nearly coplanar and with the remaining atom out of this plane. In carfecillin, C(3) is 0·42 Å out of the plane defined by S(1), C(5), N(4) and C(2) (Table 3). The thiazolidine ring is therefore very similar to that in phenoxymethylpenicillin (Abrahamsson, Crowfoot-Hodgkin & Maslen, 1963) and potassium benzylpenicillin (Crowfoot, Bunn, Rogers-Low & Turner-Jones, 1949), which is characterized by C(3) out of the common plane. The distance of N(4) from the plane defined by C(3), C(5), C(7) is 0·47 Å in carfecillin, a similar value to that of 0·44 Å for methicillin methyl ester (Blanpain, Melebeck & Durant, 1977). Thus the configuration of this N atom conforms to the pyramidal arrangement which appears to be a common feature of biologically active penicillins and cephalosporins (Sweet & Dahl, 1970; Vijayan, Anderson & Hodgkin, 1973). N(4) is also significantly (0·28 Å) out of the plane of the remaining β-lactam constituents C(5), C(6) and C(7) (Table 3).

The configuration of the side-chain asymmetric C(17) in carfecillin has been assigned by comparison of the structure reported here with the crystal structures of ampicillin anhydrate (Boles & Girven, 1976*a*), ampicillin trihydrate (James, Hall & Hodgkin, 1968) and amoxycillin trihydrate (Boles *et al.*, 1978). The asymmetric centres C(3), C(5) and C(6) of the penicillin nucleus have configuration *S*, *R* and *R* respectively as in all penicillins derived from the naturally occurring precursor, 6-aminopenicillanic acid. The configuration of the side-chain asymmetric C atom in ampicillin and amoxycillin is known to be *R* because they are synthesized from the resolved amino acids, (*R*)-α-aminophenylacetic acid and (*R*)-α-amino-4-hydroxyphenylacetic acid. Comparison of the configuration of carfecillin with the ampicillin and amoxycillin structures shows that the C(17) configuration in crystalline carfecillin is also *R*.

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Structure of Tribromotris(pyridine)indium(III) Pyridine Solvate

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Abstract. $[In(C_5H_5N)_3Br_3.C_5H_5N]$, $C_{20}H_{20}Br_3InN_4$, monoclinic, $P2_1$, $a = 9{\cdot}635$ (5), $b = 14{\cdot}87$ (5), $c = 9{\cdot}153$ (5) Å, $\beta = 118{\cdot}00$ (2)°, $Z = 2$, $D_x = 1{\cdot}923$, $D_m = 1{\cdot}91$ Mg m^{-3}. The structure has been solved from 1193 diffractometer-measured intensities with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å) and refined by full-matrix least squares to $R = 0{\cdot}0668$. The crystal structure consists of 'octahedral' $InBr_3.3(C_5H_5N)$ molecules

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with *mer* geometry and an additional pyridine molecule of crystallization.

Introduction. Pyridine adducts of the indium trihalides are well known and complexes with stoichiometries $\mathrm{In}X_3.\mathrm{C_5H_5N}$, $\mathrm{In}X_3.2(\mathrm{C_5H_5N})$ and $\mathrm{In}X_3.3(\mathrm{C_5H_5N})$ have been prepared (Carty & Tuck, 1966). Their stereochemistry has been investigated mainly by spectroscopic methods.

We have recently prepared a new complex $\mathrm{InBr_3.4(C_5H_5N)}$ and among the several interesting structural possibilities for this stoichiometry are the four-coordinate $[\mathrm{In(C_5H_5N)_4}]^{3+}$ species, the six-coordinate $[\mathrm{In(C_5H_5N)_4Br_2}]^{+}$ analogous to that found by us in the $\mathrm{GaCl_3.2(C_5H_5N)}$ complex (Sinclair, Small & Worrall, 1981), and the six-coordinate $\mathrm{InBr_3.3(C_5H_5N)}$ with an additional pyridine molecule of crystallization.

The present work was carried out to determine the stereochemistry of In in this new complex.

Using a single crystal sealed in a Lindemann-glass tube, the crystal data were obtained from Weissenberg photographs taken with Cu $K\alpha$ radiation (λ = 1·542 Å). Intensity measurements were made on a Stoe STADI-2 two-circle automatic diffractometer with graphite-monochromatized Mo $K\alpha$ radiation (λ = 0·7107 Å). Layers h = 0–9 with the crystal set about **a** were measured. The crystal size was 0·8 × 0·2 × 0·1 mm. Absorption corrections (μ = 6·02 mm^{-1}) and scaling of data were carried out with the *SHELX* suite of programs (Sheldrick, 1976); after elimination of those for which $I < 3\sigma(I)$ there remained 1193 unique reflections. An E map obtained by the *MULTAN* direct-methods program (Main, Lessinger, Woolfson, Germain & Declercq, 1977) revealed the positions of the In and Br atoms. These were used to phase $(F_o - F_c)$ maps to give the positions of the remaining atoms (apart from H) using *SHELX*. Using unit weights, full-matrix least-squares refinement of all atomic positions, anisotropic U_{ij} values for In and Br, and isotropic U values for the remaining non-H atoms were carried out until convergence was reached at R = 0·0668. Throughout the refinement y of In was constrained at a constant value as also were the C—C [1·395 (5) Å] and C—N [1·365 (5) Å] bond distances

Table 1. *Fractional atomic coordinates* (×10^4), *equivalent isotropic thermal parameters for* In *and* Br (×10^4) *and isotropic thermal parameters for* C *and* N (×10^3)

$$U_{\mathrm{eq}} = \tfrac{1}{3} \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	U_{eq}/U (Å^2)
In	6722 (2)	7300†	8362 (2)	325 (23)
Br(1)	9770 (3)	7281 (7)	9884 (4)	535 (29)
Br(2)	6623 (4)	7329 (5)	1164 (3)	468 (30)
Br(3)	6630 (4)	7259 (5)	5458 (3)	474 (28)
N(1)	4001 (23)	7364 (33)	6998 (26)	46 (7)
C(1)	3173 (31)	7051 (23)	7760 (39)	50 (11)
C(2)	1538 (30)	7000 (26)	7026 (38)	70 (13)
C(3)	786 (40)	7284 (53)	5382 (41)	80 (12)
C(4)	1524 (34)	7654 (29)	4527 (46)	80 (14)
C(5)	3162 (35)	7664 (26)	5411 (33)	63 (12)
N(2)	6587 (30)	8850 (17)	8367 (28)	24 (10)
C(6)	7581 (39)	9289 (22)	9787 (36)	56 (11)
C(7)	7572 (49)	10227 (22)	9843 (49)	69 (12)
C(8)	6559 (52)	10761 (26)	8517 (45)	73 (18)
C(9)	5560 (53)	10276 (22)	7106 (51)	76 (14)
C(10)	5544 (35)	9338 (21)	7043 (34)	45 (9)
N(3)	6606 (41)	5769 (23)	8205 (45)	55 (16)
C(11)	7562 (42)	5302 (24)	7752 (47)	54 (10)
C(12)	7592 (51)	4364 (24)	7763 (62)	90 (16)
C(13)	6498 (42)	3890 (24)	8056 (52)	43 (12)
C(14)	5511 (47)	4337 (23)	8545 (54)	72 (13)
C(15)	5562 (41)	5274 (23)	8486 (46)	54 (10)
N(4)	98 (55)	4772 (41)	5832 (50)	127 (17)
C(16)	8794 (59)	5122 (36)	4531 (50)	82 (7)
C(17)	8766 (66)	5202 (43)	2999 (57)	116 (21)
C(18)	9992 (67)	4780 (44)	2863 (66)	115 (8)
C(19)	1182 (76)	4372 (47)	4263 (71)	145 (26)
C(20)	1146 (73)	4188 (39)	5470 (72)	112 (22)

† Constrained.

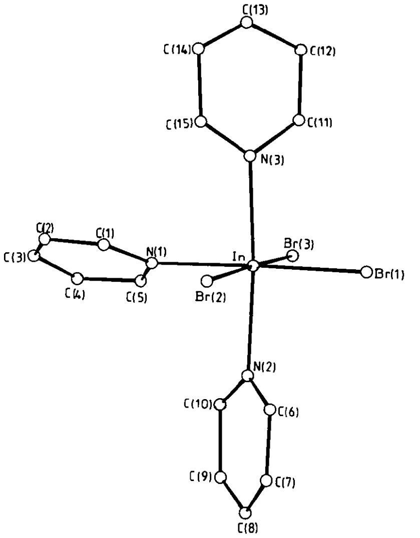

Fig. 1. Structure of $\mathrm{InBr_3.3(C_5H_5N)}$.

Table 2. *Bond distances* (Å) *and angles* (°) *around* In

In—Br(1)	2·593 (3)	In—Br(2)	2·611 (4)
In—Br(3)	2·618 (4)	In—N(1)	2·32 (2)
In—N(2)	2·31 (2)	In—N(3)	2·28 (3)
N(3)—In—N(1)	90 (1)	Br(2)—In—N(1)	88·6 (6)
Br(3)—In—N(1)	87·9 (6)	Br(1)—In—N(1)	178 (1)
N(3)—In—N(2)	175 (1)	Br(1)—In—N(2)	93·4 (7)
Br(2)—In—N(2)	87·3 (7)	Br(2)—In—N(3)	93·2 (9)
Br(2)—In—Br(1)	91·5 (1)	Br(3)—In—N(2)	92·9 (7)
Br(3)—In—N(3)	86·3 (9)	Br(3)—In—Br(1)	92·0 (1)
Br(3)—In—Br(2)	176·4 (1)	Br(1)—In—N(3)	91·9 (9)
N(2)—In—N(1)	85 (1)		

('DFIX'). As the space group is without a centre of symmetry, the enantiomorphous structure was also refined; the agreement was slightly worse ($R = 0{\cdot}0674$) although there were no significant changes in the structural parameters. Atomic scattering factors, including anomalous scattering were those of Cromer & Mann (1968) and Cromer & Liberman (1970). Final coordinates and equivalent values ($U_{\rm eq}$) of anisotropic temperature coefficients are given in Table 1.*

Discussion. The crystal-structure determination shows that $InBr_3.4(C_5H_5N)$ consists of neutral molecules $InBr_3.3(C_5H_5N)$ (Fig. 1) with additional pyridine molecules of crystallization. Bond distances and angles around In are given in Table 2. The molecule is approximately octahedral and has *mer* (*trans*) stereochemistry. The angles between least-squares planes* show that the interplane angle of *trans* pyridines [rings containing N(2) and N(3)] is 88 (6)° and these planes are at 45 (2)° to the In—Br and In—N directions in the intermediate plane.

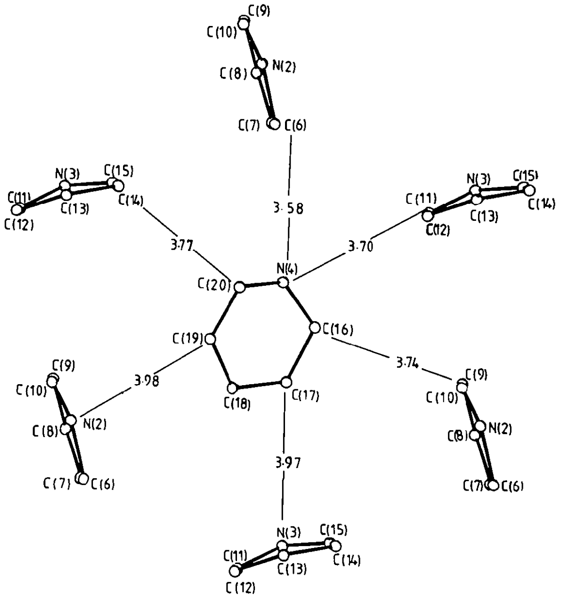

Fig. 2. Site of solvate pyridine showing the closest contacts (Å) (e.s.d. 0·10 Å).

Far-infrared spectral studies (Adams, Carty, Carty & Tuck, 1968) of the complexes $InX_3.3(C_5H_5N)$ (X = Cl, Br, I) have indicated *fac* (*cis*) geometry. Zahrobsky (1971) has discussed the stereochemical influence of non-bonded intramolecular van der Waals interactions and concludes that facial geometry should be preferred for octahedral MA_3B_3 molecules, where A and B are monoatomic ligands with substantial differences in their steric requirements. Carty & Tuck (1975) have applied Zahrobsky's method to calculate steric angles for a number of In compounds including $InCl_3.3N$ systems and their steric analysis favours *fac* geometry. Zahrobsky (1971) has pointed out that strong repulsive interactions between bulky ligands such as pyridine in *cis* positions might result in the *trans* isomer being favoured and has cited examples of Sn chemistry in support. We feel that this may be the reason why the *mer* form is the stable form of $InBr_3.3(C_5H_5N)$. We are presently determining the crystal structure of $InI_3.3(C_5H_5N)$ and preliminary results show that this also has *mer* geometry. Clearly spectroscopic results are unreliable for this series of compounds.

The stacking of $InBr_3.3(C_5H_5N)$ molecules is not completely space filling and a fourth pyridine molecule may be accommodated in the structure. Fig. 2 illustrates the position of this fourth pyridine relative to other pyridines at similar heights and the closest contacts.

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* Lists of structure factors, anisotropic U_{ij}, bond angles not involving In and angles between least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36403 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.