The structure was solved by direct phase determination. The parameters of the complete structure could be refined by full-matrix anisotropic least squares. All phenyl rings were refined without any constraints. Values of distances and angles in the rings show no significant differences from those of an ideal benzene ring. All H-atom positions were calculated using a riding model, except for that involved in the molecular N—H···O hydrogen bond, which was found from the difference Fourier maps calculated at the end of the refinement process as a small positive electron density.

Data collection: *SDP* (Frenz, 1985). Cell refinement: *SDP*. Data reduction: *REDU*4 (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON*96 (Spek, 1996). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1241). Services for accessing these data are described at the back of the journal.

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N—H···π(pyrrole) Intermolecular Interactions in 1,4-Bis(di-2-pyrrolylmethyl)benzene

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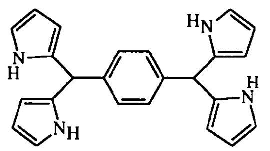
Abstract

The title compound, $C_{24}H_{22}N_4$, lies about an inversion centre which is at the centre of the 1,4-disubstituted benzene ring. Intermolecular N—H···π(pyrrole) interactions form a two-dimensional hydrogen-bonded network with shortest N···(pyrrole centroid) distances of 3.219 (3) and 3.324 (3) Å.

Comment

Dipyrromethane derivatives are useful building blocks in the synthesis of porphyrin model systems with direct approaches to *trans*-substituted porphyrins available by condensation of dipyrromethanes with aldehydes (Lee & Lindsey, 1994). The structural chemistry of these precursors has been underdeveloped, especially in relation to hydrogen-bonding studies. Interactions involving an aromatic π system as a donor or acceptor are well established (Hanton, Hunter & Purvis, 1992). We report herein the structure of 1,4-bis(di-2-pyrrolylmethyl)benzene, (I), where intermolecular N—H···π(pyrrole) interactions arise in a system with pyrrole N—H donors and only heteroaromatic pyrrole systems as acceptors.



(I)

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Molecule (I) lies about an inversion centre which is at the centre of the disubstituted benzene ring; a view of the molecule with our numbering scheme is presented in Fig. 1 and selected geometric dimensions are in Table 1. Bond lengths and angles are in accord with anticipated values (Orpen *et al.*, 1994). The two independent pyrrole rings are at angles of 79.4 (2) (N15 ring) and 67.6 (2)° (N25 ring) to the 1,4-benzene ring and are almost normal to one another [86.43 (9)°].

The pyrrole groups take part in intermolecular N—H···π(pyrrole) interactions, with each pyrrole acting as both an N—H donor and a π(pyrrole) acceptor. A view of the N—H···π(pyrrole) hydrogen-bonding process is given in Fig. 2. Molecule (I) associates about inversion centres, forming a one-dimensional chain along the *c* axis, with N25—H25···*Cg*1^{i} [*Cg*1 is the centroid of the N15 ring; symmetry code: (i) $1-x, 1-y, -z$] interactions (details in Table 2). N15—H15···*Cg*2^{ii} [*Cg*2 is the centroid of the N25 ring; symmetry code: (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$] interactions link the chains together along the **b** direction, to generate a two-dimensional network of (pyrrole)N—H···π(pyrrole) hydrogen bonds. Examination of the structure with *PLATON* (Spek, 1997*a*) showed that there were no solvent-accessible voids in the crystal lattice.

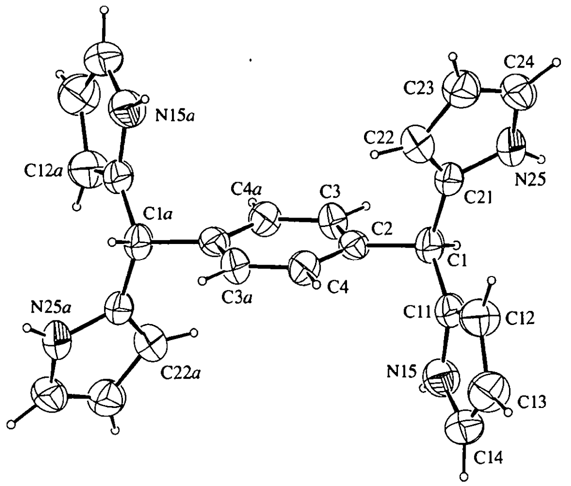

Fig. 1. View of (I) showing the atom-numbering scheme; suffix '*a*' refers to the equivalent position ($1-x, 1-y, 1-z$). Displacement ellipsoids are drawn at the 30% probability level.

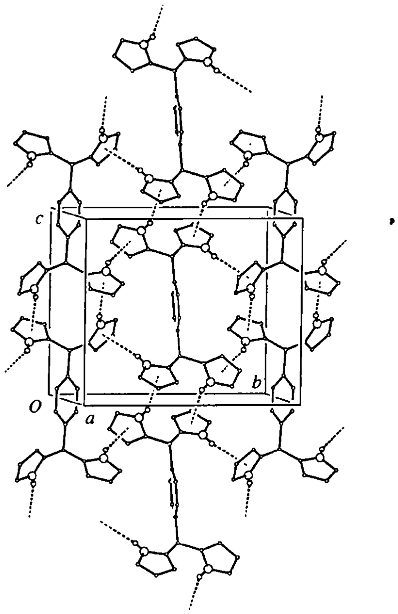

Fig. 2. A view of the N—H···π(pyrrole) interactions in the crystal structure. H atoms bonded to carbon have been omitted.

We have previously reported O—H···π(arene) interactions in 1,1,2-triphenylethanol (Ferguson *et al.*, 1994) and C—H···π(arene) interactions in a calix[5]-arene derivative (Gallagher *et al.*, 1994), where interesting crystal-packing effects are influenced largely by these interactions. Further studies are in progress on the hydrogen-bonding interactions of related porphyrin precursors.

Experimental

The title compound was prepared according to the literature method of Lee & Lindsey (1994), and recrystallized from cyclohexane.

Crystal data

$C_{24}H_{22}N_4$
$M_r = 366.46$
Monoclinic
$P2_1/c$
$a = 6.4806\,(12)$ Å
$b = 13.8928\,(15)$ Å
$c = 10.9886\,(9)$ Å
$\beta = 92.738\,(10)°$
$V = 988.2\,(2)$ Å^3
$Z = 2$
$D_x = 1.232$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.74–18.78°$
$\mu = 0.075$ mm^{-1}
$T = 294\,(1)$ K
Needle
0.28 × 0.14 × 0.10 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
2464 measured reflections
2256 independent reflections
789 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$

$\theta_{max} = 27.4°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity variation: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.104$
$S = 0.829$
2256 reflections
128 parameters

$\Delta\rho_{max} = 0.185$ e Å^{-3}
$\Delta\rho_{min} = -0.153$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0073 (13)

H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N15—C11	1.361 (3)	N25—C21	1.361 (3)
N15—C14	1.358 (3)	N25—C24	1.363 (3)
C11—C12	1.363 (3)	C21—C22	1.351 (3)
C12—C13	1.417 (4)	C22—C23	1.417 (3)
C13—C14	1.344 (3)	C23—C24	1.345 (4)
N15—C11—C1	122.1 (2)	N25—C21—C1	121.3 (2)
N15—C11—C12	106.6 (2)	N25—C21—C22	106.9 (2)
C1—C11—C12	131.3 (3)	C1—C21—C22	131.5 (2)
C11—C12—C13	107.7 (3)	C21—C22—C23	108.5 (2)
C11—N15—C14	110.4 (2)	C21—N25—C24	109.7 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N25—H25···*Cg*1^{i}†	0.86	2.42	3.219 (3)	154
N15—H15···*Cg*2^{ii}	0.86	2.52	3.324 (3)	156

Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

† *Cg*1 and *Cg*2 are the centroids of the N15 and N25 rings, respectively.

Molecule (I) crystallized in the monoclinic system: space group $P2_1/c$ from the systematic absences. H atoms were allowed for as riding atoms with N—H 0.86, and C—H 0.93 and 0.98 Å. At an intermediate stage in the analysis, the site occupancies of the atom pairs N15/C12 and N25/C22 were allowed to vary in order to check for possible N/C disorder; the occupancy factors obtained did not differ significantly from unity and accordingly in the final refinement cycles, no N/C disorder was allowed for. Difference Fourier maps in the plane of the pyrrole rings clearly supported the present atom designation.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD-4-PC Software*. Data reduction: *DATRD*2 in *NRCVAX*96 (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *NRCVAX*96 and *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *NRCVAX*96, *ORTEP*II (Johnson, 1976), *PLATON* (Spek, 1997*a*) and *PLUTON* (Spek, 1997*b*). Software used to prepare material for publication: *NRCVAX*96, *SHELXL*97 and *WordPerfect* macro *PRPCIF*97 (Ferguson, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1144). Services for accessing these data are described at the back of the journal.

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3-Methoxy-2,4,6-triphenylpyridine at 173 K

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Abstract

In the title compound, $C_{24}H_{19}NO$, the methoxy group attached at C3 avoids steric hindrance with the neighbouring phenyl rings by emerging almost orthogonally from the pyridine ring [C2—C3—O31—C31 −95.3 (2)°]. The phenyl rings show a disrotatory arrangement and the angles between them and the pyridyl ring range from 22.66 (6) to 47.91 (5)°, whereas these angles differ by only approximately 6° in 2,4,6-triphenylpyridine.

Comment

3-Methoxy-2,4,6-triphenylpyridine serves as a precursor for oxyl radicals of heterocycles. The synthesis proceeds from the title compound, (I), *via* the corresponding phenol to the triphenylpyridyloxyl (Teuber, Schütz & Bader, 1977). The crystal structure determination was performed to establish unambiguously the pattern of substitution of the heterocycle.

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