

Fig. 1. Numbering of the atoms in the title compound.

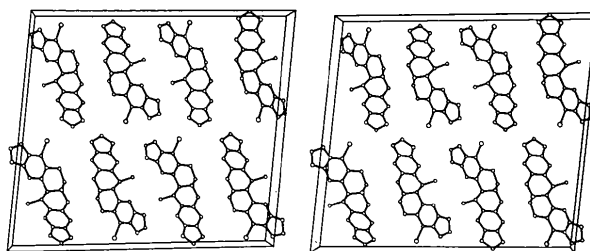


Fig. 2. ORTEP (Johnson, 1965) stereoscopic projection of the unit-cell contents. *a* is vertical, *c* is horizontal and *b* is out of the plane of the paper.

the accepted values. Atomic scattering factors were taken from Doyle & Turner (1968) except those for H which were taken from Stewart, Davidson & Simpson (1965); anomalous-dispersion data for Br were from Cromer (1965). Fig. 1 shows the numbering scheme and Fig. 2 is an ORTEP (Johnson, 1965) representation of the packing of the molecules in the unit cell.

Related literature. The structures of the title compound and 4-bromo-13 β -methyl-13 $\alpha\alpha$ -H-tetrahydropseudoptisin have been compared by Rogers, Atkinson Williams, Waight, Pai, Nagarajan, Natarajan, Suguna, Rajeswari, Chandrasekaran, Rajaraman & Manikumar (1982). Related structures are capaurimine mono-*p*-bromobenzoate (Kametani, Ihara, Honda, Shimanouchi & Sasada, 1971), capaurine hydrobromide (Shimanouchi, Sasada, Ihara & Kametani, 1969), and *cis*-5,6,13,13a-tetrahydro-3,9-dihydroxy-1,2,10-trimethoxy-8*H*-dibenzo-*[a,g]*quinolizine hydrobromide monohydrate (Shimanouchi, Sasada, Wakisaka, Kametani & Ihara, 1970). Work on protoberberines and

tetrahydroprotoberberines is described in a review by Pai, Nagarajan, Suguna & Natarajan (1977).

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6,6''-Dibromo-4'-phenyl-2,2':6',2''-terpyridine

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Abstract. C₂₁H₁₃Br₂N₃, *M_r* = 467.16, orthorhombic, *Pbcn*, *a* = 7.366 (3), *b* = 12.006 (4), *c* = 20.601 (5) Å, *V* = 1822 (1) Å³, *Z* = 4 (implying that each molecule lies on a twofold special position), *D_x* = 1.702 Mg m⁻³, λ(Cu Kα) = 1.5418 Å, μ =

5.36 mm⁻¹, *F*(000) = 920, *T* = 298 K, *R* = 0.037 for 1327 unique observed reflections with *F* > 4σ(*F*). The molecule lies on a twofold rotation axis which runs through N and one C of the central pyridyl ring. The molecule exhibits a *trans,trans* arrangement of

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Br(1)	3385 (1)	3223 (1)	5304 (1)	65 (3)
C(1)	2270 (4)	4031 (2)	4613 (1)	43 (1)
C(2)	2099 (4)	5172 (3)	4660 (1)	49 (2)
C(3)	1307 (4)	5722 (2)	4156 (2)	54 (2)
C(4)	720 (4)	5123 (2)	3618 (1)	50 (2)
C(5)	975 (3)	3975 (2)	3608 (1)	37 (1)
N(1)	1740 (3)	3426 (2)	4112 (1)	38 (1)
C(6)	448 (3)	3291 (2)	3031 (1)	38 (1)
N(2)	0	3864 (2)	2500	38 (1)
C(7)	465 (3)	2132 (2)	3053 (1)	38 (1)
C(8)	0	1526 (3)	2500	38 (2)
C(9)	0	285 (3)	2500	40 (2)
C(10)	-523 (4)	-304 (2)	3052 (2)	50 (2)
C(11)	-497 (5)	-1465 (2)	3052 (2)	66 (2)
C(12)	0	-2027 (3)	2500	67 (3)

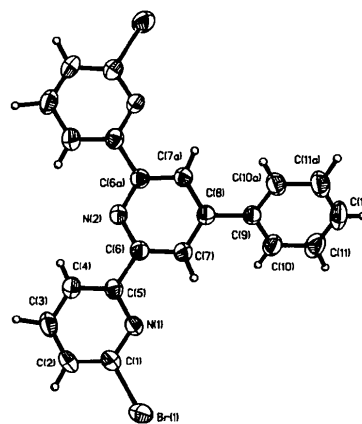
Table 2. Bond lengths (\AA) and angles ($^\circ$)

Br(1)—C(1)	1.910 (3)	C(3)—C(4)	1.391 (4)
C(1)—C(2)	1.379 (4)	C(4)—C(5)	1.391 (3)
C(1)—N(1)	1.321 (4)	C(5)—C(6)	1.497 (3)
C(2)—C(3)	1.361 (4)	N(1)—C(5)	1.352 (3)
C(6)—N(2)	1.334 (3)	N(2)—C(6a)	1.334 (3)
C(6)—C(7)	1.392 (3)	C(7)—C(8)	1.395 (3)
C(8)—C(9)	1.490 (5)	C(8)—C(7a)	1.395 (3)
C(9)—C(10)	1.394 (3)	C(9)—C(10a)	1.394 (3)
C(10)—C(11)	1.393 (4)	C(11)—C(12)	1.373 (4)
C(12)—C(11a)	1.373 (4)		
Br(1)—C(1)—C(2)	119.4 (2)	Br(1)—C(1)—N(1)	115.5 (2)
C(2)—C(1)—N(1)	125.1 (3)	C(1)—C(2)—C(3)	117.8 (3)
C(2)—C(3)—C(4)	119.4 (3)	C(3)—C(4)—C(5)	118.7 (3)
C(4)—C(5)—N(1)	121.9 (2)	C(4)—C(5)—C(6)	121.3 (2)
N(1)—C(5)—C(6)	116.7 (2)	C(1)—N(1)—C(5)	117.0 (2)
C(5)—C(6)—N(2)	115.6 (2)	C(5)—C(6)—C(7)	121.4 (2)
N(2)—C(6)—C(7)	123.0 (2)	C(6)—N(2)—C(6a)	117.9 (3)
C(6)—C(7)—C(8)	119.5 (2)	C(7)—C(8)—C(9)	121.5 (2)
C(7)—C(8)—C(7a)	117.1 (3)	C(9)—C(8)—C(7a)	121.5 (2)
C(8)—C(9)—C(10)	120.5 (2)	C(8)—C(9)—C(10a)	120.5 (2)
C(10)—C(9)—C(10a)	119.0 (3)	C(9)—C(10)—C(11)	120.3 (3)
C(10)—C(11)—C(12)	119.7 (3)	C(11)—C(12)—C(11a)	121.1 (4)

pyridyl rings about each interannular C—C bond. The three pyridyl rings are approximately coplanar, with the terminal rings making a dihedral angle of 10.9° with the central pyridyl ring. The phenyl substituent is twisted about the interannular C—C bond, and makes a dihedral angle of 35.0° with the central ring. All bond lengths and bond angles within the aromatic rings are within the expected ranges, as is the C—Br distance of $1.910(3) \text{\AA}$.

Experimental. Good-quality crystals of the title compound were obtained by slow cooling of a hot ethanolic solution. Pale-yellow tablet, $0.22 \times 0.35 \times 0.42 \text{ mm}$, Syntex $P2_1$ four-circle diffractometer, graphite-monochromated $\text{Cu } K\alpha$ X-radiation, cell parameters from 2θ values of 15 reflections measured in range $40 < 2\theta < 50^\circ$. For data collection, $T = 298 \text{ K}$, $\omega/2\theta$ scans with scan width from 1° below $K\alpha_1$ to 1° above $K\alpha_2$ and scan speed in range

$2.5\text{--}29.3^\circ \text{ min}^{-1}$, $2\theta_{\text{max}} = 125^\circ$, $h 0 \rightarrow 8$, $k -13 \rightarrow 0$, $l 0 \rightarrow 23$, two standard reflections indicated no significant crystal movement or decay in intensities, no absorption correction, 1403 unique reflections of which 1327 with $F > 4\sigma(F)$ were used in structure solution (from a Patterson synthesis followed by iterative cycles of blocked-cascade least-squares refinement and difference Fourier synthesis) and refinement [using blocked-cascade least squares on F (modified *SHELX76*; Sheldrick, 1976)]. Anisotropic thermal parameters for all non-H atoms, H-atom positions refined freely, common isotropic thermal parameter, $0.059(3) \text{\AA}^2$. At final convergence $R = 0.037$, $wR = 0.065$ for 131 parameters, $(\Delta/\sigma)_{\text{max}}$ in final cycle 0.077 , max. and min. residues in final ΔF synthesis 0.25 , -0.84 e \AA^{-3} , respectively. The



weighting scheme $w^{-1} = \sigma^2(F) + 0.001F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while bond lengths and angles appear in Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using the *SHELXTL-Plus* package (Sheldrick, 1990). Fig. 2 shows the molecular packing and was also produced with the *SHELXTL-Plus* package.

Related literature. Constable (Constable & Lewis, 1982; Constable, Lewis, Liptrot, Raithby & Schröder, 1983; Constable, Lewis, Liptrot & Raithby, 1984; Constable, Khan, Lewis, Liptrot & Raithby, 1985; Constable, Holmes & McQueen, 1987; Constable & Holmes, 1988) has reported the preparation of macrocyclic ligands and complexes derived from this and related 2,2':6',2''-terpyridines. Interest in the structures of oligopyridines arises from the conformation of the pyridyl rings, which need to change from *trans* to *cis* orientations about the interannular C—C bonds upon coordination to a metal. Although a number of structure determinations of 2,2'-

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54764 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0084]

bipyridine have been reported [see Chisholm, Huffman, Rothwell, Bradley, Kress & Woodruff (1981) for the most recent of these], few of the higher oligopyridines have been studied. To date, the only structure determinations reported are of 4'-phenyl-2,2':6',2''-terpyridine (Constable, Lewis, Liptrot & Raithby, 1990) and 2,2':6',2''':6'',2''''-quaterpyridine (Constable, Elder, Healy & Tocher, 1990).

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Structure of Octabenzene

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Abstract. [2-Hydroxy-4-(octyloxy)phenyl]phenylmethanone, $C_{21}H_{26}O_3$, $M_r = 326.4$, monoclinic, $P2_1/n$, $a = 10.186$ (1), $b = 5.546$ (1), $c = 33.232$ (2) Å, $\beta = 98.48$ (4)°, $V = 1856.8$ (2) Å³, $Z = 4$, $D_x = 1.17$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 5.74$ cm⁻¹,

$F(000) = 704$, room temperature, $R = 0.056$, $wR = 0.069$ for 1820 reflections with $I \geq 3\sigma(I)$. Bond lengths and angles are in the usual range. The molecule is in an extended configuration, the benzene and the phenol planes forming a dihedral angle of 53.8 (1)°. Intramolecular hydrogen bonding was observed between O(2) and O(3) of the carbonyl and

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