457 (2)	970 (5)	-10998 (15)	582 (58)
C(69)	-6709 (2)	992 (4)	-13010 (14)	364 (48)
C(70)	-6949 (3)	403 (4)	-13669 (15)	580 (55)
C(71)	-7190 (3)	444 (5)	-15402 (18)	684 (63)
C(72)	-7212 (3)	1018 (5)	-16562 (15)	603 (57)
C(73)	-6959 (3)	1594 (5)	-15999 (17)	716 (64)
C(74)	-6723 (3)	1568 (5)	-14226 (17)	780 (73)
Cl(2)	-177 (1)	-1534 (1)	-7138 (3)	470 (11)
N(51)	-2924 (2)	626 (4)	-8863 (10)	478 (40)
N(64)	-5308 (2)	351 (2)	-9797 (9)	305 (33)
O(53)	-2768 (1)	716 (3)	-5542 (9)	567 (35)
O(56)	-1754 (2)	-160 (3)	-7839 (8)	544 (34)
O(62)	-4599 (1)	-704 (2)	-9535 (9)	435 (31)

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Molecule A		Molecule B	
C(2)–C(4)	1.494 (8)	C(52)–C(54)	1.455 (8)
C(2)–N(1)	1.331 (10)	C(52)–N(51)	1.346 (10)
C(2)–O(3)	1.251 (8)	C(52)–O(53)	1.235 (9)
C(4)–C(5)	1.406 (10)	C(54)–C(55)	1.443 (10)
C(4)–C(10)	1.391 (9)	C(54)–C(60)	1.380 (9)
C(5)–C(7)	1.395 (10)	C(55)–C(57)	1.361 (9)
C(5)–O(6)	1.345 (8)	C(55)–O(56)	1.385 (9)
C(7)–C(8)	1.376 (11)	C(57)–C(58)	1.370 (10)
C(8)–C(9)	1.412 (11)	C(58)–C(59)	1.408 (10)
C(9)–C(10)	1.403 (8)	C(59)–C(60)	1.412 (8)
C(9)–C(11)	1.531 (9)	C(59)–C(61)	1.513 (9)
C(11)–C(13)	1.515 (8)	C(61)–C(63)	1.521 (8)
C(11)–O(12)	1.424 (7)	C(61)–O(62)	1.424 (7)
C(13)–N(14)	1.491 (9)	C(63)–N(64)	1.492 (9)
C(15)–C(16)	1.458 (14)	C(65)–C(66)	1.529 (12)
C(15)–C(17)	1.515 (11)	C(65)–C(67)	1.550 (12)
C(15)–N(14)	1.535 (8)	C(65)–N(64)	1.523 (8)
C(16)–C(18)	1.397 (17)	C(66)–C(68)	1.522 (10)
C(18)–C(19)	1.656 (16)	C(68)–C(69)	1.535 (13)
C(19)–C(20)	1.395 (14)	C(69)–C(70)	1.418 (11)
C(19)–C(24)	1.414 (14)	C(69)–C(74)	1.363 (13)
C(20)–C(21)	1.422 (15)	C(70)–C(71)	1.364 (14)
C(21)–C(22)	1.313 (14)	C(71)–C(72)	1.340 (14)
C(22)–C(23)	1.394 (16)	C(72)–C(73)	1.402 (13)
C(23)–C(24)	1.380 (15)	C(73)–C(74)	1.375 (16)
N(1)–C(2)–C(4)	120.3 (6)	N(51)–C(52)–C(54)	118.4 (6)
O(3)–C(2)–C(4)	120.5 (6)	O(53)–C(52)–C(54)	121.1 (7)
O(3)–C(2)–N(1)	119.2 (5)	O(53)–C(52)–N(51)	120.5 (6)
C(5)–C(4)–C(2)	118.0 (6)	C(55)–C(54)–C(52)	119.6 (6)
C(10)–C(4)–C(2)	122.1 (6)	C(60)–C(54)–C(52)	123.9 (6)
C(10)–C(4)–C(5)	119.9 (5)	C(60)–C(54)–C(55)	116.4 (5)
C(7)–C(5)–C(4)	120.3 (6)	C(57)–C(55)–C(55)	121.1 (6)
O(6)–C(5)–C(4)	122.2 (6)	C(57)–C(55)–O(56)	119.1 (7)
O(6)–C(5)–C(7)	117.5 (7)	C(54)–C(55)–O(56)	119.7 (9)
C(8)–C(7)–C(5)	119.7 (7)	C(58)–C(57)–C(55)	120.4 (7)
C(9)–C(8)–C(7)	121.1 (6)	C(59)–C(58)–C(57)	122.1 (6)
C(10)–C(9)–C(8)	118.9 (6)	C(60)–C(59)–C(58)	116.2 (6)
C(11)–C(9)–C(8)	120.7 (5)	C(61)–C(59)–C(58)	123.6 (5)
C(11)–C(9)–C(10)	120.3 (6)	C(61)–C(59)–C(60)	120.1 (6)
C(9)–C(10)–C(4)	120.1 (6)	C(59)–C(60)–C(54)	123.6 (6)
C(13)–C(11)–C(9)	111.0 (6)	C(63)–C(61)–C(59)	110.9 (6)
O(12)–C(11)–C(9)	107.1 (5)	O(62)–C(61)–C(59)	107.1 (5)
O(12)–C(11)–C(13)	111.4 (5)	O(62)–C(61)–C(63)	112.4 (5)
N(14)–C(13)–C(11)	111.7 (6)	N(64)–C(63)–C(61)	109.3 (6)
C(17)–C(15)–C(16)	115.5 (7)	C(67)–C(65)–C(66)	114.4 (6)
N(14)–C(15)–C(16)	112.7 (8)	N(64)–C(65)–C(66)	106.3 (6)
N(14)–C(15)–C(17)	107.9 (5)	N(64)–C(65)–C(67)	108.9 (5)
C(18)–C(16)–C(15)	114.0 (9)	C(68)–C(66)–C(65)	112.2 (7)
C(19)–C(16)–C(15)	110.6 (9)	C(69)–C(66)–C(66)	111.6 (7)
C(20)–C(19)–C(18)	117.2 (9)	C(70)–C(69)–C(68)	120.4 (8)
C(24)–C(19)–C(18)	124.9 (9)	C(74)–C(69)–C(68)	122.9 (8)
C(24)–C(19)–C(20)	117.9 (9)	C(74)–C(69)–C(70)	116.7 (8)
C(21)–C(20)–C(19)	119.5 (9)	C(71)–C(70)–C(69)	119.6 (8)
C(22)–C(20)–C(19)	121.6 (9)	C(72)–C(70)–C(69)	123.4 (9)
C(23)–C(22)–C(21)	119.7 (10)	C(73)–C(72)–C(71)	117.9 (9)
C(24)–C(23)–C(22)	121.2 (9)	C(74)–C(73)–C(72)	119.4 (9)
C(23)–C(24)–C(19)	119.6 (9)	C(73)–C(74)–C(69)	122.9 (9)
C(15)–N(14)–C(13)	113.4 (6)	C(65)–N(64)–C(63)	117.0 (6)

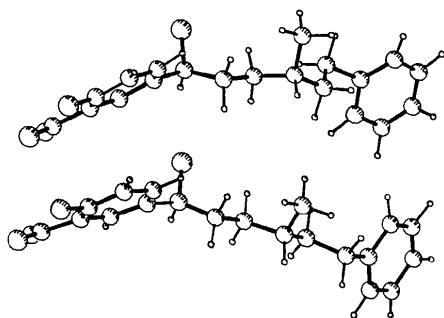


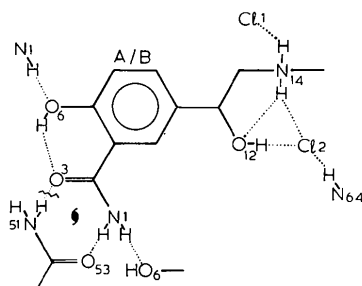
Fig. 1. Views of the two molecules of the asymmetric unit roughly parallel to the plane of the substituted phenyl ring.

Table 3. Torsion angles ( $^\circ$ ) in the backbone of labetalol hydrochloride (*e.s.d.*'s ca  $1^\circ$ )

C(8)–C(9)–C(11)–C(13)	47.6	C(58)–C(59)–C(61)–C(63)	47.7
C(8)–C(9)–C(11)–O(12)	-73.7	C(58)–C(59)–C(61)–O(62)	-75.2
C(10)–C(9)–C(11)–C(13)	-136.2	C(60)–C(59)–C(61)–C(63)	-134.7
C(10)–C(9)–C(11)–O(12)	102.4	C(60)–C(59)–C(61)–O(62)	102.2
C(9)–C(11)–C(13)–N(14)	170.5	C(59)–C(61)–C(63)–N(64)	171.2
O(12)–C(11)–C(13)–N(14)	-70.5	O(62)–C(61)–C(63)–N(64)	-68.9
C(11)–C(13)–N(14)–C(15)	164.9	C(61)–C(63)–N(64)–C(65)	176.1
C(17)–C(15)–C(16)–C(18)	-63.5	C(67)–C(65)–C(66)–C(68)	67.2
N(14)–C(15)–C(16)–C(18)	61.5	N(64)–C(65)–C(66)–C(68)	-172.5
C(16)–C(15)–N(14)–C(13)	155.5	C(66)–C(65)–N(64)–C(63)	176.5
C(17)–C(15)–N(14)–C(13)	-75.8	C(67)–C(65)–N(64)–C(63)	-59.7
C(15)–C(16)–C(18)–C(19)	176.6	C(65)–C(66)–C(68)–C(69)	-176.4
C(16)–C(18)–C(19)–C(20)	69.6	C(66)–C(68)–C(69)–C(70)	85.1
C(16)–C(18)–C(19)–C(24)	-109.6	C(66)–C(68)–C(69)–C(74)	-95.9

bond] there is a *gauche* arrangement. In this molecule the bond lengths to C(18) vary considerably from those in the *B* molecule [C(68)] and the thermal parameters of C(16) and C(18) are rather high. There are also some unexplained peaks in the C(16)–C(18) region of the final difference map, so there may be some degree of disorder in this region (but the pseudosymmetry makes it difficult to clarify this). The two molecules are very similar in shape and, apart from the C(16)–C(18) region, almost superimposable, so it is quite possible that there is a small proportion of the extended form in the *A* site. It therefore seems likely that the two conformations shown in Fig. 1 are of very similar energy.

The conformations of adrenergic and dopaminergic drugs have been reviewed recently by Duax (1979), Giesecke (1979) and Hebert (1979). The conformations are defined by two torsion angles  $\tau_1$  and  $\tau_2$  as in Fig. 3. There is considerable speculation as to the lowest energy conformation of phenylethanolamine derivatives. The calculated global energy minimum ( $\tau_1$  90°,  $\tau_2$  –60°) (Caillet, Claverie & Pullman, 1976) has not been seen in the solid state. In the crystalline state one conformation is almost always found ( $\tau_1$  90°,  $\tau_2$  180° ‘perpendicular–*trans*’). The single exception is (–)-adrenaline hydrogen (+)-tartrate (Carlström, 1973) with  $\tau_1$  0°,  $\tau_2$  180° (‘planar–*trans*’); adrenaline free



#### Intramolecular

O(6)···O(3)	2.51	O(56)···O(53)	2.54
O(12)···N(14)	3.02	O(62)···N(64)	2.99

#### Intermolecular

N(1)( $x, y, 1+z$ )···O(6)	2.88
N(1)( $-\frac{1}{2}-x, -y, \frac{1}{2}+z$ )···O(53)	2.93
N(14)( $x, y, 1+z$ )···Cl(1)	3.24
N(14)( $\frac{1}{2}+x, -\frac{1}{2}-y, -2-z$ )···Cl(2)	3.20
O(12)( $\frac{1}{2}+x, -\frac{1}{2}-y, -2-z$ )···Cl(2)	3.10
N(51)( $x, y, 1+z$ )···O(56)	2.93
N(51)( $-\frac{1}{2}-x, -y, \frac{1}{2}+z$ )···O(3)	2.98
N(64)( $x, y, 1+z$ )···Cl(1)	3.21
N(64)( $-\frac{1}{2}-x, -y, \frac{1}{2}+z$ )···Cl(2)	3.12
O(62)( $x, y, 1+z$ )···Cl(1)	3.13

Fig. 2. Lengths (Å) of hydrogen bonds in labetalol hydrochloride.

The hydrogen-bonding scheme is identical for the two independent molecules, *A* and *B* (whose atom numbers are 50 greater than the corresponding atom number in *A*). E.s.d.'s are about 0.015 Å.

base has the usual perpendicular–*trans* conformation. The present structure provides another exception, since both molecules adopt an intermediate conformation with  $\tau_1$  47°,  $\tau_2$  180° (Fig. 4). Since both independent molecules in the crystal adopt this conformation and both have virtually identical environments it seems likely that this is the result of crystal-packing forces. However, it also suggests that the barrier to rotation about C<sub>β</sub>–C<sub>γ</sub> is fairly low and that if enough structures of phenylethanolamines are studied a wide range of conformations may be observed. A general description of the effect of packing forces is given in Murray-Rust (1982) in which a preliminary account of the structure of the title compound is also included.

The crystal-structure determination cannot directly assist in deducing the conformation of labetalol required by the receptor. However, using additional evidence from rigid analogues, Hebert (1979) suggests that interaction with the adrenoceptor takes place predominantly with  $\tau_2$  180°, and the present structure is consistent with this.

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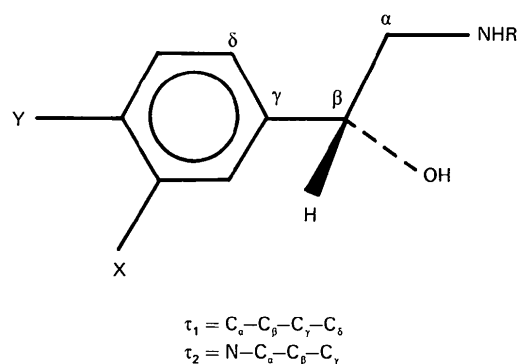


Fig. 3. Definition of the torsion angles in phenylethanolamines.

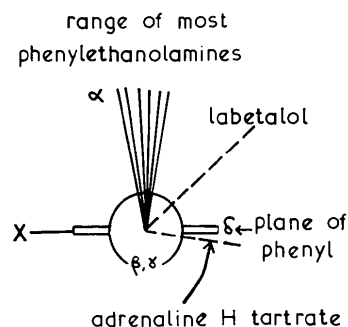


Fig. 4. Conformations of adrenergic molecules viewed down C<sub>β</sub>–C<sub>γ</sub>. Most adrenergics have  $\tau_1$  about 90° (i.e. C<sub>α</sub> lying between  $\alpha_1$  and  $\alpha_2$ ), but (–)-adrenaline hydrogen (+)-tartrate has  $\tau_1$  ~0° (at  $\alpha_A$ ) and both molecules of labetalol hydrochloride have  $\tau_1$  ~47° (at  $\alpha_2$ ).

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### Pyridin-Bortrifluorid (1/1), C<sub>5</sub>H<sub>5</sub>N.BF<sub>3</sub>

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**Abstract.**  $M_r = 146.91$ , monoclinic,  $P2_1/c$ ,  $a = 17.679$  (5),  $b = 5.914$  (1),  $c = 14.197$  (3) Å,  $\beta = 118.90$  (2)°,  $U = 1299.5$  (8) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.502$  (3) Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu(\text{Mo } K\alpha) = 0.16$  mm<sup>-1</sup>,  $F(000) = 592$ , room temperature. The crystal structure has been refined to  $R_w(F) = 0.037$  (956 reflections). The two independent molecules are similar. The boron atoms have a distorted tetrahedral coordination. The B–N bonds are 1.602 (4) and 1.604 (5) Å. The B–F bonds range from 1.330 (4) to 1.362 (3) Å.

**Einleitung.** Frühere Untersuchungen von BX<sub>3</sub>-Additionsverbindungen (X = F, Cl, Br, J) mit tertiären Aminen (Hess, 1969; Clippard, Hanson & Taylor, 1971; Geller & Hoard, 1951) zeigen mit zunehmender Grösse des Halogenatoms einen kleiner werdenden B–N-Abstand, während dieser im C<sub>5</sub>H<sub>5</sub>N.BF<sub>3</sub> nach Zvonkova (1956) kleiner ist als im C<sub>5</sub>H<sub>5</sub>N.BCl<sub>3</sub> (Töpel, Hensen & Trömel, 1981; Töpel, 1981). Allerdings wurde bereits in früheren Arbeiten (Eriks, 1963; Töpel *et al.*, 1981) auf Inkonsistenzen in den Daten von Zvonkova (1956) hingewiesen, so dass eine erneute Untersuchung angebracht schien. Die jetzige Strukturanalyse widerspricht dem Ergebnis von Zvonkova (1956).

**Experimentelles.** Die Verbindung wurde durch Addition von Pyridin zu einer Lösung von BF<sub>3</sub> in Cyclohexan dargestellt. Einkristalle wurden durch Sublimation bei 308 K und  $2,7 \times 10^2$  Pa erhalten. Die Verbindung ist hygroskopisch, so dass der untersuchte Einkristall in ein Markröhrchen eingeschmolzen wurde.

Kristall 0,58 (3) × 0,36 (2) × 0,2 (1) mm, Syntex P2<sub>1</sub>-Vierkreisdiffraktometer (Mo K $\alpha$ -Strahlung, Nb-Filter,  $\theta$ -2 $\theta$ -Abtastung, Messbereich pro Reflex 1,9°, 2,5° ≤  $\theta$  ≤ 20°, Referenzreflexe 115, 514, 115). Von den 6540 gemessenen Reflexen bildeten nach einer Profilanalyse (Blessing, Coppens & Becker, 1974) 1186 den vollständigen Datensatz (interne Konsistenz 0,049, definiert als  $\sum |I - \bar{I}| / \sum I$ ) ohne systematisch ausgelöschte (Reflexe). Die Struktur wurde mit 956 Reflexen mit  $F_o \geq 1\sigma$  ohne Absorptionskorrektur bestimmt. Die Lösung der Struktur erfolgte mit dem Programmsystem *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977), die Datenreduktion sowie die Strukturverfeinerung mit den Programmen des *XRAY* System (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) mit Atomformfaktoren nach Cromer & Mann (1968) für B, C, F, N und Stewart, Davidson & Simpson (1965) für H,  $w = 1/\sigma^2$ . Die Lagen aller Atome ausser der der Wasserstoffe