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.


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.

References
Stoe & Cie, Darmstadt, Germany.


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

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Abstract
The title compound, C24H22N4, 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···π(pyrrole centroid) distances of 3.219 (3) and 3.324 (3) Å.

Comment
Dipyrrromethane derivatives are useful building blocks in the synthesis of porphyrin model systems with direct approaches to trans-substituted porphyrins available by condensation of dipyrrromethanes 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.
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-..Tr(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-..Cg1 [Cg1 is the centroid of the N15 ring; symmetry code: (i) 1 - x, 1 - y, -z] interactions (details in Table 2). N15--H15-..Cg2 [Cg2 is the centroid of the N25 ring; symmetry code: (ii) 1 - x, y - ½, 1 - 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, 1997a) showed that there were no solvent-accessible voids in the crystal lattice.

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}  
Mr = 366.46  
Monoclinic  
P2_{1}/c  
Cell parameters from 25 reflections  
a = 6.4806 (12) Å  
b = 13.8928 (15) Å  
c = 10.9886 (9) Å  
\(\beta = 92.738 \, (10) ^{\circ}\)  
V = 988.2 (2) Å\(^{3}\)  
Z = 2  
\(D_{r} = 1.232 \, \text{Mg m}^{-3}\)  
\(D_{m} \) not measured

**Data collection**

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

**Refinement**

Refinement on \(F^{2}\)  
\(R(F^{2}) = 0.058\)  
\(wR(F^{2}) = 0.104\)  
\(S = 0.829\)  
2256 reflections  
128 parameters  
\(\Delta\rho_{max} = 0.185 \, \text{e} \, \text{Å}^{-3}\)  
\(\Delta\rho_{min} = -0.153 \, \text{e} \, \text{Å}^{-3}\)  
Extinction correction:  
SHELXL97  
Extinction coefficient:  
0.0073 (13)
Table 1. Selected geometric parameters (Å, °)

<table>
<thead>
<tr>
<th>Bond</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1--C11</td>
<td>1.361 (3)</td>
</tr>
<tr>
<td>C1--C12</td>
<td>1.363 (3)</td>
</tr>
<tr>
<td>C1--C13</td>
<td>1.364 (3)</td>
</tr>
<tr>
<td>C12--C13</td>
<td>1.417 (4)</td>
</tr>
<tr>
<td>C11--C12</td>
<td>1.363 (3)</td>
</tr>
<tr>
<td>C21--C22</td>
<td>1.351 (3)</td>
</tr>
<tr>
<td>C23--C24</td>
<td>1.345 (4)</td>
</tr>
<tr>
<td>C13--C14</td>
<td>1.344 (3)</td>
</tr>
<tr>
<td>C23--C24</td>
<td>1.345 (4)</td>
</tr>
<tr>
<td>N15--C14</td>
<td>1.358 (3)</td>
</tr>
<tr>
<td>N25--C24</td>
<td>1.363 (3)</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>D-H</th>
<th>D-A</th>
<th>D-A</th>
<th>D-H...A</th>
</tr>
</thead>
<tbody>
<tr>
<td>N25--H25...Cg11</td>
<td>0.86</td>
<td>2.42</td>
<td>3.219 (3)</td>
<td>154</td>
</tr>
<tr>
<td>N15--H15...Cg22</td>
<td>0.86</td>
<td>2.52</td>
<td>3.324 (3)</td>
<td>156</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) 1 - x, y - ½, ½ - z; (ii) 1 - x, 1 - y, -z; (iii) 1 - x, 1 - y, -z.
† Cg1 and Cg2 are the centroids of the N15 and N25 rings, respectively.

Molecule (I) crystallized in the monoclinic system: space group P21/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: SET4 and CELDIM in CAD-4-PC Software. Data reduction: DATR2D in NRCVAX96 (Gabe et al., 1989). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a). Program(s) used to refine structure: NRCVAX96 and SHELXL97 (Sheldrick, 1997b). Molecular graphics: NRCVAX96, ORTEPII (Johnson, 1976), PLATON (Spek, 1997a) and PLUTON (Spek, 1997b). Software used to prepare material for publication: NRCVAX96, SHELXL97 and WordPerfect macro PRPCIF97 (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.

References


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

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Abstract
In the title compound, C24H19NO, 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.